VDA | Verband der Automobilindustrie

19

Quality Management
In the Automotive Industry

Inspection of   Part 1
Technical Cleanliness

- Particulate contamination of functionally-relevant automotive components -

2nd Revised Edition, March2015
(former title: VDA volume 19)

Inspection of technical cleanliness

Particulate contamination of functionally-relevant automotive components

2**nd revised edition, March 2015**
(former title: Volume 19)

Verband der Automobilindustrie e. V. (VDA)

Non-binding VDA recommendations concerning standards

The Association of the German Automotive Industry (VDA) recommends its members to use the following standards when implementing and upholding quality management systems.

Exclusion of liability

This VDA volume is a recommendation which is available for general use. Anyone using it is responsible for ensuring that the information is always applied correctly.

The VDA volume takes current practices into account that were valid at the time of issue. Implementation of VDA recommendations relieves no one from the responsibility for their own actions. In this respect everyone acts at their own risk. The VDA and those involved in VDA recommendations shall bear no liability.

If, during the use of VDA recommendations, errors or the possibility of mis-interpretation are found, it is requested that the VDA be notified of these immediately so that any possible faults can be corrected.

Information concerning the use of standards

The quotations from standards identified with their DIN number and issue date are reproduced with the permission of the DIN Deutsches Institut für Normung e.V. The version with the latest issue date, available from the publishers Beuth Verlag GmbH, 10772 Berlin, is definitive for the use of the standard.

Copyright

This publication is protected by copyright. Any use outside the strict limits of copyright law is not permissible without the consent of VDA-QMC and is liable to prosecution. This applies in particular to duplication, translation, microfilming and the storing or processing in electronic systems.

Translations

This publication will also be issued in other languages. The current status can be requested from VDA QMC.

Index of Contents

Since the first VDA 19 edition was published in January 2004, the relevance of technical cleanliness has steadily increased and is now an integral quality feature in the automotive industry. In the course of this development, hundreds of cleanliness laboratories have been set up in automotive and supplier works, and numerous service laboratories have also come into being. The new job of "technical cleanliness inspector" has been created as well as a multitude of posts (often departments) within affected companies that concern themselves with and coordinate all aspects of technical cleanliness (see also VDA 19 Part 2 Technical Cleanliness in Assembly).

Over the years since it first published, due to its broad application and the wealth of knowledge possessed by the people using VDA 19, it has become necessary to amend and add supplements to the guideline.

At the same time, numbering was also made consistent: the guideline on "The "Inspection of Technical Cleanliness" is now called VDA 19.1 or VDA 19 Part 1. This brings it into line with the second volume titled "Technical Cleanliness in Assembly", which is numbered VDA 19.2 or VDA 19 Part 2.

The main reasons behind its comprehensive revision (December 2012 till June 2014) were to:

- improve the comparability of cleanliness analysis results
- include new (extraction and analysis) techniques
- consider work protection within the scope of the guideline
- provide information on how to define cleanliness specifications and explain how to react when cleanliness limit values are exceeded.

The basic methods and techniques of component cleanliness analysis that were described in the first edition have proved to be very effective and are therefore included in full in the revised version in a highly-precise and more detailed form.

The second revised and extended edition comprises the following topics and contents:

- A new informative **Chapter 2: Cleanliness specification,** in which the procedure for defining cleanliness specifications is described, taking technical requirements and the aspects of technical and economic feasibility into account.

- An extended informative **Chapter 3: Selecting the inspection method**, which explains how to choose suitable extraction procedures, analysis filtration methods and analysis techniques. Tables and graphs provide assistance in selecting the best extraction method and also show the suitability of specific analysis methods for assessing different contaminant features and cleanliness specifications.

- An abbreviated **Chapter 4: Handling test components cleanly**. The section "Preparatory steps and post-treatment of test components" that was previously located here can now be found in a more appropriate place in the chapter "Extraction". Chapter 4 also contains a reference to VDA 19 Part 2 in which numerous aspects of this subject are addressed in detail. A new paragraph has been added to this chapter that regulates the criteria which exclude components from a cleanliness inspection.

- An extended and more precise **Chapter 5: Qualification tests and blank value**, which especially describes in a clearer and more binding manner how to perform and interpret declining tests, e.g. by specifying six mandatory declining steps.

- In the **Chapter 6: Extraction methods**, the proven methods for sampling with liquid (pressure-rinsing, ultrasonics, internal rinsing and agitation) have been supplemented, where possible, by so-called start parameters; these aim to narrow down parameter diversity in a meaningful way and thus make it easier to compare different extraction procedures. A prior dissolving step is also described that can be used on components which, due to the presence of conservation agents for example, exhibit a delayed declining behavior.

  The chapter also describes two new extraction methods using air as an extraction medium – air throughflow extraction and air jet extraction. These techniques are suitable, for example, for components that may not come into contact with liquids during their manufacture or operation.

- A completely new **Chapter 7** is devoted to the topic of **filtration**. Correctly-selected and carefully-prepared analysis filters are an absolute necessity in order to attain error-free results from optical analysis methods. The chapter contains more detailed information about the selection and suitability of analysis filters.

- **Chapter 8: Analysis** is now divided into three sections: the first one is concerned with so-called standard analysis with gravimetry and/or light-optical analysis (microscope and new flatbed scanner). Conventions for systems and settings have now been fixed to improve the comparability of analysis results. Width has been added as a new feature for characterizing particles, and a standardized criterion for defining fibers is now also included.

  The second section describes the extended analysis method (process optimization, cause research), which can be used to identify materials or determine 3-D geometric shapes (only to be used to validate cleanliness specifications if specifically arranged because the effort and costs involved are significantly higher than with standard analysis). As well as SEM/EDX analysis, which was already discussed in the first edition, other methods such as LIBS, Raman and IR-spectroscopy, light-optical determination of particle height and microcomputer tomography can now be found.

  The third section deals with the shortened analysis method that does not include a filtration step. This enables analysis results to be obtained much faster and may prove useful in cases where monitoring is data-intensive. Liquid particle counters are discussed here again, together with a new system based on the optical recognition and measurement of particles occupying an analysis filter in the media flow. Due to the diverse detection principles, the results obtained from a shortened analysis cannot be directly compared with those of light-optical standard analysis. They are more intended to be used to validate cleanliness specifications if this is explicitly stated in the customer-supplier agreement.

- All the information and tables for documenting cleanliness specifications, inspection specifications and inspection reports that were listed in the appropriate chapters in the first edition of VDA 19 are now grouped together in **Chapter 9 Documentation**. This gives the new edition a clearer structure and makes it easier to read.

The particle size classes for documenting specifications or test results have now been increased by three sizes from the previous 1000 µm um to 3000 µm.

- The new **Chapter 10 Interpretation and reaction** describes the procedure to be taken when the result of a cleanliness analysis exceeds the permissible value stated in the cleanliness specification.

- **Work safety and protection of the environment** is addressed in a new **Chapter 11**, which especially considers the aspect of cold cleaners that are generally used for the extraction process. The chapter should make it easier for operators of cleanliness laboratories to make structured risk assessments for tasks carried out in the laboratory.

- **Chapter 12: Case examples** has been completely redesigned and uses examples to show how to qualify, perform and document cleanliness inspections. On the basis of practical examples, the chapter aims to give especially newcomers to the field of inspecting technical cleanliness a better understanding of the numerous topics contained in the guideline, and facilitate their implementation.

- **Chapter 13 definitions, abbreviations and equation symbols, and Chapter 14 TecSa industrial alliance** have been updated. The **Bibliography** chapter has been deleted.

Due to the restructuring and extension of the revised second edition of VDA 19, the guideline can be utilized not only to design, perform and document cleanliness inspections in the laboratory but is also suitable for use in other disciplines such as quality assurance, construction and development or supplier management to give a brief introduction to the subject. We recommend reading Chapters 2 and 3 to obtain a rough overview of technical cleanliness and understand how to inspect it.

1 SCOPE OF APPLICATION AND VALIDITY

1.1 Introduction

The guideline describes requirements for applying and documenting methods for determining particulate contamination on functionally-relevant components (cleanliness inspection).

A cleanliness inspection forms 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

- Quality control or monitoring of manufacturing processes relevant to cleanliness (e.g. cleaning, surface treatment and assembly processes).

The guideline enhances the informative quality and comparability of test results; it also regulates the standardized expression of cleanliness specifications and cleanliness test results in the quality chain of the automotive industry.

Information concerning references:

If an inspection of technical cleanliness has been carried out in accordance with this guideline, the following reference is made in such documents as catalogues, specifications and reports:

"*Standard cleanliness inspection according to VDA 19.1*" 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 VDA 19.1*" 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 VDA 19.1*" in cases where shortened analysis methods are implemented and whose application, pa-

rameterization and documentation are defined within the scope the customer-supplier relationship.

1.2 Exclusions

The following points are not discussed:

1. Detection of filmy contamination (grease, oils, etc.)
2. Application of non-quantifiable particulate detection methods on test components (e.g. visual assessment, wipe test with clean cloth, etc.)
3. Characterization of operating fluids (fuel, oils, coolants, brake fluid, etc.), see VDA 19 Part 2.

Moreover, the guideline does not state any concrete 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 should only be undertaken by specialists who not only know the component concerned but also the system it is built in to, the later conditions of use, technically-feasible practices and possible consequences for manufacturing processes and the supply chain. Tips for deriving limit values can be found in the guideline in Chapter 2: Cleanliness specifications.

1.3 Cleanliness inspection

The purpose of a cleanliness inspection is to detect as best as possible the particulate contamination generated by the manufacturing process 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 a sampling 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 analyze technical cleanliness, first of all a so-called extraction (or sampling) step is necessary. Here, the particles present are first removed from the component by means of a laboratory cleaning process. The complete extraction medium 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).

Due to the fact that in some cases a single particle may be functionally critical, it is essential that the maximum number of particles present on the surface are removed during the extraction step. As real test components with a defined level of contamination do not exist to enable the efficacy of extraction procedures to be assessed, extraction procedures first have to be qualified before technical cleanliness can be assessed:

Qualification of the extraction procedure: the appropriate extraction procedure for a test component is determined by performing qualification tests. The qualification criteria are stated in this guideline. They serve to ensure that as many particles are removed from the test component as possible. The qualification results are then documented together with the general requirements and parameters of the extraction procedure chosen.

Blank value: 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 foreign particles permitted in a test is stated in the guideline. This so-called permissible blank value is orientated towards the cleanliness requirements of the respective test component.

The cleanliness inspection (see Fig. 1-1) is made up of the following steps:

1. Receipt of the test component

2. Removal of the particles from the test component (extraction)

3. Filtration of the particles (except with shortened analysis)

4. Analysis of the particles

5. Documentation of the test and results

In order to perform technical cleanliness inspections, special equipment, suitable premises and skilled, experienced and motivated staff are required.
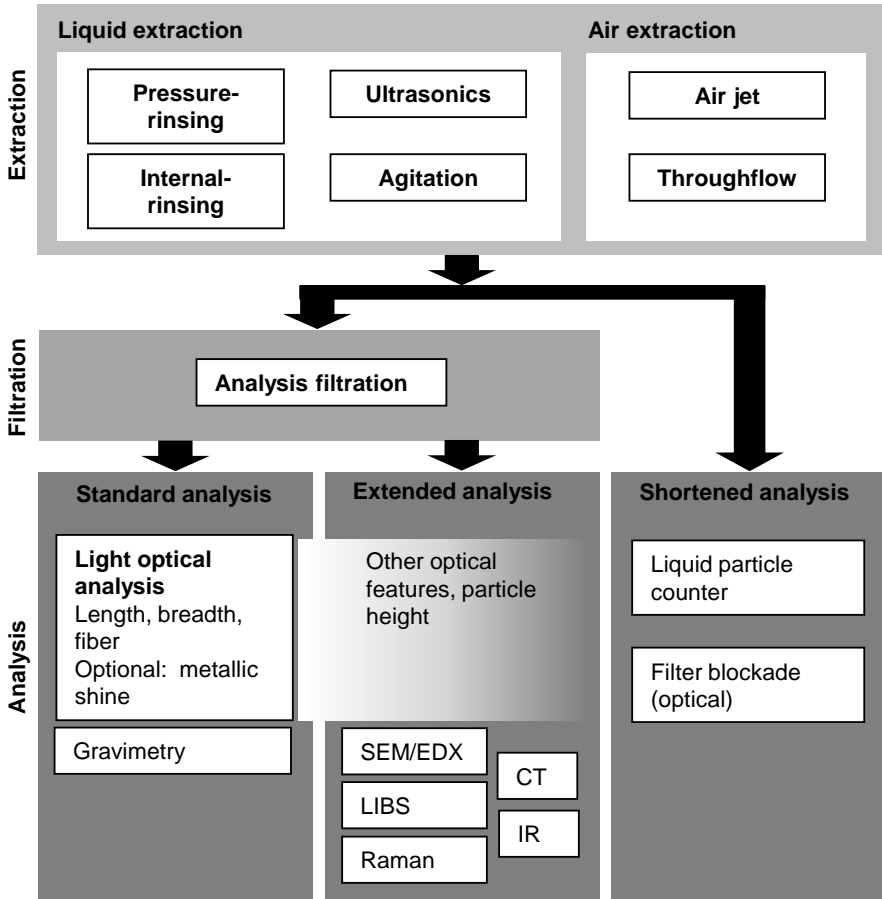
Fig. 1-1: Inspection methods according to VDA 19.1

2 CLEANLINESS SPECIFICATION (INFORMATIVE)

2.1 Principles

A cleanliness specification (or cleanliness limit value) is stated in cases where there is a significant risk that the function of a system or component could be damaged by particulate contamination. If no risk is present, a cleanliness specification should not be made.

In general, cleanliness limit values represent control limits and not tolerance limits. If a limit is exceeded, this does not necessarily lead to a malfunction although it does increase the risk. The probability of a malfunction occurring also depends on the system concerned (geometry, airflow patterns, cross-sections modifying with time, or similar).

The achievable level of cleanliness for a component or system is principally determined by its design. Before establishing cleanliness limit values, the system concerned should first be inspected to assess whether particle compatibility (robustness) can be increased, for example by channeling particles or reducing their migration within the system (by using centrifugal effects, installing a filter, etc.). Another way of realizing technically feasible and cost-effective cleanliness limit values and is to reduce particle generation in the manufacturing process. This can already be attained by selecting suitable materials and coatings for components and systems, by considering cleanability aspects in their design and by reducing the formation of chips and burrs during machining processes.

Technical cleanliness is not a constant feature, such as the diameter of a borehole, but rather a temporary state that can be influenced by external factors. In the customer-supplier relationship, it should therefore be stipulated when and where a cleanliness specification applies. This can be fixed via generally-applicable supply conditions, or be defined for each case individually (e.g. after cleaning, on delivery to the assembly area, etc.).

Any additional arrangements require verification to ensure that they do not contradict with the agreed cleanliness specifications. This may concern a suitable form of packaging, or the person responsible for cleaning reusable packaging; however, there may also be a need to assess storage locations and times, or the suitability of the assembly processes used with regard to cleanliness.

2.2 Deriving cleanliness limit values for systems

In many cases, fluid systems (liquids and gases) often contain one or more components sensitive to particles (see annex for examples of damaging mechanisms). As particles inside such systems are generally mobile, this "weakest link in the chain" usually determines the degree of cleanliness necessary for the whole system. Examples of typical fluid systems found in the automotive industry include:

- Fuel systems
- Lubricating and hydraulic oils (engine and gearbox)
- Braking systems
- Steering
- Air-conditioning and cooling systems
- Air intake systems and exhaust tracts
- Systems for post-treating exhaust gases

Note: Components may also form part of more than one fluid system. In such cases, different cleanliness specifications may apply to different sections of the same component, with the consequence that each section may need to be sampled individually in the cleanliness inspection.

The first step towards deriving a cleanliness specification is to identify the areas which are sensitive to particles, e.g. with the aid of media circulation plans. The aim is to characterize functionally-critical particulate contaminants as accurately as possible, with regard to:

- Damaging mechanisms/damaging effect (e.g. blocking, jamming, electrical shorting, etc.)
- Particle size (relevant dimensions)
- Particle count/quantity
- Material properties/critical particle materials
- Probability of a defect occurring, e.g. during a changeable state such as a valve closing
- (temporary) local concentration in the system

Where possible, the points listed in the left-hand section of Fig. 2-1 are to be taken into account.

In a next step, the requirements determined have to be examined to find out if they can be fulfilled using state-of-the-art technologies on taking the sub-points listed in the right-hand section of Fig. 2-1 into account.

Only after taking both sections into consideration (necessity and feasibility), can a meaningful cleanliness specification be drawn up for the respective system.
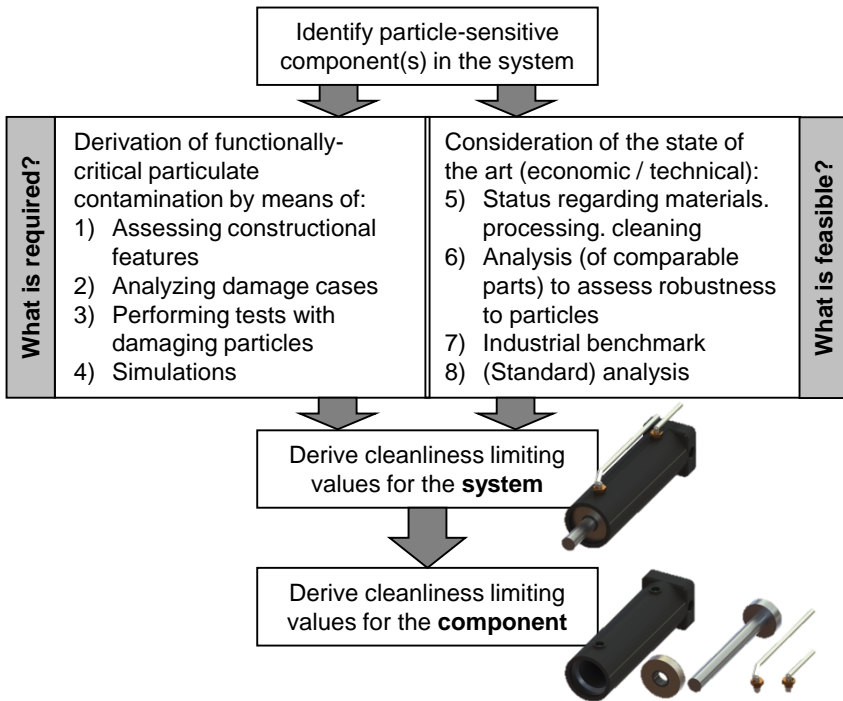
Fig. 2-1: Procedure for deriving cleanliness specifications

About 1) Assessing constructional features. In a first step, the geometric particle size that could be functionally-critical for a component in the fluid system is determined based on the system's constructional features, e.g. gap dimensions, valve stroke, filter mesh size, etc. At the same time, the direction of flow and orientation of particles in the flow is also considered. Where surfaces move against one another, e.g. sliding bearings, the degree of abrasion may be taken into account. With valves that open and close, the shearing action of particles can be considered, and in the case of electronic systems, conductivity may be relevant.

About 2) Analyzing damage cases. If experience has already been obtained with the system (or a similar one) either in practice, in test benches or ongoing serial runs, for example, the functional impairments that arose can be taken into consideration when defining the specification. Particles that caused damage can be isolated after disassembly, for example, or be visualized by computer tomography in order determine their size, geometry and material.

About 3) Testing for critical particles. In order to assess a system's ability to withstand damaging particles, tests can be performed in which the system is deliberately operated with such particles and the effects analyzed. For example, particles can be injected into the system whilst being operated under realistic conditions (pressure, temperature, revolutions, etc.) or (with heat-soluble liquid grease, etc.) before it is assembled. In such tests, the sizes, types and counts of particles can be determined which cause a system to fail completely, be irreversibly damaged or shorten its service life. This can be done by monitoring test bench parameters such as oscillations and pressures, or by inspecting component surfaces after dissembling the test system.

Test components or systems can also be built from transparent materials to visualize particle migration or accumulation in order to reach conclusions about damage risks.

About 4) Carrying out simulations. A further method of evaluating the ability of fluid systems to withstand particles is to use modified software programs to simulate particle-charged flows. This enables particle transport to be simulated and visualized.

Note: The construction of test benches or modification of software programs to assess resistance to damaging particles varies greatly for each product and may involve major technical and financial outlays.

About 5) Current materials, processing and cleaning methods. Due to the types of materials and processing methods used in automotive manufacturing and the fact that components are produced under high cost pressure in extremely short cycle times, it is not economically feasible to attain levels of cleanliness that are typical for other industries (e.g. semiconductor industry or medical technology). For example, a machined cast aluminium component manufactured in three-shift operation cannot be cleaned as thoroughly as a polished stainless component manufactured in the medical device industry. If such facts are not taken into account, it is often impossible to reliably adhere to a cleanliness specification despite major cleaning and

post-treatment efforts (see graph in the annex). Before fixing final cleanliness specifications, the state of machining and cleaning technology should be known.

Note: With current technologies, it is impossible to realize cleanliness limit values for compact particles in automotive fluid systems in the two-digit micrometer range (e.g. no particles > 50 µm permitted). The presence of (textile) fibers is still also to be reckoned with.

About 6) Analysis of particulate loads (comparable parts). In order to gain a rough idea about the level of technical cleanliness that can be attained with current manufacturing methods, cleanliness analyses according to VDA 19 can be performed on similar or comparable components. However, comparable manufacturing and cleaning processes need to be implemented and logistics processes and environmental conditions also have to be similar. These analyses may also identify any existing potentials for improving cleanliness levels.

About 7) Industrial benchmark. In the automotive and supplier industries, in order to obtain relatively standardized cleanliness specifications for comparable components with comparable conditions of use in a vehicle, it may make sense to reach a cross-company agreement on these limit values. For example, the supplier could agree to provide a standardized level of technical cleanliness for specific product groups, or the customer may demand a standardized level of cleanliness for comparable parts, thus enabling supplier processes and costs to be planned.

About 8) Analysis. Using the analysis method described in VDA 19.1, it has to be possible to validate the cleanliness specifications derived from Points 1 to 7. Even the best-founded cleanliness specification makes no sense if the supplier is unable to validate it using state-of-the-art technical equipment or if costs are excessively high. If no special arrangements have been made between the customer and the supplier, cleanliness specifications are evaluated using the standard analysis described in VDA 19.1.

2.3 Deriving cleanliness limit values for components

When deriving cleanliness limit values for components from cleanliness limit values for systems, a differentiation is made between the following:

1) The cleanliness limit value for a system is expressed as a rejection criterion, e.g. no particles larger than X μm (not including fibers) are permitted in the system. In this simple case, the cleanliness specification applies to all components relevant to the system (component surfaces coming into contact with media).

2) A specific quantity of contamination is permitted for the overall system, e.g. a gravimetric value or a particle size class. This total quantity of contamination is the sum (i=1-n) of the contaminants on the components forming the system, i.e. the sum of the limit values of the components i is the limit value of the system (see first line in Fig. 2-2). As large components are generally responsible for more contamination in a system than small ones, in a first step the amount of contamination in the system can be divided among the components in relation to their surface area (in contact with media) (see second line in Fig. 2-2). In practice, it makes more sense to apply a modified surface reference. Here, to start with, the surface reference remains the same, i.e. individual component limit values LV_{comp} are calculated from the total limit value LV_{total} divided by the total surface area SA_{total} of the system and then multiplied by the surface area of the component SA_{comp}. With this modified approach, individual component limit values are scaled by weighting factors A_i whilst keeping the system's total particle count the same. This enables information regarding cleanability or other facts relevant to cleanliness to be included in the scaling process. As a result, components such as small low-cost parts which cannot be cleaned cost-effectively, complex components difficult to deburr, or coated parts are assigned weighting factors $A > 1$ (i.e. higher component limit values) and large components, which are easy to clean and deburr, are assigned weighting factors $A < 1$ (i.e. lower component limit values). Further factors relevant to cleanliness can also be integrated into overall system cleanliness by including additional summands $B, C, etc.$, for example the particulate load of an oil poured into the system, or the amount of contamination generated during assembly (see third line in Fig. 2-2).
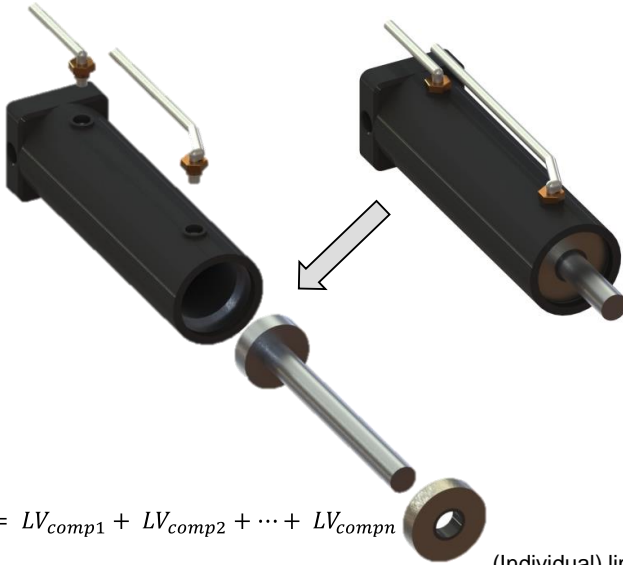
$$LV_{tot} = LV_{comp1} + LV_{comp2} + \cdots + LV_{compn}$$

(Individual) limiting value for Component *n*

$$LV_{tot} = \frac{LV_{tot}}{SA_{tot}} * SA_{comp1} + \cdots + \frac{LV_{tot}}{SA_{tot}} * SA_{compn}$$

$$LV_{tot} = A_1 * \frac{LV_{tot}}{SA_{tot}} * SA_{comp1} + \cdots + A_n * \frac{LV_{tot}}{SA_{tot}} * SA_{compn} + B + C + \cdots$$

LV_{total}: total limit value of the system

SA_{total}: total surface area of the system (in contact with media)

$LV_{Comp\ i}$: limit value of Component *i*

$SA_{Comp\ i}$: surface area of Component *i* (in contact with media)

A_i: component-specific weighting factors

$B, C, ...$: further additive quantities of contamination

Fig. 2-2: Deriving cleanliness specifications for components

2.4 Consequences drawn from cleanliness limit values

2.4.1 Inspecting technical cleanliness in quality control

When specifications for the technical cleanliness of components are arranged between the customer and the supplier, they have to be validated in accordance with the agreement. The following general principles apply when inspecting technical cleanliness:

- Special equipment is required, e.g. for the extraction and analysis procedures.

- An inspection can be compared to a laboratory test, i.e. suitable clean facilities are necessary.

- Inspections should always be performed by skilled members of staff (laboratory experience or training with a laboratory background is advisable).

- Frequent staff changes should be avoided because the results of cleanliness analyses are highly dependent upon staff experience and consistent inspection procedures.

- As some cleanliness inspections are time-consuming and involve manual tasks, much fewer of these inspections can be carried out compared to other quality inspections (e.g. reproducibility) (see also example of inspection costs in the annex). Consequently, established quality control methods cannot be applied indiscriminately to cleanliness inspections.

- The technical cleanliness of components fluctuates much more than other technical features (see INTERPRETATION and REACTION in Chapter 10)

- Technical cleanliness is a state that changes in dependence on time and is influenced by a wide range of factors. Therefore, the exact point in time and location of a cleanliness specification should be defined, as well as the procedure for removing the component from the production line and transporting it (together with packaging) to the inspection site.

Note: A technical cleanliness inspection does not necessarily have to be performed in a cleanroom. The cleanliness grade of the test environment depends on the blank value determined for the inspection, which in turn depends on the permissible cleanliness limit value for the component (see Chapter 5: QUALIFICATION TESTS AND BLANK VALUE).

2.4.2　Impact on production

Cleanliness specifications should always be derived on bearing in mind the aspects of technical feasibility and economic viability. High cleanliness demands can lead to immense costs regarding the manufacture and further processing of components along the complete supply chain. For example, higher costs can be incurred due to:

- The selection of materials or surface coatings
- Processing and deburring
- Cleaning (see also Annex 2.2)
- Logistics
- Manufacturing environment
- Staff
- Assembly equipment and joining processes.

Therefore, when deriving a cleanliness specification, only the level of cleanliness required for the component to function correctly should be specified and not more (as clean as necessary, not as clean as possible!).

Comments, information and methods regarding the consideration of assembly equipment, logistics, staff and environment from the aspect of cleanliness can be found in the guideline VDA 19 Part 2 "Technical Cleanliness in Assembly".

Annex 2   Cleanliness specification

A 2.1   Examples of damage mechanisms

Jamming of bearings
or sliding surfaces

- − Turbochargers
- − Crankshaft
  bearings
- − Dispensing
  pumps
- − Cylinder linings
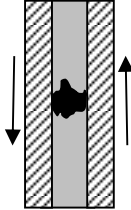
Jamming of valves

- − Anti-blocking
  systems
- − Hydraulics
- − Brake servos
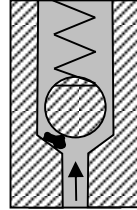
Blockage of nozzles
or filters

- − Injectors
- − Fuel lines
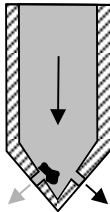
Shorting of contacts

- − Control
  electronics
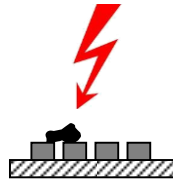
A 2.2   Rise in cleaning costs as cleanliness limit values increase
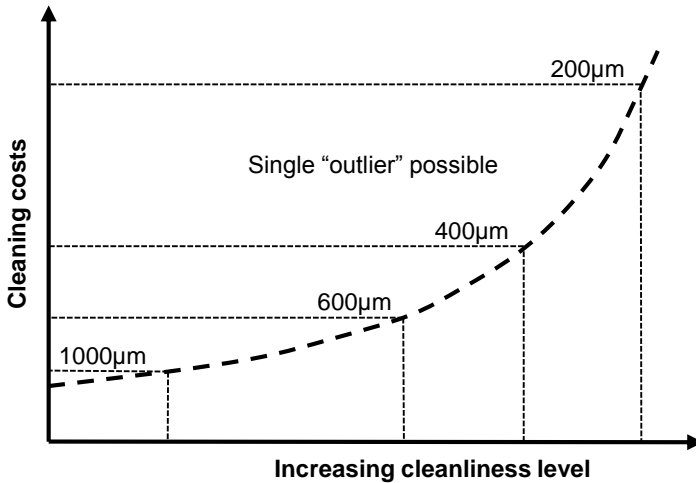
Factors affecting rising cleaning costs include:

- Higher new investments
- Bath care/filtration
- Longer cleaning times
- Lower filling quantities
- Better quality washing frames/baskets
- Maintenance
- Environment
- Trained staff

A 2.3   Example of deriving a cleanliness specification

Note: This is not a real example. Particle sizes and counts, as well as observations made and conclusions drawn, are purely exemplary and could be totally different in practice. The example only serves to illustrate how a cleanliness specification can be derived on taking several different aspects and disciplines into account.

The component in the example is an electronic subassembly fitted with contacts.

Development:

Malfunctions may occur if conductive particles bridge and short the contacts on the subassembly.

The distance between the contacts of this new product is 400 µm.

⇨ With this information, the developer initially derived the following cleanliness specification: no conductive particles ≥ 400µm permitted for the component

Consultation with quality assurance:

Malfunctions caused by conductive particles also occurred sporadically with similar subassemblies in the past. Metallic particles > 600 µm were found in the cleanliness analyses.

It should always be possible to validate a cleanliness specification using a standard analysis method because the component concerned may be manufactured at a location unable to perform extended analysis methods.

Consultation with production:

A new cleaning system was purchased to clean the components. The cleanliness specification is defined in such a way so that fluctuations in cleanliness levels, and thus the state of the cleaning system, can be determined if cleanliness inspections are performed on a regular basis.

Action taken by quality assurance:

Cleanliness levels of similar components manufactured with the same processes were analyzed. To do this, extended analysis methods were also implemented in the company's central laboratory to obtain additional information about materials and 3-D shapes. The following was determined:

- Not only metallic particles were found but also non-metallic particles from the housing material that, due to their geometry, only caused a malfunction in the installed relay if they were approx. 1000 µm or larger in size. The cleanliness specification should therefore take this fact into consideration.
- The metallic particles generated during the manufacturing process (solder beads, stamping burrs from plug connectors) were shaped in such a way that, despite a measured $Feret_{max}$ length of sometimes over 400 µm, their complete length never touched the surface and thus did not cause shorting. Only particles upwards of 600 µm represented a shorting hazard.

Action taken by development:

Test series were carried out to subject the subassemblies to particles of different sizes, different metals and originating from different production processes (e.g. on a vibration tester to simulate running conditions). The electrical function of the components was evaluated simultaneously.

The results showed that malfunctions repeatedly occurred due to metallic particles. The size of the metal particles responsible for this depended on the type of particle concerned. Thus, aluminium particles only started to cause problems upwards of 1000 µm (possibly due to the oxide skin on the surface). Tin chippings already caused malfunctions if they had a size of 600 µm or more.

The cleanliness specification

After taking into consideration the three areas of development, quality assurance and production, the following cleanliness specification was drawn up based on these requirements and this information:

Particle Size Class	All particles (excluding fibers)	Metallic shiny particles
x > 1000 µm	0	0
600 < x ≤ 1000 µm	2	0
400 < x ≤ 600 µm	6	2
200 < x ≤ 400 µm	20	8
150 < x ≤ 200 µm	40	Not specified
100 < x ≤ 150 µm	60	Not specified
50 < x ≤ 100 µm	150	Not specified

The following points are taken into account in the cleanliness specification:

- Textile fibers not regulated because they are not critical to function. However, they have to be identified in the analysis in order to separate them from other particles.

- Particles upwards of 50 µm are specified because they can be tested using the standard analysis procedure.

- Particle size classes with smaller particle sizes (< 150 µm) are used to recognize fluctuations in the manufacturing process (cleaning system) at an early stage; this is not possible with large particles because of their scarcity.

- No particles larger than 1000 µm are permitted because these are considered to be functionally-critical, even if they are non-metallic.

- No metallic shiny particles larger than 600 µm are permitted because the tests were unable to confirm that the originally-derived size of 400 µm is functionally-critical.

- Using light-optical standard analysis, it cannot be concluded without a doubt that metallic shiny particles are conductive (further material analyses would be required to determine this). Nevertheless, metallic shine can be utilized as a typing indicator if the automated analysis is subsequently double-checked by a skilled inspector.

- As the smaller particles (< 150 µm) are used to monitor trends rather than characterize the damaging potential of individual parti-

cles, these size classes are not typed (metallic shine) in the specification. This makes sense because the cost of manually confirming particle types rises as the number of smaller particles increases. Moreover, typing becomes less reliable as particle sizes decrease, and it the comparison of different light-optical analysis systems also become more difficult.

3 SELECTING THE INSPECTION METHOD (INFORMATIVE)

A cleanliness inspection is made up of three steps:

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

- 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.

- Particle analysis: relevant particle features are analyzed 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 so-called standard analysis procedure is to be applied.

Note: In the so-called shortened analysis (see diagram of the structure of VDA 19.1 in Chapter 1), there is no filtration step: Once detached by the test medium, particles are advanced directly for analysis, e.g. with a liquid particle counter.

3.1 Selecting the extraction method

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 (Fig. 3-2), the following parameters influence the extraction result:

- **test medium** (chemical cleaning aspect)

- **temperature**

- **cleaning mechanism** (extraction method with parameters)

- **time** (time the component surface is exposed to the extraction method)

A particle bonded to a component surface by forces of adhesion serves as a model for the cleaning task to be solved. The particle could adhere, for example, because of van of the Waals forces, or alternatively capillary forces subsequent to the presence of humidity in the air or process media

(coolants, lubricants or anti-corrosives). The task of the extraction method is to overcome these forces and detach the particle.
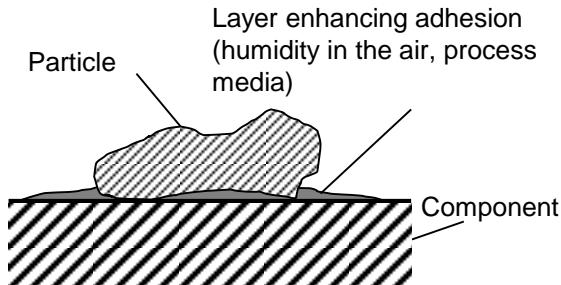
Fig. 3-1: Particle bonded to the component surface by forces of adhesion

Note: Burrs may become detached from the component during the extraction procedure, which are then also included in the cleanliness analysis. To avoid this, only components that have been carefully deburred beforehand should be analyzed. If this is not possible, an agreement must be reached to determine the procedure for analyzing and documenting detached particles.

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 room for maneuver regarding the specific parameters that can be chosen, e.g. diameter of the spray nozzle, volume flow, ultrasound frequency, etc. In order to obtain comparable extraction (and thus also analysis) results, it is advisable to commence qualification tests using the VDA 19.1 start parameters recommended for the various methods. See Chapter 6: Extraction Methods.

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

3.1.1 Test medium

Liquid extraction (extraction liquid as a cleaning medium)

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
- Cleaning agents
- Joining aids
- Operating fluids.

For all components coming into contact with these fluids, an extraction procedure with a liquid is recommended. The primary task of the extraction liquid is to overcome the adhesive forces prevailing between the particles and the component. Tests have to be performed to determine which liquid is best suited for detaching the contaminants without attacking the component surface. Table 6-1 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 cleaning liquids: tensidic aqueous neutral cleaning media and non-polar solvents. In the case of the latter, particular care must be taken with regard to work safety and protection of the environment (odorless isoparaffins, which are non-combustible at room temperature, are preferable).

Recommendations for selecting the extraction liquid:

a. Check whether aqueous neutral cleaning media 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 must be selected, e.g. ethanol (polar solvent) to clean brake components before their analysis.

The extraction liquid must be compatible with the component material (see Table 6-2).

Note: In cases where steel components are returned to the production line after a cleanliness analysis, they generally need to be treated with an anti-corrosive (applied with cleaning medium or separately after extraction).

Air extraction

Some components requiring a cleanliness inspection do not come into contact with fluids 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.

3.1.2 Temperature

Test media 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.

3.1.3 Mechanisms

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 (volume flow, ultrasound output, etc.) for the extraction.

Selecting the cleaning mechanism

This is essentially determined by the size and geometry of the component as well as the location of the test 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 also Annex 3).

Liquid extraction

Four different extraction methods can be utilized for liquid extraction – pressure-rinsing, ultrasonics, internal rinsing and agitation.

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 values, high operator influence due to largely manual procedure.
Note:	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.

Ultrasonics: ultrasound 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 ultrasound-permeable baskets in baths specially constructed for cleanliness analysis with an outlet to analysis filtration, or in beakers that are placed in standard laboratory ultrasound baths. To compensate for inhomogeneities in the ultrasound field, components should be moved gently in the bath. Movements should have an amplitude of at least half the wavelength of the ultrasound waves used.

Where ultrasound baths are utilized to clean the inner surfaces of components, it must be verified that the cleaning effect in the interior is adequate. In general, this becomes more and more difficult the smaller the aperture becomes for the ultrasound 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, possible to achieve low blank values 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 ultrasound bath (distribution of ultrasound 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.

