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Road vehicles — Cleanliness of components and systems

Véhicules routiers — Propreté des composants et des systèmes

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Foreword

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

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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 22, *Road vehicles*, Subcommittee SC 34, *Propulsion, powertrain, and powertrain fluids*.

This edition of ISO 16232 cancels and replaces the entire ISO 16232:2007 series (all parts) which have been technically revised and consolidated into a single document.

The main changes are described in Annex L.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

In order to achieve reliable performance of components and systems, control over the amount of particles introduced during the build phase is necessary, and measurement of particulate contaminants is the basis of control.

Based on a world-wide broadening and ongoing development of technical cleanliness issues this document is intended to:

- further improve the comparability of cleanliness inspection results;
- include new (extraction and analysis) techniques; and
- provide information on how to define cleanliness specifications and explain how to react when cleanliness limit values are exceeded.

This document has been prepared to fulfil the requirements of the automotive industry, since the function and performance of modern automotive components and systems are sensitive to the presence of a single or a few critically sized particles. Consequently, this document deals with the analysis of the total volume of extraction liquid and of all contaminants collected using an approved extraction method.

To be able to compare inspection results the same extraction procedure, the same test fluid and also same parameter settings for the analysis instruments are intended to be used.

This document is based on existing International Standards such as those developed by ISO/TC 131/SC 6. They have been extended, modified and new ones have been developed to produce a comprehensive suite of International Standards to measure and report the cleanliness levels of components and systems fitted to road vehicles.

Road vehicles — Cleanliness of components and systems

1 Scope

WARNING — The use of this document can involve hazardous materials, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this document to establish appropriate safety and health practices and to determine the applicability of any other restrictions prior to its use.

This document specifies requirements for applying and documenting methods for determining particulate contamination on functionally-relevant components and systems (cleanliness inspection) of road vehicles.

A cleanliness inspection comprises the basis of an assessment of technical cleanliness, which is performed, for example, under the following circumstances:

- initial inspection and evaluation;
- inspection of incoming and outgoing components; and
- quality control or monitoring of manufacturing processes relevant to cleanliness (e.g. cleaning, surface treatment and assembly processes).

This document is intended to improve the informative quality and comparability of test results. It also defines the standardized expression of cleanliness specifications and cleanliness test results in the quality chain of the automotive industry.

This document does not apply to the following:

- detection of filmy contamination (grease, oils, etc.);
- application of non-quantifiable particulate detection methods on test components (e.g. visual assessment, wipe test with clean cloth, etc.); and
- characterization of operating fluids (fuel, oils, coolants, brake fluid, etc.).

This document does not define any cleanliness limit values for specific components or systems. The degree of cleanliness required for a specific component or system is dependent on a number of highly-individual factors. Cleanliness specifications are intended to be undertaken only by specialists who not only know the component concerned but also the system it is built into, the later conditions of use, technically-feasible practices and possible consequences for manufacturing processes and the supply chain. Guidance for deriving limit values can be found in Annex H.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions 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 https://www.iso.org/obp

3.1

active component

component which may set the fluid in motion or be activated by the fluid during operation

EXAMPLE Pump, cylinder, distributor, injector, valve regulator.

3.2

aerosol formation

atomization of a liquid to create small droplets, e.g. in pressure-rinsing due to the shape of nozzles or impaction of the pressure-rinsing jet on a surface

3.3

agglomerate

two or more particles which are in intimate contact and cannot be separated by gentle stirring and the small shear forces thus generated

3.4

agitation

extraction method implemented for internal surfaces

Note 1 to entry: Its cleaning effect is based on the turbulent change in direction of the test liquid inside the component.

3.5

air cleanliness class

specification of air quality based on the concentration of particles in a defined volume of air

3.6

analysis balance

balance with a high (10^{-4} g) to very high (10^{-6} g) resolution capable of weighing minute quantities of particulate residue

3.7

analysis filter

filter membrane

thin membrane, either meshed or foamed, possessing defined deposition properties that cause particles of a specific size to be retained during filtration

3.8

analysis parameters

settings on an *analysis system* (3.9) that are used in the analysis step

3.9

analysis system

device to measure and/or characterize particles

3.10

automatic particle counter

APC

counting system that works on the light extinction principle

3.11

back-scattered electron detector

BSE detector

device that supplies scanning electron microscope (SEM) images with a high material contrast, used for the detection of particles on a membrane filter

3.12
blank level
blank value
amount of contaminant introduced from sources other than the test component, such as reagents, glassware, preparation of test units, and the environment

3.13
blank level criterion
maximum value that may not be exceeded when determining the blank level, i.e. max. 10 % of the required or expected cleanliness value

3.14
blank level test
analysis carried out in the same operating conditions as on the test component but without the test component

Note 1 to entry: The blank test enables quantification of the contamination introduced from sources other than the test component, such as reagents, glassware, preparation of test units, and the environment.

3.15
cavity
cavity in the test component, which cannot be wetted by the test liquid due to the presence of gas

3.16
clean
state of *cleanliness* (3.18) of a component or fluid that meets the specified cleanliness level

3.17
cleaning
process to make a liquid, object or extraction setup as clean as required

3.18
cleanliness
condition of a product, surface, device, liquid, etc., characterized by the absence of particulate contamination

3.19
cleanliness inspection
extraction and analysis of component contamination including documentation

3.20
cleanliness level
C_L
amount and/or nature of contaminant present on the controlled surfaces and/or in controlled volumes of a component

Note 1 to entry: The term may apply to the presumed, specified or measured extent of contamination.

3.21
cleanliness specification
documentation of permissible particle features and quantities for a component

3.22
cleanliness state
component cleanliness
cleanliness value (3.23) or values of a component that may change over time due to external influences

3.23
cleanliness value
single value specifying the cleanliness of a component, e.g. residue mass, longest particle or particle count

3.24
cleanroom
room within which the number concentration of airborne particles is controlled and classified, and which is designed, constructed and operated in a manner to control the introduction, generation and retention of particles inside the room

Note 1 to entry: The class of airborne particle concentration is specified.

Note 2 to entry: Levels of other cleanliness attributes such as chemical, viable or nanoscale concentrations in the air, and also surface cleanliness in terms of particle, nanoscale, chemical and viable concentrations might also be specified and controlled.

Note 3 to entry: Other relevant physical parameters might also be controlled as required, e.g. temperature, humidity, pressure, vibration and electrostatic.

[SOURCE: ISO 14644-1:2015, 3.1.1]

3.25
coincidence
particles grouped together, which can therefore not be analysed as single particles, e.g. by an optical particle counter or microscope

3.26
collection equipment
any device (e.g. conical flask, beaker, tray, funnel, collection surface of the extraction cabinet) with a size and a shape suited to the collection of all the extraction liquid draining from the test component

3.27
component
part, sub-assembly, or part assembly used in a road vehicle

Note 1 to entry: This definition differs from that given for the same term in ISO 5598.

3.28
component cleanliness code
CCC
alpha-numeric expression of the *cleanliness level* (3.20) of a *component* (3.27) measured or specified by the particle size distribution of the contaminants

3.29
component contamination
amount or nature of contaminants extracted from the wetted or controlled surfaces of a *component* (3.27), as measured by an applicable analysis method

3.30
conditioning
preparation step in which the *analysis filter* (3.7) is pressure-rinsed, dried and dehumidified before its tare mass is weighed

3.31
contaminant
undesirable solid substance that is in suspension in a fluid or in a component or on a *controlled surface* (3.33) of a component

Note 1 to entry: For the purposes of this document, contaminants are particles as defined in 3.77. This definition differs from ISO 5598 in its exclusion of liquids and gases.

3.32
contamination
all contaminants in a fluid, system or on a component

3.33
controlled surface
surface of a component that is subject to a cleanliness requirement

3.34
controlled volume
volume of a component that is subject to a cleanliness requirement

Note 1 to entry: The controlled volume may differ from the wetted volume.

3.35
critical particle
particle which geometric dimensions or chemical-physical nature could cause *component* (3.27) damage

Note 1 to entry: A sub-category of critical particles is formed by so-called "killer" particles, which cause an immediate component failure.

3.36
decline
decrease observed in the contamination level of a component, on repeated extraction

3.37
declining criterion
threshold for assessing the suitability of extraction conditions

3.38
declining test
procedure used to verify the efficacy and suitability of extraction parameters in which the extraction step is repeated six times on the same test component in an identical manner

3.39
declining value
D_n
quotient of the cleanliness value under consideration and the sum of all preceding cleanliness values (including that under consideration)

Note 1 to entry: It is expressed in percent.

3.40
dissolving liquid
test liquid used for extraction or another liquid with stronger solvent action to speed up dissolving step during the extraction process, compatible with the test component and inspection equipment

3.41
double inspection
procedure in which two identical extraction steps are performed in order to confirm the efficacy and suitability of qualified extraction parameters

Note 1 to entry: In this case, the declining criterion is set at 30 % instead of the 10 % that applies for declining tests.

3.42
effective filter surface area
area of the analysis filter membrane through which extraction liquid flows during the filtration step

3.43
element analysis
EDX analyses
energy dispersive X-ray spectroscopy capable of analysing elements based on their characteristic X-ray spectra

3.44
extraction
sampling
operation required to transfer as much contaminant as possible that is present within a controlled volume or on a controlled surface into a test fluid and its collection for subsequent analysis

3.45
extraction curve
declining curve
progress curve of the cleanliness level of an extraction fluid applied to the test component as a function of the number of extractions

Note 1 to entry: This is related to the extraction time or to the volume of test fluid passed through or over the test component.

3.46
extraction liquid
analysis liquid
extraction fluid
extraction air
test medium loaded with contaminants extracted from the test component

3.47
extraction method
technique to detach the particle load from the test component

3.48
extraction parameters
totality of all physical parameters influencing the extraction which can be set on or calculated from the extraction setup

3.49
extraction procedure
complete sequence of all extraction steps performed

3.50
extraction setup
extraction device
equipment used to perform the extraction

3.51
extraction step
sampling step
single work step forming part of the *extraction procedure* (3.49) that is performed either in a *declining test* (3.38) or when extracting several controlled surfaces

3.52
extraction volume
total volume of test fluid used to extract the contaminants from a component

3.53
fibre
long, thin structure which is defined by a ratio between stretched length and maximum inner circle diameter greater than 20, the width measured via maximum inner circle diameter being lower than or equal to 50 µm

Note 1 to entry: It is generally used to characterize a textile fibre (flexible, pliable, and made from organic materials) and differentiate it from compact particles.

3.54
filter background
colour of the analysis filter

3.55
filter housing
casing consisting of an upper and lower section with an inlet and outlet as well as a supporting grid; used to clamp and tension the *analysis filter* (3.7)

3.56
filter occupancy
ratio between surface area of the *analysis filter* (3.7) covered with particles to entire analysed filter area

3.57
filter pore size
<mesh filter> nominal mesh width

3.58
filter pore size
<foamed analysis filter> equivalent mesh width measured by means of the bubble point test

3.59
filtration
process in which particles are deposited on the analysis filter

3.60
final rinsing
application of fluid to remove any particulate residues from the surfaces of the extraction setup and deposit them on the analysis filter

Note 1 to entry: With "final rinsing", liquid flows over a component without any pressure being used. The step is implemented after extraction to remove any particles that may be adhering slightly to the component or surfaces of the extraction equipment due to re-sedimentation and to forward them to the analysis step. The liquid is applied to the surface using a spray.

3.61
final rinsing liquid
(test-) liquid used after the extraction step to remove any particulate residues from the surfaces of the extraction setup and deposit them on the analysis filter

3.62
fixative
liquid used to fix particles on the *analysis filter* (3.7) to prevent their loss though electrostatic charging

3.63
fan nozzle
flat jet nozzle
nozzle that generates a linear jet on impaction on a smooth surface

Note 1 to entry: Parameters include jet width and equivalent bore diameter.

3.64
functional test bench
closed circuit of test liquid onto which the test component is installed in order to be subjected to stresses or to provide similar functions to those to which it will be subjected to under final use operating conditions

3.65
gravimetry
analysis method used to determine the mass of all residues present on the analysis filter by measuring differences in mass

3.66

inspection procedure

inspection method

full sequence of all work steps including preparatory steps, extraction, filtration, analysis, carried out in the course of a *cleanliness inspection* (3.19)

3.67

inspection report

document that contains clear, summarized, general information together with details of the test component, information about the extraction, filtration and analysis steps as well as a representation of the inspection results

3.68

inspection specification

inspection document

detailed description of the *inspection procedure* (3.66)

Note 1 to entry: Where appropriate, it can also include illustrations to provide assistance.

3.69

internal rinsing

extraction method with a cleaning effect that is based on a turbulent flow of liquid inside the component

3.70

internal rinsing apparatus

technical construction consisting of a media supply and set-up to integrate the test component

Note 1 to entry: It is used exclusively to perform an internal rinsing extraction step, e.g. an adapted test bench for a hydraulic component.

3.71

isolation

screening of areas of the test component that do not form part of the controlled surface and which should not come into contact with the test fluid

Note 1 to entry: Sealing or masking are examples of isolation methods.

3.72

limit value

maximum permissible value for the residue mass, particle dimension (length, width, height) or particle quantity, which cannot be exceeded

3.73

material analysis

accurate characterization of a material by means of chemical, spectroscopic, mechanical or metallographic methods

3.74

material classification

assignment of elemental composition to a material class based on the percentages of individual elements identified

3.75

microscope

device used to magnify and visualize small objects, often containing an integrated camera system with image-processing

Note 1 to entry: A differentiation is made between material, stereo and zoom microscopes.

3.76
monitoring
inspection of cleanliness at regular intervals with the purpose of evaluating processes relevant to cleanliness

3.77
particle
tiny structure made from solid organic or inorganic matter

3.78
particle count
number of particles counted by their size or other characteristics

3.79
particle load
totality of all particles present on a surface or in a liquid

3.80
particle overlap
particles overlaying one another, which prevents them from being optically recorded as single particles

3.81
particle size
geometric feature of a particle that is stated in the cleanliness specification

Note 1 to entry: If this is not stated, the particle size corresponds with the longest dimension $\text{Feret}_{\text{max}}$.

3.82
particle size class
size class
particle size range with an upper and lower class limit

3.83
particle size distribution
particle count grouped into *particle size classes* (3.82)

3.84
particle standard
substrate marked with objects of a known shape and size used to verify that optical analysis systems are correctly calibrated

3.85
passive component
component which does not set the fluid in motion and is not actuated by the fluid

EXAMPLE Connector, tube, tank, etc.

3.86
preparatory steps
totality of all measures carried out after delivery of the component and before execution of the extraction procedure in order to enable all particles present solely on the controlled surface to be detached during the extraction step, e.g. disassembly, demagnetization, isolation, pre-cleaning

3.87
pressure-rinsing
extraction method with a cleaning effect that is generated by the kinetic energy of an open jet (impulse)

3.88
qualification report
documentation of the qualification test, including declining test and derivation of the routine inspection procedure

3.89
qualification test
performance of declining tests to work out a routine inspection

3.90
residue mass
mass of the particle load determined as the mass difference using the method of gravimetric analysis

3.91
routine inspection
cleanliness inspection performed with qualified extraction parameters (recurrent)

3.92
scanning electron microscope
SEM
high resolution microscope which images a sample by scanning it with a focused electron beam

3.93
start parameters
recommended parameters of a considered extraction method (e.g. pressure-rinsing) to commence qualification tests in order to achieve better comparability of extraction procedures and at last better comparability of cleanliness inspection results

3.94
test component
test object
test piece
single component, aggregate or system inspected for cleanliness

3.95
test conditions
circumstances under which the cleanliness inspection is performed

3.96
test liquid
test fluid
test air
medium of specified characteristics (e.g. viscosity, boiling point, cleanliness level, and solvency) used to extract contaminants from a component

3.97
test lot
all test components from which the combined particle load is measured in a cleanliness inspection

3.98
test lot size
number of test components comprising the *test lot* (3.97)

3.99
turbulent flow
flow of fluid with a cleaning effect generated by turbulences that is used to extract contaminants from internal component geometries

Note 1 to entry: An effective turbulent flow prevails in a liquid if the following applies: Reynolds number ≥4 000.

3.100
ultrasonic bath
technical device used to clean components by means of ultrasonic vibration

3.101
ultrasonic vibration
extraction method with a cleaning effect based on the formation and subsequent collapse of vapour bubbles (cavitation) that is induced by high sound pressures

Note 1 to entry: Frequencies ranging between 20 kHz and 400 kHz are used (in some cases up to 1 GHz).

3.102
vacuum rinsing
vacuum flushing
method of contaminant extraction using vacuum to flow the test fluid through the test component

3.103
validation
process by which a test method evaluates the capability of the contaminant removal process

3.104
volumetric measurement
flow rate determined by the measurement of a volume of liquid and the time required to fill a vessel to a specific level

3.105
wetted surface
A_c
surface area of the component that is exposed to a system fluid

3.106
wetted volume
V_c
volume of the component that is exposed to a system fluid

4 Symbols and abbreviated terms

A	Unit of reference "per 1 000 cm^2" when specifying the CCC
A_C	Controlled surface of all test objects in a test lot
ATR	Attenuated total reflection
APC	Automatic particle counter
BSE detector	Back-scattered electron detector
C_i	Cleanliness value
CCC	Component cleanliness code
CCD	Charge coupled device
CT	Computer tomography
d	Internal diameter (e.g. of piping)
D_n	Declining value at repeated extraction step n
EDX	Energy dispersive X-ray spectroscopy
$\text{Feret}_{\text{max}}$	the greatest possible perpendicular distance measured between two parallel lines touching the particle [µm]

FT-IR	Fourier transformation infrared spectrometer
m_C	Mass of particle load related to number of components tested
m_A	Mass of particle load in relation to controlled surface
m_V	Mass of particle load in relation to controlled volume
h	Absolute particle count
H_A	Particle count in relation to controlled surface
H_C	Particle count in relation to test lot size
H_V	Particle count in relation to controlled volume
Hz	Frequency (≙ movements per second) [1/s]
IR	Infrared
ISO-MTD	ISO medium test dust
λ	Wavelength
LIBS	Laser-induced breakdown spectroscopy
m_1	Tare mass of analysis filter
m_2	Total mass of occupied analysis filter
m	Residue mass of particle load
n	Number or test lot size
N	Unit of reference "per component" when specifying the CCC
nA	Numerical aperture
η	Dynamic viscosity [Ns/m^2]
ν	Kinematic viscosity [m^2/s]
PET	Polyethylene terephthalate
Re	Reynolds Number, $Re = w \cdot d/\nu$
SEM	Scanning electron microscope
SE detector	Secondary electron detector
U/S	Ultrasonic
V	Unit of reference “per 100 cm^3” when specifying the CCC
V_C	Wetted volume of all test objects in a test lot
w	Velocity of the fluid [m/s]
x	Variable for the particle size, e.g. 150 µm ≤ x < 200 µm
z	Particle height [µm]

5 Cleanliness inspection principles

5.1 General

The purpose of a cleanliness inspection is to detect the particulate contamination which is present on the relevant surfaces of a test component.

In contrast with other inspections, which can generally be performed directly on a test component, e.g. optical or tactile (contact) tests, the inspection of technical cleanliness is an indirect test that requires an extraction step. The relevant surfaces of functionally-critical components utilized in the automotive industry are often located inside pipes, channels, casings, tanks, pumps, valves or similar components. Fluids often flow through such components and these are capable of transporting particles to sensitive areas of a system. The inner surfaces cannot generally be accessed to allow a direct e.g. visual inspection. Furthermore, due to their material composition, roughness or lack of contrast with the particulate contamination, many surfaces are not suitable for visual inspections.

Consequently, in order to analyse technical cleanliness, first of all an extraction (or sampling) step is necessary. Here, the particles present are first removed from the component by means of a laboratory cleaning process (see Figure 1). The complete test fluid is then filtered, the particles extracted from the component deposited on an analysis filter and then subjected to analysis.

This also means that **it is impossible to repeat the cleanliness inspection on the same test component** because its cleanliness state has been altered as a result of the inspection method (extraction).

As in some cases a single particle may be functionally critical, it is essential that the collective of particles present on the component is removed as entirely as possible by the extraction step. As reference test components with a defined level of contamination do not exist to assess the efficacy of extraction procedures, the effect of extraction conditions for cleanliness inspection needs to be investigated based on the following:

- **Qualification of the extraction procedure (qualification test):** the appropriate extraction procedure for a test component is determined by performing qualification tests. The qualification criteria are stated in this document. They serve to ensure that as many of contaminant particles are removed from the test component as possible. Finally the qualification results, requirements and parameters of the extraction procedure chosen are documented.
- **Blank level:** the cleaner a test component is (i.e. the lower the number of particles expected to be determined in a cleanliness inspection), the higher the risk that external contamination introduced into the test procedure will falsify results. The quantity of extrinsic particles permitted in a test is stated in this document. This permissible blank level is orientated towards the cleanliness requirements of the respective test component.

The cleanliness inspection (see Figure 1) is made up of the following steps:

a) provision of the test component;

b) removal of the particles from the test component (extraction);

c) filtration of the particles (except with shortened analysis);

d) analysis of the particles; and

e) documentation of the inspection and results.

In order to perform cleanliness inspections, special equipment, suitable premises and skilled, experienced staff are required.

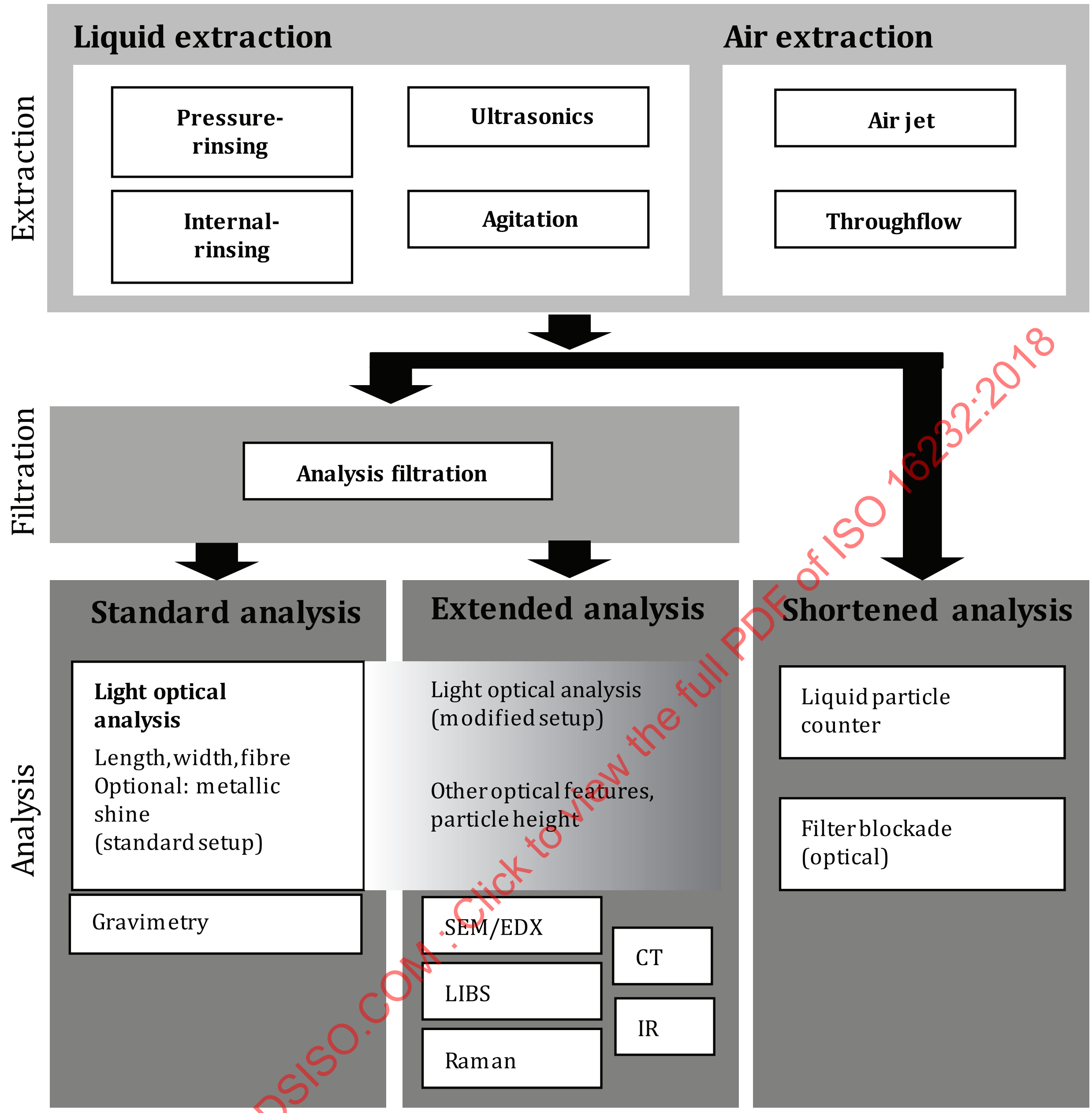


Figure 1 — Inspection methods

To be able to compare inspection results the same test fluid, the same extraction procedure and also same parameter settings for the analysis instruments shall be applied.

Annex I describes possible procedures to be taken when the result of a cleanliness inspection exceeds the permissible value stated in the cleanliness specification.

5.2 Selecting the inspection method

With respect to Figure 1 the following shall be considered.

— Particle extraction: particles are detached from the component by means of a test fluid in a cleaning process. The extraction method is determined by the features of the test component(s), such as size, shape, accessibility to the surfaces requiring inspection, component material and type and adhesion of the contamination to be removed (see Clause 7).

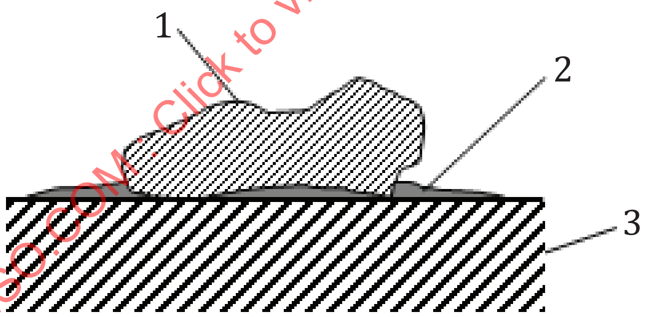
- Particle filtration: particles with a size relevant to the analysis are deposited on an analysis filter. The type of analysis filter is selected according to the size and quantity of the particles requiring analysis as stated, for example, in the cleanliness specification (see Clause 8).
- Particle analysis: relevant particle features are analysed in accordance with the information stated in the cleanliness specification, for example. Such features include aspects such as size, quantity, material composition and properties. Where no specific features are stated, the standard analysis procedure shall be applied (see 9.2).

NOTE 1 In the shortened analysis (see Figure 1) there is no filtration step. Once detached by the test liquid, particles are advanced directly for analysis, e.g. with a liquid particle counter.

The purpose of selecting an extraction method, deriving parameters for it and subsequently validating the method is to adapt and optimize a cleaning task. According to Sinner´s circle (see Figure A.2), the following parameters influence the extraction result:

- test fluid (chemical cleaning aspect);
- temperature;
- cleaning mechanism (extraction method with parameters);
- time (time the controlled surface is exposed to the extraction method).

A particle bonded to a component surface by forces of adhesion is used as a model for the cleaning purpose. The particle in Figure 2 could adhere, for example, because of Van der Waals forces, or alternatively capillary forces subsequent to the presence of humidity in the air or process media (coolants, lubricants or anti-corrosives). The purpose of the extraction method is to overcome these forces and detach the particle.



Key

1 particle

2 layer enhancing adhesion (humidity in the air, process media)

3 component

Figure 2 — Particle bonded to the component surface by forces of adhesion

NOTE 2 Burrs can detach from the component during the extraction procedure, which are then also included in the cleanliness inspection.

5.3 Start parameters

After selecting the extraction method (cleaning mechanism), which is essentially determined by the size, shape, material and location of the component surfaces requiring inspection, there is considerable options for variations regarding the specific parameters that can be chosen, e.g. diameter of the spray nozzle, flow rate, ultrasonic frequency, etc. In order to obtain comparable extraction (and thus also analysis) results, it is advisable to commence qualification tests using the start parameters recommended for the various methods (see Clause 7).

NOTE To optimize an extraction procedure for a specific component with regard to extraction time or particle detachment (without damaging the component surfaces concerned), the recommended start parameters can be modified provided they are documented appropriately.

5.4 Cleaning mechanism parameters

The extraction effect and even probability of damage to the component surface is primarily dependent on the parameters of the extraction method responsible for the mechanical action on the component (impulses, forces etc.). In order to select suitable parameters, sound knowledge of the test component, its manufacture and later usage may be necessary. However, if there are no requirements or experience known which justifies the implementation of specific extraction parameters, the start parameters recommended for the respective extraction method should be used.

For more details see Annex A.

5.5 Staff skills

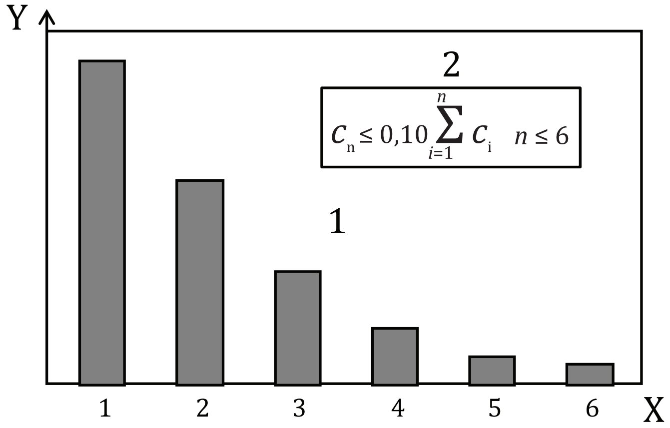
The cleanliness inspection is a very complex task including the understanding of cleanliness specifications, the selection and careful execution of extraction procedures, precise analysis and an accurate documentation. Therefore, well trained and highly motivated staff members are essential (see also Annex J).

6 Qualification tests and blank level

6.1 General

Qualification tests are carried out to define the appropriate extraction method for routine inspection. They represent the final step in the procedure that started in Annex A. The qualification tests are therefore the fourth element (time) in Sinner's Circle shown in Figure A.2. The qualification tests are carried out by means of a declining test, as illustrated in the extraction curve in Figure 3. The qualification and blank level tests shall be conducted and evaluated as described in 6.2, 6.3 and Annex B.

Qualification tests and cleanliness inspection are different tasks requiring separate test lots. For the purpose of an initial cleanliness inspection at least 2 test lots are needed, one for the qualification test and one for the subsequent routine inspection.



Key

X	consecutive extraction steps n
Y	cleanliness value C_i
1	extraction curve
2	declining criterion

Figure 3 — Cleanliness values determined by carrying out repeated extraction steps on a component (declining performance)

Determining blank levels and observing the corresponding permissible limits guarantees that the cleanliness inspection is performed under sufficiently-clean testing and environmental conditions. This ensures that any contaminants collected during the test procedure from sources other than the test component may not falsify the analysis result.

To verify that no particles are lost during the various steps of extraction, final rinsing of the extraction equipment, handling and drying of the analysis filter and analysis – with the consequence that they would be lacking in the analysis result - the recovery rate of test particles can be optionally determined.

When controlling cleanliness specifications in the customer-supplier relationship, extraction conditions shall be qualified according to this document.

Even in the case of an extended analysis, especially for microscopic analysis, qualification tests are mandatory, because any form of analysis demands an efficient and representative extraction procedure.

6.2 Qualification tests

6.2.1 Principle

In order to assess the cleanliness of a component correctly, an extraction method shall be effective. Therefore, it shall be proved that the maximum amount of detachable contaminant particles has been extracted from the test component in a cleanliness inspection. Unfortunately, there is no absolute method to determine the particle load actually present. Additionally, “norm-contaminated” products (i.e. in a defined and reproducible state of contamination), which cover the full spectrum of cleanliness-relevant automotive components as far as their geometry, material, particle content and adhesion of contaminants are concerned, simply do not exist. Consequently, to evaluate the efficacy of an extraction method, declining tests are performed.

The decline in cleanliness values is assessed in order to derive appropriate extraction conditions for a component or a group of similar components (component family) and to define an “inspection specification”.

By carrying out repeated extractions on a component, it can be determined whether the particulate load detached each time decreases and thus whether the extraction method applied is suitable (see

Annex B). The declining criterion (see Figure 3) determines whether the particulate contamination has been effectively detached. This identifies the point on the extraction curve where the current value of extracted contamination is less than 10 % of the total quantity of contamination extracted in all previous extraction steps including the current step. This point is used to develop the routine inspection procedure.

At the same time as performing the declining tests, it can also be determined whether the extraction method attacks the component, causing particles to be detached from the component surface that are not relevant contamination (see also B.2), e.g. burrs, coatings or component material.

The routine inspection procedure is qualified and specified at least once for each component or component family. Should any modifications be made to the component (constructional) or to the production process that could affect the removal of contamination from the component using the existing extraction procedure (different component geometry or different type, quantity or method of adhesion of contamination), the qualification tests shall be repeated.

Qualification tests shall never be repeated on a component which has already been analysed.

The qualification procedure comprises the following steps (see Figure B.1):

a) declining test (see 6.2.3 and Figure B.1);

b) development of the routine inspection procedure (6.2.3);

c) optional: validation of the routine inspection procedure by carrying out a double inspection;

d) determination of blank levels (see 6.3);

e) optional: test particle recovery (see 6.2.3.2).

If the blank level and declining performance meet the requirements described in Clause 6, the procedure developed is documented as a qualified inspection specification for the routine inspection.

NOTE Based on the inspection specification derived for a similar component (geometry, material, coating, etc.), in some cases the qualification test can be reduced to steps c) and d).

6.2.2 Materials and equipment

Cleaning mechanism, test fluid, and temperature of the extraction procedure, as well as filtration and analysis method are determined as described in 5.2.

Declining tests are evaluated using the analysis method that is also applied to validate the cleanliness specification (e.g. gravimetry and light optical analysis).

At the beginning of the declining tests, the start parameters recommended for the respective extraction method are set and implemented — or other parameters, if they are more suitable and strictly documented (see Clause 7).

An adequate supply of identical components (same model, same manufacturing lot) shall be available in the event that the declining test needs to be repeated.

Ensure that the test component is delivered to the test laboratory in accordance with Clause 11.

6.2.3 Procedure

6.2.3.1 Declining test

Figure 3 shows an example of an extraction curve obtained if a suitable extraction procedure is utilized.

a) Determine a test lot size estimated to be suitable.

b) Clean testing equipment as required and validate the cleanliness level by performing a blank level measurement.

Because the later routine procedure has not been determined at this stage at the start of the qualification, the blank level cannot yet be determined using the final extraction volume or extraction time. The parameters used to determine the “provisional” blank level are initially selected by the person carrying out the inspection. The respective start parameters may be useful guides to help choose these as well as the experience gained by the inspecting person with regard to the inspection equipment used. Additionally, an equipment blank level is often known, i.e. a blank level that can be attained with the extraction setup using a defined procedure and volume of liquid or period of time. If the equipment blank level is used as the “provisional” blank level for the qualification tests, it shall also fulfil the blank level criteria.

c) Using the start parameters, carefully perform the extraction procedure on the same component or test lot six times in succession and determine the cleanliness value C_i for each of the six extraction steps. The extraction setup shall be subsequently rinsed using an effective final rinsing procedure after each extraction step.

d) Then, for each cleanliness value C_i, calculate the ratio between the cleanliness value under consideration and the total of all cleanliness values including the current extraction step. The declining value for step n of declining test is calculated using Formula (1).

$$\frac{C_\mathrm{n}}{\sum_{i=1}^{n} C_i} = D_\mathrm{n} \qquad (1)$$

e) If the declining value D_n is ≤0,10 (10 %), the declining criterion has been achieved.

$$C_n \leq 0{,}10 \sum_{i=1}^{n} C_i \quad n \leq 6$$

f) Once the declining criterion has been attained, the details for the routine inspection procedure (i. e. inspection specification) are determined (see 6.2.3.2).

g) If the declining criterion is not achieved after six extraction steps, extraction parameters need to be modified, or a different extraction method chosen, and the declining test repeated with **a new component or test lot**. It may be necessary, for example, to reduce the strength of the extraction process if the component material is damaged by the extraction. If components have very low particle loads (i.e. are very clean to start with), it may be very difficult to identify a decline. In such cases, it may be wise to increase the number of components of the test lot (test lot size).

h) If, despite optimizing the extraction parameters, the declining criterion still cannot be attained, the component is regarded as an exceptional inspection case (see 6.4).

The six cleanliness values C_i obtained from each respective extraction step can be illustrated in a graph as an extraction curve, as shown in Figure 3.

A range of curve shapes that may be obtained in the course of declining tests is shown in B.2 which also describes methods for interpreting them.

To determine declining values and assess the declining performance (point d) and e)) the cleanliness values C_i stated in the cleanliness specification of the component are used. They are:

- in the case of gravimetric information, the residue mass;
- in the case of a light-optical or other method to determine particle size distribution, the particle counts in the respective particle size classes. To simplify the determination and representation of declining performance, all particle size classes included in the cleanliness specification can be added together. However, care shall be taken that there is a decreasing trend in each size class.

All particles with the exception of (textile) fibres are included in the declining test.

If fibres are part of the cleanliness specification they can be included in the declining test.

If no cleanliness specification has been defined, or if only a maximum permissible particle size is specified (e.g. no particles >x µm permitted), all particle size channels are considered in the declining test that can be reliably measured with the magnification and pixel resolution selected in the light-optical analysis method (see 9.2.3).

NOTE 1 Under certain circumstances, it can be necessary to consider particle size classes which are not included in the cleanliness specification by adding size classes with at least 20 counting events.

NOTE 2 To obtain further information, the declining performance of individual particle size classes can also be assessed without adding them together.

6.2.3.2 Developing the routine inspection

From the findings in 6.2.3.1, the appropriate routine inspection conditions are derived for the respective extraction method.

- Pressure-rinsing: the required total pressure-rinsing time, as well as the related total volume calculated on basis of the flow rate.
- Ultrasonic: the required total length of time in the ultrasonic bath.

- Internal rinsing: the required total rinsing time as well as the related total volume calculated on basis of the flow rate.
- Agitation: the required total agitation time as well as total number of shaking movements calculated on basis of the agitation frequency.
- Air jet extraction: the required total time the component is cleaned with the air jet.
- Air through-flow extraction: the required total time air flows through the component.

If *n* single extraction steps are required for the qualification (to achieve the 10 % criterion), the routine inspection procedure is carried out over the (n − 1)-fold total extraction time minimum. The aim of a qualified routine inspection is to extract at least 90 % of detachable contamination from the component within the period of time it is subjected to the extraction process.

The final rinsing procedure to remove residual particles from the extraction devices remains unchanged. The volume of liquid or final rinsing time used in the procedure is independent of the extraction of particles from the component and therefore not multiplied by (n − 1). In order to determine whether the selected final rinsing procedure is effective, the final rinsing procedure can be repeated or test particles utilized, for example (see Annex C).

Once qualified and determined, the routine inspection (see Figure B.3) is utilized to extract particles from components in order to validate the component cleanliness and thus also cleanliness specifications defined between the customer and the supplier.

6.2.3.3 Verification of the routine inspection/double inspection (informative)

To further verify a qualified routine inspection procedure, a double inspection can be performed.

- The routine inspection developed in the preceding section is carried out twice on another non-inspected test lot and the cleanliness values C_1 and C_2 determined.
- If the second cleanliness value is lower than/equal to 30 % of the sum of both cleanliness values, the applicability of the routine inspection conditions developed is verified and can be documented in the inspection specification.

  $$C_2 \leq 0{,}30\ (C_1 + C_2)$$

- If the criterion is not fulfilled, it should be investigated whether it is possible to implement more appropriate extraction parameters.

6.3 Blank level

6.3.1 Principle

The use of extraction methods to inspect the cleanliness of components involves the risk that not only particles from the test component are included in analysis results but also extrinsic particles are introduced into the test. If the proportion of extrinsic particles (blank level) is too high, this could lead to a faulty assessment of component cleanliness. The blank level represents the total value of contamination that does not originate from the component. Origins of such interfering particles include:

- test fluid,
- extraction equipment (baths, basins, tubing, valves, etc.),
- all objects coming into contact with the component and test liquid,
- handling processes during preparation, extraction and analysis,
- environment and staff.

The cleanliness grade of the test environment shall be appropriate for the required cleanliness state of a component.

If the required blank level is not achieved, the cause shall be investigated among the above-mentioned influencing factors.

NOTE Tips on optimizing cleanliness suitability, which can also be transferred to a cleanliness laboratory, are described in Reference [8].

To ensure that the blank level does not have an adverse influence on the test result,

- it shall be correctly determined, and
- it may not exceed a specific maximum ratio as far as the required/derived component cleanliness is concerned.

A blank level test is not mandatory before each routine inspection. However, a blank level test shall be performed, for example, if there is a risk that the cleanliness state of the extraction equipment is unsuitable or unknown, e.g.:

- if extraction equipment has not been used for a prolonged period of time, if stopped overnight, over the weekend or for longer periods;
- on changing from a component inspection with high particle content to a component with lower particle content, because there is a major risk of transferring an impermissible quantity of particles to the clean component.

6.3.2 Deriving blank levels

The permissible blank level is calculated on the basis of a component's cleanliness values.

The blank level may not exceed 10 % of the required/expected cleanliness values for the component. Further conditions apply for the blank level regarding the maximum permissible particle size (see Table 1).

If the cleanliness values of a component are not known — e.g. on inspection for the first time — the basis for calculating the blank levels can only be attained after performing the qualification tests.

