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Cleanrooms and associated controlled environments —

Part 13:
Cleaning of surfaces to achieve defined levels of cleanliness in terms of particle and chemical classifications

Salles propres et environnements maîtrisés apparentés —

Partie 13: Nettoyage des surfaces afin d'obtenir des niveaux de propreté par rapport aux classifications particulaire et chimique

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 209, *Cleanrooms and associated controlled environments*.

A list of all parts in the ISO 14644 series can be found on the ISO website.

Introduction

The term surface refers to the interface between two phases. For the purpose of this document, the surface is a solid. A "clean surface" is where one or more of the contamination categories (particles, chemical) are under control due to cleaning/decontamination. The degree of cleanliness is specified in the corresponding surface cleanliness classifications (see ISO 14644-9 and ISO 14644-10). Different cleaning methods are necessary depending on the degree of cleanliness (cleanliness class) required. This document gives guidance on the selection of cleaning methods to achieve specified cleanliness levels. For the selection procedure, the aspects of surface description, cleanliness specifications, types of contamination, cleaning techniques, material compatibility, and assessment methodology are taken into consideration. Most of the methods are suitable for removal of more than one contamination category at the same time; therefore, a common standard for the selection of a cleaning method for both particles, as well as chemical contamination, is needed.

Cleanrooms and associated controlled environments —

Part 13:
Cleaning of surfaces to achieve defined levels of cleanliness in terms of particle and chemical classifications

1 Scope

This document gives guidelines for cleaning to a specified degree on cleanroom surfaces, surfaces of equipment in a cleanroom and surfaces of materials in a cleanroom. Under consideration are all surfaces (external or internal) that are of interest. It provides guidance on the assessment of cleaning methods for achieving the required surface cleanliness by particle concentration (SCP) and surface cleanliness by chemical concentration (SCC) classes and which techniques should be considered to achieve these specified levels.

The appropriateness of cleaning techniques will make reference to the cleanliness classes and associated test methods found in ISO 14644-9 and ISO 14644-10.

The following matters of general guidance will be provided:

— expected surface cleanliness levels;

— suitability of cleaning methods;

— compatibility of surfaces with the cleaning technique;

— assessment of cleaning appropriateness.

The following will be excluded from this document:

— classification of cleaning methods;

— product produced within a cleanroom;

— specific surface-related cleaning methods;

— detailed description of cleaning mechanisms, methods and procedures of various cleaning methods;

— detailed material characteristics;

— description of damage mechanisms by cleaning processes and time-dependent effects;

— references to interactive bonding forces between contaminants and surfaces or generation processes that are usually time-dependent and process-dependent;

— other characteristics of particles such as electrostatic charge, ionic charges, etc.;

— chemical reactions between molecular contaminants and surfaces;

— microbiological aspects of surface cleanliness;

— radioactive aspects of contamination;

— health and safety considerations;

— environmental aspects such as waste disposal, emissions, etc.;

- selection and use of statistical methods.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 14644-8, *Cleanrooms and associated controlled environments — Part 8: Classification of air cleanliness by chemical concentration (ACC)*

ISO 14644-9, *Cleanrooms and associated controlled environments — Part 9: Classification of surface cleanliness by particle concentration*

ISO 14644-10, *Cleanrooms and associated controlled environments — Part 10: Classification of surface cleanliness by chemical concentration*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 14644-9, ISO 14644-10 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp

3.1
cleanliness
<of a solid surface>condition of a solid surface where the amount of *contamination* (3.4) (particle, chemical) is controlled to a specific level

3.2
cleaning appropriateness
relation between the required *cleanliness* (3.1) and the accomplished cleanliness under controlled conditions

Note 1 to entry: In some languages, the term cleaning efficacy is used to indicate cleaning appropriateness.

Note 2 to entry: In case of real operational conditions or monitoring, the term cleaning effectiveness is used.

3.3
cleaning efficiency
fraction of specific contaminants removed from a surface by a cleaning process

Note 1 to entry: The fraction is determined by the accomplished surface cleanliness in respect to the initial surface cleanliness.

3.4
contamination
unwanted matter in an undesirable location

3.5
particle
minute piece of matter with defined physical boundaries

[SOURCE: ISO 14644-1:2015, 3.2.1]

3.6

particle contamination

particles (3.5) having the potential to affect the process, the product, the personnel or the facilities

3.7

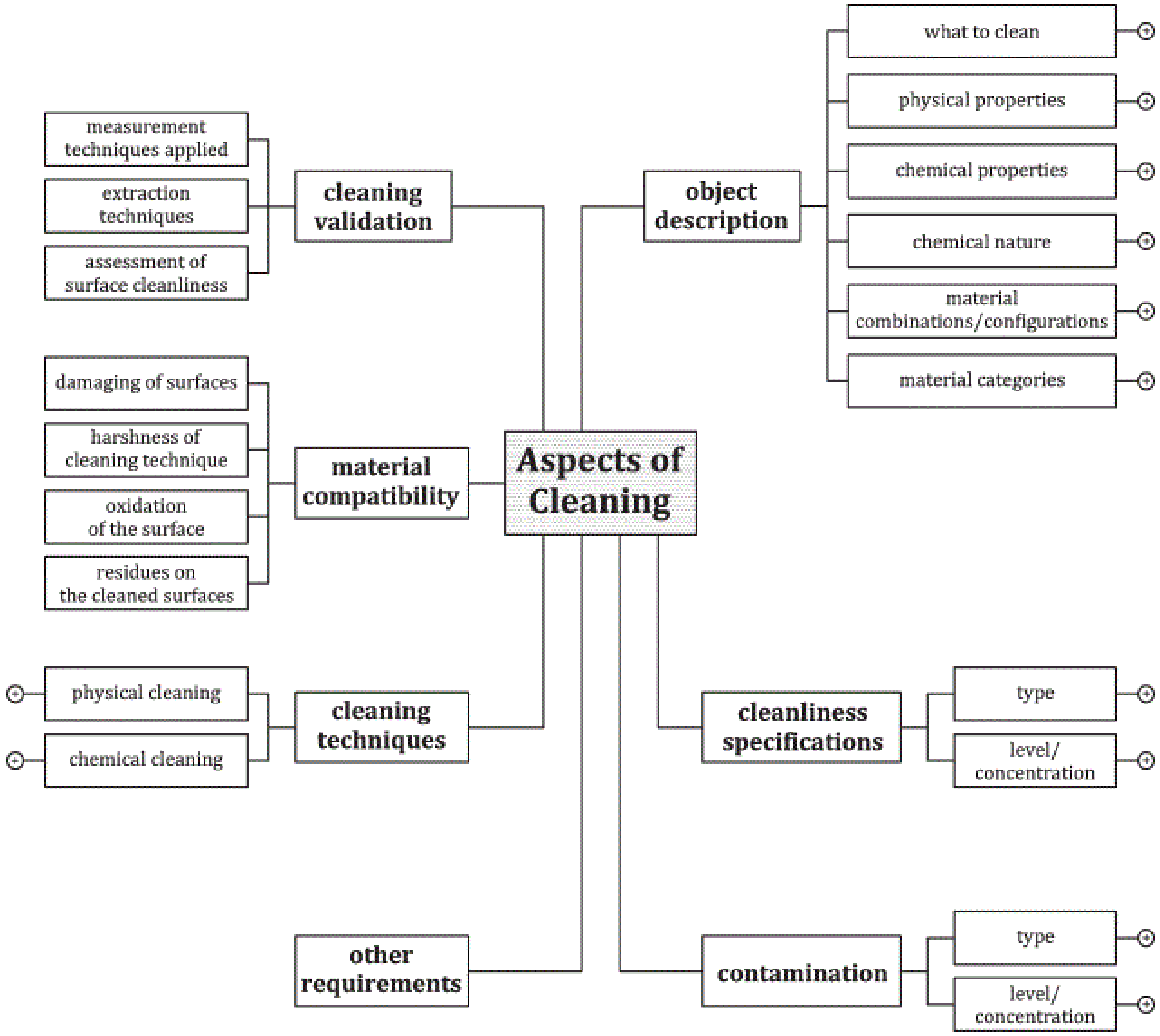
particle size

diameter of a sphere that produces a response, by a given particle-sizing instrument, that is equivalent to the response produced by the *particle* (3.5) being measured

4 General methodology

4.1 Overview

Multiple aspects need to be considered for cleaning. Figure 1 provides an overview of the factors that contribute to the suitability of cleaning methods to achieve a defined level of surface cleanliness. For more details, see Annex A.



Key

\+ more details in Figure A.1

Figure 1 — Overview of cleaning aspects (non-exhaustive)

4.2 Methodology

Appropriateness of a cleaning technique for an application depends on many factors. For complex objects, it is advised to follow the sequence described in this clause and in the decision tree (see Figure 2). By using this procedure, it can be assured that all important issues are covered. For simple objects or surfaces, deviations from the sequence are allowable, as long as the critical information is documented.

The approach starts with describing the object that has to be cleaned. The description should cover, among others, composition of materials, chemical characteristics, surface finish and shape factors like geometrical complexity and size (step 1). In the second step, the objective of the cleaning procedure is to be specified in terms of the desired cleanliness of the object. To have a starting point, the initial contamination level should be assessed (step 3) and other requirements shall be listed (step 4). Based on type of contaminants and the required removal efficiency, a cleaning technique or a combination of techniques can be selected (step 5). The cleaning methodology shall be checked against the materials from the first step in order to avoid material compatibility issues (step 6). At the last step, a validation method shall be carried out (step 7). The validation shall at least comprise methods for determination of the cleaning performance and material compatibility. The performance of the cleaning shall be checked against the specification.