- 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 extraction 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 test surfaces concerned by adapting rinsing lines, low blank values 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: 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 apparatus) 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 extraction medium cannot be sufficiently agitated to remove particles effectively.

Advantages: Simple, very cheap, low blank values 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 (weight of test object + extraction liquid).

Note: 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 foreseen in VDA 19 (risk of component abrasion).

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 throughflow extraction where air flows through the test component, which is comparable with internal rinsing as a form of liquid extraction.

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 throughflow method cannot be applied, or for logistics packaging such as blister packs, small load carriers or cardboard.

Advantages: Closed chambers for liquid extraction procedures can be easily and cheaply converted to chambers 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 values are difficult to attain due to the large internal surfaces of the extraction apparatus, high operator influence because the procedure is generally carried out manually.

Air throughflow extraction: with this type of extraction, air is usually guided through the test piece using specially-constructed test apparatus. 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 centimeters 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 medium leaves no residues in the component.
Disadvantages:	Complex test apparatus, need for component-specific adapters, time-consuming execution due to the preparation of primary analysis filters for air which then have to be manually re-prepared as secondary analysis filters for liquid.

Cleaning mechanism parameters

The intensity of the extraction effect and even 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.

3.1.4   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 - so-called declining tests (see Chapter 5: Qualification tests and blank value). Even in cases where the volume of extraction liquid is used for validation purposes, the actual parameter that is qualified is the length of time the extraction method acts on the component surface.
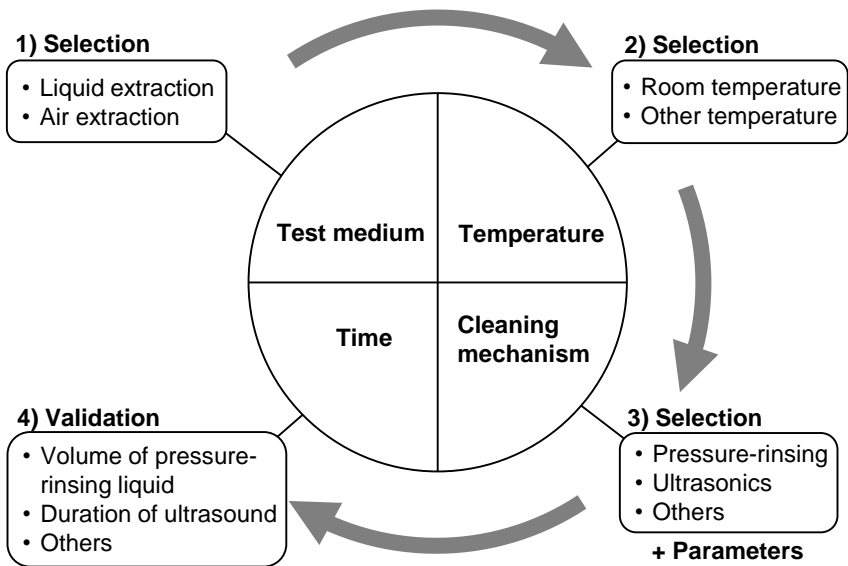
Fig. 3-2: Procedure for selecting the extraction method

3.2 Selecting the 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 extraction 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 may be necessary to verify whether the analysis filter and analysis method are compatible with one another (e.g. if high radiation levels are used).

3.3 Selecting the analysis method

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:

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
- 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, this method is 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 at on a regular basis.

3.3.1 Standard analysis

Used to validate cleanliness limit values in the customer-supplier relationship. The two following analysis methods are used in a standard analysis procedure:

Gravimetry:

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

- fiber shape
- metallic shine (optional)

In order to implement light-optical analysis methods, skilled staff are required. They must be able to operate the equipment and also be prepared to 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 analysis, the analysis is no longer considered to be standard and the respective points have to be agreed on and documented in the customer-supplier relationship.

3.3.2 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.

3.3.3 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.

The following tables list the analysis methods described in this chapter and their fields of application.

Table 3-1: 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	++ *)	+	+
		3-D	- *)	++	--
	Computer tomography		- *)	++	--
	SEM/EDX		0 *)	++	-
	LIBS		0 *)	++	-
	Raman		0 *)	++	-
	Infrared		-*)	++	--
Shortened analysis	Liquid particle counter		0 *)	-	++
	Filter-blocking (optical)		0 *)	0	++

*) Can be applied if arranged in the customer-supplier relationship provided analysis parameters are specified

++ highly suitable

+ suitable

0 suitable in principle

- less suitable, or not available for automated analysis, or too expensive for the intended purpose (if new equipment has to be purchased)

-- unsuitable, not available for automated analysis, too expensive for the intended purpose (if new equipment has to be purchased)

Table 3-2: Analysis methods, information obtained and limits

Method	Overall particle mass	Length	Width	Height	Metallic shine	Material	Limitations
		Particle size			Other features		
Gravimetry	x						Gives no information about single particles
Scanner		x	x		(x)[2]		Can only be verified on the image, greater optical magnification or other contrast methods not possible
Light microscope		x	x	(x)[1]	(x)[2]		Not all features usable with one lens
Computer-tomography		x	x	x[5]			Prolonged analysis times
SEM/EDX		x	x			x	No differentiation between organic/inorganic particles
LIBS		x[3]	x[3]			x	No differentiation between organic/inorganic particles
Raman		x[3]	x[3]			x	Metals not detected, limitations with fluorescing and black particles
Infrared		x[3]	x[3]			x	Only organic particles, no fully-automated analysis
Liquid particle counter		x[4]					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

[1] Possible with material microscopes with small depth of field (focusing position between background of analysis filter and highest point of particle)

[2] Pre-requisite: manual double-check

[3] Material analysis coupled with automated light-optical particle detection

[4] Size often determined by the diameter of a circle with an equivalent surface area

[5] Here, all size information can be determined (volume, thickness, cross-section, etc.)

Annex 3   Selecting the inspection method

A 3.1   Liquid extraction: example illustrating the suitability of component groups for the various extraction methods

External component surfaces

Single parts. e.g.
• Conrods
• Pistons
• Toothed wheels
• Housing parts
• Crankshafts
  (exterior)
• …

Small bulk goods, e.g.
• Ball bearing beads
• Washers
• Screws
• …

Ultrasonics

Pressure rinsing

Internal component surfaces

Large openings, accessible internal areas
(use pressure-rinsing lances)

Few. easy-to-seal openings.
simple cavities, e.g.
• Compressed air tanks
• Coolant containers
• Short fat pipes. etc.

Internal areas with good throughflow
properties and small cross-sections at
connection points, e.g.
• Tubing
• Filter housings
• Small heat exchangers
• Oil ducts (engine block, crankshaft)
• Etc.

Internal Rinsing

Agitation

Pressure rinsing

A 3.2 Liquid extraction: examples of component features and suitable extraction methods

Extraction method	Component features	Examples
Pressure-rinsing or ultrasonics	**Small components**, several millimeters to a few centimeters in size, difficult to handle individually, for extraction in bulk (total component surface area)	Ball bearings, gaskets, screws, springs, o-rings, etc. …
Pressure-rinsing	**Small to large components,** a few centimeters to over one meter in size (total component surface area)	Conrods, pistons, toothed wheels, housing parts, crankshafts, etc.
Internal rinsing	**Component areas with good throughflow properties,** with connector cross-sections and interior cross-sections sized between a few millimeters and several centimeters (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 centimeter 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: weight of component + extraction liquid)	Compressed air tanks, coolant containers, etc.
Agitation or internal rinsing	**Inner surfaces** of components with **good throughflow properties** and few easy-to-seal openings (cross-sections of apertures and inner areas in lower centimeter 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.

48

A 3.3 Air extraction: 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 throughflow extraction, packaging (SLC, blisters, cardboard), etc.
Air throughflow extraction	**Inner component surfaces with good throughflow 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

4   HANDLING COMPONENTS CLEANLY

4.1   Principles

Technical cleanliness is not a constant feature of a component but rather a temporary condition that is influenced by external factors. It should therefore be stipulated in the customer-supplier relationship when and where a cleanliness specification is to be performed (for example, after cleaning, on arrival at the assembly line or at another point in time).

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, sequences and conditions have to 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 performing such inspections must 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.
Detailed assistance on evaluating influencing factors and maintaining the cleanliness state of components is provided in **VDA 19 Part 2**.

4.2 Selected measures and recommendations

4.2.1 Staff

Responsibilities regarding the complete inspection process, from removal of the test component until its return, are to be defined.

Members of staff involved in the inspection are to be instructed how to handle test components cleanly.

4.2.2 Packaging

Packaging is to 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, leak proofness 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 must 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, must 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 is to be avoided.

4.2.3   Storage and transport

General requirements:

- 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.

4.2.4   Facilities for inspecting cleanliness

The test environment must 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 are to be handled and, if required, disassembled under environmental conditions conform to the cleanliness requirements.

The methods used to clean floors, equipment and workplaces and the frequency of cleaning processes depends 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 may not be required. It is only important that the blank value is reliably attained because this may also reflect possible environmental influences.

4.3 Exclusion from an inspection – invalid inspection

4.3.1 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. Recommended further action:

- Document the discrepancy or deviation from the means of packaging specified
- Inform all people involved before or after the inspection
- Label the component and proceed with relevant instructions, e.g. return or destroy 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 must be documented in the inspection report.

4.3.2 Deviations and errors in the inspection procedure

Any deviations to or reflected lights occurring during the actual cleanliness inspection are to be documented in the inspection report.

If there is a possibility that they may have falsified results, 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 words "inspection error" are written, possibly with an explanation or justification of the error.

5 QUALIFICATION TESTS AND BLANK VALUE

5.1 Principles

Qualification tests are carried out to refine the extraction method (routine inspection) and thus represent the final step in the procedure that started in Chapter 3.1 with "Selecting the extraction method". The qualification test is therefore the fourth element (time) in Sinner's Circle shown in Fig. 3-2. This step is carried out by means of a test - a so-called declining test – as illustrated in the diagram in Fig. 5-1.
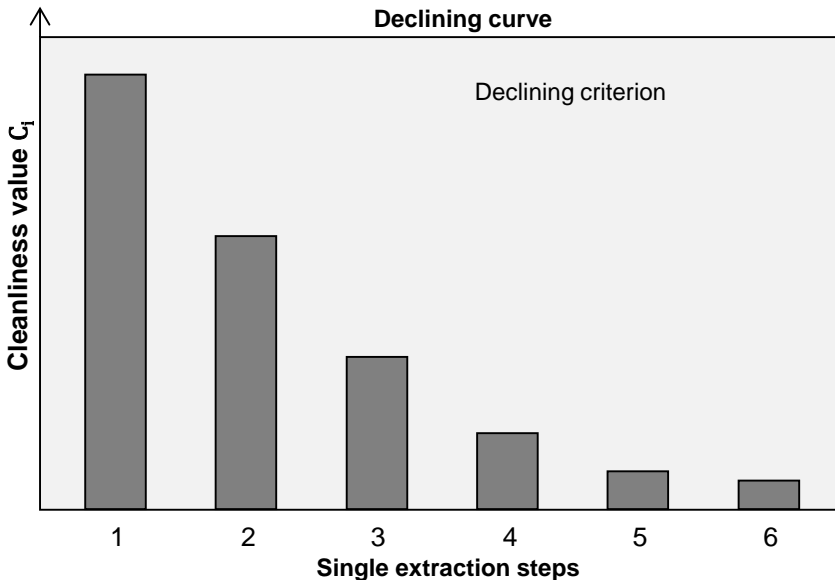
Fig. 5-1: Cleanliness value determined on sampling a component repeatedly (declining behavior)

Determining blank value**s** 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 introduced into the test procedure from sources other than the test piece may not unduly distort 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 of test particles** can be **optionally** determined.

Note 1: When validating cleanliness specifications in the customer-supplier relationship, extraction conditions must be qualified according to VDA 19.

Note 2: Even in the case of an extended or shortened analysis, a qualification test is recommended. This is because any form of analysis demands an efficient and comprehensive extraction procedure.

5.2   Qualification tests

5.2.1   Principle

In order to assess the cleanliness of a component correctly, an extraction method must be effective. Therefore, it must be proved that the maximum number 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, 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 with a defined state of contamination. 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 repeat 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. The so-called declining criterion (see Fig. 5-1) determines whether the contamination to be analyzed has been effectively detached. This identifies the point on the declining curve where the last value of extracted contamination is less than 10% of the total quantity of contamination extracted up till that moment. 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 par-

ticles to be detached from the component surface that are not relevant contamination (see also Annex A 5.2).

Note: 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 procedure must be repeated.

The qualification procedure comprises the following steps (Annex A 5.1):

1. Declining test (5.2)

2. Development of the routine inspection procedure (5.2)

3. Optional: validation of the routine inspection procedure by carrying out a double inspection

4. Determination of blank values (5.3)

5. Optional: test particle recovery (5.4)

If the blank value and declining behavior are in order, 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 procedure can be reduced to Steps 3 and 4.

Examples of Points 1 – 4 can be found in Chapter 12: Case examples.

5.2.2   Materials and equipment

Extraction parameters for the test medium, temperature and cleaning mechanism of the extraction procedure, filtration and analysis method are determined as described in Chapter 3: Selecting the inspection method.

Declining tests are analyzed using the analysis method that is also applied to validate the cleanliness specification.

At the beginning of the declining tests, start parameters appropriate for the respective extraction method are set and implemented - or more suitable parameters if they are justified and documented - (see Chapter 6: Extraction method).

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

Note: Qualification tests may never be repeated on a component which has already been analyzed.

5.2.3   Procedure

Declining test

Fig. 5-1 (see Section 5.1) shows an example of a declining curve obtained if a suitable extraction procedure is utilized.

Note: Ensure that the test component is delivered to the test laboratory in an appropriately clean state (see also Chapter 4: Handling test components cleanly).

1. Determine a test lot size estimated to be suitable

2. Clean testing equipment as required and validate the level of cleanliness by performing a blank value measurement.

Note: Because the later routine procedure has not been determined at this stage at the start of the qualification, the blank value cannot yet be determined using the final extraction volume or extraction time. The parameters used to determine the "provisional" blank value 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, a so-called "equipment blank value" is often known, i.e. a blank value that can be attained with the extraction apparatus using a defined procedure and volume of liquid or period of time. If the "equipment blank value" is used as the "provisional" blank value for the qualification procedure, it also has to fulfill the blank value criteria.

3. 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 apparatus must be subsequently rinsed using an effective final rinsing procedure after each extraction step.

4. Then, for each cleanliness value, calculate the ratio between the cleanliness value under consideration and the total of all cleanliness values excluding the current extraction step:

$$\frac{C_n}{\sum_{i=1}^{n} C_i} = declining\ value, Step\ n\ of\ declining\ test$$

5. If this declining value < 0.1, the declining criterion has been achieved.

$$C_n \leq 0.1 \sum_{i=1}^{n} C_i \ n \leq 6$$

6. Once the declining criterion has been attained, the routine inspection is developed (see next subtitle)

7. If the declining criterion is not achieved after six extraction steps, extraction parameters need to be adjusted accordingly, 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 attacked 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 size of the test lot.

8. If, despite optimizing the extraction parameters, the declining criterion still cannot be attained, the component is regarded as an exceptional inspection case (see Chapter 5.5).

Note: The six cleanliness values C_i obtained from each respective extraction step can be depicted in a graph as a declining curve, as shown in Fig. 5-1. Annex 5.2 includes a range of curve shapes that may be obtained in the course of declining tests and also describes methods for interpreting them.

To determine declining values and declining criteria (Point 4. and 5.), 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 weight.

  – 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 portrayal of declining behavior, all particle size classes included in the cleanliness specification can be added together. However, care is to be taken to ensure that a decline takes place in each particle size class.

  All **particles with the exception of fibers** are included in the declining test.

  If no cleanliness specification has been made, 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 Chapter 8.2.2 Light-optical analysis).

Note 1: Size classes in which at least 20 counting events are registered should be selected. Under certain circumstances, it may be necessary to consider particle size classes which are not included in the cleanliness specification.

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

Developing the routine inspection

From the information contained in Point 5.2.3, suitable routine test conditions are derived for the respective extraction method:

- Pressure-rinsing: the total pressure-rinsing time, equivalent to the total volume of rinsing liquid used if the flow rate is known

- Ultrasonics: the total length of time in the ultrasound bath

- Internal rinsing: the total rinsing time, which is equivalent to the total volume of rinsing liquid used if the flow rate is known

- Agitation: the total agitation time, which is equivalent to the number of agitation movements if the agitation frequency is known

- Air jet extraction: the total time the component is cleaned with the air jet

- Air throughflow extraction: the total time air flows through the component.

If **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.

Note: Die final rinsing procedure to remove particles from the extraction apparatus 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 (see 5.4) utilized, or example.

Once developed and qualified, the routine inspection is utilized to extract particles from components in order to validate the cleanliness specifications defined between the customer and the supplier.

Validating the routine inspection / double inspection (informative)

To optimally validate a qualified routine inspection procedure, a so-called 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 routine test conditions developed are suitable and can be documented as an inspection specification.

$$C_2 \leq 0.3(C_1 + C_2)$$

- If the criterion is not fulfilled, it should be investigated whether it is possible to adjust extraction conditions / extraction parameters appropriately.

5.3 Blank value

5.3.1 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 foreign particles are introduced into the test. If the proportion of foreign particles (blank value) is too high, this could lead to a faulty assessment of component cleanliness. The blank value represents the total value of contamination that does not originate from the component. Origins of such foreign particles include:

- extraction liquid and rinsing liquids

- extraction equipment (baths, basins, tubing, valves, etc.)

- handling processes during extraction and analysis

- environment and staff

- all objects coming into contact with the component and extraction liquid

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

Note 1: If the required blank value is not achieved, the cause has to be investigated among the above-mentioned influencing factors.

To ensure that the blank value does not have a decisive influence on the test result,

- it must be correctly determined and

- it may not exceed a specific maximum ratio as far as the re-quired/derived component cleanliness is concerned.

A blank value test is not necessary if a comparable test has been carried out immediately beforehand. However, a blank value test should 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 a high particle con-tent to a component with a lower particle content, because there is a major risk of transferring an impermissible quantity of particles to the clean component.

5.3.2   Deriving blank values

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

The blank value may not exceed 10% of the required/anticipated cleanli-ness values for the component. Further conditions apply for the blank value regarding the maximum permissible particle size (see Table 5-1).

Note: If the cleanliness values of a component are not known – e.g. on sampling for the
first time – the basis for calculating the blank values can only be attained after per-
forming the qualification test.

Table 5-1: Blank value criterion and examples

Cleanliness specification	Blank value criterion	Example			
		Cleanliness specification		Blank value derived	
Gravimetric	10% of permissible or measured residue weight*)	7 mg		0.7 mg	
Particle size distribution	10% of the quantity of permissible or measured particles in each size class (without exception, digits after the decimal point are to be rounded off)	Size channel [µm]	Permissible quantity	Size channel [µm]	Quantity permitted in blank value
		100 ≤ x < 150	90	100 ≤ x < 150	9
		150 ≤ x < 200	28	150 ≤ x < 200	2
		200 ≤ x < 400	6	200 ≤ x < 400	0
		400 ≤ x < 600	0	400 ≤ 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 µm ≤ x < 400 µm Therefore: no particles ≥ 200 µm permitted in the blank value	
None	No particles > 50 µm permitted **)	-		No particles > 50 µm permitted	

*) Take resolution of balance into account, see Chapter 8.2.1 for more details

**) Only applies if no information is available, such as no cleanliness specification, no analysis result from the component, no experience with similar components.

The blank value always refers to the concrete component inspection. In order to be able to calculate permissible blank values where cleanliness specifications are stated per surface area or wetted volume, or in the case of coded cleanliness specifications, the permissible quantity of contamina-

tion for the test lot analyzed (one or more components) must always be calculated first.

If different cleanliness features are combined in one cleanliness specification, e.g. residue weight and particle size distribution, the blank value is to be calculated and attained for all features specified.

If two possible blank values are determined, e.g. with a specified particle size distribution and a maximum permissible particle size, the more stringent blank value derived applies.

Note: When validating cleanliness specifications, it is not permitted to deduct the calculated blank value from the analysis result of a component cleanliness inspection.

5.3.3   Materials and equipment

To determine blank values, exactly the same equipment, materials, settings and test parameters are to be used in the extraction process as well as in the filtration and analysis steps.

5.3.4   Procedure

1. Calculate the permissible blank value either on the basis of the cleanliness specification or on the basis of the cleanliness values derived in Section 5.2.3

2. Perform exactly the routine inspection developed in Section 5.2.3 without a component and determine the cleanliness value (= blank value)

3. Check that the blank value derived is within the permitted limit.

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

Another reason why blank values 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 value test.

5.4 Recovering test particles (informative)

5.4.1 Principle

The blank value 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 apparatus 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
- – or training of inspection staff.

5.4.2 Material and equipment

For this procedure, as well as all the materials and equipment utilized for the derivation of blank values 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 apparatus 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 can be clearly identified in the analysis method. For example, the particles must be deposited reproducibly on an analysis filter to enable 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 analysis.

These particles thus correspond with particles actually occurring during production and should also be found in daily laboratory operation in the routine analysis.

5.4.3 Procedure

- Procure a supply of known test particles

- Introduce the test particles into the extraction apparatus

- Give the extraction apparatus a final rinse on completion of the specified procedure

- Proceed with analysis filtration, filter removal, filter drying and filter analysis

- Determine the recovery rate, compare it with the desired value:

    a. If specially-produced test particles are used, the recovery rate should be 100%

    b. 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.

- If the desired recovery rate is not achieved, the chain, i.e. final rinsing, filtration, filter handling and analysis procedure and/or equipment is to be optimized and the inspection with test particles repeated.

5.5 Exceptional cases

If it is proven that the qualification criteria for the declining and/or blank value 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.
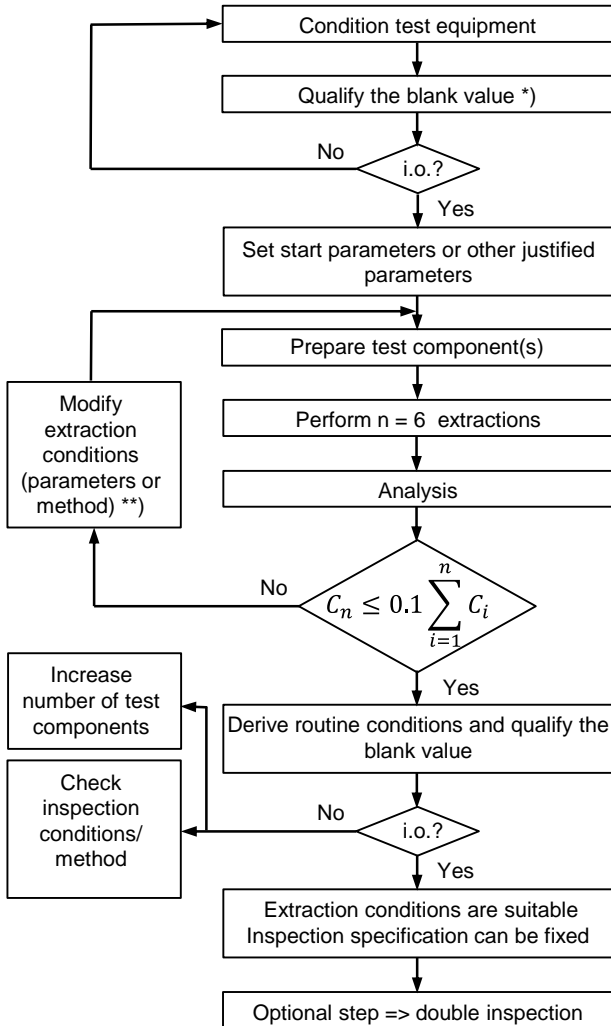
Note: Before a component cleanliness inspection is declared to be an exceptional case, firstly all possible causes of error are to be investigated and remedied, e.g. incorrect handling, particles transmitted from non-relevant component areas, etc.

The following may prove to be exceptional cases:

- Active component groups which have to be operated during extraction (e.g. valves, pumps, injectors, etc.). In such cases, detached contaminant particles and particles generated during running-in may overlie one another with the consequence that the decline in particulate contamination cannot be observed separately.

- Components that emit particles, e.g. from the component material or from sacrificial coatings.

- Extremely clean components, with which a qualification cannot be achieved despite increasing the test lot size and optimizing blank values.

Annex 5 Qualification tests and blank value

### A 5.1   Qualification test procedure

```
        ┌──────────────────────────────────┐
   ┌───►│     Condition test equipment     │
   │    └──────────────────────────────────┘
   │                    │
   │    ┌──────────────────────────────────┐
   │    │     Qualify the blank value *)   │
   │    └──────────────────────────────────┘
   │                    │
   │   No          ◄ i.o.? ►
   └────────────────    │  Yes
                        ▼
        ┌──────────────────────────────────┐
        │  Set start parameters or other   │
        │      justified parameters        │
        └──────────────────────────────────┘
                        │
   ┌────────────────────┤
   │    ┌──────────────────────────────────┐
   │    │     Prepare test component(s)    │
   │    └──────────────────────────────────┘
   │                    │
┌──────────┐ ┌──────────────────────────────────┐
│ Modify   │ │     Perform n = 6  extractions   │
│extraction│ └──────────────────────────────────┘
│conditions│                │
│(parameters│┌─────────────────────────────────┐
│or method)│ │            Analysis             │
│   **)    │ └──────────────────────────────────┘
└──────────┘                │
   │   No       ◄  Cn ≤ 0.1 Σ Ci  ►
   └────────────            │  Yes
```

$$C_n \leq 0.1 \sum_{i=1}^{n} C_i$$

```
┌──────────────┐
│  Increase    │     ┌──────────────────────────────────┐
│ number of    │◄────│ Derive routine conditions and    │
│test components│    │    qualify the blank value       │
└──────────────┘     └──────────────────────────────────┘
                                     │
┌──────────────┐      No        ◄ i.o.? ►
│   Check      │◄──────────────       │  Yes
│ inspection   │                      ▼
│ conditions/  │     ┌──────────────────────────────────┐
│  method      │     │  Extraction conditions are       │
└──────────────┘     │  suitable Inspection specification│
                     │       can be fixed               │
                     └──────────────────────────────────┘
                                     │
                     ┌──────────────────────────────────┐
                     │ Optional step => double inspection│
                     └──────────────────────────────────┘
```

*) This is a provisional blank value, see note in Section 5.2.3

**) See also different forms of declining curves and their possible interpretations in Annex A 5.2
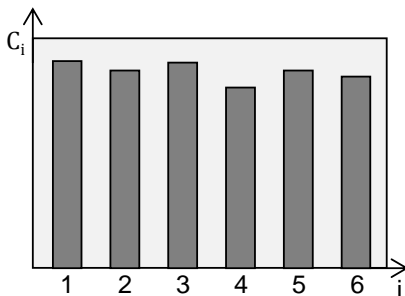
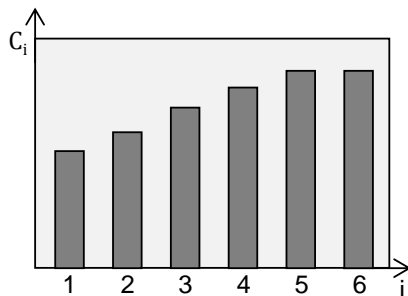## A 5.2 Various shapes of declining curve

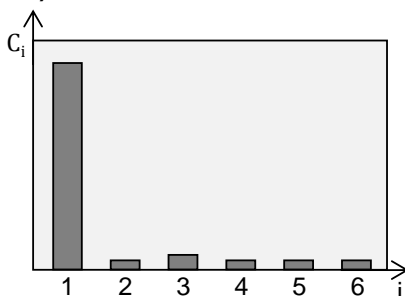### 1) Constant decline
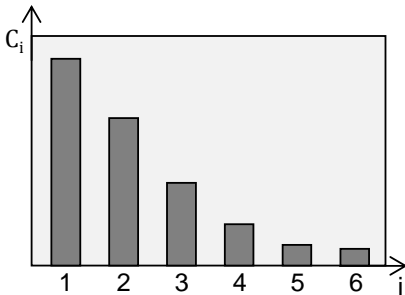
2) Delayed decline
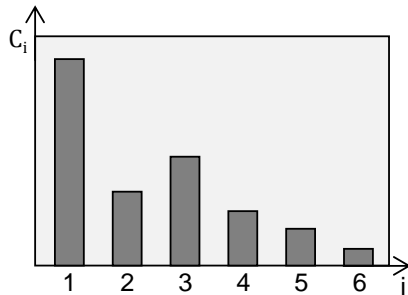
3) No decline

4) Increase

5) Immediate decline

Interpretation of different declining curves

About 1) Constant decline: contamination is detached at a uniform rate, no further interpretation required, the routine inspection procedure can be fixed.

About 2) Delayed decline "humped curve": detachment is not uniform due to delayed dissolving of contamination or process media such as pre-servatives by the extraction 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 1).

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 extraction liquid) for an appropriate length of time without any physical cleaning assistance such as pressure-rinsing or ultrasonics. Alternatively, components can be filled with the solvent and sealed if interior surfaces are to 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 analysis liquid.

A further method to accelerate particle detachment during the extraction procedure is to use a extraction liquid with a stronger dissolving power. Care is to be taken to follow the relevant safety instructions.

About 3) 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 ultrasonics on cast materials, coatings flaking off, burrs detaching or solder beads adhering firmly to the surface. In such a case, extraction parameters are to be modified to avoid **"component attack"** or a different extraction method is to be selected. This type of declining behavior 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 value. In this case, an extraction procedure with a lower particle emission level from the component/lower blank value needs to be established. If the blank value 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 is to 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 value 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 piece remain in the test apparatus and are transmitted to later extraction steps.

About 4) Increase: the quantity of contamination found in each extraction step increases. This may also be due to several factors:

- As in 3), 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 2). 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 apparatus, or incorrect handling.

On 5) 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 value.

- 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 sampled 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 declining curves may also be due to faulty handling or unsuitable test apparatus.

A 5.3 Routine inspection

```
        ┌─────────────────────────────────┐
        │ Conditions appropriate to       │
        │ inspection specification        │
        └────────────────┬────────────────┘
                         ▼
        ┌─────────────────────────────────┐
   ┌───▶│    Condition test apparatus     │
   │    └────────────────┬────────────────┘
   │                     ▼
   │    ┌─────────────────────────────────┐
   │    │    Prepare test component(s)     │
   │    └────────────────┬────────────────┘
   │  ┌──────────────┐   │
   ├─▶│ Clarify and  │──▶│
   │  │ remedy cause │   │
   │  └──────────────┘   ▼
   │    ┌─────────────────────────────────┐
   │    │  Check blank value (case by case)│
   │    └────────────────┬────────────────┘
   │         No          ▼
   └─────────────────◆ i.o.? ◆
                         │ Yes
                         ▼
        ┌─────────────────────────────────┐
        │            Extraction            │
        └────────────────┬────────────────┘
                         ▼
        ┌─────────────────────────────────┐
        │             Analysis             │
        └────────────────┬────────────────┘
                         ▼
        ┌─────────────────────────────────┐
        │      Expression of results       │
        └─────────────────────────────────┘
```
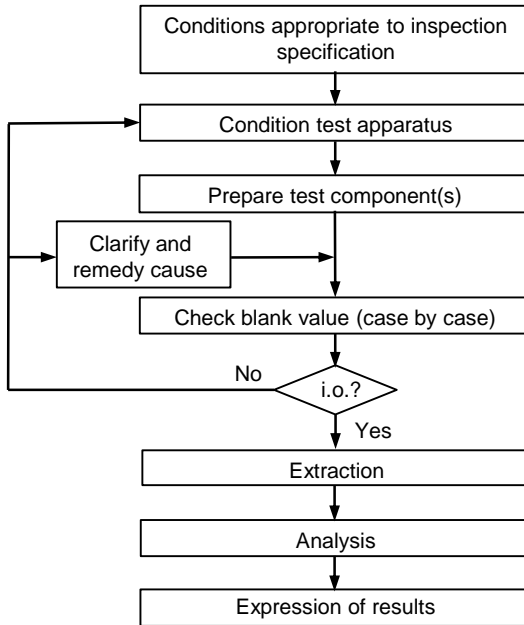
6   EXTRACTION METHOD

6.1   Principles

Because of their geometry and surface characteristics, in general functionally-relevant components in automotive fluid systems cannot be fully inspected directly for the presence of particles (e.g. using a direct visual or microscopic technique). For this reason, this chapter describes the method of removing particles from components by so-called 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 Chapter 5: Qualification tests and blank value.

Depending on the test component, several different extraction methods may have to be implemented. It may also be necessary to activate parts of a component mechanically in order to sample the test 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 test surfaces that have different cleanliness specifications and which therefore have to be sampled separately.

After detaching the particles from test surfaces with a aid of a liquid or possibly air (see also Chapter 3: Selecting the inspection method), 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 analyzed directly in the medium.

Because the result of a cleanliness inspection is highly dependent on the care with which the (invariably manual) extraction procedure is performed, it is essential that inspection staff are well-trained and motivated. The inspection specification must contain a detailed description of the extraction procedure.

6.2 General requirements of extraction equipment

All items of the extraction apparatus coming into contact with the test medium, and all surfaces thereof whose cleanliness could affect the result of the component cleanliness inspection, are to be constructed and kept clean in such a way to ensure that that the permissible blank value is not exceeded. The following points are to 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 collection vessels and angled piping (as short as possible) from collection vessel to analysis filtration

- no particles emitted from the apparatus (or only particles which are not relevant to the component inspection), also when components are activated, moved or driven.

To facilitate the attainment of and compliance with blank values, the surfaces of extraction apparatus in contact with media that are located between the purification filter of the test medium and the analysis filtration unit should be kept as small and as geometrically simple as possible.

6.3 Preparatory steps and post-treatment of test components

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 different suitable workplace.

6.3.1 Unpacking

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

When opening packaging and removing test pieces, care is to be taken to avoid contaminants adhering to the outside of the packaging from being released and reaching the test surfaces of components. Examples of appropriate action include:

 – Cleaning external packaging surfaces before opening

 – Changing gloves between opening the packaging and removing the test pieces

 – Ensuring that packaging is opened by one person and test pieces removed by another

 – Onion skin principle (two layers of packaging and lock concept)

6.3.2 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). Two facts need to be clarified:

 – The exact location of the test 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 test surface(s). This is especially important if the cleanliness specification is normed or coded, e.g. per 1000 cm² or 100 cm³. In such cases, the permissible quantity of contamination for the test piece is calculated using the real size of the test surface, and subsequently used to derive the permissible blank value for the test. Additionally, when developing a declining test, the required quantity of extraction liquid can be determined on the basis of the start parameters.

6.3.3 Preparatory steps

Where test surfaces are not the full size of the component, or where a component possesses several relevant test surfaces with different cleanliness specifications (see also 6.3.2), it may be necessary to isolate the various component areas from one another. In this way, extraction liquid cannot inadvertently come into contact with another test surface during the procedure and transfer particles to it that are not relevant.

Areas not requiring inspection are to be isolated, for example by plugging openings, coating, masking, sealing or marking them. Isolation measures are to be carried out in an appropriately clean manner.

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

For the extraction, it may still be necessary to fix the test piece 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 Point 6.3.4 Disassembly.

Note: In some cases, it may make sense to design adapters for components in bright colors. If particles of this color then appear in the analysis, the particles originate from the adapter and are not due to an inadequate level of cleanliness of the test piece.

6.3.4 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 will be generated and released, e.g. from joins. To prevent particles generated during disassembly from reaching component test surfaces, disassembly tasks are to be carefully planned and performed in an appropriately clean manner. Particles originating from disassembly steps are to be removed (e.g. by suction cleaning or wiping) the moment they are generated/released or immediately afterwards. All tools and auxiliary equipment are to 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 test surfaces being altered by disassembly processes, disassembly steps are to be carefully documented; alternatively, precise instructions for disassembly are to be given.

6.3.5   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 media 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, this is to 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 are to 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 VDA 19.

6.3.6   Post-treatment

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

6.4 Liquid extraction

As described in Chapter 3.1.1 Selecting the extraction method, liquid extraction is suitable for the majority of functionally-relevant components encountered in the automotive industry.

6.4.1 Extraction liquid

The extraction liquid (and final rinsing liquid) has to 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 extraction liquid is mainly selected in dependence on the component material and the characteristics of the contaminants expected. Among others, these are dependent on the previous history of the component and its manufacturing process (see also Annex A 6.4.1.1). Particles relevant to the inspection may, for example, be bound in assembly greases or preservatives.

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

Note: Purification filters must reliably retain particles of a size that is maximum 10% of the smallest particle size specified for a component. If the smallest size to be assessed is, e.g. 100 µm, the purification filter must at least be capable of reliably retaining particles ≥10 µm (minimum deposition rate of 99%). An alternative to this is to filter liquids repeatedly.

Warning: With aqueous cleaning media, for example, active cleansing substances present in the extraction liquid could be removed by the filter.

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

In order to re-use an extraction liquid (e.g. in circulatory systems), it has to be treated so that it does not impair the quality of inspection results. The following points are to be observed:

- 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.

Annex 6.4.1   Extraction liquid

A 6.4.1.1   Suitability and compatibility of extraction liquids

The following tables only give a simplified overview of extraction liquids. Individual applications still require technical clarification. The tables do not include special cleaning media that are not grouped into the categories mentioned.

Table 6-1: Ability of extraction liquids to dissolve accompanying contaminants from components that have a potential to bind particles

Substance (contaminant)	Extraction liquid		
	Aqueous tensidic neutral	Polar solvents *)	Non-polar solvents **)
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 (AV-GO)	O	+	+
Silicon oil	-	-	+
Anti-corrosives, amine-based	+	+	O
Anti-corrosives, other	O/+	+	+
Wax	O Only above melting point	O	+

+ suitable, O partially suitable, - unsuitable

*) polar solvents, e.g. alcohols

**) non-polar solvents, e.g. cold cleaners or benzene

Table 6-2: Compatibility of extraction liquids with materials

Material (test component and inspection equipment)	Extraction liquid		
	Aqueous tensidic neutral	Solvents polar *)	Solvents non-polar **)
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

6.4.2   Pressure-rinsing

Principle

Pressure-rinsing describes the localized application of a suitable extraction 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 extraction 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 Annex A 6.4.2.2).

With most applications, a full-jet nozzle with a round cross-section is utilized. However, depending on the geometry of the component requiring extraction, it may be more practical to use different-shaped nozzles (see Annex A 6.4.2.1).

Note: 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 must be taken to ensure that the complete internal area is filled and wetted with extraction liquid. The extraction effect in this case is no longer that of pressure-rinsing but rather of rinsing because the surface is not impacted by an open jet (see Chapter 6.4.4 Internal rinsing).

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

- characteristics of the extraction liquid
- volume flow 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 sampling the object surfaces
- time per surface or forward feed
- repeat number of extractions per surface.

Note: The pressure of the jet can only be used to characterize its cleaning effect to a limited extent. Although the volume flow 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), may vary significantly from

the actual pressure at nozzle level and are therefore unsuitable as parameters. On the other hand, the volume flow remains constant regardless of where it is measured. Even if the volume flow 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 Annex 6.4.2.1 Diagrams 1), 4) and 6)).