Table 1 — Blank level criterion and examples

Cleanliness specification	Blank level criterion	Example			
		Cleanliness specification		Blank level derived	
Residue mass	10 % of permissible or measured residue mass[a]	7 mg		0,7 mg	
Particle size distribution (also CCC)	10 % of the quantity of permissible or measured particles in each size class (without exception, digits after the decimal point shall be rounded off)	Size channel [µm]	Permissible quantity	Size channel [µm]	Quantity permitted in blank level
		$100 \le x < 150$	90	$100 \le x < 150$	9
		$150 \le x < 200$	28	$150 \le x < 200$	2
		$200 \le x < 400$	12	$200 \le x < 400$	1
		$400 \le x < 600$	0	$400 \le x < 600$	0
Largest permissible particle	Maximum permissible particle size is halved; no particles permitted in this size class or larger	No particles >500 µm permitted		500 µm/2 = 250 µm, lies in the size class H $200\ \mu m \le x < 400\ \mu m$ Therefore: no particles ≥ 200 µm permitted in the blank level	
None	No particles >50 µm permitted[b]	—		No particles >50 µm permitted	

[a] Take resolution of balance into account, see 9.2.2 for more details.

[b] Only applies if no information is available in terms of a cleanliness specification, analysis results from the component, experience with similar components.

The blank level always refers to the specific component inspection. In order to be able to calculate permissible blank levels where cleanliness specifications are stated per controlled surface or wetted volume, or in the case of coded cleanliness specifications, the permissible quantity of contamination for the test lot analysed (one or more components) shall always be calculated first.

If different cleanliness features are combined in one cleanliness specification, e.g. residue mass and particle size distribution, the blank level shall be calculated and attained for all features specified.

If two possible blank levels are determined, e.g. with a specified particle size distribution and a maximum permissible particle size, both blank levels shall be fulfilled.

When validating cleanliness specifications, it is not permitted to subtract the calculated blank level from the analysis result of a component cleanliness inspection.

6.3.3 Materials and equipment

To determine blank levels, exactly the same equipment, materials, settings and parameters for extraction, filtration and analysis shall be used as defined in the inspection specification for the component under test.

6.3.4 Procedure

a) Calculate the permissible blank level either on the basis of the cleanliness specification or on the basis of the cleanliness values determined in 6.2.3.

b) Perform exactly the routine inspection developed in 6.2.3 without a component and determine the cleanliness value (= blank level).

c) Check that the attained blank level is within the permitted limit.

If blank level criteria are not fulfilled, elements of the testing equipment or environment are unsuitable and require optimization.

Another reason why blank levels cannot be attained may be due to an inadequate final rinsing step; as a result, particles remain in the testing apparatus and are only detected in the course of the blank level test.

6.4 Exceptional cases

If it is proven that the qualification criteria for the declining and/or blank level cannot be fulfilled after the repeated optimization of extraction conditions, the inspection procedure best-suited for the task can be specified for the routine inspection in the customer-supplier relationship.

Before a cleanliness inspection procedure (failing the qualification criteria) is declared to be an exceptional case, firstly all possible causes of error shall be investigated and remedied, e.g. incorrect handling, particles transmitted from non-relevant component areas, etc.

The following may prove to be exceptional cases.

- Active components and assemblies which need to be operated during extraction (e.g. valves, pumps, injectors, etc.). In such cases, detached contaminant particles and particles generated during break-in may overlie one another with the consequence that the decline in particulate contamination cannot be observed separately.
- Components that release particles, e.g. from the component material or from sacrificial coatings.
- Extremely clean components, with which an assessment of the declining performance cannot be achieved despite increasing the test lot size and optimizing blank levels.

For further details see Annex B.

7 Extraction methods

7.1 Principles

Because of their geometry and surface characteristics, functionally-relevant components in automotive systems cannot usually be fully inspected directly for the presence of particles (e.g. using a direct visual or microscopic technique). In such case, particles are removed from components by extraction or sampling.

The aim is not just to detect a representative quantity of particles but to ensure that the maximum number of detachable particles is detected, including the reliable detachment of single critical particles. Procedures for qualifying and validating this requirement are the same as those described in Clause 6.

Depending on the test component, several different extraction methods may need to be implemented. It may also be necessary to activate parts of a component mechanically in order to inspect the controlled surfaces concerned.

In some cases, the packaging coming directly into contact with the component may also need to be included in the cleanliness inspection.

A component may possess controlled surfaces that have different cleanliness specifications and which therefore shall be inspected separately.

After detaching the particles from controlled surfaces using a liquid or possibly air (see also 5.2), the contamination is deposited on an analysis filter membrane, which is then investigated using a standard and/or extended analysis method. In the case of shortened analysis, no filtration step is required and particles are analysed directly in the fluid (usually liquid).

Because the result of a cleanliness inspection is highly dependent on the care with which the always manual extraction procedure is performed, it is essential that inspection staff are well-trained. The inspection specification shall contain a detailed description of the extraction procedure. To design and develop applicable extraction procedures, the principal settings and configurations described in Annex D shall be considered.

7.2 General requirements of extraction equipment

All items of the extraction setup coming into contact with the test fluid, and all surfaces thereof whose cleanliness could affect the result of the component cleanliness inspection, shall be constructed and kept clean in such a way to ensure that the permissible blank level is not exceeded. The following points shall be taken into account in their design and operation:

- low degree of surface roughness;
- high chemical and mechanical resistance;
- no particle traps, such as dead zones or undercuts;
- easy to clean;
- non-magnetic;
- not electrostatically chargeable;
- tilted surface and angled piping (as short as possible) from extraction cabinet or collection funnel to analysis filtration;
- no particles emitted from the apparatus (or only particles which are not relevant to the component inspection), including when components are activated, moved or driven.

To facilitate achievement of and compliance with blank levels, the surfaces of extraction setup in contact with fluid that are located between the purification filter of the test fluid and the analysis filtration unit should be kept as small and as geometrically simple as possible.

7.3 Preparatory steps and post-treatment of test components

7.3.1 General

Although the preparation step should be performed near the test environment, if there is a risk of particles being generated or released when preparing components for a cleanliness inspection, it should be carried out at a different suitable workplace.

7.3.2 Unpacking

Test components shall be supplied for inspection in an appropriately clean manner. In many cases, suitable packaging is required. From the moment it is removed from the respective manufacturing step and arrives at the cleanliness inspection laboratory, the packaging protects the test component against re-contamination during storage or transportation.

When opening packaging and removing test components, care shall be taken to avoid contaminants adhering to the outside of the packaging from being released and reaching the controlled surfaces of components. Appropriate action includes miscellaneous options such as:

- cleaning external packaging surfaces before opening;
- changing gloves between opening the packaging and removing the test components;
- ensuring that packaging is opened by one person and test components removed by another;
- onion skin principle (two layers of packaging and lock concept).

7.3.3 Clarifying which surfaces require inspection

In many cases, only part of the surface area of a component is relevant to cleanliness or has been designated a cleanliness specification (e.g. only the interior of the casing for an electronic device).

Alternatively, more than one area of a component is relevant and these have different cleanliness requirements (e.g. refined oil inlet and outlet). The following needs to be specified.

- The exact location of the controlled surface(s) to derive further steps, e.g. possible disassembly or other preparation processes, and plan the extraction strategy (e.g. sequence of the various extraction steps, pressure-rinsing procedure, etc.).
- The exact size of the controlled surface(s). This is especially important if the cleanliness specification is normed or coded, e.g. per 1 000 cm^2 or 100 cm^3. In such cases, the permissible quantity of contamination for the test component is calculated using the actual size of the controlled surface, and subsequently used to derive the permissible blank level for the test. Additionally, when developing a declining test, the required quantity of test liquid can be determined on the basis of the start parameters.

7.3.4 Preparatory steps

Where controlled surfaces are not the full size of the component, or where a component possesses several relevant controlled surfaces with different cleanliness specifications (see also 7.3.3), it may be necessary to isolate the various component areas from one another. In this way, test liquid cannot inadvertently come into contact with another controlled surface during the procedure and transfer particles to it that are not relevant.

Areas not requiring inspection shall be isolated, for example by plugging openings, coating, masking, sealing or marking them. Isolation measures shall be carried out in an appropriately clean manner and used materials should not affect the cleanliness inspection results.

Design and material of isolation items shall be cleanliness suitable.

If non-relevant areas of a test component do not require pre-cleaning, care shall be taken to ensure that no particles or other substances are transferred to the relevant controlled surfaces or removed from them.

For the extraction, it may still be necessary to fix the test component in mounting, lifting or sampling devices, or to use adapters for items such as rinsing lines, drives or electrical connections. This may also cause particles to be generated or released. Consequently, the same recommendations apply as those mentioned in 7.3.5.

In some cases, it can be useful to design adapters for components in bright colours. If coloured particles then appear in the analysis, the particles originate from the adapter and are not due to an inadequate cleanliness level of the test component.

7.3.5 Disassembly

In some cases, it may be necessary to partially or fully dismantle the test component before the cleanliness inspection in order to gain access to the functional surfaces relevant to the extraction.

During disassembly, there is a high risk that particles be generated and released, e.g. from joints. To prevent particles generated during disassembly from reaching component controlled surfaces, disassembly tasks shall be carefully planned and performed in an appropriately clean manner. Particles originating from disassembly steps shall be removed (e.g. by suction cleaning or wiping) the moment they are generated/released or immediately afterwards. All tools and auxiliary equipment shall be kept clean and designed with low abrasion levels.

There is also a risk during disassembly that particles from external surfaces that are not relevant to the inspection are transferred to internal surfaces that are relevant to component cleanliness. This can essentially be avoided by cleaning external surfaces before carrying out the disassembly process.

Due to the risk of the cleanliness state of controlled surfaces being altered by disassembly processes, disassembly steps shall be carefully documented; alternatively, precise instructions for disassembly shall be given.

7.3.6 Demagnetization

Magnetism causes magnetizable particles to adhere more strongly to the test component. Therefore, ferromagnetic test components should be assessed for residual magnetism and, where necessary, demagnetized before commencing tests.

EXAMPLE The non-critical residual magnetism value of components of diesel injection systems in contact with fluid is 2,5 Gauss or 200 A/m.

Machining steps during a component's manufacture may cause it to become magnetized. If a product requires demagnetization, it shall be documented. Features relevant to the function of a component may be destroyed by demagnetization processes.

Components which cannot be demagnetized for technical reasons are usually exceptional cases; these shall be specified in the customer-supplier relationship. In such cases, extraction with liquid or air is generally only suitable to a very limited extent and methods highly-specific to the component concerned need to be applied in order to detach particles; such methods are not addressed in this document.

7.3.7 Post-treatment

It shall be documented whether a tested component shall be scrapped or returned to the production line after inspection. Any post-treatment steps, such as removing residual test liquid, drying the component or applying a preservative shall be specified.

7.4 Liquid extraction

7.4.1 General

As described in Annex A, liquid extraction is suitable for the majority of functionally-relevant components encountered in the automotive industry.

7.4.2 Test liquid

The test liquid (and final rinsing liquid) shall be compatible with the component as well as with the extraction and filtration equipment, including all seals and filters used.

As far as the cleaning effect is concerned, the test liquid is mainly selected in dependence on the component material and the characteristics of the contaminants expected and with a suitable kinematic viscosity e.g. $\nu \leq 5$ mm^2/s. Among others, these are dependent on the history of the component and its manufacturing process (see also D.1). Particles relevant to the inspection may, for example, be bound in assembly greases or preservatives.

If the test liquid and final rinsing liquid are not adequately clean, purification filters shall be used.

WARNING — With aqueous test liquid, for example, active cleansing substances present in the test liquid could be removed by the filter.

All volumes of liquid used in the extraction and final rinsing steps shall be included in the analysis.

In order to re-use a test liquid (e.g. in circulatory systems), it shall be treated so that it does not impair the quality of inspection results. The following points should be taken into account.

- The physical/chemical cleaning effect of the liquid may not be affected, e.g. by the depletion of detergents or the consumption of active chemical ingredients.
- No substances may be added which could impair the inspection results; e.g. greases or preservatives.
- No substances may be added which could damage the inspection equipment or component; e.g. water, acids, alkalis.
- Viscosity of blended liquid might change during usage.

Annex K considers risk assessments for tasks carried out in the laboratory, in particular with the use of cold cleaners for the extraction process.

7.4.3 Pressure-rinsing

7.4.3.1 Principle

Pressure-rinsing describes the localized application of a suitable test liquid to a component via an open jet. The cleaning effect is mainly due to the momentum transferred by the jet on impacting on the test component. As a rule, a rinsing effect also takes place as the test liquid runs off the component.

The method is suitable for cleaning external geometries and easily-accessible inner geometries. If coarse-meshed baskets or filters are used, small components with simple geometries can also be extracted one at a time or in bulk (see also Figure D.2).

With most applications, a full-jet nozzle with a round cross-section is utilized. However, depending on the geometry of the test component, it may be more practical to use different-shaped nozzles (see Figure D.1).

Pressure-rinsing tools (nozzles) can also be implemented to clean the inner surfaces of components if they are applied to the aperture of a boring or tubing. Care shall be taken to ensure that the complete internal area is filled and wetted with test liquid. The extraction effect in this case is no longer that of pressure-rinsing but rather of internal rinsing because the surface is not impacted by an open jet (see 7.4.5).

The most important parameters influencing the efficacy of the pressure-rinsing method are the:

- characteristics of the test liquid,
- flow rate combined with the cross-sectional area of the nozzle,
- geometry of nozzles and lances,
- distance away from the object and the angle of the jet,
- sequence implemented when extracting the object surfaces,
- time per surface or forward feed, and
- repetition rate of extractions per controlled surface.

NOTE The pressure of the jet can only be used to characterize its cleaning effect to a limited extent. Although the flow rate of a nozzle with a given nozzle diameter is proportional to the pressure at the nozzle, it is often difficult to measure the pressure directly at nozzle level. Liquid feed pressures measured in other areas of the system (e.g. in a pressure tank or behind a pump), can vary significantly from the actual pressure at nozzle level and are therefore unsuitable as parameters. On the other hand, the flow rate remains constant regardless of where it is measured. Even if the flow rate is not measured via a sensor in the liquid feeding system, it can be simply calculated by means of volumetric measurement (e.g. filling rate of a beaker into which the pressure-rinsing jet is directed) and is thus suitable as a parameter.

In the cases of nozzles with round outlet cross-sections and a non-widened cylindrical jet (or several such nozzles in a pressure-rinsing tool), the efficacy of the pressure-rinsing jet is much less dependent on its distance away from the component as would be the case with a widening flat-jet nozzle (fan nozzle). This makes it easier to perform a defined extraction on the test component (see also D.2, Figure D.1 a), d) and e)).

The efficiency of the pressure-rinsing method is very dependent on the extraction steps that are generally carried out manually. The pressure-rinsing procedure suitable for the respective test shall be documented in the inspection specification.

If the pressure-rinsing method is implemented using a solvent as a test liquid, aerosols could be generated. These aerosols may form a combustible mixture even in cases where the flashpoint of the test liquid is much higher than the testing temperature/room temperature. If there is a risk of aerosol formation, the extraction should be performed in a suitable extraction cabinet which does not contain any potential ignition sources.

To find out when aerosols may occur, refer to Figure D.4.

Figure D.4 can be used as a guide to determine the combinations of nozzle diameter and flow rate which may lead to aerosol formation. The graph was developed for round full-jet nozzles. Due to the fact that aerosol formation is generally to be expected if flat-jet nozzles (fan nozzles) or other nozzles that widen and mist the pressure-rinsing jet are used, it is essential that all relevant work safety directives are followed.

7.4.3.2 Start parameters

In order for extraction equipment to be used for as many components as possible, pressure-rinsing start parameters shall be selected (see 5.3) so as to generate a jet with a maximum cleaning efficacy without the risk of aerosol formation (see Table 2).

Table 2 — Start parameters for pressure-rinsing

Parameter	Start value/Condition
Nozzle shape	Round full-jet nozzle
Nozzle diameter	2,5 mm
Flow rate	1,5 l/min
Distance from component	Max. 15 cm
Pressure-rinsing volume/controlled surface	5 ml/cm^2
When inspecting larger surfaces, several nozzles (e.g. combined in a pressure-rinsing tool) can be used together to reduce the extraction time. However, care shall be taken to ensure that the above-mentioned parameters apply for each nozzle aperture. Due to the way existing extraction equipment is constructed, it may not be possible to avoid the risk of the operator being sprayed with liquid reflected back from the component or of detached particles from being lost if the recommended parameters are used. In these justified cases, which shall be documented, the initial flow rate of the pressure-rinsing liquid can be decreased to 1,0 l/min.	

When using pressure-rinsing for small components that shall be inspected in bulk it is recommended that the test lot contains a minimum controlled surface of 200 cm^2 (see Figure D.2 c)).

As part of the qualification tests/declining tests (see Clause 6), the final pressure-rinsing volume per controlled surface is determined for the routine inspection in dependence on after which extraction step the declining criterion is attained. If the declining criterion is not achieved using these start parameters or if other parameters are more suitable and strictly documented, start parameters may be modified.

7.4.3.3 Materials and equipment

The materials and equipment used in the extraction step shall comply with the general requirements of extraction equipment described in 7.2.

a) Test liquid, kinematic viscosity $\nu \leq 5$ mm^2/s.

b) Pick-up for component: e.g. rest, tweezers or mount for single objects; coarse-meshed basket for small components; manipulators for large components.

c) Extraction setup consisting of:

  - pressure-rinsing tool(s): e.g. nozzle pen, which can be controlled manually during the component extraction process and final rinsing of the extraction setup, lances for component inner surfaces, or similar;
  - media supply with container for test liquid, purification filter, pump or pressure supply.

d) Collection equipment: e.g. pressure-rinsing cabinet, funnel or container to collect extraction liquid.

e) Suction unit (optional): to remove residual liquid from component cavities.

f) Where required, graduated vessel to measure volumes: e.g. beaker or measuring cylinder and stopwatch to control extraction times or calculate the flow rate if volumetric measurement is used for the pressure-rinsing step.

Laboratory wash bottles shall not be used for component extraction by pressure-rinsing, because it is not possible to attain a defined and stable pressure-rinsing parameter and is thus not a reproducible extraction step. However, in many cases, laboratory wash bottles are suitable for the final rinsing of extraction or filtration equipment.

7.4.3.4 Procedure

The following procedure shall be adapted to the features of the test component.

a) Procure all resources required for the test.

b) Pre-clean all surfaces of the extraction setup in contact with fluid; where appropriate, determine blank levels.

c) Prepare the test component as described in 7.3.

d) Place the test component in the extraction apparatus; if necessary, position the test component in such a way so as to enable the liquid to flow easily into the collection container or collection funnel.

e) Perform the pressure-rinsing procedure and adhere to all parameters, times and exact pressure-rinsing sequence. The careful execution of the procedure is crucial to the result of the inspection. Avoid loss of liquid or wetting component areas that are not relevant to the test.

f) Remove residual liquid (if required, repeatedly) from all areas of the component where liquid and particles can accumulate. Use suction apparatus to remove liquid from objects with cavities that are difficult to access (this liquid shall be included in the analysis).

g) Where applicable (if specified): sample the inner surfaces of packaging (this liquid is also included in the analysis).

h) Carry out a final rinse on all areas of the extraction equipment (chambers, mounting devices, etc.) that were in contact with the test liquid (this liquid is also included in the analysis). A suitable procedure should be elaborated, tested and defined for this final rinsing step.

i) Transfer all the liquid for filtration and analysis.

j) Fill out the inspection report.

7.4.3.5 Documentation

See Clause 10.

7.4.4 Ultrasonic vibration

7.4.4.1 Principle

Extraction by ultrasonic vibration describes the action of mechanical oscillations ranging between 20 kHz and 400 kHz exerted on the surface of a test component via a liquid. The cleaning procedure is performed in immersion baths fitted with ultrasonic oscillating elements.

The particle-detaching effect of extraction by ultrasonic vibration is caused by high pressure peaks that form when cavitation bubbles implode.

The ability of ultrasonic vibration to detach contaminants is due mainly to the ultrasonic frequency and changes in ultrasonic pressure, which are influenced by the ultrasonic output and geometric features. As a rule, the higher the ultrasonic output and the lower the ultrasonic frequency, the stronger the physical cleaning forces become.

In the event that too-high quantities of particles are detached from the component surface as a result of unsuitable ultrasonic parameters, this can be corrected by setting a higher frequency or lower output. Special care shall be taken with cast iron because graphite may be released. Care is also required with cast aluminium surfaces, painted or coated surfaces and sintered materials, as the selection of unsuitable ultrasonic parameters can cause material damage with the result that non-relevant particles are released from the component surface.

Ultrasonic baths are suitable for inspecting external geometries as well as internal surfaces, provided the sound waves can penetrate component apertures with sufficient intensity. The smaller the openings for the sound waves and the larger the inner cavities become in proportion, the poorer the cleaning effect inside a component. Therefore, when extracting contaminants from the interior and exterior of a component at the same time, the cavitation threshold inside the component shall be exceeded without the component material on the outside being attacked.

Ultrasonic baths are especially suitable for extracting contamination from small components that are difficult to handle individually because they can be inspected in bulk. The use of small ultrasonic extraction baths or beakers reduces the total surface area wetted during the extraction step, which has a positive impact on achievable blank levels.

Ultrasonic vibration can also be used to enhance internal rinsing procedures. Here, the interior of the test component, which is generally pipe-shaped (no sound-absorbing materials), is subjected to a through-flow of test liquid and additionally immersed in an ultrasonic bath and treated externally with oscillations.

The main parameters influencing extraction by ultrasonic vibration are:

- characteristics and temperature of the test liquid,
- ultrasonic frequency,
- ultrasonic density,
- arrangement of ultrasonic oscillators in the bath,
- orientation of the component in relation to the ultrasonic source,
- extraction time, and
- movement of the component.

7.4.4.2 Verifying performance features of ultrasonic baths

To assess relative stability over time (aging) or to compare different ultrasonic baths, the efficacy of the ultrasonic system can be verified.

This is achieved for example by quantitatively determining the perforation of a sheet of aluminium foil by cavitation. The procedure shall be carried out under defined conditions (foil thickness, location and orientation of the foil in the bath).

WARNING — Erosion of aluminium foil may contaminate the ultrasonic bath.

NOTE A simple method to check the ultrasonic energy is the use of a commercially available indicator liquid in vials which is applied e.g. in pharmaceutical industry. After adding the vial to the ultrasonic bath a colour change shows if the ultrasonic bath is working.

7.4.4.3 Start parameters

The start parameters for extraction by ultrasonic vibration shall be carefully selected (see Table 3) in order to avoid highly-aggressive low ultrasonic frequencies, achieve a good cleaning effect with the power density set without subjecting the component to excessive stress and also to attain short start times for the declining tests (see 5.3.)

Table 3 — Start parameters for ultrasonic vibration

Parameter	Start value/Condition
Ultrasonic frequency	35 kHz to 40 kHz
Power density	10 W/l
Time	30 s to 60 s

The power density is set via an output regulator on the ultrasonic bath (if available) or via the filling quantity of the bath (without the component). In general, double half-wave devices are used. The power density documented shall correspond with the effective power density and not the peak power density.

When using ultrasonic vibration on small components that are inspected in bulk, the test lot shall contain sufficient components so as to achieve a minimum controlled surface of 200 cm^2 (see Figure D.5 a) and b)).

During the qualification tests/declining tests (see Clause 6), the required duration in the ultrasonic bath is determined for the routine inspection depending on after which extraction step the declining criterion is attained. If the declining criterion cannot be achieved with these start parameters or if other parameters are more suitable and strictly documented, the recommended start parameters may be modified.

7.4.4.4 Materials and equipment

The materials and equipment used in the extraction step shall comply with the general requirements of extraction equipment described in 7.2.

a) Test liquid, kinematic viscosity $\nu \leq 5$ mm^2/s.

b) Ultrasonic bath: the bath shall be made from stainless steel or other suitable material. Data and performance features shall be documented, especially the output, frequency and bath volumes or filling level. As a rule, the bases and/or walls of ultrasonic baths are fitted with transducers to optimize the homogeneity of ultrasonic distribution and cleaning effect.

   — Mounting device to secure the test component in the bath (no contact with the base or walls); materials shall be permeable to ultrasonic waves (e.g. baskets with very coarse mesh, no mounting devices made from compact absorbent plastics).

   — Where appropriate: beaker for holding small components and corresponding mounting device for beaker.

   — Device (manual/automated) to oscillate the components by half the wavelength vertically in relation to the surface emitting the ultrasonic waves. This compensates for inhomogeneities in the ultrasonic effect due to the formation of stationary waves (oscillation velocity ≤1 cm/s).

c) Where appropriate: additional containers (funnels, beakers) to collect the extraction liquid.

d) Pressure-rinsing test set-up for the final rinsing of components and equipment and/or for dispensing test liquid (see 7.4.3).

e) Graduated vessel to measure volumes; e.g. beaker or measuring cylinder.

f) Suction unit (optional): for removing residual liquid from component cavities.

WARNING — In addition to general safety requirements applicable to all extraction methods the following points shall be considered when using ultrasonic baths:

- **Due to the coupling of ultrasonic vibration in the test liquid and the resulting energy input, liquids may become hot if subjected to ultrasonic waves for an extended period of time. This shall be given special consideration and validated accordingly in cases where combustible liquids are used (temperature shall be kept 20 °C below respective flashpoint).**
- **During the application of ultrasonic vibration, do not place hands or any other part of the body in the test liquid (risk of embolus or destruction of skin cells!).**

7.4.4.5 Procedure

The following procedure shall be adapted to the features of the respective test component.

a) Procure all resources required for the test.

b) Pre-clean all surfaces of the extraction setup in contact with fluid; where required, determine blank levels.

c) Prepare the test component as described in 7.3.

d) Fill the extraction device with clean test liquid.

   1) For tests carried out directly in the ultrasonic bath: fill the bath and set the output to achieve the required power density in the bath (10 W/l with start parameters). If the output cannot be adjusted, fill to the level which corresponds with the desired power density.

   2) In a beaker: fill the bath with water and a small amount of cleaning agent/detergent (to improve the wetting capacity). The water only serves to transmit the ultrasonic waves to the beaker and is not later analysed. The power density can be adjusted using an output regulator or, if not available, by altering the filling level of the bath. The volume subjected to ultrasonic vibration, which shall be known in order to calculate the power density, is the volume of water plus the contents of the beaker. Fill the beaker with clean test liquid.

e) Place the test component(s) directly in the ultrasonic bath, or in recipients such as baskets, or in the filled beaker, which is also secured in the ultrasonic bath by a mounting device.

f) Commence the ultrasonic extraction step using the power density, frequency and time stated in the inspection specification. Move the component or beaker while the ultrasonic step is in progress.

   If the component shall be subjected to ultrasonic vibration from several sides or several positions, care shall be taken when changing its position (turning over, rotating, etc.) to avoid additional particles from being generated by this handling step.

   If the component or beaker is not moved slowly through the sound field of the ultrasonic bath (min ½ wavelengths of the sound waves in the bath), the cleaning effect may not be homogeneous.

g) Remove the test component(s), hold over the ultrasonic bath or beaker and carry out a final rinsing step with clean test liquid.

h) Where applicable, empty objects to remove any residual liquid and particles contained inside.

i) If required, extract particles from the inner surfaces of packaging (pressure-rinsing). The resulting liquid forms part of the extraction liquid.

j) Empty the beaker; alternatively, where technically possible, drain the extraction liquid out of the bath.

   When using laboratory ultrasonic baths, care shall be taken to ensure that no particles detached from the component remain in the baths. There is often a risk of particles sedimenting at the outlet of a bath where, for manufacturing reasons, there is often a gap or step hindering drainage. If taps or similar drainage elements are fitted to the ultrasonic bath, it shall be checked that these fixtures do not generate particles when used as this could falsify analysis results (blank level).

k) Then perform a final rinsing step on all surfaces wetted by the test liquid (e.g. beaker and/or baths) using a sufficient volume of test liquid. The resulting liquid is included in the analysis.

l) Transfer the total volume of liquid for filtration and analysis.

m) Fill out the inspection report.

7.4.4.6 Documentation

See Clause 10.

7.4.5 Internal rinsing

7.4.5.1 Principle

Internal rinsing describes the extraction of contamination by means of a flow of liquid passed through the inner surfaces of a component. In the process, the entire (or almost entire) test component is completely filled with test liquid. In order to ensure that particles are detached effectively from the walls of the component, the flow through the interior should be turbulent and not laminar. A pulsed through-flow further enhances the efficacy of this extraction technique.

Consequently, internal rinsing is a suitable extraction method for automotive components that will conduct fluids in later use. Examples include:

— passive components such as tubing, lines, channels, filters and heat exchangers, and

— active components needing to be actuated for the through-flow process, such as valves and injectors, or even driven, such as pumps.

With internal rinsing, at least one point of the test component is connected hermetically to a line holding test liquid. Once the fluid has flowed through the test component, the liquid can be removed from the component via one or more lines connected to it. The liquid can flow into a closed rinsing system or be allowed to drain off into an extraction basin without the influence of pressure.

Depending on the test component concerned, the set-up for extraction by internal rinsing can be very simple. For example, a pressure-rinsing set-up such as that illustrated in Figure D.3 can also be used to rinse fairly short sections of piping provided pressure-rinsing tools are replaced by rinsing devices. In the case of active components, internal rinsing test benches may be highly complex if components need to be powered, driven or subjected to high pressure.

Where components have wide cross-sections for connections, e.g. cooling systems of commercial vehicles, extraction by internal rinsing often reaches its limits because a major effort is required to construct the rinsing setup with the necessary volumes and flow rates of liquid. It may make sense in such cases to revert to extraction by agitation or pressure-rinsing.

The advantage of internal rinsing is that only inner areas are wetted, thus there is no risk of the test fluid flowing over external surfaces of the component that are not relevant to the extraction procedure. However, adapters for connections/rinsing tubing may generate and release particles due to friction. In the case of active components, there is a risk of excessive contamination being generated from running-in processes and frictional wear when the components are re-activated, which would falsify results.

The main parameters influencing the extraction method of internal rinsing are:

— characteristics of the test liquid,

— flow rate and rinsing time,

— geometry and model of the test component,

- pulsation and pulsation frequency, and
- type and frequency of movement (for active components).

7.4.5.2 Start parameters

As opposed to other liquid extraction methods, there are no meaningful start parameters for internal rinsing. This is because the flow rate required for a turbulent flow (calculable via the Reynolds number >4 000) is dependent upon the geometry of the test component and upon the test fluid used.

However, the minimum throughput volume shall be several times the inner volume of the component.

Table D.4 lists examples of flow velocities for two different test liquids (cold cleaners) with different viscosities. These rates are required for through-flows through tubes or lines of varying diameters in order to achieve a fully-turbulent flow and thus maximize the efficacy of the extraction.

7.4.5.3 Materials and equipment

The materials and equipment used in the extraction step shall comply with the general requirements of extraction equipment described in 7.2.

a) Test liquid, kinematic viscosity $\nu \leq 5$ mm^2/s.

b) Where appropriate, use a component mount: e.g. fixing device or clamp. For active components, use switching, actuating or drive devices, etc.

c) Internal rinsing apparatus consisting of:

   - adapters for filling the component, for the through-flow and for draining the liquid from component, and
   - media supply with container for test liquid, purification filter, pump or pressure supply; where appropriate, with pulsation mechanism.

   NOTE Internal rinsing systems can also be designed as vacuum rinsing systems, i.e. the liquid is not advanced through the component under positive pressure but rather sucked through using a vacuum device.

d) If required, use additional containers (funnels, beakers) to collect the extraction liquid.

e) Pressure-rinsing apparatus for final rinse of equipment with test liquid using g).

f) Graduated vessel to measure volumes; e.g. beaker or measuring cylinder.

g) Suction device (optional): to remove residual liquid from component cavities.

With a closed internal rinsing circuit, it makes senses to place the analysis filter directly behind the test component. However, this is not usually possible because the analysis filter has a high flow resistance which would prevent the flow velocities required inside the test component from being achieved. These flow velocities are necessary in order for the extraction step to be efficient.

Figure D.10 illustrates an example of an internal rinsing system.

7.4.5.4 Procedure

The following procedure shall be adapted to the features of the respective test component.

a) Procure all resources required for the test.

b) Pre-clean all surfaces of the extraction setup in contact with fluid; where required, determine blank levels.

c) Prepare the test component as described in 7.3.

d) Fit adapters to the test component for rinsing tubes; if required, position the test component in such a way so as to enable the liquid to flow easily into the extraction cabinet or collection container.

e) Where appropriate: with active components, connect power, switching or drive device to the test component.

f) Perform the extraction procedure on observing all internal rinsing parameters, times and, in the case of active components, operating parameters.

g) Empty residual liquid from component areas in which liquid and particles could collect (if required, repeatedly). For objects with cavities that are difficult to access, use suction equipment (the resulting liquid is included in the analysis).

h) Carry out a final rinsing step on all surfaces of rinsing setup, collection basins, etc., in contact with the liquid. A suitable procedure shall be elaborated, tested and defined for this final rinsing step (the resulting liquid forms part of the extraction liquid).

i) Transfer all the liquid for filtration and analysis.

j) Fill out the inspection report.

7.4.5.5 Documentation

See Clause 10.

7.4.6 Agitation

7.4.6.1 Principle

The test component is partially filled with test liquid and all openings sealed. By agitating this liquid, particles adhering to the inner controlled surfaces are detached and transferred to the liquid. The agitation process enables the liquid to exert a force on the particles in different directions and also enables dead zones and undercuts to be reached effectively by the liquid. Detached particles are then held in suspension.

The method is suitable for objects with cavities that can be accessed at least via one opening and whose size and mass allow the test areas to be adequately agitated. The method is not suitable for inspecting narrow inner geometries, such as tubes or capillaries, because the impulse created by agitation is too low to be effective.

The set-up chosen for the agitation procedure depends on the shape, size and mass of the component concerned.

The method is not suitable for foaming liquids.

Agitation can be carried out manually or with the aid of an automated device.

It is not useful to place components in a sealed vessel in order to agitate the vessel manually or by means of a vibrating table. This is because particles can be generated if the components strike against one another. Ultrasonic vibration or pressure-rinsing are preferred methods in such cases.

The main parameters influencing extraction by agitation are:

- characteristics of the test liquid,
- volume of test liquid used,
- duration, amplitude and frequency of the agitation step,
- number of fillings, and
- final rinsing of the test component.

7.4.6.2 Start parameters

The start parameters for extraction by agitation shall be selected (see Table 4) to maximize the extraction effect (see 5.3) whilst still allowing the process to be performed manually if desired.

Table 4 — Start parameters for agitation

Parameter	Start value/Condition
Filling volumes	30 % to 40%
Amplitude	approx. 30 cm
Frequency	1 Hz[a]
Time	15 s

[a] 1 Hz is one movement per second.

During the qualification tests/declining tests (see Clause 6), the agitation procedure or number of agitation movements is determined for the routine inspection depending on after which extraction step the declining criterion is attained. If the declining criterion is not achieved with these start parameters or if other parameters are more suitable and strictly documented, these start parameters may be modified.

However, a filling volume of 30 % to 40 % for components shall not be altered because a too-low or too-high filling quantity does not create the necessary mechanical action to generate an efficient cleaning effect.

7.4.6.3 Materials and equipment

a) Test liquid, kinematic viscosity $\nu \leq 5$ mm^2/s.

b) Pressure-rinsing apparatus for final rinsing of components and equipment and/or dispensing the test liquid (see 7.4.3).

c) Graduated vessel to measure volumes, e.g. beaker or measuring cylinder.

d) Funnel: for filling and/or emptying test liquid.

e) Appropriately sized abrasion-resistant seals/stoppers for the test component.

f) (Optional) Automated agitation device, e.g. vibrating table fitted with mounting device for the test component.

g) Clock to control the duration of the agitation step.

h) Sampling container, sampling vessel or analysis filter unit (dependent on application) to hold the extraction liquid.

i) Suction equipment (optional): to remove residual liquid from component cavities.

7.4.6.4 Procedure

The following procedure shall be adapted to the features of the respective test component.

a) Procure all resources required for the test.

b) Pre-clean all surfaces of the extraction devices in contact with fluid; where appropriate, determine blank levels.

c) Determine the internal volumes of the test component.

d) Prepare the test component in accordance with 7.3; special care shall be taken to clean the exterior of the component well and to seal all component openings with suitable clean, abrasion-resistant closing devices.

e) Remove the closing device from one opening that can be used to fully empty the test component.

f) Determine the volume of test liquid required, i.e. 30 % to 40 % of the volume of the interior of the test component.

g) Pour the test liquid into the object and seal the opening.

h) Perform the agitation step on observing all the specified conditions (frequency, amplitude, duration).

i) Remove one closing device and pour the liquid contained in the test component into a sampling vessel or filtration unit with the aid of a funnel. Avoid any loss of liquid or wetting of external component surfaces.

j) Carefully perform a final rinsing step on the interior of the test component. This final rinsing procedure may take the form of a further agitation step using test liquid or a pressure-rinsing step. The resulting volume of liquid forms part of the extraction liquid.

k) Ensure that the test component is fully emptied.

l) Then, using a sufficient volume of test liquid, carry out a final rinsing step on all surfaces wetted by the test liquid (sampling vessel, filtration equipment, etc.). The resulting liquid is included in the analysis.

m) Transfer all the liquid for filtration and analysis.

n) Fill out the inspection report.

7.4.6.5 Documentation

See Clause 10.

7.4.7 Dissolving

7.4.7.1 Principle

In cases where components have been treated with preservatives or grease, for example, effective particle extraction can only be achieved after first dissolving the layer of preservative or grease present. In consequence, a delayed decline may take place in the course of the qualification tests.

In such cases, **dissolving** can be carried out as a preparatory step before performing the actual extraction procedure. Here, the test component is first immersed in a suitable solvent (e.g. the test liquid used for the inspection) for an appropriate length of time without the aid of further physical cleaning effects, such as pressure-rinsing or ultrasonic vibration. Alternatively, components can also be completely filled and sealed if inner surfaces shall be inspected.

The volume of liquid used in the dissolving step as well as the final rinsing liquid for the vessel in which the dissolving step is carried out forms part of the extraction liquid and shall be included in the subsequent analysis.

The dissolving step can be speeded up during the extraction process by using a test liquid with a stronger chemical solvent action (verify compatibility with component).

The main parameters influencing the dissolving step are:

— characteristics of the dissolving liquid, and

— duration of the dissolving procedure.

7.4.7.2 Materials and equipment

a) Dissolving liquid (e.g. test liquid, or solvent with stronger dissolving properties).

b) Pressure-rinsing equipment to perform final rinsing step on the apparatus and/or to dispense the test liquid (see 7.4.3).

c) Where appropriate: funnel for filling or removing dissolving liquid.

d) If required: suitable abrasion-resistant seals/stoppers for the test component.

e) Clock to control the duration of the dissolving step.

f) Dissolving container, beaker or bath from pressure-rinsing or ultrasonic device.

7.4.7.3 Procedure

The following procedure shall be adapted to the features of the respective test component.

a) Procure all resources required for the test.

b) Pre-clean all surfaces of the dissolving container in contact with fluid, where required, determine blank levels.

c) Place the component in the dissolving container.

d) Fill the dissolving container with dissolving liquid until the component is fully immersed. Ensure that all surfaces are completely wetted, e.g. components with cavities.

e) Leave the component in the dissolving liquid during the dissolving period without the use of any further physical forces.

f) Remove the component and transfer to the next step of the extraction procedure.

g) Transfer the dissolving liquid to the filtration process.

h) Then, using a sufficient volume of test liquid, perform a final rinsing step on all surfaces wetted by the dissolving liquid (sampling vessel, filtration equipment, etc.). The resulting liquid forms part of the extraction liquid.

i) Transfer all the liquid for filtration and analysis.

j) Fill out the inspection report.

If the dissolving procedure is only carried out on the inner surfaces of a component, the described procedure shall be modified accordingly. The component shall be completely filled with dissolving liquid (ensure that all surfaces are fully wetted and that no air bubbles are present) and sealed during the dissolving step (e.g. with stoppers). The component is then emptied and the dissolving liquid transferred to the filtration process.

7.4.7.4 Documentation

See Clause 10.

7.5 Air extraction

7.5.1 General

Some components built into vehicles do not come into contact with liquids during their operation (and also some during their manufacture). Such components may even be damaged if they come into contact with a liquid, e.g. air filters, with the consequence that liquid cannot be used for the extraction process. Extraction using air is an alternative method for such components.

7.5.2 Air jet extraction

7.5.2.1 Principle

With this extraction method, particles are detached from the test component by means of a jet of clean, oil-free compressed air (see ISO 8573-1). The tools and procedure are similar to those required for liquid extraction by pressure-rinsing.

The method is suitable for external surfaces, or for the inner surfaces of components accessible with a jet of compressed air. It can be utilized, for example, to extract contaminants from electronic components, single components of engine air intake tracts that cannot be cleaned using the liquid through-flow method, or also to remove contamination from logistics packaging, such as blister packs, small load carriers or cardboard.

For this form of extraction, a completely-sealed cabinet with the possibility to intervene manually is required (e.g. glove box) as well as connections for compressed air. It shall be possible to vent the cabinet to prevent a positive pressure from being created inside it.

Air jet extraction is performed in two steps:

a) Particles are detached from the test component with a jet of compressed air and subsequently adhere to the walls of a cabinet wetted with liquid. The test component is then removed from the cabinet.

b) The particles adhering to the cabinet walls are rinsed off and transferred for analysis using a process similar to the final rinsing step performed after extraction by pressure-rinsing.

The main parameters influencing air jet extraction are (see also Table 5):

- pressure of the jet of compressed air,
- geometry of the jet nozzle,
- distance away from the object and the angle of the jet,
- sequence used when extracting the controlled surfaces,
- duration of jet per surface or feed rate,
- repeats per surface.