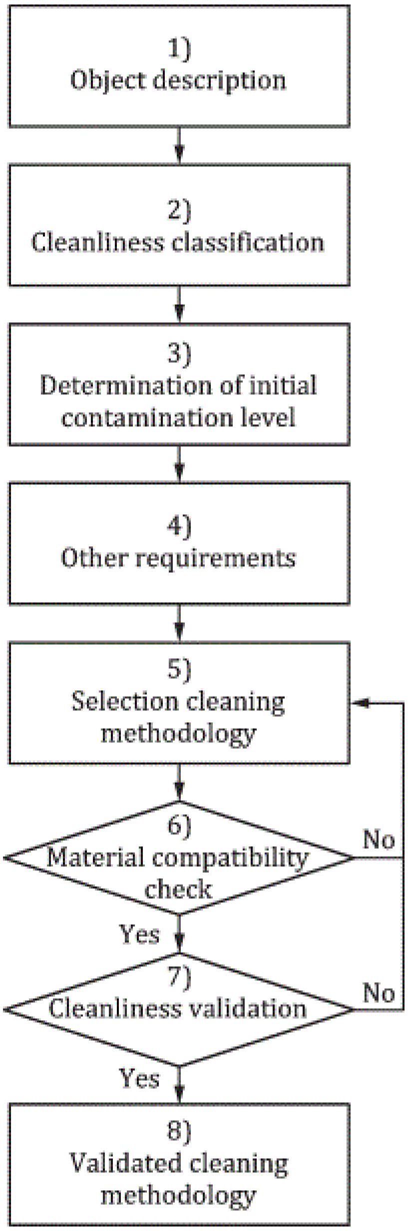


Figure 2 — Decision tree

5 Object description

In the object description, the following aspects shall be considered.

Physical properties of the object:

- physical dimensions;
- form/shape/complexity of the object;
- critical surfaces.

A general description of the object shall be given. Size, shape, complexity and identification of the critical surfaces set many boundary conditions for a cleaning method.

Surface(s):

- material composition of the object surface(s);
- complexity;
- materials/critical materials (sensitive to cleaning agents);
- thin/atomic surface layers (e.g. protective layers);
- other physical properties (e.g. electrostatic).

The composition of the object could be simple (one or more similar material(s)) or a system consisting of a range of different materials (e.g. metal/plastic/glass composition). Each individual material should be taken into account when choosing a cleaning method. Some combinations of materials can be very difficult to clean as one cleaning method may be suitable for one material but not the other. The selection of the cleaning method shall be the best compromise between minimal damage of the materials at maximum cleaning efficiency.

Chemical properties of the surface(s):

- chemical composition;
- state of the surface (hydrophobic, hydrophilic, lypophobic, lipophilic, etc.);
- energetic state of the surface (zeta potential).

Physical and chemical properties of a surface have a large influence on the choice of a cleaning method. Choice of a method shall take into account all involved materials/chemical composition of the surface(s) and the combination of materials undergoing cleaning.

The activity of the surface is determined by the chemical configuration of the final atomic layer. This is an important parameter in determining the choice of method. These layers may be hydrophilic (water wettable-lipophobic) or hydrophobic (water repellent but oil-wettable-lipophilic). The cleaning method used can influence the surface activity. The surface activity also influences the electrostatic properties of a material — a hydrophilic surface will have fewer tendencies to electrically charge.

Morphology:

Cleaning is influenced by the morphological aspects of a surface such as shape, size, structure, surface roughness or porosity. Morphological aspects may further complicate cleaning due to the accessibility of the surfaces to be cleaned and to the retention of cleaning agents and materials used for cleaning.

Object specific requirements:

- intended use of the object;

- environmental conditions, both pre- and post-cleaning;
- critical surfaces — surfaces with either high importance in the application or sensitive to cleaning.

6 Cleanliness specifications

The rationale of determining appropriateness of the cleaning method should include the contaminants of concern, as well as acceptable levels of contamination for the application.

The required cleanliness shall be specified in terms of particle concentration according to ISO 14644-9 and/or in terms of the concentration of a specific group of chemicals according to ISO 14644-10.

The specification of the targeted cleanliness class can be determined in various ways:

- cleanliness class required by customer;
- common cleanliness class required for similar objects;
- by performing an analysis on the impact of contamination of the surface in the (future) function of a product or process where the cleaned surface will be used;
- by performing tests or simulations with various cleanliness classes of the considered surface and/or;
- by performing an analysis of the contamination that caused failure or quality loss.

7 Determination of initial contamination level

7.1 General

To select the cleaning procedure for achieving a required surface cleanliness level, the initial surface cleanliness shall be established. The initial surface cleanliness by chemical and/or particle concentration shall be determined for the surfaces of interest. This can be determined qualitatively or quantitatively.

Qualitative evaluation is a non-numerical assessment (e.g. a visual inspection).

Where the quantitative surface cleanliness is determined, it can be related to the efficiency of the cleaning procedure in respect to the required surface cleanliness by particle concentration and/or chemical concentration. The quantified contamination (particle and/or chemical) shall be related to the area of the representative surface. This evaluation allows determination of the initial cleanliness class.

7.2 Determination of initial contamination level with respect to particle concentration

The initial surface cleanliness by particle concentration shall be determined for the different surfaces. Depending on the required cleanliness class, a qualitative assessment of the initial cleanliness level may be sufficient. For quantitative assessment, the number and size of particles shall be characterized by a measurement method as described in Clause 12. The surface cleanliness classification as outlined in ISO 14644-9 shall be applied.

The quantified particle concentration shall be related to the area of the representative surface. This evaluation allows determination of the initial cleanliness class.

7.3 Determination of initial contamination level with respect to chemical concentration

The initial surface cleanliness by chemical concentration shall be determined for the different surfaces. Depending on the specific application, a qualitative assessment (e.g. water break test) of the initial cleanliness level might be sufficient. For quantitative assessment of the mass and nature of the chemical contaminants, a measurement technique according to Clause 12 and ISO 14644-10 shall be applicable.

The quantified chemical contamination shall be related to the area of the representative surface. This evaluation allows determination of the initial cleanliness class.

8 Other requirements

In addition to requirements for cleaning appropriateness and product integrity, many other elements influence the applicability of the cleaning method. In step 5 of the decision tree (see Figure 2), these other aspects shall be listed and requirements shall be defined. The contents of these requirements are strongly related to the object or surface and its application.

Typical examples for other requirements are time available for cleaning, number of items that will be cleaned, available footprint, waste, investment and labour costs, level of operator skill and environmental aspects.

9 Selection of a cleaning methodology

9.1 Selection procedure

Requirements for cleaning are defined in Clause 5 to Clause 8. These requirements shall be considered during the selection process for the cleaning methodology to ensure the greatest uniformity between the requirements and the method.

The final decision of the choice of cleaning methodology shall be based on a combination of all aspects to find the technically and financially optimal, most efficient solution. The customer, the clean technology supplier and the process owner of the surface/object/component to be cleaned should be involved in this selection process. The decision optimizes the various parameters and shall assure specified surface quality. Table B.1 and Table B.2 can aid the selection process.

9.2 Cleaning methodologies

9.2.1 Cleaning method

A cleaning methodology consists of a cleaning technique (e.g. wiping, plasma cleaning and ultrasonic cleaning) and a cleaning process. The cleaning process is linked to the cleaning techniques and involves the amount of mechanical energy, chemical energy, temperature and process time. By using a balanced combination of these four process parameters, the optimum cleaning result can be achieved. Final cleanliness can be limited by substrate damage and/or selection criteria defined in Clause 8.

The required cleanliness level and the initial cleanliness level in particle and/or chemical concentration lead to a selection of cleaning methods. In case the initial cleanliness class is high and the required cleanliness class is low, a combination of methods may be required.

9.2.2 Categories of cleaning techniques

The cleaning techniques can be divided depending on the major cleaning mechanism into physical and chemical cleaning which additionally could be divided into wet and dry cleaning techniques. Whether a process is categorized as physical/chemical or as wet/dry depends on the dominant mechanism used for the cleaning. A non-exhaustive list of physical cleaning techniques can be found in B.2 (mechanical cleaning), B.3 (fluidic cleaning), and B.4 (blasting cleaning techniques). A list of chemical cleaning techniques can be found in B.5 (chemical cleaning). References to the categories of cleaning techniques are given in Table 1.

Table 1 — Categorization of cleaning techniques as described in Annex B

Cleaning technique	Physical	Chemical
Dry	B.2, B.4 dry	B.5 dry
Wet	B.3, B.4 wet	B.5 wet

The numbers refer to subclauses within Annex B that provide a brief description of the major cleaning techniques.

Table B.1 and Table B.2 support the choice of an appropriate cleaning technique (physical/chemical) that fulfils the cleanliness specifications. With the help of feasibility tests on material compatibility (see Clause 10) and empirical values/evaluation, the best suitable technique shall be chosen for validation (see Clause 11).

9.3 Cleaning process

In Table B.1 and Table B.2, an overview is given on the working ranges for different cleaning techniques. In these ranges, optimum process conditions are assumed. Since material properties and other requirements might force use of less than optimal conditions, the actual cleaning efficiency and appropriateness might be lower. The process settings shall be defined either by an experimental investigation or from experience.

10 Material compatibility check

The compatibility of materials with cleaning agents shall be considered. These cleaning agents (e.g. chemicals, solvents, high pressure gas or liquid) shall be selected with respect to their compatibility with the materials or items to be cleaned and their efficiency in removing different kinds of contaminants: particle or chemical contaminants.

Detrimental effects on surfaces can be caused by the cleaning agents and/or the cleaning technique. The chemical nature of the materials of the surface — individually or in combination depending on the composition of the surface — should be evaluated technically.

The following describes such direct, indirect and long duration effects.

— Direct effects are changes of intrinsic material properties (physical and/or chemical) as a consequence of the interaction with a process parameter from the cleaning technique: chemical nature of a solvent, time of exposure and temperature.

EXAMPLE 1 The change of the chemical surface structure may strongly change the physical behaviour. Change of the hydrophilic character of the last atomic layer to hydrophobic through the cleaning process may change the physical behaviour of the cleaned surface, i.e. wettability and electrostatic charge. Hydrophobic surface is not wettable and is easily charged which may attract particles.

EXAMPLE 2 Change in surface roughness.

— Indirect effects can be caused by different physical-chemical mechanisms as a consequence of secondary interactions (e.g. chemical reactions with specific compounds of the substrate) or as an interaction with a non-process parameter (post-degradation or oxidation by O_2 of air, deposits of residues as a new contamination).

NOTE An indirect effect might not be observed immediately.

— Long duration effects result from a slow process (e.g. induced corrosion from chemical conversion after chemical cleaning, ageing and weakening).

Annex C gives an overview of compatibility with chemical solvents; it is a non-exhaustive list. In the case of a lack of information for a specific cleaning solvent, the chemical resistance of the material shall be assessed through an appropriate test.