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

If the pressure-rinsing method is implemented using a solvent as an extraction liquid, aerosols could be generated. These aerosols may form a combustible mixture even in cases where the flashpoint of the extraction 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 chamber which does not contain any potential ignition sources.

To find out when aerosols may occur, please refer to the information given in the following documents:

- Leaflet M 043 02/2007 published by BG Chemie

- EN 12921-3:2005+A1:2008 (international C-norm ISO 12921-3)

These documents explain that there is no risk of aerosol formation provided the pressure at the level of the rinsing nozzle is below 0.7 bars. On the basis of this guideline, the graph illustrated in Annex A 6.4.2.4 was developed by way of experiments when VDA 19 was compiled. The graph can be used as a guide to determine the combinations of nozzle diameter and volume flow 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 (see also Chapter 11: Work safety and protection of the environment).

Start parameters

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

Table 6-3: Start parameters for pressure-rinsing

Parameter	Start Value
Nozzle shape	Round full-jet nozzle
Nozzle diameter	2.5 mm
Volume flow	1.5 l/min
Distance from component	Max. 15 cm
Pressure-rinsing volume / component surface area	5 ml/cm²

Note 1: When sampling larger surfaces, several nozzles (e.g. combined in a pressure-rinsing tool) can be used together to reduce the extraction time. However, care is to be taken to ensure that the above-mentioned parameters apply for each nozzle aperture.

Note 2: Due to the way an existing rinsing apparatus 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 are to be documented, the initial volume flow of the pressure-rinsing liquid can be decreased to 1.0 l/min.

When using pressure-rinsing for small components that are to be inspected in bulk, the test lot must contain sufficient components to give a minimum **extraction surface area of 200 cm²**.

As part of the qualification test/declining tests (see Chapter 5), the final pressure-rinsing volume per component surface area 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 more suitable parameters are justified and documented, start parameters may be modified.

Materials and equipment

The materials and equipment used in the extraction step must comply with the general requirements of extraction equipment described in Chapter 6.2.

1. Extraction liquid

2. Recipient for component: e.g. rest, tweezers or mount for single objects; coarse-meshed basket for small components; manipulators for large components

3. Pressure-rinsing apparatus consisting of:

    – pressure-rinsing tool(s): e.g. nozzle stylus, which can be controlled manually during the component extraction process and final rinsing of the extraction apparatus, lances for component inner surfaces, or similar.

    – media supply with container for extraction liquid, purification filter, pump or pressure supply

4. Sampling container: e.g. pressure-rinsing chamber, funnel or sampling vessel to collect analysis liquid

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

6. 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

Note: In principle, laboratory wash bottles can also be used for component extraction by pressure-rinsing. However, this is not recommended 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.

Procedure

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

1. Procure all resources required for the test

2. Pre-clean all surfaces of the extraction apparatus in contact with media; where appropriate, determine blank values

3. Prepare the test component as described in Chapter 6.3

4. Place the test component in the extraction container/extraction chamber; if necessary, position the test component in such a way so as to enable the liquid to flow easily into the sampling container or collecting vessel

5. Perform the pressure-rinsing extraction 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

6. 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 is to be included in the analysis)

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

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

9. Transfer all the liquid for filtration and analysis

10. Fill out the inspection report.

Documentation

See Chapter 9 Documentation and also Chapter 12 Case examples

Annex 6.4.2   Pressure-rinsing

A 6.4.2.1   Examples of various pressure-rinsing techniques

1) Round full-jet nozzle for localized extraction

Shape of jet

2) Pressure-rinsing lance for extracting contamination from blind holes
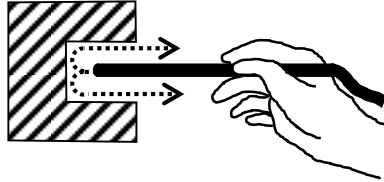
3) "Spiked lance" for extracting contamination from internal component areas
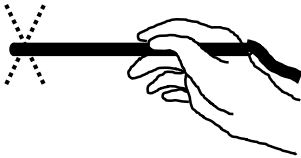
4) Parallel jet nozzle for extracting contamination from larger surface areas
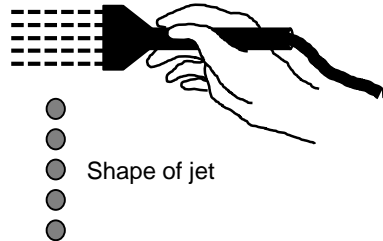
Shape of jet

5) Flat-jet nozzle (fan nozzle) for extraction of larger surface areas (care with aerosol formation)

Shape of jet

A 6.4.2.2 Examples of use of pressure-rinsing

1) Cleaning single components
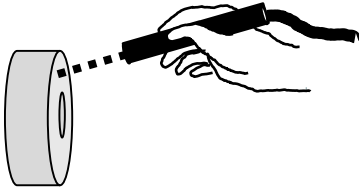
2) Cleaning large surfaces
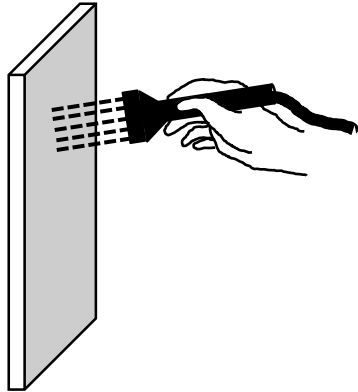
3) Cleaning small components (bulk goods)
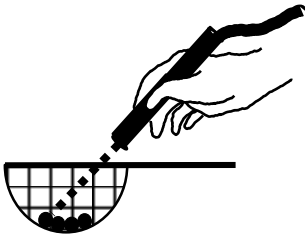
4) Cleaning accessible internal areas of components
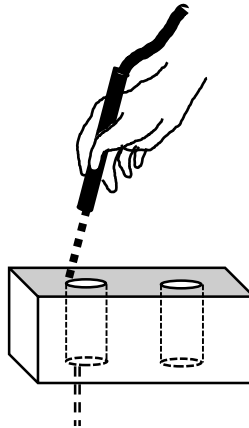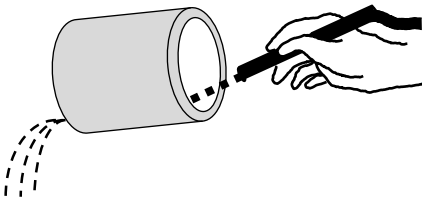
A 6.4.2.3 Example of pressure-rinsing apparatus

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 inspection liquid

   • Purification filter

   • Pump or pressure supply

7) Control unit for pressure-rinsing
   programs

8) Foot pedal
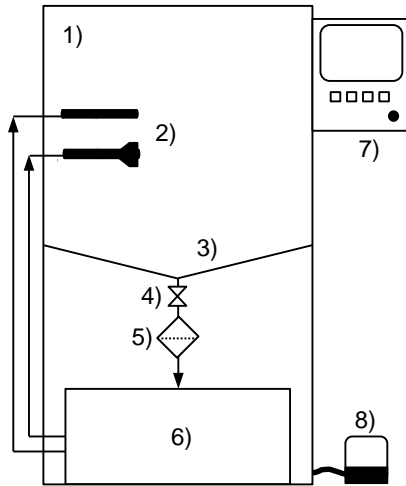
A 6.4.2.4 Information on nozzle diameters and flow rates

Jet pressure at nozzle level in relation to nozzle diameter (round full-jet nozzle) and volume flow as well as risk of aerosol formation
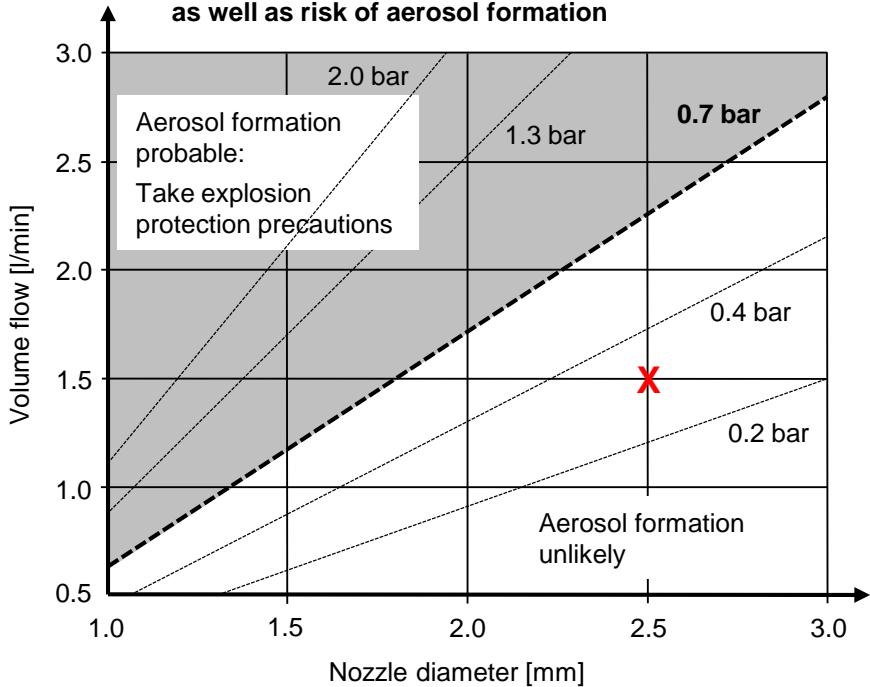
The start parameter values for the nozzle cross-section and volume flow are marked with an **X** and lie in an area where there is a risk of aerosol formation. This information is based on:

– Leaflet M 043 02/2007 published by BG Chemie
– EN 12921-3:2005+A1:2008 (international C-norm ISO 12921-3)

6.4.3   Ultrasonics

Principle

Extraction by ultrasound describes the action of mechanical oscillations ranging between 20 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 ultrasonics is caused by high pressure peaks that form when cavitation bubbles implode.

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

Note: In the event that too-high quantities of particles are detached from the component surface as a result of unsuitable ultrasound parameters, this can be corrected by setting a higher frequency or lower output. Special care is to 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 ultrasound parameters may cause material damage with the result that non-relevant particles are released from the component surface.

Ultrasound baths are suitable for sampling 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 must be exceeded without the component material on the outside being attacked.

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

Ultrasonics 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 throughflow of extraction

liquid and additionally immersed in an ultrasound bath and treated externally with oscillations.

The main parameters influencing extraction by ultrasound are:

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

Start parameters

The start parameters for extraction by ultrasonics are carefully selected in order to avoid highly-aggressive low ultrasound 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.

Table 6-4: Start parameters for ultrasonics

Parameter	Start Value
Ultrasound frequency	35-40 kHz
Power density	10 W/l
Time	30-60 s

Note: The power density is set via an output regulator on the ultrasound 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 should correspond with the effective power density and not the peak power density.

When using ultrasonics on small components that are inspected in bulk, the test lot must contain sufficient components so as to achieve a minimum **extraction surface area of 200 cm²**.

During the qualification test/ declining tests (see Chapter 5), the required duration in the ultrasound 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 more suitable parameters are justified and documented, the recommended start parameters may be modified.

Materials and equipment

The materials and equipment used in the extraction step must comply with the general requirements of extraction equipment described in Chapter 6.2.

1. Extraction liquid, dynamic viscosity $\eta \leq 5$ mm²/s

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

   - Mounting device to secure the test component in the bath (no contact with the base or walls); materials must be permeable to ultrasound 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 ultrasound effect due to the formation of stationary waves (oscillation velocity ≤ 1 cm/s)

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

4. Pressure-rinsing test set-up for the final rinsing of components and equipment and/or for dispensing extraction liquid (see Chapter 6.4.2 Pressure-rinsing)

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

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

Warning: As well as the general safety requirements applicable to all extraction methods (see Chapter 11), two further points are to be considered when using ultrasound baths:

- Due to the coupling of ultrasonics in the extraction liquid and the resulting energy input, liquids may become hot if subjected to ultrasonic waves for an extended period of time. This is to be given special consideration and validated accordingly in cases where combustible liquids are used (temperature must be kept 20° below respective flashpoint).

- During the application of ultrasound, do not place hands or any other part of the body in the extraction liquid (risk of embolus or destruction of skin cells!).

Procedure

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

1. Procure all resources required for the test

2. Pre-clean all surfaces of the extraction apparatus in contact with media; where required, determine blank values

3. Prepare the test component as described in Chapter 6.3

4. Fill the extraction apparatus with clean extraction liquid:

   a. For tests carried out directly in the ultrasound 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.

   b. In a beaker: fill the bath with water and a small amount of cleaning medium/tensides (to improve the wetting capacity). The water only serves to transmit the ultrasound waves to the beaker and is not later analyzed. Again here, 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 ultrasound, which must 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 extraction liquid.

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

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

Note 1: If the component is to be subjected to ultrasound from several sides or several positions, care is to be taken when changing its position (turning over, rotating, etc.) to avoid additional particles from being generated by this handling step

Note 2: If the component or beaker is not moved slowly through the sound field of the ultrasound bath (min ½ wavelength of the sound waves in the bath), the cleaning effect may not be homogeneous.

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

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

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

10. Empty the beaker; alternatively, where technically possible, drain the analysis liquid out of the bath.

Note: When using laboratory ultrasound baths, care is to 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 ultrasound bath, it must be checked that these fixtures do not generate particles when used as this could falsify analysis results (blank value).

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

12. Transfer the total volume of liquid for filtration and analysis

13. Fill out the inspection report

Documentation

See Chapter 9 Documentation and also Chapter 12 Case examples

Annex 6.4.3   Ultrasonics

A 6.4.3.1   Examples of use of extraction by ultrasound

1) Ultrasound extraction procedure for small components (bulk goods in a beaker)
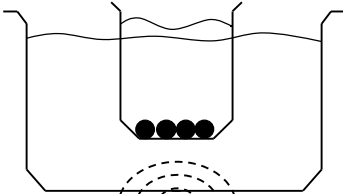
2) Ultrasound extraction procedure for small components (bulk goods in a mesh basket)
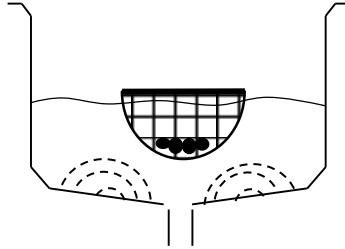
3) Ultrasound extraction procedure for single components
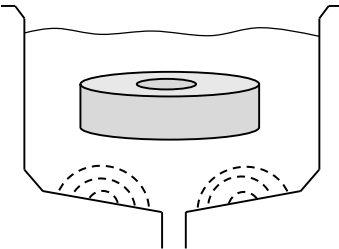
A 6.4.3.2   Verifying performance features of ultrasound baths

To assess relative stability over time (aging) or to compare different ultrasound baths, the efficacy of the ultrasound system can be verified. This is achieved by quantitatively determining the perforation of a sheet of aluminium foil by cavitation. The procedure must be carried out under defined conditions (foil thickness, location and orientation of the foil in the bath).

A 6.4.3.3   Volumes and power densities requiring documentation
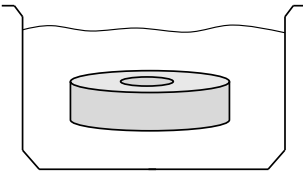
Volume analyzed = volume subjected to ultrasound = filling quantity of bath

Power density in bath =

Output of ultrasonic bath /

Filling quantity of bath
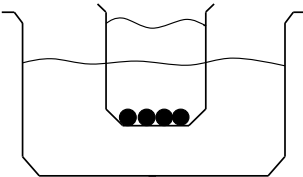
Volume analyzed = filling quantity of beaker
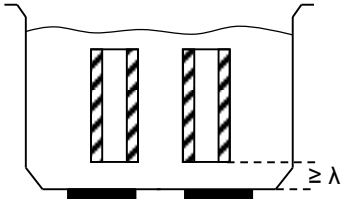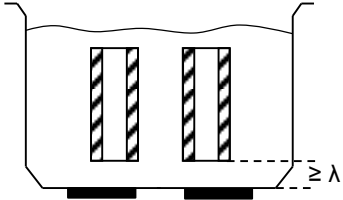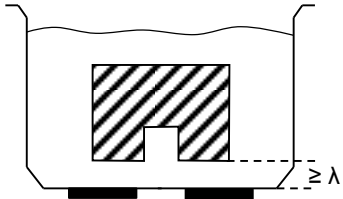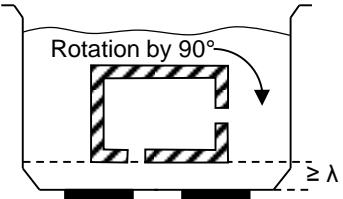
Power density in bath =

Output of ultrasonic bath /

(filling quantity of bath + filling quantity of beaker)

A 6.4.3.4   Examples of ultrasound wavelengths

Frequency	Wavelength λ
25 kHz	61 mm
35 kHz	43 mm
40 kHz	38 mm
120 kHz	12 mm

A 6.4.3.5 Examples of arranging components in the ultrasound immersion bath

Extraction	Arrangement in bath	Remark
External extraction	 Surface emitting ultrasound waves	Distance between component and sound source should be ≥ λ (see Annex A 6.4.3.3) For distances > 400 mm between the surface to be cleaned and the sound-emitting surface, components will have to be rotated.
Simple inner cavity	 Surface emitting ultrasound waves	Orientation of component opening to sound-emitting surface
Blind hole or similar shape	 Surface emitting ultrasound waves	Orientation of component opening to sound-emitting surface Ensure cavity is filled
Inner cavity	 Rotation by 90° Surface emitting ultrasound waves	Orientation of component opening to sound-emitting surface Ensure cavity is filled

6.4.4   Internal rinsing

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 piece is completely filled with extraction 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 throughflow further enhances the efficacy of this extraction technique.

Note: With so-called "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 resedimentation after detachment from the test component, and to forward them to the analysis step. The liquid is applied to the surface using a spray. "Final rinsing" is therefore completely different from the extraction method of "internal rinsing" described here, which can only be utilized for internal component geometries.

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

- active components needing to be actuated for the throughflow 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 extraction liquid. Once the medium has flowed through the test piece, 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 Annex A 6.4.2.3 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 extraction apparatus 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 medium 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 extraction liquid
- volume flow and rinsing time
- geometry and model of the test component
- pulsation and pulsation frequency
- type and frequency of movement (for active components).

Start parameters

As opposed to other liquid extraction methods, there are no meaningful start parameters for internal rinsing. This is because the volume flow required for a turbulent throughflow (calculable via the Reynolds number > 4000) is dependent upon the geometry of the test component and upon the test medium used.

However, the minimum throughflow volume must be several times the inner volume of the component.

In Annex 6.4.4.3, a table of examples lists flow velocities for two different extraction liquids (cold cleaners) with different viscosities. These rates are required for throughflows through tubes or lines of varying diameters in order to achieve a fully-turbulent flow and thus maximize the efficacy of the extraction.

Materials and equipment

The materials and equipment used in the extraction step must comply with the general requirements of extraction equipment described in Chapter 6.2.

1. Procure the extraction liquid

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

3. Internal rinsing apparatus consisting of:

   - Adapters for filling the component, for the throughflow and for draining the liquid from component

   - Media supply with container for extraction 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.

4. If require, use additional containers (funnels, beakers) to collect the extraction liquid

5. Pressure-rinsing apparatus for final rinse of equipment with extraction liquid using 7.

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

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

With a closed internal rinsing circuit, it make 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 piece from being achieved. These flow velocities are necessary in order for the extraction step to be efficient.

Annex A 6.4.4.4 illustrates an example of an internal rinsing system in a diagram.

Procedure

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

1. Procure all resources required for the test

2. Pre-clean all surfaces of the extraction apparatus in contact with media; where required, determine blank values

3. Prepare the test component as described in Chapter 6.3

4. 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 sampling container or collection vessel

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

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

7. 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)

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

9. Transfer all the liquid for filtration and analysis

10. Fill out the inspection report

Documentation

See Chapter 9 Documentation and also Chapter 12 Case examples

Annex 6.4.4   Internal rinsing

A 6.4.4.1   Various internal rinsing examples

1) Internal rinsing in a closed system (adapters for tubing on both sides)
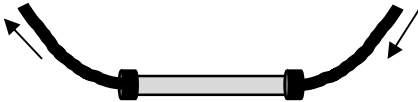
2) Internal rinsing by adapting a rinsing line (pressureless drainage)
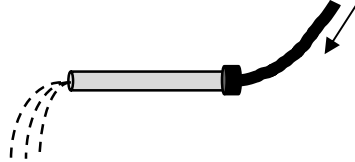
3) Internal rinsing by adapting a pressure-rinsing tool (pressureless drainage)
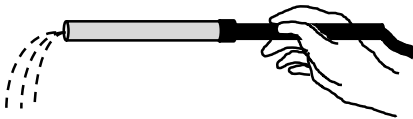
A 6.4.4.2   Examples of use of internal rinsing

1) Internal rinsing pipes or lines
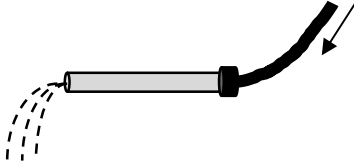
2) Internal rinsing of inner component areas (e.g. oil borings)
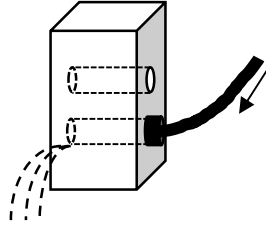
3) Internal rinsing components (e.g. heat exchangers or filter housings)
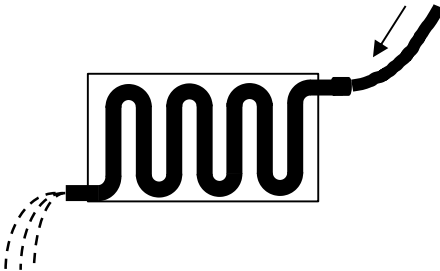
A 6.4.4.3 Examples of turbulent throughflow parameters

	Example - Cold cleaner Medium 1	Example - Cold cleaner Medium 2
Flashpoint [°C]	62	105
Kinematic viscosity [mm²/s]	1.9	3.2
Tube diameter [mm]	**Volume flow [l/min]**	**Volume flow [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 > 4000, which is defined via the equation Re = w *d /v, (w: velocity of the flow, d: tube diameter, v: kinematic viscosity) is used. The turbulent volume flow (dV/dt) for other liquids can be calculated on converting this to dV/dt = Re * d * π * v /4.

A 6.4.4.4 Example of an internal rinsing system
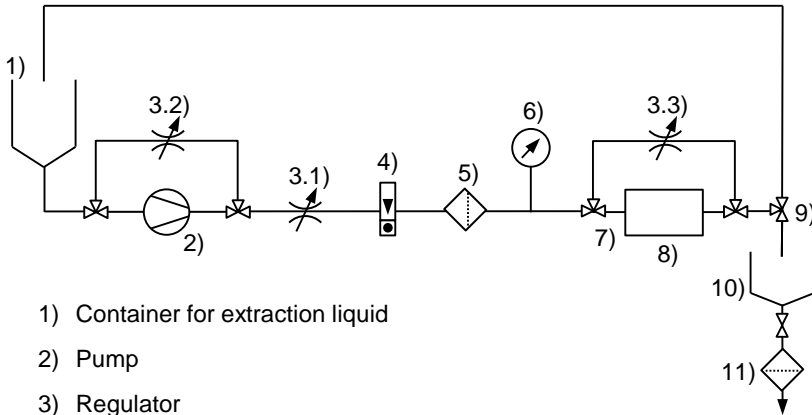
1) Container for extraction liquid
2) Pump
3) Regulator

    3.1 Main regulator

    3.2 Regulator at level of pump bypass

    3.3 Regulator at level of test component bypass

4) Throughflow meter
5) Purification filter
6) Manometer
7) Mounting device to hold test component
8) Test component
9) Three-way ball valve
10) Collection vessel for extraction liquid
11) Analysis filter

6.4.5   Agitation

Principle

The test component is partially filled with extraction liquid and all openings sealed. By agitating this liquid, particles adhering to the inner test 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 weight allow the test areas to be adequately agitated. The method is not suitable for sampling 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 weight 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.

Note: It is not recommended to place simply-shaped, small components in a vessel filled with extraction liquid and subsequently agitate the sealed vessel manually or by means of a vibrating table. This is because particles can be generated if the components strike against one another. Ultrasonics or pressure-rinsing are preferred methods in such cases.

The main parameters influencing extraction by agitation are:

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

Start parameters

The start parameters for extraction by agitation are selected to maximize the extraction effect whilst still allowing the process to be performed manually if desired.

Table 6-5: Start parameters for agitation

Parameter	Start Value
Filling volumes	30-40%
Amplitude	approx. 30 cm
Frequency	1 Hz
Time	15 sec

During the qualification test/ declining tests (see Chapter 5), 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 more suitable parameters are justified and documented, these start parameters may be modified.

However, a filling volume of 30 - 40% for components should 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.

Materials and equipment

1. Extraction liquid

2. Pressure-rinsing apparatus for final rinsing of components and equipment and/or dispensing the extraction liquid (see Chapter 6.4.2)

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

4. Funnel: for filling and/or emptying extraction liquid

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

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

7. Clock to control the duration of the agitation step

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

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

Procedure

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

1. Procure all resources required for the test

2. Pre-clean all surfaces of the extraction apparatus in contact with media; where appropriate, determine blank values

3. Determine the internal volumes of the test component

4. Prepare the test component in accordance with Chapter 6.3; special care is to be taken to clean the exterior of the component well and to seal all component openings with suitable clean, abrasion-resistant closing devices

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

6. Determine the volume of extraction liquid required, i.e. 30-40% of the volume of the interior of the test piece

7. Pour the extraction liquid into the object and seal the opening

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

9. 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.

10. 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 clean extraction liquid or a pressure-rinsing step. The resulting volume of liquid forms part of the analysis liquid.

11. Ensure that the test component is fully emptied

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

13. Transfer all the liquid for filtration and analysis

14. Fill out the inspection report

Documentation

See Chapter 9 Documentation and also Chapter 12 Case examples

Annex 6.4.5   Agitation

A 6.4.5.1   Examples of use of extraction by agitation

1) Agitation of a pressure tank
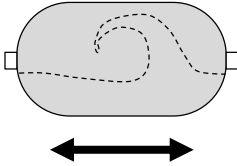
2) Agitation of a section of piping (short fat cross-section)
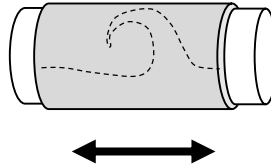
3) Agitation of a container
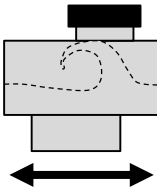
4) Agitation of small components in a container: not suitable
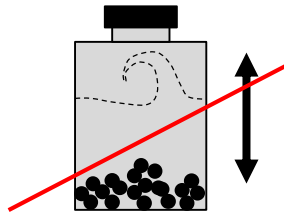
6.4.6 Dissolving

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 cleaning medium used for the inspection) for an appropriate length of time without the aid of further physical cleaning effects, such as pressure-rinsing or ultrasonics. Alternatively, components can also be completely filled and sealed if inner surfaces are to be sampled.

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 analysis liquid.

The dissolving step can be speeded up during the extraction process by using an extraction liquid with a stronger chemical solvent action (verify compatibility with component). In such cases, the respective safety regulations apply.

The main parameters influencing the dissolving step are:

    – characteristics of the dissolving liquid

    – duration of the dissolving procedure

Materials and equipment

1. Dissolving liquid (e.g. extraction liquid, or medium with stronger dissolving properties)

2. Pressure-rinsing equipment to perform final rinsing step on the apparatus and/or to dispense the extraction liquid (see Chapter 6.4.2)

3. Where appropriate: funnel for filling or removing dissolving liquid

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

5. Clock to control the duration of the dissolving step

6. Dissolving container, beaker or bath from pressure-rinsing or ultrasound extraction apparatus

Procedure

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

1. Procure all resources required for the test

2. Pre-clean all surfaces of the dissolving container in contact with media, where required, determine blank values

3. Place the component in the dissolving container

4. 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

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

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

7. Transfer the dissolving liquid to the filtration process

8. Then, using a sufficient volume of extraction 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 analysis liquid.

9. Transfer all the liquid for filtration and analysis

10. Fill out the inspection report

If the dissolving procedure is only carried out on the inner surfaces of a component, the described procedure is to be modified accordingly. The component must 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.

Documentation

See Chapter 9 Documentation

6.5 Air extraction

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 become 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.

6.5.1 Air jet extraction

Principle

With this extraction method, particles are detached from the test piece by means of a jet of clean, oil-free compressed air. 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 throughflow method, or also to remove contamination from logistics packaging, such as blister packs, small load carriers or cardboard.

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

Air jet extraction is performed in two steps:

1. First of all, particles are detached from the test piece with a jet of compressed air and subsequently adhere to the walls of a chamber wetted with liquid. The test piece is then removed from the chamber.
2. In a second step, the particles adhering to the chamber 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:

- 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 sampling the component 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.

Start parameters

Table 6-6: Start parameters for air jet extraction

Parameter	Start Value
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 / component surface area	1 sec/cm²

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

During the qualification test/ declining tests (see Chapter 5), the duration of application of the jet per component 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 more suitable parameters are justified and documented, these start parameters may be modified.

Materials and equipment

The materials and equipment used in the extraction step must comply with the general requirements of extraction equipment described in Chapter 6.2.

1. Supply of clean, oil-free compressed air (cleanliness of the compressed air must fulfill the blank value 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)

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

3. 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 extraction liquid, purification filter, pump or pressure supply

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

5. 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

Procedure

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

1. Procure all resources required for the test

2. Prepare the test component in accordance with Chapter 6.3

3. Use extraction liquid to pre-clean all surfaces of the air jet chamber coming into contact with compressed air and media as well as other extraction equipment; where required, determine blank values. In order to bind detached particles, all the walls of the air jet chamber must be wetted with the test medium before commencing the air jet extraction procedure.

4. Place the test component in the air jet chamber and secure it if necessary

5. 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.

6. Remove the test piece from the air jet chamber

7. Using extraction liquid, perform a final rinsing step on all inner surfaces of the air jet chamber 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.

8. Transfer all the liquid for filtration and analysis

9. Fill out the inspection report

Documentation

See Chapter 9 Documentation

Annex 6.5.1   Air jet extraction

A 6.5.1.1   Principle and procedure for air jet extraction

Step 1: Extract particles from test component using a jet of **compressed air**
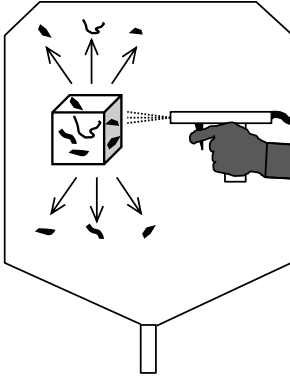
Step 2: Remove the test component
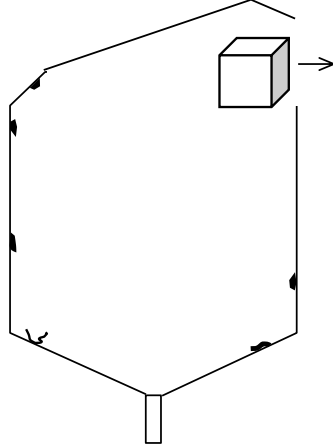
Step 3: Perform final rinsing step on chamber without test component using **liquid**

Analysis filter
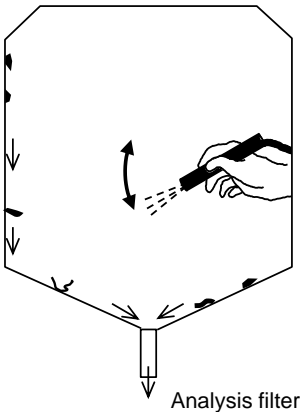
6.5.2 Air throughflow extraction

Principle

With this form of air extraction, the test piece is subjected to a throughflow 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 centimeters in diameter.

Air throughflow extraction is divided into two steps (see also Annex A 6.5.2.1):

1. Primary extraction step: the test component is installed in the test bench using component-specific adapters. Particles extracted by the clean (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 so-called primary analysis filter is removed in an appropriately clean manner after the primary extraction step and forwarded to the secondary extraction step.

2. Secondary extraction step: in this step, liquid extraction by means of pressure-rinsing (see Chapter 6.4.2) is carried out to remove the particles originating from the test piece 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: The air throughflow 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 millimeters in width. The basic set-up is illustrated in the form of a diagram in Annex A 6.5.2.2. Due to the fact that very little experience had been gained about this extraction method at the time this version of VDA 19 was written, a detailed description has not been included. In the example shown, the pneumatic component is subjected to a throughflow of clean, oil-free compressed air. After the extraction step, the air is guided into the liquid-filled bath of a pressure-rinsing extraction apparatus. The particles extracted from the component are thus bound in the liquid and can subsequently be deposited onto an analysis filter and analyzed.

The main parameters influencing the air throughflow extraction method are:

- – air throughflow rate
- – geometry and type of the test component
- – form and frequency of actuation (in the case of active components)
- – duration of air throughflow

Start parameters

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

Two minutes are recommended as a parameter for the start time for each extraction step in the declining test/qualification test.

Materials and equipment

The materials and equipment used in the extraction step must comply with the general requirements of extraction equipment described in Chapter 6.2.

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

2. Air throughflow 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) must 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

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

4. Pressure-rinsing extraction apparatus (see Chapter 6.4.2) to perform the secondary extraction step on the primary analysis filter

5. Where appropriate: if the pressure-rinsing apparatus mentioned in Point 4 is located elsewhere and cannot be used here, further pressure-rinsing equipment to perform final rinsing step on the primary filtration unit.

Procedure

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

1. Procure all resources required for the test

2. Pre-clean all relevant surfaces of the extraction apparatus; where required, determine blank values

3. Prepare the test component in accordance with Chapter 6.3

4. Use component-specific adapters to connect the test piece to the test set-up

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

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

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

8. Drain off final rinsing liquid at the lowest point of the primary filtration unit

9. Remove the primary analysis filter and transport it to the pressure-rinsing extraction apparatus in an appropriately clean manner

10. Perform a full pressure-rinsing extraction step (in accordance with the procedure described in Chapter 6.4.2 Pressure-rinsing) using the primary filter membrane as the test piece. Transfer the particles to the secondary analysis filter.

11. Fill out the inspection report

Documentation

See Chapter 9 Documentation

Annex 6.5.2 Air throughflow extraction

A 6.5.2.1 Principle set-up required for air throughflow extraction on components such as engine air intake components

1) Air filter

2) Adapter to test component

3) Test component

4) Primary filtration unit

5) Primary analysis filter

6) Drainage system for final rinsing liquid

7) Sensors to monitor volume flow

8) Pump

9) Pressure rinsing chamber

10) Secondary analysis filter
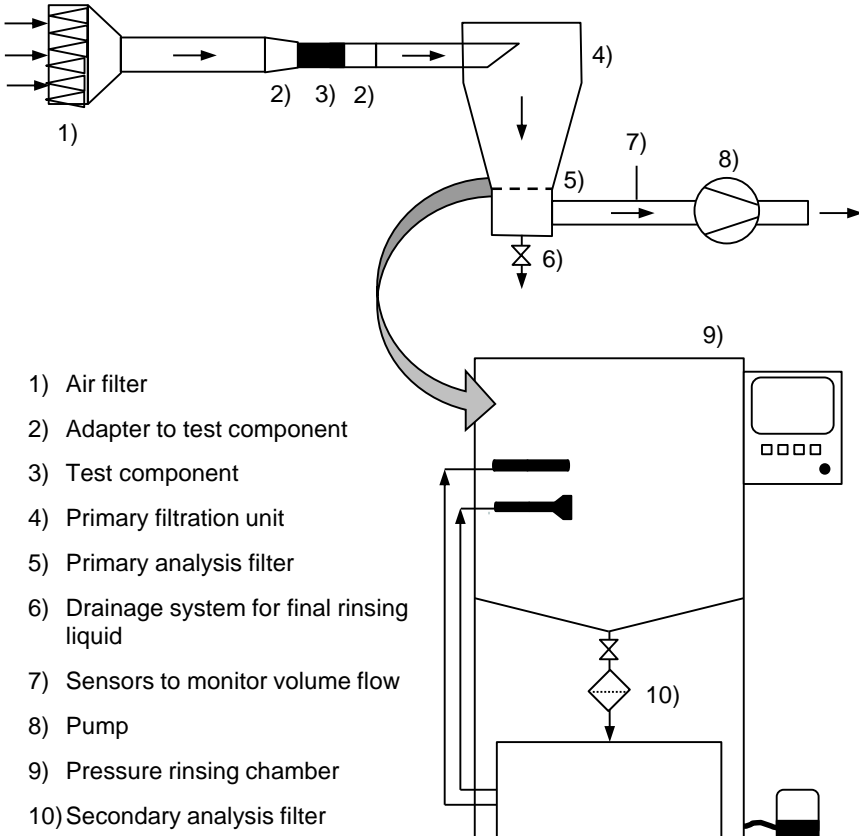
A 6.5.2.2 Principle set-up required for air throughflow extraction on components such as parts of pneumatic systems

1) Intake oil-free compressed air

2) Regulator

3) Air filter

4) Manometer

5) Mounting device to hold test component

6) Test component

7) Actuator for test component (only for active components)

8) Collection vessel for extraction liquid

9) Analysis filter
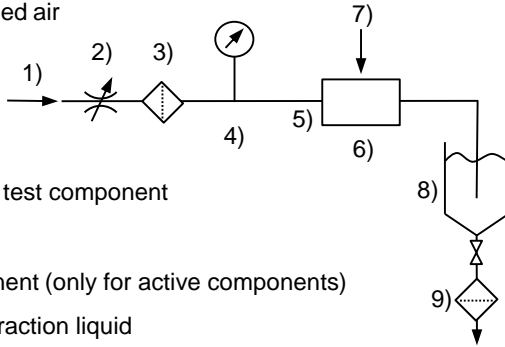
7 ANALYSIS FILTRATION

7.1 Principles

The task of analysis filtration is to deposit the particles extracted from the component and suspended in the analysis liquid onto the surface of an analysis filter so that they can be analyzed. 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.

7.2 Selecting the analysis filtration method

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 analysis is carried out within the scope of the standard analysis procedure in VDA 19, i.e. detection of particles upwards of 50 μm with gravimetric and/or light-optical analysis, the followings analysis filter is recommended:

5 μm PET mesh filter

7.2.1 Chemical resistance

All of the equipment used in the filtration process as well as the analysis filter utilized must be compatible with the extraction liquid implemented (where appropriate, also the final rinsing liquid or particle-fixative) (see also Annex A 7.4).

7.2.2 Particle retention capacity

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

must 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 Annex A 7.3 Selecting the filter pore size).

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 Annex A 7.2).

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 are retained by a finer filter downstream. If gravimetric analysis is required, all of the filters in the cascade need to be weighed.

7.2.3 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. color) 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.

7.3   Handling analysis filters

In order to avoid adverse influences from handling analysis filters, the following points should be observed:

- Always store analysis filter membranes in sealed containers to minimize contamination from the environment.