NOTE As opposed to extraction by pressure-rinsing, in which the pressure measured and information obtained are highly dependent on the place of measurement (at the nozzle, behind the pump, etc.), the pressure in air jet extraction fluctuates less at the various sites and can thus be considered as a suitable extraction parameter. However, the flow rate of the air cannot be measured by means of simple volumetric measurement.

7.5.2.2 Start parameters

Table 5 — Start parameters for air jet extraction (see 5.3)

Parameter	Start value/Condition
Nozzle shape	Round, full-jet nozzle
Nozzle diameter	1,5 mm
Pressure	1,5 bar
Distance between nozzle and component	Max. 10 cm
Duration of application of jet / controlled surface	1 s/cm^2

NOTE When inspecting larger surface areas, the extraction time can be reduced by using several nozzles (e.g. combined in an air jet tool).

During the qualification tests/ declining tests (see Clause 6), the duration of application of the jet per controlled surface is determined for the routine inspection depending on after which extraction step the declining criterion is attained. If the declining criterion is not achieved with these start parameters or if other parameters are more suitable and strictly documented, these start parameters may be modified.

7.5.2.3 Materials and equipment

The materials and equipment used in the extraction step shall comply with the general requirements of extraction equipment described in 7.2.

a) Supply of clean, oil-free compressed air (cleanliness of the compressed air shall fulfil the blank level criteria), control valve to regulate pressure and manometer to display the pressure. Air jet tool with nozzle and switching option (finger switch on air jet pistol or foot pedal in compressed air line).

b) If required, device to secure the component: e.g. rest, mounting device.

c) Pressure-rinsing apparatus for the final rinsing procedure comprising:

  - pressure-rinsing tool: e.g. nozzle stylus, which can be guided by hand to rinse the extraction equipment, parallel jet or fantail nozzle;
  - media supply with container holding test liquid, purification filter, pump or pressure supply.

d) Completely-sealed air jet and pressure-rinsing cabinet with intervention option for guiding air jet and final rinsing tools (e.g. glove box) and handling the test components. The box shall contain a pressure vent to prevent a positive pressure from being created inside the cabinet. Extracted particles may not escape via the vent.

e) Where appropriate, graduated vessel to measure volumes: e.g. beaker or measuring cylinder, and stopwatch to adhere to extraction times or determine by means of volumetric measurement the flow rate used in the final rinsing step.

7.5.2.4 Procedure

The following procedure shall be adapted to the features of the respective test component.

a) Procure all resources required for the test.

b) Prepare the test component in accordance with 7.3.

c) Use test liquid to pre-clean all surfaces of the air jet cabinet coming into contact with compressed air and fluid as well as other extraction equipment; where required, determine blank levels. In order to bind detached particles, all the walls of the air jet cabinet shall be wetted with the test fluid before commencing the air jet extraction procedure.

d) Place the test component in the air jet cabinet and secure it if necessary.

e) Perform the air jet extraction procedure on observing all the specified parameters, times and following the exact sequence. The careful execution of this procedure is decisive to the inspection results.

f) Remove the test component from the air jet cabinet.

g) Using test liquid, perform a final rinsing step on all inner surfaces of the air jet cabinet to remove all the extracted particles now adhering to the walls and transfer them for analysis. A suitable procedure should also be elaborated, tested and defined for this final rinsing step.

h) Transfer all the liquid for filtration and analysis.

i) Fill out the inspection report.

7.5.2.5 Documentation

See Clause 10.

7.5.3 Air through-flow extraction

7.5.3.1 Principle

With this form of air extraction, the test component is subjected to a through-flow of air using a procedure similar to that of internal rinsing in the case of liquid extraction.

This method is suitable for inspecting the internal surfaces of air conducting components, e.g. engine air intake tract. With this method, large volumes of air are guided through components with cross-sections several centimetres in diameter.

Air through-flow extraction is divided into two steps (see also Figure D.13).

a) Primary extraction step: the test component is installed in the test bench using component-specific adapters. Particles extracted by the pre-filtered air as it flows through the component are deposited onto a large-surface (high flow rates!), fine-meshed metallic membrane filter located in a primary filtration unit. This primary analysis filter is removed in an appropriately clean manner after the primary extraction step and forwarded to the secondary extraction step.

b) Secondary extraction step: in this step, liquid extraction by means of pressure-rinsing (see 7.4.3) is carried out to remove the particles originating from the test component that are present on the primary analysis filter and to transfer them to a conventional secondary analysis filter suitable for use with microscopy and/or gravimetry.

NOTE 1 The air through-flow extraction method can also be implemented to inspect pneumatic components in the automotive industry. Such components are systems that conduct compressed air and have cross-sections several millimetres in width. The basic set-up is illustrated in Figure D.14. In the example shown, the pneumatic component is subjected to a through-flow of clean, oil-free compressed air. After the extraction step, the air is guided into the liquid-filled bath of a pressure-rinsing extraction device. The particles extracted from the component are thus bound in the liquid and can subsequently be deposited onto an analysis filter and analysed.

NOTE 2 The air through-flow extraction method can also be used with direct filtration of the air flow with an analysis filter downstream the test component, especially if low flow rate is sufficient for extraction.

The main parameters influencing the air through-flow extraction method are:

- air through-flow rate,
- geometry and type of the test component,

- form and frequency of actuation (in the case of active components), and
- duration of air through-flow.

7.5.3.2 Start parameters

The flow rate of air through the test component cannot be used as a general start parameter (see 5.3) because it depends on the rate at which air will flow through the component when in operation. These values shall be specified in the customer-supplier relationship.

A start time of 2 min is recommended as a parameter for each extraction step in the declining tests/qualification tests.

7.5.3.3 Materials and equipment

The materials and equipment used in the extraction step shall comply with the general requirements of extraction equipment described in 7.2.

a) Where appropriate: secure component: e.g. mounting device or clamps. In the case of active components: use devices for switching, actuating, etc.

b) Air through-flow test set-up comprising:

   - air pre-filter;
   - system of pipes to guide air through the test component;
   - primary filtration unit with primary analysis filter (e.g. 10 µm metallic mesh filter). The interior of the primary filtration unit (walls) shall be given a final rinse with liquid and the liquid drained off at the lowest point;
   - pipe system with flow gauge and regulator;
   - pump to generate the required flow rate of air for the test;

c) Component-specific adapters (may need to be constructed in some cases) to connect the test component to the tubing of the test set-up.

d) Pressure-rinsing extraction device (see 7.4.3) to perform the secondary extraction step on the primary analysis filter.

e) Where appropriate: if the pressure-rinsing apparatus mentioned in d) is located elsewhere and cannot be used here, further pressure-rinsing equipment to perform final rinsing step on the primary filtration unit.

7.5.3.4 Procedure

The following procedure shall be adapted to the test component and test set-up.

a) Procure all resources required for the test.

b) Pre-clean all relevant surfaces of the extraction setup; where required, determine blank levels.

c) Prepare the test component in accordance with 7.3.

d) Use component-specific adapters to connect the test component to the extraction set-up.

e) Where appropriate: in order to actuate active test components, connect switching, power or drive devices.

f) Carry out the air through-flow extraction procedure on observing the flow rate and duration; in the case of active components, adhere to operating parameters.

g) Perform final rinsing step on the interior of the primary filtration unit with final rinsing liquid to transfer any particles that may have sedimented on the walls to the primary analysis filter.

h) Drain off extraction liquid at the lowest point of the primary filtration unit.

i) Remove the primary analysis filter and transport it to the pressure-rinsing extraction setup in an appropriately clean manner.

j) Perform a full pressure-rinsing extraction step (in accordance with the procedure described in 7.4.3) using the primary filter membrane as the test component. Transfer the particles to the secondary analysis filter.

k) Fill out the inspection report.

7.5.3.5 Documentation

See Clause 10.

8 Analysis filtration

8.1 Principles

The purpose of analysis filtration is to deposit the particles extracted from the component and suspended in the extraction liquid onto the surface of an analysis filter so that they can be analysed. Vacuum filtration is generally used for this.

In recent years, particle-counting techniques and extended analysis methods, which both require particles to be located singly without overlapping on an analysis filter, have become much more popular than gravimetry. Consequently, the filtration step is now crucial to the analysis result. The careful selection of filtration equipment and analysis filter(s) as well as the precise execution of the filtration procedure are decisive steps that play a major role on the quality of the subsequent analysis results. Recommendations for the design and application of analysis filtration are described in Annex E.

8.2 Selecting the analysis filtration method

8.2.1 General

The properties of analysis filters, such as resistance to chemicals, capacity to retain particles, and also further aspects such as optical characteristics, may vary considerably depending on the component and analysis task concerned. If the cleanliness inspection is carried out within the scope of the standard analysis procedure of this document, i.e. detection of particles upwards of 50 µm with gravimetric and/or light-optical analysis, the following analysis filter is recommended:

5 µm mesh filter

NOTE 1 E.g. PET can be used as a filter material.

NOTE 2 Very thin particles may pass the mesh filter and thus not be captured in the subsequent analysis.

8.2.2 Chemical resistance

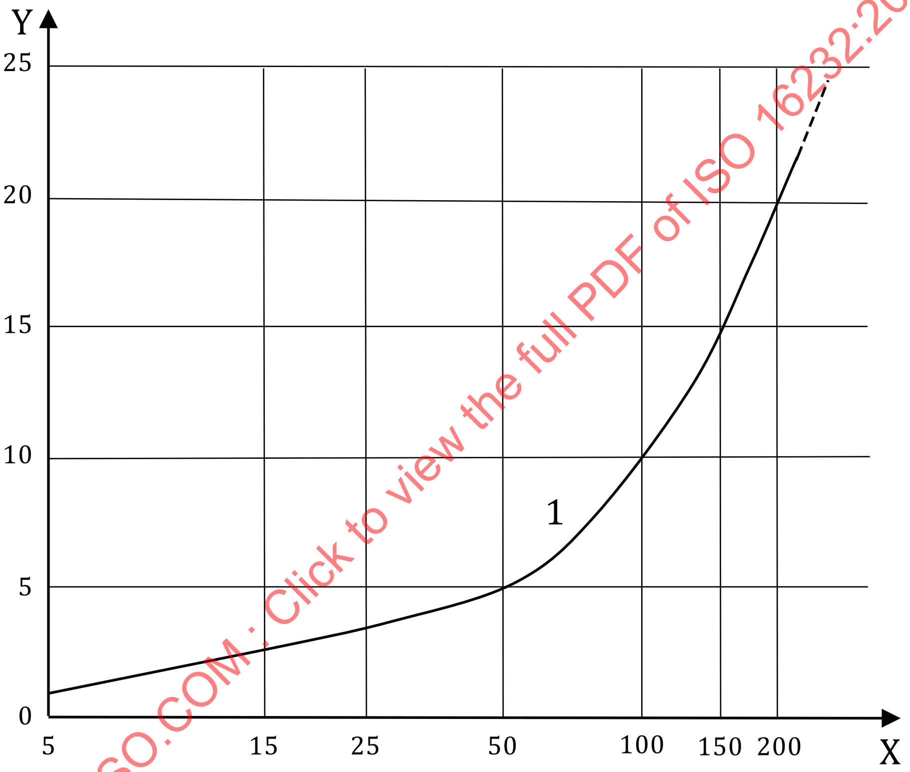
All of the equipment used in the filtration process as well as the analysis filter utilized shall be compatible with the test liquid implemented (where appropriate, also the final rinsing liquid or particle-fixative) (see also E.2).

8.2.3 Particle retention capacity

8.2.3.1 Filter pore size

The function of the analysis filter is to retain the particles which are relevant to the analysis (ideally only these particles). The pore size of the analysis filter is selected according to the cleanliness specification, i.e. the filter shall be capable of reliably retaining the smallest particle size stipulated in the cleanliness specification. To ensure that elongated particles are also retained, the following rule of thumb applies:

Filter pore size = 1/10 to 1/5 the size of the smallest particle size specified, with 1/10 being recommended for larger particles (>50 µm) and 1/5 for smaller particles (<50 µm). This is because smaller particles generally have a more compact shape than larger particles, which tend to have a highly diverse range of shapes (see also graph in Figure 4).



Key

X smallest particle size requiring detection, µm

Y filter pore size, µm

1 filter pore size = 1/5 to 1/10 of the smallest particle size requiring detection

Figure 4 — Selecting the filter pore size

8.2.3.2 Filter cascade

To pre-select particle sizes, analysis filtration can be performed in several steps using filters with different pore sizes. To do this, a filter holder into which several filters can be fitted is used (see Figure E.2).

For example, the use of a filter cascade enables only large particles to be deposited on a coarser-meshed membrane filter, thus facilitating counting by microscopy. The high number of small particles is retained by a finer filter downstream. If gravimetric analysis is required, all of the filters in the cascade shall be weighed.

8.2.4 Further properties of analysis filters

An analysis filter generally has to withstand the typical temperatures it is subjected to during the drying process in the oven and also be mechanically robust (tear-resistant) when handled with tweezers or dried by vacuum.

The following filters can be implemented to adapt and optimize analysis filtration for specific particle types or special analysis methods.

- Filters with special optical characteristics (e.g. colour) to give a good light-optical contrast, or made from specific materials to give a high material contrast or good conductivity or resistance in the scanning electron microscope.
- Filters that do not generate a measuring signal of their own as this could overlay particle signals, e.g. Raman or FT-IR inactive filter materials.

8.3 Handling analysis filters

In order to avoid adverse influences regarding analysis filters, the following points shall be observed.

- Always store analysis filter membranes in sealed containers to minimize contamination from the environment.
- Visually inspect the analysis filters before use. Discard damaged filters. Pre-clean the analysis filter with test liquid except it is otherwise safeguarded that the filter is not contaminated.
- Always handle analysis filter membranes with clean tweezers and only hold the edge of the analysis filter membrane. Sharp or pointed objects may damage analysis filter membranes.
- The surface of the analysis filter containing the particles may never come into contact with another surface (potential particle loss). Exception: lids for fixing particles for the purpose of microscopic analysis or archiving. The lids of analysis filters for microscopic analysis may alter the shape of particles due to clamping. If the glass lid is removed after light-optical analysis, e.g. in order to carry out an extended analysis, there is a risk that particles will be lost through adhesion to the glass lid.

  NOTE The material of the lids that are used can have an impact on the detection of particles during microscopic analysis, especially for polarization, bright field illumination and transmitted light.

- On completion of filtration, careful drying and subsequent analysis, analysis filters shall be stored in a suitable container (e.g. Petri dish with a supporting screen to aid drying and removal of the analysis filter). To ensure the correct assignment of the analysis filter to a specific component, the storage container should be clearly marked.

8.4 Materials and equipment

a) Analysis filter, selected on the basis of the features of the respective cleanliness inspection.

b) Filtration equipment including vacuum pump. The items required for this depend on the type of filtration procedure selected (see E.1).

   If the filtration flow rate is too high, particles of importance could pass through the analysis filter or the filter could become damaged.

c) Tweezers to handle the analysis filter membrane.

   The tweezers used shall not be magnetic nor become electrostatically charged. This applies to all items of equipment coming into contact with the analysis filter because this could cause particle loss due to magnetism or electrostatic charging.

   NOTE Analysis filters can also be handled with a clean spatula. All appropriate formulations also apply to spatulas.

d) Test liquid for the final rinsing step after the filtration procedure (using a wash bottle).

e) Petri dish or similar clean, sealable container for handling and storing analysis filters.

f) Drying oven with controlled temperature (no fan) or other suitable equipment like vacuum apparatus, microwave device etc.

The temperature and duration of the drying process shall be modified in dependence on the combination of analysis filter and test liquid (see 9.2.2).

WARNING — When drying analysis filters wetted with solvents, ensure an adequate air exchange inside the warming cabinet to prevent an explosive mixture from being formed (keep well below the lower explosion limit).

g) Fixative (optional): a substance to fix the collected particles onto the analysis filter.

If a fixative is used to prevent particle loss, it shall be utilized only after gravimetric analysis.

8.5 Procedure

a) Procure all resources required for the filtration step.

b) Prepare the filtration equipment. Depending on the apparatus used in the extraction procedure, analysis filtration can be performed in two ways (see Figure E.1).

   i) Direct filtration: Filtration takes place directly at the level of the extraction setup: the filter unit is situated immediately behind the outlet of the basin for collecting the extraction liquid.

   ii) Separate filtration: The extraction liquid is first collected in a clean vessel (e.g. beaker or sampling vessel) and then filtered through a suction filter.

c) Depending on the analysis method required, the analysis filter may need to be conditioned first.

   i) Microscopic analysis: The analysis filter does not require conditioning.

   ii) Gravimetric analysis: The tare mass of the analysis filter shall be determined (see 9.2.2). To avoid negative residue masses, the analysis filter shall be conditioned.

d) Place the analysis filter in the filter holder with the aid of tweezers.

NOTE In the case of especially thin analysis filters (e.g. polycarbonate), it can be helpful to place a filtration aid beneath it (e.g. suitable analysis filter as a support) as this promotes a more uniform particle load for microscopy (avoid “support grid effect”).

e) Filtration of the extraction liquid.

   i) Direct filtration: Using clean liquid, carefully perform final rinsing step on the collection container (e.g. pressure-rinsing basin or ultrasonic bath) to avoid any particle loss. The final rinsing liquid is included in the analysis.

   ii) Separate filtration: Pour the liquid from the sampling vessel (e.g. beaker from ultrasonic extraction) into the filter funnel, then perform a final rinsing step on the sampling vessel. The final rinsing liquid forms part of the extraction liquid. The size of the vacuum flask limits the volume of liquid which can be filtered.

Sometimes the extraction step and filtration step are carried out at different locations (far away from one another) with the consequence that the sampling vessel needs to be transported a long distance. In such a case, care shall be taken with the following: before commencing filtration, clean the exterior of sampling vessels to exclude any contamination present from reaching the extraction liquid during the analysis step. If the extraction liquid is stored for a prolonged period of time, particles may sediment and agglomerate. With counting analysis methods, it is essential to break up the agglomerated particles before commencing the analysis step. This can be achieved by agitating the extraction liquid or placing the vessel in an ultrasonic bath. However, the method chosen may not modify the original sizes of particles in the liquid. Stored sampling vessels generally require gentle agitation to return sedimented particles into suspension. Sampling vessels (including closing devices) shall be thoroughly rinsed with final rinsing liquid to avoid any particle loss. The final rinsing liquid forms part of the extraction liquid.

f) Filter the extraction liquid.

The uniform distribution of particles on the filter (important for microscopic analysis) can be aided if a "cushion" of liquid is always present on the analysis filter. However, if the extraction liquid only passes through the margins of the filtration unit, there is a risk that the particles will remain at the edge and overlap one another.

g) To avoid particle loss, perform a final rinsing step on all surfaces in contact with the extraction liquid.

Do not pour a stream of liquid directly onto the surface of the filter when preparing it for analysis by counting as this could impair homogeneous particle distribution.

h) Analysis filter for gravimetric analysis (optional): wash away undesired chemical residues by post-treating the analysis filter with a suitable solvent.

i) Analysis filter for microscopic analysis (optional): fix the particles with the aid of a fixative.

WARNING — Not suitable for gravimetric analysis filters because the fixative alters the residue mass.

j) Remove the upper section of the filter holder in the presence of a vacuum.

k) Transfer any particles present on the upper section of the filter holder to the analysis filter by rinsing.

l) Where appropriate, aerate the filtration apparatus. Carefully remove the suction-dried analysis filter with tweezers (risk of particle loss) and place it in a clean labelled Petri dish (hold horizontally).

m) Dry the analysis filter by keeping the Petri dish partly open (in the case of analysis filters to be analysed gravimetrically, dry the analysis filter until the mass remains constant (see 9.2.2).

8.6 Verifying particle occupancy on the analysis filter

To use automated optical systems for particle analysis, the analysis filter shall be prepared in such a way so as to ensure that particles are evenly distributed over the surface of the analysis filter without overlapping or agglomerating (see e.g. Figure E.4 and E.5).

The following points are important in this regard:

- the filter occupancy in percent, i.e. the proportion of the surface of the analysis membrane occupied by particles;
- the homogeneity of occupancy, i.e. the uniformity of particles distributed over the surface of the analysis filter (are agglomerates of particles present or are the particles mainly located at the margin?);
- occupancy with fibres: fibres are often much longer than other particles. Fibres are capable of covering large areas of the analysis filter and therefore often touch or overlap numerous particles and render individual analysis impossible.

With light-optical analysis systems, the filter occupancy in percent should be calculated and documented. The critical occupation density depends on the type of filter implemented. This is shown in E.5 for mesh filters and foamed membrane filters.

If the filter occupancy is such that it cannot be analysed, the extraction procedure shall be repeated with another component of the same type, and a further filtration step carried out. To optimize filter occupancy and thus filter analysability, the following alternatives are available.

a) Use an analysis filter with a larger diameter (provided the filtration unit and analysis system used technically allow this);

b) Distribute the extraction liquid over several analysis filters.

NOTE If the analysis filter retains a high quantity of very small particles that are not relevant to the analysis result, an analysis filter with a larger pore size can be selected. This decreases the occupation density and lowers the risk of particles overlapping one another. This also helps to maintain the contrast between detected particles and the analysis filter background.

If gravimetric analysis of the analysis filter is required as well as microscopic analysis, this is problematic because a conflict arises between the reduction in filter occupancy as far as particles overlapping and the lower detection limit of the balance are concerned (see 9.2.2).

c) Use a filter cascade: The filter cascade contains analysis filters with different pore sizes to separate small and large particles.

Some examples of incorrectly-prepared analysis filters together with possible remedies are given in E.4.

9 Analysis methods

9.1 Principles

This clause describes the various techniques which can be implemented to analyse particulate contamination. The selection of a suitable analysis method and its field of use are described in 5.2.

To design and apply procedures for microscopic analysis, the specific settings and configurations described in Annex F shall be considered.

9.2 Standard analysis

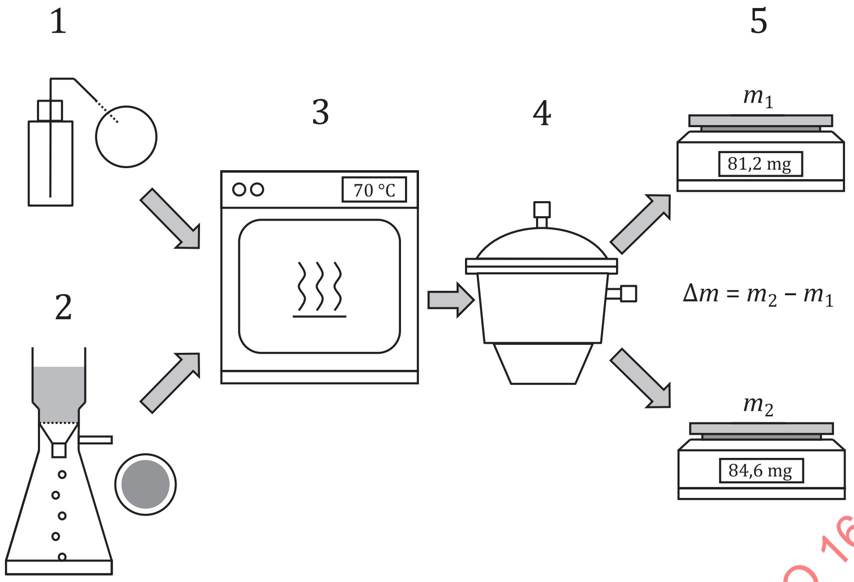
9.2.1 General

Provided that no other arrangements have been made, standard analysis is used to validate the cleanliness specifications stated in the customer-supplier relationship. Standard analysis comprises the methods of gravimetry and/or light-optical analysis. In order to optimize the comparability of analysis results within the technical possibilities of the method used, there are limitations regarding the application of the method and the settings and analysis parameters which can be used.

9.2.2 Gravimetry

9.2.2.1 Principle

As shown in Figure 5, the total mass of the particle load extracted from a test component can be determined by weighing the analysis filter with an analysis balance before and after the extraction liquid is filtered.



Key

1	pre-conditioning (as required)	4	cooling
2	filtration	5	weighing
3	drying		

Figure 5 — Diagram of the gravimetric analysis procedure

The result of the gravimetric analysis is the residue mass and represents an integral value. Its magnitude depends on the total quantity and size of the particles extracted as well as on their respective material composition. Gravimetric analysis does not give any information about the quantity, size or nature of the particles concerned and thus provides very little information about their damage potential.

To accurately determine the mass difference, the analysis filter shall be carefully prepared, dried and cooled to room temperature. If very small differences in mass need to be determined, climatic conditions in the room need to be regulated more precisely, and the design of the weighing area becomes more complex.

If additional analyses are required as well as gravimetric analysis, e.g. light-optical analysis, the gravimetric residue mass shall always be determined first e.g. in order to avoid change of the mass of the filter due to air humidity.

A dilemma is often encountered when preparing an analysis filter for simultaneous gravimetric analysis to determine mass and light-optical microscopy to determine particle size distribution. This is because the analysis filter should contain a maximum quantity of particulate contamination in order to obtain a reliable gravimetric result, but low numbers of single, separate particles for light-optical analysis.

Mass constancy: the temperature and duration of the drying process vary according to the material the analysis filter is made of and the type of test and final rinsing liquids implemented. Drying conditions are determined as being suitable when mass constancy of the analysis filter is achieved, i.e. when the analysis filter does not lose any more mass as the result of a longer drying period. The respective time and temperature settings need to be determined at least once for each combination of filter/test fluid used (e.g. cellulose nitrate filter combined with cold cleaner, 150 min at 70 °C).

NOTE 1 Mass constancy is attained if no change in mass relevant to the gravimetric result occurs after the same analysis filter has been subjected to a second drying step and weighed again.

NOTE 2 Drying conditions (time and temperature) can vary for each combination of test liquid and analysis filter. See manufacturer's instructions for details.

Non-particulate residues: to stabilize the tare mass of the clean analysis filter before commencing the inspection, it may be necessary to rinse it briefly with test liquid before performing the first drying process in order to remove any volatile substances present in the filter material.

In some cases, the analysis filter may need to be rinsed with an appropriate liquid after the filtration step in order to remove any chemical residues that are not particles but which could influence the gravimetric result. Examples of such residues include tensides from aqueous test liquids, or greases or wax originating from the component that are difficult to dissolve.

In some cases, particles may need to be fixed on the analysis filter for the purpose of further analysis, e.g. with the aid of a fixative. This may only be applied once gravimetric analysis has been completed.

9.2.2.2 Material and equipment

See Clause 8. The following are also required.

a) Desiccator.

   The desiccator prevents the analysis filter from absorbing any humidity from the environment during the cooling process after it has been dried.

   Depending on cleanliness requirements, or when relatively high residue masses (>> 10 mg) need to be determined, the use of a desiccator may not be necessary. In order for this condition to apply, the maximum permissible blank level may not be exceeded.

b) Ionization unit (if it is known that the weighing process is affected by electrostatic interference).

   The ionization unit generates positively and negatively charged ions to neutralize the electrostatic charge on the analysis filter as this could impair the weighing result. The ionization unit shall be located as close as possible to the weighing platform. Ionization units with fans are not suitable.

c) Analysis balance.

   The minimum readability requirement is 0,1 mg = 0,000 1 g (four-digit balance). If a higher gravimetric resolution of 0,01 mg is required, use a 5 digit balance in a controlled environment regarding temperature and humidity.

9.2.2.3 Environmental conditions

The lower detection limit or sensitivity of gravimetric analysis is not solely determined by the capabilities of the analysis balance. Constant levels of humidity and temperature of the immediate environment and also the cleanliness of the air have a significant influence. In a non-controlled environment, the result is thus affected by the length of time a dried analysis filter is exposed to the environment before being weighed. Consequently, time periods shall be kept as identical as possible during the procedure.

Where possible, the weighing surface of the balance should not transmit any vibrations, be non-magnetic and not electrostatically chargeable (no steel, plastic or glass). The room in which the balance is located shall be low-vibration and the temperature in the room kept constant. The relative humidity of the air should be kept between 45 % and 60 %.

If a six-digit balance is used (± 1 µg), it is recommended to keep the relative humidity of the test environment permanently controlled.

Direct sunlight and draughts (even in cleanrooms) shall be avoided. The balance may not be positioned near air-conditioning units, heaters or doors.

9.2.2.4 Lower detection limit of the gravimetric analysis

If a four-digit balance is used in rooms where the temperature and relative humidity are not controlled, the lower detection limit is 1 mg. In connection with the requirement that the percentage of the blank level may be maximum 10 percent of the total load, only particle loads with a residue mass (*m*) upwards of 10 mg can be detected.

In order to be able to use gravimetry to assess the technical cleanliness of components with particle loads below 10 mg, the lower detection limit of the analysis set-up (balance including environment) shall be less than 1 mg in order to fulfil the blank level criterion of ≤10 %. This is achieved by using a balance with a higher resolution in a room with controlled temperature and relative humidity.

These more complex measures enable particle loads as low as 3 mg to be reliably detected using gravimetry. If a lower mass is determined in a gravimetric analysis, the result is expressed as $m < 3$ mg.

Another way of analysing components with very small particle loads by means of gravimetry is to increase the residue mass (*m*). This is achieved by inspecting several components simultaneously, or by filtering the extraction liquid from several components through one analysis filter.

9.2.2.5 Procedure

a) Procure all resources required for preparing the analysis filter and for the subsequent analysis procedure.

b) Condition the analysis filter to stabilize its mass.

   In case of large gravimetric load the conditioning may be not necessary because measurement error in relation to load is not critical.

   To stabilize the mass of an analysis filter, first of all it is treated with test liquid before the first drying process in order to remove any substances that can be washed out from the filter material. This would otherwise occur during the analysis filtration step and lead to a lower actual residue mass. This shall be verified before using the combination of test liquid and analysis filter for the first time.

   i) Place the membrane in the filter holder and filter a sufficient volume of clean test liquid through it to remove any soluble substances contained in the analysis filter.

   ii) Pre-dry the membrane by sucking air through the analysis filter with the vacuum pump.

c) Pick up the analysis filter with tweezers and place it in a marked clean Petri dish.

d) Determine the initial mass m_1 (tare mass).

   i) Place the partly-covered Petri dish containing the analysis filter (= goods to be dried) in the pre-heated drying oven; verify the temperature and time required to dry the analysis filter.

   ii) Remove the dried goods and place immediately in the desiccator; check the amount of time required to cool the analysis filter.

   iii) Take the dried goods out of the desiccators; remove the analysis filter immediately with tweezers and place it on the weighing surface of the analysis balance.

   iv) Read off and document the value shown for the initial mass m_1 of the analysis filter (tare mass).

e) Using tweezers put the analysis filter back into the Petri dish and close the lid. The analysis filter is now ready for use in an analysis filtration step.

f) Perform the analysis filtration step (see Clause 8).

g) Determine the final mass m_2 (gross mass).

   i) Place the partly-covered Petri dish containing the analysis filter (= goods to be dried) in the pre-heated drying oven; verify the temperature and time required to dry the analysis filter.

  - ii) Remove the dried goods and place immediately in the desiccator; check the amount of time required to cool the analysis filter.
  - iii) Take the dried goods out of the desiccators. Remove the analysis filter with tweezers immediately and place it on the weighing surface of the analysis balance.

IMPORTANT — Ensure that no particles present on the analysis filter are lost in the process.

h) Read off and document the value displayed for the final mass m_2 of the analysis filter (gross mass).

i) Using tweezers put the analysis filter back into the Petri dish and close the lid.

j) Calculate the residue mass (net mass), which is the difference between m_2 and m_1.

9.2.2.6 Calibration

Follow the manufacturer’s instructions to calibrate the weighing balance.

9.2.2.7 Documentation

See Clause 10.

9.2.3 Light-optical analysis

9.2.3.1 General

As opposed to gravimetric analysis, which only gives information about the total particle load extracted from the component, more detailed information can be obtained using light-optical analysis. With this method, particles can be measured and counted and also characterized.

Depending on requirements, different types of microscopes or flatbed scanners can be used for light-optical analysis (see Figure 6).

Because the function of components may already be impaired by one or only a few particles with specific features, it is imperative that such particles are detected reliably. In order to do this, the entire effective surface area of the analysis filter is inspected.

Analysis diameter shall be larger than filtration area to ensure that all particles on the analysis filter are analysed.

Light-optical systems are not only used in a standard analysis procedure with fixed parameters in order to validate cleanliness specifications in the customer-supplier relationship (provided that no other agreements have been made); they can also be utilized for the purpose of extended analysis.

Light-optical analysis of an entire analysis filter membrane is generally performed using a fully-automated process. If only a small quantity of large particles need to be measured, this can be done manually.

9.2.3.2 Principle

With light-optical analysis, the analysis filter containing the particles extracted from the test component is illuminated in image fields using a suitable light source and reproduced by a magnifying lens, generally in the form of pixels on a camera sensor. Image-processing is used to detect particles and determine size features. However, objects can only be detected if they differ optically (in brightness) from the background. Thus, for example, white particles cannot be detected if they are on a white analysis filter membrane, and very pale particles can only be partially identified (see also F.1.5). This is not a fault or shortcoming of the light-optical system concerned but is rather due to the nature of the detection method and its principle of function.

Which particles are recognized in detail and how they are measured and characterized is highly individual and depends on several factors:

- imaging lens (magnification and resolution);
- type and individual design of illumination (for example bright field, dark field, reflected light, transmitted light, polarization);
- illumination of the image fields;
- threshold value(s) which enables the image-processing software to differentiate between particles and the filter background (binarization threshold);
- definition criteria for measuring and characterizing particles as well as the exact algorithms used to implement them;
- individual composition and optical characteristics of the respective particle (colour, brightness, homogeneity, surface topography and roughness, etc.).

All these factors influence the result of a light-optical particle analysis. For this reason, it is only possible to fully compare light-optical analysis results if identical systems with identical settings are used to analyse the same type of particles.

9.2.3.3 Light-optical standard analysis

In order to obtain a good degree of comparability with different analysis systems (which all have their strong points) a *standard analysis* is recommended for light-optical particle analysis.

To apply standard light-optical analysis meaningfully, the following four general requirements shall be fulfilled:

- the light-optical analysis system fulfils the respective requirements (see section on materials and equipment);
- the cleanliness specification only considers particles ≥50 µm;
- the analysis filter is well-prepared with particles being evenly distributed and filter occupancy being not too high (see Clause 8);
- the task is performed by specially-trained skilled staff.

Regardless of the light-optical system used (material microscope, stereo microscope, zoom microscope or scanner system with suitable illumination) and thus irrespective of the way the image is generated, the basic idea is not to determine any conventions until image-settings and analysis processes are determined. The conventions aim to enable standardized analysis and thus improve the comparability of analysis results.

The application of light-optical standard analysis alone does not guarantee the full comparability of analysis results from different systems. The level of comparability achieved, especially with regard to the contaminant particles under investigation, shall be considered individually in each case.

When changing from a light-optical analysis with different parameters to a standard analysis, the analysis results may vary.

In some cases, it may make sense to modify the parameters of the standard analysis, e.g. if:

- the cleanliness specification requires the analysis of particles smaller than 50 µm;
- the cleanliness specification is concerned with features and types other than those considered in the standard analysis, e.g. specifications regarding particle material or a third particle dimension;

- optimization is necessary or desired to in order to improve the detection of specific particles; this can be achieved using other contrast methods and/or parameters than those stated in the standard analysis.

These modifications shall be mutually defined and documented if intended for use to validate cleanliness specifications in the customer-supplier relationship.

Standard analysis shall be used in cases where a cleanliness specification fulfils the criteria of the standard analysis and provided that no other arrangements have been made within the customer-supplier relationship.

9.2.3.4 Detecting, measuring and characterizing particles with the aid of light-optical standard analysis

For the light-optical standard analysis of particles over 50 µm, several different light-optical systems can be utilized, e.g. material microscope, stereo microscope, zoom microscope or flatbed scanner, each with reflected light illumination.

As shown in Figure 7 areas of the analysis filter membrane (and thus the particle) are magnified by an imaging lens and reproduced on a camera sensor (or line camera in the case of a scanner). The camera sensor is made up of individual, light-sensitive elements (pixels). In this way, via the degree of magnification selected/set, a specific length in the analysis filter plane is assigned the size of a camera pixel (see example of micrometre scale in Figure 7). This gives an image scale in µm/pixels which is used to measure particles.

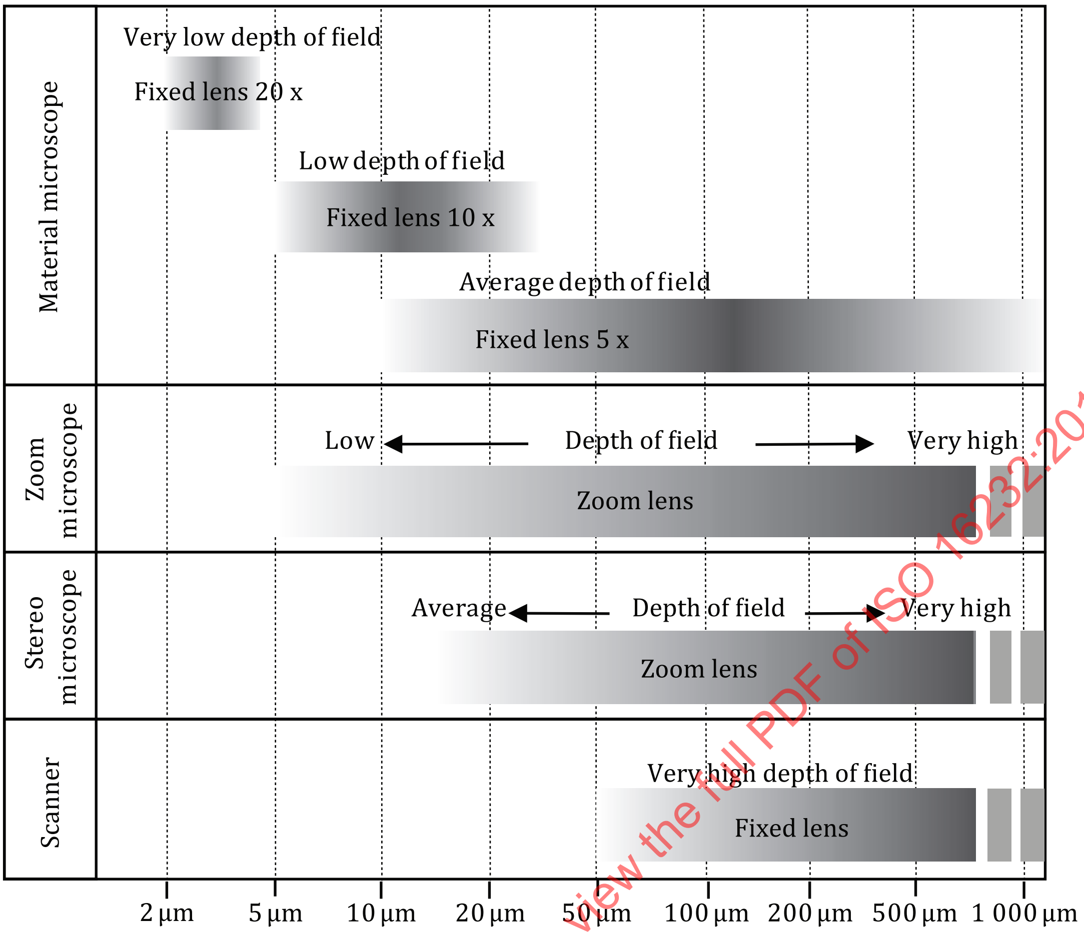
To determine particle sizes sufficiently accurately, the longest dimension of a particle requiring analysis should be reproduced on **10 pixels** of the camera image. In a standard analysis where the smallest particles to be measured are 50 µm in size, this therefore results in the necessary pixel resolution of maximum 5 µm/pixel.

The number of pixels and the size of the camera are not the only factors that determine the accuracy of a particle measurement; the optical characteristics of the imaging lens play a more important role. For example, the lens or lens setting (zoom level) of the microscope determines the resolving power and depth of field. Figure 6 gives an overview of the suitability of different lenses for analysing different-sized particles, as well as giving information on the depth of field.

The higher the degree of magnification and resolution selected/set, the smaller the size of particles that can be measured. However, because higher degrees of magnification reduce the depth of field, it may become difficult to depict large particles clearly. In cases with high resolutions and a low depth of field, a motorized z-drive can be used to compensate for uneven features on the surface of the analysis filter.

NOTE 1 A higher degree of magnification demands a larger quantity of images fields in order to inspect the analysis filter comprehensively. This increases the analysis time as well as the volume of data to be processed or archived.

When selecting the degree of magnification, a compromise should always be made between a high optical resolution on the one hand and a good depth of field, short analysis time and low data volume on the other hand.



Key

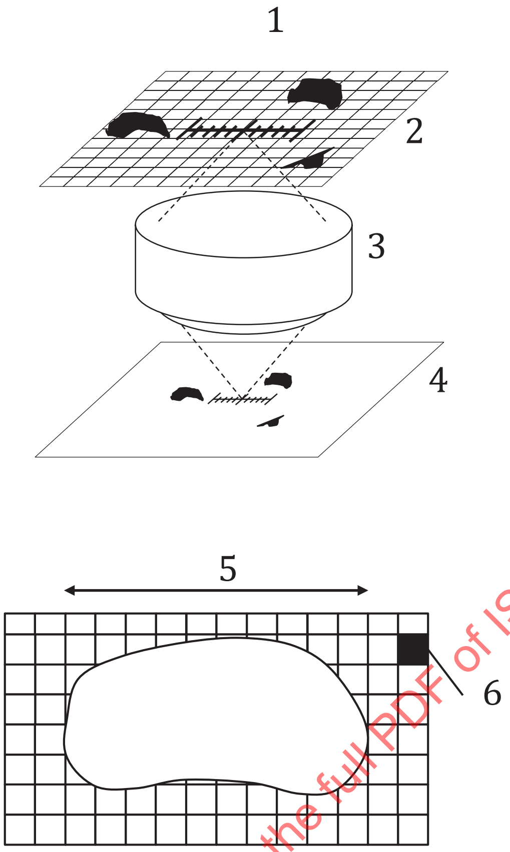
▨ recommended suitability for particle size range

Depth of field:	Very low	1 to 10 µm
	Low	10 to 50 µm
	Average	50 to 100 µm
	High	100 to 1 000 µm
	Very high	≥1 000 µm

Figure 6 — Overview of suitability of different lenses for particle analysis

In the same way as stereo microscopes, zoom microscopes or macroscopes also have a zoom lens with variable magnification. With a stereo microscope, the eyes of the observer view the sample from slightly different angles via a common lens (parallax). This gives the sample a three-dimensional appearance when viewed through the lens. If stereo microscopes are used for automated measurements, the beam path for the camera should be directed in such a way so as to prevent this parallax from occurring, or it should be corrected. With a zoom microscope, the beam path through the lens is identical for both eyes as well as for the camera. However, with solely video zoom microscopes, there is no eyepiece at all, only a beam path for the camera. Here, the image is viewed exclusively on a screen.