11 Cleaning validation

11.1 General

To assess the suitability of a selected cleaning procedure, the cleaning efficiency and the cleaning appropriateness should be considered.

For the validation of a cleaning process to reach the required surface cleanliness with respect to particle concentration and/or chemical concentration, the achieved surface cleanliness SCP and/or SCC levels should be determined.

Other performance-related aspects, such as repeatability, reproducibility and operator influences, shall also be considered. In Clause 8, a set of requirements are defined that are not related to cleaning performance. Although these requirements are taken into account during the selection of the cleaning method, the validation process shall verify that these requirements are fulfilled.

Figure 3 and Figure 4 are depictions of the terms cleaning efficiency and cleaning appropriateness.

Cleaning efficiency = 1 − final cleanliness level/initial cleanliness level (expressed as a percentage)

Cleaning appropriateness = required cleanliness level/final cleanliness level

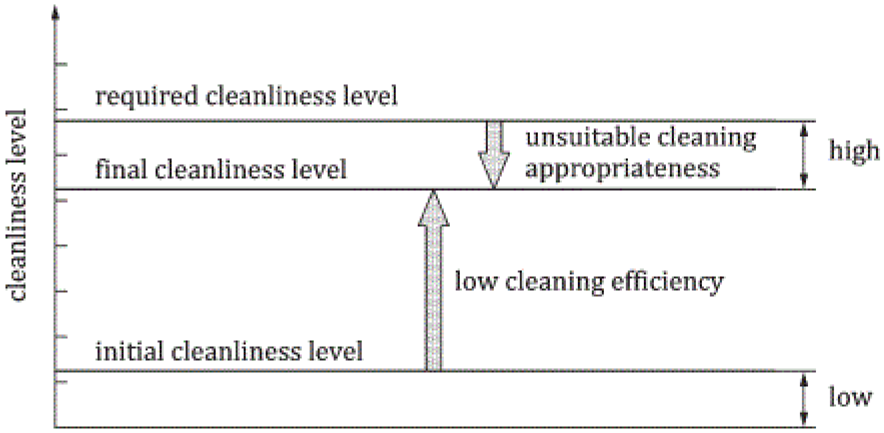


Figure 3 — Example for a method with low cleaning efficiency leading to unsuitable cleaning appropriateness

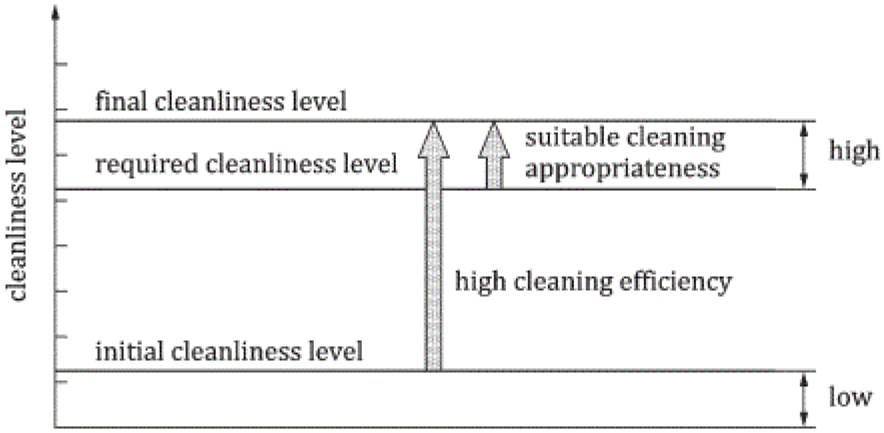


Figure 4 — Example for a method with high cleaning efficiency leading to suitable cleaning appropriateness

11.2 Cleaning efficiency

11.2.1 Evaluation

For evaluation of the cleaning efficiency, the initial surface cleanliness level and final cleanliness level shall be determined.

11.2.2 Cleaning efficiency with respect to particle concentration

The cleaning efficiency is calculated by Formula (1):

$$C_{\mathrm{eff}} = 1 - \frac{C_{\mathrm{SCP};D}^{\mathrm{fin}}}{C_{\mathrm{SCP};D}^{\mathrm{ini}}} \tag{1}$$

where

D is the particle size, in micrometres (µm);

$C_{\mathrm{SCP};D}^{\mathrm{ini}}$ is the initial surface concentration, in particles per square metre of surface, of particles that are equal to or larger than the considered particle size D in µm;

$C_{\mathrm{SCP};D}^{\mathrm{fin}}$ is the final surface concentration, in particles per square metre of surface, of particles that are equal to or larger than the considered particle size D in µm.

In accordance with ISO 14644-9, the relation with the surface cleanliness class is given by Formula (2):

$$C_{\mathrm{SCP};D} = k\,\frac{10^{\mathrm{SCP};N}}{D} \tag{2}$$

where

$C_{\mathrm{SCP};D}$ is the maximum permitted total surface concentration, in particles per square metre of surface, of particles that are equal to or larger than the considered particle size;

$\mathrm{SCP};N$ is the SCP classification number;

D is the considered particle size, in micrometres;

k is the constant 1, in micrometres.

Combining Formula (1) and Formula (2) gives the cleaning efficiency in terms of SCP, in Formula (3):

$$C_{\mathrm{eff}} = 1 - \frac{k\,\dfrac{10^{\mathrm{SCP};N_{\mathrm{fin}}}}{D}}{k\,\dfrac{10^{\mathrm{SCP};N_{\mathrm{ini}}}}{D}} = 1 - \frac{10^{\mathrm{SCP};N_{\mathrm{fin}}}}{10^{\mathrm{SCP};N_{\mathrm{ini}}}} = 1 - 10^{\left(\mathrm{SCP};N_{\mathrm{fin}} - \mathrm{SCP};N_{\mathrm{ini}}\right)} \tag{3}$$

where

D is the particle size, in micrometres;

$\mathrm{SCP};N$ is the SCP classification number;

$\mathrm{SCP};N_{\mathrm{fin}}$ is the SCP classification number of the final surface cleanliness of particles;

$\mathrm{SCP};N_{\mathrm{ini}}$ is the SCP classification number of the initial surface cleanliness of particles.

NOTE The cleaning efficiency can also be expressed as a percentage (%).

EXAMPLE In this example, the smallest particle size that could be measured with the applied measurement method is 5 µm. Therefore, the concentration of the number of particles ≥5 µm is used.

The initial concentration of particles ≥5 µm per m² $C_{\text{SCP;5}}^{\text{ini}}$ is 600 000. Therefore, the SCP;N_{ini} = lg (600 000 × 5) = 6,5.

The initial surface cleanliness class number SCP;N_{ini} = 6,5 for particles equal to or larger than 5 µm.

After cleaning, the final concentration of particles ≥5 µm per m² $C_{\text{SCP;5}}^{\text{fin}}$ is 1 000. Therefore, the SCP;N_{fin} = lg (1 000 × 5) = 3,7.

The final surface cleanliness class number SCP;N_{fin} = 3,7 for particles equal to or larger than 5 µm.

The cleaning efficiency in terms of particle concentration = 1 − (1 000)/(600 000) = 1 − 1/600 = 0,998 or 99,8 % for particles ≥5 µm.

Similarly, the cleaning efficiency in respect to the SCP class numbers = 1 − $10^{(3,7-6,5)}$ = 1 − $10^{-2,8}$ = 0,998 4 or 99,8 % for particles ≥5 µm.

11.2.3 Cleaning efficiency with respect to chemical contamination

The cleaning efficiency is calculated by Formula (4):

$$C_{\text{eff}} = 1 - \frac{C_{\text{SCC}}^{\text{fin}}}{C_{\text{SCC}}^{\text{ini}}} \tag{4}$$

where

$C_{\text{SCC}}^{\text{ini}}$ is the initial concentration of the considered chemical (g/m²);

$C_{\text{SCC}}^{\text{fin}}$ is the final concentration of the considered chemical (g/m²).

According to ISO 14644-10, the relation with the surface cleanliness class by chemical concentration (SCC) is given by Formula (5):

$$C_{\text{SCC}} = 10^{\text{SCC};N} \tag{5}$$

where

SCC;N is the surface cleanliness of chemical concentration by classification number.

Combining Formula (4) and Formula (5) gives the cleaning efficiency in terms of SCC, in Formula (6):

$$C_{\text{eff}} = 1 - \frac{10^{\text{SCC};N_{\text{fin}}}}{10^{\text{SCC};N_{\text{ini}}}} = 1 - 10^{(\text{SCC};N_{\text{fin}} - \text{SCC};N_{\text{ini}})} \tag{6}$$

NOTE The cleaning efficiency can also be expressed as a percentage (%).

EXAMPLE The initial concentration of the hydrocarbons is 50 mg per m^2 for hydrocarbons.

Therefore, the

$$\text{SCC};N_{\text{ini}} = \lg\left(50 \times 10^{-3}\right) = -1{,}3$$

for hydrocarbons.

The final concentration of the hydrocarbons is 10 µg per m^2.

Therefore, the

$$\text{SCC};N_{\text{fin}} = \lg\left(10 \times 10^{-6}\right) = -5{,}0 .$$

The cleaning efficiency in terms of chemical concentration =

$1 - 10 \times 10^{-6}/50 \times 1\,000 \times 10^{-6} = 1 - 10/50\,000 = 0{,}999\,8$ or 99,98 % for hydrocarbons.

Similarly, the cleaning efficiency in respect to the SCC classes = $1 - 10^{(-5{,}0\,+\,1{,}3)} = 1 - 10^{-3{,}7} = 0{,}999\,8$ or 99,98 % for hydrocarbons.

11.3 Cleaning appropriateness

11.3.1 Assessment

For evaluation of the cleaning appropriateness, the final cleanliness level obtained shall be determined and compared to the required surface cleanliness.

11.3.2 Cleaning appropriateness with respect to particle concentration

The cleaning appropriateness is calculated by Formula (7):

$$C_{\text{app}} = \frac{C_{\text{SCP};D}^{\text{requ}}}{C_{\text{SCP};D}^{\text{fin}}} \tag{7}$$

where

$C_{\text{SCP};D}^{\text{requ}}$ is the required concentration of particles equal to or larger than D µm;

$C_{\text{SCP};D}^{\text{fin}}$ is the final particle concentration obtained of particles equal to or larger than D µm.

D is the particle size, in micrometres.