- Visually inspect the cleanliness and condition of analysis filter membranes before use. Where appropriate, discard damaged filters and pre-clean soiled filters with the same extraction liquid that will be used in the test.

- Always handle analysis filter membranes with clean tweezers and only hold the edge of the analysis filter membrane.

Note: 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.

Note: 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.

- On completion of filtration, careful drying and subsequent analysis, analysis filters should 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.

7.4 Materials and equipment

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

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

Note: If the negative pressure is too high, particles could pass through the analysis filter or the filter could become damaged.

3. Tweezers to handle the analysis filter membrane

Note 1: The tweezers used may 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 2: Analysis filters may also be handled with a clean spatula. All appropriate formulations also apply to spatulas.

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

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

6. Drying oven with controlled temperature (no fan)

Note: The temperature and duration of the drying process are to be modified in dependence on the combination of analysis filter and analysis liquid / extraction liquid (see Chapter 8.2.1: Gravimetry).

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).

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

Note: A fixative is used to prevent particle loss when handling and analyzing the analysis filter. In the case of gravimetric analysis, a fixative may only be utilized once the residue weight has been determined.

7.5 Procedure

1. Procure all resources required for the filtration step

2. Prepare the filtration equipment. Depending on the apparatus used in the extraction procedure, analysis filtration can be performed in two ways (see Annex A 7.1)

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

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

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

   3.1 Microscopic analysis:
   The analysis filter does not require conditioning.

   3.2 Gravimetric analysis:
   The tare weight of the analysis filter has to be determined (see Chapter 8.2.1: Gravimetry). To avoid negative residue weights, the analysis filter must be conditioned.

4. 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 may 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").

5. Filtration of the analysis liquid

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

   5.2 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 analysis liquid. The size of the vacuum flask limits the volume of liquid which can be filtered.

6. Filter the analysis 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 analysis 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.

7. To avoid particle loss, perform a final rinsing step on all surfaces in contact with the analysis liquid.

Note: 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.

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

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

Warning: Not suitable for gravimetric filters because the fixative alters the residue weight.

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

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

12. 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 labeled Petri dish (hold horizontally)

13. Dry the analysis filter by keeping the Petri dish partly open (in the case of analysis filters to be analyzed gravimetrically, dry the analysis filter until the weight remains constant. See Chapter 8.2.1: Gravimetry)

7.6   Verifying particle occupancy on the analysis filter

To use automated optical systems for particle analysis, the analysis filter has to be prepared in such as way so as to ensure that particles are evenly distributed over the surface of the analysis filter without overlapping or agglomerating. The following points are important in this regard:

- the occupation density 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 nests of particles present or are the particles mainly located at the margin?)

- occupancy with fibers: fibers are often much longer than other particles. Fibers 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 density of particle occupation on the filter in percent should be calculated and documented. The critical occupation density depends on the type of filter implemented. This is shown in Annex A 7.7 for mesh filters and foamed membrane filters.

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

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

2. Distribute the analysis 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 Chapter 8.2.1: Gravimetry).

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

Annex A 7.6 shows some examples of incorrectly-prepared analysis filters together with possible remedies.

Annex 7 Filtration

A 7.1   Different analysis filtration methods

A) Direct filtration

E.g.
- Collection vessel
- Ultrasound bath

Analysis filter

Filter holder

Vacuum source

B) Separate filtration

Filter funnel

Analysis filter

Filter holder

Vacuum source

Vacuum flask
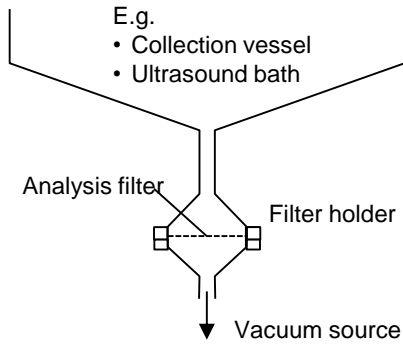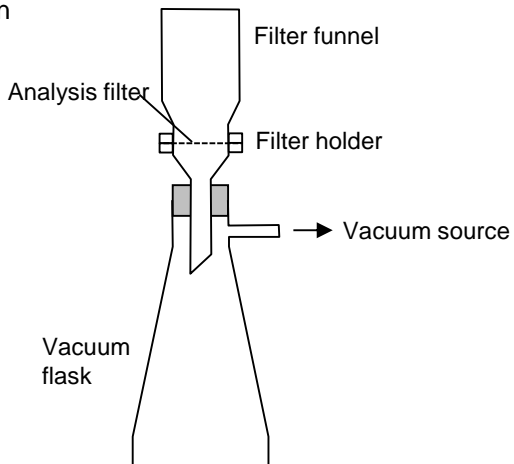
A 7.2 Filter cascade

Filter cascade

2 or 3 analysis
filters with
different pore
sizes

A 7.3 Selecting the filter pore size

**Filter pore size = 1/5 – 1/ 10 of the smallest
particle size requiring detection**

Filter pore size [μm]

25

20

15

10

5

0

5   15   25   50   100 150 200

Smallest particle size requiring detection [μm]
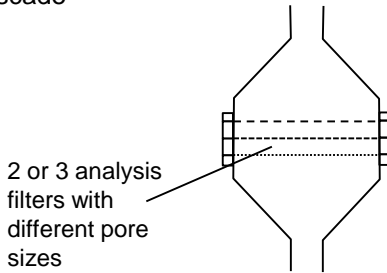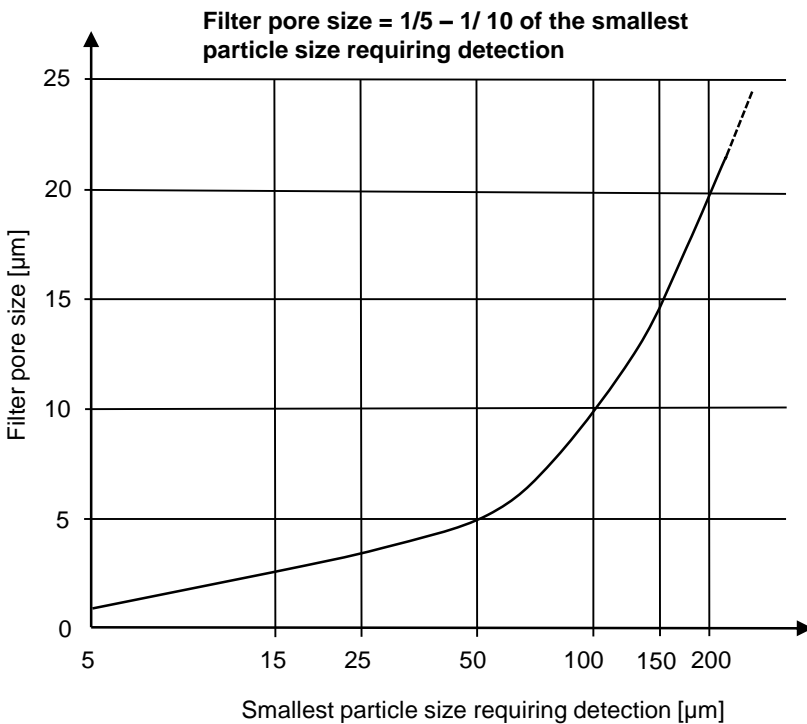
A 7.4   Compatibility of analysis filters with extraction liquids

Filter material		Medium (test or final rinsing liquid)				
		Aqueous neutral cleaner	Isopropanol	Ethanol (spirit)	Aliphatic hydrocar-bons, e.g. also cold cleaners	Ketones, e.g. ace-tone
Foamed membrane	Cellulose nitrate	+	-	-	+	-
	Cellulose acetate	+	+	-	+	-
	Polyamide/nylon	+	+	+	+	+
Mesh	Polyester	+	+	+	+	+
	Polyamide/nylon	+	+	+	+	+

Note: *The table lists only a few of the most common types of analysis filter. In some cases, e.g. if resistance to a certain inspection medium is required, or if a certain type of filter is necessary for the analysis method, different filter types may have to be selected. If no special conditions are specified, the use of the analysis filter suggested in Section 7.2. is recommended.*

A 7.5   Examples of analysis filters

8 µm cellulose nitrate
foamed membrane filter
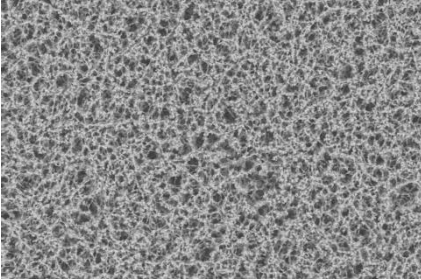
15 µm PET mesh filter
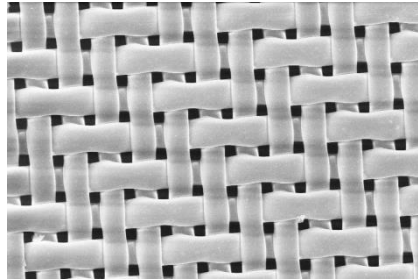
⊢ 20µm Magnification 200x

Foamed membrane filter

> 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 (gray or black analysis filter). This type of filter often absorbs humidity (impairs gravimetric analysis).

Mesh membrane filter

> 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 artifacts or reflections and impair light-optical analysis.

A 7.6 Examples of problems encountered with analysis filtration and possible remedies

Good filter occupancy:

- Uniform
- Most particles isolated

Excessive filter occupancy:

- Extract fewer components per filter
- Use coarser analysis filter
- Use filter cascade
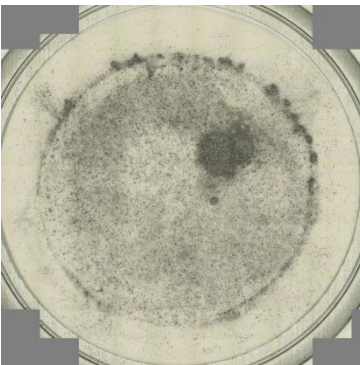
Inhomogeneous filter occupancy:

- Optimize filter holder or filtration process
- Post-process filter (re-float particles or gently shake filter holder with a small amount of liquid)

Examples of filter images
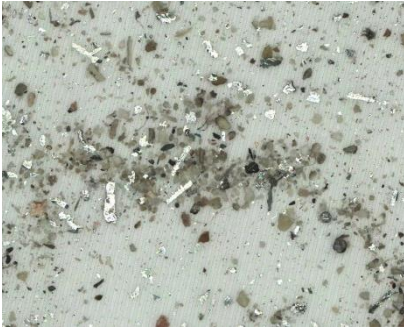
Excessive filter occupancy:

- Extract smaller component lots per filter
- Use coarser analysis filter
- Use filter cascade

Filter completely clogged:

- Not analyzable optically
- Only analyzable by gravimetry

Pieces of cardboard on the filter:

- Test component packaging may be unsuitable

Large number of fibers on filter

- Storage location of test components may be unsuitable

A 7.7 Filter occupancy and analyzability

Feasibility of light optical systems to analyze analysis filters

| 0.1 | 0.2 | 0.5 | 1 | 2 | 5 | 10 | 20 | 50 | 100 |
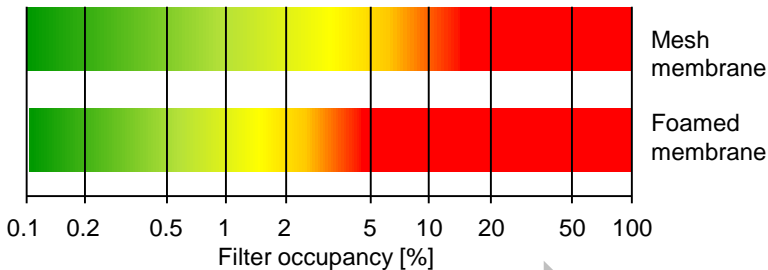
Filter occupancy [%]

- Number of measurement errors increases
- Number of manual post-processing steps increases
- Comparability of different systems decreases

Light-optical analysis starts to become difficult when mesh membrane filters have a filter occupancy upwards of 3% and foamed membrane filters an occupancy of 1.5% and above. 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 analyze filters also depends on other factors (homogeneous occupancy, percentage of fibers, etc.). Consequently, these values only serve as a rough indication and require individual assessment in each case.

Note: If filter occupancy is extremely homogeneous, it may be possible to analyze filters with a higher particle occupancy.

8 ANALYSIS METHOD

8.1 Principles

This section of the guideline describes the various techniques which can be implemented to analyze particulate contamination. The selection of a suitable analysis method and its field of use are described in Chapter 3: Selecting the inspection method.

8.2 Standard analysis

Provided 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.

8.2.1 Gravimetry

Principle

As shown in following diagram, 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 analysis liquid is filtered.
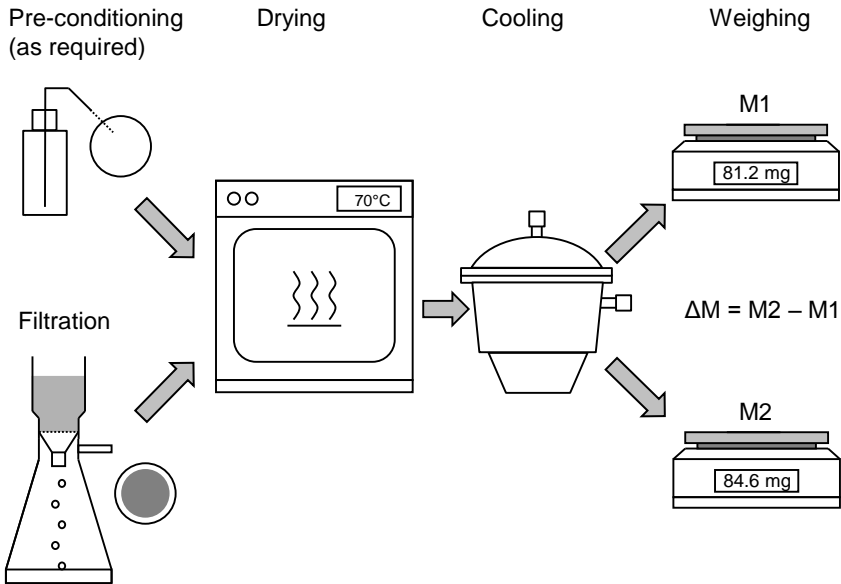
Fig. 8-1: Diagram of the gravimetric analysis procedure

The result of the gravimetric analysis is called the residue weight 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 weight difference, the analysis filter has to 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 weight must always be determined first.

A dilemma is often encountered when preparing an analysis filter for simultaneous gravimetric analysis to determine weight and light-optical microscopy to determine particle size distribution. This is because the analysis filter should contain a maximum quantity of particulate contamination in

138

order to obtain a reliable gravimetric result, but low numbers of single, separate particles for light-optical analysis.

Weight 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 weight constancy of the analysis filter is achieved, i.e. when the analysis filter does not lose any more weight 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 medium used (e.g. cellulose nitrate filter combined with cold cleaner, 150 minutes at 70°C).

Note 1: Weight constancy is attained if no change in weight 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) may vary for each combination of extraction liquid and analysis filter. See manufacturer's instructions for details.

Non-particulate residues: to stabilize the tare weight of the clean analysis filter before commencing the inspection, it may be necessary to rinse it briefly with extraction 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 cleaning media, or greases or wax originating from the component that are difficult to dissolve.

In some cases, particles may have 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.

Material and equipment

See **Chapter 7: Filtration**. The following are also required:

1. Desiccator

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

Note:	Depending on cleanliness requirements, or when relatively high residue weights have to be determined, the use of a desiccator may not be necessary. In order for this condition to apply, the maximum permissible blank value may not be exceeded.

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

   Note: Models with beta emitters must be monitored.

   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 must be located as close as possible to the weighing platform. Ionization units with fans are not suitable.

3. Analysis balance

   The minimum readability requirement is 1 d (1 digit) = 0.1 mg = 0.0001 g (four-digit balance).

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 are to 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 must be low-vibration and the temperature in the room kept constant. The relative humidity of the air should be kept between 45 and 60 %.

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

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 guideline requirement that the percentage of the blank value may be max. 10 percent of the total load, only particle loads with a residue weight (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) has to be less than 1 mg in order to fulfill the blank value 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 weight is determined in a gravimetric analysis, the result is expressed as M < 3 mg.

Another way of analyzing components with very small particle loads by means of gravimetry is to increase the residue weight (M). This is achieved by sampling several components simultaneously, or by filtering the analysis liquid from several components through one analysis filter.

Procedure

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

2. Condition the analysis filter to stabilize its weight (optional):

Note: To stabilize the weight of an analysis filter, first of all it is treated with analysis 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 weight. This is to be verified before using the combination of analysis liquid and analysis filter for the first time.

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

2.2 Pre-dry the membrane by sucking air through the analysis filter with the vacuum pump

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

4. Determine the initial weight M1 (tare weight):

    4.1 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

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

    4.3 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

    4.4 Read off and document the value shown for the initial weight M1 of the analysis filter (tare weight)

5. 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.

6. Perform the analysis filtration step (see **Chapter 7: Filtration**)

7. Determine the final weight M2 (gross weight):

    7.1 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

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

    7.3 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.

8. Read off and document the value displayed for the final weight M2 of the analysis filter (gross weight)

9. Using tweezers, put the analysis filter back into the Petri dish and close the lid.

10. Calculate the residue weight (net weight), which is the difference between M2 and M1

Calibration

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

Documentation

See Chapter 9 Documentation and also Chapter 12 Case examples

8.2.2 Light-optical analysis

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.

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.

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 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.

Principle

With light-optical analysis, the analysis filter containing the particles extracted from the test piece 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. So-called 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 Annex A 8.2.2.5). 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 (bright field, dark field, reflected light, transmitted light)
- 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 (color, brightness, homogeneity, surface topography and roughness, etc.)
- Others.

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 analyze the same type of particles.

Light-optical standard analysis

In order to obtain a good degree of comparability with different analysis systems (which all have their strong points) despite this, the VDA 19.1 guideline recommends a so-called standard analysis for light-optical particle analysis.

To apply standard light-optical analysis meaningfully, four general requirements have to be fulfilled:

- the light-optical analysis system fulfills 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 particle occupancy being not too high (see Chapter 7: Filtration)
- 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.

Note 1: 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, must be considered individually in each case.

Note 2: When changing from a light-optical analysis with different parameters to a standard analysis, the analysis results may vary.

In some cases, for several reasons 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 are to be mutually defined and documented if intended for use to validate cleanliness specifications in the customer-supplier relationship.

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

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

For the light-optical standard analysis of particles upwards of 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 Fig. 8-3, 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 micrometer scale in Fig. 8-3). 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 at least 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. Fig. 8-3 gives an overview of the suitability of different lenses for analyzing 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: 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.

| | | 2 µm | 5 µm | 10 µm | 20 µm | 50 µm | 100 µm | 200 µm | 500 µm | 1000 µm |

Material microscope

Very low depth of field — Fixed lens 20x

Low depth of field — Fixed lens 10x

Average depth of field — Fixed lens 5x

Zoom microscope

Low ←— Depth of field —→ Very high

Zoom lens

Stereo microscope

Average ←— Depth of field —→ Very high

Zoom lens

Scanner

Very high depth of field

Fixed lens

Recommended suitability for particle size range

Depth of field:
Very low	1 – 10 µm
Low	10 – 50 µm
Average	50 – 100 µm
High	100 – 1000 µm
Very high	> 1000 µm

Fig. 8-2: Overview of suitability of different lenses for particle analysis

Note: 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 cam-

148

era. 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.
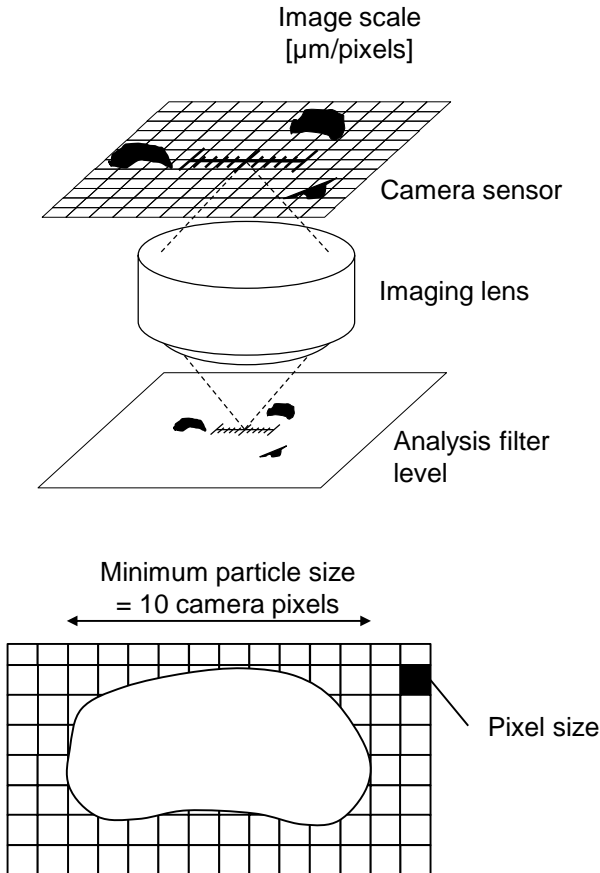
Fig. 8-3: Pixel resolution of the light-optical image

As already mentioned, in order to apply the optical method effectively to determine particle size ranges, analysis filters have to 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 analyze 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 Chapter 7.6: Verifying filter occupancy.

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 gray values varying between black and white. In the case of particle analysis, 256 shades of gray have become established. If the quantity of pixels in the image possessing specific gray values are spread out over this range, a so-called "histogram" is obtained, as shown in Fig. 8-4.

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:

1. **Light microscopes with polarizers**. With these, the light used to illuminate the analysis filter is linearly polarized by a polarizer. Light rays reflected from the test component are detected by the magnification lens and directed through a second polarization filter rotated by 90° (analyzer). 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 gray value range of the filter background (the histogram maximum) is shifted to 55% ± 5% of the total gray 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 gray values.

The binarization threshold is set at 70 % of the maximum gray value, see also Fig. 8-4. Structures to the left of this gray value threshold are recognized as particles.

Microscopes with crossed polarizers

A)

B)
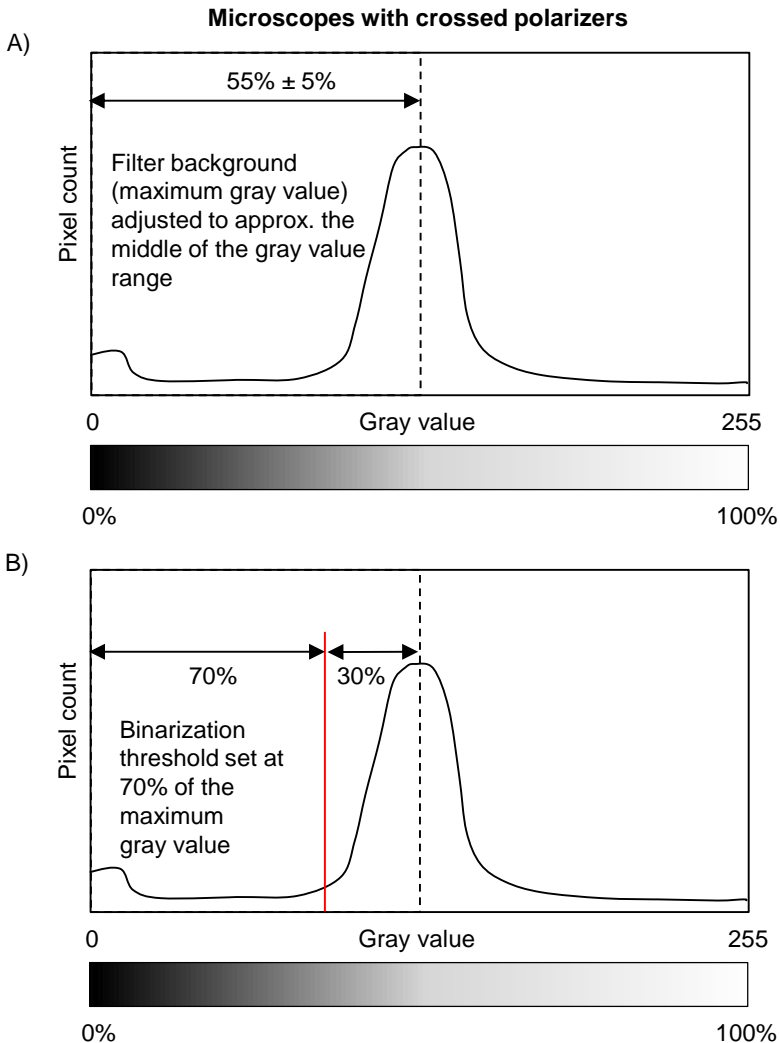
Fig. 8-4: Setting the image brightness and binarization threshold for light microscopes with polarizers

2. **Scanners 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 gray value range.

To detect particles, two binarization thresholds relative to the gray value maximum are selected, one at 70% and the other one at 145% (see also Fig. 8-5). Structures located to the left of the left-hand gray value threshold and to the right of the right-hand gray value threshold are recognized as particles. The mid-range values of the filter background are practically "cut out".

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 Fig. 8-6, image-processing methods (dilatation and erosion) are used to join up these areas of the particle again. In Figure A), an example of a particle with pale and dark areas is shown together with gray 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 1: 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 2: The targeted form of illumination used by scanners may cause reflections from the mesh filaments to occur when analyzing mesh filters, which could be recognized as (small) bright particles. Appropriate methods ("despeckle") are used to ensure that artifacts or reflections are not included in the particle results.

Scanner (without polarizers)

A)

B)
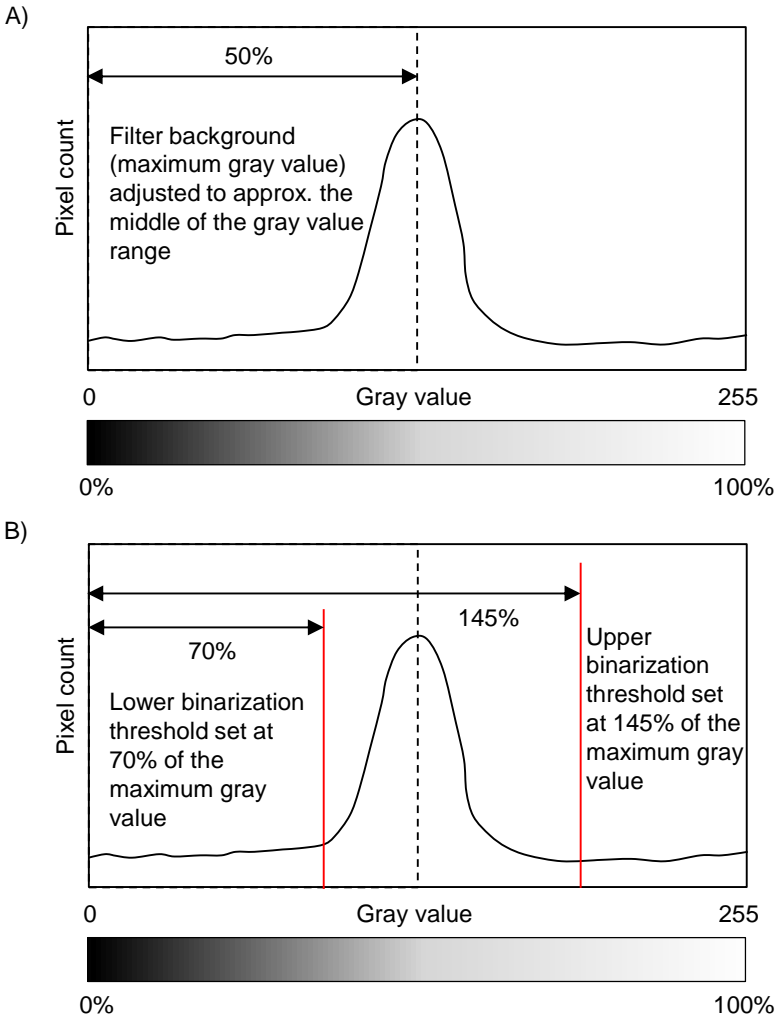
Fig. 8-5: Setting the brightness and binarization thresholds for light-optical scanner systems without polarizers
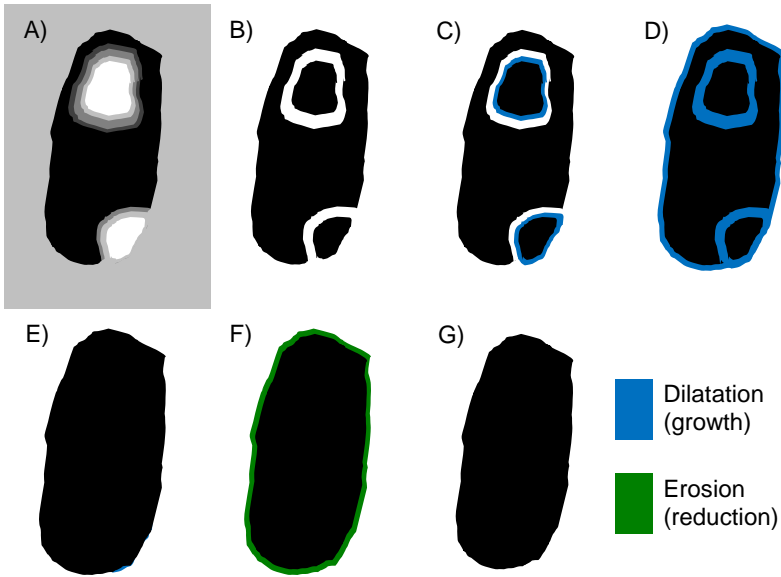
Fig. 8-6: Joining up separated particle structures with dilatation and erosion

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 the following figure, in image-processing this value is known as Feret$_{max}$. 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.
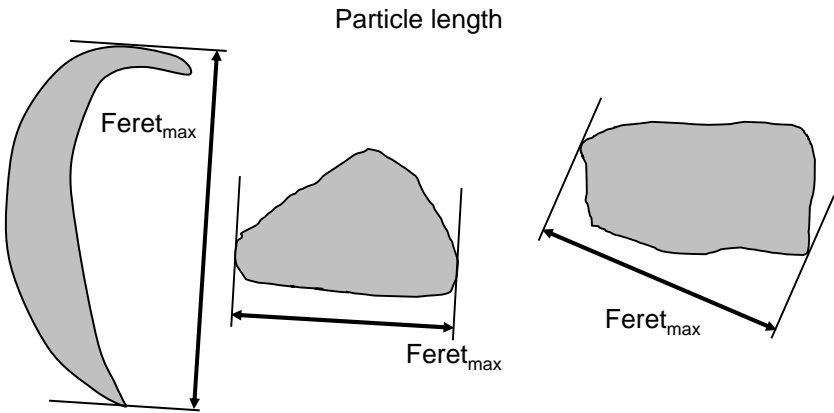
Particle length

Fig. 8-7: definition of the particle length as Feret_{max}

Particle width: the smallest possible perpendicular distance measured between two parallel lines touching the particle. Shown in the following figure, in image-processing this value is known as Feret_{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.
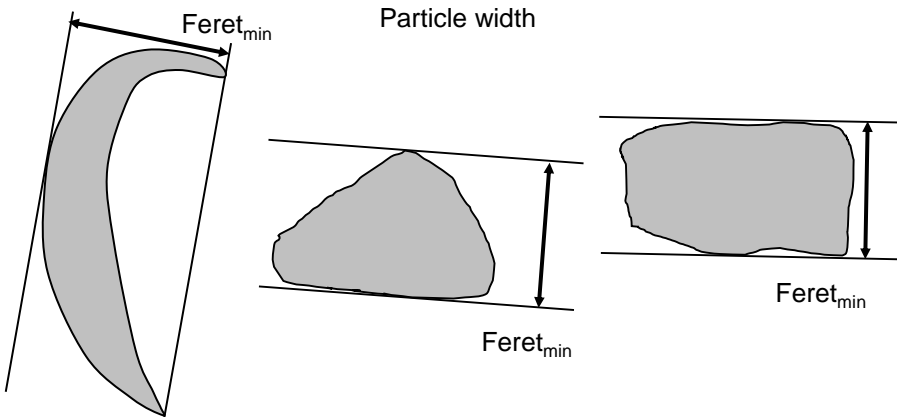
Particle width

Fig. 8-8: Definition of particle width as Feret_{min}

Note: 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°.

Annex 8.2.2.1 illustrates two additional particle widths that can also be measured in the standard analysis.

2. **Characterizing and measuring fibers**: In any manufacturing or laboratory environment where people are present, fibers are emitted from clothing materials and are later found in component cleanliness analyses. Because material fibers 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.

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

   – stretched length / maximum inner circle diameter > 20

   – width measured via the maximum inner circle diameter ≤ 50μm
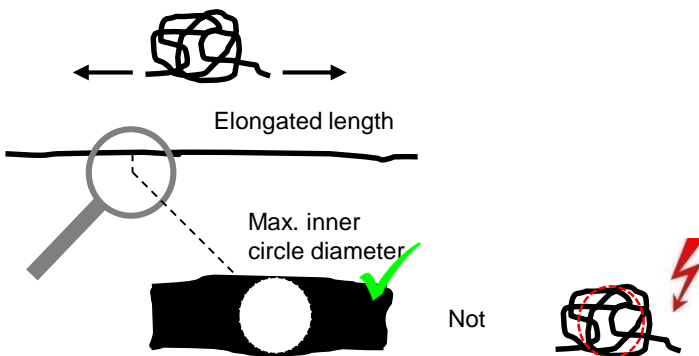
Fig. 8-9: Characterizing fibers

The length of a fiber can be measured in two ways. Firstly, in the same way as with the length of any other particle, the length of the fiber is expressed as $Feret_{max}$.

Length = Feret$_{max}$

Length = elongated length
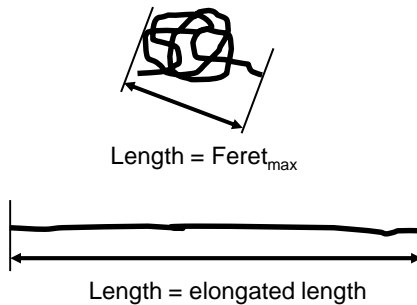
Fig. 8-10: Measuring fibers

As opposed to other particles, material fibers tend not to have a fixed shape, with the result that their orientation, position, bend 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 fiber can alternatively be expressed as the so-called "elongated length". This length, which corresponds with an untangled, elongated fiber, can be calculated using computational image-processing methods.

Warning: The elongated length of a fiber 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 fiber as a particle or vice versa, is only possible if both are measured and documented using the same size criterion.

3. **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 color, 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 analyzed 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 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 and shiny during an automated analysis. At the time this version of VDA 19.1 went to press, it was 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 analyze 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 must 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.

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 Chapter 7.6 and Annex A 7.6 + A 7.7), 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 may also occur when different types of particle overlap, e.g. if a small metallic shiny particle touches a fiber. 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 are to be documented and saved to enable later transparency.

Material and equipment

1. Light-optical system, e.g. material microscope, zoom microscope, stereo microscope or flatbed scanner, featuring the components and characteristics described in the section **"Detecting, measuring and characterizing particles with the aid of light-optical standard analysis"**.

2. 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 must 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. Artifacts due to reflections from the filaments of mesh filters, for example, are to be avoided.

   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).

3. Sample holder: with automated light-optical analysis, the analysis filter must be secured so that it does not slip out of place when the sampling table is moved. All areas of the analysis filter and particles contained on it must 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

160

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 must be secured with a glass lid.

Note 1: If, for example, the analysis filter is inspected in a tensioning device without a lid, the environment must be sufficiently clean to ensure that the analysis filter is not contaminated by further particles or fibers in the specified size range during the analysis. The tensioning device may not cover any part of the effective filter surface.

Note 2: 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.

Note 3: 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.

Note 4: If an analysis filter that has translucent areas, such as the mesh of a coarser membrane filter, is analyzed using a light-optical method, the sample holder should be of a similar color 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.

4. Motorized positioning unit: positioning axes need to be extremely accurate for the following reasons:

   – In an automated analysis, the effective filter surface has to be fully analyzed without it slipping out of place (see also Annex A 8.2.2.2 and 8.2.2.3)

   – Particles have to 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 sampling table has a wider range of movement, filter mounts can be realized that hold several analysis filters, which can then be subsequently analyzed automatically in succession (the focal plane over horizontal distances must remain identical).

5. Camera: the number of pixels of the camera sensor (in the case of flat-bed scanners, the line camera) must be adapted to the optical resolution of the magnification lens, i.e. the 10-pixel criterion is to 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 must be operated with defined, fixable sensitivity settings. Automatic functions that correct the brightness are to be deactivated.

6. System control and image processing: the programs used to control the system and analyze the images must possess the features described in the section **"Detecting, measuring and characterizing particles with the aid of light-optical standard analysis"**.

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.

Procedure

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

1. Procure all resources required for the test

2. 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 is to be secured with a glass lid, verify the state of the lid and clean it beforehand if necessary.

Note 1: Conditioning is not required if analysis filters are inspected only by light-optical analysis and not gravimetry.

Note 2: Unlike with gravimetry, filters for light-optical analysis do not have to be dried until weight constancy is attained. The cooling 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.

3. Fix the sample holder onto sampling table (or place the sample holder in the flatbed scanner)

4. 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.

5. Where appropriate, check that the sample and sampling table are level

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

7. Perform the automated analysis

8. Verify the analysis results in accordance with Section "**Double-check"**

9. Document the results

Documentation

See Chapter 9 Documentation and also Chapter 12 Case examples

Annex 8.2.2   Light-optical analysis

A 8.2.2.1   Other (optional) particles widths measured using light-optical standard analysis

Maximum inner circle diameter

Largest vertical cross-section
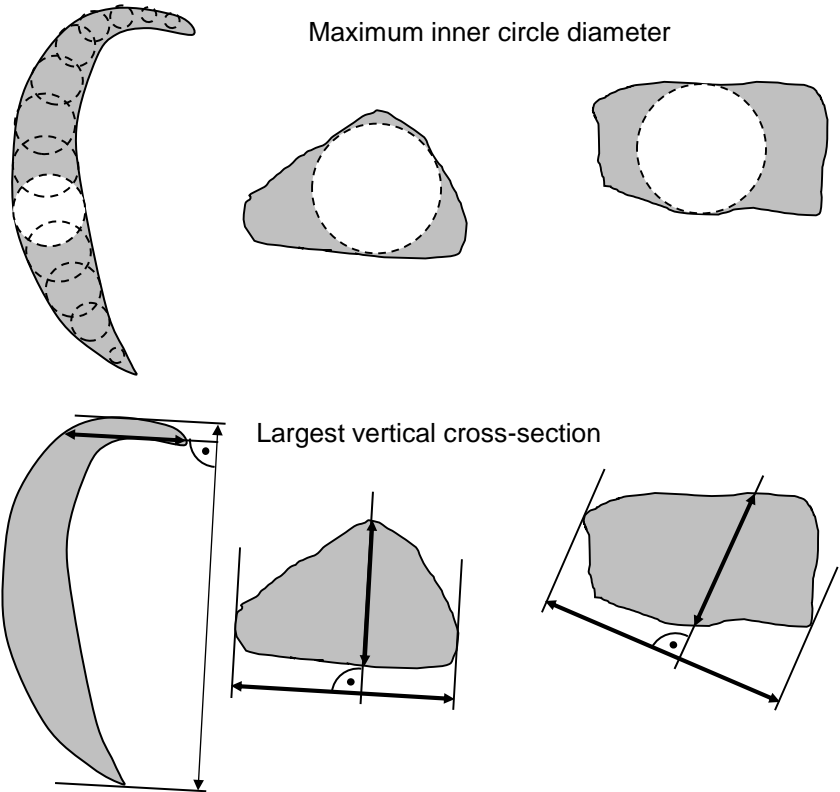
Light-optical standard analysis can also be applied as an option to determine two other particle widths:

– the **maximum inner circle diameter**: this identifies the damaging potential of a particle, or the size of the narrowest channel it can still pass through.