Where very small particles need to be measured (beyond the range of standard analysis), such as 5 µm or 15 µm, it may make sense not to apply the 10 pixel criterion and to reproduce such particle sizes on 5 pixels instead.



Key

1	image scale, µm/pixel	4	analysis filter level
2	camera sensor	5	minimum particle size = 10 camera pixels
3	imaging lens	6	pixel size

Figure 7 — Pixel resolution of the light-optical image

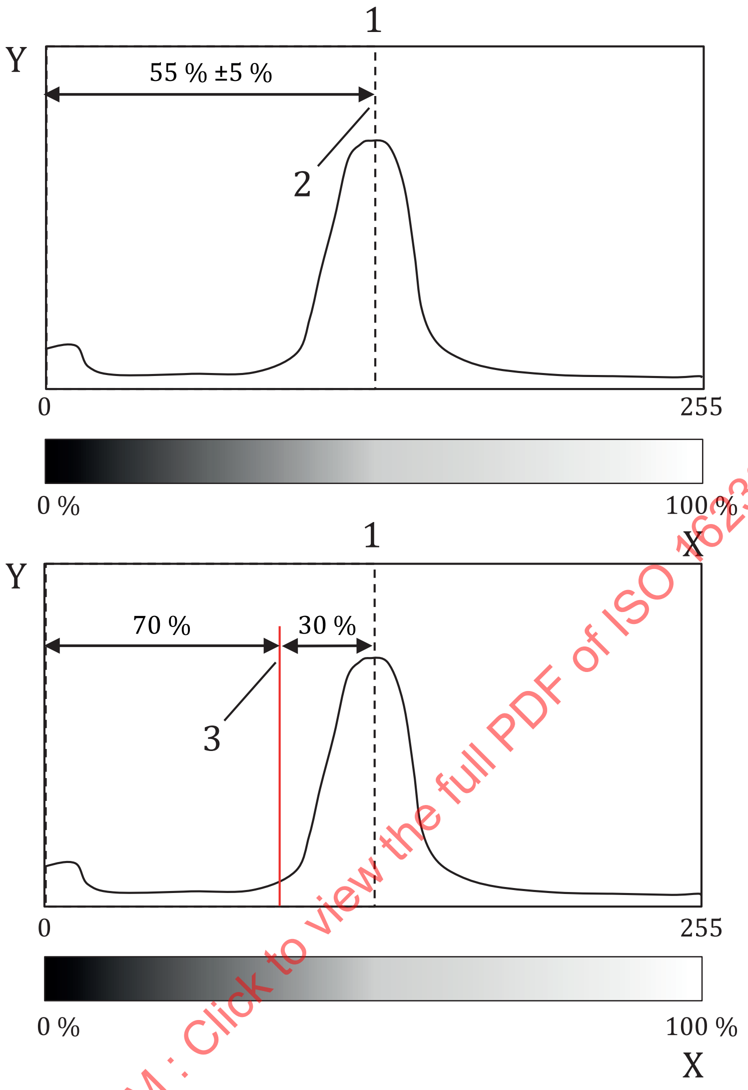
In order to apply the optical method effectively to determine particle size ranges, analysis filters shall be well-prepared with a particle occupancy that is not too dense and with particles neither overlapping nor touching one other (isolated cases cannot be avoided but can be corrected through manual intervention). One criterion for being able to analyse an analysis filter membrane accurately is the occupancy of particles on the filter in percent. This value should be read off and documented as an aid for the user when applying the light-optical analysis method. The percentage of filter occupancy encompasses all areas of images that are considered to be particles (all types) after binarization (see below). This applies not only to the particle sizes mentioned in the cleanliness specification but also to all pixels in the filter image. For further information on filter occupancy, see 8.6.

When **detecting particles**, the image-processing software assigns some image areas to particles and the remaining areas to the background of the analysis filter. To do this, the brightness range of the image is generally divided into grey values varying between black and white. In the case of particle analysis, 256 shades of grey have become established. If the quantity of pixels in the image possessing specific grey values is spread out over this range, a “histogram” is obtained, as shown in Figure 8.

In order to set the image brightness and thus define the shape and position of the histogram and also set one or two threshold values, which are essential in order to differentiate between particles and background (so-called binarization thresholds), further conventions are made to increase the comparability of analysis results. A differentiation is made between light microscopes with polarizers and scanners without polarizers:

a) **Light microscopes with polarizers**. The light used to illuminate the analysis filter is linearly polarized by a polarizer. Light rays reflected from the specimen are detected by the magnification lens and directed through a second polarization filter rotated by 90° (analyser). The crossed polarization filters make particles darker and erase reflections from metallic particles. As a result, the particles requiring detection appear dark on a pale filter background. The brightness of the image is then adjusted until the maximum grey value range of the filter background (the histogram maximum) is shifted to 55 % ± 5 % of the total grey value range (via the brightness of the illumination, camera exposure time, or similar). The relevant information in the image (dark particles) is located to the left of this maximum value in the form of darker grey values.

   The binarization threshold is set at 70 % of the maximum grey value, see also Figure 8. Structures to the left of this grey value threshold are recognized as particles.



Key

X	grey value
Y	pixel count
1	microscopes with crossed polarizers
2	filter background (maximum grey value) adjusted to approx. the middle of the grey value range
3	binarization threshold set at 70 % of the maximum grey value

Figure 8 — Setting the image brightness and binarization threshold for light microscopes with polarizers

b) **Scanners and microscopes without polarizers**. As opposed to an image obtained with crossed polarizers, an image obtained without the use of polarizers contains relevant information to the left and right of the histogram maximum, i.e. structures of objects that are darker and brighter than the filter background. In consequence, with scanner systems, image brightness is set so that the histogram maximum is 50 % of the total grey value range.

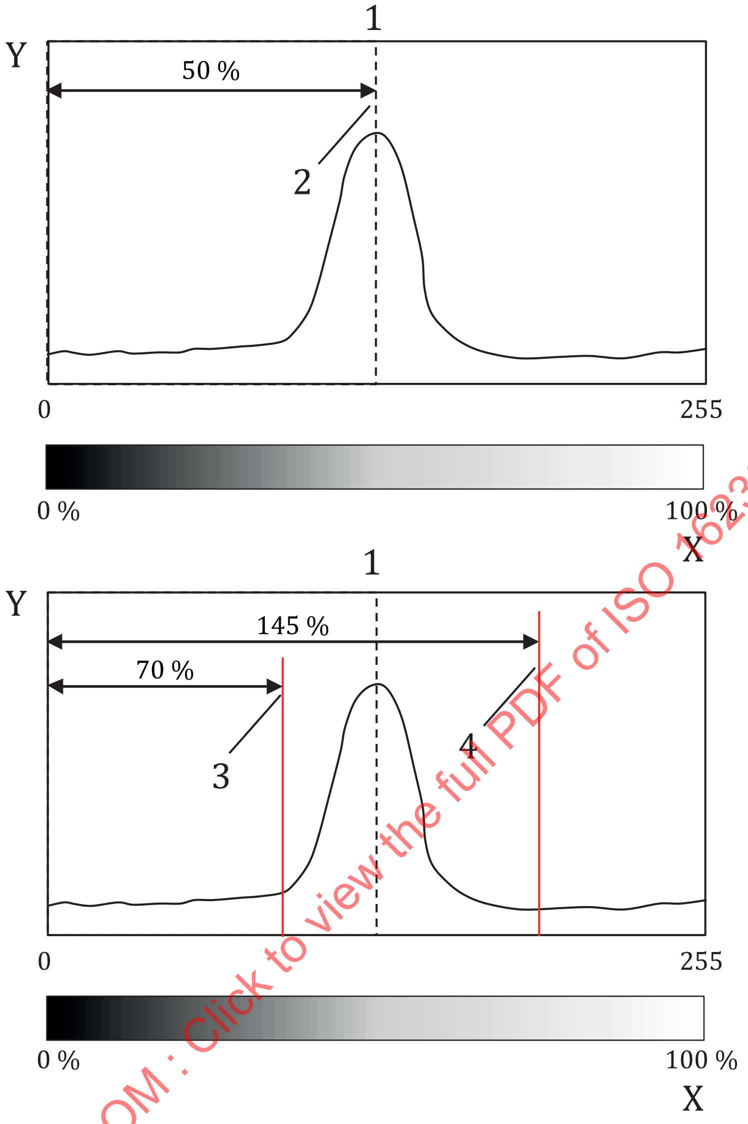
To detect particles, two binarization thresholds relative to the grey value maximum are selected, one at 70 % and the other one at 145 % (see also Figure 9). Structures located to the left of the left-hand grey value threshold and to the right of the right-hand grey value threshold are recognized as particles. The mid-range values of the filter background are practically “cut out”.

NOTE 2 It can be necessary to modify the procedure for "joining up separated particle structures with dilatation and erosion" described in the following paragraph and shown in Figure 10 in regard to the optical resolution and settings that are used.

One disadvantage of this method compared to systems with crossed polarizers is that particles with pale and dark areas are often divided up to form more than one particle because the transition between pale and dark areas on the particle is in the brightness range of the filter background (which is cut out). As illustrated in Figure 10, image-processing methods (dilatation and erosion) are used to join up these areas of the particle again. In Figure 10 a) an example of a particle with pale and dark areas is shown together with grey value transitions. After binarization, the particle is divided into three separate sections, b). In an initial dilatation step, the originally pale areas of the particle are magnified by one pixel, c). In a second dilatation step, all areas of the particle are magnified by one pixel and the separated structures are joined together again d). As the overall contour of the particle is now enlarged e), an erosion step reduces it in size by one pixel f) to result in the particle structure g), which is then measured.

NOTE 3 When applying this method to join separated particles, there is a risk that particles which are very close to one another but physically separate could also be joined up, thus distorting results as far as particle size and quantity are concerned. Structures inside a particle may also be connected, which, under certain circumstances, could distort the size and shape.

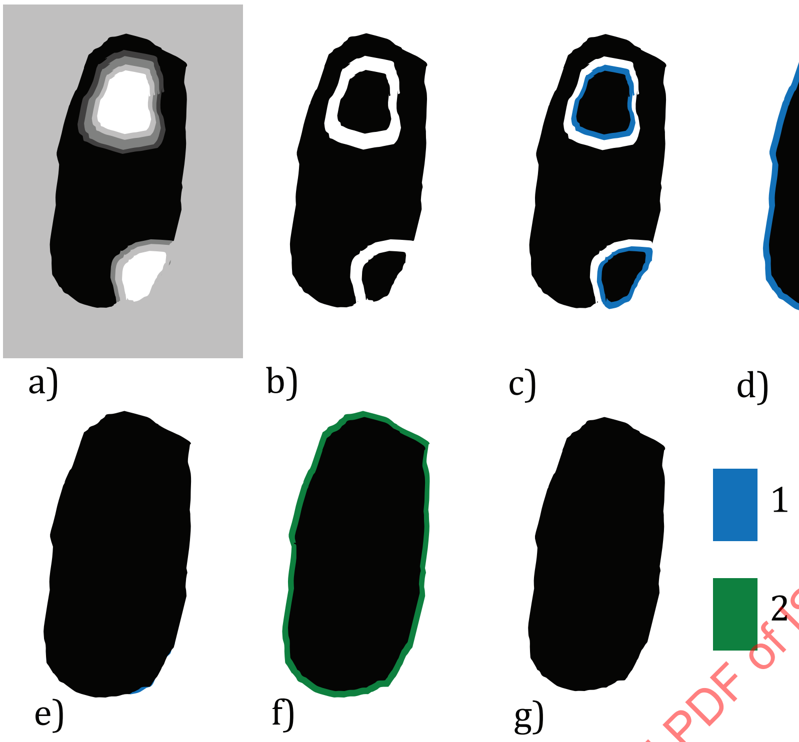
NOTE 4 The targeted form of illumination used by scanners may cause reflections from the mesh filaments to occur when analysing mesh filters, which could be recognized as (small) bright particles. Appropriate methods are used to ensure that artefacts or reflections are not included in the particle results.



Key

X	grey value
Y	pixel count
1	light-optical microscope and scanner system without polarizers
2	filter background (maximum grey value) adjusted to approx. the middle of the grey value range
3	lower binarization threshold set to 70 % of the maximum grey value
4	upper binarization threshold set at 145 % of the maximum grey value

Figure 9 — Setting the brightness and binarization thresholds for light-optical microscope and scanner systems without polarizers



Key

a) particle with pale and dark areas

b) after binarization, the particle is divided into three separate sections

c) in an initial dilatation step, the originally pale areas of the particle are magnified by one pixel

d) in a second dilatation step, all areas of the particle are magnified by one pixel and the separated structures are joined together again

e) overall contour of the particle is now enlarged

f) an erosion step reduces it in size by one pixel

g) real particle structure, which is then measured

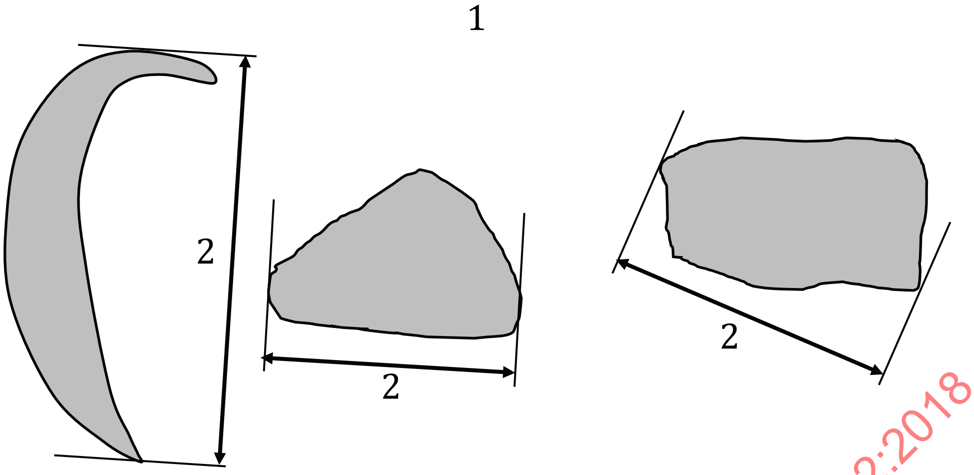
1 dilatation (growth)

2 erosion (reduction)

Figure 10 — Joining up separated particle structures with dilatation and erosion

Light-optical standard analysis can be used to determine the following particle size **measurements and features**.

1. **Particle length**: the greatest possible perpendicular distance measured between two parallel lines touching the particle. Illustrated in Figure 11, in image-processing this value is known as $\text{Feret}_{\text{max}}$. The length corresponds with the “worst-case damaging potential” of a particle capable of bridging a sensitive gap in an automotive system, e.g. contact distance between two electronic elements.



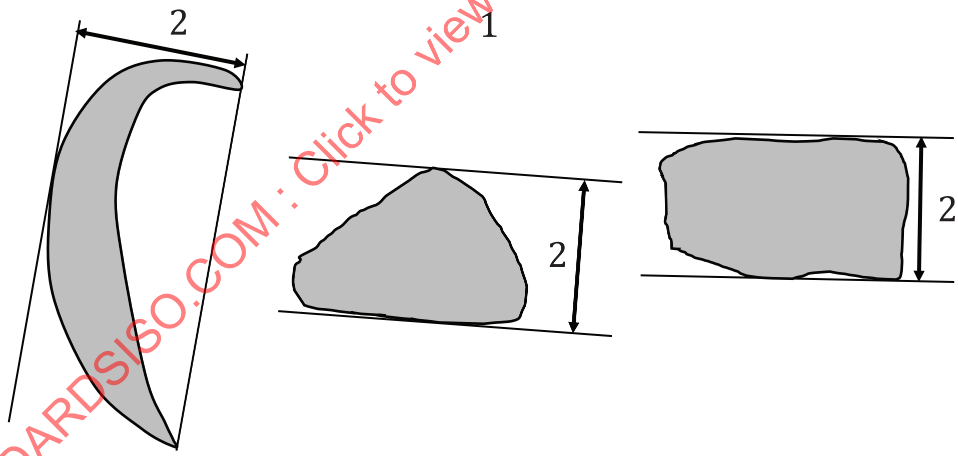
Key

1 particle length

2 $\text{Feret}_{\text{max}}$

Figure 11 — Definition of the particle length as $\text{Feret}_{\text{max}}$

2. **Particle width:** the smallest possible perpendicular distance measured between two parallel lines touching the particle. Shown in Figure 12 in image-processing this value is known as $\text{Feret}_{\text{min}}$. As far as the damaging potential is concerned, it corresponds with the width of a channel, for example, through which a particle of this size could still pass.



Key

1 particle width

2 $\text{Feret}_{\text{min}}$

Figure 12 — Definition of particle width as $\text{Feret}_{\text{min}}$

When measuring the Feret length and width, the accuracy of the measurement depends on the angle increments implemented on rotating the two parallel lines around the respective particle to determine the maximum or minimum position. The angle of rotation per increment should be ≤5°.

Two additional particle widths that can also be measured in the standard analysis are illustrated in Figure F.1.

3. **Characterizing and measuring fibres**: In any manufacturing or laboratory environment where people are present, fibres are emitted from clothing materials and are later found in component cleanliness analyses. Because textile fibres are usually very long but less critical to the function

of many technical systems, it is important to separate them from compact, potentially-damaging particles.

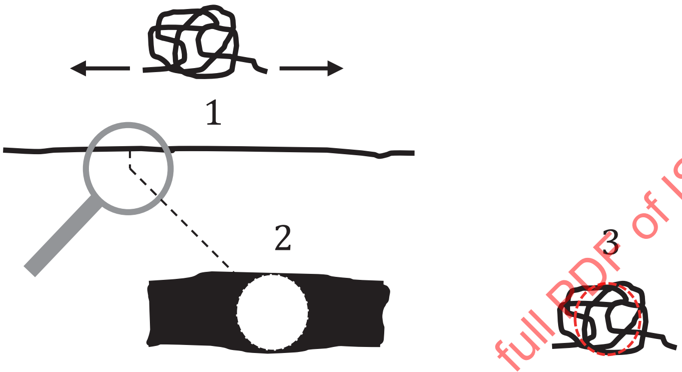
Universal, reliable separation of textile fibres and fibre-shaped potentially-damaging particles simply using light-optical techniques is not possible.

The geometric criteria for **characterizing** a structure as a fibre are:

— elongated length/maximum inner circle diameter >20 (see Figure 13), and

— width measured via the maximum inner circle diameter ≤50 µm.

WARNING — Hard fibre-shaped structures which are detected as fibres may be harmful particles. It shall be clearly documented if non textile fibres are counted as critical particles.



Key

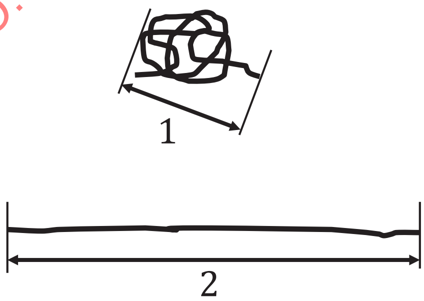
1 elongated length

2 maximum inner circle diameter as width of the fibre

3 wrong width measurement

Figure 13 — Characterizing fibres

The length of a fibre can be measured in two ways. Firstly, in the same way as with the length of any other particle, the length of the fibre is expressed as Feret$_{max}$ (see Figure 14).



Key

1 length = Feret$_{max}$

2 length = elongated length

Figure 14 — Measuring fibres

As opposed to other particles, (textile) fibres tend not to have a fixed shape, with the result that their orientation, position, bends or twist on an analysis filter is generally quite haphazard. With such an arbitrary shape, stating the length as Feret$_{max}$ is less suitable for characterizing such particles. Therefore, the length of a fibre can alternatively be expressed as the “elongated length" (see Figure 14). This length, which corresponds with an untangled, elongated fibre, can be calculated using computational image-processing methods.

WARNING — The elongated length of a fibre should only be documented after it has been verified and on completion of any manual correction and/or re-characterization steps. Re-characterization, e.g. of a fibre as a particle or vice versa, is only possible if both are measured and documented using the same size criterion.

4. **Characterizing metallic shiny particles (optional)**: When determining technical cleanliness, it may be very important to characterize metallic particles. This is partly due to the fact that metals are key materials in the manufacture of functionally-relevant systems; they thus represent the majority of contaminant particles originating from processing or assembly steps that are found on test components. Secondly, they are classified as being functionally-critical in a wide range of applications because of their material properties (e.g. hardness or conductivity).

   Metal particles can only be determined with certainty by applying extended analysis methods. Universal, reliable detection simply using light-optical techniques is not possible. This is due to their varying optical appearance, which depends on the metal and processing step the metallic particles originate from. Consequently, the colour, brightness, surface structure, shape and homogeneity of a particle's optical characteristics may differ significantly.

   Despite this, many metallic particles display a common feature: provided the particle surface is not matt, oxidized or soiled with soot or other contaminants, a metallic reflection or metallic shine occurs. This effect can be utilized to aid characterization.

   The freely-moving electrons in the metal are responsible for the feature of metallic shine. To identify such particles, two different images are analysed and compared. One analysis is carried out using crossed polarizers to detect all the particles present (pole image) and another analysis is performed using either parallel polarizers or no polarizers (bright image). If bright spots (close to or the same as the white value in the histogram) in the bright image are detected in the same areas that the pole image assigned to particles, and if these spots are dark in the pole image, such particles can be classified as having a metallic shine. If the dark value selected is close to the black value of the histogram, a clear difference can be made between particles with a metallic shine and particles with another form of shine.

   For scanners or microscope without a polarization unit, this analysis technique using a pole image and a bright image cannot be applied. Nevertheless, the occurrence of bright reflections (areas of a particle with a white value close to or the same as the white value in the histogram) can be used to pre-characterize metallic shiny particles. However, there is a risk that particles with another form of shine may be classified as being metallic shiny particles.

   A number of system requirements and parameters determine whether a particle is characterized as being metallic shiny during an automated analysis. At present it is still not possible to standardize these:

   — type of imaging lens;

   — degree of magnification or zoom level;

   — type and exact geometry of illumination;

   — procedure for characterizing metallic shine and exact parameter settings;

   — individual properties of metallic particles and individual particle orientation.

   Therefore, results of the automated characterization of metallic shiny particles can only be fully compared with one another if identical systems and identical parameter settings are used, and if particle characteristics are identical.

Despite its limitations, in many cases the characterization of metallic shine can be a very practical aid. In order to be able to use it to analyse technical cleanliness and validate cleanliness specifications, the following points need to be clarified or adhered to by the user/operator.

- Metallic particles found in components during routine inspections in a cleanliness laboratory shall be of such a kind that they can be principally characterized as metallic shiny by the respective system with the parameter settings used. To assess this, pre-tests using the extended analysis method may be required.
- When double-checking the analysis results from each filter, it should be assessed whether the results from the automated characterization can be visually confirmed by a skilled operator or whether some particles require re-characterization.

9.2.3.5 Double-check

Even when analysis filters have been prepared well and feature a uniform distribution of particles that are not too close to one another (see also 8.6, E.4 and E.5), the following unavoidable errors may occur due to optical/technical limitations:

- particles are divided up if they have areas possessing the same degree of brightness as the filter background;
- particles touch or overlap one another and are classified as being a single particle;
- particles are wrongly characterized.

NOTE Incorrect characterization can also occur when different types of particle overlap, e.g. if a small metallic shiny particle touches a fibre. In such cases, the particle is characterized as being a coherent metallic shiny object.

Due to this risk of error, the results of light-optical, automated cleanliness analyses should always be double-checked by a skilled operator. Additionally, the analysis system should be capable of joining up divided particles again, separating particles touching one another and re-characterizing particles.

It may be helpful during the double-checking step to re-inspect particles using a higher degree of magnification, a different form of illumination or another contrast method, etc., in order to verify that measurements and characterizations are correct. Although this is possible with light-optical microscopes, the mode of function of flatbed scanners does not allow it. Here, the image can only be inspected using a higher zoom level.

Any corrective steps carried out by the analysis system shall be documented and saved to enable later transparency.

9.2.3.6 Material and equipment

a) Light-optical system, e.g. material microscope, zoom microscope, stereo microscope or flatbed scanner, featuring the components and characteristics described in 9.2.3.4.

b) Reflected light illumination: when performing automated analyses, it is essential that the entire image field is homogenously illuminated at all times irrespective of the degree of magnification selected. The degree of illumination shall also remain constant during the entire procedure. In order to prevent the illumination from being altered inadvertently, it should be integrated into the light-optical analysis system. Artefacts due to reflections from the filaments of mesh filters, for example, shall be avoided.

c) Any inhomogeneities occurring on illuminating the image fields can also be compensated for by the image-processing system (shading correction with microscopes, white balance with flatbed scanners or other brightness balance, provided no information from the image that is relevant to the particle is altered).

d) Sample holder: with automated light-optical analysis, the analysis filter shall be secured so that it does not slip out of place when the sample stage is moved. All areas of the analysis filter and particles contained on it shall be within the range of the depth of field of the imaging lens. This is essential in order to obtain a good image and accurate analysis. To achieve this, the analysis filter can be tensioned, for example (like an ear drum) or pressed flat with a glass lid. With flatbed scanner systems, where the analysis filter is placed face down for the inspection, the filter shall be secured with a glass lid.

   If, for example, the analysis filter is inspected in a tensioning device without a lid, the environment shall be sufficiently clean to ensure that the analysis filter is not contaminated by further particles or fibres in the specified size range during the analysis. The tensioning device may not cover any part of the effective filter surface.

   NOTE If the analysis filter is not clamped in place by a glass lid, there is a risk that large particles could shift when the sample table is moved and be counted twice.

   When removing a glass lid used to secure an analysis filter it is probable that particles will adhere to the lid and be removed from the analysis filter. If the analysis filter is then subjected to an extended analysis, the use of a glass lid should either be avoided, the extended analysis be carried out beforehand, or the particles adhering to the lid be rinsed back onto the analysis filter (on a suction filter) with extreme care using a laboratory wash bottle.

   If an analysis filter that has translucent areas, such as the mesh of a coarser membrane filter, is analysed using a light-optical method, the sample holder should be of a similar colour and brightness as the analysis filter itself. Otherwise there is a risk that dark translucent areas will be counted as particles. The easiest way to avoid this problem is to place a non-transparent filter membrane beneath the mesh filter.

e) Motorized positioning unit: positioning axes need to be extremely accurate for the following reasons:

   - in an automated analysis, the effective filter surface shall be fully analysed without it slipping out of place (see also F.1.2 and F.1.3);
   - particles shall be reliably positioned under the lens for a manual double-check (this is not possible with flatbed scanners because of their design).

   Positioning accuracy should be in the same range as the smallest particles requiring detection.

   In cases where the sample stage has a wider range of movement, filter mounts can be realized that hold several analysis filters, which can then be subsequently analysed automatically in succession (the focal plane over horizontal distances shall remain identical).

f) Camera: the number of pixels of the camera sensor (in the case of flatbed scanners, the line camera) shall be adapted to the optical resolution of the magnification lens, i.e. the 10-pixel criterion shall be observed for the smallest particle size to be measured.

   The light sensitivity of the camera has a similar impact on the analysis image as the intensity of illumination. The camera shall be operated with defined, fixable sensitivity settings. Automatic functions that correct the brightness shall be deactivated.

g) System control and image processing: the programmes used to control the system and analyse the images shall possess the features described in 9.2.3.4.

   Additional software filters for processing the analysis images, such as for enhancing contrast or sharpen edges, may be useful for the viewer but should not be used in a light-optical standard analysis because their influence is often not known.

9.2.3.7 Procedure

The following procedure shall be adapted to the features of the respective light-optical analysis system.

a) Procure all resources required for the test.

b) Secure the analysis filter in the sample holder. In doing so, use tweezers to carefully remove the analysis filter from the drying receptacle or transport container (e.g. Petri dish) and position it as required, making sure that no particles are lost. If the analysis filter shall be secured with a glass lid, verify the state of the lid and clean it beforehand if necessary.

   NOTE Conditioning is not required if analysis filters are inspected only by light-optical analysis and not gravimetry.

   Unlike with gravimetry, filters for light-optical analysis do not need to be dried until mass constancy is attained. The step in the desiccator is therefore not necessary. Although, in principle, wet or moist analysis filters can be inspected by light-optical analysis, there is a risk that reflections due to humidity or changes in the image may occur as the filter dries under the microscope. If analysis on wet filters shall be done, nothing may be placed upon the filter surface (lid, frame, etc.).

c) Fix the sample holder onto sample stage (or place the sample holder in the flatbed scanner).

d) Set all parameters including illumination (if necessary, allow for warming-up time). Parameters can be set manually and/or (partially) automatically by the analysis system.

e) Where appropriate, check that the sample holder and sample stage are levelled.

f) As required, focus the imaging lens on the surface of the analysis filter.

g) Perform the automated analysis.

h) Verify the analysis results in accordance with 9.2.3.5.

i) Document the results.

9.2.3.8 Documentation

See Clause 10.

9.3 Extended analysis

With extended analysis, methods are applied to obtain more information about particle shape (third dimension) and/or particle composition. For example, extended analysis methods are implemented within the scope of process optimization and cause research in order to gain supplementary information about the origin of particles. Extended analyses can also be performed to better evaluate a particle's damaging potential and thus determine the most suitable action to be taken should limit values be exceeded. This is because these methods provide more detailed information than standard analysis methods.

If extended analysis methods are to be applied to validate cleanliness specifications, this shall be documented in the customer-supplier relationship together with detailed parameters of the method used. The implementation of extended analysis methods may involve significantly higher costs than those associated with standard analysis methods.

Information about physical features such as hardness, abrasiveness or other material characteristics can only be gained using extended analysis methods. In many cases, the characteristic of metallic shine, which can be determined in the standard analysis, may be a good indication of a metallic and thus of a conductive particle. However, if the metallic nature of a particle needs to be determined with certainty, extended analysis methods shall be implemented.

In some cases the light optical analysis with *modified setup* can be used where suitable setting for microscopy hardware, illumination and image analysis are chosen to capture the relevant particle structures.

9.3.1 Further light-optical analyses

9.3.1.1 Light optical analysis (modified setup)

The aim of the light optical analysis with modified setup could be a more accurate detection and measurement of particles e.g. with low contrast to the filter background. This includes a very rigorous double check after the automated counting.

When performing a light optical analysis with modified setup, a more detailed knowledge of microscopy and image analysis is necessary, than in the standard analysis with standard setup.

Like in the standard analysis it is very important to have:

— a well prepared analysis filter (see 8.6),

— a defined illumination without uncontrolled influence of foreign light,

— well focused field of view, and

— a stable illumination (e.g. warming up of light source if necessary).

NOTE If using the light optical analysis with modified setup the analysis results can vary from results of the standard analysis.

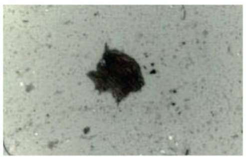
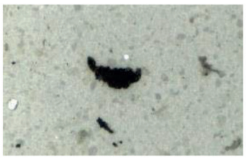
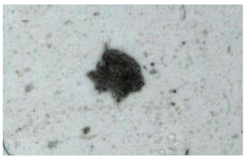
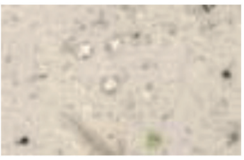
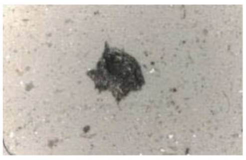
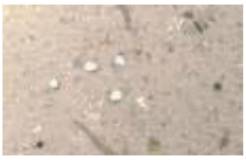
The following not exhaustive list gives examples for settings that can be used to optimize particle measurement.

a) **Illumination modes** e.g. incident/reflected light (bright field or dark field), transmitted light, polarized illumination etc. In some cases a special filter holder is needed e.g. for transmitted light (see Table 6).

b) **Background correction** is a way to correct heterogeneous lighting on the image field.

c) **Binarization thresholding**, the threshold value can be changed or an adaptive thresholding can be used.

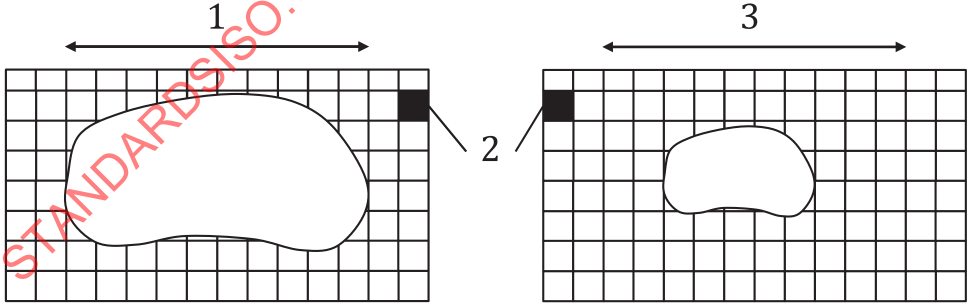
Table 6 — Examples of how particles are visualized depending on the illumination mode

Illumination mode	Types of particles			
	Dark particles	Transparent/clear particles	“Metallic shiny” particles	Fibres
Incident light *90 ° cross-polarization*				
Incident light *Dark field*				
Incident light *Bright field*				
Transmitted light *Bright field*				

1) **Sizing of particles:**

— if necessary, other size definitions for particle length and width than described in the standard analysis can be used if agreed between parties;

— other pixel resolution for sizing particles than described in the standard analysis (10 Pixel per particle) can be used e.g. if a more precise measurement is needed more pixels can be used or for counting 5 µm and 15 µm particles it can be suitable to use 5 Pixels (see Figure 15).



Key

1 minimum particle size = 10 camera pixels

2 pixel size

3 minimum particle size = 5 camera pixels

Figure 15 — Example for counting small particles with a resolution of 5 camera pixels per particle

2) **Image processing**: For some reasons like increasing of contrast, sharpness, separating of neighbour particles, filling of holes etc. further image treatment operations could be helpful, like:

   - closing (dilation followed by erosion using the same size pixel matrix for both operations); it consists in gap filling with outline smoothing and connection of nearby objects;
   - opening (erosion followed by dilation using the same size pixel matrix for both operations); it consists in removal of small artefacts with outline smoothing;
   - filling; it consists in filling in all holes, no matter what their size is, while preserving the outlines of particles.

   Care shall be taken, because the original image information is changed when image treatment operations are performed.

9.3.1.2 Additional optical features

In the extended analysis, optical features such as shape, colour and surface texture can be used to characterize particles.

9.3.1.3 Particle height — Principle

With this analysis method, the height of a particle is determined with the aid of the depth of field of a microscope lens (T). In simple terms, the depth of field can be expressed as the ratio between the wavelength (a value of 550 nm can generally be assumed here) and numerical aperture (nA) of the lens used.

$$T = \frac{550}{nA^2}$$

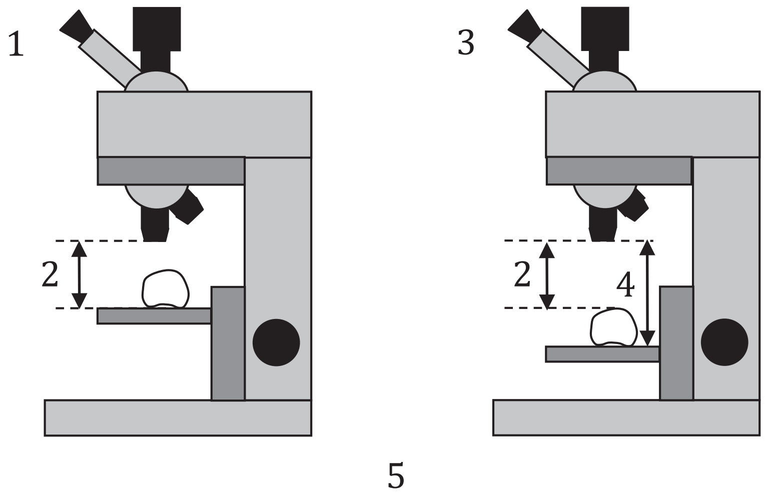
The numerical aperture influences the lateral resolving power of the lens as well as its depth of field. As a rule, the numerical aperture increases if fixed lenses with mounting degrees of magnification are utilized. Therefore, as magnification increases (2,5 × -> 5 × -> 10 × -> 20 ×), so also does the lateral optical resolving power, whereas the depth of field decreases, i.e. the range within which structures of different heights can be clearly visualized in a plane (focal plane).

Microscope systems with fixed, high-resolution lenses possess degrees of magnification upwards of approx. 20 × (200-fold total magnification at lens level) and a depth of field which is low enough to prevent objects with varying heights from being visualized clearly in one focal plane. As a result, there is a difference between the upper and lower focal plane. This difference indicates an object’s height.

The theoretical accuracy of this method depends on the numerical aperture of the lens used and can be said to be 2 × the depth of field.

To assess height, first of all the bottom of the particle (i.e. filter background) is brought into focus. Then the lens is adjusted along the z-axis until the top of the particle is in focus.

The particle height is the difference between these two focal planes, or the distance travelled by the lens in the z-direction. It is calculated indirectly via the screw pitch and stepmotor of the z-axis (see Figure 16).



Key

1 step 1 focus on the background of the analysis filter
2 focal level z_1
3 step 2 focus on the highest point of the particle
4 focal level z_2
5 particle height z = 4 − 2

Figure 16 — Principle of assessment of particle height

In order to best use the method for assessing particle height via the difference between the focus positions of the filter background and the highest point of the particle, the particle should lie flat on a planar analysis filter. If a mesh filter is used, care shall be taken to ensure that the particle does not “protrude” into the pore structure as this would result in a lower height being measured.

9.3.1.4 Material and equipment

a) Light microscope with parallel beam path.

b) Lens with a numerical aperture >0,4. This generally corresponds with a 20-fold degree of optical magnification (200-fold total magnification at lens level).

c) High-resolution motorized fine focus (resolution in submicron range).

d) Software to read the focus positions.

e) Filter holder without cover.

Analysis filters may not be covered with a lid after the filtration step as this could press particles into the analysis filter and falsify results.

9.3.1.5 Procedure

a) General analysis with a low-magnification lens, e.g. 2,5 × or 5 × (to determine two-dimensional information about particle length, width, surface area, etc.).

b) After the scanning step, measure the height of critical particles using a lens with a higher degree of magnification (numerical aperture >0,4).

c) The particle concerned is automatically brought into focus, selected and memorized.

d) Bring the lens with the higher degree of magnification into position.

e) Adjust the contrast to enhance particle topography.

f) Focus on the background of the analysis filter near the particle to be analysed.

g) Confirm this 1st focus position.

h) Software reads the 1st focus position.

i) Focus on the highest point the particle.

j) Confirm this 2nd focus position.

k) Software reads the 2nd focus position.

l) Analyse the focus positions to determine the difference in height.

m) Document difference in height in overall results.

9.3.1.6 Documentation

The type of optical system used shall be clearly documented in the analysis report as zoom-based systems are not generally suitable for this analysis method.

The results obtained shall be listed in a separate table in the analysis report. In order to be able to estimate the damaging potential of the particles measured, not only the height of particles but also their length and width should be documented.

9.3.2 SEM/EDX

9.3.2.1 Principle

SEM/EDX analysis is a combined analysis technique which counts and measures particles using a scanning electron microscope (SEM) and determines their material composition by means of energy-dispersive X-ray spectroscopy (EDX). SEM/EDX analyses can also be performed manually on individual particles or on particles pre-selected in the light microscope if particle coordinates are transferred to the SEM/EDX system. This section of the document only addresses the automated analysis of complete analysis filters. By assigning particles and their size to specific material classes (derived from their elemental composition), supplementary information can be gained about their damaging potential or origin.

As with light-optical analysis, to ensure that single critical particles are detected, the entire filter surface shall be inspected.

If used for monitoring purposes, in order to detect high particle counts in small particle size channels, only a part of the analysis filter is inspected and this value is then extrapolated to the total filter surface area. However, particle occupancy in the areas analysed shall be representative.

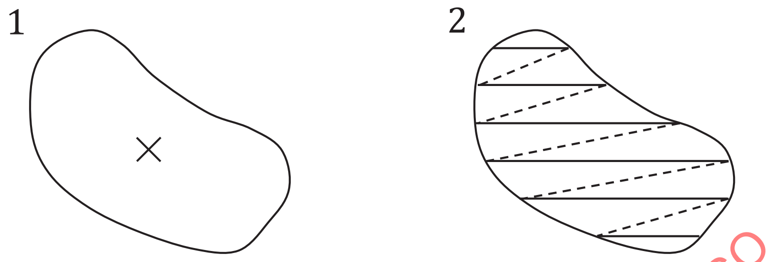
Again, as with light-optical analysis, in order for particles to be recorded, measured and analysed correctly, the analysis filter shall be carefully prepared to ensure that particles are isolated from one another and do not overlap on the filter surface.