According to ISO 14644-9, the relation with the surface cleanliness class is given by Formula (8):

$$C_{\text{SCP};D} = k\,\frac{10^{\text{SCP};N}}{D} \tag{8}$$

where

$C_{\text{SCP};D}$ is the concentration of particles equal to or larger than D µm;

D is the particle size, in micrometres;

SCP;N is the SCP classification number.

Combining Formula (7) and Formula (8) gives the cleaning appropriateness in terms of SCP class numbers, in Formula (9):

$$C_{\text{app}} = \frac{10^{\text{SCP};N_{\text{requ}}}}{10^{\text{SCP};N_{\text{fin}}}} = 10^{\left(\text{SCP};N_{\text{requ}} - \text{SCP};N_{\text{fin}}\right)} \tag{9}$$

If the cleaning appropriateness is less than 1, the chosen cleaning procedure was not suitable.

If the cleaning appropriateness is equal or greater than 1, the chosen cleaning procedure was suitable and results in a surface at the required cleanliness class.

NOTE The cleaning appropriateness can also be expressed as a percentage (%).

EXAMPLE In this example, the smallest particle size that could be measured with the applied measurement method is 10 µm. Therefore, the concentration of the number of particles ≥ 10 µm is used.

The required SCP;N_{requ} = 4,0 for particles equal to or larger than 10 µm.

This means that $C_{\text{SCP};10}^{\text{requ}}$ (10 µm) = 10 000/10 µm = 1 000 particles/m².

The final concentration of particles obtained ≥ 10 µm $C_{\text{SCP};10}^{\text{fin}}$ is 1 250 particles/m².

Cleaning appropriateness = $C_{\text{SCP};10}^{\text{requ}} / C_{\text{SCP};10}^{\text{fin}}$ = 1 000/1 250 = 0,8 → 80 % for particles ≥10 µm.

In terms of SCP:

$\text{SCP};N_{\text{fin}} = \lg\left(1\,250 \times 10\right) = 4{,}1$.

The cleaning appropriateness = $10^{(4{,}0 - 4{,}1)} = 10^{-0{,}1}$ = 0,8 or 80 %.

The cleaning appropriateness does not meet the requirements.

11.3.3 Cleaning appropriateness with respect to chemical concentration

The cleaning appropriateness is the ratio of the required surface cleanliness by chemical concentration under controlled conditions to the (final) chemical concentration after cleaning, given by Formula (10):

$$C_{\text{app}} = \frac{C_{\text{SCC}}^{\text{requ}}}{C_{\text{SCC}}^{\text{fin}}} \tag{10}$$

where

$C_{\text{SCC}}^{\text{requ}}$ is the required maximum concentration of the considered chemical (g/m²);

$C_{\text{SCC}}^{\text{fin}}$ is the final concentration of the considered chemical (g/m²).

According to ISO 14644-10, the relation with the surface cleanliness class by chemical concentration (SCC) is given by Formula (11):

$$C_{\text{SCC}} = 10^{\text{SCC};N} \tag{11}$$

where

C_{SCC} is the maximum allowable concentration of the specified chemical substance or group of substances, expressed in g/m²;

SCC;N is the common logarithm index of concentration C_{SCC}.

Combining both Formula (10) and Formula (11) gives the cleaning appropriateness in terms of SCC, in Formula (12):

$$C_{\text{app}} = \frac{C_{\text{SCC}}^{\text{requ}}}{C_{\text{SCC}}^{\text{fin}}} = 10^{\left(C_{\text{SCC}}^{\text{requ}} - C_{\text{SCC}}^{\text{fin}}\right)} \tag{12}$$

where

$C_{\text{SCC}}^{\text{requ}}$ is the required maximum concentration of the considered chemical (g/m^2);

$C_{\text{SCC}}^{\text{fin}}$ is the final concentration of the considered chemical (g/m^2).

If the cleaning appropriateness is smaller than 1, the chosen cleaning procedure was not appropriate. If the cleaning appropriateness is equal to or greater than 1, the chosen cleaning procedure was appropriate and results in a surface at the required cleanliness class.

NOTE The cleaning appropriateness can also be expressed as a percentage (%).

EXAMPLE The required SCC;$N_{\text{requ}} = -4{,}0$ for hydrocarbons. SCC;$N_{\text{requ}} = 10^{-4}$ g/m^2 = 100 µg/m^2.

The resulting concentration of the hydrocarbons is 70 µg per m^2.

Cleaning appropriateness = 100/70 = 1,43 is 143 %.

In terms of SCC class numbers:

$$\text{SCC};N_{\text{requ}} = \lg\left(70 \times 10^{-6}\right) = -4{,}15\,.$$

The cleaning appropriateness = $10^{(-4{,}0\,+\,4{,}15)} = 10^{0{,}15} = 1{,}41$ is 141 %.

The cleaning appropriateness does meet the requirements.

12 Measurement methods

12.1 General

The particle concentration and/or concentration of chemicals on a cleaned surface can be measured in a direct or indirect way. Direct surface cleanliness measurement may be appropriate in situations where the surface is accessible. The surface under study shall be brought to the measurement instrumentation or the instrumentation shall be brought to the surface.

The area of the surface selected for measurement shall be both representative of the entire area being assessed and the level particle and/or chemical contamination present.

The selected surface cleanliness measurement method can be applied to determine the initial and/or final surface cleanliness class.

Applicable measurement methods are described in ISO 14644-9 and ISO 14644-10.

For further information on measurement methods, see Annex D.

12.2 Direct measurement methods

12.2.1 General

A direct measurement method determines the surface cleanliness with respect to particle concentration or chemical concentration on the surface of interest of the object that has been cleaned. The resulting concentration of particles or the chemical of interest shall be expressed as either concentrations or surface cleanliness classes (SCP and SCC).

12.2.2 Direct measurement methods with respect to SCP

Count the number of particles >D per surface area. For D, a set of different size bins (D_i and D_{i+1}) could be selected. The smallest size D_1 shall be equal to or smaller than the particle size of interest.

By using a particular magnification by a magnifying glass or (stereo) microscope, smaller particle sizes can be counted, but the field of view will be also smaller. When the field of view is too small, the surface may need to be scanned to measure a representative surface area. A representative area is determined by the area that may contain at least 20 particles of the size of interest within the intended SCP class [see Formula (13)].

$$A \geq 20 \times \frac{D}{10^{SCP}} \qquad (13)$$

12.2.3 Direct measurement methods with respect to SCC

Use a spectroscopic or advanced surface analysis technique to measure the chemical contamination of interest on the surface.

In an enclosed heating or vacuum system, the SCC can be determined by measuring the outgassing of the surface using mass spectroscopy and associated residual gas analysis techniques.

In some cases, a part of the surface could be removed from the object to fit into an advanced measurement system.

12.3 Indirect measurement methods

12.3.1 General

An indirect method should be used when the surface to be measured cannot be reached by, is too small, or too large for the available measurement system.

12.3.2 Indirect measurement methods with respect to SCP

For this method, particles should be removed from the surface into either another medium (air, liquid) or onto another surface.

The transfer efficiency shall be taken into account and will depend on the transfer method, particle size and surface properties.

For particles suspended in a liquid or a gas, either a filtration method with subsequent microscopic analysis or discrete particle counting method can be used. For transfer surfaces, a direct measurement method can be used.

Count the number of particles >D per surface area. For D, a set of different size bins (D_i and D_{i+1}) could be selected. The smallest size D_1 shall be equal to or smaller than the particle size of interest.

To determine the original surface contamination, the measured number of particles should be divided by the surface area from which the particles are removed.

A smaller particle size can be counted by using a higher magnification. However, the field of view will be also smaller. If the field of view is too small, the surface may need to be scanned to measure a representative surface area. A representative area is determined by an area that may contain at least 20 particles of the size of interest of the intended SCP class [see Formula (11)].

12.3.3 Indirect measurement methods with respect to SCC

To measure the concentration of chemicals of interest on the surface of an object, the chemicals can be extracted by the chemical contamination being dissolved into a solvent. The solvent containing the extracted chemicals can then be analysed by an appropriate chemical analysis technique. In a heating or vacuum system, the chemical contaminants can be concentrated in a sorbent and measured by an appropriate chemical analysis technique.

The transfer efficiency should be taken into account and depends on the transfer method, the chemicals of interest and the surface properties.

The mass of the chemicals of interest shall be divided by the surface area from which the chemicals are removed to determine the original concentration.

13 Documentation

The results from each step in the decision tree (see Figure 2) shall be recorded and submitted as a comprehensive report, along with a statement of compliance or non-compliance with the specified goals, as described in Clause 5 to Clause 12. For simple cases, steps may be combined and reported in one document.

The report shall include the following:

a) the name and address of the executive organization;

b) the staff identification and the date on which tests were performed or decisions were made;

c) a reference to this document, i.e. ISO 14644-13:2017;

d) the specified designation criteria for the object;

e) the boundary conditions and assumptions;

f) the applied methods and procedures, e.g. ISO 14644-9 for surface particle cleanliness and ISO 14644-8 for classification air cleanliness by chemical concentration and ISO 14644-10 surface cleanliness by chemical concentration;

g) the measuring instruments applied and proof of calibration;

h) the results, either from assessments/selection procedures or experimental results. For the results on cleaning validation, the documentation requirements of ISO 14644-9 and ISO 14644-8 and/or ISO 14644-10 shall be followed;

i) a transfer statement for starting the next step.

NOTE In the decision tree (see Figure 2) assessments, selection procedures and experimental studies are involved. Therefore, the text refers to executive organization and staff identification instead of the often used terms testing organization and operator identification.