– the **largest perpendicular cross-section**: this is determined perpendicular to the longest dimension measured and cannot be directly linked to the damaging potential of a specific particle size.

A 8.2.2.2 Terminology regarding analysis membranes
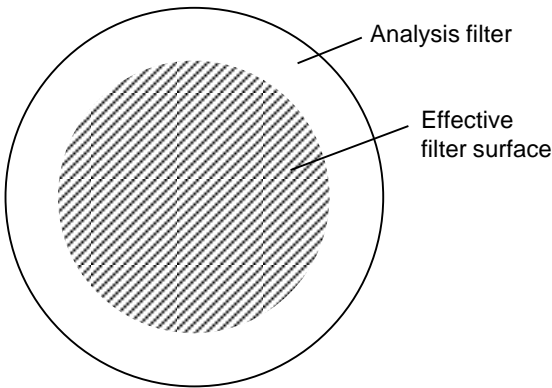
The effective filter surface is the area onto which particles are deposited that is wetted during the filtration process.
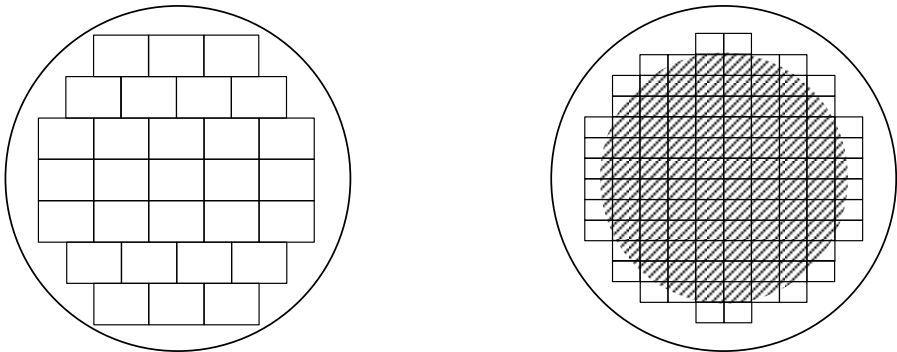
The measuring fields are to be located to enable the entire effective filter surface to be analyzed. The diagram shows two examples of microscope grids: the one on the left for a lower degree of magnification and the one on the right for a higher degree of magnification. A linear grid is formed with scanners because they have line cameras.

Note: Occasionally, due to errors in the filtration process or during handling, some particles may be deposited outside the effective filter surface. Where possible, these particles are also to be included in the analysis.

A 8.2.2.3   Particle reconstruction

To avoid particles projecting over the edge of an image field from being excluded in the analysis, counted twice or only measured in part, the light-optical analysis system must be capable of reconstructing such "margin particles" to their full shape and of measuring and characterizing them on the basis of the complete particle. To achieve this, image analysis and the axis control unit need to cooperate with one another, the sampling table has to be accurate and motorized and the translation axes of the sampling table and the camera must be finely adjustable. The following figure b) shows a typical error that occurs if the translation axes of the camera and sampling table are not optimally aligned with one another, or if sizes are calibrated inaccurately.

a) Correct particle reconstruction
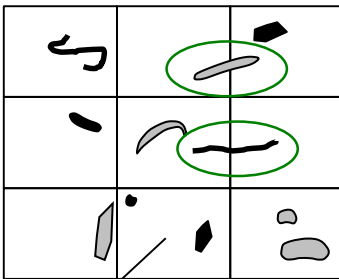
b) Incorrect particle reconstruction
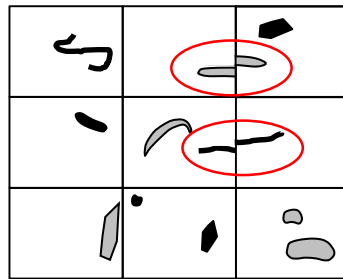
Single image field

A 8.2.2.4   Calibration and maintenance

To calibrate magnification, a specific unit of length in the sampling plane (object scale) is assigned to the corresponding unit of length on the sample image (in the camera sensor or overall image, or eyepiece scale in the case of manual measurements) (see also Fig. 8-3).
The calibration process must be repeated for all degrees of magnification used to analyze the filter. Where systems have a zoom function, e.g. stereo microscopes, the degrees of magnification have to be lockable in a defined position.

A so-called object scale can be used (glass plates with graduations marked on them) to perform a simple calibration of length.

To verify other basic functions of automated light-optical analysis systems, such as the accurate automated measurement of length, the orientation of the camera in relation to the sampling table or the structure of image fields, as well as their correct magnification, so-called particle standards can be implemented. These are generally glass substrates marked with defined geometric particle structures in full black/white contrast.

The particle standards used must be certified and traceable.

It is not possible to compare light-optical analysis systems with one another using particle standards with ideal geometries and contrasts because brightness settings and binarization thresholds cannot be assessed in this way.

A 8.2.2.5 Functioning principle and limitations of light-optical analysis

In the analysis of particles using light optical microscopes, only particle structures with values below the binarization threshold are detected (darker than the filter background).

Therefore, in order to detect metals, crossed polarizers are used; these extinguish shiny areas on particles and make the particles appear slightly darker. This generally enables effective detection (see Graph A).

Other materials, such as abrasives, plastics and textile fibers, usually have a broad spectrum of colors and levels of brightness. This makes it impossible to accurately detect bright particles or bright parts of particles (see Graphs B), C) and D)).

Metal particle → Crossed polarizers

A)

Metals
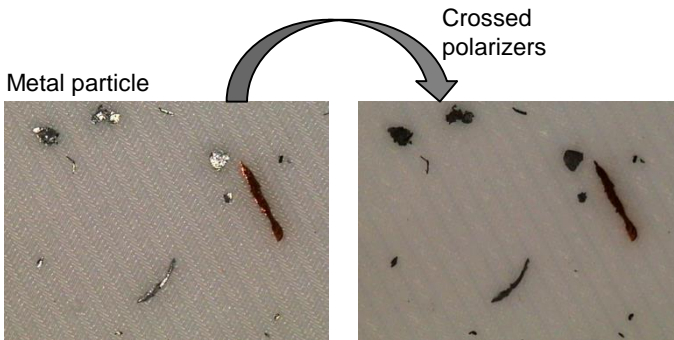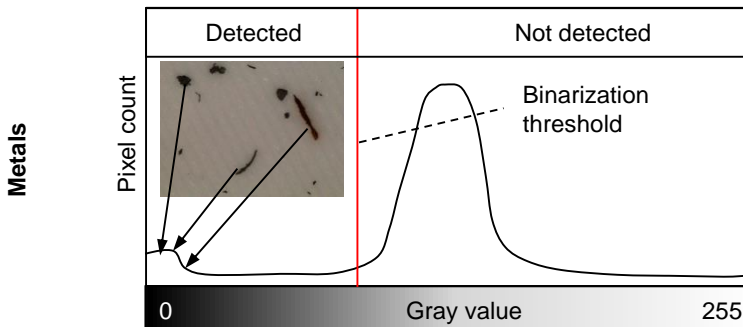
B)
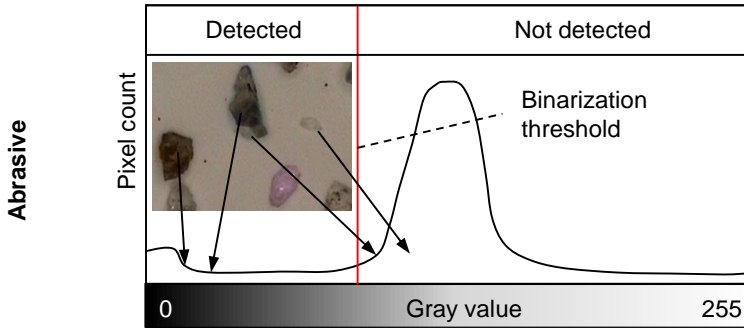
C)
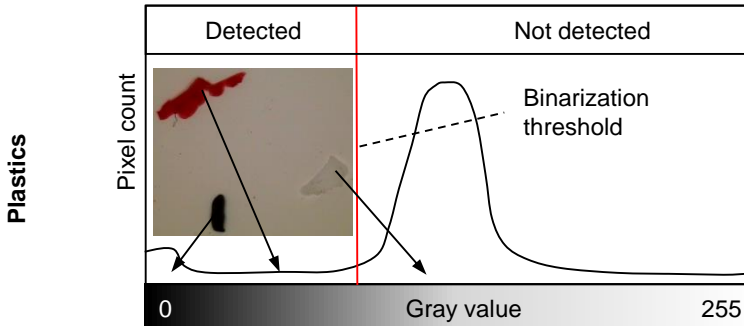
D)
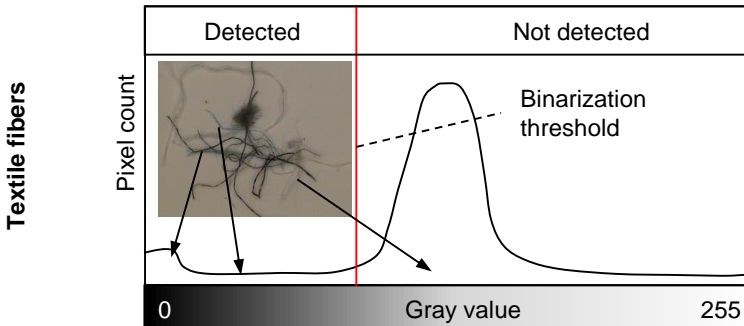
8.3 Extended analysis

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 is to 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 should be implemented.

8.3.1 Further light-optical analyses

Optical features

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

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.

170

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

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 x -> 5 x -> 10 x -> 20 x), 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 x (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 x the depth of field.

To measure 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 traveled by the lens in the z-direction. It is calculated indirectly via the screw pitch and stepmotor of the z-axis (see Fig. 8-11).

Particle height $z = z_2 - z_1$
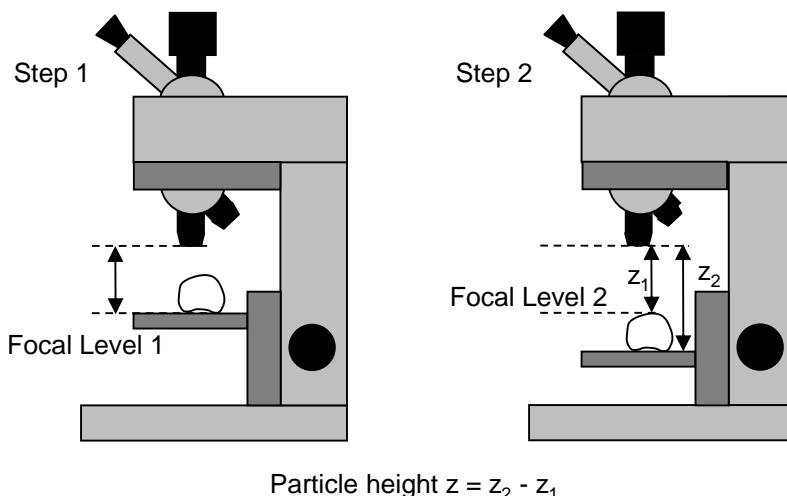
Fig. 8-11: Principle of measuring particle height

Note: In order to best use the method for measuring 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 is to be taken to ensure that the particle does not "protrude" into the pore structure as this would result in a lower height being measured.

Material and equipment

- Light microscope with parallel beam path
- 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)
- High-resolution motorized fine focus (resolution in sub μm-range)
- Software to read the focus positions
- 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.

Procedure

- General analysis with a low-magnification lens, e.g. 2.5 x or 5 x (to determine two-dimensional information about particle length, width, surface area, etc.)

- After the scanning step, measure the height of critical particles using a lens with a higher degree of magnification (numerical aperture > 0.4).

- The particle concerned is automatically brought into focus, selected and memorized

- Bring the lens with the higher degree of magnification into position

- Adjust the contrast to enhance particle topography

- Focus on the background of the analysis filter near the particle to be analyzed

- Confirm this 1st focus position

- Software reads the 1st focus position

- Focus on the highest point the particle

- Confirm this 2nd focus position

- Software reads the 2nd focus position

- Analyze the focus positions to determine the difference in height

- Document difference in height in overall results.

Documentation

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

The results obtained are to 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.

8.3.2   SEM/EDX

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 guideline 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 has to be inspected.

Note: 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 analyzed must be representative.

Again, as with light-optical analysis, in order for particles to be recorded, measured and analyzed correctly, the analysis filter has to 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.

Warning: 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 the following figure.

Fixed position of electron beam: not suitable for correct particle analysis

Correct analysis by measuring the full surface area of the particle e.g. with the electron beam moving along a grid structure
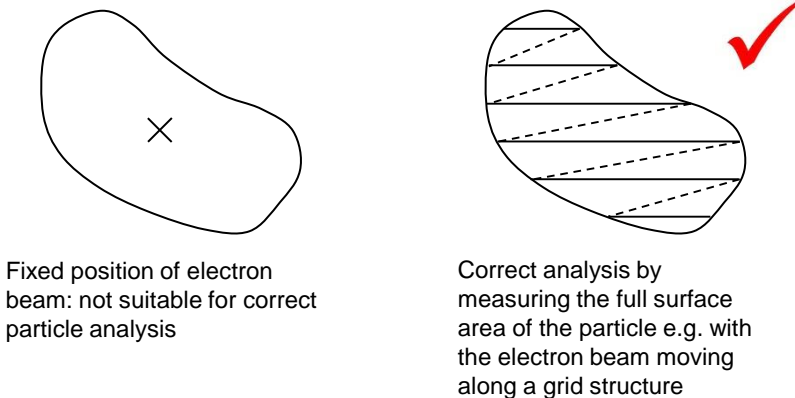
Fig. 8-12: Movement of the electron beam in EDX analysis

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 and 25 mm).

Note: As high numbers of particles are generally analyzed 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 are 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.

Material and equipment

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

Procedure

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

- 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.

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

- The z-position: set the height of the sampling table so that the analysis filter is located at the working distance specified for the system.

- 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 is to be documented.

- 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.

- If necessary: warm the cathode to stabilize the beam current

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

- Start the automated analysis

Documentation

See Chapter 9 Documentation

Annex 8.3.2   SEM/EDX

A 8.3.2.1   Material classification

In order to classify detected particles into material groups, the SEM/EDX system must be capable of establishing the necessary links. The following table gives examples of how specific percentages of elemental compositions are assigned to a material class.

Material Class	Element	Amount in %
Steel A	Fe	>95
	Mn	<1
	Si	<1
Steel B	Fe	>85
	Cr	<10
	Ni	<5
	Mn	<1
	Si	<2
Steel C	Fe	50-80
	Cr	10-40
	Ni	0-15
	W	1-10
	Mo	1-10
	V	1-10
Brass	Cu	20-80
	Zn	20-80
Bronze	Cu	20-80
	Sn	20-80

The material classification made should then be confirmed by analyzing known material samples (e.g. typical alloys from component manufacturing processes).

Note: If required, different material classes can be quantified together with regard to their physical characteristics, for example (conductivity, hardness, abrasiveness).

A 8.3.2.2 Calibration

Systems should be calibrated (measurement of length in the case of SEM, measurement of energy with EDX) once a year, or if a major intervention is carried out, such as an adjustment or modification.

A 8.3.2.3 Maintenance

Especially in cases where samples emit gases and/or chamber pressures are too high, all components inside the vacuum system of the SEM, e.g. sampling chamber, detectors, etc., could become contaminated. These components are to be regularly cleaned at specific intervals or if the image quality becomes impaired.

Components subject to aging or wear, in particular parts of the electron lens such as the cathode, are to be replaced or renewed either as required or in compliance with the manufacturer's recommendations.

A 8.3.2.4 Material contrast and filter occupancy

As opposed to light-optical systems, the brightness contrast in an image is not determined by the optical characteristics of particles but rather by their material contrast. In consequence, heavy elements appear bright and lightweight elements appear dark.
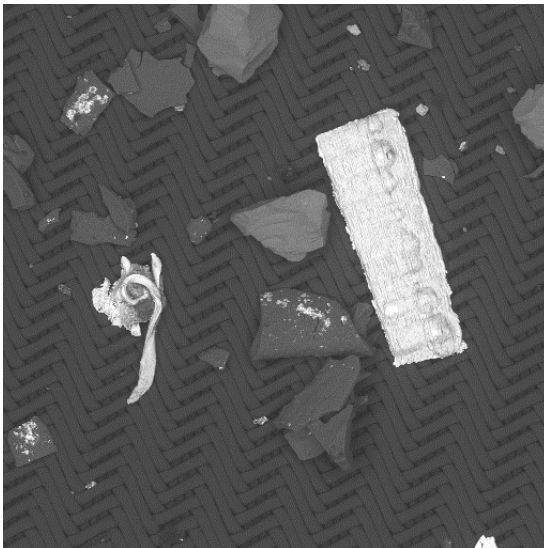
In the same way as with light-optical systems, if filter occupancy is too high measurement errors may occur due to particles lying too close to or on top of one another. The following figure shows a metallic particle that is optically divided into two because of an organic fiber lying over it.

300µm
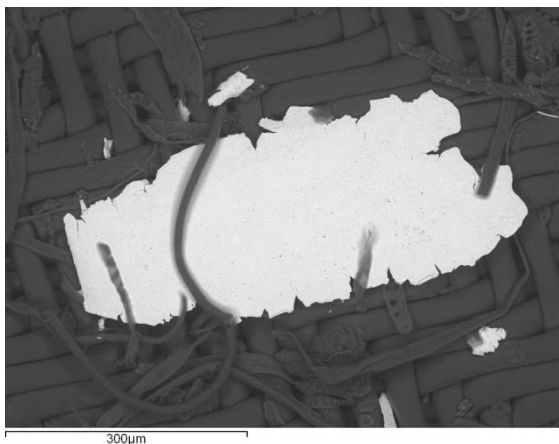
8.3.3 LIBS

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 analyzed 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 and 1064 nm. The laser pulse vaporizes the material and generates plasma. Depending on the system, the wavelength range inspected lies between 400 and 800 nanometers. Depending on the measuring system implemented, the resolution of the spectrum, i.e. the quantity of points per spectrum, lies between 0.01 and 1 nanometer.

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 analyzing particles with a minimum size of approx. 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 approx. 10 μm is analyzed 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.

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 1064 nm.

Systems with an integrated microscope are capable of automated particle detection (see light microscopy) and determine the coordinates of a particle's center 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 analyzes 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 1000.

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, 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 analyze deeper layers.

Documentation
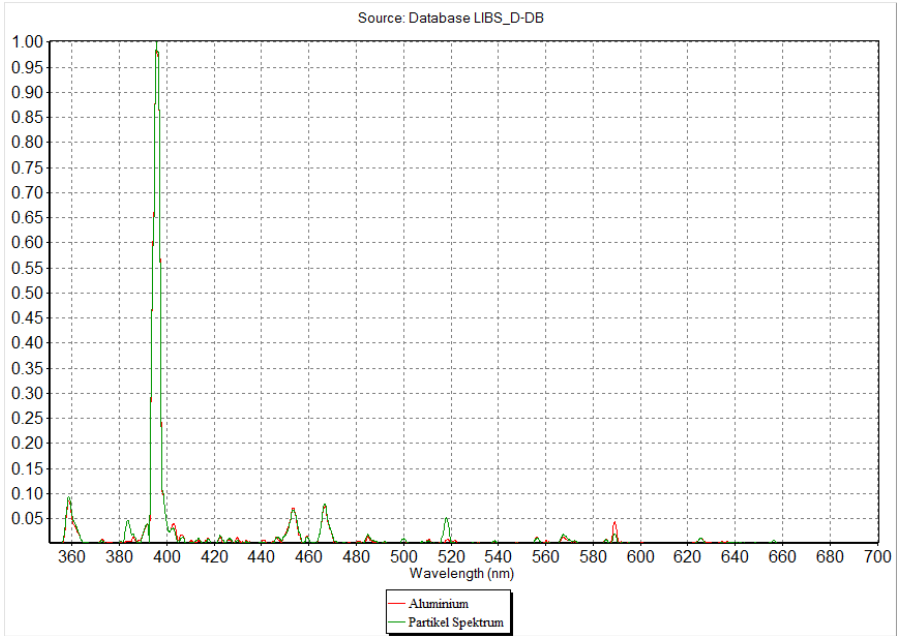
Fig. 8-13: Comparison of LIBS spectrum for aluminium (green) with the particle spectrum. Match: 996 points, quality of the spectrum: 171, laser 331 nm

A 8.3.3.1   Diagram of a spectroscopic set-up which can be used for LIBS analysis and Raman spectroscopy
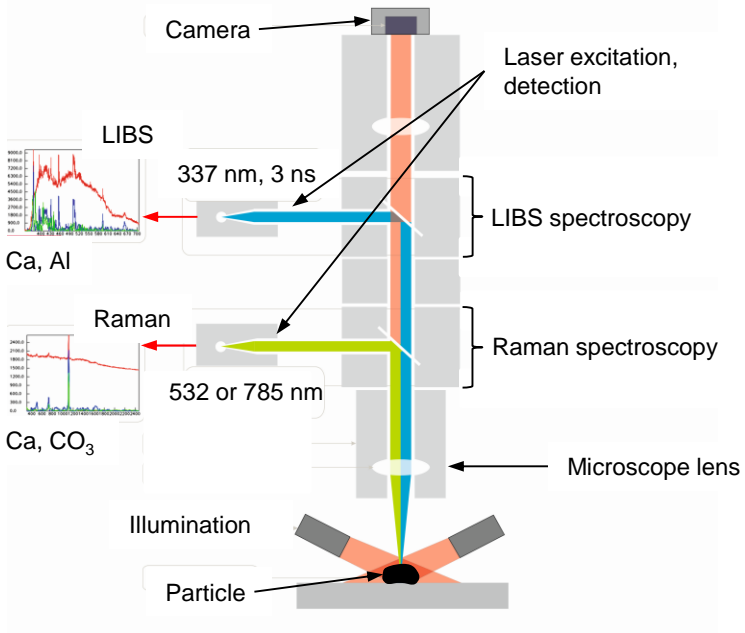
8.3.4 Raman spectroscopy

Principle

Raman spectroscopy is a form of oscillation spectroscopy, which enables the structure of a particle to be analyzed. 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 (Charge Coupled Device). The signal is known as a spectrum. Depending on the system, the wavelength range considered lies between 400 and 3500 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 analyzing 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 and 8 µm.

Typical analysis times per particle range between 30 and 60 seconds. In some cases, fluorescence may be excited by the laser beam. This could overlay the Raman signal and hamper classification.

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 center 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 (Charge Coupled Device). A microcomputer analyzes 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 1000.

Procedure

The operator places the test filter 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 to 60 seconds 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.

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 operator can adapt the laser output and exposure time to the respective particle material.

Documentation
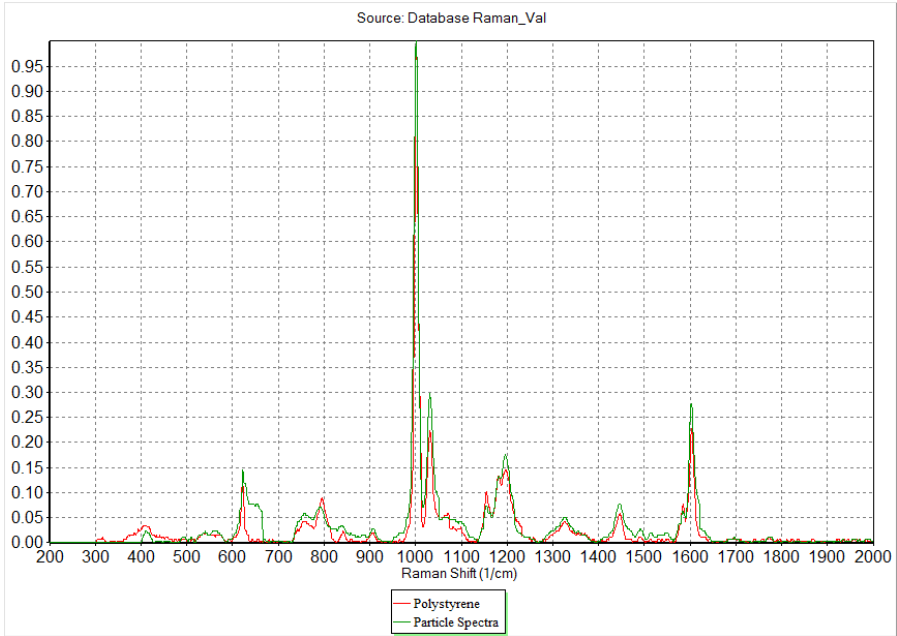
Fig. 8-14: Comparison of Raman spectra for polystyrene (green) with particle spectrum. Match: 966, quality of spectrum: 101. Exposure time: 60s, laser 785 nm, intensity 30%

For a diagram of a Raman system, see Annex A 8.3.3.1

8.3.5   IR (infrared spectroscopy)

Principle

If organic molecules are bombarded with electromagnetic waves in the infrared range (wave number 4000 - 400 cm^{-1} or wavelength approx. 2.5 – 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 Section 8.3.4).

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 analyzing particles: 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: here, the IR beam is coupled <u>and</u> 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 1-3 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.

Material and equipment

- FT-IR spectrometer or FT-IR microscope linked to a databank.

- If required: tweezers or needle to transfer the particle from the analysis filter to the analysis device

- Where appropriate: diamond pressing cell (with transmission)

- To transfer individual particles from the analysis filter, a stereo microscope can be used, for example.

Procedure

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

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 Fig. 8-15).

As well as listing all the equipment used in the test, precise details of the IR-spectrometer and associated microscope (manufacturer, model, etc.) are also to be included in the report.
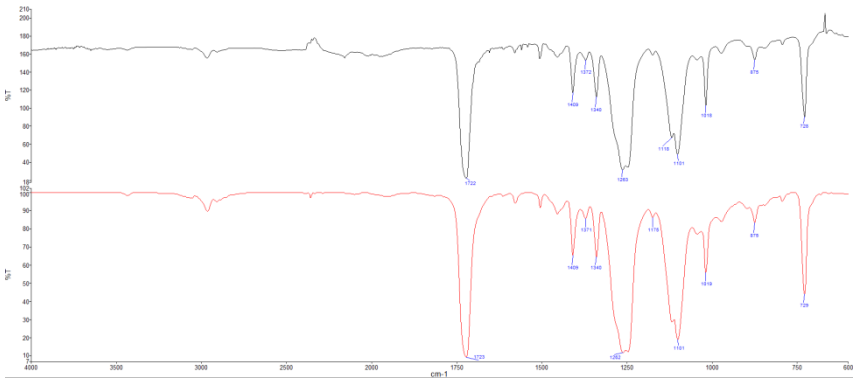
Fig. 8-15: IR-spectrum of PET (black, top) and nearest match in the data library (red, bottom)

Bibliography

- IR-Spektroskopie, Eine Einführung (Helmut Günzler, Hans-Ulrich Gremlich), ISBN 978-3-527-30801-9, Wiley-VCH

- IR-Spectroscopy (Helmut Günzler), ISBN 978-3-527-28896-0, Wiley-VCH

- Course notes on the interpretation of infrared and Raman spectra (Dana W. Mayo, Foil A. Miller, Robert W. Hannah), ISBN 978-0-471-24823-1, Wiley

8.3.6　X-ray microtomography

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 diagram). During data capture, the test component is rotated step-by-step and an x-ray image recorded at each new angle.
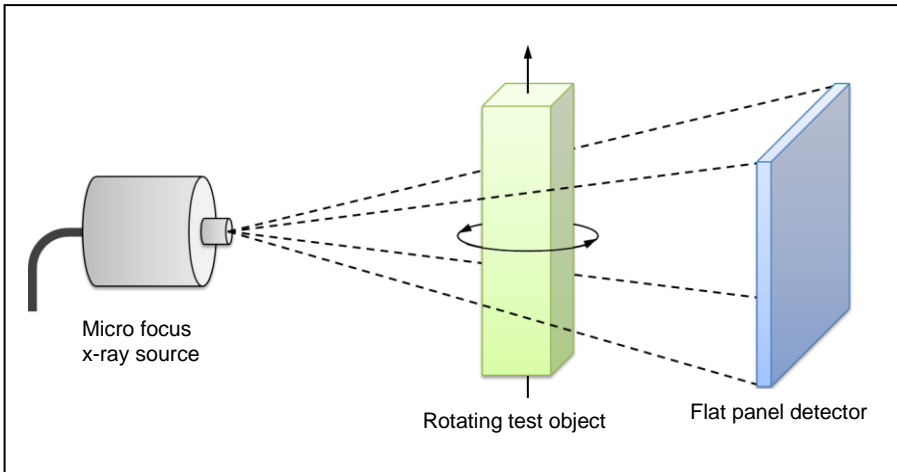
Fig. 8-16: Principle of computer tomography

Using a computer-aided method according to Feldkamp, the x-ray attenuation from all the projection images is reconstructed to form a 3-D image (computer tomography). As the attenuation corresponds approximately with the density of the material, the reconstruction represents the spatial distribution of density. The reconstructed gray value gives an indication of particle density. By applying image-processing methods, the geometric structure of the test component can be calculated from the density distribution.

The test object analyzed is a suitably-prepared filter membrane charged with particles. By means of segmentation, particles are separated from the background and then analyzed individually. In the analysis, not only is the geometry of a particle measured 3-dimensionally, but the analysis of the gray 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).

Material and equipment

To analyze micro-particles, a high-resolution x-ray microtomography system is required ("micro CT"). The system must 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 analyze 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 analyzed by segmenting the reconstructed gray value data with suitable software. The software also has to be able to measure the particle's dimensions of interest (length, thickness, elongation, surface volume, bounding box, density index, etc.) from the segmentation.

Procedure

1. Particle fixation / compaction of analysis filter

2. X-ray scan and reconstruction

3. Segmentation and particle analysis

4. Compilation of report

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.

8.4 Shortened analysis

In this section, 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 analyzed 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.

8.4.1 Liquid particle counter

Principle

Extinction particle counters (optical particle counters / OPC) 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 frequency of particles sized between 5 µm and approx. 200 µm.

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 realtime particle-counting or sample analysis can also be programmed for immediate further processing in data capture systems.

Due to the detection principle, the test liquid has to flow through the particle sensor with a defined volume flow. With "standing" samples, such as liquids contained in a beaker after extraction by ultrasound, 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 Fig. 8-17 for an example).

Particle to be measured

Circle with equivalent surface area

Surface area = 490 µm²

Length = Feret$_{max}$ = 50µm

Length = D= 25µm
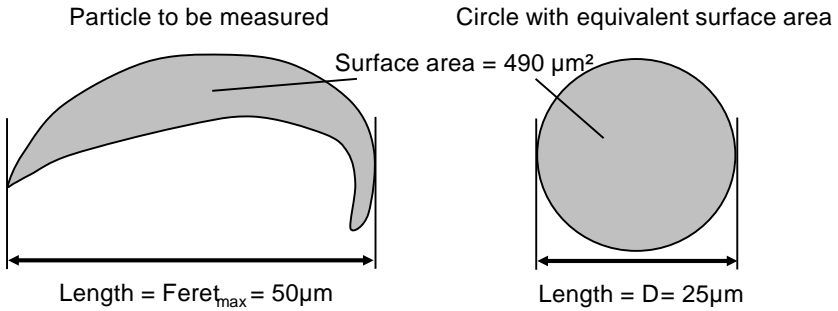
Fig. 8-17: Diameter of a circle with an equivalent surface area as a measure of particle size

The more a particle is shaped like a thin chip or fiber, the greater the deviation between the particle size displayed by the particle counter and the Feret$_{max}$ length determined.

Gas bubbles or droplets of foreign liquids in the test liquid are (falsely) identified and documented as solid particles.

If using an OPC 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 analysis liquid or liquid sample are analyzed. The statistical application is based on the assumption that particles are distributed fairly homogeneously in the analysis 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 must be taken into consideration (dilution of the particle load).

Although the analysis devices are easy to operate, special care is to be taken when preparing and feeding the analysis 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 chambers in the future; this will make it possible to analyze extraction liquid in so-called realtime without the need for manual sample preparation steps.

To calibrate OPCs, ISO-MTD in oil (Mean Test Dust) 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.

Material and equipment

As well as the analysis device and corresponding sample feed unit, sealable sample containers, beakers and filtered extraction liquid are required. The filtered liquid is used to condition the system before use and determine its blank value. If particle concentrations in samples exceed the measuring range of the OPC, this liquid is also utilized to dilute the sample in a defined manner. Protective eyewear and gloves may also be required. An ultrasound 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).

Procedure

The sample is analyzed in compliance with standards such as **U.S.P Pharmacopeia** 788 **PARTICULATE MATTER IN INJECTIONS 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 analyzed, all standing samples must 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 must 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 volume flow is usually necessary.

Documentation

The following information is to 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 actual particles.
- When listing absolute quantities (not concentrations), the volume of liquid actually analyzed is to be stated
- When giving information about concentrations, the original volume of liquid used in the extraction step must be stated
- Volume analyzed expressed in percent in cases where the sensor effectively only detects/analyzes a fraction of the throughflow liquid.
- Dilution factor for highly-concentrated samples
- Total volume of liquid analyzed
- Coincidence concentration.

8.4.2 Filter-blocking (optical)

Principle

With this analysis method, particles from the flow of liquid (extraction medium containing particles) are deposited directly on an analysis filter that is situated in the field of view of a camera. 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 extraction apparatus 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 analyzed. 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.
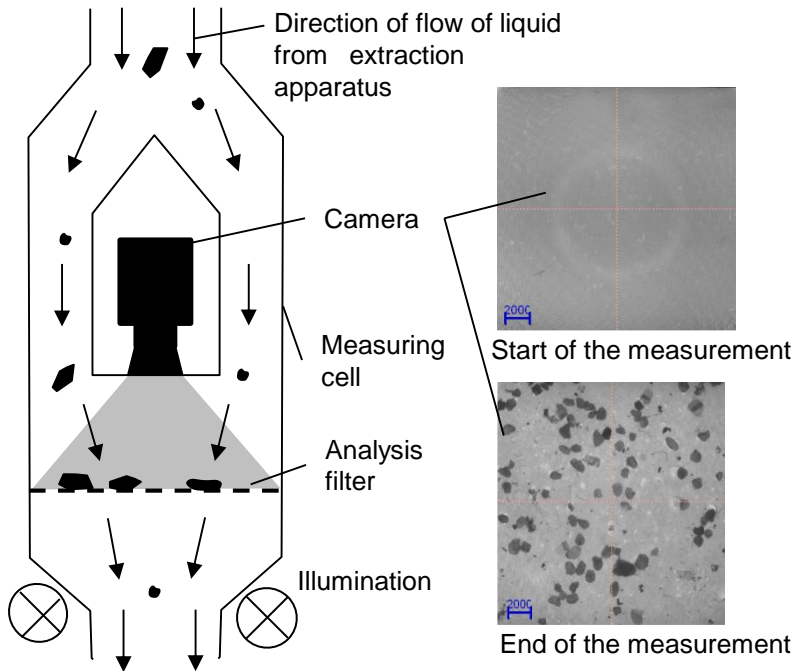
Fig. 8-18: Cell for the optical analysis of the filter blockade

Material and equipment

- Measuring cell with:
    - camera
    - analysis filter
    - (transmitted light) illumination
    - system for backwashing the analysis filter
    - image processing and analysis
- Adapter to extraction apparatus and return feed of extraction media

Procedure

The following procedure is to be adapted to the features of the respective extraction apparatus and analysis system.

- Record and analyze an image of the analysis filter to assess its initial state

- 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.

- Record and analyze an image of the analysis filter after the extraction step

- The difference between the two analyses equates to the particle load extracted from the test component.

- Carry out the next extraction or backwash the analysis filter if particle occupancy is too high.

Documentation

Particles are analyzed and documented in accordance with the procedure for light-optical analysis and grouped into the particle size classes stated in Chapter 9 Documentation.

9 DOCUMENTATION

9.1 Principles

Different documents are generated in the course of a cleanliness inspection (see Fig. 9-1). 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 test (declining series), with the latter being used to derive the routine inspection procedure.

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 third document, 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. Further references (e.g. supplementary agreements) are optional.

At least, the formal correct document must contain all the information mentioned in the sub-chapters. Further information is to be added as required.
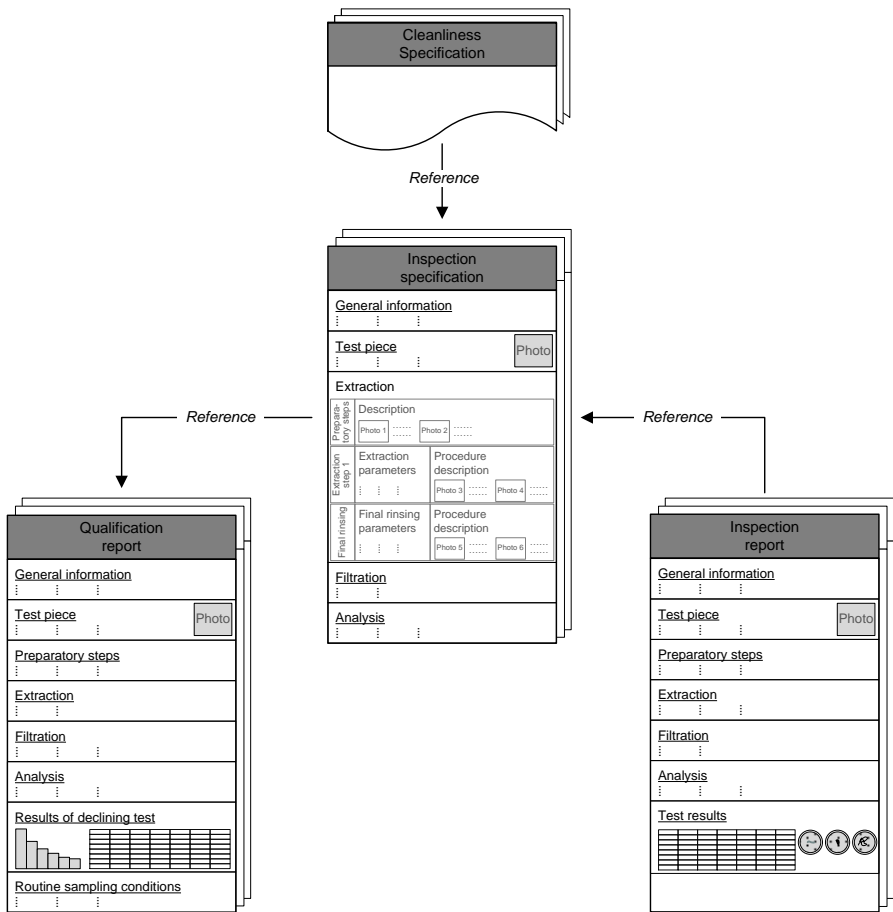
Fig. 9-1: Cleanliness inspection documents (overview and connections to one another)

9.2 General information

The section "General information" contains higher level information to aid the classification and administration of the documents supplied.