A pre-requisite for detecting and visualizing particles effectively with SEM is a good material contrast, which is obtained due to the difference in the intensity of electrons that are back-scattered from the various chemical elements.

IMPORTANT — Because their principles of detection are based on different contrasting methods, it is not possible to compare the analysis results obtained from light-optical microscopy with those from scanning electron microscopy.

Again, in the same way as with light-optical analysis, a threshold value (binarization threshold) is set to define which image areas should be allocated to particles and which to the filter background. Due to the different contrast ratios in SEM, the background of filters containing carbon appears dark with particles containing elements heavier than carbon appearing paler (the heavier the elements in the particle, the paler they appear).

At the points on images which have been assigned to particles, an EDX-spectrum is recorded. The energetic position of the lines in the X-ray spectrum is characteristic for the chemical element they originate from. The analysis of the line spectrum enables the corresponding elements to be quantified. From the results, conclusions can be drawn about the chemical composition of the particles detected. In order to correctly characterize a particle in its totality, the electron beam of the EDX analysis may not stop at any one spot on the particle but instead scan the full surface of the particle, as shown in Figure 17.



Key

1 NOK: A fixed position of the electron beam is not suitable for correct particle analysis

2 OK: The electron beam passing the entire surfaces of the particle e.g. moving along a grid structure is required for correct particle analysis

Figure 17 — Movement of the electron beam in EDX analysis

To obtain an accurate quantitative result, it is important that the distance specified for the respective device is maintained between the lower edge of the pole shoes and the surface of the sample (typically between 10 mm and 25 mm).

As high numbers of particles are generally analysed in automated particle analysis with SEM/EDX, an EDX analysis time of between one and a few seconds per particle is selected to compromise between the counting rate of X-ray impulses needed to identify the particle and the shortest time required for the analysis. To confirm an element analysis, it should be possible to approach single particles again after the fully-automated analysis in order to verify the analysis result using a longer measuring time and higher rate of X-ray impulses.

Limitations: particles are assigned a specific material class solely on the basis of their elemental composition. Due to the limited energy resolution of EDX detectors, some results may be ambiguous.

The lack of material contrast offered by the background of organic analysis filters makes it difficult to detect organic particles. In consequence, organic substances are detected but their carbon content invariably prevents them from being classified in more detail. An exception to this is plastics which contain not only carbon, nitrogen and oxygen but also other elements, such as halogenated plastics (chlorine in PVC, fluorine in PTFE).

NOTE By preparing samples on metal membranes, organic particles, plastics, boron nitride and boron carbide can be measured and counted.

9.3.2.2 Material and equipment

a) Scanning electron microscope with:

  - adjustable vacuum; the residual air molecules in the vacuum chamber cause charges in the sample due to the electron beam to be discharged. Therefore, if low-vacuum devices are used, samples do not require sputtering or vaporizing;
  - automated x-y table;

- mounting device to hold one or several analysis filters;
- back-scattered electron detector (BSE) to record images;
- constant beam current (<1 % fluctuation per hour).

b) X-ray detector with analysis system (EDX system) to enable element analysis

c) Where appropriate: particle fixative to bind particles to the analysis filter membrane and discharge any electrostatic charges (if particles become charged, image errors may occur or particles may "jump" due the electrostatic forces, causing them to be excluded from the analysis or counted twice).

d) The same requirements apply for these systems as for light-optical analysis systems with regard to the resolving power, the option to reconstruct images and particles and the particle dimensions to be measured.

9.3.2.3 Procedure

The following procedure shall be adapted to the features of the respective SEM/EDX system.

a) Where appropriate: fix particles onto the analysis filter using a suitable particle fixative. If gravimetric analysis is also required, the fixative may only be applied after the gravimetric analysis has been performed.

b) Place the analysis filters in the SEM/EDX system and evacuate the chamber until a stable working pressure is attained.

c) The z-position: set the height of the sample stage so that the analysis filter is located at the working distance specified for the system.

d) Set the acceleration voltage of the SEM to 20 kV for the element analysis (standard setting). This enables an X-ray spectral range for the analysis to be used that keeps X-ray lines well apart from one another and allows elements to be clearly identified. In specific cases, the acceleration voltage may need to be adjusted; this shall be documented.

e) The beam current selected for the analysis depends on the compatibility of the sample, the counting rate of the EDX detector and the desired analysis time per particle.

f) If necessary: warm the cathode to stabilize the beam current.

g) Set the brightness and contrast of the BSE image so that all relevant elements are detected and the dynamic range of the image processing system is optimally used.

h) Start the automated analysis.

9.3.2.4 Documentation

See Clause 10.

9.3.3 LIBS

9.3.3.1 Principle

Laser induced breakdown spectroscopy (LIBS) is a form of emission spectroscopy that enables the elemental composition of particles to be determined. When inspecting technical cleanliness, the material composition of particles is analysed on the filter membrane or adhesive pad on the basis of the emission lines in the spectrum. This enables direct component analysis and, because material is removed, an in-depth analysis of sample.

In the analysis, the lens focuses a laser pulse onto a particle. The laser pulses used have a wavelength ranging between 331 nm and 1 064 nm. The laser pulse vaporizes the material and generates plasma.

Depending on the system, the wavelength range inspected lies between 400 nm and 800 nm. Depending on the measuring system implemented the resolution of the spectrum, i.e. the quantity of points per spectrum, lies between 0,01 nm and 1 nm.

Pure metals generally emit a strong signal. The strength of the signal is mainly determined by the point of vaporization of the material concerned. Thus, the higher the quantity of material vaporized by the laser pulse, the stronger the signal becomes. Because elements each have a specific spectrum, metals and inorganic materials can be clearly identified by means of a spectrum databank. Polymers and elastomers can only be identified if they are mixed with inorganic materials (if present). If a stronger laser pulse is used, fragments of organic molecules can be visualized, thus facilitating the classification of polymers.

Modern systems are capable of analysing particles with a minimum size of approximately 15 µm. The quality of the signal varies significantly according to the numerical aperture of the lens and the light throughput of the spectrometer. The size of the focal point of the laser is also determined by the numerical aperture of the lens and the quality of the laser beam. An impact crater with a diameter of 20 µm and a depth of approximately 10 µm is analysed per particle. The analysis time is less than one second. The method is quantitative and can also be used to identify and classify alloys. Depending on the resolution of the spectrum, individual constituents of an alloy may overlay other materials in the spectrum and impair their identification.

9.3.3.2 Material and equipment

As 47 mm or 25 mm round filters made from cellulose nitrate or polyester mesh filters only generate weak LIBS signals, they are therefore an ideal background for detecting specific interference-free LIBS signals from particles.

It has proved useful to fix the membrane and particles with a fixative emulsion. This prevents particles from being moved by the impulse generated by the laser.

The main constituent of the LIBS microscope is an infinity-corrected microscope. A laser is coupled with a mirror in front of the imaging camera and the LIBS signal is decoupled. Typical laser wavelengths are 331 nm and 1 064 nm.

Systems with an integrated microscope are capable of automated particle detection (see light microscopy) and determine the coordinates of a particle's centre of gravity. Particles are then aligned with the laser focus.

The light is split into spectra by a grid and recorded by a CCD (Charge coupled device). A microcomputer analyses the signal, processes the spectra and compares them with the spectra contained in the databank. The latter compares the similarity of the two spectra and weights the result as a point score. A successful classification is assigned a point score >700 with a maximum score being 1 000.

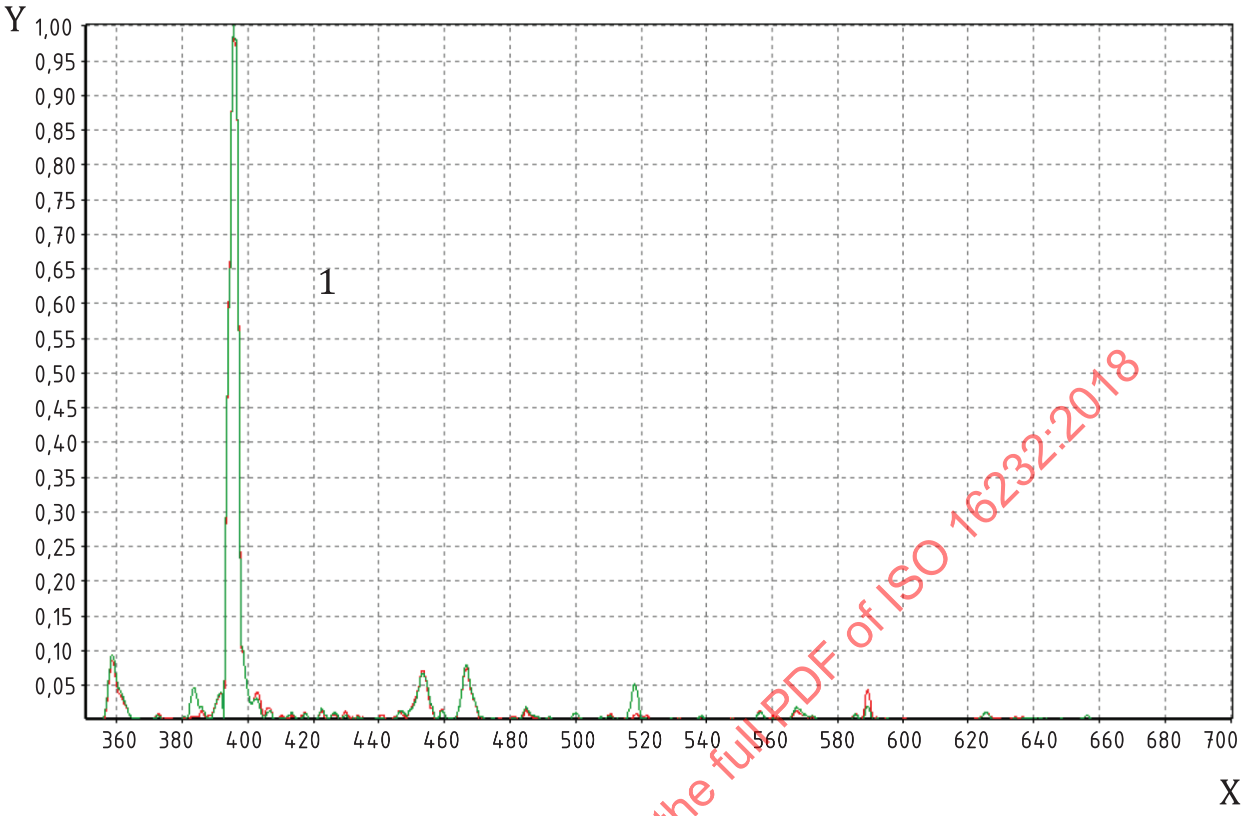
9.3.3.3 Procedure

The operator places the test filter in the analysis device and starts the automated analysis. Particles for analysis are selected according to their length or other parameter of shape. The operator then enters an appropriate databank to enable automated interpretation of the spectra obtained. If the match of a particle spectrum exceeds the score of 700 (see Figure 18), the name of the material is displayed by the device and documented in the test report.

Some analysis systems also allow a manual double-check to be performed on completion of the automated particle analysis. The operator can then select additional measuring points or different focus points or choose to bombard the same point repeatedly in order to penetrate further into the material and analyse deeper layers.

9.3.3.4 Documentation

An example of a LIBS analysis comparing the particle spectrum with a reference spectrum is shown in Figure 18.



Key

X wavelength, nm

Y signal height

1 red: aluminium spectrum, green: particle spectrum

Figure 18 — Comparison of LIBS spectrum for aluminium (green) with the particle spectrum. Match: 996 points, quality of the spectrum: 171, laser 331 nm

For a diagram of a spectroscopic set up see F.3.

9.3.4 Raman spectroscopy

9.3.4.1 Principle

Raman spectroscopy is a form of oscillation spectroscopy, which enables the structure of a particle to be analysed. The atoms in the molecule and their arrangement relative to one another create a specific material fingerprint.

In the analysis, a laser beam is focused onto a particle by a lens. The interaction of the matter with the laser beam generates a signal that is recorded by a CCD camera. The signal is known as a spectrum. Depending on the system, the wavelength range considered lies between 400 and 3 500 wave numbers. The resolution of the spectrum, i.e. the quantity of points per spectrum, varies between 2 and 12 wave numbers according to the system implemented.

The system is only capable of analysing molecules (pure metals do not generate a signal). Organic and inorganic materials, provided they are Raman active, can be clearly identified by a spectrum databank because their structure generates a specific spectrum.

The minimum size of particles for analysis depends on the factor of magnification of the analysis lens. In principle, particles upwards of 0,5 µm can be detected. The quality of the signal is strongly influenced by the numerical aperture of the lens and the light throughput of the spectrometer. The size of the laser focus point also depends on the numerical aperture and ranges between 0,5 µm and 8 µm.

Typical analysis times per particle range between 30 s and 60 s. In some cases, fluorescence may be excited by the laser beam. This could overlay the Raman signal and hamper classification.

9.3.4.2 Material and equipment

As metals do not generate a Raman signal, they form an ideal background for detecting a specific Raman signal from a particle without any interference. Special metalized plastic mesh filters are available for this purpose. Materials from particles upwards of 0,5 µm emit clear signals if they are prepared for analysis on such filters.

47 mm or 25 mm round filters made from cellulose nitrate or polyester mesh filters can also be used with larger particles without the need for additional sample preparation. However, if this procedure is implemented, the particles should effectively screen the filter background to prevent these organic materials from contributing significantly to the Raman signal.

The main constituent of the Raman microscope is an infinity-corrected microscope. A laser is coupled with a mirror in front of the imaging camera and the Raman signal is decoupled. Typical laser wavelengths are 532 nm and 785 nm. The laser output is adjustable. During the Raman analysis, the sample can be observed with a digital camera.

Systems with an integrated microscope are capable of automated particle detection (see light microscopy) and determine the coordinates of a particle’s centre of gravity. The particles are then aligned with the laser focus.

The light is split into spectra by a grid and recorded by a cooled CCD. A microcomputer analyses the signal, processes each spectrum and compares it with the spectra in the databank. The latter compares the similarity of the two spectra and weights the result as a point score. A successful classification is assigned a point score >700 with a maximum score being 1 000.

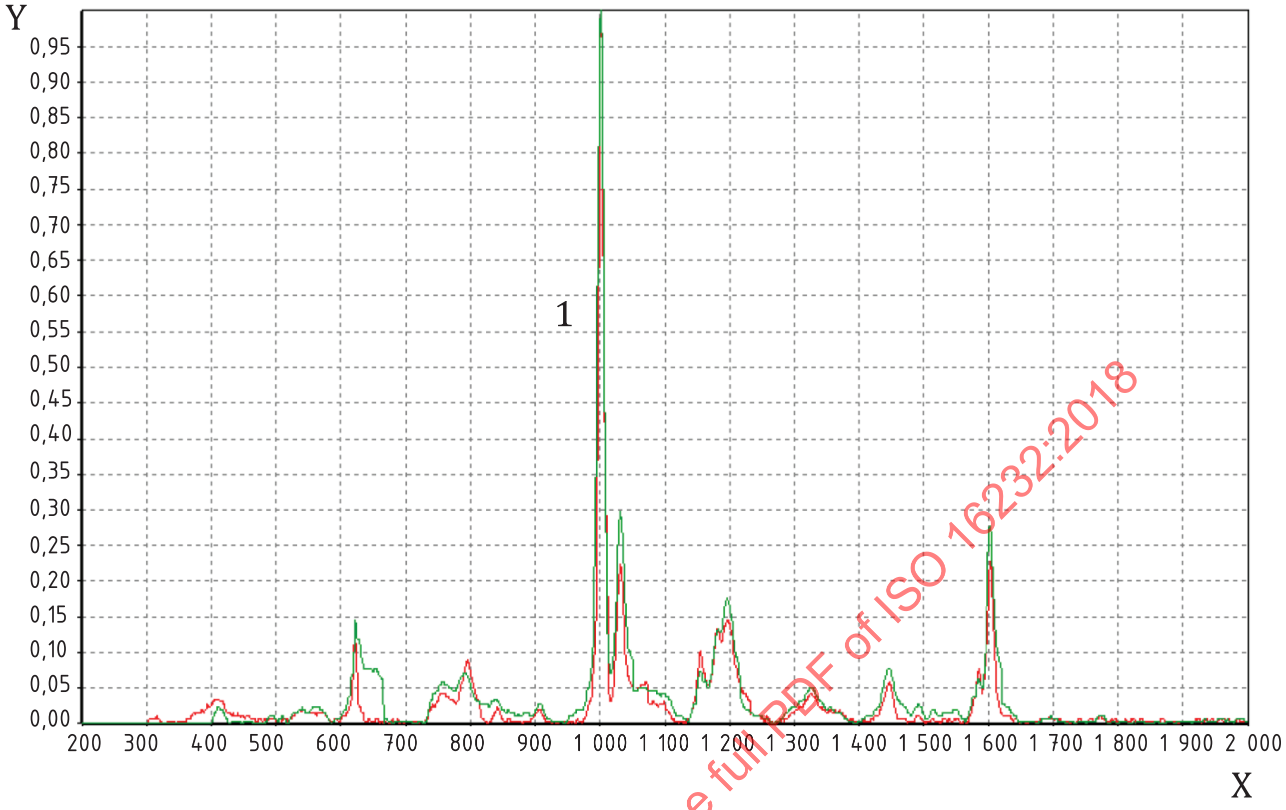
9.3.4.3 Procedure

The test filter is placed in the analysis device and starts the automated analysis. Particles for analysis are selected depending on their length or other parameter of shape. Standard parameters for spectroscopy are a laser output of 5 mW to 10 mW and an exposure time of 30 s to 60 s per particle. The operator then enters an appropriate databank to enable automated spectrum interpretation. If the match of the particle spectrum exceeds the score of 700, the name of the material is displayed by the device and documented in the test report (see Figure 19).

Some analysis systems also allow a manual double-check to be performed on completion of the automated particle analysis. The operator can then select additional measuring points or different focus points on the particle. In this mode, the laser output and exposure time can be adapted to the respective particle material.

9.3.4.4 Documentation

An example of a Raman analysis comparing a particle spectrum with a reference spectrum is shown in Figure 19.



Key

X Raman shift, 1/cm
Y signal height
1 red: polystyrene spectrum, green: particle spectrum

Figure 19 — Comparison of Raman spectra for polystyrene (green) with particle spectrum. Match: 966, quality of spectrum: 101. Exposure time: 60 s, laser 785 nm, intensity 30 %

For a diagram of a Raman system, see F.3.

9.3.5 IR (infrared spectroscopy)

9.3.5.1 Principle

If organic molecules are bombarded with electromagnetic waves in the infrared range (wave number 4 000 cm^{-1} to 400 cm^{-1} or wavelength approximately 2,5 µm to 25 µm), specific energies are absorbed. The energies absorbed depend on the oscillation energy/frequency of the excited groups of atoms or complete molecule. In the process, different forms of oscillation occur, such as stretching oscillation or deformation oscillation. All symmetric forms of oscillation are Raman-active but not IR-active (see 9.3.4).

Fourier transform infrared spectrometers are generally utilized. These feature a shorter analysis time and a better signal-interference ratio than dispersive spectrometers.

The resulting IR-spectrum is generally shown as a transmission graph, i.e. the ability of the excitation beam to penetrate the sample (expressed in percent) against the reciprocal value of the wavelength (wave number, unit cm^{-1}).

As the energies absorbed by the structure or functional groups are dependent on the substances contained in the sample, the method can be used to detect all functional groups present that are infrared-active. Although individual structural elements can be identified via the absorbed energies or bands forming in the spectrum, a library search is usually carried out with the complete IR-spectrum obtained.

Depending on the size of the databank used, most organic compounds or materials can be identified. Among others, these include plastics as well as process media, such as greases and oils. In order to identify as many unknown samples as possible, it is recommended that the existing databank be extended by adding all the in-house operating and process media used.

The two common FT-IR technologies implemented for particle analysis are IR microscopy and the ATR method (attenuated total reflexion).

Two different FT-IR analysis techniques are suitable for analysing particles (see Table 7): the ATR method (attenuated total reflexion) or transmission analysis. In principle, both methods can be carried out using a simple IR spectrometer or an IR microscope (combined with imaging).

ATR method: The IR beam is coupled and de-coupled again via a crystal which is placed/pressed on the particle requiring characterization. Due to the functioning principle of the method, information can only be gained to a penetration depth of 1 µm to 3 µm. Placing the sample is not difficult. The method is especially suitable for investigating strongly-absorbent materials.

Transmission: with this method, an IR beam passes through the particle requiring analysis (which has been pressed thin enough to enable this) and the absorption spectrum is recorded. The spectral information originates from the complete particle volume.

9.3.5.2 Material and equipment

a) FT-IR spectrometer or FT-IR microscope linked to a databank.

b) If required: tweezers or needle to transfer the particle from the analysis filter to the analysis device.

c) Where appropriate: diamond pressing cell (with transmission).

d) To transfer individual particles from the analysis filter, a stereo microscope can be used, for example.

9.3.5.3 Procedure

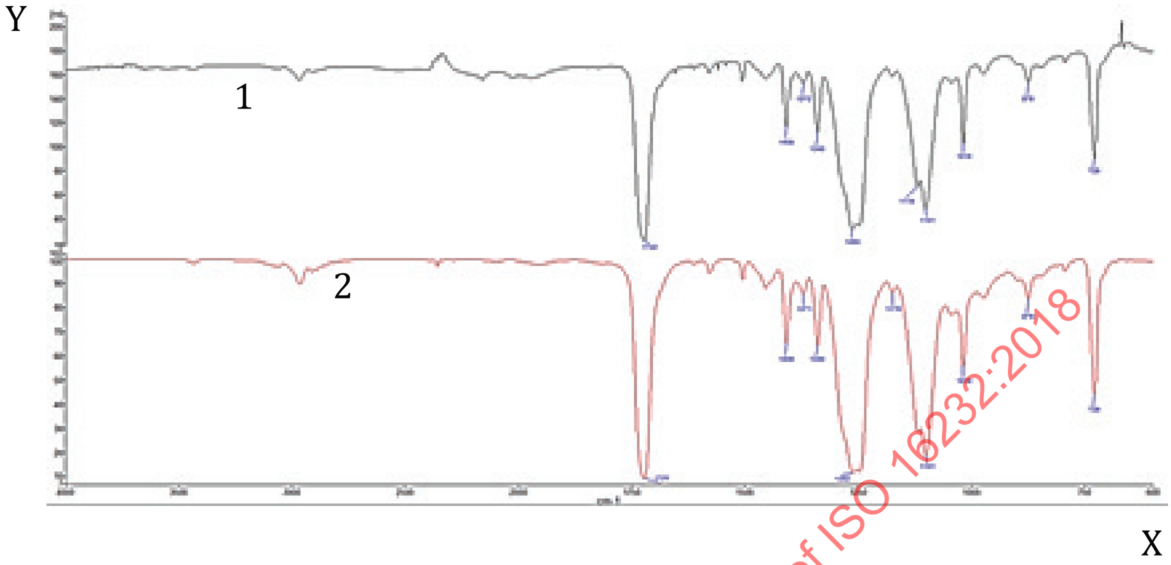
Table 7 — FT-IR analysis techniques for analysing particles

Analysis method	IR spectrometer	IR microscope
ATR	Pick up particle from analysis filter with tweezers/needle and place it on the ATR crystal. Use stamp to press particle onto ATR crystal.	Approach particle on analysis filter (no manual particle transfer required). ATR crystal is lowered onto/placed on particle.
Transmission	—	Pick up particle from analysis filter with tweezers/needle and place it on window of the diamond pressing cell. Apply opposing part of pressing cell and make the particle thinner by pressing it. Place pressing cell in the beam path of the IR microscope and perform analysis.

9.3.5.4 Documentation

When documenting results, the comparison of IR-spectra is generally included in the report, i.e. the spectrum of the sample compared with the nearest match in the databank (see Figure 20).

As well as listing all the equipment used in the test, precise details of the IR-spectrometer and associated microscope (manufacturer, model, etc.) shall be included in the report.



Key

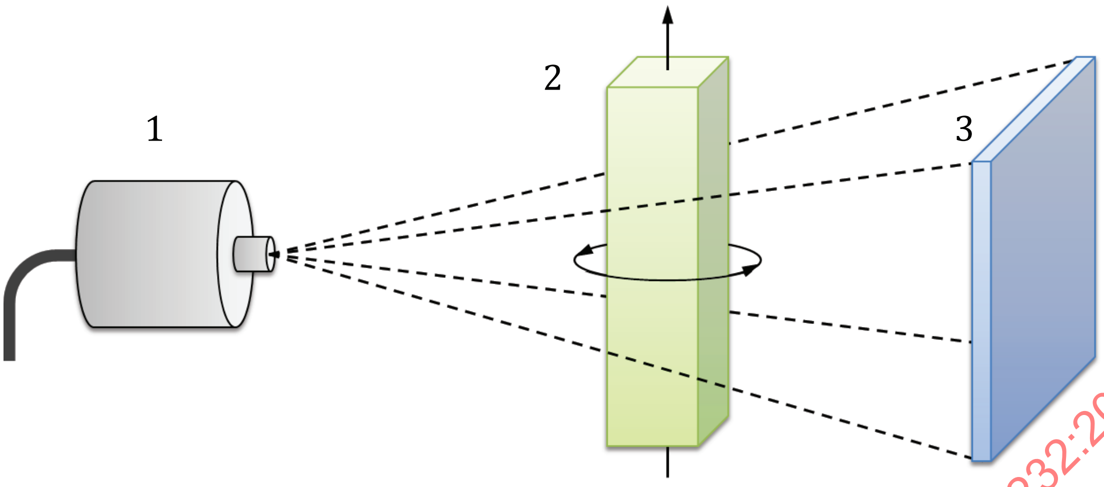
X	wave number, 1/cm
Y	IR-signal
1	IR-spectrum of PET (black)
2	nearest match in the database (red)

Figure 20 — IR-spectrum of PET (black, top) and nearest match in the data library (red, bottom)

9.3.6 X-ray microtomography

9.3.6.1 Principle

With X-ray microtomography, a finely-focused X-ray beam is used to project an image of the test component into an X-ray camera (see Figure 21). During data capture, the test component is rotated step-by-step and an X-ray image recorded at each new angle.



Key

1	micro focus X-ray source
2	rotating test object
3	flat panel detector

Figure 21 — Principle of computer tomography

The test object analysed is a suitably-prepared filter membrane charged with particles. By means of segmentation, particles are separated from the background and then analysed individually. In the analysis, not only is the geometry of a particle measured 3-dimensionally, but the analysis of the grey values also enables conclusions to be drawn about particle density. Consequently, a differentiation can be made between lightweight and heavy materials, which may help to identify the origin of particles. However, the use of X-ray microtomography does not allow finely-resolved material analysis, such as with spectroscopic methods (EDX, IR and Raman).

9.3.6.2 Material and equipment

To analyse micro-particles, a high-resolution X-ray microtomography system is required ("micro CT"). The system shall be equipped with a high-resolution X-ray camera and a micro-focus X-ray source. The diameter of the focal point of the X-ray corresponds with the smallest detectable particle size. In order to be able to analyse the complete filter in a single scan, a surface detector serves as the X-ray camera.

Particles can be examined singly on the analysis filter or in their entirety. If single particles are examined, they are fixed onto a piece of adhesive tape or polystyrene substrate. The analysis filter is then compacted by rolling or folding it to minimize the sample diameter. This enables a high special resolution to be achieved on the complete analysis filter.

The scan is analysed by segmenting the reconstructed grey value data with suitable software. In addition, the software shall be able to measure the particle's dimensions of interest (length, thickness, elongation, surface volume, bounding box, density index, etc.) from the segmentation.

9.3.6.3 Procedure

a) particle fixation/compaction of analysis filter;

b) X-ray scan and reconstruction;

c) segmentation and particle analysis;

d) compilation of report.

9.3.6.4 Documentation

With X-ray microtomography, it is especially important that recording parameters are documented: X-ray energy (kV), tube current (µA), size of focal point (µm), voxel size (µm), material and density of the X-ray filter, material of the density reference, if used.

9.4 Shortened analysis

9.4.1 General

Two analysis methods are described which require an extraction step but not a filtration step for the actual analysis. This enables analysis results to be obtained much faster, i.e. higher quantities of components can be analysed within the same period of time. Consequently, shortened analysis methods are highly suitable for the purpose of process monitoring.

The results of the two shortened analysis methods addressed are not as detailed as those of a standard analysis. Therefore, they may not be used to validate cleanliness specifications unless this has been explicitly arranged in the customer-supplier relationship.

9.4.2 Liquid particle counter

9.4.2.1 Principle

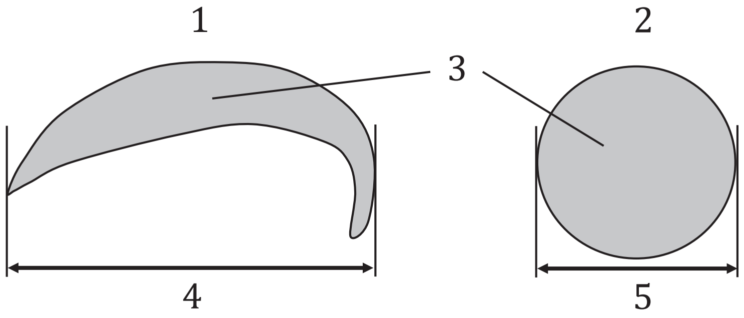
Extinction particle counters [automatic particle counters (APC)] can be utilized to classify the size and number of particles present in a liquid. These devices are not used to detect single relatively large killer particles, but rather to determine the occurrence of particles sized between 5 $\mu m_{(c)}$ and approximately 70 $\mu m_{(c)}$.

NOTE The use of $\mu m_{(c)}$ means that particle size measurements are carried out using an automatic particle counter which has been calibrated in accordance with ISO 11171.

The fact that particles are detected directly in a liquid makes these devices very easy to use and enable their implementation in a largely-automated analysis step. Continuous real time particle-counting or sample analysis can also be designed for immediate further processing in data monitoring systems.

Due to the detection principle, the test liquid has to flow through the particle sensor with a defined flow rate. With non-flowing samples, such as liquids contained in a beaker after extraction by ultrasonic vibration, a sample feed unit is used — often on the basis of an automated pressure-rinsing system. If the liquid flows through tubing, as is the case when internally rinsing a component, the sensor can in principle be connected up directly to the tubing.

The sensor contains a capillary-shaped measuring cell, through which a beam of light is shone. The intensity of the beam is registered continuously by a photodetector. Any particles contained in the liquid as it flows through the cell cause the light impulse to weaken and this weakening is proportional to the particle's projected surface area. The geometry of the particle, such as length or width, cannot be determined with this method. The degree of weakness in the light signal (shaded detection area) is equated to the surface area of an equivalently-sized circle and its diameter is then expressed as the particle size (see Figure 22 for an example).



Key

1	particle to be measured	4	length = $\text{Feret}_{\text{max}}$ = 50 µm
2	circle with equivalent surface area	5	length = diameter = 25 µm
3	surface area = 490 μm^2		

Figure 22 — Diameter of a circle with an equivalent surface area as a measure of particle size

The more a particle is shaped like a thin chip or fibre, the greater the deviation between the particle size displayed by the particle counter and the $\text{Feret}_{\text{max}}$ length determined.

Gas bubbles or droplets of foreign liquids in the test liquid are (falsely) identified and documented as solid particles.

If using an APC for monitoring purposes, there is no need to detect or visualize a range of different particle features. Furthermore, in general only (small) amounts of the respective extraction liquid or liquid sample are analysed. The statistical application is based on the assumption that particles are distributed fairly homogeneously in the extraction liquid and that any changes in the cleanliness quality or deviations in a process will show up as an alteration in the mean particle size distribution. In order to assess or compare component cleanliness on the basis of particle concentrations measured in random liquid samples, the total volume of liquid used in the extraction procedure shall be taken into consideration (dilution of the particle load).

Although the analysis devices are easy to operate, special care shall be taken when preparing and feeding the extraction liquid in order to avoid the formation of air bubbles, for example. If the maximum concentration is exceeded for the device concerned, numerous counting errors will arise due to particles overlapping in the measuring cell (coincidence concentration). This can be avoided by diluting samples before carrying out the analysis.

Current developments in complex sample feed systems should enable the use of such devices directly at the outlets of extraction cabinets in the future; this will make it possible to analyse extraction liquid in realtime without the need for manual sample preparation steps.

To calibrate APCs, silica test dust in oil or spherical polystyrene particles in water are utilized.

Counting functions are verified using liquid samples containing defined quantities of particles.

With the aid of tests or theoretical considerations, it is possible to assess when “sedimentation losses” may occur. There are physical limitations to the ability of heavy, generally relatively-large particles to be transported through sample feed lines.

9.4.2.2 Material and equipment

As well as the analysis device and corresponding sample feed unit, sealable sample containers, beakers and filtered test liquid are required. The filtered liquid is used to condition the system before use and determine its blank level. If particle concentrations in samples exceed the measuring range of the APC, this liquid is also utilized to dilute the sample in a defined manner. Protective eyewear and gloves may also be required. An ultrasonic bath can optionally be used to homogenize liquid samples and remove any gas contained in them. Samples can also be homogenized with the aid of an automatic swilling or agitation unit. To keep particles in standing samples better in suspension, a stirrer should be utilized. Magnetic stirrers are unsuitable because they bind ferromagnetic contaminant particles and exclude them from the analysis (falsification of results).

9.4.2.3 Procedure

The sample is analysed in compliance with standards such as USP 788 or ISO 11500. When preparing and configuring the analysis set-up, it is essential that no air bubbles are present in the sample.

Standing liquid samples:

Before being analysed, all standing samples shall be agitated to homogenize particle distribution. A subsequent resting phase is then required to enable any air bubbles to rise and dissipate; at the same time, the sample should be kept homogeneous by stirring it at an appropriate speed.

NOTE Floating particles (e.g. lightweight fragments of plastic) cannot be detected with this analysis technique.

Use with pressurized tubing:

This method is generally used in conjunction with a functional test bench or apparatus for rinsing the interior of components. The apparatus and procedure shall prevent the presence of any air bubbles in the system. To ensure this, where possible the test component (fuel line, etc.) is “flooded” with liquid before commencing the actual internal rinsing step. To enable accurate analysis results, a specified constant flow rate is usually necessary.

9.4.2.4 Documentation

The following information shall be included:

- general description of the analysis device and sample feed system;
- date and type of the last calibration (polystyrene or ISO-MTD);
- sampling technique: sample container or direct connection to rinsing line;
- explicit remark to make it clear that the quoted particle sizes refer to equivalent sizes and not dimensions like e.g. $\text{Feret}_{\text{max}}$;
- when listing absolute quantities (not concentrations), the volume of liquid actually analysed shall be stated;
- when giving information about concentrations, the original volume of liquid used in the extraction step shall be stated;
- volume analysed expressed in percent in cases where the sensor effectively only detects/analyses a fraction of the through-flow liquid;
- dilution factor for highly-concentrated samples;
- total volume of liquid analysed;
- coincidence concentration.

9.4.3 Filter-blocking (optical)

9.4.3.1 Principle

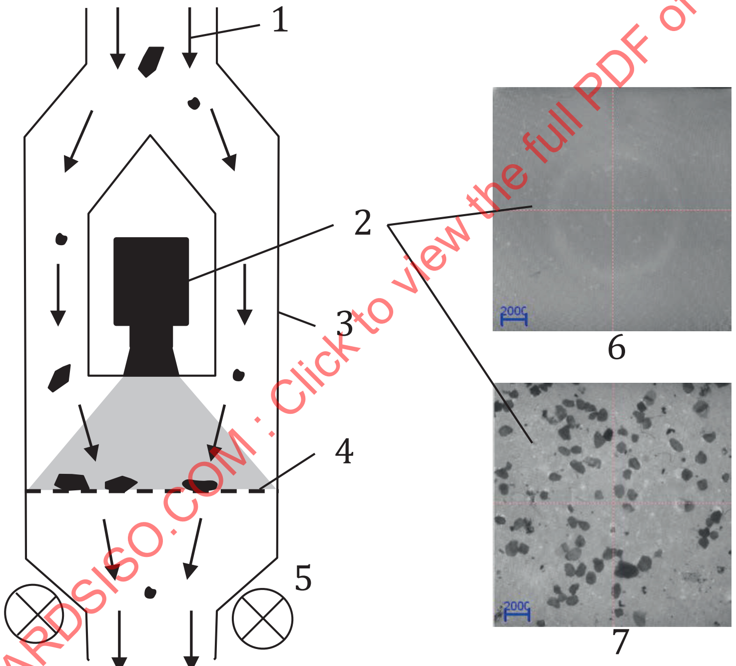
With this analysis method, particles from the flow of liquid (extraction liquid containing particles) are deposited directly on an analysis filter that is situated in the field of view of a camera (see Figure 23).

The pore size of the analysis filter is selected to enable only relevant particle sizes to be retained and smaller particles to pass through the filter. The measuring cell, which is located for example at the outlet of an extraction setup where the filter is normally clamped, is fitted with a (transmitted light) illumination source. This provides a high degree of contrast between the particles deposited on the filter and the filter background and assures their detection by the camera.

Further image processing and analysis steps are carried out conform to the light-optical analysis method (binarization and particle measurement). As opposed to automated microscope systems, instead of several image fields being combined to form an overall image, in this case only one image field is analysed. As a result, this system does not offer the optical resolution of microscope systems but is quite suitable for detecting largish single particles.

In each analysis, two images are evaluated — one before the extraction step and one after deposition of the contaminant particles on the filter. The difference between the two corresponds with the particle load extracted from the component.

If particle occupancy on the analysis filter is too high and analysis errors occur due to particles touching or overlapping one another, the analysis filter is backwashed to remove (the majority of) the particle load. Backwashing is performed by reversing the flow of liquid with the aid of suitable pumps and valves or by putting the analysis filter in the other way round.



Key

1	direction of flow of liquid from extraction apparatus	5	illumination
2	camera	6	start of the measurement
3	measuring cell	7	end of the measurement
4	analysis filter		

Figure 23 — Cell for the optical analysis of the filter blockade

9.4.3.2 Material and equipment

a) Measuring cell with:

- — camera,
- — analysis filter,

- (transmitted light) illumination,
- system for backwashing the analysis filter, and
- image processing and analysis.

b) Adapter to extraction setup and return feed of test liquid.

9.4.3.3 Procedure

The following procedure shall be adapted to the features of the respective extraction device and analysis system.

a) Record and analyse an image of the analysis filter to assess its initial state.

b) Perform the extraction procedure, including the final rinsing step. The complete extraction liquid and final rinsing liquid is then guided through the measuring cell and analysis filter.

c) Record and analyse an image of the analysis filter after the extraction step.

d) The difference between the two analyses equates to the particle load extracted from the test component.

e) Carry out the next extraction or backwash the analysis filter if particle occupancy is too high.

9.4.3.4 Documentation

Particles are analysed and documented in accordance with the procedure for light-optical analysis and grouped into the particle size classes stated in Clause 10.

10 Documentation

10.1 Overview

This clause specifies the content of the inspection documents, not the format.

Different documents are generated in the course of a cleanliness inspection (see Figure 24). Depending on the type of cleanliness inspection performed, either a qualification report or an inspection report is written.

The qualification report documents the test conditions, extraction parameters and the results of the qualification tests (declining tests), with the latter being used to determine the routine extraction procedure and thus the related inspection specification.

The inspection report briefly lists the extraction parameters and test conditions as well as the results of routine inspections. In the inspection report, only information related to the inspection performed is required.

The inspection specification contains details of the extraction and analysis parameters together with a clear and easily understandable description of the inspection procedure. The description can be written in a summarized or full version with photos or illustrations being added if desired. The inspection specification may include proof of the qualification or refer to a separate document, the qualification report (for example see Annex G). Further references (e.g. supplementary agreements) are optional.

The cleanliness inspection document(s) shall contain all the information mentioned in 10.2 to 10.8 that is necessary to reproduce the cleanliness inspection and assess the results. Further information shall be added as required.