Annex A
(informative)

Aspects of cleaning

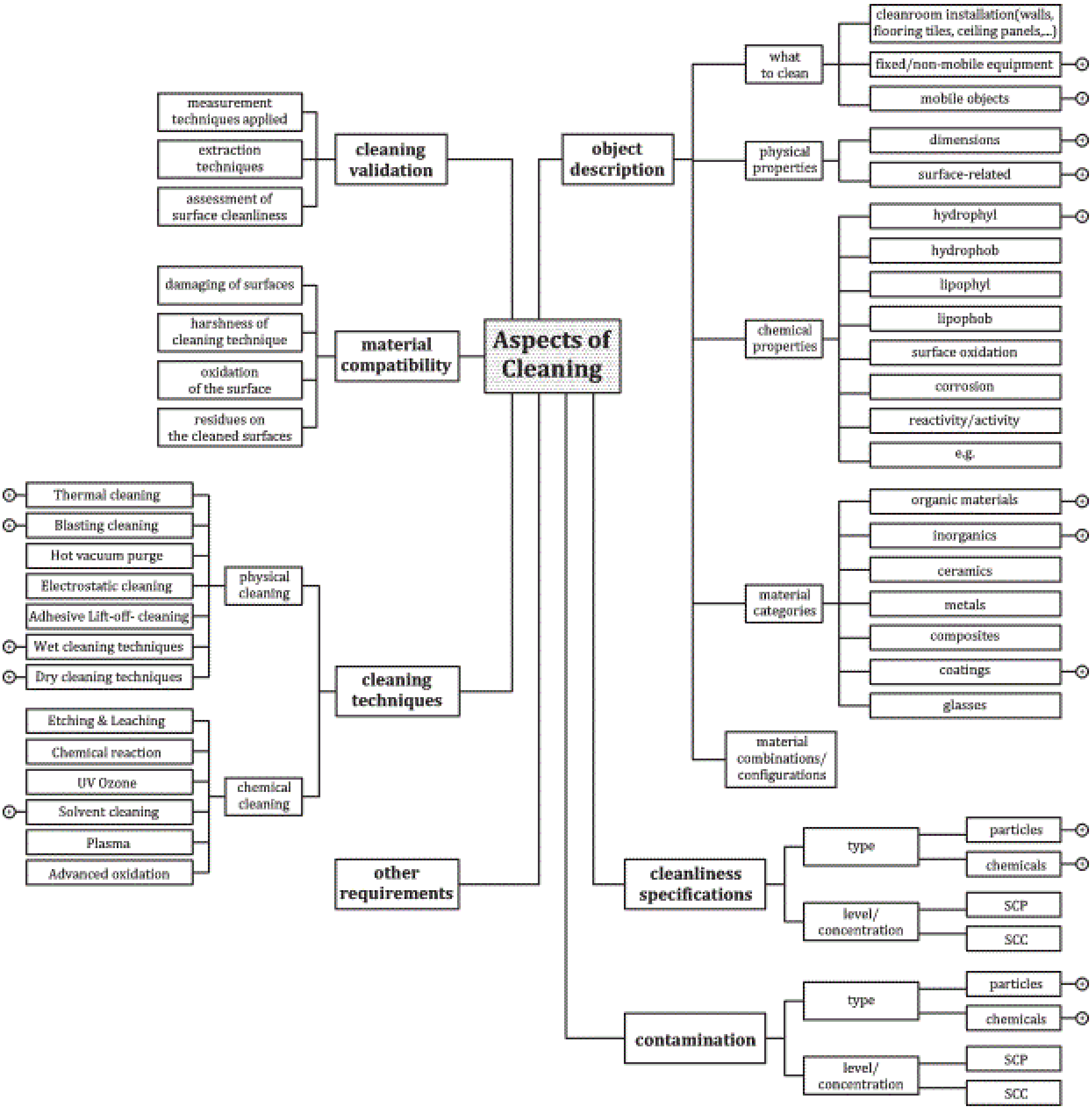


Figure A.1 — Detailed non-exhaustive overview of cleaning aspects

Annex B
(informative)

Cleaning methods

B.1 General

It is important to note that some cleaning methods are only for application outside the cleanroom for pre-cleaning.

The number of available techniques is extensive. This overview gives an indication of the available cleaning methods. The methods are divided into:

- mechanical cleaning;
- fluidic cleaning;
- chemical cleaning;
- blasting cleaning techniques.

In some cases, combinations of the described cleaning methods can be applied.

For all dry cleaning techniques, tribo-electrical effects may cause charging of the surface. Electrostatic attraction plays an important role in the adhesion of dust particles to a product surface. In order to eliminate this adhesive force, an equal number of positive and negative ions can be generated in ionized air and transported to the surface of the product where they neutralize the electrostatically charged dust particles and the surface itself. By neutralizing the surface particle re-deposition is avoided.

B.2 Mechanical cleaning

B.2.1 Wiping

Principle: Soft, non-abrasive, non-cross contaminating materials can be utilized to remove lightly-adhering contaminants (dust, dirty water, etc.). If a wiping process is performed using a cleaning fluid, the chemical action of the cleaning agent is essentially responsible for the cleaning effect. In order to detach and remove contaminants with a wipe or a supported wipe, the adhesive forces between the contamination and object surface need to be overridden by frictional movements.

Areas of application: Wiping is especially effective for removing liquid and particle contamination. It is usually a manual process.

B.2.2 Brushing/sweeping

Principle: The cleaning technique of brushing/sweeping is the purely mechanical wiping off or abrading of generally solid contaminants from an object surface through the action of bristles. Brushes with soft bristles made of plastic or natural fibres are used to remove easily-detachable contamination, such as dust. For strongly-adhering contaminants such as oxides, scale or paint, stiffened, non-shedding bristles may be utilized. All materials which are not damaged by the chosen brush or where a minimum degree of surface abrasion is tolerated can be cleaned in this way.

Areas of application: Brushing/sweeping is frequently used to clean flat surfaces.

B.2.3 Scraping/abrading

Principle: Scraping or abrading is a mechanical separation technique in which adhering solids are lifted up by a tool placed beneath them (e.g. blade or scraper) and then removed.

Areas of application: Scraping/abrading is implemented to remove strongly-adhering material residues from essentially smooth surfaces.

B.2.4 Grinding

Principle: Grinding is the removal of superficial contamination by abrasion, where usually rotating grinding discs or high-speed circular grinding belts are used. Grinding is capable of removing strongly-adhering contaminants such as scales, oxides, rust, coatings or paints quickly and effectively. The fact that a certain amount of base material will be abraded as well should be taken into account.

Areas of application: As the grinding discs used are made of grains of hard material (e.g. silicon carbide, diamonds) embedded in a bonding material, the selection of the grain/bonding material is decisive for the grinding process.

B.3 Fluidic cleaning

B.3.1 Washing/rinsing/drying

Principle: Cleaning with the aid of liquid agents often involves washing in order to remove the detached/emulsified contamination, as well as the active ingredients of the solvent cleaning agent itself, e.g. surfactants and emulsifiers, from the object surface. This prevents contaminants from being transferred to the next cleaning step or causing re-contamination in a subsequent rinsing or drying process.

Rinsing is applied to remove unwanted residues of the washing process.

Drying is generally the final step. It removes the remains of the cleaning medium of the previous process. It does not remove contamination from the previous process.

Areas of application: Rinsing processes are principally used to achieve improved cleanliness levels after cleaning with aqueous fluids.

B.3.2 Compressed gas cleaning

Principle: With this cleaning technique, surfaces are cleaned by the action of a jet of gas which is directed at an object at high speed via a nozzle. The gas jet exerts an aerodynamic force on the particle. If the aerodynamic resistance of a particle exceeds the adhesive force between the particle and the surface, the particle is detached from the surface and carried away with the flow of gas. In particular, the technique allows dry, loosely-adhering contaminants, such as dust, to be removed effectively. Particles in the micrometre size range can no longer solely be removed by use a high-speed jet of gas. Particles are suspended in the gas and can be re-deposited at other locations. Ionized gas can suppress the electrostatic charge of the surface.

Areas of application: If only dry contamination is present, good cleaning results can be achieved for particles larger than 5 µm. Liquid or paste-like contamination can also be removed partially as long as higher pressures, e.g. by using supersonic (Venturi) nozzles, are applied.

B.3.3 Vacuum cleaning

Principle: A suction nozzle is placed above the object's surface to be cleaned and being connected to a pump creates a negative pressure which causes an air flow away from the object's surface. This airflow exerts an aerodynamic force on the particle contamination. If the aerodynamic resistance of the particle exceeds the adhesive force between the particle and the surface, the particle is detached from the surface and carried away with the airflow. Vacuum cleaning is especially suitable for removing dry contamination such as dust particles. Usually, this technique is unsuitable for removing particles in the micrometre range.

Areas of application: Vacuum cleaning is used in controlled environments and for hard to reach areas.

B.3.4 Sonic cleaning

B.3.4.1 Ultrasonic cleaning

Principle: With ultrasonic cleaning, the objects to be cleaned are suspended in a bath filled with a suitable cleaning agent. Ultrasound waves are created in the cleaning bath via oscillating walls that spread longitudinally through the cleaning agent. Imploding cavitation bubbles create very high pressures of approximately 1 000 bar which will blast off contamination from surfaces which are then dissolved or dispersed into the cleaning fluid. After cleaning, the surfaces need to be rinsed and dried.

Ultrasonic action is also active at internal surfaces that are otherwise hard to clean.

Areas of application: Ultrasonic cleaning achieves extremely high quality results within a very short time, even for complex-shaped objects, crevices and drill holes. In general, ultrasonic cleaning is a gentle cleaning technique which can be used to clean delicate surfaces without damaging them.

B.3.4.2 Megasonic cleaning

Principle: Megasonic cleaning relates to the group of sonic cleaning. As megasonic cleaning uses higher frequencies (often in the range of 800 kHz to 2 000 kHz) than ultrasonic cleaning (20 kHz to 500 kHz) it creates less cavitation and therefore causes less damage to the surfaces to be cleaned. Acoustic waves in the cleaning fluid generate pressure variations which remove contamination from surfaces. Only the surface of the object facing the transducer(s) is cleaned.

Areas of application: Medical implant, flat panel and wafer manufacturing or industrial parts cleaning.

B.3.5 Spray cleaning

Principle: Cleaning liquid enters under increased pressure in the cleaning environment (chamber). A wide range of cleaning agents can be used in combination with water. The cleaning action is characterized by a high impact of cleaning spray and a continuous refreshment of the cleaning agent. In a spray cleaner, either the object to be cleaned or the nozzle can be moved in order to cover the entire object surface.

Areas of application: Spray cleaning is used for a wide range of objects.

B.4 Blasting cleaning techniques

B.4.1 General

The blasting cleaning techniques are divided into the main medium used to deliver the blast: gas and liquid.

Blasting cleaning techniques should only be applied to robust surfaces.