General information			
Order No.: ……………………………		Report Nor.: ……………………………………	
Customer: ……………………………		Date of analysis: ……………………………………	
		Contact partner: ……………………………………	
Test ordered: ☐ Routine inspection ☐ Double inspection ☐ Qualification test			
Reason for test: ☐ Initial assessment ☐ Process control ☐ Modifications, constructional ☐ ………………			

9.3 Information about the test piece

In this section, the test component is described in more detail and identified clearly. Additional information which may be useful could include:

- The material the test component is made from,
- The presence of filmy residues on the test component

Test piece			
Description: ……………………	Condition on delivery: …………………	Photo on delivery	Photo test piece
Parts Number: ……………………	Test lot size: …………………		
Batch Number: ……………………	Control area: …………………		
Date removed: ……………………	A_C = ……… cm²		
Time removed: ……………………	V_C = ……… cm³		
Removed from: ……………………	Inspection specification: …………………		

9.4 Information about preparatory steps

Information about preparatory steps indicates if and when supplementary work steps are required. If no information about preparatory steps is mentioned, test results could be wrongly evaluated or misinterpreted.

Preparatory steps	
☐ None	☐ Isolate control areas (e.g. sealing, masking)
☐ Disassembly	☐ Preclean contaminated surfaces not relevant to control area
☐ Demagnetization	☐ ……………………………………………………………………

9.5 Information about the extraction

The way the extraction step is performed may vary significantly according to the expertise of inspection staff involved. In this section, the degree of freedom regarding the extraction step is reduced by documenting the inspection procedure as clearly as possible. This aims to make the extraction step reproducible and improve the comparability of test 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 environment:	☐ Undefined
	☐ Test piece inclusive packaging		☐ Cleanliness Room Grade (VDA 19-2)
			☐ Cleanroom Class (ISO 14644-1): .....

Extraction method:	☐ Pressure rinsing	☐ Internal rinsing	☐ Air jet
	☐ Ultrasonics	☐ Agitation	☐ Air throughflow

Solvent: Liquid .... Duration .... Temperature ....

9.5.1 Pressure rinsing	9.5.3 Internal rinsing	9.5.5 Air jet
....	....	....
....	....	....

9.5.2 Ultrasonics	9.5.4 Agitation	9.5.6 Air throughflow
....	....	....
....	....	....

Final rinse, Control Area 1

Manufacturer, extraction device: ....
Model, extraction device: ....

Manufacturer, nozzle: ....
Type, nozzle: ☐ Round jet ☐ Parallel jet
  ☐ Flat jet ☐ ....
Dimensions, nozzle: ....

Extraction 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

Final rinse, Control Area 2

Manufacturer, extraction device: ....
Model, extraction device: ....

Manufacturer, nozzle: ....
Type, nozzle: ☐ Round jet ☐ Parallel jet
  ☐ Flat jet ☐ ....
Dimensions, nozzle: ....

Extraction 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

9.5.1 Pressure-rinsing

Pressure-rinsing has the highest number of influencing parameters. This should therefore be taken into account in the documentation. Among other things, the cleaning effect of pressure-rinsing is influenced by the (pressure-rinsing) 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 borings* for a parallel jet nozzle

Additional information can be documented as follows:

Pressure-Rinsing	
Control Area 1	**Control Area 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: ....
Extraction liquid: ☐ Solvent .... ☐ Neutral cleaner .... ☐ Other	Extraction 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

9.5.2 Ultrasonics

With ultrasonics, 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.

It is not sufficient to note the ultrasound output because this depends on the filling level of the ultrasound device. Instead, information about the power density (output per filling volume) should be given.

Ultraschall			
Control Area 1		**Control Area 2**	
Manufacturer, extraction device:	....	Manufacturer, extraction device:	....
Model, extraction device:	....	Model, extraction device:	....
Extraction liquid:	☐ Solvent	Extraction liquid:	☐ Solvent ....
	☐ Neutral cleaner		☐ Neutral cleaner ....
	☐ Other		☐ Other ....
Filling level, US bath:	 l	Filling level, US bath:	 l
Filling level, beaker:	 l	Filling level, beaker:	 l
Frequency:	.... Hz	Frequency:	.... Hz
Power density:	.... W/l	Power density:	.... W/l
Duration:	.... min	Duration:	.... min

9.5.3 Internal rinsing

Internal rinsing may take different forms. 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 extraction liquid flows through the control area in one direction with a periodically-changing flow rate, this is known as pulsation. The pulsation frequency is dependent on time and is to be stated.

Internal rinsing			
Control Area 1		**Control Area 2**	
Manufacturer, extraction device:	....	Manufacturer, extraction device:	....
Model, extraction device:	....	Model, extraction device:	....
Manufacturer, nozzle:	....	Manufacturer, nozzle:	....
Type, nozzle:	☐ Round jet ☐ Parallel jet	Type, nozzle:	☐ Round jet ☐ Parallel jet
	☐ Flat jet ☐		☐ Flat jet ☐ ....
Dimensions, nozzle:	....	Dimensions, nozzle:	....
Dimensions, adapter:	....	Dimensions, adapter:	....
Extraction liquid:	☐ Solvent	Extraction liquid:	☐ Solvent ....
	☐ Neutral cleaner		☐ Neutral cleaner ....
	☐ Other		☐ Other ....
Volume flow:	.... l/min	Volume flow:	.... l/min
Volume of liquid:	 l	Volume of liquid:	 l
Duration:	.... min	Duration:	.... min
Pulsation frequency:	.... s^{-1}	Pulsation frequency:	.... s^{-1}
Volume flow, max:	.... l/min	Volume flow, max:	.... l/min
Volume flow, min:	.... l/min	Volume flow, min:	.... l/min
Reverse flow rinse:	....	Reverse flow rinse:	....

9.5.4 Agitation

Agitation is essentially a manual extraction method. The frequency and amplitude of the arm movements can neither be fixed nor measured but are to be approximated as best as possible in the documentation. Here, the term frequency describes the up-and-down and side-to-side movement of the test piece. 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		
Control Area 1		**Control Area 2**
Manufacturer, extraction device:		Manufacturer, extraction device: ....
Model, extraction device:		Model, extraction device: ....
Extraction liquid: ☐ Solvent .... ☐ Neutral cleaner .... ☐ Other		Extraction liquid: ☐ Solvent .... ☐ Neutral cleaner .... ☐ Other ....
Filling volume: l		Filling volume: l
Frequency: .... Hz		Frequency: .... Hz
Amplitude: .... mm		Amplitude: .... mm
Duration: .... min		Duration: .... min
No. of fillings:		No. of fillings: ....

9.5.5 Air jet extraction

Air jet extraction is the same as pressure-rinsing except for the fact that air is used instead of an extraction liquid.

Air Jet Extraction		
Control Area 1		**Control Area 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: ....
Pressure: .... bar		Pressure: .... bar
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

9.5.6 Air throughflow extraction

Air throughflow extraction is the same as rinsing except for the fact that air is used instead of an extraction liquid.

Air throughflow extraction	
Control Area 1	**Control Area 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: ....
Dimensions, adapter:	Dimensions, adapter: ....
Pressure: .... bar	Pressure: .... bar
Duration: .... min	Duration: .... min
Pulsation frequency: .... s^{-1}	Pulsation frequency: .... s^{-1}
Volume flow, max: .... l/min	Volume flow, max: .... l/min
Volume flow, min: .... l/min	Volume flow, min: .... l/min
Reverse flow rinse:	Reverse flow rinse: ....

9.6 Information about filtration

As well as documenting the actual filtration details, information about drying and any post-treatment steps should also be noted.

Filtration		
Filter 1	**Filter 2**	**Filter 3**
Manufacturer:	Manufacturer:	Manufacturer: ....
Type:	Type:	Type: ....
Filter material:	Filter material:	Filter material: ....
Diameter: .... mm	Diameter: .... mm	Diameter: .... mm
Pore size: .... µm	Pore size: .... µm	Pore size: .... µm

Drying		Pre-conditioning	
Devices:	☐ None (environment) ☐ Drying cabinet ☐ Exsiccator	☐ Yes	☐ No
		Post-treatment	
Temperature:	.... °C	Liquid	☐ Solvent .... ☐ Neutral cleaner .... ☐ Other ....
Duration:	.... min		
		Volume of liquid:	 l

9.7 Information about the analysis

Here, 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 is to be documented in the same way as shown in 9.5 Information about extraction.

9.7.1 Standard analysis

9.7.1.1 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.

Gravimetry	
Manufacturer: ……………………………	Date of last calibration: ………………………………………………
Model: ……………………………	Resolution of balance: ☐ ± 0,1 mg ☐ ± 0,01 mg ☐ ± 0,001 mg

9.7.1.2 Light-optical analysis

Light-optical devices can be calibrated using particle standards. The date of the last calibration is to be documented. The scale is to be stated as the magnification per pixel in [μm/pixel].

Light-Optical Analysis	
Manufacturer: ……………………………	Particle standard: ……………………………………………
Model: ……………………………	Date of last check: ……………………………………………
Scale / magnification: …………………………………………………………………………………… μm/Pixel	
Analysis diameter: …………………………………………………………………………………………… mm	
Brightness: ……………………………………………………………………………………………………… %	
Threshold value: …………………………………………………………………………………………………	

9.7.2 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 below.

208

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	

9.7.3 Shortened analysis (informative)

The information shown below regarding a shortened analysis refers to an optical particle counter.

Optical Particle Counter (OPC)	
Manufacturer:	Coincidence limit: ....
Model:	Date of last calibration: ....
	Calibration material: ....
Analysis liquid: .... ml	
Dilution factor:	
Measuring interval: .... min^{-1}	
Nominal volume flow: .... ml/min	
Detection volume: .... ml	

9.8 Information about test results

Information concerning test results is only correct if related to one of the following units of reference:

Number of components tested $n = [1]$

Wetted surface area of test component $A_c = [cm^2]$

Wetted volume of test component $V_c = [cm^3]$

9.8.1 Gravimetric analysis

The result of a gravimetric analysis is the residue weight $M = M2 - M1$, with the last digit being mathematically rounded. The weights M2 and M1 are read off using the resolving capacity of the weighing balance. The resulting particle mass G, which is calculated from the difference between the residue weight and the unit of reference, is to be documented.

Mass related to number of components tested

$$G_C = \frac{M}{n} = \left[\frac{mg}{1}\right]$$

The unit is expressed as $mg\ per\ (n)component(s)$.

Mass related to wetted surface area

$$G_A = \frac{M * 1000}{A_C} = \left[\frac{mg}{1000\ cm^2}\right]$$

Mass related to wetted volume

$$G_V = \frac{M * 1000}{V_C} = \left[\frac{mg}{100\ cm^3}\right]$$

9.8.2 Light-optical analysis

Light-optical systems determine the length and width of individual particles. The particles are also classified into a specified length/width interval (also: size class). This gives the particle size distribution, which may contain all or only selected (particle) size classes.

Length/width intervals other than the size classes listed below should only be documented if absolutely necessary, e.g. 100 ≤ x < 200 μm, 200 ≤ x < 300 μm, etc. (added effort).

The size classes used in the past have been extended by the letters L, M and N, see Table 9-1.

210

Table 9-1: (Particle) size classes

Size class	Size x [µm]
B	5 ≤ x < 15
C	15 ≤ x < 25
D	25 ≤ x < 50
E	50 ≤ x < 100
F	100 ≤ x < 150
G	150 ≤ x < 200
H	200 ≤ x < 400
I	400 ≤ x < 600
J	600 ≤ x < 1000
K	1000 ≤ x < 1500
L	1500 ≤ x < 2000
M	2000 ≤ x < 3000
N	3000 ≤ x

The particle size distribution must 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 is expressed as $particles\ per\ (n)component(s)$.

If, for example, a test lot of five components is inspected (n = 5) and 1 particle in Size Class H (200 ≤ x < 400 µm) is detected, this gives the normed particle count $H_C = 0.2$ rounded to at least one position after the decimal point.

Particle count related to wetted surface area

$$H_A = \frac{h * 1000}{A_C} = \left[\frac{1}{1000\ cm^2}\right]$$

The unit is expressed as $... particles\ per\ ... 1000\ cm^2$.

If, for example, 89 particles are counted in Size Class H (200 ≤ x < 400 µm) for a component with a wetted surface area 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

$$H_V = \frac{h * 100}{V_C} = \left[\frac{1}{100\ cm^3} \right]$$

If, for example, 2 particles in Size Class K (1000 ≤ x < 1500 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 table (see example of test results on next page). When norming, the resulting comma positions can often be mathematically rounded to one digit after the decimal point. The specification of one position after the decimal point 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 value calculation always refers to the test lot.

A second addition is information about categories. Here, among other things, 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 is to be documented. 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.

Particle size distribution according to categories

Length, Feret$_{Max}$ [µm]	Particle count per 1000 cm²												
	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 < 1000	1000 ≤ x < 1500	1500 ≤ x < 2000	2000 ≤ x < 3000	3000 <
Category — All particles excluding fibers													
Category — Metallic shiny particles													
Category — Fibers													

Width, Feret$_{Min}$ [µm]	Particle count per 1000 cm²												
	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 < 1000	1000 ≤ x < 1500	1500 ≤ x < 2000	2000 ≤ x < 3000	3000 <
Category — All particles excluding fibers													
Category — Metallic shiny particles													
Category — Fibers													

Image series

Largest particle — Second largest particle — Largest metallic shiny particle — Second largest metallic shiny particle — Largest fiber — Seconlargest fiber — Filter occupancy [%]

The third addition concerns documentation of the largest particle. Images of the longest or widest particle in each category are to 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.

9.8.3 Extended analysis

The way inspection results are presented depends upon the method selected. In the case of as SEM/EDX analysis, for example, information about a material class can also be documented.

Inspection results

Particle size distribution according to material classes

Length, Feret$_{Max}$ [µm]		Particle count per 1000 cm²											
		B	C	D	E	F	G	H	I	J	K	L	M	N
		5	15	25	50	100	150	200	400	600	1000	1500	2000	3000
		≤ x <	≤ x <	≤ x <	≤ x <	≤ x <	≤ x <	≤ x <	≤ x <	≤ x <	≤ x <	≤ x <	≤ x <	<
		15	25	50	100	150	200	400	600	1000	1500	2000	3000	
Material class	Steel, high-alloy													
	Steel, non-alloyed													
	Brass													
	Copper													
	Aluminium													
	Lead													
	Mineral													
	Glass mineral fiber													
	⋮													

Image series

Largest particle steel

Second largest particle steel

Largest particle copper

Second largest particle copper

Largest mineral glass fiber

Second largest mineral glass fiber

9.8.4 Shortened analysis

Inspection results

Particle size distribution

Equivalent length [µm]	Particle count per 1000 cm²												
	B	C	D	E	F	G	H	I	J	K	L	M	N
	5	15	25	50	100	150	200	400	600	1000	1500	2000	3000
	≤ x <	≤ x <	≤ x <	≤ x <	≤ x <	≤ x <	≤ x <	≤ x <	≤ x <	≤ x <	≤ x <	≤ x <	<
	15	25	50	100	150	200	400	600	1000	1500	2000	3000	

9.8.5 Optional coding (informative)

This sub-chapter deals with coding particle size distributions in accordance with ISO 16232 (as of 2007) with the original intention of abbreviating cleanliness values. It can only be used for particle size distributions, although it is not stated in the ISO norm whether coding may be applied to individual types of particle (fibers, metallic shiny, etc.). The norm also does not state whether particle widths can be coded or not.

Component Cleanliness Code

Coding is used to assign the particle count to a cleanliness level, see Table 9-2.

The Component Cleanliness Code is noted as follows:

$$CCC = \underbrace{A}_{\substack{Unit\ of \\ reference}}\ (\ \underbrace{B}_{\substack{Size \\ class}}\ \underbrace{20}_{\substack{Contam- \\ ination \\ level}}\ \underbrace{/}_{Separator}\ C16/D18/\dots)$$

The unit of reference states whether the particle count is related to:

      $1000\ cm^2$ wetted surface area of the test component (marked A),

      $100\ cm^3$ wetted volume of the test component (marked V) or

      $1\ component$ (marked N).

See Table 9-1 for size classes and Table 9-2 for cleanliness levels.

Table 9-2: Particle concentration classes

Cleanliness Level	Particle count (per 1000 cm² or pro 100 cm³)	
	including	up to
00	-	0
0	0	1
1	0	2
2	0	4
3	0	8
4	0	16
5	0	32
6	0	64
7	0	130
8	0	250
9	0	500
10	0	1×10^3
11	0	2×10^3
12	0	4×10^3
13	0	8×10^3
14	0	16×10^3
15	0	32×10^3
16	0	64×10^3
17	0	130×10^3
18	0	250×10^3
19	0	500×10^3
20	0	1×10^6
21	0	2×10^6
22	0	4×10^6
23	0	8×10^6
24	0	16×10^6

Simplified ways of writing Component Cleanliness Codes

The following example shows the various methods permitted for documenting the CCC. The following particle size distribution is documented:

Particle count per 1000 cm²												
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 < 1000	1000 ≤ x < 1500	1500 ≤ x < 2000	2000 ≤ x < 3000	3000 <
634981	49735	36791	938	502	394	48	7	5	2	1	1	0

As particle counts are expressed in a normed way, they can be read of as a Cleanliness Level directly from Table 9-2, resulting in the following Component Cleanliness Code:

$$CCC = A(B20/C16/D12/E10/F10/G9/H6/I3/J2/K1/L0/M0/N00)$$

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 neighboring size classes are identical, this can be abbreviated to:

$$CCC = A(B20/C16/D12/\underline{EF}10/G9/H6/I3/J2/K1/\underline{LM}10/N00)$$

However, the situation changes if several different size classes are combined in the analysis:

Particle count per 1000 cm²							
B - D	E	F	G	H	I	J	K - N
5 ≤ x < 50	50 ≤ x < 100	100 ≤ x < 150	150 ≤ x < 200	200 ≤ x < 400	400 ≤ x < 600	600 ≤ x < 1000	1000 <
721507	938	502	394	48	7	5	4

The corresponding simplified code is then expressed as follows:

$$CCC = A(\underline{B-D}20/E10/F10/G9/H6/I3/J2/\underline{K-N}2)$$

Exceptional case, unit of reference N

The CCC per *component* may only be stated if it refers to exactly 1 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 are to be rounded.

$$CCC = N(B755840/C43720/D36791/E938/F502/$$
$$G394/H48/I7/J5/K2/L1/M1/N0)$$

9.9 Scope of the various documents

9.9.1 Inspection specification

The inspection specification is the main document of the cleanliness analysis. It is written after the qualification test has been completed and once inspection parameters have been derived for the routine inspection. The inspection specification comprises:

General information

Information about the test component

*Information about preparatory steps (detailed)

*Information about extraction (detailed)

Information about filtration

Information about analysis

Whereabouts of the test component

The inspection specification may contain additional information about the qualification test or refer to an independent document – the qualification report.

The inspection specification contains a clearly understandable description of the test procedure (especially with regard to the sections marked with *). 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 must 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 14.6.1.

No inspection results are documented in the inspection specification except for the results of the qualification test if no qualification report is available. Cleanliness requirements or limit values may be included in an inspection specification if no independent cleanliness specification exists or if no appropriate sketches are available.

9.9.2 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 also no inspection report. The qualification report comprises:

General information

Information about the test component

Information about preparatory steps

Information about extraction

Information about filtration

Information about analysis

*Results of the declining test

*Verification of the blank value

*Information on routine test conditions and inspection parameters

The qualification report contains a graph of the declining test and information about routine test conditions and parameters (marked with *). As an option, the results of a subsequent double inspection can also be included.

The routine test 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 test 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 test is included in the inspection specification (see preceding list).

9.9.3   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:

General information

Information about the test component

Information about preparatory steps

Information about extraction

Information about filtration

Information about analysis

*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 it is always understandable how test conditions and parameters were determined, the inspection report must 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 they have been archived or saved, the inspection specification and qualification report do not have to be included when handing over the inspection report.

Annex:

Example of an inspection report about a test component with a control area. The extraction method of pressure-rinsing was used. Filtration was carried out with a three-step cascade. *Gravimetry* and *light microscopy* were utilized for the analysis.

Inspection report

General information

Order No.:	....	Report Nor.:	....
Customer:	....	Date of analysis:	....
		Contact partner:	....

Test ordered: ☐ Routine inspection ☐ Double inspection ☐ Qualification test

Reason for test: ☐ Initial assessment ☐ Process control ☐ Modifications, constructional ☐ ....

Test piece

Description:	....	Condition on delivery:	....
Parts Number:	....	Test lot size:	....
Batch Number:	....	Control area:	....
Date removed:	....	A_C = ... cm²	
Time removed:	....	V_C = ... cm³	
Removed from:	....	Inspection specification:	....

Photo on delivery

Photo test piece

Preparatory steps

☐ None ☐ Isolate control areas (e.g. sealing, masking)

☐ Disassembly ☐ Preclean contaminated surfaces not relevant to control area

☐ Demagnetization ☐ ....

Extraction

Scope of extraction: ☐ Test piece exclusive packaging Test environment: ☐ Undefined
☐ Test piece inclusive packaging ☐ Cleanliness Room Grade (VDA 19-2)
☐ Cleanroom Class (ISO 14644-1): .....

Extraction method: ☐ Pressure rinsing ☐ Internal rinsing ☐ Air jet
☐ Ultrasonics ☐ Agitation ☐ Air throughflow

Solvent: Liquid .... Duration .... Temperature ....

Pressure-rinsing, Control Area 1	Final rinse, Control Area 1
Manufacturer, extraction device:	Manufacturer, extraction device: ....
Model, extraction device:	Model, extraction device: ....
Manufacturer, nozzle:	Manufacturer, nozzle: ....
Type, nozzle: ☐ Round jet ☐ Parallel jet	Type, nozzle: ☐ Round jet ☐ Parallel jet
☐ Flat jet ☐	☐ Flat jet ☐ ....
Dimensions, nozzle:	Dimensions, nozzle: ....
Extraction liquid: ☐ Solvent	Extraction liquid: ☐ Solvent ....
☐ Neutral cleaner	☐ Neutral cleaner ....
☐ Other	☐ 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

Inspection report

Filtration

Filter 1	Filter 2	Filter 3
Manufacturer:	Manufacturer:	Manufacturer: ....
Type:	Type:	Type: ....
Filter material:	Filter material:	Filter material: ....
Diameter: .... mm	Diameter: .... mm	Diameter: .... mm
Pore size: .... µm	Pore size: .... µm	Pore size: .... µm

Drying

Devices:
☐ None (environment)
☐ Drying cabinet
☐ Exsiccator

Temperature: .... °C
Duration: .... min

Pre-conditioning

☐ Yes   ☐ No

Post-treatment

Liquid
☐ Solvent   ....
☐ Neutral cleaner   ....
☐ Other   ....

Volume of liquid: l

Analysis

Gravimetry

Manufacturer: ....
Model: ....

Date of last calibration: ....
Room temperature: .... °C
Resolution of balance: ☐ ± 0,1 mg
                       ☐ ± 0,01 mg
                       ☐ ± 0,001 mg

Light microscopy

Manufacturer: ....
Model: ....

Particle standard: ....
Date of last check: ....
Scale: .... µm/pixel
Analysis diameter: .... mm
Brightness: .... %
Threshold value: ....

Inspection results

Residue weight

M = ... G_C = ... G_A = ... G_V = ...

Particle size distribution according to categories

Length, $Feret_{Max}$ [µm] or Width, $Feret_{Min}$ [µm]	Particle count per 1000 cm²										
	D	E	F	G	H	I	J	K	L	M	N
	25 ≤ x < 50	50 ≤ x < 100	100 ≤ x < 150	150 ≤ x < 200	200 ≤ x < 400	400 ≤ x < 600	600 ≤ x < 1000	1000 ≤ x < 1500	1500 ≤ x < 2000	2000 ≤ x < 3000	3000 <
Category All particles excluding fibers											
Metallic shiny particles											
Fibers											

Image series

Largest particle

Second largest particle

Third largest particle
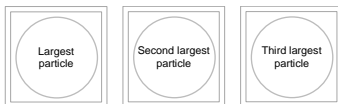
Filter occupancy

Filter occupancy [%]

10 INTERPRETATION AND REACTION (INFORMATIVE)

10.1 Interpretation

The technical cleanliness of components fluctuates much more than any features created specifically. This applies both to the quantities of particles (gravimetric or as counts in particle size classes) and especially the length of the largest particle.

This is due to the fact that contaminant particles are not specifically generated but are rather produced in many shapes and sizes in an uncontrolled manner. This applies to almost all forms of contamination arising in the course of the manufacturing process or during transport and storage. In chipping processes, for example when machining metal components, chips in a wide range of lengths, widths and shapes are generated. Some of these may be found again on the component in the form of residual particulate contamination.

Note: Even with chips specially produced in the laboratory by means of precision chipping, tests have shown that lengths still fluctuate by about 20%.

Although an industrial parts cleaning plant can reduce the overall quantity of component contamination highly efficiently, the existing fluctuation margin can only be reduced to a limited extent. One of the reasons for this is the technical characteristics of filters (filtration curve). For example, a filter in a cleaning system, though which the washing or rinsing medium flows, retains particles upwards of a specific particle width or height that are orientated in the direction of flow. However, the length of particles passing through the filter membrane may still vary significantly. This can often be seen by the major fluctuation in largest particle size in cleanliness analysis results.

This explains why <u>standard quality assurance criteria</u>, such as the values otherwise applying typically to the reliability of processes in the automotive and supplier industries, <u>cannot be applied to technical cleanliness!</u>

However, as already explained in Chapter 2.1, cleanliness limit values are not tolerance limits but rather intervention limits. If a cleanliness limit value for a component is exceeded, this does not necessarily mean that the system the component has been installed into will fail. However, the probability of this occurring will increase.

A particle determined to be critical will only cause a malfunction if is located in the sensitive area of the system at the right moment in time and possibly if it is orientated in a specific direction.

The aim of a VDA 19 cleanliness analysis is not primarily to detect such random events caused by strong fluctuations in cleanliness characteristics but rather to identify systematic errors arising, for example, due to:

- Errors in cleaning processes/plants
- Incorrectly-adjusted or worn tools
- Unsuitable storage or packaging

10.2 Reaction

For the reasons mentioned in the preceding section, the basis of a reaction and of each escalation scenario when a limit value is exceeded is first of all to carry out a double-check. To start with, the test conditions and testing equipment are carefully checked. Then an inspection is performed on a second component from the same batch (B-sample).

Further escalation measures are only taken if this second test also shows that limit values have been exceeded; these measures are not specified in VDA 19.1 and are to be defined individually between the customer and the supplier. Additional testing steps which could be taken within the scope of an escalation procedure could include:

- Testing more components
- A more detailed examination of the respective contaminant particles by applying an extended analysis method, e.g. to determine the third particle dimension or determine material composition.

Note: When testing components, the appropriate action to be taken in the event of a deviation is to be arranged individually between the customer and supplier.

11 WORK SAFETY AND PROTECTION OF THE ENVIRONMENT

Due to the increasing importance of technical cleanliness, over the last few years a new profession has come into being, that of a "technical cleanliness inspector". The respective job description often entails the handling of hydrocarbon-based degreasing extraction liquids for prolonged periods of time. As the potential risks associated with this have only been covered to a certain extent by other safety regulations up till now, the book addresses this issue in more detail at this point.

11.1 General remarks

The application of this guideline involves the use of materials, facilities and equipment which could represent hazards to man and the environment. The guideline does not claim to deal with or exclude all risks associated with its implementation. It is the responsibility of the operator to ensure that health and safety measures are imposed and that legal requirements are upheld.

11.2 Hazards concerning tests involving solvents

The sphere of responsibility of a person running a cleanliness laboratory/testing area includes assessing risks associated with the tasks carried out therein. This section of the book helps operators to assess the typical risks involved, especially those associated with handling solvents when performing an extraction step as part of a cleanliness inspection. The main hazards arising when handling solvents are:

- Hazards associated with inhalation or skin contact:
  - ➢ Vapors or aerosols may be absorbed via the respiratory tract
  - ➢ Degreasing effect on the skin
- Hazards associated with physical-chemical characteristics:
  - ➢ Fire
  - ➢ Dangerous explosive atmosphere

As the majority of non-aromatic cold cleaners used in cleanliness analyses have a flashpoint > 60°C, the above-mentioned risk of fire is not generally considered to be a hazard. However, if the cold cleaner used is in vapor or aerosol form and the lower explosion limit (typical values for cold cleaners 0.6-0.7%) is exceeded, an explosive atmosphere results. The common workplace limit value for vapors from cold cleaners is 600mg/m³. Provided this is observed at all times, there is no risk of explosion because the lower explosion limit is well below the workplace limit value. In Fig. 11-1 shown below, the other risks mentioned – contact with liquid, inhalation and risk of explosion – are linked to various tasks that are typically performed in a cleanliness laboratory.
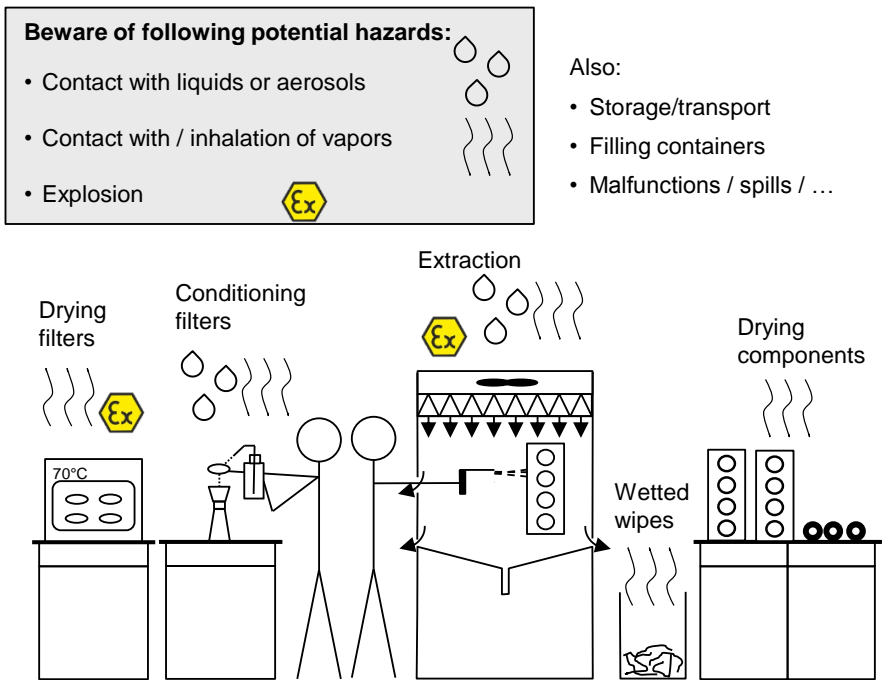
Fig. 11-1: Hazards in the cleanliness laboratory associated with solvents

Additional hazards may be created through the use of ultrasonics, e.g. when warming an extraction liquid in the bath (see Chapter 6.4.3).

Concerning the storage of solvents, the directive TRGS 510 applies.

The directive TRGS 900 provides information on workplace limit values.

If the risk assessment in the cleanliness laboratory identifies potential hazards, protective measures are to be taken to minimize them. They are listed as follows in order of priority (with No. 1 being given the highest priority). The other points listed are merely non-prioritized examples of measures that could be taken to reduce or eliminate hazards.

1. **Substitution** (examples):

   – Replace with aqueous cleaning media (provided extraction efficacy is not impaired)

   – Use solvents (e.g. hydrocarbons) with a flashpoint > 60 °C

   – Substitute solvent mixtures by non-aromatic isoparaffins as pure substances

   – Replace pressure-rinsing extraction from inner surfaces (with associated risk of aerosol formation) with an internal rinsing process using a contained extraction medium

   – Etc.

2. **Technical measures** (examples):

   – Forced ventilation in the laboratory

   – Use enclosed extraction workplace with air exhaust system, explosion-proof if appropriate

   – Fit component-drying areas with air exhaust system

   – Use lower temperatures to dry analysis filters

   – Use closed waste containers for wiping cloths soaked in solvents

   – Monitor temperature in ultrasound baths; automatic shutdown if critical temperatures are reached

   – Exclude sources of ignition in extraction chambers

- Use pressures below 0.7 bar at nozzle level (according to EN 12921-3:2005 +A1:2008, lower risk of aerosol formation) (see also diagram in Annex A 6.4.2.4)

- Etc.

3. **Organizational measures** (examples):

- Restrict the number of analysis filters soaked in solvent that are placed in the drying oven to a non-critical number

- Keep component-drying processes separate from other work-places

- Remove wiping cloths soaked in solvent from the laboratory at regular intervals

- Etc.

4. **Personal safety measures** (examples):

- Breathing protection (this may not be a permanent measure)

- Face mask

- Solvent-proof protective clothing

- Etc.

Note: This list of key points to aid risk assessment does not claim to be complete. It does not release operators of cleanliness laboratories from their duty to obtain comprehensive information about all valid regulations, laws, guidelines and hazards, and to take them appropriately into account in risk assessments.

Annex 11.1 contains examples of different types of chamber for use in conjunction with pressure-rinsing extraction as well as the respective points to be considered in risk assessments. In particular, completely open pressure-rinsing chambers represent a major risk. In such cases, technical protective measures are to be taken to avoid the need for personal protective gear to be worn constantly. See also TRGS 900.

Annex 11   Work safety and the environment

A 11.1   Potential hazards associated with extraction steps using solvents (examples)

A) Fully-closed pressure rinsing chamber:

On opening after extraction step, assess

− exposure to aerosols

− occupational exposure limits
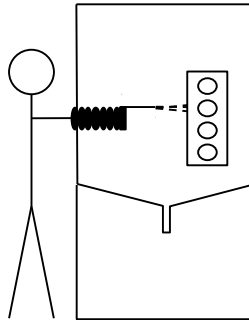
− explosion hazard

B) Closed pressure-rinsing chamber with air supply and leaks:

During extraction (and after), assess

− occupational exposure limits
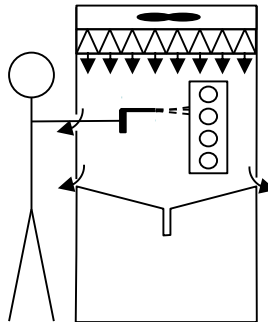
− explosion hazard

C) Closed pressure-rinsing chamber with air supply, leaks and air removal:

Assess

− Explosion hazard in air exhaust system
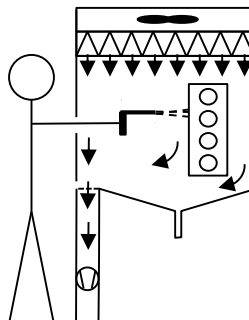
D) Open pressure-rinsing chamber:

- Assess occupational exposure limits and
- Explosion hazard.
- Exposure to aerosols cannot be reliably avoided
- Contact with solvent highly probable

Only permitted if personal protection gear is worn and if not performed for prolonged periods of time
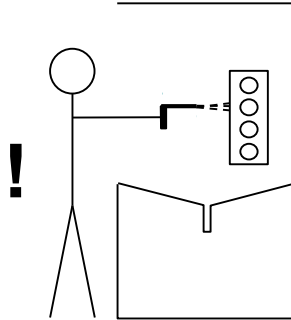
E) Manual pressure-rinsing extraction step without chamber:

Risks as in D) but lower due to small quantities and lower rinsing pressure

- Assess occupational exposure limits
- Exposure to aerosols cannot be reliably avoided
- Contact with solvent probable
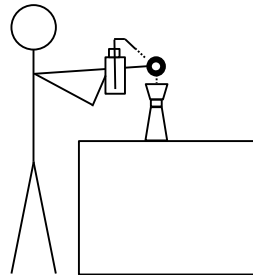
F) Manual pressure-rinsing extraction step under hood:

- Very low risks
- Contact with solvent possible
- Observe blank value because of airflow from environment to the inside
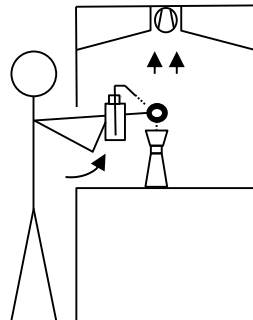
12 CASE EXAMPLES

12.1 Overview

In this section, the following cases are described:

Example / test object	Extraction method	Analysis method	Aim
12.2 Compressed air tank	Agitation	Gravimetry	Devise an inspection specification, declining test
12.3 Piston rings, coated	Ultrasonics	Light microscope (automated)	Devise an inspection specification, declining test
12.4 Plastic housing	Pressure-rinsing	Light microscope (automated)	Devise an inspection specification, declining test and verify by means of double inspection
12.5 Cast cylinder	Pressure-rinsing	Gravimetry, Light microscope (automated)	Devise an inspection specification, declining test, consideration of a cleanliness specification and determination of the blank value
12.6 Crankshaft	Pressure-rinsing + Internal rinsing	Light microscope (automated)	Verify an existing inspection specification by means of double inspection, perform routine inspections on consideration of a cleanliness specification

12.2 Example 1 – Compressed air tank

12.2.1 Aim

To devise an inspection procedure that fulfils the declining criterion, thus enabling it to be established as an inspection specification suitable for use as a basis for developing recurrent routine inspections.

12.2.2 Declining test

In a declining test, the extraction procedure is performed six times on the same component. The suitability of extraction parameters is determined from the results of the declining test shown below.

Extraction conditions

Due to the geometry and location of the control area (internal surface) of the component, agitation was chosen as the extraction method and a component (test lot size n = 1) subsequently tested. The component is made from steel and has a volume of 6 liters. In the extraction step, the container was filled to 30 % with a solvent and agitated 20x per minute at an amplitude of 30 cm.

Analysis conditions

After the sampling procedure, the analysis liquid was filtered through an analysis filter (cellulose nitrate) with a pore size of 5 µm and a diameter of 47 mm. The analysis was performed by means of gravimetry using a five-digit balance.