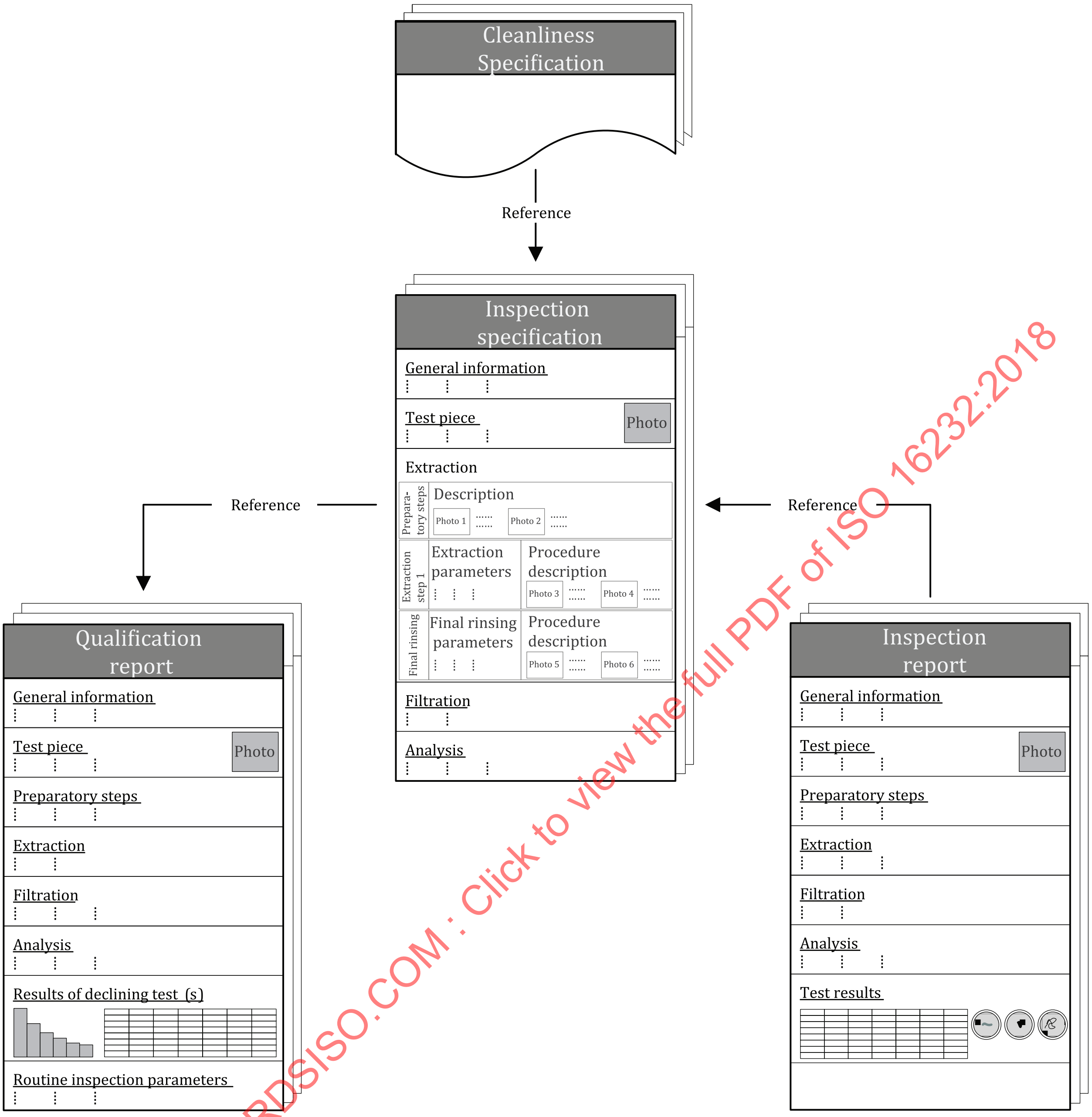


Figure 24 — Cleanliness inspection documents (overview and interrelations)

10.2 General information

“General information” (see Figure 25) covers higher level information to aid the classification and administration of the documents supplied.

General information

Order No.: ………………………………… Report No.: ……………………………………………
Customer: ………………………………… Date of analysis: ……………………………………………
Contact partner: ……………………………………………

Test ordered: ☐ Routine inspection ☐ Double inspection ☐ Qualification test
Reason for test: ☐ Initial assessment ☐ Process control ☐ Modifications, constructional ☐ ………………..

Figure 25 — General Information

10.3 Information about the test component

The test component is described in more detail and identified clearly (see Figure 26). Additional information which may be useful could include:

- the material the test component is made from, and
- the presence of filmy residues on the test component.

Test component

Description: ....
Parts Number: ....
Batch Number: ....
Date removed: ....
Time removed: ....
Removed from: ....

Condition on delivery: ....
Test lot size: ....
Controlled surface : ....
A_C = ... cm^2
V_C = ... cm^3
Inspection specification: ....

Photo
on delivery

Photo
test piece

Figure 26 — Information about the test component

10.4 Information about preparatory steps

Information about preparatory steps indicates if and when supplementary work steps are required (see Figure 27). If no information about preparatory steps is mentioned, inspection results could be wrongly evaluated or misinterpreted.

Preparatory steps

☐ None
☐ Disassembly
☐ Demagnetization

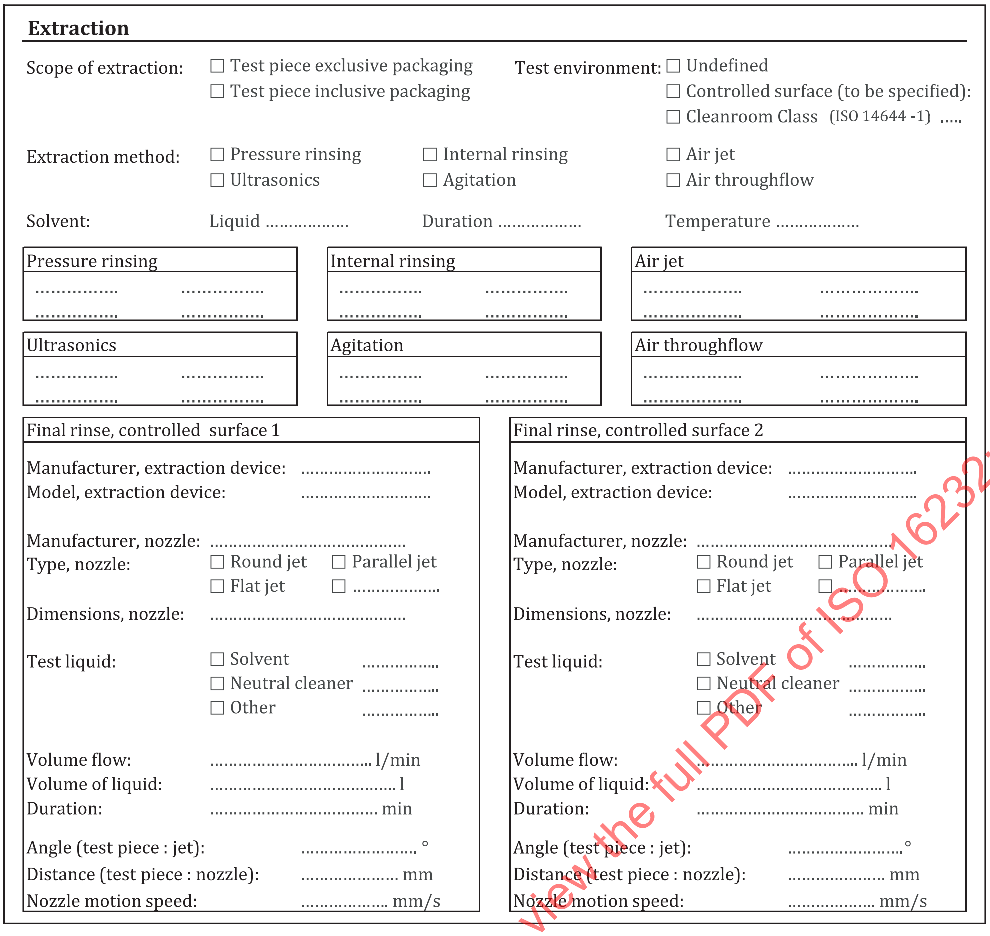
☐ Isolate controlled surfaces (e.g. sealing, masking)
☐ Preclean contaminated surfaces not relevant to controlled surface
☐ ....

Figure 27 — Information about preparatory steps

10.5 Information about the extraction

10.5.1 General

The way the extraction step is performed may vary significantly according to the expertise of inspection staff involved. The degree of freedom regarding the extraction step is reduced by documenting the inspection procedure as clearly as possible (see Figure 28). This aims to make the extraction step reproducible and improve the comparability of inspection results. The number of possible answers should be restricted by the use of checkboxes or dropdown lists. Because the angle or feed rate of an open jet may fluctuate markedly during the extraction step, this information is optional.



Extraction

Scope of extraction: ☐ Test piece exclusive packaging ☐ Test piece inclusive packaging

Test environment: ☐ Undefined ☐ Controlled surface (to be specified): ☐ Cleanroom Class (ISO 14644 -1) .....

Extraction method: ☐ Pressure rinsing ☐ Ultrasonics ☐ Internal rinsing ☐ Agitation ☐ Air jet ☐ Air throughflow

Solvent: Liquid .... Duration .... Temperature ....

Pressure rinsing		Internal rinsing		Air jet	
....	....	....	....	....	....
....	....	....	....	....	....

Ultrasonics		Agitation		Air throughflow	
....	....	....	....	....	....
....	....	....	....	....	....

Final rinse, controlled surface 1	Final rinse, controlled surface 2
Manufacturer, extraction device:	Manufacturer, extraction device: ....
Model, extraction device:	Model, extraction device: ....
Manufacturer, nozzle:	Manufacturer, nozzle: ....
Type, nozzle: ☐ Round jet ☐ Parallel jet ☐ Flat jet ☐	Type, nozzle: ☐ Round jet ☐ Parallel jet ☐ Flat jet ☐ ....
Dimensions, nozzle:	Dimensions, nozzle: ....
Test liquid: ☐ Solvent .... ☐ Neutral cleaner .... ☐ Other	Test liquid: ☐ Solvent .... ☐ Neutral cleaner .... ☐ Other ....
Volume flow: .... l/min	Volume flow: .... l/min
Volume of liquid: l	Volume of liquid: l
Duration: .... min	Duration: .... min
Angle (test piece : jet): .... °	Angle (test piece : jet): .... °
Distance (test piece : nozzle): .... mm	Distance (test piece : nozzle): .... mm
Nozzle motion speed: .... mm/s	Nozzle motion speed: .... mm/s

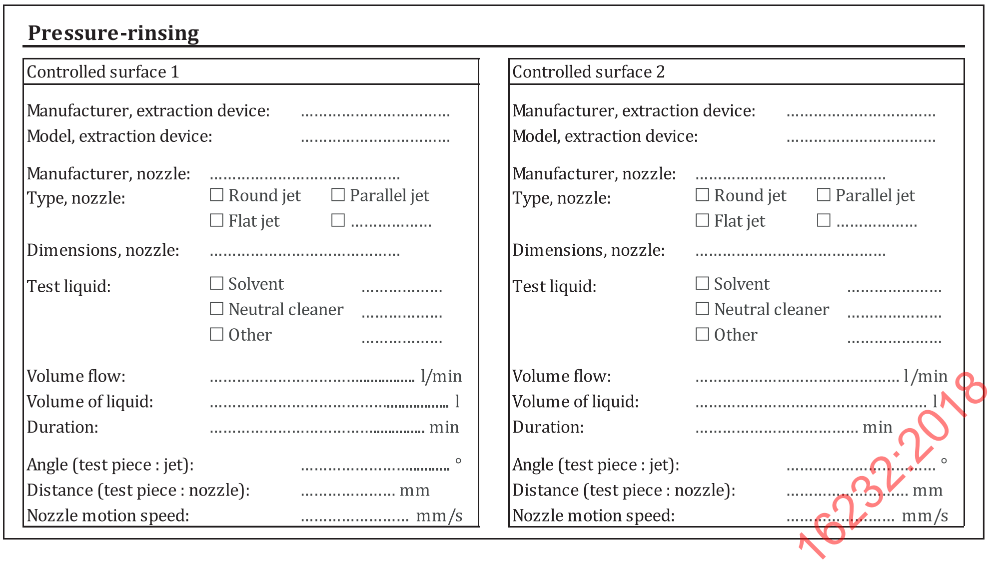
Figure 28 — Information about the extraction

10.5.2 Pressure-rinsing

Pressure-rinsing has the highest number of influencing parameters. Among other things, the cleaning effect of pressure-rinsing is influenced by the nozzle selected. Depending on the nozzle type, the following information should be noted in the section *Dimensions, nozzle*:

- *nozzle cross-section in mm* for a round jet (full jet) nozzle;
- *pressure-rinsing angle in* ° and *equivalent nozzle cross-section in mm* for a flat jet (fan-jet) nozzle;
- *nozzle cross-section in mm* and *number of bores* for a parallel jet nozzle.

Additional information can be documented as shown in Figure 29.



Pressure-rinsing

Controlled surface 1

Manufacturer, extraction device: ....
Model, extraction device: ....

Manufacturer, nozzle: ....
Type, nozzle: ☐ Round jet ☐ Parallel jet
☐ Flat jet ☐ ....

Dimensions, nozzle: ....

Test liquid: ☐ Solvent ....
☐ Neutral cleaner ....
☐ Other ....

Volume flow: .... l/min
Volume of liquid: l
Duration: .... min

Angle (test piece : jet): .... °
Distance (test piece : nozzle): .... mm
Nozzle motion speed: .... mm/s

Controlled surface 2

Manufacturer, extraction device: ....
Model, extraction device: ....

Manufacturer, nozzle: ....
Type, nozzle: ☐ Round jet ☐ Parallel jet
☐ Flat jet ☐ ....

Dimensions, nozzle: ....

Test liquid: ☐ Solvent ....
☐ Neutral cleaner ....
☐ Other ....

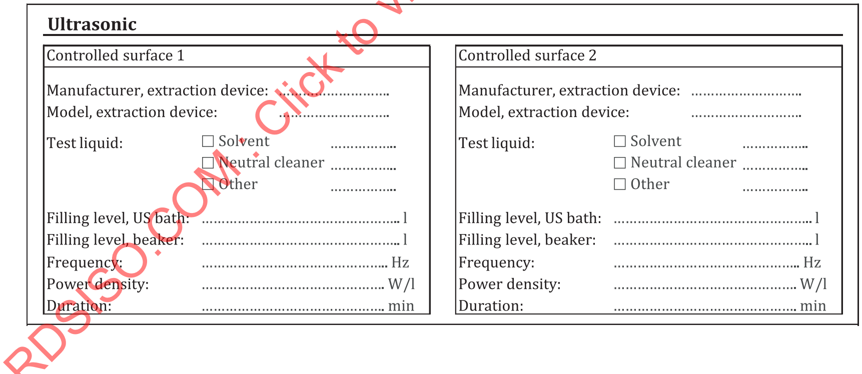
Volume flow: .... l/min
Volume of liquid: l
Duration: .... min

Angle (test piece : jet): .... °
Distance (test piece : nozzle): .... mm
Nozzle motion speed: .... mm/s

Figure 29 — Information about pressure-rinsing

10.5.3 Ultrasonic vibration

With ultrasonic vibration, more influencing parameters are known than are documented. As it is not easy to determine fluctuating sound pressures, pressure peaks and other variables, only details of the extraction parameters shown are documented (see Figure 30). It is not sufficient to note the ultrasonic output because this depends on the filling level of the ultrasonic device. Instead, information about the power density (output per filling volume) should be given.



Ultrasonic

Controlled surface 1

Manufacturer, extraction device: ....
Model, extraction device: ....

Test liquid: ☐ Solvent ....
☐ Neutral cleaner ....
☐ Other ....

Filling level, US bath: l
Filling level, beaker: l
Frequency: .... Hz
Power density: .... W/l
Duration: .... min

Controlled surface 2

Manufacturer, extraction device: ....
Model, extraction device: ....

Test liquid: ☐ Solvent ....
☐ Neutral cleaner ....
☐ Other ....

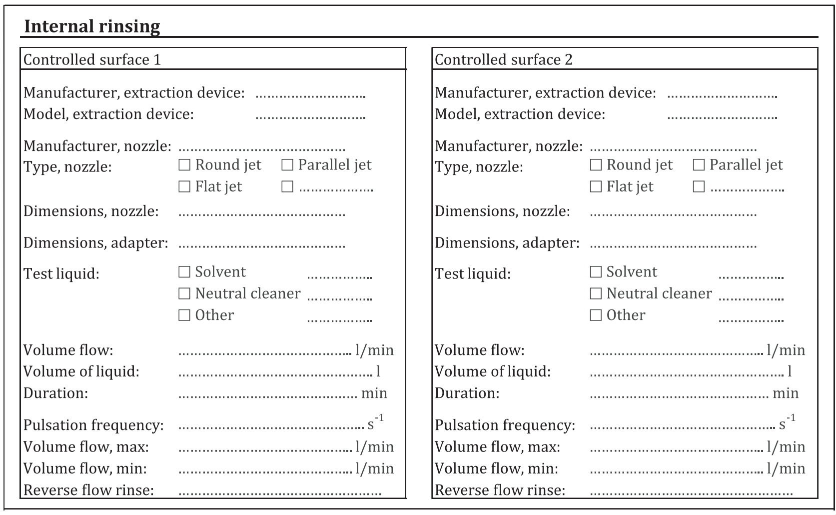
Filling level, US bath: l
Filling level, beaker: l
Frequency: .... Hz
Power density: .... W/l
Duration: .... min

Figure 30 — Information about ultrasonic vibration

10.5.4 Internal rinsing

Internal rinsing may take different forms (see Figure 31). They range from simple internal rinsing, with the (pressure-rinsing) nozzle being applied hermetically to an opening, right up to complex internal rinsing systems, where parameters such as direction of flow, pulsation, etc. are all adjustable.

If the test liquid flows through the controlled surface in one direction with a periodically-changing flow rate, this is known as pulsation. The pulsation frequency is dependent on time and shall be stated.



Internal rinsing

Controlled surface 1

Manufacturer, extraction device: ....
Model, extraction device: ....

Manufacturer, nozzle: ....
Type, nozzle: ☐ Round jet ☐ Parallel jet
☐ Flat jet ☐ ....

Dimensions, nozzle: ....

Dimensions, adapter: ....

Test liquid: ☐ Solvent ....
☐ Neutral cleaner ....
☐ Other ....

Volume flow: .... l/min
Volume of liquid: l
Duration: .... min

Pulsation frequency: .... s^{-1}
Volume flow, max: .... l/min
Volume flow, min: .... l/min
Reverse flow rinse: ....

Controlled surface 2

Manufacturer, extraction device: ....
Model, extraction device: ....

Manufacturer, nozzle: ....
Type, nozzle: ☐ Round jet ☐ Parallel jet
☐ Flat jet ☐ ....

Dimensions, nozzle: ....

Dimensions, adapter: ....

Test liquid: ☐ Solvent ....
☐ Neutral cleaner ....
☐ Other ....

Volume flow: .... l/min
Volume of liquid: l
Duration: .... min

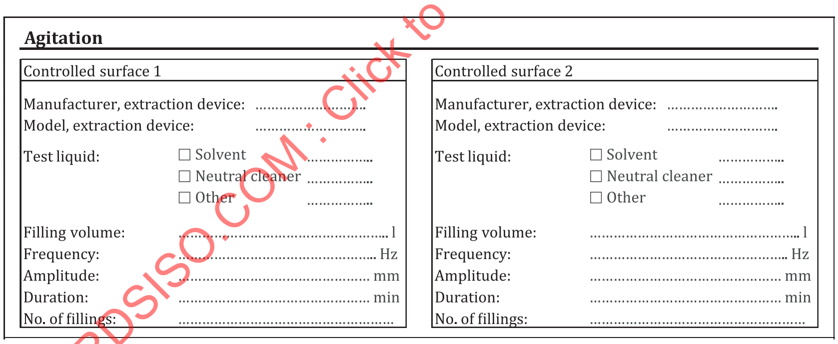
Pulsation frequency: .... s^{-1}
Volume flow, max: .... l/min
Volume flow, min: .... l/min
Reverse flow rinse: ....

Figure 31 — Information about internal rinsing

10.5.5 Agitation

Agitation is essentially a manual extraction method. The frequency and amplitude of the arm movements can neither be fixed nor measured but shall be approximated as best as possible in the documentation (see Figure 32).

The term frequency describes the up-and-down and side-to-side movement of the test component. The amplitude describes the distance of travel of the component during this movement, and the number of fillings indicates how often the test component was (re-)filled with clean liquid during the extraction step.



Agitation

Controlled surface 1

Manufacturer, extraction device: ....
Model, extraction device: ....

Test liquid: ☐ Solvent ....
☐ Neutral cleaner ....
☐ Other ....

Filling volume: l
Frequency: .... Hz
Amplitude: .... mm
Duration: .... min
No. of fillings: ....

Controlled surface 2

Manufacturer, extraction device: ....
Model, extraction device: ....

Test liquid: ☐ Solvent ....
☐ Neutral cleaner ....
☐ Other ....

Filling volume: l
Frequency: .... Hz
Amplitude: .... mm
Duration: .... min
No. of fillings: ....

Figure 32 — Information about agitation

10.5.6 Air jet extraction

Air jet extraction is the same as pressure-rinsing except for the fact that air is used instead of a test liquid (see Figure 33).

Air jet extraction

Controlled surface 1

Manufacturer, extraction device: ....
Model, extraction device: ....

Manufacturer, nozzle: ....
Type, nozzle: ☐ Round jet ☐ Parallel jet
☐ Flat jet ☐ ....

Dimensions, nozzle: ....

Pressure: .... bar
Duration: .... min

Angle (test piece : jet): .... °
Distance (test piece : nozzle): .... mm
Nozzle motion speed: .... mm/s

Controlled surface 2

Manufacturer, extraction device: ....
Model, extraction device: ....

Manufacturer, nozzle: ....
Type, nozzle: ☐ Round jet ☐ Parallel jet
☐ Flat jet ☐ ....

Dimensions, nozzle: ....

Pressure: .... bar
Duration: .... min

Angle (test piece : jet): .... °
Distance (test piece : nozzle): .... mm
Nozzle motion speed: .... mm/s

Figure 33 — Information about air jet extraction

10.5.7 Air through-flow extraction

Air through-flow extraction is the same as rinsing except for the fact that air is used instead of a test liquid (see Figure 34).

Air throughflow extraction

Controlled surface 1

Manufacturer, extraction device: ....
Model, extraction device: ....

Manufacturer, nozzle: ....
Type, nozzle: ☐ Round jet ☐ Parallel jet
☐ Flat jet ☐ ....

Dimensions, nozzle: ....

Dimensions, adapter: ....

Pressure: .... bar
Duration: .... min

Pulsation frequency: .... s^{-1}
Volume flow, max: .... l/min
Volume flow, min: .... l/min
Reverse flow rinse: ....

Controlled surface 2

Manufacturer, extraction device: ....
Model, extraction device: ....

Manufacturer, nozzle: ....
Type, nozzle: ☐ Round jet ☐ Parallel jet
☐ Flat jet ☐ ....

Dimensions, nozzle: ....

Dimensions, adapter: ....

Pressure: .... bar
Duration: .... min

Pulsation frequency: .... s^{-1}
Volume flow, max: .... l/min
Volume flow, min: .... l/min
Reverse flow rinse: ....

Figure 34 — Information about air through-flow extraction

10.6 Information about filtration

As well as documenting the actual filtration details, information about drying and any post-treatment steps should also be noted (see Figure 35).

Filtration

Filter 1
Manufacturer: ....
Type: ....
Filter material: ....
Diameter: .... mm
Pore size: .... µm

Filter 2
Manufacturer: ....
Type: ....
Filter material: ....
Diameter: .... mm
Pore size: .... µm

Filter 3
Manufacturer: ....
Type: ....
Filter material: ....
Diameter: .... mm
Pore size: .... µm

Drying

Devices: ☐ None (environment)
☐ Drying cabinet
☐ Desiccator

Temperature: .... °C
Duration: .... min

Pre-conditioning

☐ Yes ☐ No

Post-treatment

Liquid ☐ Solvent ....
☐ Neutral cleaner ....
☐ Other ....

Volume of liquid: l

Figure 35 — Information about analysis filtration

10.7 Information about the analysis

10.7.1 General

Details and settings of the analysis system are documented. If the extraction step and analysis are performed in different places or different room qualities, the test environment shall be documented in the same way as shown in 10.5.

10.7.2 Standard analysis

10.7.2.1 Gravimetric analysis

Among other things, the weighing result of a gravimetric analysis is dependent on the room temperature. If this is not documented anywhere else, the room temperature should be noted in the gravimetric analysis (see Figure 36).

Gravimetry

Manufacturer: ....
Model: ....
Date of last calibration: ....
Resolution of balance: ☐ ± 0,1 mg ☐ ± 0,01 mg ☐ ± 0,001 mg

Figure 36 — Information about balance for gravimetric analysis

10.7.2.2 Light-optical analysis

Light-optical devices can be calibrated using particle standards. The date of the last calibration shall be documented. The scale shall be stated as the magnification per pixel in [µm/pixel] (see Figure 37).

Light optical analysis

Manufacturer: ....
Model: ....
Particle standard: ....
Date of last check: ....

Pixel resolution: .... µm/Pixel
Analysis diameter: .... mm
Brightness: .... %
Threshold value: ....

Figure 37 — Information about light optical analysis

10.7.3 Extended analysis (informative)

Information about extended analyses may vary depending on the analysis system utilized. An example of information about a scanning electron microscope with EDX detector is shown in Figure 38.

Scanning Electron Microscopy / Energy Dispersive X-ray spectroscopy (SEM / EDX)

Manufacturer: ……………………………… Particle standard: ………………………………
Model: ……………………………… Date of last check: ………………………………

Acceleration voltage: ……………………………… keV
Analysis diameter: ……………………………… mm
Working distance: ……………………………… mm
Process: ☐ Automated ☐ Manual

Figure 38 — Information about SEM/EDX for extended analysis

10.7.4 Shortened analysis (informative)

The information shown in Figure 39 regarding a shortened analysis refers to an automatic particle counter.

Optical particle counter (OPC)

Manufacturer: ……………………………… Coincidence limit: ………………………………
Model: ……………………………… Date of last calibration: ………………………………
Calibration material: ………………………………

Extraction liquid: ……………………………… ml
Dilution factor: ………………………………
Measuring interval: ……………………………… min^{-1}
Nominal volume flow: ……………………………… ml/min
Detection volume: ……………………………… ml

Figure 39 — Information about optical particle counter for shortened analysis

10.8 Reporting of the inspection results

10.8.1 General

Information about cleanliness values shall be related to one of the following units of reference:

- number of components tested $n = [1]$;
- wetted surface area of test component $A_C = [\text{cm}^2]$;
- wetted volume of test component $V_C = [\text{cm}^3]$.

10.8.2 Gravimetric analysis

The result of a gravimetric analysis is the residue mass $m = m_2 - m_1$, with the last digit being mathematically rounded. The masses m_2 and m_1 are read off using the resolving capacity of the weighing balance. The resulting particle mass m, which is calculated from the difference between the residue mass and the unit of reference, shall be documented.

Mass related to number of components tested

$$m_C = \frac{m}{n} = \left[\frac{\text{mg}}{1}\right]$$

The unit used is mg per (n) component(s).

Mass related to controlled surface

$$m_A = \frac{m \times 1\,000}{A_C} = \left[\frac{\text{mg}}{1\,000\ \text{cm}^2}\right]$$

Mass related to controlled volume

$$m_V = \frac{m \times 100}{V_C} = \left[\frac{\text{mg}}{100\ \text{cm}^3}\right]$$

10.8.3 Light-optical analysis

Light-optical systems determine the length and/or width of individual particles. The particles are also classified into a specified length or width interval (or size class).

Length/width intervals other than the size classes listed in Table 8 should only be documented if absolutely necessary, e.g. 100 µm ≤ x < 200 µm, 200 µm ≤ x < 300 µm, etc. (added effort).

The size classes now include letters L, M and N (see Table 8)

Table 8 — (Particle) size classes

Size class	Size x [µm]
B	$5 \le x < 15$
C	$15 \le x < 25$
D	$25 \le x < 50$
E	$50 \le x < 100$
F	$100 \le x < 150$
G	$150 \le x < 200$
H	$200 \le x < 400$
I	$400 \le x < 600$
J	$600 \le x < 1\,000$
K	$1\,000 \le x < 1\,500$
L	$1\,500 \le x < 2\,000$
M	$2\,000 \le x < 3\,000$
N	$3\,000 \le x$

The particle size distribution shall include information about the unit of reference. The normed particle count H is obtained from the ratio between the absolute particle count h and the unit of reference.

Particle count related to the number of components tested

$$H_C = \frac{h}{n} = [1]$$

The unit used is particles per (n) component(s).

EXAMPLE A test lot of 4 components is inspected (n = 4) and 13 particles in Size Class G (150 ≤ x < 200 µm) are detected (h = 13). This gives the normed particle count H_c = 13/4 = 3,25. Rounding to at least one position after the decimal point finally results in H_c = 3,3.

Particle count related to controlled surface

$$H_{\mathrm{A}} = \frac{h \times 1\,000}{A_{\mathrm{C}}} = \left[\frac{1}{1\,000\ \mathrm{cm}^2} \right]$$

The unit used is particles per 1 000 cm^2.

If, for example, 89 particles are counted in Size Class H (200 µm ≤ x < 400 µm) for a component with a controlled surface of A_{C} = 263 cm^2, on implementing the equation this gives the normed particle count H_{A} = 338,4 rounded to at least one position after the decimal point.

Particle count related to wetted volume

$$H_{\mathrm{V}} = \frac{h \times 100}{V_{\mathrm{C}}} = \left[\frac{1}{100\ \mathrm{cm}^3} \right]$$

If, for example, 2 particles in Size Class K (1 000 µm ≤ x < 1 500 µm) are counted for a component with a wetted volume of V_{C} = 320 cm^3, on implementing the equation, this gives the normed particle count H_{A} = 0,6 rounded to at least one position after the decimal point.

For each unit of reference, particle counts are entered directly into the appropriate boxes in the report (see Figure 40). When norming, the resulting comma positions can often be mathematically rounded to one digit after the decimal comma. The specification of one position after the decimal comma takes outliers into account which occur when limit value requirements are made for higher size classes and smaller absolute particle quantities.

If no unit of reference is stated, the information or blank level calculation always refers to the test lot.

Information about categories of particles (i.e. fibres, "metallic shiny", etc.) is also added. The physical effects of reflected light are used to characterize particles more precisely. This enables a differentiation to be made between the category "metallic shiny" and "not (metallic) shiny" particles, and can be documented. Categories of particles to be counted should be agreed between parties. However, light-optical systems do not allow particles to be categorized as soft or hard, electrically conductive or non-conductive, or magnetic or non-magnetic.

NOTE Metal particles can only be determined with certainty by applying extended analysis methods.

Inspection results

Particle size distribution according to categories

	Length, Feret$_{max}$ [µm]	Particle count per 1 000 cm^2												
		B	C	D	E	F	G	H	I	J	K	L	M	N
		5 ≤ x < 15	15 ≤ x < 25	25 ≤ x < 50	50 ≤ x < 100	100 ≤ x < 150	150 ≤ x < 200	200 ≤ x < 400	400 ≤ x < 600	600 ≤ x < 1 000	1 000 ≤ x < 1 500	1 500 ≤ x < 2 000	2 000 ≤ x < 3 000	3 000 ≤ x
Category	All particles excluding fibres													
	Metallic shiny particles													
	Fibres													

	Length, Feret$_{min}$ [µm]	Particle count per 1 000 cm^2												
		B	C	D	E	F	G	H	I	J	K	L	M	N
		5 ≤ x < 15	15 ≤ x < 25	25 ≤ x < 50	50 ≤ x < 100	100 ≤ x < 150	150 ≤ x < 200	200 ≤ x < 400	400 ≤ x < 600	600 ≤ x < 1 000	1 000 ≤ x < 1 500	1 500 ≤ x < 2 000	2 000 ≤ x < 3 000	3 000 ≤ x
Category	All particles excluding fibres													
	Metallic shiny particles													
	Fibres													

Image series

Largest particle

Second largest particle

Largest metallic shiny particle

Second largest metallic shiny particle

Largest fibre

Second largest fibre

Overview picture incl. filter occupancy [%]

Figure 40 — Documentation of inspection results regarding particle size distribution

Documentation of the largest particle respectively particle images shall be included. The report shall include an overview picture of the entire analysis filter and should include the value of filter occupancy.

Images of the longest or widest particle in all specified categories shall be recorded with the image-processing software of the light-optical system in order to be able to better assess the damaging potential of particles. There is no fixed minimum number of images to be recorded in each category.

Documentation of particle size distributions determined by shortened analysis with optical particle counter is shown in Figure 42.

10.8.4 Extended analysis

The way inspection results are presented depends on the method selected. In the case of as SEM/EDX analysis, for example, information about a material class can also be documented (see Figure 41).

Inspection results

Particle size distribution according to material classes

Length, Feret$_{Max}$ [µm]		Particle count per 1 000 cm^2												
		B	C	D	E	F	G	H	I	J	K	L	M	N
		5 $\le x <$ 15	15 $\le x <$ 25	25 $\le x <$ 50	50 $\le x <$ 100	100 $\le x <$ 150	150 $\le x <$ 200	200 $\le x <$ 400	400 $\le x <$ 600	600 $\le x <$ 1 000	1 000 $\le x <$ 1 500	1 500 $\le x <$ 2 000	2 000 $\le x <$ 3 000	3 000 $\le x$
Material class	Steel, high-alloy													
	Steel, non-alloyed													
	Brass													
	Copper													
	Aluminium													
	Lead													
	Mineral													
	Glass mineral fibre													
	⋮													

Image series

Largest particle steel

Second largest particle steel

Largest particle copper

Second largest particle copper

Largest mineral glass fibre

Second largest mineral glass fibre

Figure 41 — Documentation of inspection results regarding material classes

10.8.5 Shortened analysis

Inspection results

Particle size distribution

Equivalent length [µm]	Particle count per 1 000 cm^2											
	B	C	D	E	F	G	H	I	J	K	L	M	N
	5 $\le x <$ 15	15 $\le x <$ 25	25 $\le x <$ 50	50 $\le x <$ 100	100 $\le x <$ 150	150 $\le x <$ 200	200 $\le x <$ 400	400 $\le x <$ 600	600 $\le x <$ 1 000	1 000 $\le x <$ 1 500	1 500 $\le x <$ 2 000	2 000 $\le x <$ 3 000	3 000 $\le x$

Figure 42 — Documentation of particle size distribution in case of shortened analysis

10.8.6 Optional coding (informative)

This clause deals with the optional coding of particle size distributions with the intention of abbreviating cleanliness values of automotive parts. Coding can only be used for particle size distributions. If not otherwise indicated the code refers to the particle length Feret$_{max}$ and the category "all particles excluding fibres".

NOTE Different coding systems are used for cleanliness measurement in different standards (e.g. hydraulic industry).

Component Cleanliness Code

Coding is used to assign the particle count to a cleanliness level, see Table 9.

The component cleanliness code is noted as follows:

$$\text{CCC} = \underbrace{A}_{\substack{\text{Unit of}\\ \text{reference}}} \ (\underbrace{\text{B}}_{\substack{\text{Size}\\ \text{class}}}\ \underbrace{20}_{\substack{\text{Contamination}\\ \text{level}}}\ \underbrace{/}_{\text{Separator}}\ \text{C16/D18/...})$$

The unit of reference states whether the particle count is related to:

- 1 000 cm^2 controlled surface of the test component (marked *A*),
- 100 cm^3 controlled volume of the test component (marked *V*) or,
- 1 component (marked *N*).

See Table 8 and Table 9 for cleanliness levels.

Table 9 — Particle concentration classes

Cleanliness level	Particle count (per 1 000 cm^2 or 100 cm^3) up to including
00	0
0	1
1	2
2	4
3	8
4	16
5	32
6	64
7	130
8	250
9	500
10	1×10^3
11	2×10^3
12	4×10^3
13	8×10^3
14	16×10^3
15	32×10^3
16	64×10^3
17	130×10^3
18	250×10^3
19	500×10^3
20	1×10^6
21	2×10^6
22	4×10^6
23	8×10^6
24	16×10^6

Simplified ways of writing component cleanliness codes

The following particle size distribution is documented (see Table 10):

Table 10 — Example of documentation of a particle size distribution

Size class												
B	C	D	E	F	G	H	I	J	K	L	M	N
Particle count per 1 000 cm^2 (x = particle size in µm)												
5 ≤ x < 15	15 ≤ x < 25	25 ≤ x < 50	50 ≤ x < 100	100 ≤ x < 150	150 ≤ x < 200	200 ≤ x < 400	400 ≤ x < 600	600 ≤ x < 1 000	1 000 ≤ x < 1 500	1 500 ≤ x < 2 000	2 000 ≤ x < 3 000	3 000 ≤ x
634 981	49 735	3 679	938	502	394	48	7	5	2	1	1	0
Cleanliness level												
20	16	12	10	10	9	6	3	3	1	0	0	00

As particle counts are expressed in a normed way, they can be read of as a cleanliness level directly from Table 9 resulting in the following component cleanliness code:

CCC = A(B20/C16/D12/E10/F10/G9/H6/I3/J3/K1/L0/M0/N00)

If individual size classes do not need to be stated, e.g. if no specification or analysis result exists for these size classes, they are omitted in the CCC:

CCC = A(D12/E10/F10/G9/H6/I3/J2/K1/L0/M0)

If the cleanliness levels of neighbouring size classes are identical, this can be abbreviated to:

CCC = A(B20/C16/D12/EF10/G9/H6/I3/J2/K1/LM0/N/00)

In this case the maximum count for size class E is 1×10^3. The maximum count for size class F is also limited to 1×10^3. The maximum count for size class L is limited to 1 as well as the maximum counts in size class M are restricted to 1.

The situation changes if several different size classes are combined to form a larger size interval (for example, see Table 11).

Table 11 — Example of documentation of a particle size distribution

Size class							
B – D	E	F	G	H	I	J	K – N
Particle count per 1 000 cm^2 (x = particle size in µm)							
5 ≤ x < 50	50 ≤ x < 100	100 ≤ x < 150	150 ≤ x < 200	200 ≤ x < 400	400 ≤ x < 600	600 ≤ x < 1 000	1 000 ≤ x
721 507	14 688	873	361	56	12	4	1
Cleanliness level							
20	14	10	9	6	4	2	0

The corresponding simplified code is then expressed as follows:

CCC = A(B – D20/E14/F10/G9/H6/I4/J2/K – N0)

Exceptional case, unit of reference N

The CCC per *component* may only be stated if it refers to exactly one component. Cleanliness levels may not be used for this unit of reference; therefore, the particle count is adopted from the particle size distribution in a non-encoded form. Particle counts with a position after the decimal point shall be rounded.

CCC = *N*(B755840/C43720/D36791/E938/F502/G394/H48/I7/J5/K2/L1/M1/N0)

10.9 Description of content of the various documents

10.9.1 Inspection specification

The inspection specification is the main document of the cleanliness inspection. It is written after the qualification tests have been completed and once inspection parameters have been derived for the routine inspection. The inspection specification comprises:

a) general information,

b) information about the test component,

c) information about preparatory steps (detailed),

d) information about extraction (detailed),

e) information about filtration, and

f) information about analysis.

The inspection specification may contain additional information about the qualification tests or refer to an independent document (the qualification report).

The inspection specification contains a clearly understandable description of the test procedure. Illustrations or photos are a helpful supplement to the description. In general, as the test procedure becomes more complex, the more detailed the description should be. With simple test procedures, a point-by-point description is sufficient.

The inspection specification shall enable the cleanliness inspection to be performed independently of the time, place and inspecting member of staff. An example of an inspection specification is shown in Annex G (see Figure G.1, Figure G.2 and Figure G.3).

No inspection results are documented in the inspection specification except for the results of the qualification tests if no qualification report is available. Cleanliness requirements or limit values may be included in an inspection specification if no separate cleanliness specification document or drawing entry exists.

10.9.2 Qualification report

The first document prepared is the qualification report. Without the qualification of test conditions and inspection parameters, there can be no inspection specification and thus no inspection report. The qualification report comprises:

a) general information,

b) information about the test component,

c) information about preparatory steps,

d) information about extraction,

e) information about filtration,

f) information about analysis,

g) results of the declining test,

h) verification of the blank level, and

i) information on routine inspection conditions and inspection parameters.

The qualification report contains a graph of the declining test and information about routine inspection conditions and parameters. As an option, the results of a subsequent double inspection can also be included.

The routine inspection conditions and parameters derived from the declining test, which are contained in the qualification report, are also included in the inspection specification and inspection report. The origin of the routine inspection conditions and parameters remains transparent because of the mandatory reference to the inspection specification. The reference is not necessary if all the required information about the qualification tests is included in the inspection specification (see list above).

10.9.3 Inspection report

The inspection report is the last document written. A routine inspection can only be performed once qualified test conditions and parameters have been determined. All settings and results are documented briefly and clearly in the inspection report. The inspection report comprises:

a) general information,

b) information about the test component,

c) information about preparatory steps,

d) information about extraction,

e) information about filtration,

f) information about analysis, and

g) results of the routine inspection.

The most important part of the inspection report is the results. With recurrent routine inspections, these are the only things that change and all other information remains identical.

To ensure that the procedure used for determination of the test conditions and parameters is clear, the inspection report shall contain a reference to the inspection specification. This may be of particular interest if the declining test was not successful — i.e. in the event of an exceptional case — and in cases where no qualified inspection parameters exist.

Provided that they have been archived or saved, it is not necessary to include the inspection specification and qualification report when handing over the inspection report.

11 Handling components cleanly

11.1 Principles

Technical cleanliness is not a constant feature of a component but rather a temporary condition that is influenced by multiple external factors. It should therefore be stipulated in the customer-supplier relationship when and where a cleanliness specification shall be performed (for example, after cleaning, on arrival at the assembly line or at another point in the process chain).

Therefore, the cleanliness state of a test component may not be altered between its removal from the designated point in the production chain and its arrival at the laboratory for a cleanliness inspection.

In order for test components to be received in a representative state, procedures and conditions shall be rigidly upheld from the point of removal up to the point of arrival at the testing facility. This also applies to deviations from serial conditions when transporting a test component to the test location (such as the use of different packaging or a different transport pathway). As it is essential that test components are handled in a clean manner and the reason, workers contributing to cleanliness inspections shall be appropriately trained and instructed.

The following factors may influence the state of cleanliness of the test component on its removal from the process chain:

- handling;
- packaging;
- transport;
- environmental conditions;
- storage;
- preparatory steps before the test (e.g. inserting plugs, disassembly or affixing mounts).

In cases where tested components are returned to the production line or re-used, appropriate cleanliness regulations may be required, e.g. treatment with an anti-corrosive or the use of an unused plastic bag.

11.2 Selected measures and recommendations

11.2.1 Staff

Responsibilities regarding the complete inspection process, from removal of the test component until its return, shall be defined.

Members of staff involved in the inspection shall be instructed how to handle test components cleanly.

11.2.2 Packaging

Packaging shall be designed so that the test component is appropriately protected in accordance with cleanliness requirements, e.g. regarding:

- resistance to abrasion, particle emission from the component, leakproofness and electrostatics;
- type and duration of transport, handling and storage;
- environmental conditions (e.g. climatic fluctuations) between the time of manufacture and the cleanliness inspection.