B.4.2 Gas blasting

B.4.2.1 Compressed air blasting

Principle: Surfaces are cleaned through the action of various blasting agents which are carried in the compressed gas blast and applied at fixed or fluctuating speeds to the object to be cleaned. The method is highly versatile as it removes contamination, debris, corrosion, scale and paint. The high kinetic energy transferred by the blasting agent impacting onto the surface may cause damage.

Areas of application: Suitable for cleaning components and structures of every size and description, only areas in direct contact with the impacting media will be cleaned properly.

B.4.2.2 Wet compressed air blasting

Principle: The method is similar to that of compressed gas blasting. However, here a blasting jet composed of air is used to accelerate water and a blasting agent.

Areas of application: The method is appropriate for cleaning surfaces and structures of every size and shape. After application, the surface to be cleaned will be covered with a slurry which needs to be removed with an air or water jet after the cleaning is completed.

B.4.2.3 CO_2 pellet cleaning

Principle: Dry ice pellets at approximately −80 °C (diameter/length in millimetre range) are accelerated to almost supersonic speed (300 m/s). The rapid cooling of the surface leads to embrittlement of the surface layers. During sublimation of CO_2, adhered contamination can be easily removed from the surface. It is less abrasive than pressurized fluid blasting.

Areas of application: The method can be utilized on nearly all materials. As the method has abrasive character, some "soft" surfaces might be visibly roughened.

B.4.2.4 Accelerated CO_2 snow cleaning

Principle: Accelerated CO_2 snow cleaning is a dry and solvent-free process. As carbon dioxide expands rapidly; it changes from liquid CO_2 to a mixture of solid CO_2 (dry ice) and gaseous CO_2. The dry ice is then blasted onto the surface to be cleaned accelerated by pure nitrogen. CO_2 snow cleaning is a further development of the CO_2 pellet cleaning. CO_2 snow cleaning is less abrasive than CO_2 pellet cleaning.

Areas of application: CO_2 snow cleaning has a broad scope of usage, ranging from precision cleaning right up to the cleaning of large surface areas. It can be utilized to clean highly sensitive surfaces. Particle contamination can be removed, as well as oil films, grease, fingerprints and flux residues.

B.4.3 Liquid blasting

B.4.3.1 Pressurized liquid blasting

Principle: A jet of liquid under high pressure is directed via a nozzle at the surface to be cleaned. A solid blasting material can be added to the liquid to increase the abrasive action. Ideally, even single layers of a coating system can be cleaned off. After application, the surface to be cleaned will be covered with a slurry which needs to be removed with an air or water jet after the cleaning is completed.

Areas of application: Suitable for all sizes and shapes of cleaning surfaces and structures; only the areas in direct contact with the impacting media will be cleaned properly.

B.4.3.2 Low-pressure water jet blasting

Principle: Method similar to that of compressed air blasting. However, very little water is added to the blasting agent/air mixture in front of the nozzle. In contrast with compressed air blasting, a surface prepared using this method is initially wet.

Areas of application: Suitable for all sizes and shapes of cleaning surfaces and structures; only the areas in direct contact with the impacting media will be cleaned properly. It is invariably used in cases where dust and water levels need to be kept low.

B.4.3.3 Elutriation blasting

Principle: A fine-grained material dispersed in water or other liquid is blasted at high speed onto the surface to be cleaned. In contrast to wet compressed air, the liquid accelerates the blasting agent.

Elutriation blasting is used to clean and micro-finish surfaces at the same time. After elutriation cleaning, rinsing is necessary to get rid of any adhering blasting agent residues. Elutriation blasting is suitable for producing a fine and even surface profile.

Areas of application: Precision casting, micro-/precision deburring, polishing surfaces, cleaning surgical instruments.

B.4.3.4 Steam blasting

Principle: Heated, pressurized water expands in a nozzle, resulting in a hot vapour spray being applied to surfaces. This highly efficient method for cleaning oily surfaces can be enhanced by adding detergents.

Areas of application: Steam blasting is applied where strong mechanical washing and rinsing actions are required.

B.5 Chemical cleaning

B.5.1 General

With chemical cleaning techniques, a cleaning agent reacts with the contamination present to remove it. The cleaning agent then either evaporates or can be easily removed.

B.5.2 Etching

Principle: Etching is the removal of the surface layer with contamination by chemical reaction.

Areas of application: Removal of coatings and final cleaning of components.

B.5.3 Chemical reaction

Principle: Specifically selected chemicals are used in a targeted way to convert contamination through a chemical reaction into other compounds which are either volatile or easy to remove.

Areas of application: With this principle, surfaces can be descaled, for example, by using acidic aqueous solutions.

B.5.4 Vapour cleaning

Principle: Vapour cleaning is often used in combination with ultrasonic cleaning in a fast drying environment-friendly solvent. The effect can be enhanced by using a mixture of chemicals. After immersion, a film of solvent covers the surface of the object. In this film, the remaining contamination is dissolved or dispersed. By moving the object through a cold zone, the film is stripped from the surface. The object leaves this zone with a dry surface.

Areas of application: Metallic objects, electronic parts, degreasing.

B.5.5 Plasma cleaning

Principle: In plasma cleaning, a mixture of reactive species is created that sputter the surface of the object. This cleaning action releases contamination from the surface and additionally creates a reaction that transfers contamination into a gas phase which can be removed by a vacuum system.

A plasma cleaning process is executed in a vacuum system. However, there are also atmospheric solutions available. The agitation energy is either in the RF range (13,6 MHz) or in the microwave range (2,4 GHz). As a cleaning gas, several pure gases or combinations can be selected: argon, nitrogen, oxygen, halogens, etc.

Areas of application: Electronic parts, plastic objects, metallic objects, optics.

B.5.6 Ozone cleaning

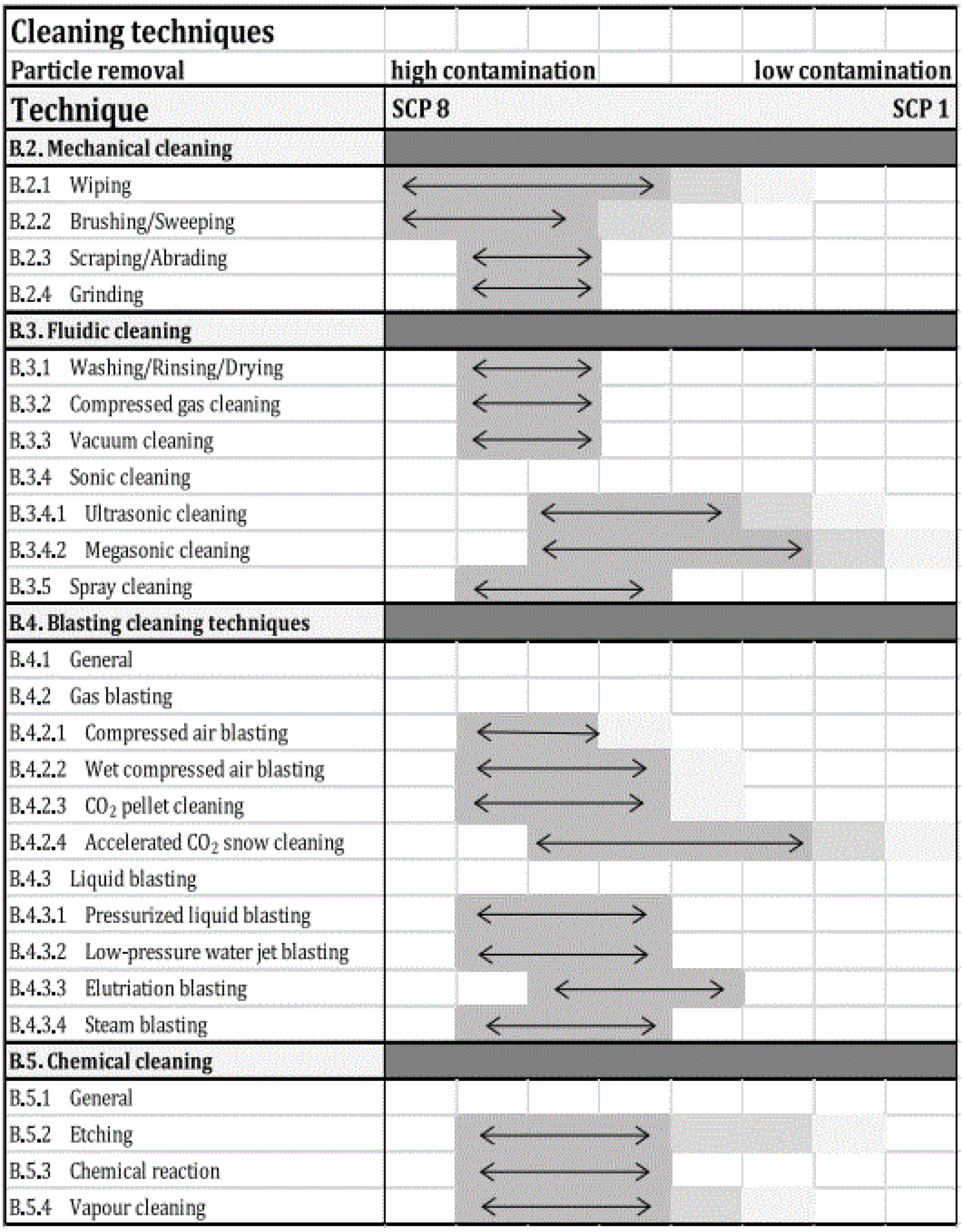
Principle: In ozone, reactive O_3 species are used to oxidize organic contamination. The ozone is generated and activated by UV light.

Areas of application: Flat objects like glass plates, optics.