Results of the declining test:

Calculation of the declining test: particulate residue weight					
	Cleanliness value in mg	Cleanliness value in mg (added together)	Declining value		Declining value ≤ 10 % attained
Extraction #1	6.0	---	---		---
Extraction #2	4.8	10.8	0.44	44 %	No
Extraction #3	1.7	12.5	0.14	14 %	No
Extraction #4	1.2	13.7	0.09	9 %	Yes
Extraction #5	0.7	14.4	0.05	5 %	Yes
Extraction #6	0.2	14.6	0.01	1 %	Yes
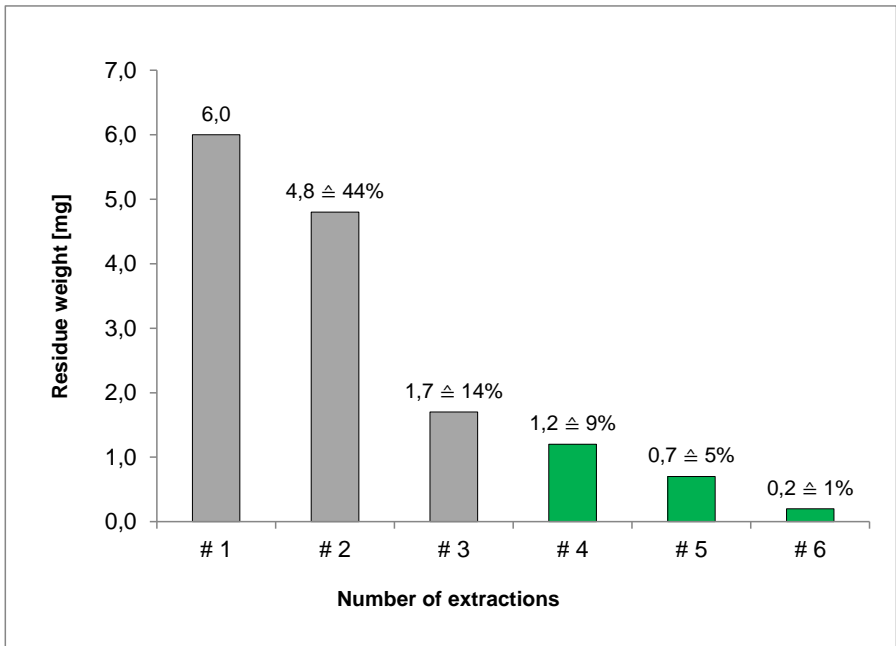
Declining behavior 5.2

The ratio between the fourth extraction step and the sum of the first four extractions is 0.09 (9 %) and therefore fulfils the declining criterion of maximum 0.1 (10 %). In the inspection specification, for recurrent routine cleanliness analyses the extraction parameters need to be adapted to the results of the qualification test. In consequence, the extraction time has to be tripled in order to detach all relevant particles.

12.2.3   Inspection specification

General information

Order No.:	1234567	Report No.:	#Insp. spec._compressed air tank_date#
Customer:	John Smith	Date of analysis:	31.12.2013
		Contact partner:	Mr. Cleanliness

Test ordered: ☐ Routine inspection ☐ Double inspection ☒ Qualification test

Reason for test: ☒ Initial assessment ☐ Process control ☐ Modifications, constructional ☐ ………………

Test piece

Description:	Compressed air tank	Condition on delivery:	Connecting ports closed
Parts number:	987654	Test lot size:	1
Batch number:	654321	Control area:	Internal area
Date removed:	23.12.2013		A_C = ……… cm²
Time removed:	11:30		V_C = ……… cm³
Removed from:	Packing station		
Insp. specification:	#Ins. spec._compressed air-tank_date#		

Photo

Preparatory steps

☐ None   ☐ Isolate control areas (e.g. sealing, masking)

☐ Disassembly   ☒ Pre-clean contaminated surfaces not relevant to control area

☐ Demagnetization   ☒ Clean surfaces with wiping cloth

Extraction

Scope of extraction: ☒ Test piece exc. packaging   Test environment   ☐ Undefined
                                       ☐ Test piece inc. packaging   ☒ Cleanliness Room Grade (VDA 19-2)
                                                                                                                                    ☐ Cleanroom Class (ISO 14644-1) ………

Extraction method: ☐ Pressure-rinsing   ☐ Internal rinsing   ☐ Air jet
                                       ☐ Ultrasonics   ☒ Agitation   ☐ Air throughflow

Solvent: Liquid ………………   Duration ………………   Temperature ………………

Agitation

Control Area 1

Manufacturer, extraction device: ---
Model, extraction device: Laboratory workplace

Extraction liquid: ☒ Solvent   Degreaser
                  ☐ Neutral cleaner ....
                  ☐ Other ....

Filling volume: 1,8 l (30% volume of tank)
Frequency: 20x / min
Amplitude: 30 cm
Duration of agitation: 3 min
Number of fillings: ---

Control Area 2

Manufacturer, extraction device: ....
Model, extraction device: ....

Extraction liquid: ☐ Solvent ....
                  ☐ Neutral cleaner ....
                  ☐ Other ....

Filling volume: ....
Frequency: ....
Amplitude: ....
Duration of agitation: ....
Number of fillings: ....

Control Area 1

Final rinsing liquid: Solvent degreaser
Nozzle geometry: Round jet nozzle 2.5 mm
Volume flow: 1 l/min
Volume of liquid: 0.5 l
Final rinsing time: 0.5 min

Control Area 2

Final rinsing liquid: ....
Nozzle geometry: ....
Volume flow: ....
Volume of liquid: ....
Final rinsing time: ....

Example

Control Area 1

Control Area 2

12.3   Example 2 – Piston rings

12.3.1   Aim

To develop an inspection procedure that fulfils the declining criterion, thus enabling it to be established as an inspection specification suitable for use as a basis for developing recurrent routine inspections.

12.3.2   Declining test

In a declining test, the extraction procedure is performed six times on the same component. The suitability of extraction parameters is determined from the results of the declining test shown in the following.

Extraction conditions

Due to the geometry and material of the component, ultrasonics using a solvent was selected as the extraction method. Based on experience, 10 piston rings were chosen as a test lot size. In a first approximation, the recommended start parameters were selected with a duration of the ultrasound of 1 min, a frequency of 40 kHz and a power density of 10 W/l.

Analysis conditions

After the extraction step, the analysis liquid was filtered through an analysis filter (PET mesh membrane) with a pore size of 5 µm and a diameter of 47 mm. The analysis was performed by means of automated light microscopy with a particle detection size upwards of Size Class E (particles ≥ 50 µm).

Results of the declining test:

Particle count per size class										
Size Class	**E**	**F**	**G**	**H**	**I**	**J**	**K**	**L**	**M**	**N**
Length $Ferret_{max}$ in µm	$50 \leq x < 100$	$100 \leq x < 150$	$150 \leq x < 200$	$200 \leq x < 400$	$400 \leq x < 600$	$600 \leq x < 1000$	$1000 \leq x < 1500$	$1500 \leq x < 2000$	$2000 \leq x < 3000$	$3000 \leq x$
Extraction #1	3593	746	272	129	12	4	2	0	0	0
Extraction #2	889	180	64	33	7	2	0	0	0	0
Extraction #3	1031	224	70	27	7	0	0	0	0	0
Extraction #4	1208	349	126	59	2	1	0	0	0	0
Extraction #5	875	204	80	34	4	0	0	0	0	0
Extraction #6	1157	242	78	28	2	0	0	0	0	0

Remark:

To calculate the declining behavior, Size Classes E – N were added together.

Calculation of the declining test: particle count					
	Cleanliness value Particle count	Cleanliness value Particle count added together	Declining value		Declining value ≤ 10 % attained
Extraction #1	4758	---	---		---
Extraction #2	1175	5933	0.20	20 %	No
Extraction #3	1359	7292	0.19	19 %	No
Extraction #4	1745	9037	0.19	19 %	No
Extraction #5	1197	10234	0.12	12 %	No
Extraction #6	1507	11741	0.13	13 %	No
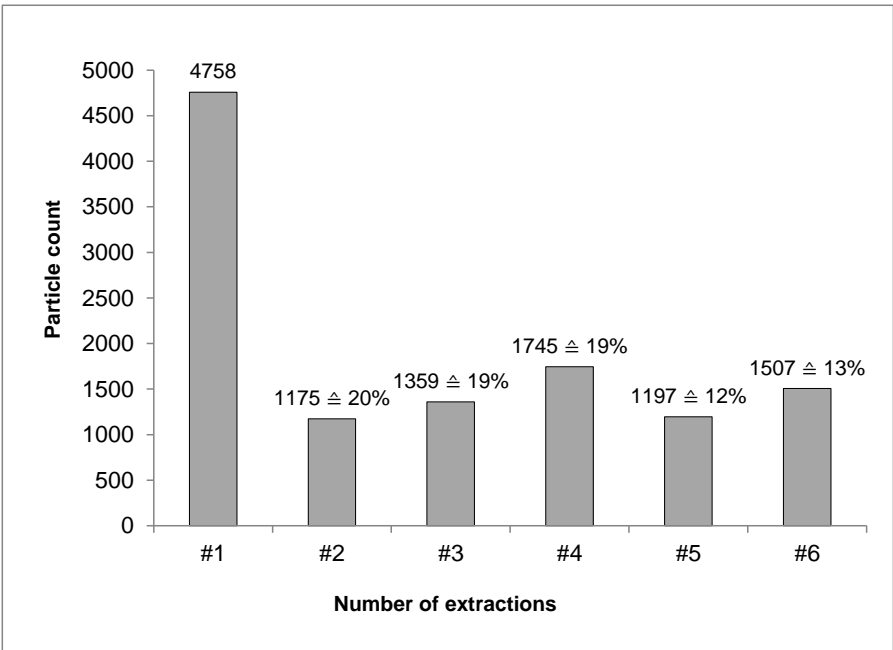
Declining behavior 5.3

The declining criterion of 0.1 (10 %) could not be achieved with the chosen extraction procedure. Consequently, this extraction procedure is not suitable for recurrent routine cleanliness inspections. A new qualification test with different parameters or different extraction method needs to be carried out.

Conclusion:

The results suggest that the procedure destroys the component coating because high particle counts were repeatedly recorded in each declining test. For this reason, a further declining test with an alternative extraction method is required.

12.3.3   Declining test – alternative method

In a declining test, the extraction procedure is performed six times on the same component. The suitability of the extraction conditions is determined from the results of the declining test shown in the following.

Extraction conditions

Based on the results of the first declining test, pressure-rinsing with a solvent was selected as an alternative extraction method. The test lot size was decreased to 5 piston rings. In a first approximation, the recommended start parameters were selected: round jet nozzle with a diameter of 2.5 mm, volume flow of 1.5 l/min and extraction time of 1 min.

Analysis conditions

After the extraction step, the analysis liquid was filtered through an analysis filter (PET mesh membrane) with a pore size of 5 μm and a diameter of 47 mm. The analysis was carried out by means of automated light microscopy with a particle detection size upwards of Size Class E (particles ≥ 50 μm).

Results of the declining test:

Particle count per size class										
Size Class	**E**	**F**	**G**	**H**	**I**	**J**	**K**	**L**	**M**	**N**
Length $Ferret_{max}$ in μm	50 ≤ x < 100	100 ≤ x < 150	150 ≤ x < 200	200 ≤ x < 400	400 ≤ x < 600	600 ≤ x < 1000	1000 ≤ x < 1500	1500 ≤ x < 2000	2000 ≤ x < 3000	3000 ≤ x
Extraction #1	1121	191	113	47	21	6	0	0	0	0
Extraction #2	180	35	22	8	0	0	0	0	0	0
Extraction #3	349	23	11	11	1	0	0	0	0	0
Extraction #4	101	34	19	7	0	0	0	0	0	0
Extraction #5	79	21	7	3	0	0	0	0	0	0
Extraction #6	87	31	9	4	1	0	0	0	0	0

Remark:

To calculate the declining behavior, Size Classes E – N were added together.

Calculation of the declining test: particle count					
	Cleanliness value Particle count	Cleanliness value Particle count (added together)	Declining value		Declining value ≤ 10 % attained
Extraction #1	1499	---	---		---
Extraction #2	245	1744	0.14	24 %	No
Extraction #3	395	2139	0.18	18 %	No
Extraction #4	161	2300	0.07	7 %	Yes
Extraction #5	110	2410	0.05	5 %	Yes
Extraction #6	132	2542	0.05	5 %	Yes
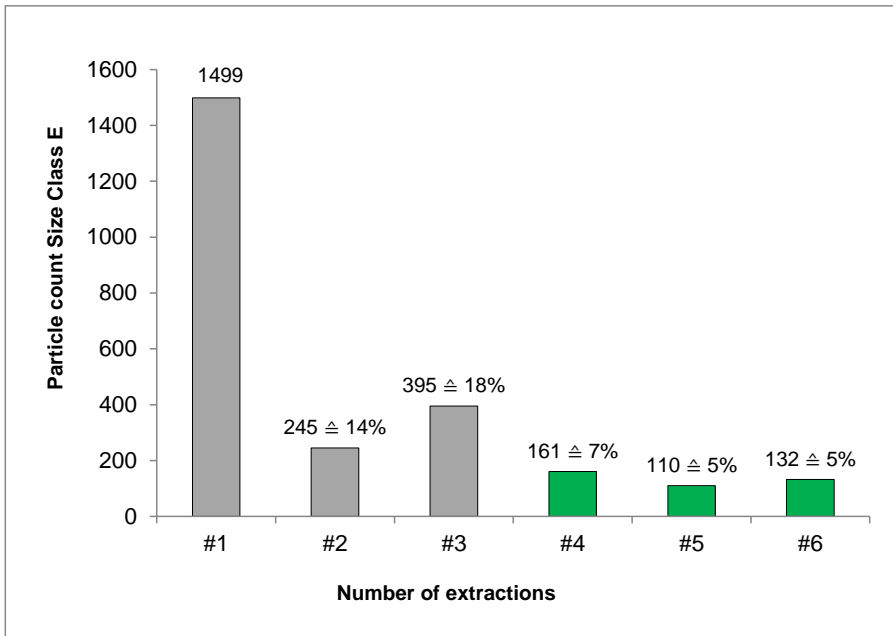
Declining behavior 5.3 Alternative method

The ratio between the fourth extraction step and the sum of the first four extraction steps is 0.07 (7 %), thus fulfilling the declining criterion of maximum 0.1 (10 %). In the inspection specification, for recurrent routine cleanliness analyses the extraction parameters need to be adapted to the results of the qualification test. In consequence, the extraction time has to be tripled in order to detach all relevant particles.

Conclusion:

The pressure-rinsing method does not damage the component in any way during the extraction. The inspection procedure can therefore be fixed.

12.3.4 Inspection specification

General information			
Order No.:	1234567	Report No.:	#Insp. spec._piston rings_date#
Customer:	John Smith	Date of analysis:	31.12.2013
		Contact partner:	Mr. Cleanliness
Test ordered:	☐ Routine inspection ☐ Double inspection ☒ Qualification test		
Reason for test:	☒ Initial assessment ☐ Process control ☐ Modifications, constructional ☐ ………………		

Test piece			
Description:	Piston rings	Condition on delivery:	Packed in VCI foil bags, each containing 50 rings
Parts number:	756890	Test lot size:	5
Batch number:	121212	Control area:	Complete ring
Date removed:	31.12.2013	A_C = ……… cm²	
Time removed:	11:30	V_C = ……… cm³	On delivery
Removed from:	Packing station		
Insp. specification:	#Insp. spec._piston rings_date#		

Preparatory steps	
☒ None	☐ Isolate control areas (e.g. sealing, masking)
☐ Disassembly	☐ Pre-clean contaminated surfaces not relevant to control area
☐ Demagnetization	☐ Clean outer surfaces with wiping cloth

Extraction	

Scope of extraction: ☒ Test piece exc. packaging   Test environment: ☐ Undefined
                  ☐ Test piece inc. packaging   ☐ Cleanliness Room Grade (VDA 19-2)
                                                  ☒ Cleanroom Class (ISO 14644-1) …8.….

Extraction method: ☒ Pressure-rinsing   ☐ Internal rinsing   ☐ Air jet
                  ☐ Ultrasonics   ☐ Agitation   ☐ Air throughflow

---

Pressure-rinsing

Control Area 1	Control Area 2
Manufacturer, extraction device: CLEAN	Manufacturer, extraction device: ……………………
Model, extraction device: 2000	Model, extraction device: ……………………
Manufacturer, nozzle: CLEAN	Manufacturer, nozzle: ……………………………………
Type, nozzle ☒ Round jet ☐ Parallel jet	Type, nozzle: ☐ Round jet ☐ Parallel jet
☐ Flat jet ☐ ……………….	☐ Flat jet ☐ ……………….
Dimensions, nozzle 2.5 mm	Dimensions, nozzle: ……………………………………
Extraction liquid: ☒ Solvent Degreaser	Extraction liquid: ☐ Solvent …………………
☐ Neutral cleaner …………………	☐ Neutral cleaner …………………
☐ Other …………………	☐ Other …………………
Volume flow: 1.5 l/min	Volume flow: ……………………………………
Volume of liquid: 4.5 l	Volume of liquid: ……………………………………
Duration: 3 min	Duration: ……………………………………
Angle (test piece : jet): ---	Angle (test piece : jet): …………………………
Distance between test piece & nozzle: 15 cm	Distance between test piece & jet: …………………
Nozzle motion speed: ---	Nozzle motion speed: …………………………

Control Area 1	Control Area 2
Final rinsing liquid: Solvent degreaser	Final rinsing liquid: …………………………………………
Nozzle geometry: Round jet nozzle 2.5 mm	Nozzle geometry: …………………………………………
Volume flow: 1.5 l/min	Volume flow: …………………………………………
Volume of liquid: 4.5 l	Volume of liquid: …………………………………………
Duration: 3 min	Duration: ……………………………………………

Example

Control Area 1	Control Area 2

Filtration

Filter 1	Filter 2	Filter 3
Manufacturer: SIEB	Manufacturer: …………………	Manufacturer: …………………
Type: Filter	Type …………………	Type: …………………
Filter material: PET mesh	Filter material: …………………	Filter material: …………………
Diameter: 47 mm	Diameter: …………………	Diameter: …………………
Pore size: 5 µm	Pore size: …………………	Pore size: …………………

Drying

Preparatory steps

Devices:
☐ None (environment)
☒ Drying cabinet
☐ Exsiccator

☐ Yes   ☒ No

Post-treatment

Temperature: 60 °C
Duration: 1 h

Liquid:
☐ Solvent ………………
☐ Neutral cleaner ………………
☐ Other ………………

Volume of liquid: …………………………………… l

Light-optical analysis

Manufacturer: XYZ   Particle standard: V1.X
Model: XYZ   Date of last check: 31.12.2013

Scale / magnification: ≤ 5 µm/pixel
Analysis diameter: 44 mm
Brightness: Standard parameters
Threshold value: Standard parameters

12.4 Example 3 – Plastic housing for electronics

12.4.1 Aim

To develop an inspection procedure that fulfils the declining criterion, thus enabling it to be established as an inspection specification suitable for use as a basis for developing recurrent routine inspections.

12.4.2 Declining test

In a declining test, the extraction procedure is performed six times on the same component. The suitability of extraction parameters is determined from the results of the declining test shown in the following.

Extraction conditions

Due to the geometry and material of the component, pressure-rinsing with an aqueous neutral cleaning medium was chosen as the extraction method. The component, which is 10 x 10 cm in size, is an injection-molded plastic part with an inserted elastomer washer. A test lot size of 5 housing components was selected. In a first approximation, the recommended start parameters were selected: round jet with a diameter of 2.5 mm, volume flow of 1.5 l/min and an extraction time of 0.5 min per component was chosen. The customer requirement states that the component packaging is also to be included in the analysis. The packaging was therefore also sampled by pressure-rinsing using an aqueous neutral cleaning medium with a volume flow of 1.5 l/min per minute.

Analysis conditions

After the extraction step, the analysis liquid was filtered through an analysis filter (PET mesh membrane) with a pore size of 5 µm and a diameter of 47 mm. The analysis was performed by means of automated light microscopy with a particle detection size upwards of Size Class E (particles ≥ 50µm).

Results of the declining test:

Particle count per size class										
Size Class	E	F	G	H	I	J	K	L	M	N
Length Ferret$_{max}$ in µm	50 ≤ x < 100	100 ≤ x < 150	150 ≤ x < 200	200 ≤ x < 400	400 ≤ x < 600	600 ≤ x < 1000	1000 ≤ x < 1500	1500 ≤ x < 2000	2000 ≤ x < 3000	3000 ≤ x
Extraction #1	467	170	74	40	11	7	0	0	0	0
Extraction #2	44	11	5	4	2	0	0	0	0	0
Extraction #3	36	6	1	1	0	0	0	0	0	0
Extraction #4	40	7	6	3	0	0	0	0	0	0
Extraction #5	24	2	2	1	0	0	0	0	0	0
Extraction #6	57	9	0	0	0	0	0	0	0	0

Remark:

To calculate the declining behavior, Size Classes E - N were added together.

Calculation of the declining test: particle count					
	Cleanliness value Particle count	Cleanliness value Particle count (added together)	Declining value		Declining value ≤ 10 % attained
Extraction #1	769	---	---		---
Extraction #2	66	835	0.08	8 %	Yes
Extraction #3	44	879	0.05	5 %	Yes
Extraction #4	56	935	0.06	6 %	Yes
Extraction #5	29	964	0.03	3 %	Yes
Extraction #6	66	1030	0.06	6 %	Yes
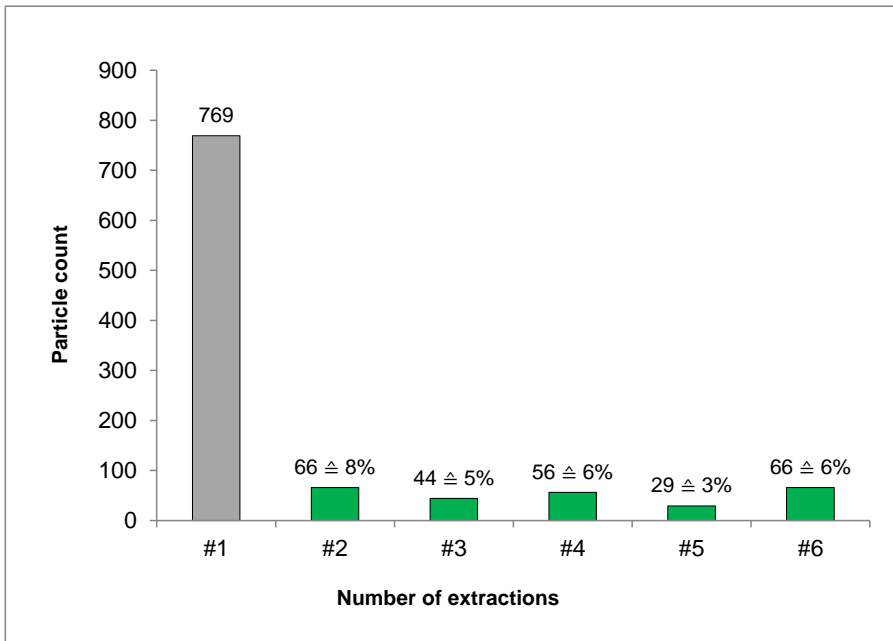
Declining behavior 5.4

The ratio between the second extraction step and the sum of the first two extraction steps is 0.08 (8 %), thus fulfilling the declining criterion of maximum 0.1 (10 %). In the inspection specification, for recurrent routine cleanliness analyses the extraction parameters need to be adapted to the results of the qualification test. In consequence, the extraction procedure is suitable for detaching all relevant particles.

12.4.3 Verification of the inspection procedure (double inspection)

To determine whether all relevant particles are still effectively detached from the components without a short reaction time between each declining test, a double inspection is carried out.

The procedure is carried out twice on the same component. The blank value is determined before and after each procedure. To determine the blank value, the preceding extraction and analysis procedures are performed without the component.

Results of the double inspection:

Particle count per size class										
Size Class	E	F	G	H	I	J	K	L	M	N
Length $Ferret_{max}$ in µm	50 $\leq x <$ 100	100 $\leq x <$ 150	150 $\leq x <$ 200	200 $\leq x <$ 400	400 $\leq x <$ 600	600 $\leq x <$ 1000	1000 $\leq x <$ 1500	1500 $\leq x <$ 2000	2000 $\leq x <$ 3000	3000 $\leq x$
Blank Value 1	39	5	2	0	0	0	0	0	0	0
Extraction #1	398	182	61	48	13	6	0	0	0	0
Extraction #2	24	8	2	0	0	0	0	0	0	0
Blank Value 2	15	1	0	0	0	0	0	0	0	0

Remark:

In the calculation, Size Classes E -N were added together.

	Cleanliness value Particle count	Cleanliness value Particle count (added together)	Double in-spection declining value		Double inspection declining value ≤ 30 % attained
Calculation of the double inspection: particle count					
Extraction #1	708	---	---		---
Extraction #2	34	742	0.05	5 %	Yes
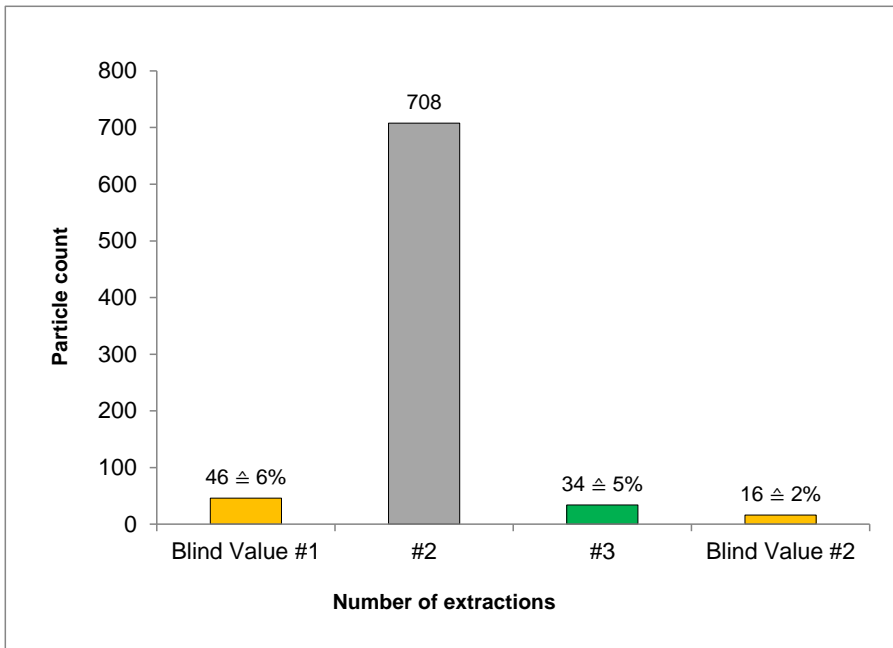
Double inspection 5.4

The ratio between the second extraction step and the sum of the first two extractions is 0.05 (5 %), thus fulfilling the double inspection criterion of maximum 0.3 (30 %).

Verification of the blank value:

Particle count per size class										
Size Class	**E**	**F**	**G**	**H**	**I**	**J**	**K**	**L**	**M**	**N**
Length Ferret$_{max}$ in µm	50 ≤ x < 100	100 ≤ x < 150	150 ≤ x < 200	200 ≤ x < 400	400 ≤ x < 600	600 ≤ x < 1000	1000 ≤ x < 1500	1500 ≤ x < 2000	2000 ≤ x < 3000	3000 ≤ x
Extraction #1	398	182	61	48	13	6	0	0	0	0
Max. permissible blank value 10 %	39	18	6	4	1	0	0	0	0	0
Blank Value #1	39	5	2	0	0	0	0	0	0	0
Blank Value i.O.	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
Blank Value #2	15	1	0	0	0	0	0	0	0	0
Blank Value i.O.	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes

Remark:

As no cleanliness specification with limit values exists for the component, the limits for the blank value are determined from the cleanliness values determined (Extraction #1) (10 % of the values, rounded).

Conclusion:

The inspection procedure developed from the declining tests enables component cleanliness to be accurately determined.

12.4.4   Inspection specification

General information

Order No.:	1234567	Report No.:	#Insp. spec._plastic housing_date#
Customer:	John Smith	Date of analysis:	31.12.2013
		Contact partner:	Mr. Cleanliness

Test ordered: ☐ Routine inspection   ☒ Double inspection   ☒ Qualification test
Reason for test: ☒ Initial assessment   ☐ Process control   ☐ Modifications, constructional   ☐ ………………

Test piece

Description:	Plastic housing	Condition on delivery:	Packed in plastic bags, each containing 5 pieces
Parts Number:	10892-15	Test lot size:	5
Batch Number:	13131313	Control area:	Complete housing
Date removed:	23.12.2013	A_C = ……… cm²	
Time removed:	11:30	V_C = ……… cm³	
Removed from:	Packing station		
Insp. specification:	#Insp. spec._plastic housing_date#		

Preparatory steps

☒ None   ☐ Isolate control areas (e.g. sealing, masking)
☐ Disassembly   ☐ Pre-clean contaminated surfaces not relevant to control area
☐ Demagnetization   ☐ Clean outer surfaces with wiping cloth

Extraction

Scope of extraction:	☐ Test piece exc. packaging	Test environment:	☐ Undefined
	☒ Test piece inc. packaging		☒ Cleanliness Room Grade (VDA 19-2)
			☐ Cleanroom Class (ISO 14644-1): ………
Extraction method:	☒ Pressure-rinsing	☐ Internal rinsing	☐ Air jet
	☐ Ultrasonics	☐ Agitation	☐ Air throughflow
Solvent:	Liquid ………………	Duration………………	Temperature ………………

Pressure-Rinsing

Control Area 1	**(component surface)**

Manufacturer, extraction device: CLEAN
Model, extraction device: 2000

Manufacturer, nozzle: CLEAN
Type, nozzle: ☒ Round jet ☐ Parallel jet
☐ Flat jet ☐ ....
Dimensions, nozzle: 2.5 mm

Extraction liquid: ☐ Solvent ....
☒ Neutral cleaner, aqueous
☐ Other ....

Volume flow: 1.5 l/min
Volume of liquid: 0.75 l per comp. / 3.75 l in total
Duration: 0.5 min per comp. / 2.5 min total

Angle (test piece : jet): 45°
Distance (test piece : nozzle): 15 cm
Nozzle motion speed: ---

Control Area 2	**(component packaging)**

Manufacturer, extraction device: CLEAN
Model, extraction device: 2000

Manufacturer, nozzle CLEAN
Type, nozzle: ☒ Round jet ☐ Parallel jet
☐ Flat jet ☐ ....
Dimensions, nozzle: 2.5 mm

Extraction liquid: ☐ Solvent ....
☒ Neutral cleaner, aqueous
☐ Other ....

Volume flow: 1.5 l/min
Volume of liquid: 1.5 l
Duration: 1 min

Angle (test piece : jet): 45°
Distance between test piece & nozzle: 15 cm
Nozzle motion speed: ---

Control Area 1	

Final rinsing liquid: Neutral cleaner, aqueous
Nozzle geometry: Round jet nozzle 2.5 mm
Volume flow: 1.5 l/min
Volume of liquid: 3 l
Duration: 2 min

Control Area 2	

Final rinsing liquid: ....
Nozzle geometry: ....
Volume flow: ....
Volume of liquid: ....
Duration: ....

Example

Control Area 1	Control Area 2
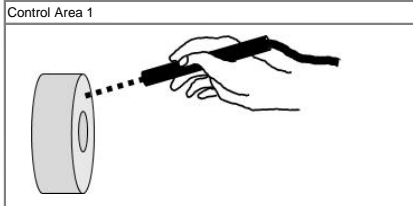
Filtration

Filter 1

Manufacturer: SIEB
Type: Filter
Filter material: PET mesh
Diameter: 47 mm
Pore size: 5 µm

Filter 2

Manufacturer: …………………
Type: …………………
Filter material: …………………
Diameter: …………………
Pore size: …………………

Filter 3

Manufacturer: …………………
Type: …………………
Filter material: …………………
Diameter: …………………
Pore size: …………………

Drying

Devices: ☐ None (environment)
              ☒ Drying cabinet
              ☐ Exsiccator

Temperature: 90 °C
Duration: 1 h

Preparatory steps

☐ Yes   ☒ No

Post-treatment

Liquid: ☐ Solvent   ………………
               ☐ Neutral cleaner ………………
               ☐ Other   ………………
Volume of liquid: …………………………………… l

Light-optical analysis

ManufacturerXYZ   Particle standard: V1.X
Model: XYZ   Date of last check: 31.12.2013

Scale / magnification: ≤ 5 µm/pixel
Analysis diameter: 44 mm
Brightness: Standard parameters
Threshold value: Standard parameters

12.5 Example 4 – Cast cylinder for hydraulic applications

12.5.1 Aim

To develop an inspection procedure that fulfils the declining criterion, thus enabling it to be established as an inspection specification suitable for use as a basis for developing recurrent routine inspections. A cleanliness specification with limit values has already been defined for the component.

12.5.2 Cleanliness specification:

The following cleanliness specification exists for the test component.

Cleanliness specification									
Component	Description:		Number:			Parts Number:			
	Cast cylinder		**1234567**			**9876-54321**			
			Surface area:	800cm²		Volume:			
Unit of reference	☐ Component		☒ $A_C = 1000cm^2$			☐ $V_C = 100cm^3$			
Inspection specification									
Particle count per size class									
Size Class	**F**	**G**	**H**	**I**	**J**	**K**	**L**	**M**	**N**
Length Ferret$_{max}$ in µm	100 ≤ x < 150	150 ≤ x < 200	200 ≤ x < 400	400 ≤ x < 600	600 ≤ x < 1000	1000 ≤ x < 1500	1500 ≤ x < 2000	2000 ≤ x < 3000	3000 ≤ x
All particles excluding fibers	n.a.	n.a.	n.a.	50	10	5	0	0	0
Metallic shiny particles	n.a.	n.a.	n.a.	10	5	0	0	0	0
Fibers	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
Gravimetry									
Residue weight mg	12								

12.5.3   Declining tests

In a declining test, the extraction procedure is performed six times on the same component. The suitability of extraction parameters is determined from the results of the declining test shown in the following.

Extraction conditions

Due to the geometry and material of the component, pressure-rinsing with a solvent was chosen as the extraction method. A test lot size of one component was selected. In a first approximation, the recommended start parameters were used, i.e. round jet nozzle 2.5 mm in diameter and a volume flow of 1.5 l/min. Based on the surface area of the test component (800 cm²), a volume of liquid of (5 ml/cm² x 800 cm²) → 4 l was required. This corresponds with a calculated extraction time of 2 minutes and 36 seconds per component.

Analysis conditions

After the extraction step, the analysis liquid was filtered via a filter cascade comprising a first analysis filter (cellulose nitrate) with a pore size of 5µm and a second analysis filter (PET mesh) with a pore size of 21 µm, each with a diameter of 47 mm. The 5 µm filter was analyzed by means of gravimetry and the 21 µm filter by automated light microscopy with a particle detection size upwards of Size Class I (particles ≥ 400 µm).

Results of the gravimetric declining test:

	Cleanliness value in mg		Cleanli-ness value in mg (added together)	Cleanli-ness values in mg added together	Declining value		Declin-ing value ≤ 10 % attained
	Coarse filter	Fine filter					
Extraction #1	4.8	3.6	8.4	---	---		---
Extraction #2	1.2	2.8	4.0	12.4	0.32	32 %	No
Extraction #3	1.3	1.6	2.9	15.3	0.19	19 %	No
Extraction #4	0.8	0.9	1.7	17.0	0.10	10 %	No
Extraction #5	0.3	0.6	0.9	17.9	0.05	5 %	Yes
Extraction #6	0.1	0.1	0.2	18.1	0.01	1 %	Yes

Calculation of the declining test: particle residue weight
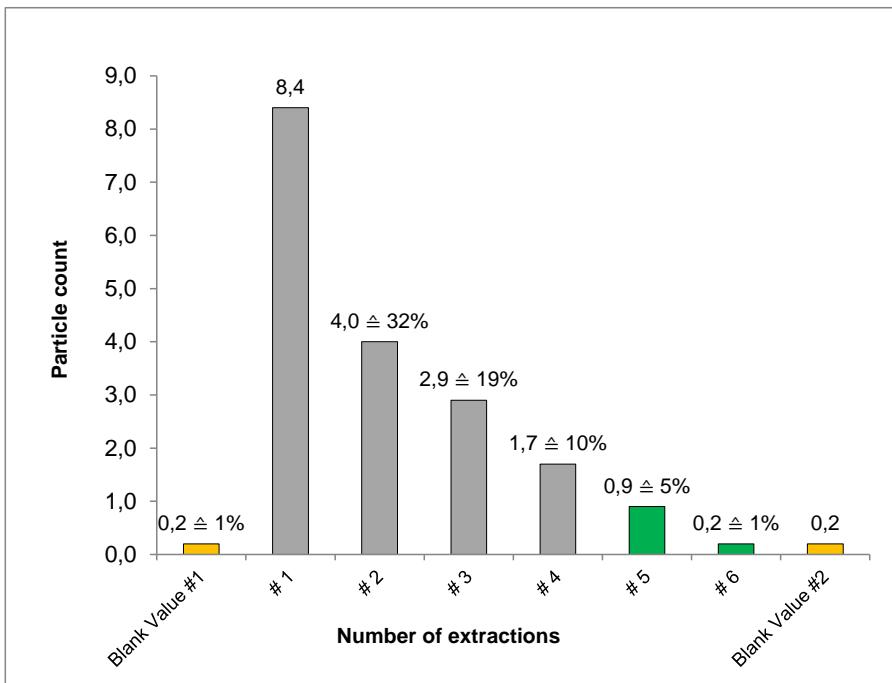
Declining behavior 5.5 Gravimetry

The ratio between the fifth extraction step and the sum of the first five extraction steps is 0.05 (5%), thus fulfilling the declining criterion of maximum 0.1 (10%). In the inspection specification, for recurrent routine cleanliness analyses the extraction parameters need to be adapted to the results of the qualification test.

Verification of the blank value:

Gravimetry	
Permissible residue weight in mg	12
Blank Value in mg (max. permissible) 10%	1.2
Blank Value #1	0.2
Blank Value i.O.	Yes
Blank Value #2	0.2
Blank Value i.O.	Yes

Results of the light microscopy declining test:

Particle count per size class									
Size Class	F	G	H	I	J	K	L	M	N
Length Ferret$_{max}$ in µm	$100 \leq x < 150$	$150 \leq x < 200$	$200 \leq x < 400$	$400 \leq x < 600$	$600 \leq x < 1000$	$1000 \leq x < 1500$	$1500 \leq x < 2000$	$2000 \leq x < 3000$	$3000 \leq x$
Blank Value #1	1	0	0	0	0	0	0	0	0
Extraction #1	1235	627	171	56	4	1	0	0	0
Extraction #2	954	376	15	19	2	1	0	0	0
Extraction #3	623	212	12	7	1	0	0	0	0
Extraction #4	324	159	9	1	1	0	0	0	0
Extraction #5	189	79	7	0	0	0	0	0	0
Extraction #6	98	46	7	1	0	0	0	0	0
Blank Value #2	0	0	0	0	0	0	0	0	0

Remark:

To calculate the declining behavior, Size Classes H - K are added together.