Foils and bags that are in direct contact with the test object shall be clean and unused (disposable packaging). Cardboard is not suitable as a direct means of packaging for test components because it generates large quantities of particles.

Reusable packaging, which objects are placed into directly, shall be cleaned before use.

The same applies for closures. They may not cause any mechanical damage to the test component. Self-cutting closures may not be utilized.

If several components are placed together loose in a packaging unit, there is a risk of them striking/abrading against each other during transport (material abrasion) and causing particles to detach. This shall be avoided.

11.2.3 Storage and transport

The following general requirements apply:

- shortest possible pathways;

- shortest possible lengths of time;
- minimum vibration;
- components protected against damage inside the packaging;
- packaging protected against damage;
- where applicable, additional protection from wetness, humidity and fluctuations in temperature.

It is recommended that outer packaging and storage areas intended for test components are specially marked.

11.2.4 Facilities for inspecting cleanliness

The test environment shall be completely separate from areas where contamination is generated, e.g. cutting or welding processes. Compressed air may not be used in the inspection area to clean/dry objects as this could spread contamination.

Objects shall be handled and, if required, disassembled under environmental conditions conforming to the cleanliness requirements.

The methods used to clean floors, equipment and workplaces and the frequency of cleaning processes depend on the contamination levels concerned and on the cleanliness requirements of the respective components. Separate clean areas are required for highly-sensitive inspections (e.g. clean workbench).

The suitability of the test environment is not necessarily dependent on a (specific) cleanroom class. The installation of clean air technology to create a defined air cleanliness class according to ISO 14644-1 may not be required. It is only important that the blank level is reliably attained because this may also reflect possible environmental influences.

11.3 Exclusion from an inspection — invalid inspection

11.3.1 Deviations from required state on delivery

If discrepancies regarding the test component or its packaging are noticed, which may have affected its state of cleanliness, the inspection may not be performed. The following further actions are recommended:

- document the discrepancy or deviation from the means of packaging specified;
- inform all people involved in the inspection order;
- label the component and proceed with relevant instructions, e.g. return or scrap it.

Typical exclusion criteria include the absence of or damaged inner packaging, unsuitable packaging materials, humidity, corrosion, component damage, visible foreign contamination, e.g. wood chippings, etc.

If a component is inspected despite such discrepancies, this shall be documented in the inspection report.

11.3.2 Deviations and errors in the inspection procedure

Any deviations or anomaly occurring during the actual cleanliness inspection shall be documented in the inspection report.

If there is a possibility that results may have been falsified, the determination of cleanliness values is meaningless and their documentation in the report not permitted.

The test component concerned is rejected and not suitable for a renewed inspection. Instead of documenting cleanliness values in the respective report, the term “inspection error” is written, possibly with an explanation or justification of the error.

12 Designation

If an inspection of technical cleanliness has been carried out in accordance with this document, the following reference is made in such documents as product drawings, catalogues, specifications, and reports:

"standard cleanliness inspection according to ISO 16232" in cases where standard analysis methods and parameters are used and no other agreements regarding methods and parameters have been made within the scope of the customer-supplier relationship;

“*extended cleanliness inspection according to ISO 16232*” in cases where more detailed analysis methods have been defined, implemented, parameterized and documented within the scope of the customer-supplier relationship, or in the event that modified or extended standard analysis methods are implemented, which have also been agreed upon and documented;

“shortened cleanliness inspection according to ISO 16232” in cases where shortened analysis methods are implemented and whose application, parameterization and documentation are defined within the scope the customer-supplier relationship.

Annex A
(informative)

Selecting contamination extraction and analysis procedures

A.1 Test fluid

A.1.1 Liquid extraction

The majority of functionally-relevant automotive components come into contact with fluids during their manufacture or later operation. Capable of binding or detaching particles, examples of such fluids include:

- cooling lubricants,
- preservatives,
- washing fluids/cleaning agents,
- joining aids, and
- operating fluids.

For all components coming into contact with these fluids, an extraction procedure with a liquid is recommended. The primary task of the test liquid is to overcome the adhesive forces prevailing between the particles and the component. Tests are performed to determine which liquid is best suited for detaching the contaminants without damaging the component surface. Table D.1 gives a general overview. It can be seen here that the majority of process fluids used in production can be removed with the aid of two groups of test liquids: tensidic aqueous neutral cleaning liquids and non-polar solvents. In the case of the latter, particular care should be taken with regard to work safety and protection of the environment (odourless isoparaffins, which are non-combustible at room temperature, are preferable). For more information, see Annex K.

Recommendations for selecting the test liquid:

a) Check whether aqueous neutral test liquids containing tensides are suitable: they can generally be used to remove aqueous emulsions of cooling lubricants, animal and vegetable greases and oils and also amine-based anti-corrosives (ensure they do not foam).

b) If they cannot be used for any reason, check whether non-polar solvents such as those described above can be utilized. They are especially suited for removing mineral oil-based lubricants and anti-corrosives.

c) Should neither of the above be suitable, a special cleaning medium should be selected, e.g. ethanol (polar solvent) to clean brake components before their analysis.

The compatibility of test liquid and component material is a prerequisite to prevent damage of the component material and related falsification of test results (see Table D.2).

In cases where steel components are returned to the production line after a cleanliness inspection, they generally need to be treated with an anti-corrosive (applied with cleaning medium or separately after extraction).

A.1.2 Air extraction

Some components requiring a cleanliness inspection do not come into contact with liquids during their manufacture or subsequent operation (e.g. engine air intake components). Also, some materials are damaged if they come into contact with liquids, such as air filters, or paper and cardboard used in packaging materials, which may also influence cleanliness. If particles can be removed from such components without the need for a chemical solvent, an extraction procedure using air can be performed.

A.2 Temperature

Test fluids should be used at room temperature to minimize inspection costs and efforts (safety, comfort, heating times, energy costs, etc.). In exceptional cases, a different temperature may be required.

A.3 Mechanisms

A.3.1 General

Especially when removing particles, the use of mechanical forces to assist cleaning processes plays a major role. Cleaning mechanisms usually function in two ways.

- They detach firmly-adhering contaminants with the aid of strong mechanical forces, i.e. the actual cleaning task.
- They remove the detached particles that may have started to re-sediment and are now adhering only slightly (to the surface of the component or extraction equipment) with the aid of relatively weak mechanical forces, i.e. a rinsing process.

This third step to be considered when selecting the extraction method is made up of two sub-points — “Selecting cleaning mechanisms”, i.e. the extraction method itself, and “Cleaning mechanism parameters”, i.e. deriving parameters (flow rate, ultrasonic output, etc.) for the extraction.

The selecting of the cleaning mechanism is essentially determined by the size and geometry of the component as well as the location of the controlled surfaces (internal or external surfaces). For components with several functional surfaces that may have varying cleanliness specifications, different extraction methods may be used to inspect the respective areas of the same component (see Figure A.1 and Table A.1).

A.3.2 Liquid extraction

Four different extraction methods can be utilized for liquid extraction — pressure-rinsing, ultrasonic vibration, internal rinsing and agitation (for examples see Figure A.1 and Table A.1).

Pressure-rinsing: pressure-rinsing with spray nozzles is suitable for directly accessible surfaces with large areas that can be cleaned with parallel jet nozzles or fan nozzles. Internal surfaces or external areas that are difficult to access can only be cleaned to a certain extent with nozzles. For geometries that cannot be accessed directly with a spray jet, spray lances with a small diameter may be useful, e.g. inserted into blind holes.

Advantages:	Highly universal application, can be adapted to many extraction tasks.
Disadvantages:	Detached particles are distributed over a large area → costly final rinsing step required, the large wetted surface areas of the extraction equipment make it more difficult to attain low blank levels, high operator influence due to largely manual procedure.

NOTE 1 In some cases spray nozzles can also be used for internal extraction by inserting them into the ends of tubing, borings and pipe apertures with small diameters, for example. However, this only applies for the extraction method “internal rinsing”, where the spray nozzle merely functions as an adapter to connect the rinsing line to the component.

Ultrasonic vibration: ultrasonic baths, into which components are completely immersed, are used to clean objects with simple external geometries. With this extraction method, which is especially suitable for small bulk goods, components are either placed in ultrasonic-permeable baskets in baths specially constructed for cleanliness inspection with an outlet to analysis filtration, or in beakers that are placed in standard laboratory ultrasonic baths. To compensate for inhomogeneities in the ultrasonic field, components should be moved gently in the bath. Movements should have amplitude of at least half the wavelength of the ultrasonic waves used.

Where ultrasonic baths are utilized to clean the inner surfaces of components, it is important to verify that the cleaning effect in the interior is adequate. In general, this becomes more and more difficult the smaller the aperture becomes for the ultrasonic waves to enter, the deeper the internal geometry to be cleaned penetrates into the component, and the broader the dimensions are of the cavity concerned.

Advantages:	Easy to perform, good reproducibility, marginally influenced by inspection staff, cheap testing equipment for small parts, possibility to achieve low blank levels due to low wetted surface areas.
Disadvantages:	If inappropriate parameters are set, cavitation effects may cause material to detach from the test component, which is then falsely recognized in the analysis as particulate contamination, difficult to validate cleaning parameters in the ultrasonic bath (distribution of ultrasonic waves, cavitation effect).

Internal rinsing: internal rinsing is suitable for extracting particles from the inner surfaces of a wide range of components, e.g.

— active sub-assemblies (e.g. pumps, injectors), inspected in internal rinsing systems while activating the components concerned, and

— pipe-shaped passive components that are much longer than the size of the inlet/outlet and whose internal cross-section is similar to the diameter of the inlet and outlet.

With internal rinsing, the complete inner geometry of the component to be cleaned is always wetted and the test liquid is fed into the component via adapters. After flowing through the interior of the component, via an adapter and tubing or piping, the liquid is then advanced to the filtration unit, or drains off the component directly into an extraction bath (liquids may not come into contact with the exterior of components). In order for the internal rinsing step to be effective, a turbulent flow through the interior of the component is required.

Advantages:	Easy to limit the method to the controlled surfaces concerned by adapting rinsing lines, low blank levels attained due to low wetted surface areas, marginally influenced by inspection staff, for many fluid-conducting components this is the best extraction method for simulating real vehicle conditions.
Disadvantages:	Test set-ups/internal rinsing systems may be highly complex where high flow rates (components with large internal cross-sections) or active components (e.g. switched, powered or driven) are concerned. By adapting rinsing lines to the test components, abrasion from joining processes (screwing, pushing, pressing, etc.) may occur, which could be falsely recognized as contamination originating from the component.

NOTE 2 The term “internal rinsing” always refers to the rinsing of inner surfaces; consequently, there is no rinsing extraction procedure for external component surfaces. The term “final rinsing” refers to a pressure-rinsing step which removes particles that have re-sedimented (onto the test component or in the extraction setup) after detachment and forwards them for filtration.

Agitation: agitation is a suitable method for extracting particles from simply-shaped, spacious inner areas of components without narrow cross-sections or apertures that are difficult to seal. It is not suitable for narrow pipes or capillaries because the test liquid cannot be sufficiently agitated to remove particles effectively.

Advantages:	Simple, very cheap, low blank levels due to low wetted surface areas.
Disadvantages:	Only compatible for use on a very small spectrum of components. Cannot be performed manually on large test components (mass of test object plus test liquid).

NOTE 3 The term “agitation” always refers to extraction from inner surfaces; a procedure for extracting particles from the outer surfaces of components (one or several) by immersing them in a receptacle filled with liquid and subsequently carrying out an agitation or swilling step is not covered in this document (risk of component abrasion).

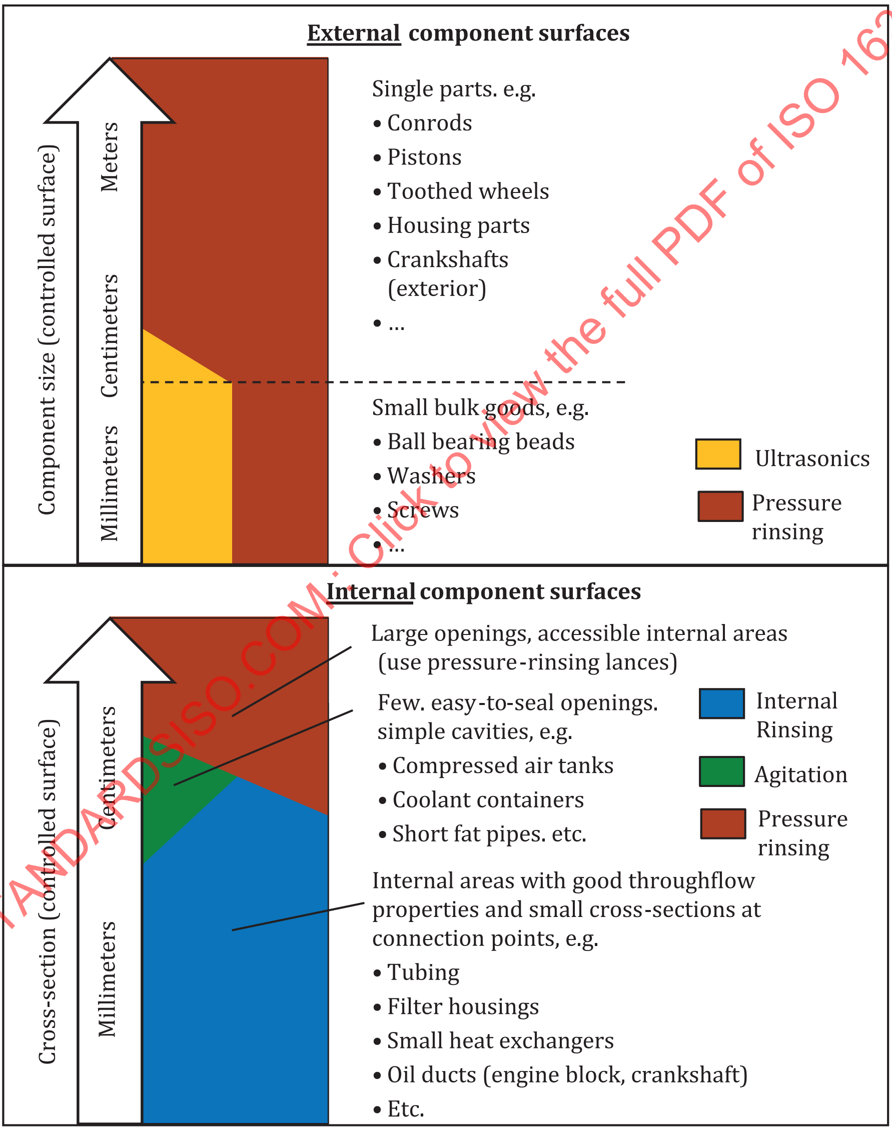


Figure A.1 — Suitability of extraction methods depending on features of test components

Table A.1 — Examples for suitability of extraction methods regarding component features

Extraction method	Component features	Examples
Pressure-rinsing or ultrasonic vibration	**Small components**, several millimetres to a few centimetres in size, difficult to handle individually, for extraction in bulk (total controlled surface)	Ball bearings, gaskets, screws, springs, O-rings, etc.
Pressure-rinsing	**Small to large components,** a few centimetres to over one meter in size (total controlled surface)	Connection rods, pistons, toothed wheels, housing parts, crankshafts, etc.
Internal rinsing	**Component areas with good through-flow properties,** with connector cross-sections and interior cross-sections sized between a few millimetres and several centimetres (inner area)	Tubing, piping, filter housings, heat exchangers, etc.
Internal rinsing or pressure-rinsing	**Inner component surfaces** with connector cross-sections and interior cross-sections in the centimetre range; accessible by internal rinsing and by pressure-rinsing jet	Hydraulic valve blocks, pipe or tube connections, turbocharger housing, rails, etc.
Internal rinsing and pressure-rinsing	Components with **separate functional areas**, of which some are suitable for extraction by pressure-rinsing (exterior) and others by internal rinsing (interior), (one after the other on different analysis filters)	Cylinder crank housing, crankshafts, etc.
Agitation	Geometrically **simple inner surfaces** of components without narrow cross-sections and with few easy-to-seal openings (handling limit with manual extraction: mass of component plus test liquid)	Compressed air tanks, coolant containers, etc.
Agitation or internal rinsing	**Inner surfaces** of components with **good through-flow properties** and few easy-to-seal openings (cross-sections of apertures and inner areas in lower centimetre range)	Piping, simple heat exchangers (with cross-sections that are not too narrow), etc.
Agitation or pressure-rinsing	Geometrically **simple inner surfaces** of components without narrow cross-sections, with few easy-to-seal openings and apertures with large cross-sections	Tanks, short fat pipes, etc.

A.3.3 Air extraction

Air extraction can be performed in two ways, either as air jet extraction, which corresponds with pressure-rinsing in liquid extraction, or as through-flow extraction where air flows through the test component, which is comparable with internal rinsing as a form of liquid extraction (for examples see Table A.2).

Air jet extraction: With this extraction method, particles are removed from the component by a jet of clean, oil-free compressed air. The method is suitable for external surfaces, or for inner component surfaces that can be easily accessed by the jet of compressed air. It is used for electronic components, components forming part of engine air intake systems where the through-flow method cannot be applied, or for logistics packaging such as blister packs, small load carriers or cardboard.

Advantages: Closed cabinets for liquid extraction procedures can be easily and cheaply converted to cabinets for air jet extraction. The simple test method can also be applied to components that would be damaged if they came into contact with a liquid.

Disadvantages: The detached particles are spread over a large area → a thorough final rinse (with liquid) is required, low blank levels are difficult to attain due to the large internal surfaces of the extraction setup, high operator influence because the procedure is generally carried out manually.

Air through-flow extraction: with this type of extraction, air is usually guided through the test component using specially-constructed extraction setups. The method is suitable for evaluating the cleanliness of inner surfaces of air conducting components, such as parts of engine air intake systems. With this procedure, large volumes of air are guided through component cross-sections several centimetres in diameter.

Advantages:	Easy to separate the test area from non-relevant component surfaces by using adapters connected to piping systems. Low operator influence: for air intake components, the best extraction method for simulating real vehicle conditions. Air filters, which are not compatible with liquids, can be inspected using this method. The test fluid leaves no residues in the component.
Disadvantages:	Complex extraction setup, need for component-specific adapters, time-consuming execution due to the preparation of primary analysis filters for air which then need to be manually re-prepared as secondary analysis filters for liquid.

Table A.2 — Examples of components and suitable extraction methods

Extraction method	Component features	Examples
Air jet extraction	**External areas** or **inner surfaces of components accessible by a jet of compressed air,** which do not come into direct contact with liquid during their manufacture or operation and which would be damaged if they came into contact with liquid	Electronic printed circuits, single components of air intake tracts not suitable for air through-flow extraction, packaging (SLC, blisters, cardboard), etc.
Air through-flow extraction	**Inner component surfaces with good through-flow properties** which do not come into direct contact with liquid during their manufacture or operation and which would be damaged if they came into contact with liquid	Tubing, housing, filters, manifolds, bellows in air intake tract

A.4 Time

As opposed to other extraction parameters, extraction times are optimized for each component and not derived from features such as component geometry or type of contamination. The required time is determined by repeating cleanliness analyses with short exposure times and registering the decline in the particulate load extracted - known as declining tests (see Clause 6). Even in cases where the volume of test liquid is used for validation purposes, the actual parameter that is qualified is the length of time the extraction method acts on the controlled surface.

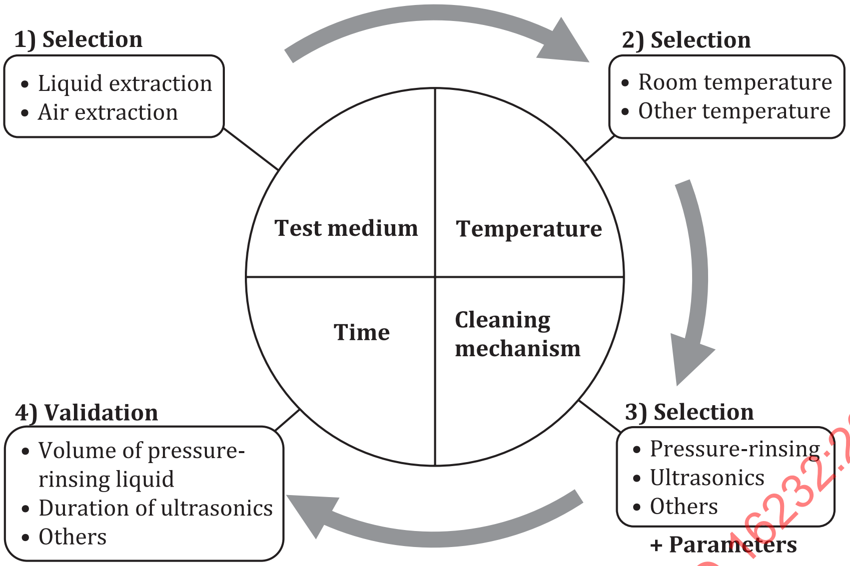


Figure A.2 — Procedure for selecting the extraction method

A.5 Filtration method

Through the increasing use of optical analysis, in which particles are counted and measured on analysis filters, the careful selection and execution of the filtration procedure are crucial to the quality of the later analysis step. Analysis filters are chosen according to their compatibility with the test liquid and their capacity to retain particles. To ensure that the correct particles are retained by the analysis filter, the particle sizes stated in the cleanliness specification are of utmost importance.

NOTE If further analyses are required, it could be necessary to verify whether the analysis filter and analysis method are compatible with one another (e.g. if high radiation levels are used).

A.6 Analysis method

A.6.1 General

The analysis method is selected according to the particle features stated in the cleanliness specification for the respective test component and also depends on the purpose of the analysis (see also Table A.3 and Table A.4).

Standard analysis: this verifies the cleanliness limit values arranged between the customer and the supplier. In the standard analysis, the analysis method and the particle features requiring analysis are determined. Additionally, parameters for the analysis method are specified and particle features defined. Standard analysis can be implemented without further agreements having to be made between the customer and supplier.

Extended analyses: these supply information about the type and thus possible origin of particles. They are used:

— to optimize processes,

— to research into causes (source of critical particles),

— to react to limit values being exceeded, and

— if the requirements made in a cleanliness specification go beyond the features covered by standard analysis.

Extended analyses are often associated with more effort and higher costs.

Shortened analyses: due to the rapid availability of results and therefore higher sampling quantities possible, these methods are highly suitable for the purpose of process monitoring.

NOTE Standard or extended analysis methods can also be used to monitor processes if they are performed on a regular basis.

A.6.2 Standard analysis

Standard analysis is used to validate cleanliness limit values in the customer-supplier relationship. The two following analysis methods are used in a standard analysis procedure.

Gravimetry:

It is used to determine the total mass of all particles extracted from the component and deposited on the analysis filter.

Light-optical analysis:

With light-optical standard analysis, microscopes or flatbed scanners are used to determine particle size classes and measure or characterize particles according to the following features.

Measurement of particles >50 µm with regard to:

- length and/or
- width.

Characterization of particles with regard to:

- fibre shape and
- metallic shine (optional).

In order to implement light-optical analysis methods, skilled staff is required. Staff should be able to operate the equipment and scrupulously check measurement and characterization results.

In order to improve the comparability of results from different types of analysis systems as well as from identical systems handled by different operators, it is essential that the stated equipment, parameters, procedures and features are implemented and observed. If specified conventions are deviated from in a cleanliness inspection, the analysis is no longer considered to be standard and it is of importance that the respective points are agreed on and documented in the customer-supplier relationship.

A.6.3 Extended analyses

Extended analyses are carried out if the following additional information on contaminant particles is required:

- exact measurement of particles <50 µm;
- geometric features of particles other than length and width, e.g. the third dimension;
- material identification or characteristics derived from this, such as the degree of particle hardness.

The aim of the respective analysis, the method applied, the procedure implemented and the processing and interpretation of analysis results are all to be arranged and documented in the customer-supplier relationship.

A.6.4 Shortened analysis

The term “shortened analysis” refers to analysis techniques which, although needing an extraction step, do not require filtration for the actual analysis. This enables analysis results to be available much faster, i.e. larger quantities of components can be assessed within the same period of time. Results obtained from the two shortened analysis methods mentioned do not correspond exactly with those obtained from a standard analysis. Consequently, shortened analyses are not used to validate cleanliness limit values unless this has been explicitly arranged in the customer-supplier relationship.

Tables A.3 and A.4 list the analysis methods described in A.6 and their fields of application.

Table A.3 — Analysis methods and possible applications

Analysis method			**Purpose of the analysis**		
			Validate cleanliness specification	**Cause research for process optimization**	**Process monitoring**
Standard analysis	Gravimetry		++	−−	+
	Scanner 2-D		++	+	+
	Light microscope	2-D ≥ 50 µm	++	+	+
Extended analysis		2-D < 50 µm	++[a]	+	+
		3-D	−[a]	++	−−
	Computer tomography		−[a]	++	−−
	SEM/EDX		o[a]	++	−
	LIBS		o[a]	++	−
	Raman		o[a]	++	−
	Infrared		−[a]	++	−−
Shortened analysis	Liquid particle counter		o[a]	−	++
	Filter-blocking (optical)		o[a]	o	++

[a] Can be applied if arranged in the customer-supplier relationship provided analysis parameters are specified.

++ highly suitable

\+ suitable

o suitable in principle

− less suitable, or not available for automated analysis, or too expensive for the intended purpose (if new equipment needs to be purchased)

−− unsuitable, not available for automated analysis, too expensive for the intended purpose (if new equipment needs to be purchased)

Table A.4 — Analysis methods, information obtained and limits

Method	Information						Limitations
	Overall particle mass	Particle size			Other features		
		Length	Width	Height	Metallic shine	Material	
Gravimetry	x						Gives no information about single particles
Scanner		x	x		(x)[b]		Can only be verified on the image, greater optical magnification or other contrast methods not possible
Light microscope		x	x	(x)[a]	(x)[b]		Not all features usable with one lens
Computer-tomography		x	x	x[e]			Prolonged analysis times
SEM/EDX		x	x			x	No differentiation between organic/inorganic particles
LIBS		x[c]	x[c]			x	No differentiation between organic/inorganic particles
Raman		x[c]	x[c]			x	Metals not detected, limitations with fluorescing and black particles
Infrared		x[c]	x[c]			x	Only organic particles, no fully-automated analysis
Liquid particle counter (OPC)		x[d]					Results may be inaccurate if air bubbles are present
Filter-blocking (optical)		x	x				Results may be inaccurate if residues are on the lens

( ) Depending on lenses and model.

[a] Possible with material microscopes with small depth of field (focusing position between background of analysis filter and highest point of particle).

[b] Pre-requisite: manual double-check.

[c] Material analysis coupled with automated light-optical particle detection.

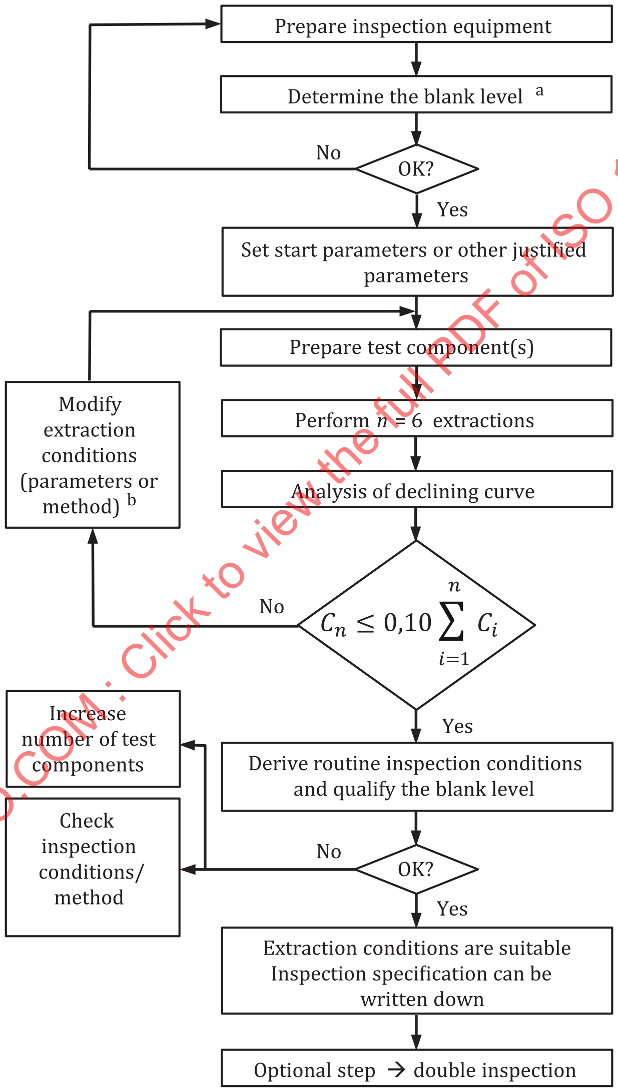
[d] Size often determined by the diameter of a circle with an equivalent surface area.

[e] Here, all size information can be determined (volume, thickness, cross-section, etc.).

Annex B
(normative)

Qualification tests and blank level

B.1 Qualification test procedure (synopsis)

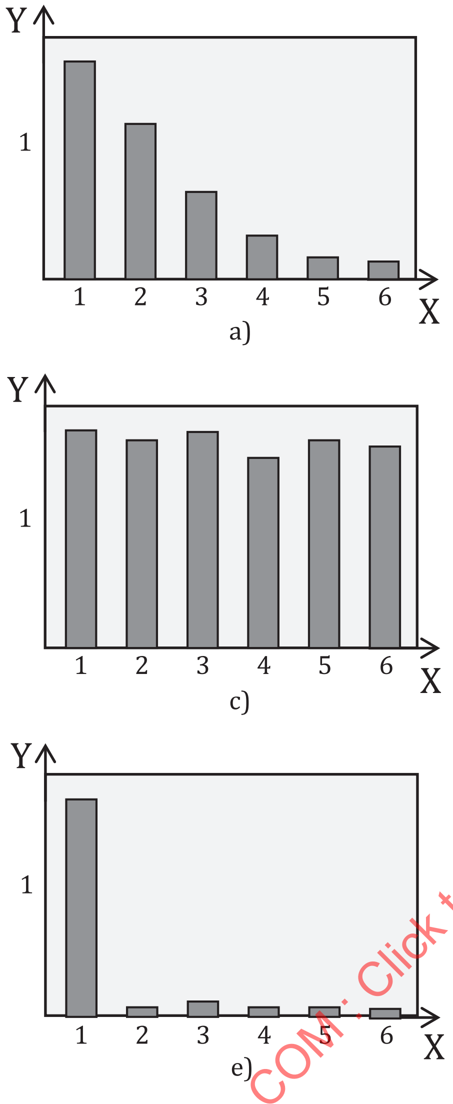


[a] This is a provisional blank level, see 6.2.3.1 b)

[b] See also different forms of extraction curves and their possible interpretations in Figure B.2.

Figure B.1 — Synopsis of qualification test procedure (declining test)

B.2 Various shapes of extraction curve



Key

X	consecutive extraction steps
Y	cleanliness value C_i
a)	constant decline
b)	delayed decline
c)	no decline
d)	increase
e)	immediate decline

Figure B.2 — various shapes of extraction curve

Interpretation of different extraction curves (see Figure B.2)

About a) Constant decline: contamination is detached at a uniform rate, no further interpretation required, the routine inspection procedure can be fixed.

About b) Delayed decline "humped curve": detachment is not uniform due to delayed dissolving of contamination or process media such as preservatives by the test liquid.

If the declining criterion is attained after a maximum of 6 single tests, the routine inspection procedure can be drawn up in the same way as in a).

However, if the declining criterion is not achieved because the detachment of particles is severely delayed, the dissolving step can be carried out separately before commencing the actual extraction. To do this, the test component is immersed in a suitable solvent (e.g. the test liquid) for an appropriate length of time without any physical cleaning assistance such as pressure-rinsing or ultrasonic vibration. Alternatively, components can be filled with the solvent and sealed if interior surfaces shall be tested.

Both the volume of liquid used in the dissolving step, as well as the liquid used to finally rinse the receptacle in which the dissolving process was carried out, count as extraction liquid.

A further method to accelerate particle detachment during the extraction procedure is to use a test liquid with a stronger dissolving power. Care shall be taken to follow the relevant safety instructions.

About c) No decline: a similar quantity of particulate contamination is detached during each extraction step. There may be several reasons for this.

- The extraction effect (chemical detachment or mechanical influence) is so high that particles are extracted which do not count as adhering contaminants but originate instead from the component itself (material). For example, such particles could originate from using ultrasonic vibration on cast materials, coatings flaking off, burrs detaching or solder beads adhering firmly to the surface. In such a case, extraction parameters shall be modified to avoid **detrimental effects on component material** or a different extraction method shall be selected. This type of declining performance may also indicate that the required cleanliness values cannot be attained for the respective component because of its nature or the way it has been processed; it is therefore not suitable for use in clean areas in its present state.
- The amount of contamination found in each extraction step is the same as the blank level. In this case, an extraction procedure with a lower particle emission level from the component/lower blank level needs to be established. If the blank level is already very low, it could be that the component is so clean that a decline in the particle load is no longer possible because single particles are only detached sporadically during extraction steps. In such cases this shall be documented.
- Especially in the case of very clean components, another effective method which can be used to clearly differentiate between the particle charge requiring detection and the blank level percentage in the inspection (and thus to achieve a decline) is to increase the size of the test lot.
- Another reason for the absence of a decline may be residual magnetism in the case of ferromagnetic components. If this applies, components should be demagnetized before commencing the extraction procedure.
- The absence of a decline may also be due to an inadequate final rinsing procedure, i.e. some of the particles extracted from the test component remain in the extraction setup and are transmitted to later extraction steps.

About d) Increase: the quantity of contamination found in each extraction step increases. This may also be due to several factors.

- As in c), the problem could be caused by severe "component attack".

- — The extraction effect is so weak that adhering particles can only be detached after an intensive dissolving step as in b). This is the case, for example, if dried-on or resinous preservatives are treated with a weak solvent cleaner.
- — Accumulation due to insufficient final rinsing of the component or extraction setup, or incorrect handling.

About e) Immediate decline: the total amount of detachable particles is removed from the component in the course of the first declining step. Subsequent declining steps only show particle quantities in the same range as the blank level.

Exactly the same parameters and extraction quantities or times can be used for the routine inspection as for the individual declining steps.

The component may be "over-extracted", i.e. in the first extraction step, it is extracted for a longer period of time than that required to detach the particle load. In this case, the declining test can be repeated using lower volumes/shorter times for each extraction step in order to record a decline and derive the actual extraction time/volume necessary.

NOTE Any deviation in the shape of extraction curves can also be due to faulty handling or unsuitable extraction equipment.

B.3 Routine inspection procedure (synopsis)

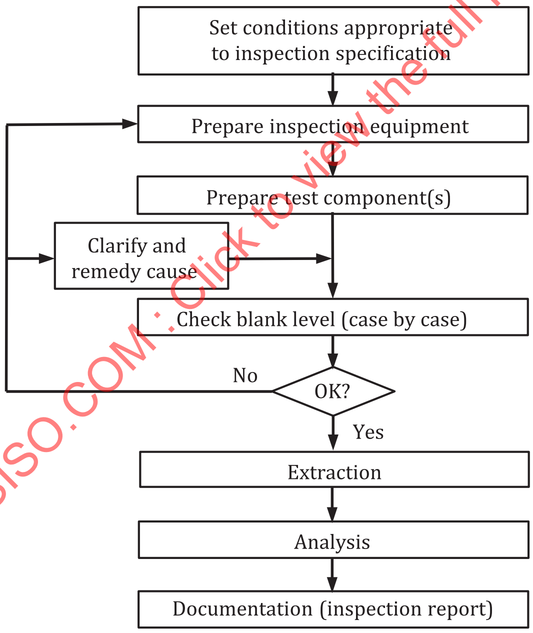


Figure B.3 — Synopsis of routine inspection procedure

Annex C
(informative)

Recovering test particles

C.1 Principle

The blank level test ensures that only foreign particles in quantities that are not critical to the result of a specific cleanliness inspection are introduced into the chain of extraction, final rinsing, filtration, handling of the analysis filter and analysis.

If there is a requirement to verify that no relevant particles are lost in the chain, an optional procedure with test particles can be performed. Here, a known size and quantity of particles are placed in the extraction setup and the final rinsing procedure and all further steps are carried out to assess in the analysis whether all the added particles are found again. The method can be applied, for example, to validate and optimize:

- final rinsing procedures;
- constructional features of extraction and filtration units;
- handling of analysis filters on removal, transport and drying;
- training of inspection staff.

C.2 Material and equipment

For this procedure, as well as all the materials and equipment utilized for the derivation of blank levels or for inspections, particles of a specific quantity and size are required. These particles are prepared in such a way so that they can be introduced into the extraction setup without loss or without the inadvertent addition of any foreign particles. Two types of particle can be used for this purpose.

a) Test particles produced in a defined way that they can be clearly characterized in the analysis method. For example, the design of the shape of the particles should enable a reproducible deposition on an analysis filter to provide the same image area to always be measured by light-optical analysis. Furthermore, in order to avoid “class hoppers” (particles that are sometimes assigned to one particle size class and sometimes to another), particle sizes may not be too close to the limit of a particle size class.

b) “In-house” particles originating, for example, from a company’s backwashed mesh filter from a component cleanliness inspection. These particles correspond with particles actually occurring during production and thus are also being found in daily laboratory operation in the routine inspection.

C.3 Procedure

a) Procure a supply of known test particles.

b) Introduce the test particles into the extraction setup.

c) Give the extraction setup a final rinse as specified in the routine procedure.

d) Proceed with analysis filtration, filter removal, filter drying and filter analysis.

e) Determine the recovery rate, compare it with the target value:

   i) in case of specially-produced test particles with defined features, the recovery rate should be 100 %;

   ii) if "in-house" particles are used, the potentially-attainable recovery rate depends very much on the quantity, size and geometry of the particles used as well as on the extraction and filtration equipment implemented. This should be defined individually for each case.

f) If the desired recovery rate is not achieved, the chain, i.e. final rinsing, filtration, filter handling and analysis procedure and/or equipment should generally be optimized and the evaluation with test particles repeated.

Annex D

(normative)

Extraction

D.1 Suitability and compatibility of test liquids

Table D.1 and Table D.2 only give a simplified overview of test liquids. Individual applications still require technical clarification. Tables D.1 and D.2 do not include special test liquids that are not grouped into the categories mentioned.

Table D.1 — Ability of test liquids to dissolve accompanying contaminants

Substance (contaminant)	Test liquid		
	Aqueous tensidic neutral	Polar solvents[a]	Non-polar solvents[b]
Salts (water-soluble)	+	–	–
Mineral oil-based lubricants (MBS)	–	+	+
Coolants/lubricants – aqueous emulsions (aqueous CLB)	+	+	o
Brake fluids (glycols, higher alcohols)	+	+	+
Animal and vegetable greases and oils (AVGO)	o	+	+
Silicon oil	–	–	–
Anti-corrosives, amine-based	+	+	o
Anti-corrosives, other	o/+	+	+
Wax	o Only above melting point	o	+

\+ suitable, o partially suitable, – unsuitable

[a] Polar solvents, e.g. alcohols.

[b] Non-polar solvents, e.g. cold cleaners or benzene.

Table D.2 — Compatibility of test liquids with materials

Material (test component and inspection equipment)	Test liquid		
	Aqueous tensidic neutral	Solvents polar[a]	Solvents non-polar[b]
Plastics	+	o	o
Elastomers	+	o	o
Coated surfaces	o	o	o
Magnesium and magnesium alloys	o With inhibitors	+	+
Aluminium, Al-alloys and cast aluminium, chromated aluminium	+	+	+
Zinc, zinc alloys and cast zinc	+	+	+
High-grade steel	+	+	+
Steel and cast iron	+	+	+
Hot-dip galvanized iron, electroplated and chromated	+	+	+
Non-ferrous metals (copper, brass, bronze, etc.)	+	+	+
Glass	+	+	+

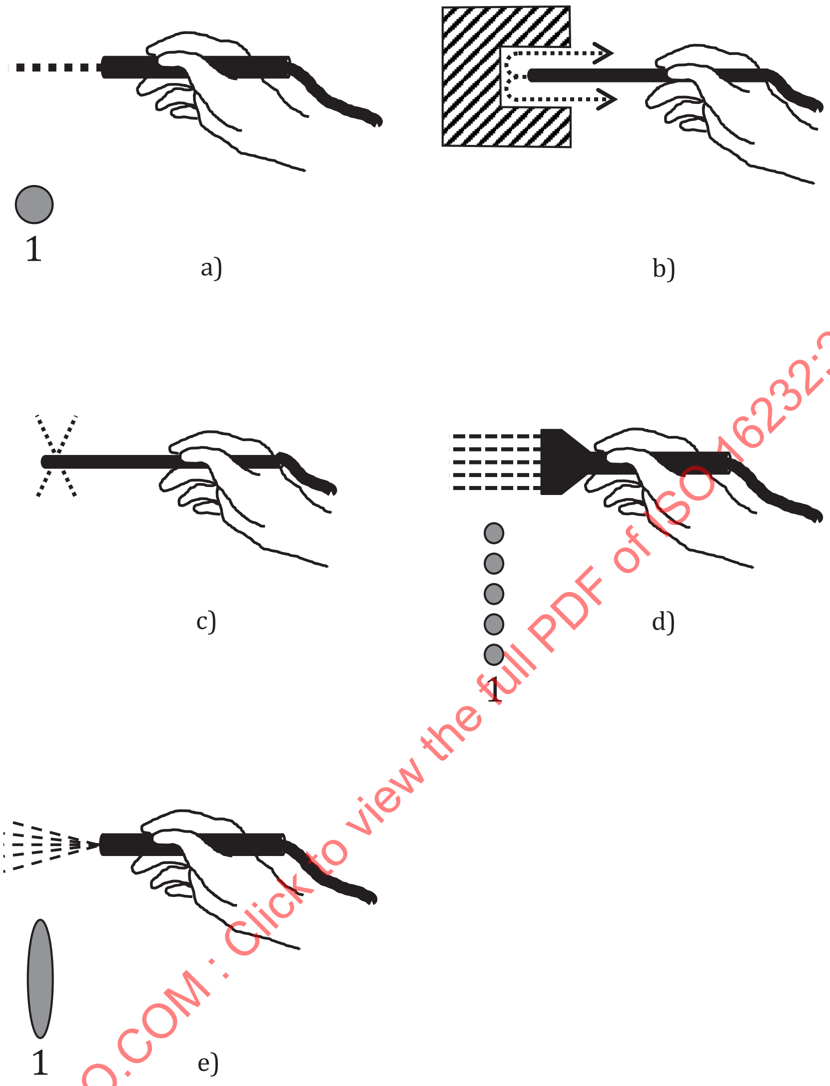
+ suitable, o partially suitable, – unsuitable

[a] Polar solvents, e.g. alcohols.