B.6 Working ranges of described cleaning techniques

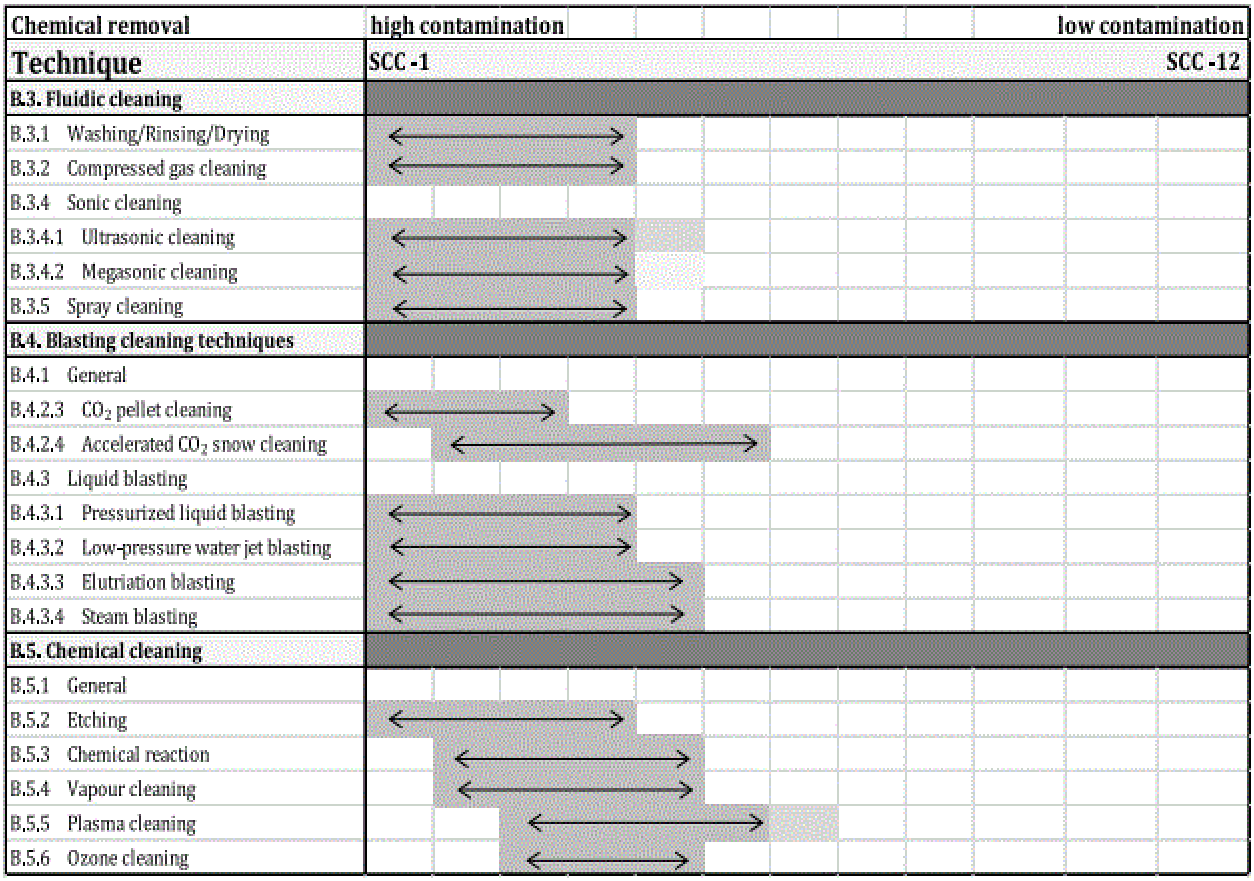
In Table B.1 and Table B.2, an overview is given of typical working ranges for the applicability of some cleaning techniques. The grey area within the tables is the area where these techniques can be applied.

Table B.1 — Working ranges of described cleaning techniques for particle removal



Cleaning techniques								
Particle removal	high contamination					low contamination		
Technique	**SCP 8**							**SCP 1**
B.2. Mechanical cleaning								
B.2.1 Wiping	▓	▓	▓	▓	░	░		
B.2.2 Brushing/Sweeping	▓	▓	▓	░				
B.2.3 Scraping/Abrading		▓	▓					
B.2.4 Grinding		▓	▓					
B.3. Fluidic cleaning								
B.3.1 Washing/Rinsing/Drying		▓	▓					
B.3.2 Compressed gas cleaning		▓	▓					
B.3.3 Vacuum cleaning		▓	▓					
B.3.4 Sonic cleaning								
B.3.4.1 Ultrasonic cleaning			▓	▓	▓	░		
B.3.4.2 Megasonic cleaning			▓	▓	▓	▓	░	░
B.3.5 Spray cleaning		▓	▓	▓				
B.4. Blasting cleaning techniques								
B.4.1 General								
B.4.2 Gas blasting								
B.4.2.1 Compressed air blasting		▓	▓	░				
B.4.2.2 Wet compressed air blasting		▓	▓	▓	░			
B.4.2.3 CO_2 pellet cleaning		▓	▓	▓	░			
B.4.2.4 Accelerated CO_2 snow cleaning			▓	▓	▓	▓	░	░
B.4.3 Liquid blasting								
B.4.3.1 Pressurized liquid blasting		▓	▓	▓				
B.4.3.2 Low-pressure water jet blasting		▓	▓	▓				
B.4.3.3 Elutriation blasting			▓	▓	▓			
B.4.3.4 Steam blasting		▓	▓	▓				
B.5. Chemical cleaning								
B.5.1 General								
B.5.2 Etching		▓	▓	▓	░	░	░	
B.5.3 Chemical reaction		▓	▓	▓				
B.5.4 Vapour cleaning		▓	▓	▓	░	░		

Table B.2 — Working ranges of described cleaning techniques for chemical removal



Chemical removal	high contamination											low contamination
Technique	SCC -1											SCC -12
B.3. Fluidic cleaning												
B.3.1 Washing/Rinsing/Drying	▨	▨	▨	▨								
B.3.2 Compressed gas cleaning	▨	▨	▨	▨								
B.3.4 Sonic cleaning												
B.3.4.1 Ultrasonic cleaning	▨	▨	▨	▨	░							
B.3.4.2 Megasonic cleaning	▨	▨	▨	▨	░							
B.3.5 Spray cleaning	▨	▨	▨	▨								
B.4. Blasting cleaning techniques												
B.4.1 General												
B.4.2.3 CO_2 pellet cleaning	▨	▨	▨									
B.4.2.4 Accelerated CO_2 snow cleaning		▨	▨	▨	▨	▨						
B.4.3 Liquid blasting												
B.4.3.1 Pressurized liquid blasting	▨	▨	▨	▨								
B.4.3.2 Low-pressure water jet blasting	▨	▨	▨	▨								
B.4.3.3 Elutriation blasting	▨	▨	▨	▨	▨							
B.4.3.4 Steam blasting	▨	▨	▨	▨	▨							
B.5. Chemical cleaning												
B.5.1 General												
B.5.2 Etching	▨	▨	▨	▨								
B.5.3 Chemical reaction		▨	▨	▨	▨							
B.5.4 Vapour cleaning		▨	▨	▨	▨							
B.5.5 Plasma cleaning			▨	▨	▨	▨	░					
B.5.6 Ozone cleaning			▨	▨	▨							

Annex C
(informative)

Material compatibility with cleaning agents

Chemical compatibility of materials with cleaning liquids has been tested extensively. Table C.1 gives examples of compatibility for a variety of materials with the most common organic cleaning liquids. This table gives insights into liquids that can be used for cleaning with minimal damage to the object. This table only applies for room temperature. Chemical blends, elevated temperatures and high mechanical action may still result in damage. Note that tables like Table C.1 can be used only as an indication. It is strongly recommended to conduct experimental compatibility tests. Same materials (notably polymeric materials) from different suppliers are likely to behave differently. Even batch-to-batch variation can be expected.

Other sources for material compatibility are available (see Bibliography).

Table C.1 — Examples of compatibility of various solvents with listed materials (indicative only)

Type of material	Abbreviation	Methanol	Ethanol	Isopropylic alcohol	Acetone	Methyl Ethyl Ketone	Dichloromethane	Chloroform
polymer								
Acetal (polyoxymethylene)	ACL	A	A	A	A	C		A
Epoxy		B	A	A	B	C		C
Ethylene-chlorotrifluoroethylene copolymer	E-CTFE	A	A	A	B	A	C	A
Ethylene-tetrafluoroethylene	ETFE	A	A	A	B	A	B	A
Fluoroethylene propylene	FEP	A	A	A	A	A	A	A
Tetrafluoroethylene	TFE	A	A	A	A	A	A	A
Perfluoroalkoxy	PFA	A	A	A	A	A	A	A
Polyamide		D	D	D	A	A	B	C
Polycarbonates	PC	B	A	A	D	D	D	D
Low density Polyethylene	LDPE	A	B	A	D	D	D	C
High density Polyethylene	HDPE	A	A	A	D	D	C	C
Polyimide		B	B	B	B	B	B	B
Polymethyl methacrylate	PMMA	D	D	D	D	D	D	D
Polyketone	PK (PEEK)	A	A	A	A			C
Polystyrene	PS	B	A	A	D	D	D	D
Polysulfone	PSF	A	A	B	D	D	D	D
Polyethylene terephthalate	PET	A	A	A	C	A	D	D
Polypropylene	PP	A	A	A	B	B	C	D
Polyurethane	PUR	C	C	C	D	D	D	D
Polyvinylidene fluoride	PVDF	A	A	A	D	D	A	A
Silicone		A	C	A	D	D	D	D
Vinylidene fluoride – hexafluoropropylene		C	A	A	D	D		A

Table C.1 *(continued)*

Type of material	Abbreviation	Methanol	Ethanol	Isopropylic alcohol	Acetone	Methyl Ethyl Ketone	Dichloromethane	Chloroform
metal								
Aluminium		A	B	B	A	B		B
Copper		B	A	B	A	A		A
316 Stainless steel		A	A	B	A	A		A
Titanium		B	A	B	A	A		A
miscellaneous								
Carbon graphite		A	A	A	A	A		A
Al_2O_3		A	A	A	A	A		A
SiC		A	A	A	A	A		A

Ratings on chemical effects at 20 °C:

- A: Excellent — no damage after 30 days of constant exposure;
- B: Resistant — little or no damage after 30 days of constant exposure;
- C: Fair to poor — some effect after 7 days of constant exposure;
- D: Not recommended — immediate damage may occur;
- Blank: No data available.

Annex D
(informative)

Cleanliness measurement

D.1 General

The surface cleanliness can be determined qualitatively by visual inspection or quantitatively by a direct or indirect measurement method. For indirect measurement, the contaminants are extracted from the surface.

D.2 Visual inspection

Visual inspection can be used to determine initial surface cleanliness. If contamination is detected at this stage, more extensive and expensive detection processes can be deferred until the surface cleanliness is improved by pre-cleaning.

Visual inspection can be executed, for example, by using ordinary light or with black light (ultraviolet) to detect fluorescing contaminants. A hand magnifier or digital microscope may increase the ease of observation of contamination.

Chemical contamination can be observed by a discolouration or by diffraction in thin layers. Other visual observation methods like contact angle measurements or a water break test can be used. Bead formation and high-contact angles may indicate the presence of organic contaminants. While a large contact angle is usually an indication of residual contamination, the converse is not necessarily true. Salts or residues from cleaning agents can result in a low contact angle, giving a false indication of cleanliness.

D.3 Direct surface cleanliness measurement

Direct surface observation may be appropriate in situations where the surface is accessible. The surface under study will either be brought to the measurement instrumentation or the instrumentation will be portable and brought to the surface.

Potential measurement methods are described in D.5 and in ISO 14644-10 in more detail.

D.4 Indirect surface cleanliness measurement by extraction

D.4.1 General

Where the performance of the cleaning process cannot readily be performed on the surface of the object to be evaluated, extractive methods are appropriate. Examples of instances where direct analysis is not appropriate include evaluation of objects with complex structures such as blind holes, situations where the analyte(s) of concern occur in a mixture and should be separated from each other and objects that have physical dimensions that cannot readily be analysed with the pertinent equipment.

D.4.2 Extraction techniques

The extraction process may include chemicals, heat and force. Extraction may be performed directly from the substrate or it may involve separation of the contaminant or analyse from a swab or wipe. The rationale of choices for sampling and extraction should be documented.

Sampling technique should be defined. The areas of the surface selected for extraction should include areas difficult to clean (e.g. blind holes or around seals). The amount of contaminant collected from the object needs to be sufficient to allow detection. The area sampled should be representative for the total surface area of the object including the range of material components.

If there is a single contaminant of concern, using a single extraction agent may be adequate. Usually, it is necessary to use at least two extraction agents to account for different polarities of the contaminant. Even with extraction of particles, because particles are held in place using both physical and chemical forces, it may be necessary to use several different extraction agents.

In selecting extraction solvents, a number of factors should be considered and those factors should be documented. Examples of such factors include purity of the extraction solvent, solubility parameters, wettability (surface tension, viscosity) and compatibility with the substrate or with the wipe or swab used for sampling.

D.4.3 Extraction from the object

The sampling technique and extraction technique should be defined.

Extraction from the object should be performed using a combination of chemicals and a process that will remove potential contaminants from all areas of the object without undue material-compatibility problems, i.e. without significant damage to the object in question. The group performing the validation should define and document any significant damage that occurs.

D.4.4 Extraction from the wipe

Sampling and extraction can show variability. Wipes and swabs contain potentially interfering materials. Therefore, controls (blanks, where swabs or wipes are subjected to a given extraction technique) should be documented.

D.5 Measurement methods

D.5.1 General

This subclause gives an overview of the most important methods for determination of particle or chemical contaminant concentrations. In ISO 14644-9 and ISO 14644-10, a more complete list is given.

D.5.2 Measurement methods for particles

D.5.2.1 Visual inspection

For objects that require a low cleanliness level, a visual inspection can be used. The inspection can be enhanced by using a flash light under a glancing angle or a UV lamp. Under ideal conditions, particles larger than 25 µm can be observed. The minimum size of detectable particles increases enormously when some surface roughness is present. Since detection depends on the observation skills of the operator, this technique is semi-quantitative only.

D.5.2.2 Light microscopy

Light microscopes are used for measuring particles larger than 1 µm. Direct measurement of surface particle cleanliness often requires microscopes having automatic stages and autofocus systems and/or using backlit systems. These systems can make series of images of subsequent fields. This allows measurement of a much larger surface area and evaluation of surfaces with low surface particle concentrations.

In indirect measurements, microscopes are used to determine particle concentrations on filter materials.

D.5.2.3 Oblique-, glancing- and side-light measurement systems

As with light microscopy, the image of a surface is depicted on a digital camera using the required magnification. By using oblique illumination with parallel light on the surface, existing surface structures are only minimally illuminated and light scattered by particles enters the camera. This technique is also known as dark field microscopy. This method enables detection of sub-resolution particles on a relatively large area. Size information is not reliable because the amount of scattered light depends on both the particle size and refractive index. The size of the smallest detectable particle depends on the surface roughness.

D.5.2.4 Scanning electron microscopy (SEM)

SEM is capable of imaging very small particles (as small as 10 nm). The sample is exposed to an electron beam in vacuum. The secondary electrons are detected. The smallest particles can only be imaged at a large magnification. At those conditions, the depth of focus enables imaging of low roughness samples in a small field of view. Therefore, measurement of the surface cleanliness by particles with a SEM is limited to high SCP classes only.

D.5.2.5 Optical particle counting

When an indirect method is used, particles are transferred to air, gases or liquids and the particle concentration is measured by optical particle counting. Particles that are present in the medium generate scattered light that is registered by photo-detectors and analysed, on passing through the laser.

An equivalent particle size is calculated from the intensity of the scattered light. In air and gasses, particles larger than 100 nm can be measured using an airborne particle counter. For a high particle concentration, a Scanning Mobility Particle Sizer (SMPS) can be used. When only particle concentrations are required, a Condensation Particle Counter (CPC) can be used. The sample air is lead through saturated (water, alcohol) gas. The component condenses on the particle which can be detected more easily. Particles as small as 5 nm can be detected. The size information is lost.

Particles in liquid can also be detected by optical particle counting. Equipment for detecting particles larger than 50 nm is commercially available.

D.5.2.6 Light-extinction particle counting

In both methods of detecting particles (extinction and scattering of light), a change in light intensity measured by the detector is converted to an electrical signal. Light extinction is useful for particles 1 µm and greater in size. In this method, the detector looks directly into the light source and measures the size of the “shadow” of the particle as it passes through the beam.

D.5.2.7 Scattered-light surface scanning device

Scattered-light scanners are implemented especially to examine surfaces with a very low roughness (e.g. silicon wafers, glass). A focused laser scans the component surface using a defined beam angle. The light reflected directly from the surface is guided into a light trap and thus eliminated. Particles present on the surface cause the laser light to be diffusely scattered. The scattered light is registered by a photomultiplier and amplified. Based on the intensity of scattered light, detected conclusions can be drawn about the size and location of the particles. The size is often expressed as the equivalent sphere diameter. The detection limit of scattered-light scanners lies at a particle size of >0,05 µm. Since a large surface area can be scanned, this method is suitable for low SCP classes.

D.5.2.8 Analysis by atomic force microscopy (AFM)

The atomic force microscope is a very-high-resolution type of scanning probe microscope, with demonstrated resolution of fractions of a nanometre, more than 1 000 times better than the optical diffraction limit. The AFM consists of a micronscale cantilever with a sharp tip (probe) at its end that is used to scan the specimen surface.

This method can only be applied on a small detection area (100 µm^2 × 100 µm^2) and particles larger than 1 µm cannot be measured correctly. Because of the small measurement area, it is difficult to select a representative measurement location.

D.5.2.9 Gravimetry

Gravimetry is a method that is used for indirect measurement of particles. The particle load on a surface or test object transferred to a gas or a liquid which is filtered. The increase in mass of an analysis filter (differential weight) is a standard for the surface particle cleanliness. The measurement limit lies at approximately 0,1 mg per analysis filter. Therefore, this method can be used for high SCP classes only.

D.5.3 Measurement methods for chemical contamination

D.5.3.1 General

This subclause describes measurement methods for chemical contamination. More information can be found in ISO 14644-10.

Most methods for measurement of chemical contamination can be applied on hydrocarbons. In this subclause, a selection of methods is described that can either be applied on the relevant surface itself (direct measurement) or can be used after extracting the contamination (indirect methods). Most direct measurement methods can also be applied in an indirect procedure.

D.5.3.2 Direct measurements

D.5.3.2.1 Secondary ion mass spectrometry (SIMS)

Mass spectrometry is used to measure the mass-to-charge quotient and abundance of secondary ions emitted from a sample after ion bombardment. It is a quantitative method using standard materials that characterizes trace contamination of surfaces, thin films, multilayer structures and interfaces.

SIMS is often combined with a time-of-flight method (TOF-SIMS) for analysing the mass.

D.5.3.2.2 Total reflection X-ray Fluorescence spectroscopy (TXRF)

TXRF is an X-ray spectrometer measuring energy distribution of fluorescent X-rays emitted from surface irradiated by primary X-rays under condition of total reflection. TXRF gives a qualitative elemental screening of unknown samples, metallic surface contamination on semiconductor wafers and composition ratios of binary thin films.

D.5.3.2.3 Fourier Transform infrared spectroscopy (FTIR)

FTIR is a measurement for infrared absorption spectra in which a spectrometer is used to measure the infrared energy. It is used for identification of chemical structure of organic compounds, films, particles, powders and liquids. Quantification is possible by using a standard.

D.5.3.3 Microgravimetry

Devices, in which piezoelectric quartz crystals respond to the condensation of gasses by reducing operating frequency in a linear mass-to-frequency relationship, can be used for direct deposition flux measurement of any chemical thin films in the range of nm to µm. Depending on their sensitivity, either a quartz crystal microbalance (QCM) or a surface acoustic wave sensor (SAW), which is 100 times more sensitive than a QCM, can be selected.

D.5.3.4 Indirect measurements

D.5.3.4.1 General

The indirect method is a combination of an extraction method and an analysis technique.

D.5.3.4.2 Solvent dissolution–gas chromatography/mass spectrometry (SD-GC/MS)

The contaminants on the substrate surface are dissolved with appropriate solvent. A portion of solvent is analysed with gas chromatography/mass spectrometry (GC/MS). This method gives compositions and quantity of organic contaminants on a surface.

D.5.3.4.3 Thermal desorption–gas chromatography/mass spectrometry TD-GC/MS

In this method, contaminants on substrate surface are thermally desorbed and concentrated into an absorption column. Concentrates are then introduced to a gas chromatograph with mass spectrometer. The method can be used for trace analysis of organic species, evaluation of contaminants, e.g. on silicon wafers.

D.5.3.4.4 Solvent dissolution–high performance liquid chromatography (SD-HPLC)

The contaminants are dissolved with appropriate solvent and are introduced to high-performance liquid chromatograph to be separated from each other and evaluated.

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