[b] Non-polar solvents, e.g. cold cleaners or benzene.

D.2 Pressure-rinsing

Type and shape of the rinsing nozzle can be adapted to the size and geometry of surface to be inspected (see Figure D.1).

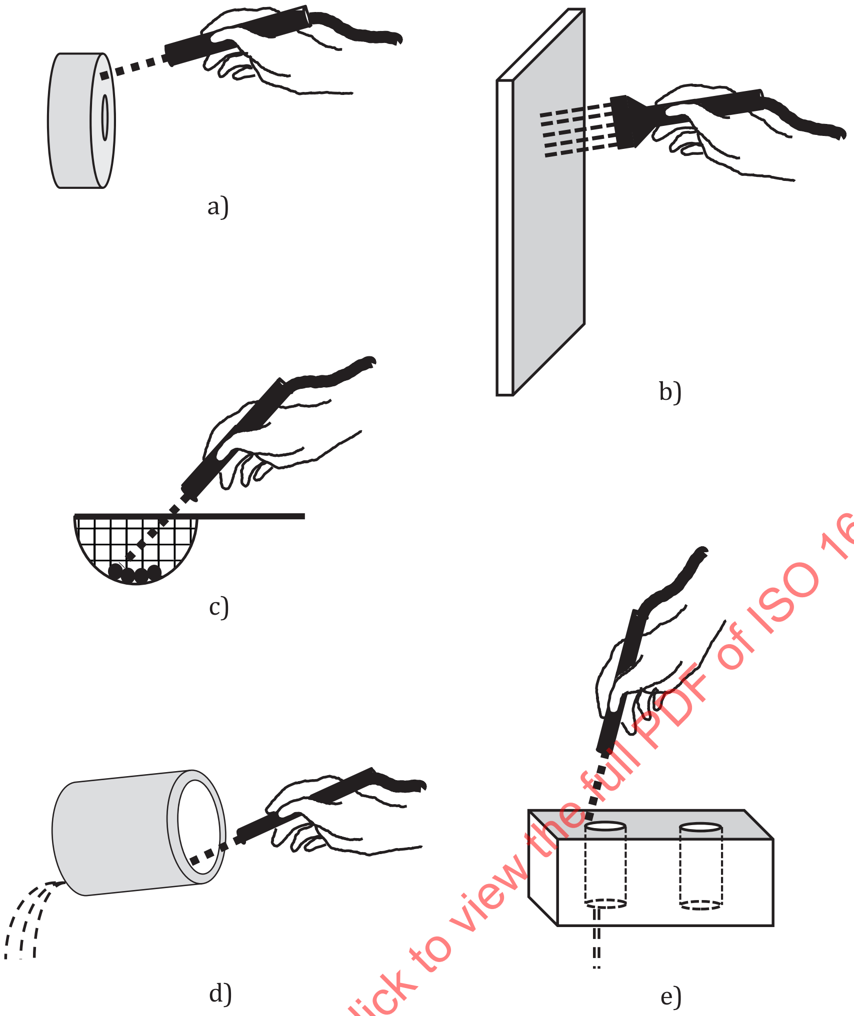


Key

a) round full-jet nozzle for localized extraction
b) pressure rinsing lance for extracting contamination from blind holes
c) "spiked lance" for extracting contamination from internal component areas
d) parallel-jet nozzle for extracting contamination from larger surface areas
e) flat-jet nozzle (fan nozzle) for extraction of larger surface areas (care with aerosol formation)
1 shape of jet

Figure D.1 — Examples of various pressure-rinsing techniques

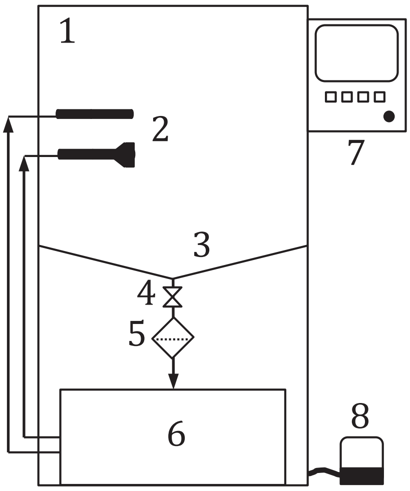
Figure D.2 illustrates examples of using pressure rinsing techniques on different components. A setup of a pressure rinsing cabinet is shown in Figure D.3.



Key

a) extracting single components
b) extracting large surfaces
c) extracting small components (bulk goods)
d) extracting accessible internal areas of component
e) extracting accessible internal areas of component

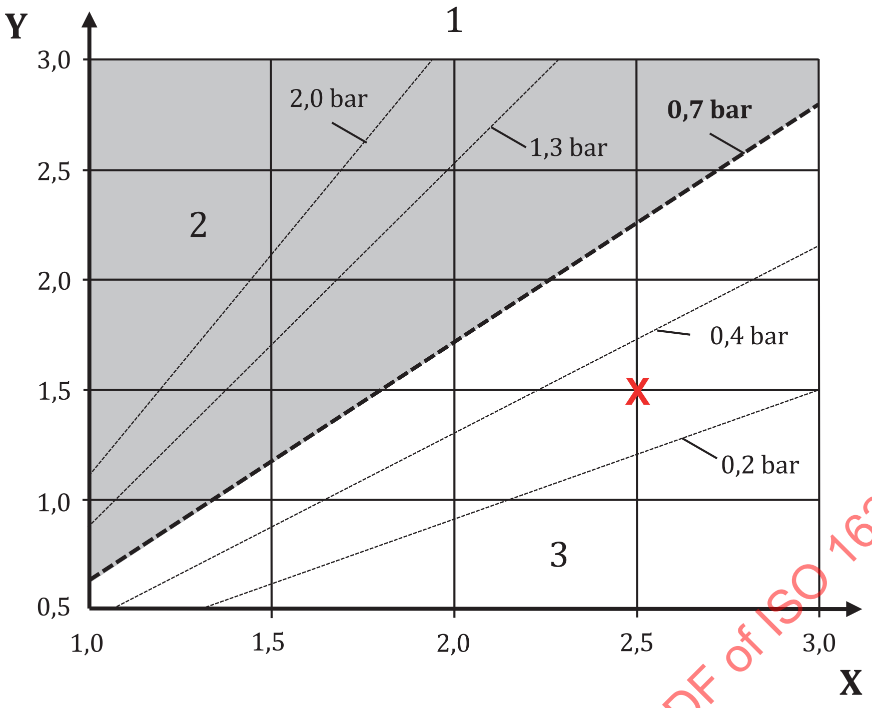
Figure D.2 — Examples of use of pressure-rinsing



Key

1	pressure-rinsing chamber (clean air area)
2	manual pressure-rinsing tools
3	collecting basin
4	stopcock
5	analysis filter
6	media supply with container for test liquid, purification filter, pump or pressure supply
7	control unit for pressure-rinsing programmes
8	foot pedal

Figure D.3 — Example of pressure-rinsing cabinet



Key

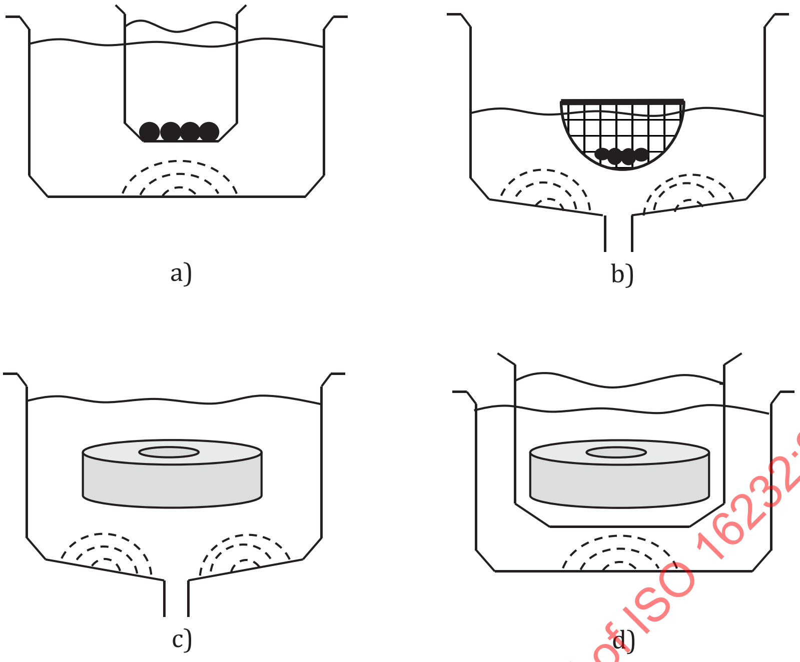
X	nozzle diameter, mm
Y	volume flow, l/min
1	jet pressure at nozzle level in relation of nozzle diameter (round full-jet nozzle) and volume flow as well as risk of aerosol formation
2	pressure larger 0,7 bar at nozzle level → aerosol formation probable, take explosion protection precautions (grey area in figure)
3	pressure smaller 0,7 bar at nozzle level → aerosol formation unlikely (white area in figure)

Figure D.4 — Information on nozzle diameters and flow rates

The start parameter values for the nozzle cross-section and flow rate are marked with an **X** (see Figure D.4) and lie in an area where there is no risk of aerosol formation.

D.3 Ultrasonic vibration

Figure D.5 illustrates arrangements for particle extraction on single parts and collectives of parts.

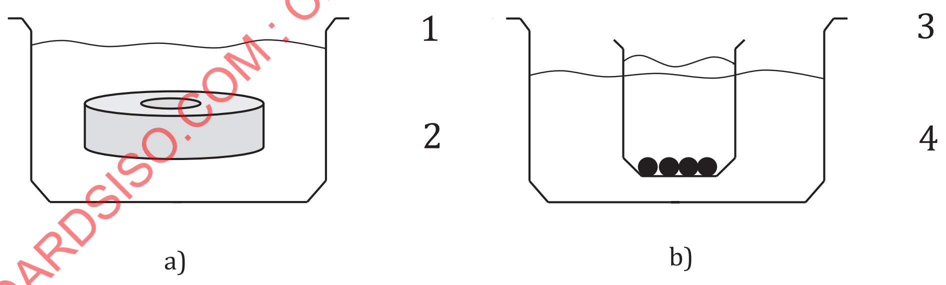

a) b) c) d)

Key

a) ultrasonic extraction procedure for small components (bulk goods in a beaker)
b) ultrasonic extraction procedure for small components (bulk goods in a mesh basket)
c) ultrasonic extraction procedure for single components
d) ultrasonic extraction procedure for single components (in a beaker)

Figure D.5 — Examples of use of extraction by ultrasonic vibration

Typical volumes and power densities requiring documentation are illustrated in Figure D.6.



a) b)

Key

a) component immersed in ultrasonic bath
b) component(s) immersed in beaker
1 volume analysed (extraction liquid) = volume subjected to ultrasonics = filling quantity of the bath
2 power density in bath = power of ultrasonic device/filling quantity of bath
3 volume analysed (extraction liquid) = filling quantity of beaker
4 power density in bath = power of ultrasonic device/(filling quantity of bath + filling quantity of beaker)

Figure D.6 — Volumes and power densities requiring documentation

With increasing frequency of the ultrasonic the related wavelength λ decreases (see Table D.3)

Table D.3 — Examples of ultrasonic wavelengths

Frequency	Wavelength λ
25 kHz	61 mm
35 kHz	43 mm
40 kHz	38 mm
120 kHz	12 mm

Examples of arranging components in the ultrasonic immersion bath are shown in Figure D.7.

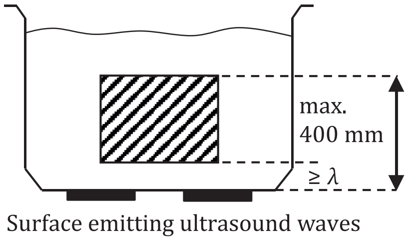
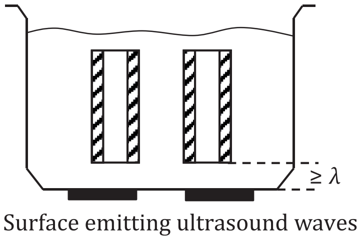
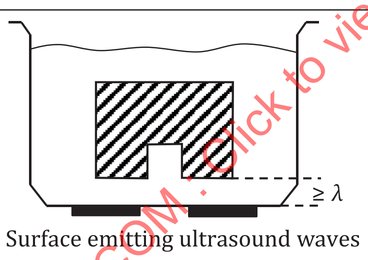
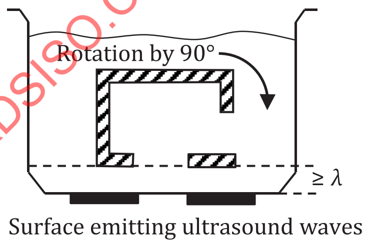
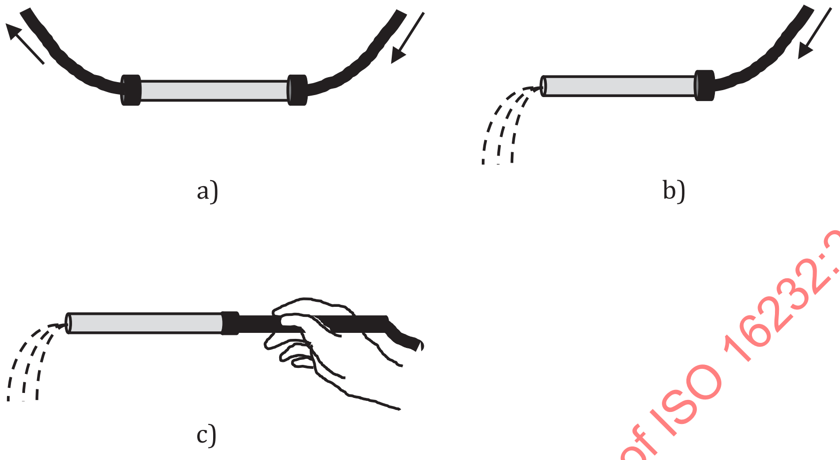
Extraction	Arrangement in bath	Remark
External Extraction	 	Distance between component and sound source should be ≥ λ. For distances >400 mm between the surface to be cleaned and the sound-emitting surface, components will need to be rotated.
Simple inner cavity	 	Orientation of component opening to sound-emitting surface
Blind hole or similar shape	 	Orientation of component opening to sound-emitting surface Ensure cavity is filled
Inner cavity	 	Orientation of component opening to sound-emitting surface Ensure cavity is filled

Figure D.7 — Examples of arranging components in the ultrasonic immersion bath

D.4 Internal rinsing

There are various configurations for applying internal rinsing technique (see Figure D.8 and Figure D.9).



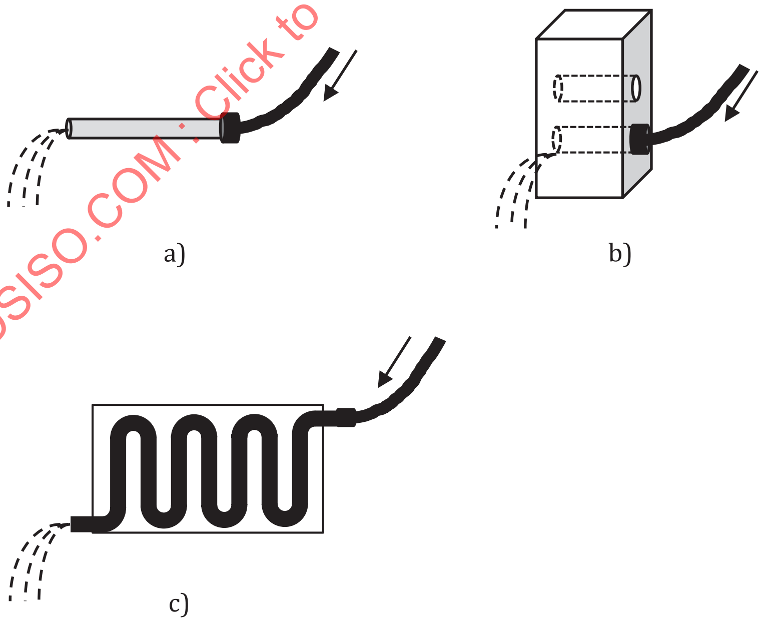
Key

a) internal rinsing in a closed system (adapters for tubing on both sides)

b) internal rinsing by adapting a rinsing line (pressureless drainage)

c) internal rinsing by adapting a pressure-rinsing tool (pressureless drainage)

Figure D.8 — Various internal rinsing examples



Key

a) internal rinsing of pipes or lines

b) internal rinsing of inner component areas (e.g. oil borings)

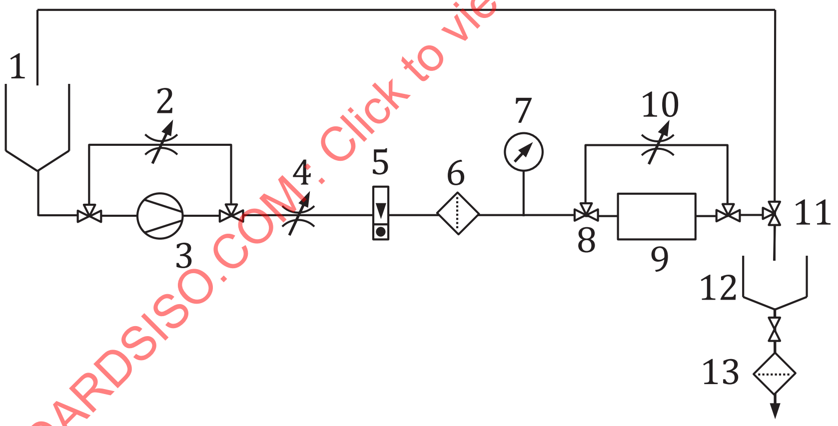
c) internal rinsing of assembly (e.g. heat exchangers or filter housings)

Figure D.9 — Examples of use of internal rinsing

Table D.4 — Examples of turbulent through-flow parameters

	Example: cold cleaner liquid 1	**Example: cold cleaner liquid 2**
Flashpoint [°C]	62	105
Kinematic viscosity [mm²/s]	1,9	3,2
Tube diameter [mm]	**Flow rate [l/min]**	**Flow rate [l/min]**
4	1,4	2,4
5	1,8	3,0
6	2,2	3,6
8	2,9	4,8
10	3,6	6,0
12	4,3	7,2
15	5,4	9,0
20	7,2	12,1
25	9,0	15,1
30	10,8	18,1
40	14,3	24,1
50	17,9	30,2

To calculate the turbulent flow through the tube, the Reynolds Number $Re > 4\,000$, which is defined via the equation $Re = w \cdot d/\nu$, (w: velocity of the flow, d: tube diameter, ν: kinematic viscosity) is used. The turbulent flow rate (dV/dt) for other liquids can be calculated on converting this to $dV/dt = Re \cdot d \cdot \pi \cdot \nu/4$.



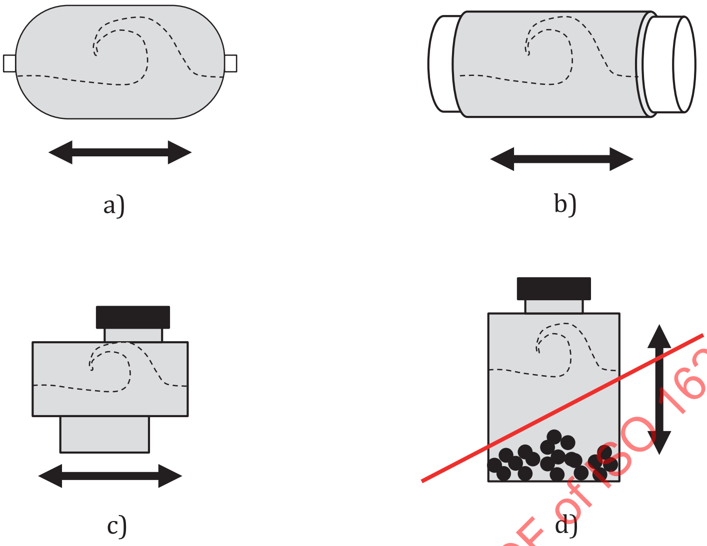
Key

1	container for test liquid
2	regulator at level of pump bypass
3	pump
4	main regulator
5	throughflow meter
6	purification filter
7	manometer
8	mounting device to hold test component
9	test component
10	regulator at level of test component bypass
11	three-way valve
12	collection vessel for extraction liquid
13	analysis filter

Figure D.10 — Example of an internal rinsing system

D.5 Agitation

Agitation is used to extract particles from the inner surface of the respective component (see Figure D.11).



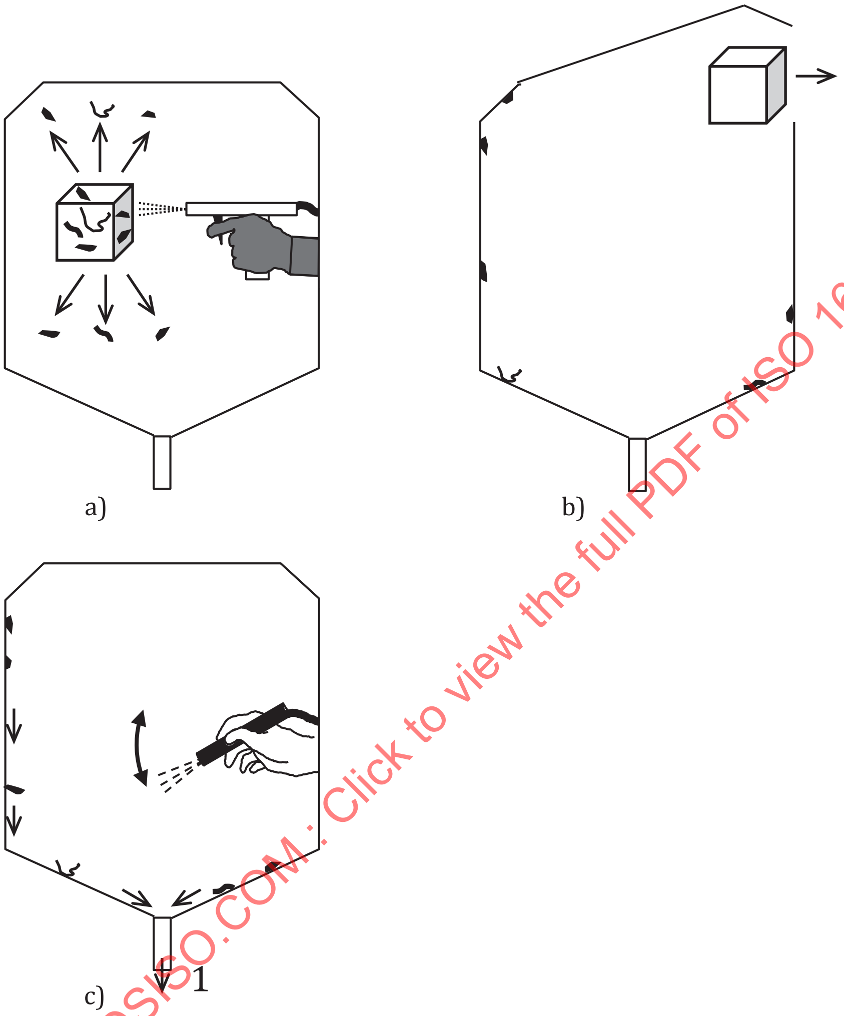
Key

a) agitation of a (pressure) tank
b) agitation of a section of piping (short fat cross-section)
c) agitation of container
d) agitation of small components in a container: not suitable

Figure D.11 — Examples of use of extraction by agitation

D.6 Air jet extraction

The air jet extraction procedure includes an extraction step on the component with compressed air and a final rinsing step with test liquid on the cabinet (see Figure D.12).



a)

b)

c)

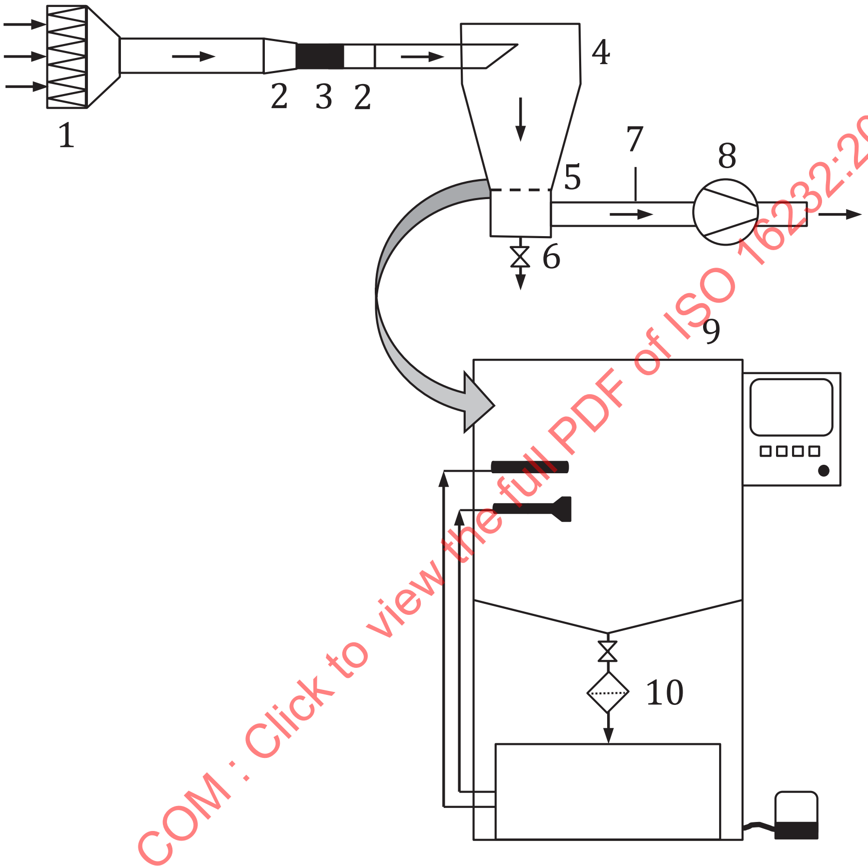
Key

a)	step 1: extract particle from test components using a jet of compressed air
b)	step 2: remove the test component
c)	step 3: perform final rinsing step on cabinet without test component using test liquid
1	analysis filter

Figure D.12 — Principle and procedure for air jet extraction

D.7 Air through-flow extraction

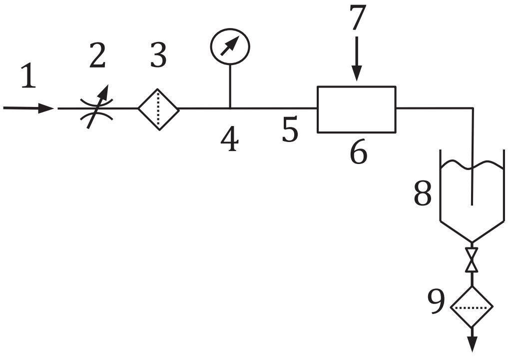
One principle of the air through-flow extraction method is based on air extraction with subsequent preparation of a secondary analysis filter by means of liquid extraction (see Figure D.13). Another variant is based on collecting the extracted particles in test liquid (impinge principle) followed by an analysis filtration step (see Figure D.14).



Key

1	air filter	6	drainage system for final rinsing liquid
2	adapter to test component	7	sensor to monitor volume flow
3	test component	8	pump
4	primary filtration unit	9	pressure rinsing cabinet
5	primary analysis filter	10	secondary analysis filter

Figure D.13 — Principle set-up required for air through-flow extraction on components such as engine air intake components



Key

1	intake oil-free compressed air	6	test component
2	regulator	7	actuator for test component (only for active components)
3	air filter	8	container for extraction liquid
4	manometer	9	analysis filter
5	mounting device to hold test component		

Figure D.14 — Principle set-up required for air through-flow extraction on components such as parts of pneumatic systems

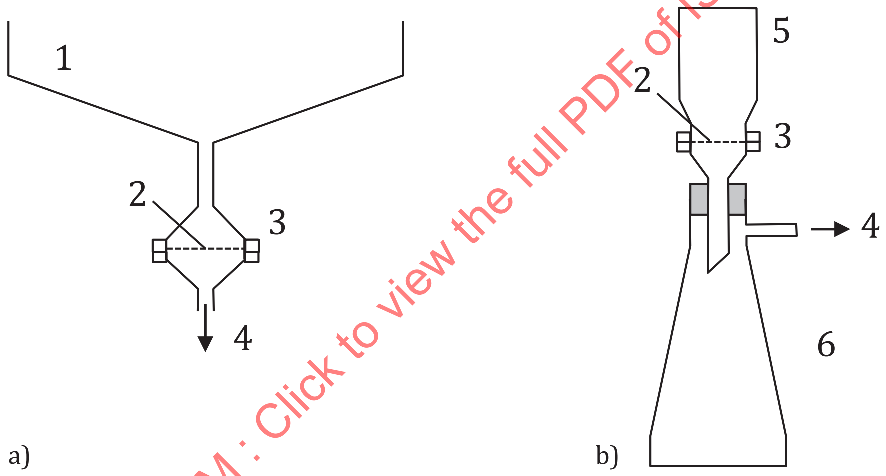
Annex E
(informative)

Filtration

E.1 Filtration methods

Direct filtration of extraction fluid (see Figure E.1 a)) is often used in combination with pressure rinsing cabinets or ultrasonic bath with drain. The separate filtration setup (see Figure E.1 b)) is used if the extraction liquid is e.g. contained in a beaker used for extraction of bulks of small components in an ultrasonic bath (see Figure D.5 a)).

To pre-select particle sizes, analysis filtration can be performed using cascades of filters with different pore sizes as shown in Figure E.2.

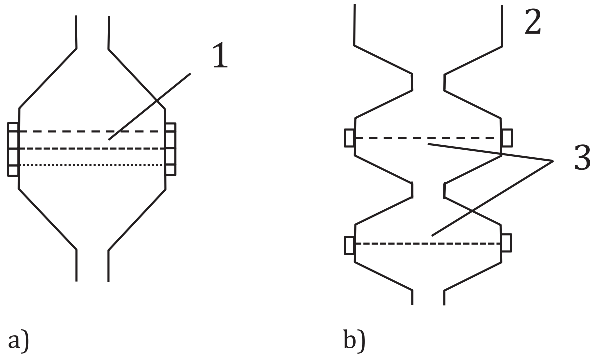


a)   b)

Key

a)	direct filtration	3	filter holder
b)	separate filtration	4	vacuum source
1	for example collection vessel or ultrasonic bath	5	filter funnel
2	analysis filter	6	vacuum flask

Figure E.1 — Different analysis filtration methods



Key

a)	filter holder with cascade of filters
b)	cascade of filter holders
1	2 or 3 analysis filters with different pore size
2	filter funnel
3	2 analysis filters with different pore size

Figure E.2 — Filter cascades

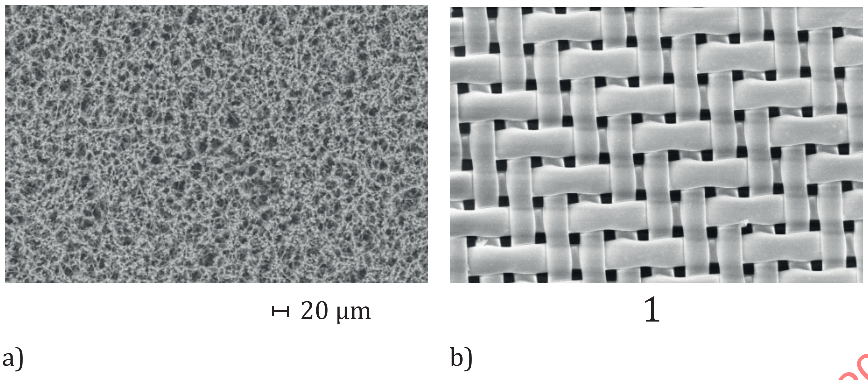
E.2 Compatibility of analysis filters with test liquids

Table E.1 — Compatibility of analysis filters with test liquids

Filter material		Medium (test or final rinsing liquid)				
		Aqueous neutral cleaner	Isopropanol	Ethanol (spirit)	Aliphatic hydrocarbons, e.g. also cold cleaners	Ketones, e.g. acetone
Foamed membrane	Cellulose nitrate	+	–	–	+	–
	Cellulose acetate	+	+	–	+	–
	Polyamide/ nylon	+	+	+	+	+
Mesh	Polyester	+	+	+	+	+
	Polyamide/ nylon	+	+	+	+	+
+ suitable, o partially suitable, – unsuitable						

Table E.1 lists only a few of the most common types of analysis filters. In some cases, e.g. if resistance to a certain test or final rinsing liquid is required, or if a certain type of filter is necessary for the analysis method, different filter types may need to be selected. If no special conditions are specified, the use of the analysis filter suggested in 8.2 is recommended.

E.3 Examples of analysis filters



Key

a)	8 µm cellulose nitrate foamed membrane filter
b)	15 µm PET mesh filter
1	magnification 200 ×

Figure E.3 — Examples of surface structure of analysis filters

E.3.1 Foamed membrane filter (Figure E.3 a))

Advantages: relatively smooth surface; highly-suitable for light-optical analysis.

Disadvantages: the undefined, foam-like structure of the material also retains pigment particles that are much smaller than the nominal pore size of the filter. These are not relevant to the cleanliness specification but may optically darken the analysis filter (high number of tiny particles), thus severely impairing light-optical analysis (grey or black analysis filter). This type of filter often absorbs humidity (impairs gravimetric analysis).

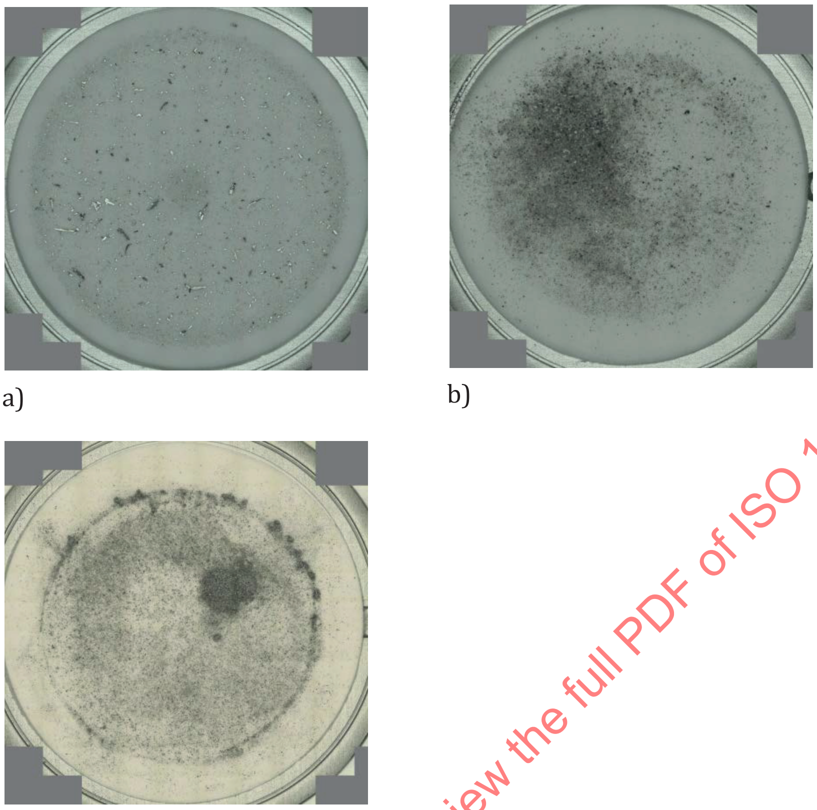
E.3.2 Mesh membrane filter (Figure E.3 b))

Advantages: defined geometric pore size and separation limit. In consequence, fewer pigment particles are retained, which facilitates light-optical analysis. In general, less humidity is absorbed by this type of filter (more reliable gravimetric results).

Disadvantages: with high degrees of magnification or very poor illumination, the structure of the mesh filaments may cause artefacts or reflections and impair light-optical analysis.

Very thin particles may pass the mesh filter and thus not be captured in the subsequent analysis.

E.4 Examples of problems encountered with analysis filtration and possible remedies



a)

b)

c)

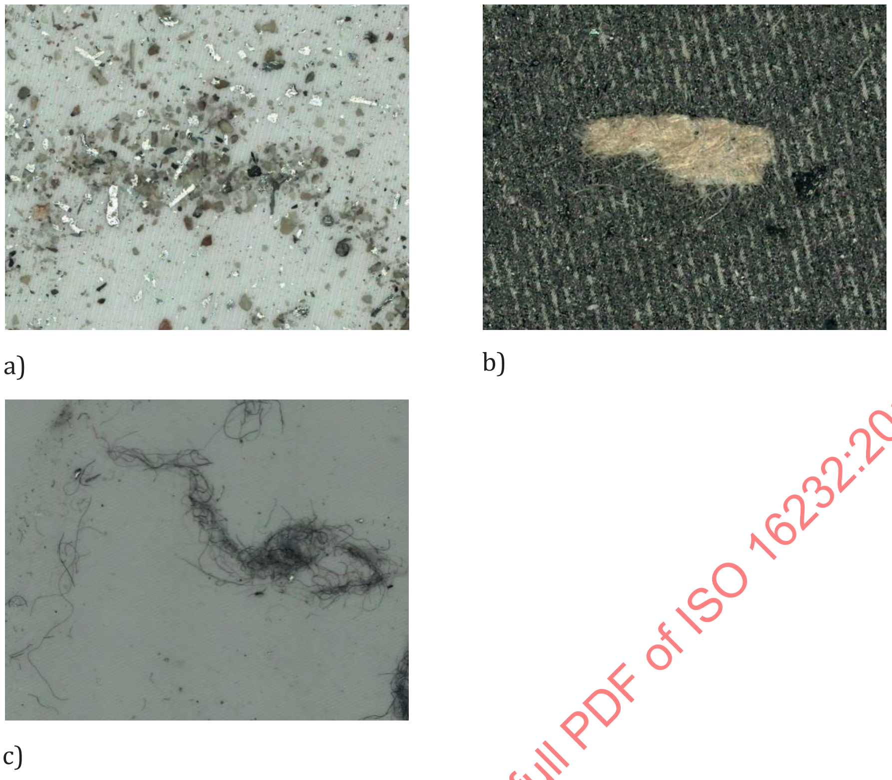
Key

a) good filter occupancy
  - — uniform
  - — most particles isolated

b) excessive filter occupancy
  - — extract fewer components per filter
  - — use filter cascade
  - — use coarser analysis filter

c) inhomogeneous filter occupancy
  - — optimize filter holder or filtration process
  - — post-process filter (refloat particles or gently shake filter holder with a small amount of liquid)

Figure E.4 — Examples of variants of filter occupancy



Key

a) excessive filter occupancy
  - extract fewer components per filter
  - use filter cascade
  - use coarser analysis filter

b) filter completely clogged
  - not analysable optically
  - only analysable by gravimetry

c) large number of fibres on filter
  - storage location of test components may be unsuitable

pieces of cardboard on the filter
  - test component packaging may be unsuitable

Figure E.5 — Examples of problems with filter occupancy

E.5 Filter occupancy and analysability

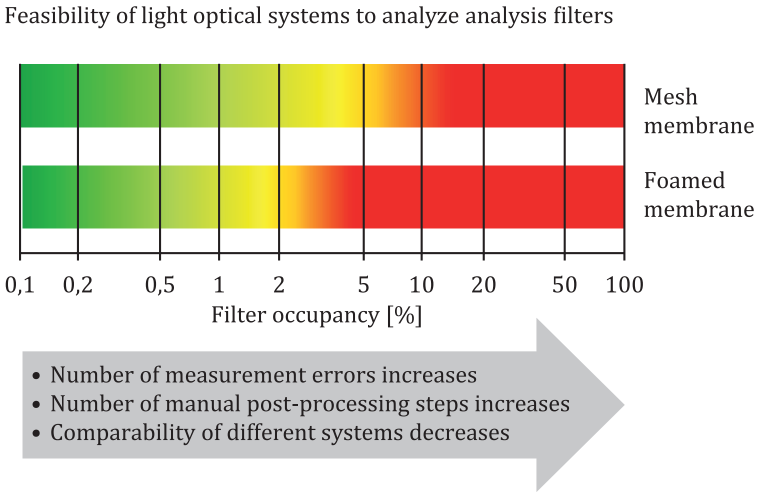


Figure E.6 — Relation of filter occupancy and analysability

Light-optical analysis becomes difficult when mesh membrane filters have a filter occupancy upwards of 3 % and foamed membrane filters an occupancy of 1,5 % and above (see Figure E.6). The number of measurement errors and quantity of manual corrective steps increase while the comparability of results from analyses performed using different systems decreases. The ability to analyse filters also depends on other factors (homogeneous occupancy, percentage of fibres, etc.). Consequently, these values only serve as a rough indication and require individual assessment in each case.

If filter occupancy is extremely homogeneous, it may be possible to analyse filters with higher particle occupancy.