Calculation of the declining test: particle count					
	Cleanliness value Particle count	Cleanliness value Particle count (added together)	Declining value		Declining value ≤ 10 % attained
Extraction #1	232	---	---		---
Extraction #2	37	269	0.14	14 %	No
Extraction #3	20	289	0.07	7 %	Yes
Extraction #4	11	300	0.04	4 %	Yes
Extraction #5	9	309	0.03	3 %	Yes
Extraction #6	8	317	0.03	3 %	Yes
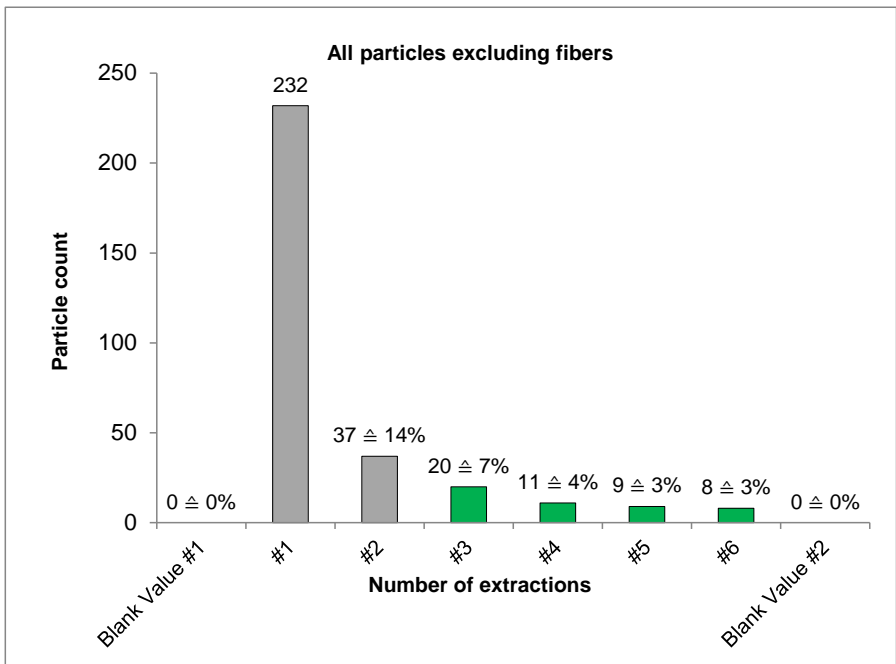
Declining behavior 5.5 Light microscopy

The ratio between the third extraction step and the sum of the first three extraction steps is 0.07 (7%), thus fulfilling the declining criterion of maximum 0.1 (10%). In the inspection specification, for recurrent routine cleanliness analyses the extraction parameters need to be adapted to the results of the qualification test.

Verification of the blank value:

Size Class	F	G	H	I	J	K	L	M	N
Particle count per size class									
Length Ferret$_{max}$ in µm	100 ≤ x < 150	150 ≤ x < 200	200 ≤ x < 400	400 ≤ x < 600	600 ≤ x < 1000	1000 ≤ x < 1500	1500 ≤ x < 2000	2000 ≤ x < 3000	3000 ≤ x
Cleanliness value per 1000 cm²	n.a.	n.a.	n.a.	50	10	5	0	0	0
Blank value 10% max. permitted per 1000 cm²	n.a.	n.a.	n.a.	5	1	0	0	0	0
Blank value max. permitted per component 800 cm²	n.a.	n.a.	n.a.	4	0.8	0	0	0	0
Blank Value #1	1	0	0	0	0	0	0	0	0
Blank value i.O.	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
Blank Value #2	0	0	0	0	0	0	0	0	0
Blank value i.O.	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes

Remark:

When calculating maximum permissible blank values, it may not be forgotten that the cleanliness specification states limit values that are normed to a surface area of 1000 cm². As the actual component surface area is 800 cm², this must be taken into account when calculating the max. permissible blank value per component.

Results of the light microscopy declining test:

Metallic shiny particles

Particle count per size class									
Size Class	**F**	**G**	**H**	**I**	**J**	**K**	**L**	**M**	**N**
Length $Ferret_{max}$ in µm	100 ≤ x < 150	150 ≤ x < 200	200 ≤ x < 400	400 ≤ x < 600	600 ≤ x < 1000	1000 ≤ x < 1500	1500 ≤ x < 2000	2000 ≤ x < 3000	3000 ≤ x
Blank Value #1	0	0	0	0	0	0	0	0	0
Extraction #1	1098	589	132	8	2	1	0	0	0
Extraction #2	943	298	15	2	0	0	0	0	0
Extraction #3	578	197	11	0	0	0	0	0	0
Extraction #4	267	136	7	0	0	0	0	0	0
Extraction #5	127	72	5	0	0	0	0	0	0
Extraction #6	98	46	5	0	0	0	0	0	0
Blank Value #2	0	0	0	0	0	0	0	0	0

Remark:

To calculate the declining behavior, Size Classes H - K are added together.

Calculation of the declining test: particle count				
	Cleanliness value Particle count	Cleanliness value Particle count (added together)	Declining value		Declining value ≤ 10 % attained
Extraction #1	143	---	---		---
Extraction #2	17	160	0.11	11 %	No
Extraction #3	11	171	0.06	6 %	Yes
Extraction #4	7	178	0.04	4 %	Yes
Extraction #5	5	183	0.03	3 %	Yes
Extraction #6	5	188	0.03	3 %	Yes
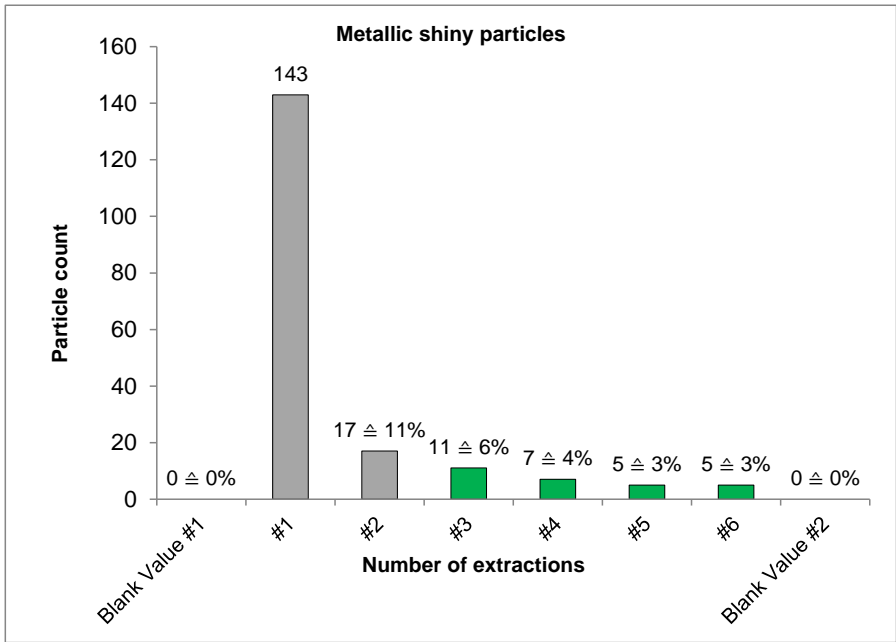
Declining behavior 5.5 Metallic shiny particles

The ratio between the third extraction step and the sum of the first three extraction steps is 0.06 (6%), thus fulfilling the declining criterion of maximum 0.1 (10%). In the inspection specification, for recurrent routine cleanliness analyses the extraction parameters need to be adapted to the results of the qualification test.

Verification of the blank value:

Particle count per size class									
Size Class	**F**	**G**	**H**	**I**	**J**	**K**	**L**	**M**	**N**
Length Ferret$_{max}$ in µm	100 ≤ x < 150	150 ≤ x < 200	200 ≤ x < 400	400 ≤ x < 600	600 ≤ x < 1000	1000 ≤ x < 1500	1500 ≤ x < 2000	2000 ≤ x < 3000	3000 ≤ x
Cleanliness value per 1000 cm²	n.a.	n.a.	n.a.	10	5	0	0	0	0
Blank value 10% max. permitted per 1000 cm²	n.a.	n.a.	n.a.	1	0	0	0	0	0
Blank value max. permitted per component 800 cm²	n.a.	n.a.	n.a.	0.8	0	0	0	0	0
Blank Value #1	0	0	0	0	0	0	0	0	0
Blank value i.O.	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
Blank Value #2	0	0	0	0	0	0	0	0	0
Blank value i.O.	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes

Remark:

When calculating maximum permissible blank values, it may not be forgotten that the cleanliness specification states limit values that are normed to a surface area of 1000 cm². As the actual component surface area is 800 cm², this must be taken into account when calculating the max. permissible blank value per component.

Conclusion:

As it has to be ensured that all relevant particles are reliably removed from the component, the parameters used in the qualification test which caused the declining behavior to take place after the highest number of repetitions need to be determined. In this case, this was the gravimetric analysis of the 5 µm filter in the cascade. Therefore, in order to remove all relevant particles, the extraction time needs to be quadrupled.

12.5.4   Inspection specification

General information

Order No.:	1234567	Report No.:	#Insp. spec._cast cyclinder_date#
Customer:	John Smith	Date of analysis:	31.12.2013
		Contact partner:	Mr. Cleanliness

Test ordered: ☐ Routine inspection   ☐ Double inspection   ☒ Qualification test

Reason for test: ☒ Initial assessment   ☐ Process control   ☐ Modifications, constructional   ☐ ………………

Test piece

Description:	Cast cylinder	Condition on delivery:	Lying horizontally in SLCs without added packaging
Parts Number:	25-854-19	Test lot size:	1
Batch Number:	1414141	Control area:	Internal area
Date removed:	23.12.2013		$A_C = 800$ cm²
Time removed:	11:30		$V_C = ………$ cm³
Removed from:	Packing station		
Insp. specification.:	#Insp. spec._cast cylinder_date#		

On delivery

Preparatory steps

☐ None

☐ Disassembly

☐ Demagnetization

☐ Isolation of control areas (e.g. sealing, masking)

☒ Pre-clean contaminated surfaces not relevant to control area

☒ Clean outer surfaces with wiping cloth

Extraction

Scope of extraction: ☒ Test piece exc. packaging   Test environment: ☐ Undefined

☐ Test piece inc. packaging   ☒ Cleanliness Room Grade: (VDA 19-2)

☐ Cleanroom Class (ISO 14644-1): ………

Extraction method: ☒ Pressure-rinsing   ☐ Internal rinsing   ☐ Air jet

☐ Ultrasonics   ☐ Agitation   ☐ Air throughflow

Solvent: Liquid ………………   Duration ………………   Temperature ………………

Pressure-rinsing

Control Area 1	Control Area 2
Manufacturer, extraction device:　CLEAN	Manufacturer, extraction device:　………….………
Model, extraction device:　2000	Model, extraction device:　………….………
Manufacturer, nozzle: CLEAN	Manufacturer, nozzle:……………………….…………
Type, nozzle:　☒ Round jet　　☐ Parallel jet	Type, nozzle:　☐ Round jet　　☐ Parallel jet
☐ Flat jet　　☐ ………………	☐ Flat jet　　☐ ………………
Dimensions, nozzle: 2.5 mm	Dimensions, nozzle:……………………….…………
Extraction liquid:　☒ Solvent　　Degreaser	Extraction liquid:　☐ Solvent　　…………………
☐ Neutral cleaner　…………………	☐ Neutral cleaner　…………………
☐ Other　　…………………	☐ Other　　…………………
Volume flow:　　　1.5 l/min	Volume flow:　　　…………………
Volume of liquid:　14.6 l	Volume of liquid:　…………………
Duration:　　　　9 min 44 sec	Duration:　　　　…………………
Angle (test piece : jet):　　　　45°	Angle (test piece : jet):　　　…………………
Distance between test piece & nozzle: 15 cm	Distance between test piece & nozzle:………………
Nozzle motion speed:　　　　---	Nozzle motion speed:　　　…………………

Control Area 1	Control Area 2
Final rinsing liquid:　Degreaser	Final rinsing liquid:　………………………………………
Nozzle geometry:　Round jet nozzle 2.5 mm	Nozzle geometry:　………………………………………
Volume flow:　　1.5 l/min	Volume flow:　　………………………………………
Volume of liquid:　3 l	Volume of liquid:　……………....................................
Duration:　　　2 min	Duration:　　　……………....................................

Example

Control Area 1	Control Area 2

Filtration

Filter 1

Manufacturer: FEIN
Type: Filter
Filter material: Cellulose nitrate
Diameter: 47 mm
Pore size: 5 µm

Filter 2

Manufacturer: SIEB
Type: Mesh filter
Filter material: PET membrane
Diameter: 47 mm
Pore size: 21 µm

Filter 3

Manufacturer: …………………
Type: …………………
Filter material: …………………
Diameter: …………………
Pore size: …………………

Drying

Devices: ☐ None (environment)
          ☒ Drying cabinet
          ☒ Exsiccator

Temperature: 60 °C
Duration: 1 h

Preparatory steps

☐ Yes   ☒ No

Post-treatment

Liquid: ☐ Solvent   ………………
            ☐ Neutral cleaner ………………
            ☐ Other   ………………
Volume of liquid: ………………………………… l

Gravimetry

Manufacturer: XYZ   Date of last calibration: 31.12.2013
Model: 2013   Resolution of balance: ☐ ± 0.1 mg   ☒ ± 0.01 mg   ☐ ± 0.001 mg

Light-optical analysis

Manufacturer XYZ   Particle standard: V1.X
Model: XYZ   Date of last check: 31.12.2013

Scale / magnification: ≤ 40 µm/pixel
Analysis diameter: 44 mm
Brightness: Standard parameters
Threshold value: Standard parameters

12.6 Example 5 - Routine inspections – crankshaft

The following inspection specification describes the inspection procedure for a specific type of crankshaft to be used as a basis for developing recurrent routine cleanliness inspections. There is also a cleanliness specification which defines limit values for the component.

12.6.1 Inspection specification

General information

Order No.:	1234567	Report No.:	#Insp. spec._crankshaft372_31.12.13#
Customer:	John Smith	Date of analysis:	20.01.2014
		Contact partner:	Mr. Cleanliness

Test ordered:	☒ Routine inspection	☒ Double inspection	☐ Qualification test
Reason for test:	☐ Initial assessment	☐ Process control	☐ Modifications, constructional ☐ ………………

Test piece

Description:	Crankshaft372	Condition on delivery:	Supplied in special load carriers, packed separately
Parts Number:	372-1284	Test lot size:	1
Batch Number:	20140120	Control area:	Complete piece /
		A_C = ………….. cm²	Functional area
		V_C = ………… cm³	

Insp. specification: #Insp. spec._crankshaft372_31.12.13#
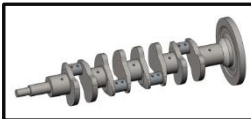
Test accessory

Description:	Stopper 275A	Condition on delivery:	100 pieces in plastic bag
Parts number:	275-2014	Color:	Red
Material:	Polyamide (PA)		
Manufacturer:	STOPPER STAR		

Preparatory steps

☐ None   ☒ Isolation of control areas (e.g. sealing, masking)
☐ Disassembly   ☐ Pre-clean contaminated surfaces not relevant to control area
☐ Demagnetization   ☒ Oil borings must be sealed before performing extraction on complete component

Preparation for extraction: seal oil borings using 13 stoppers
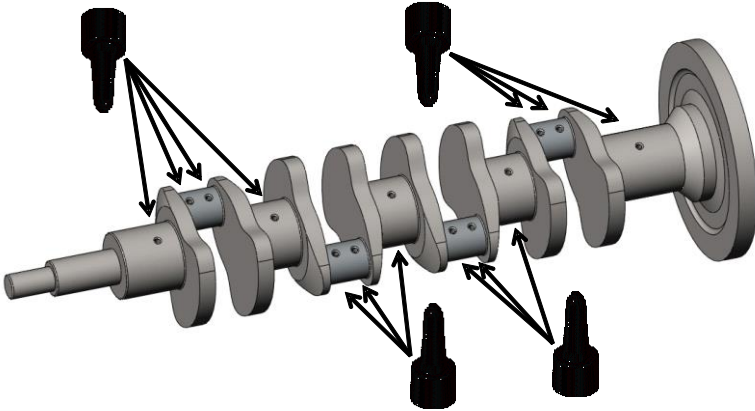
Extraction

Scope of extraction:	☒ Test piece exc. packaging	Test environment:	☐ Undefined
	☐ Test piece inc. packaging		☒ Cleanliness Room Grade: (VDA 19-2)
			☐ Cleanroom Class: (ISO 14644-1) ………
Extraction method:	☒ Pressure-rinsing	☒ Internal rinsing	☐ Air jet
	☐ Ultrasonics	☐ Agitation	☐ Air throughflow

Pressure-rinsing

Control Area 1   (complete component)

Manufacturer, extraction device:	CLEAN
Model, extraction device:	2000

Manufacturer, nozzle: CLEAN
Type, nozzle: ☐ Round jet   ☒ Parallel jet
                 ☐ Flat jet   ☐ ………………

Dimensions, nozzle: 16 borings / 1 mm

Extraction liquid: ☒ Solvent   Degreaser
                    ☐ Neutral cleaner …………………
                    ☐ Other   …………………

Volume flow:	3.0 l/min
Volume of liquid:	9 l
Duration:	3 min

Angle (test piece : jet):   20 - 45°
Distance between test piece & nozzle: 15 cm
Nozzle motion speed: ---

Control Area 2

Manufacturer, extraction device:	………………..……
Model, extraction device:	………………..……

Manufacturer, nozzle: …………………………………………
Type, nozzle: ☐ Round jet   ☐ Parallel jet
                 ☐ Flat jet   ☐ ………………

Dimensions, nozzle: …………………………………………

Extraction liquid: ☐ Solvent   …………………
                    ☐ Neutral cleaner …………………
                    ☐ Other   …………………

Volume flow:	…………………
Volume of liquid:	…………………
Duration:	…………………

Angle (test piece : jet):   …………………
Distance between test piece & nozzle: ………………
Nozzle motion speed: …………………

Control Area 1

Rotate component half-way through extraction procedure

Final rinse extraction device

Final rinsing liquid:	Solvent degreaser
Nozzle geometry:	Parallel jet nozzle
Volume flow:	3 l/min
Volume of liquid:	6 l
Duration:	2 min

Control Area 2

Final rinsing liquid:	…………………………………………
Nozzle geometry:	…………………………………………
Volume flow:	…………………………………………
Volume of liquid:	……………..
Duration:	……………..

Internal rinsing

Control Area 1   (functional area: 13 oil borings)

Manufacturer, extraction device: CLEAN
Model, extraction device: 2000

Manufacturer, nozzle: CLEAN
Type, nozzle: ☒ Round jet   ☐ Parallel jet
                 ☐ Flat jet   ☐ ………………
Dimensions, nozzle: 3 mm plastic

Dimensions, adapter: --

Extraction liquid: ☒ Solvent   Degreaser
                      ☐ Neutral cleaner   …………………
                      ☐ Other   …………………

Volume flow: 1 l/min
Volume of liquid: 1 l per boring / total 13 l
Duration: 1 min per boring / total 13 min

Pulsation rate: ---
Volume flow, max: ---
Volume flow, min: ---
Reverse flow rinse: ---

Control Area 2

Manufacturer, extraction device: ………………………
Model, extraction device: ………………………

Manufacturer, nozzle: …………………………………………
Type, nozzle: ☐ Round jet   ☐ Parallel jet
                 ☐ Flat jet   ☐ ………………
Dimensions, nozzle: …………………………………………

Dimensions, adapter: …………………………………………

Extraction liquid: ☐ Solvent   …………………
                      ☐ Neutral cleaner   …………………
                      ☐ Other   …………………

Volume flow: …………………………………………
Volume of liquid: …………………………………………
Duration: …………………………………………

Pulsation rate: …………………………………………
Volume flow, max: …………………………………………
Volume flow, min: …………………………………………
Reverse flow rinse: …………………………………………
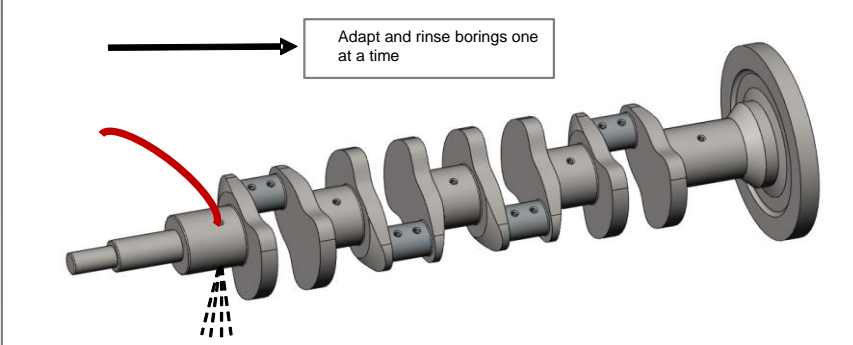
Control Area 1

Adapt and rinse borings one at a time

Final rinse extraction device

Final rinsing liquid:	Solvent degreaser
Nozzle geometry:	Round jet nozzle
Volume flow:	1 l/min
Volume of liquid:	3 l
Duration:	3 min

Control Area 2

Final rinsing liquid:	……………………………………
Nozzle geometry:	……………………………………
Volume flow:	……………………………………
Volume of liquid:	……………………………………
Duration:	……………………………………

Filtration

Filter 1

Manufacturer: FEIN
Type: Filter
Filter material: Cellulose nitrate
Diameter: 47 mm
Pore size: 5 µm

Filter 2

Manufacturer: ....
Type: ....
Filter material: ....
Diameter: ....
Pore size: ....

Filter 3

Manufacturer: ....
Type: ....
Filter material: ....
Diameter: ....
Pore size: ....

Drying

Devices:
☐ None (environment)
☒ Drying cabinet
☐ Exsiccator

Temperature: 60 °C
Duration: 1 h

Preparatory steps

☐ Yes   ☒ No

Post-treatment

Liquid:
☐ Solvent   ....
☐ Neutral cleaner   ....
☐ Other   ....

Volume of liquid: l

Light-optical analysis

Manufacturer: XYZ
Model: XYZ

Particle standard: V1.X
Date of last check: 31.12.2013

Scale / magnification: ≤ 2.5 µm/pixel
Analysis diameter: 44 mm
Brightness: Standard parameters
Threshold value: Standard parameters

Scanning electron microscopy / energy dispersive x-ray spectroscopy (SEM / EDX)

Manufacturer: ELEMENTS
Model: LOOKING FOR

Particle standard: V3.X
Date of last check: 31.12.2013

Acceleration voltage: 20 keV
Analysis diameter: 44 mm
Working distance: 5 mm
Sequence: ☒ Automated   ☐ Manual

12.6.2   Cleanliness specification

Cleanliness specification										
Component	Description:			Number:			Parts Number:			
	Crankshaft			**56789**			**54321-98**			
				Surface area:			Volume:			
Unit of reference	☒ Component			☐ $A_C = 1000 cm^2$			☐ $V_C = 100 cm^3$			
Inspection specification	**Insp. Spec.crankshaft-56789_31.12.2013**									
Particle size class										
Size Class	D	E	F	G	H	I	J	K	L	M
Size in µm	25 ≤ x < 50	50 ≤ x < 100	100 ≤ x < 150	150 ≤ x < 200	200 ≤ x < 400	400 ≤ x < 600	600 ≤ x < 1000	1000 ≤ x < 1500	1500 ≤ x < 2000	2000 ≤ x
Total component surface area without functional area – <u>length</u>, Feret$_{max}$ [µm]										
All particles excluding fibers	5000	2500	1000	500	250	100	50	10	1	0
Metallic shiny particle	2500	1000	500	250	100	50	10	1	0	0
Fibers	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
Functional area (oil borings) – <u>width</u>, Feret$_{min}$ [µm]										
All particles excluding fibers	500	250	100	50	10	5	0	0	0	0
Metallic shiny particle	250	100	50	10	5	0	0	0	0	0
Fibers	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
Additional requirements (material classification) only functional area – <u>width</u>, Feret$_{min}$ [µm]										
Steel	n.a.	n.a.	16	8	4	0	0	0	0	0
Corundum	n.a.	n.a.	2	0	0	0	0	0	0	0
Etc.										

Remark:

Details of the particle width Feret$_{min}$ are required for the functional area of the component.

12.6.3 Aim

Before commencing routine inspections, the inspection procedure is re-verified by carrying out a double inspection. The aim of this is to assess whether the specified test procedure fulfils the declining behavior and blank value criteria in the recommended guideline.

12.6.4 Validation of the test procedure (double inspection)

The procedure is performed twice on the same component, with the blank value being determined beforehand and afterwards in each case. To determine the blank value, the extraction and analysis steps specified are carried out without the component.

Results of the double inspection – complete component:

Particle count per size class										
Size Class	**D**	**E**	**F**	**G**	**H**	**I**	**J**	**K**	**L**	**M**
Length $Ferret_{max}$ in µm	25 ≤ x < 50	50 ≤ x < 100	100 ≤ x < 150	150 ≤ x < 200	200 ≤ x < 400	400 ≤ x < 600	600 ≤ x < 1000	1000 ≤ x < 1500	1500 ≤ x < 2000	2000 ≤ x
Blank Value 1	390	125	98	5	0	0	0	0	0	0
Extraction #1	3980	1820	657	481	198	64	49	8	0	0
Extraction #2	1237	478	235	132	74	24	10	1	0	0
Blank Value 2	386	98	80	4	0	0	0	0	0	0

Calculation of the double inspection					
	Cleanliness value Particle count	Cleanliness value Particle count added together	Declining value double inspection		Declining value double inspection ≤ 30 % attained
Extraction #1	7257	---	---		---
Extraction #2	2191	9448	0.23	23 %	Yes

Remark:

In the calculation, Size Classes D – M are added together.
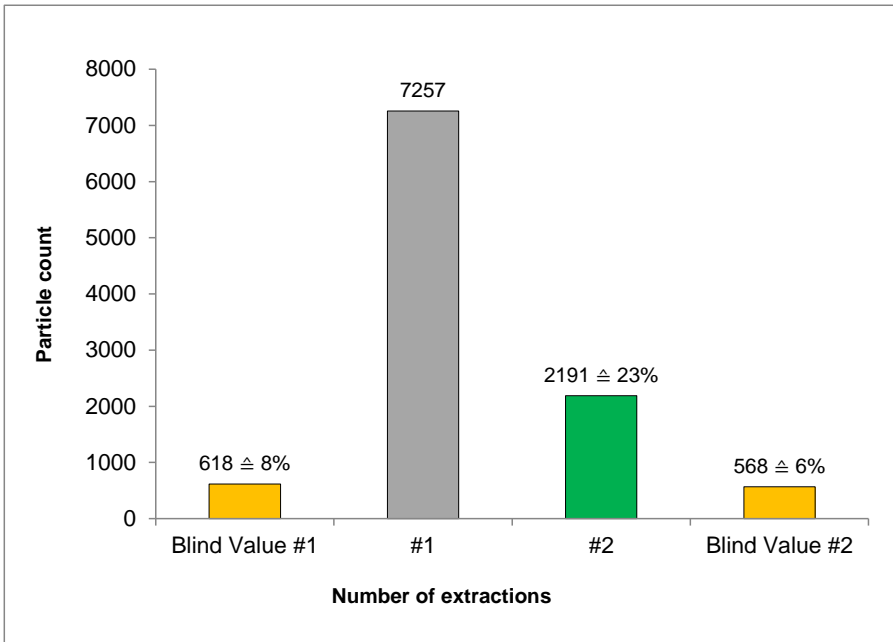
Double inspection 5.6 Complete component

The ratio between the second extraction step and the sum of the first two extractions is 0.23 (23%). This is below the permissible maximum value of 0.3 (30%). In principle, the extraction procedure is therefore suitable for inpecting the cleanliness of the component.

Verification of the blank value:

Particle count per size class										
Size Class	D	E	F	G	H	I	J	K	L	M
Length $Ferret_{max}$ in µm	25 ≤ x < 50	50 ≤ x < 100	100 ≤ x < 150	150 ≤ x < 200	200 ≤ x < 400	400 ≤ x < 600	600 ≤ x < 1000	1000 ≤ x < 1500	1500 ≤ x < 2000	2000 ≤ x
Cleanliness value	5000	2500	1000	500	250	50	5	1	0	0
Max. permissible blank value 10 %	500	250	100	50	25	5	0	0	0	0
Blank Value #1	390	125	98	5	0	0	0	0	0	0
Blank value i.O.	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
Blank Value #2	386	98	80	4	0	0	0	0	0	0
Blank value i.O.	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes

Remark:

The maximum permissible blank values were determined based on the required limit values for each size class.

Results of the double inspection – functional area:

Particle count per size class										
Size Class	D	E	F	G	H	I	J	K	L	M
Width $Ferret_{min}$ in µm	25 ≤ x < 50	50 ≤ x < 100	100 ≤ x < 150	150 ≤ x < 200	200 ≤ x < 400	400 ≤ x < 600	600 ≤ x < 1000	1000 ≤ x < 1500	1500 ≤ x < 2000	2000 ≤ x
Blank Value 1	54	34	7	0	0	0	0	0	0	0
Extraction #1	398	182	65	37	8	4	0	0	0	0
Extraction #2	65	24	13	2	0	0	0	0	0	0
Blank Value 2	54	17	5	0	0	0	0	0	0	0

Calculation of the double inspection					
	Cleanliness value Particle count	Cleanliness value Particle count cumulated	Ratio		Ratio ≤ 30 % attained
Extraction #1	694	---	---		---
Extraction #2	104	798	0.13	13 %	Yes

Remark:

In the calculation, Size Classes D – M are added together.
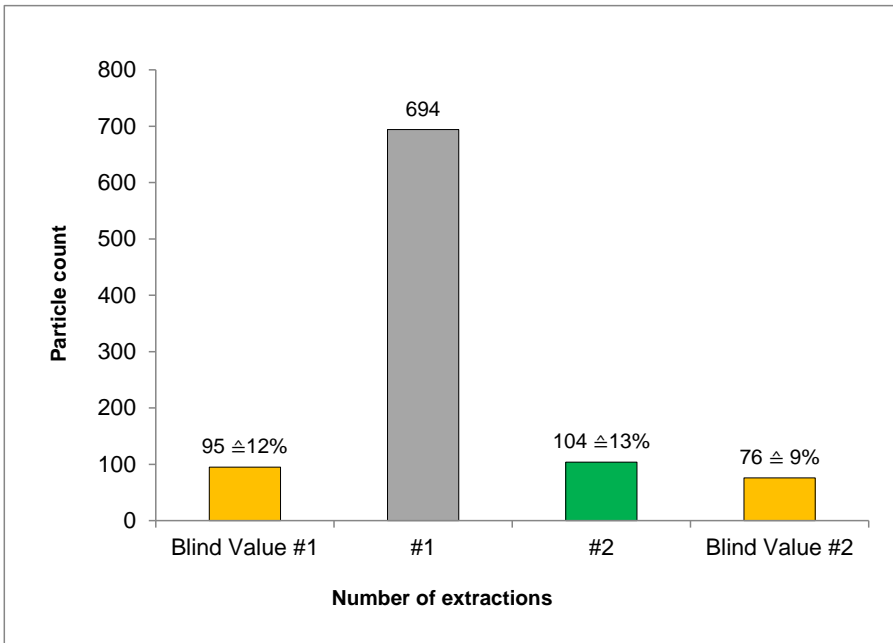
Double inspection 5.6 Functional area

The ratio between the second extraction step and the sum of the first two extractions is 0.13 (13%), thus fulfilling the double inspection criterion of maximum 0.3 (30%). In principle, the extraction procedure is suitable for inpecting the cleanliness of the component.

Verification of the blank value:

Particle count per size class										
Size Class	D	E	F	G	H	I	J	K	L	M
Length Ferret$_{max}$ in µm	25 ≤ x < 50	50 ≤ x < 100	100 ≤ x < 150	150 ≤ x < 200	200 ≤ x < 400	400 ≤ x < 600	600 ≤ x < 1000	1000 ≤ x < 1500	1500 ≤ x < 2000	2000 ≤ x
Cleanliness value	500	250	100	50	10	5	0	0	0	0
Max. permissible blank value 10 %	50	25	10	5	1	0	0	0	0	0
Blank Value #1	54	34	7	0	0	0	0	0	0	0
Blank value i.O.	No	No	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
Blank Value #2	54	17	5	0	0	0	0	0	0	0
Blank value i.O.	No	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes

Remark:

The maximum permissible blank values were determined based on the required limit values for each size class. When evaluating one of the size classes, it can be observed that blank value limits were sometimes exceeded.

Conclusion:

The inspection procedure for the functional areas (oil borings) enables component cleanliness to be accurately assessed because the double inspection criterion of 0.3 (30%) is upheld.

In order to fulfil the blank value criterion demanded in the guideline, measures need to be taken to reduce the blank value percentage. One possible measure which could be taken regarding the extraction equipment would be to decrease the wetted surface area of the testing equipment, for example.

12.6.5 Routine inspections

Both of the component areas of the crankshaft were inspected to assess their respective overall contamination levels and possible damaging potential. A total of 5 crankshafts were investigated. In the routine inspections, all particles > 25 µm were considered. An element analysis of the residue particles from the functional areas was also performed by means of SEM/EDX.

Results of the routine inspections – total component surface:

All particles excluding fibers

Particle count per size class										
Size Class	D	E	F	G	H	I	J	K	L	M
Length Ferret$_{max}$ in µm	25 $\leq$ x < 50	50 $\leq$ x < 100	100 $\leq$ x < 150	150 $\leq$ x < 200	200 $\leq$ x < 400	400 $\leq$ x < 600	600 $\leq$ x < 1000	1000 $\leq$ x < 1500	1500 $\leq$ x < 2000	2000 $\leq$ x
Component #1	4986	2375	976	481	198	87	43	7	0	0
Component #2	5231	2288	1053	475	189	64	49	8	1	0
Component #3	4871	2653	876	512	242	75	38	6	1	0
Component #4	4754	2198	956	489	232	85	27	4	0	0
Component #5	5103	2474	931	432	179	91	34	7	2	0

All particles excluding fibers > 25µm
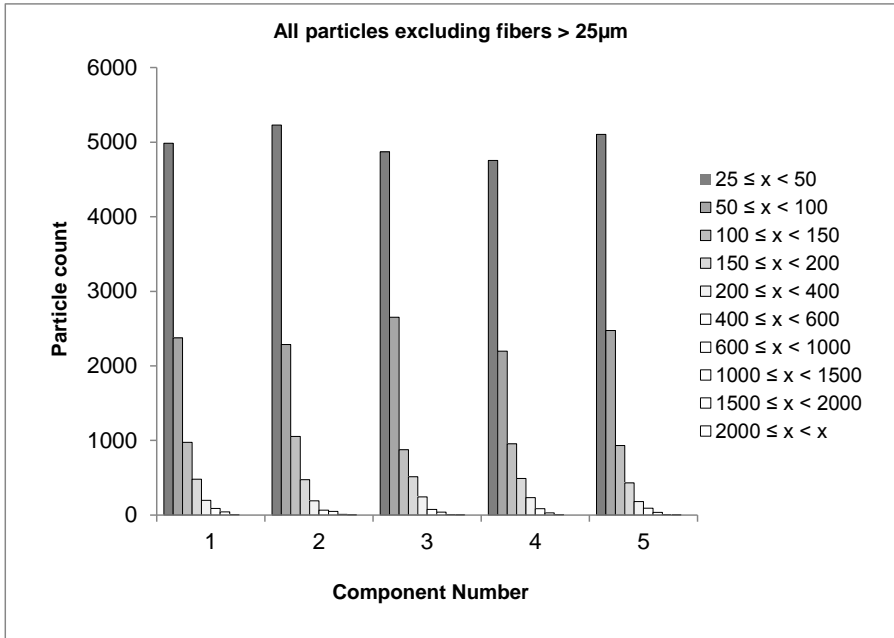
Quantity of residue particles excluding fibers 5.6 Overall component

Results of the routine inspections – total component surface:

Metallic shiny particles

Particle count per size class										
Size Class	D	E	F	G	H	I	J	K	L	M
Length Ferret$_{max}$ in µm	25 ≤ x < 50	50 ≤ x < 100	100 ≤ x < 150	150 ≤ x < 200	200 ≤ x < 400	400 ≤ x < 600	600 ≤ x < 1000	1000 ≤ x < 1500	1500 ≤ x < 2000	2000 ≤ x
Component #1	2632	879	481	189	98	41	4	0	0	0
Component #2	2477	987	521	238	87	36	8	1	0	0
Component #3	2164	574	378	156	81	25	5	0	0	0
Component #4	2576	789	403	221	67	29	3	1	0	0
Component #5	2298	831	289	176	78	27	8	0	0	0
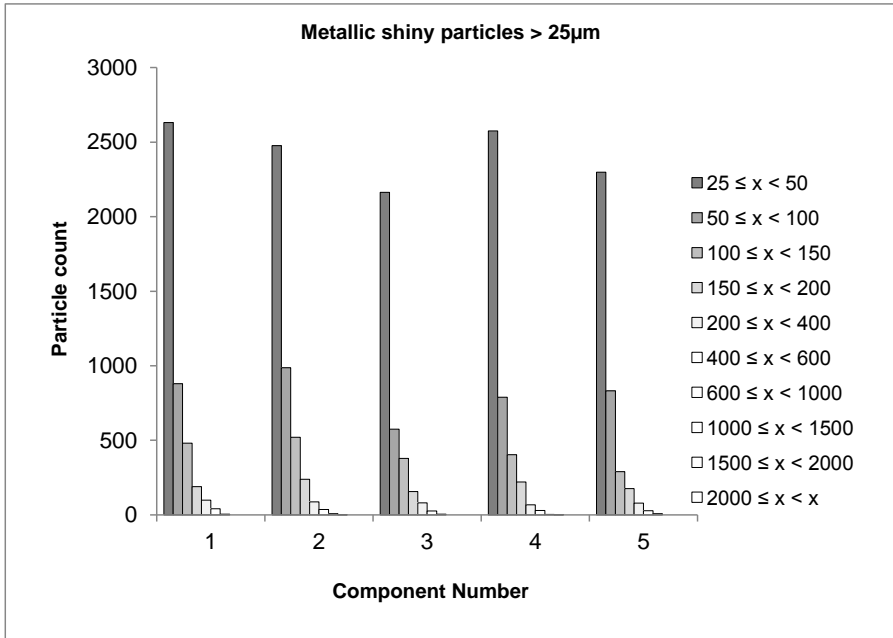
Quantity of metallic shiny residue particles 5.6 Overall component

Results of the routine inspections – functional area:

All particles excluding fibers

Particle count per size class										
Size Class	**D**	**E**	**F**	**G**	**H**	**I**	**J**	**K**	**L**	**M**
Width Ferret$_{min}$ in µm	25 ≤ x < 50	50 ≤ x < 100	100 ≤ x < 150	150 ≤ x < 200	200 ≤ x < 400	400 ≤ x < 600	600 ≤ x < 1000	1000 ≤ x < 1500	1500 ≤ x < 2000	2000 ≤ x
Component #1	498	237	97	48	9	6	1	0	0	0
Component #2	523	228	105	47	7	5	0	0	0	0
Component #3	487	265	87	51	12	7	0	0	0	0
Component #4	475	219	95	48	2	2	1	0	0	0
Component #5	510	247	93	43	7	1	0	0	0	0

All particles excluding fibers > 25µm

Legend:
- 25 ≤ x < 50
- 50 ≤ x < 100
- 100 ≤ x < 150
- 150 ≤ x < 200
- 200 ≤ x < 400
- 400 ≤ x < 600
- 600 ≤ x < 1000
- 1000 ≤ x < 1500
- 1500 ≤ x < 2000
- 2000 ≤ x <
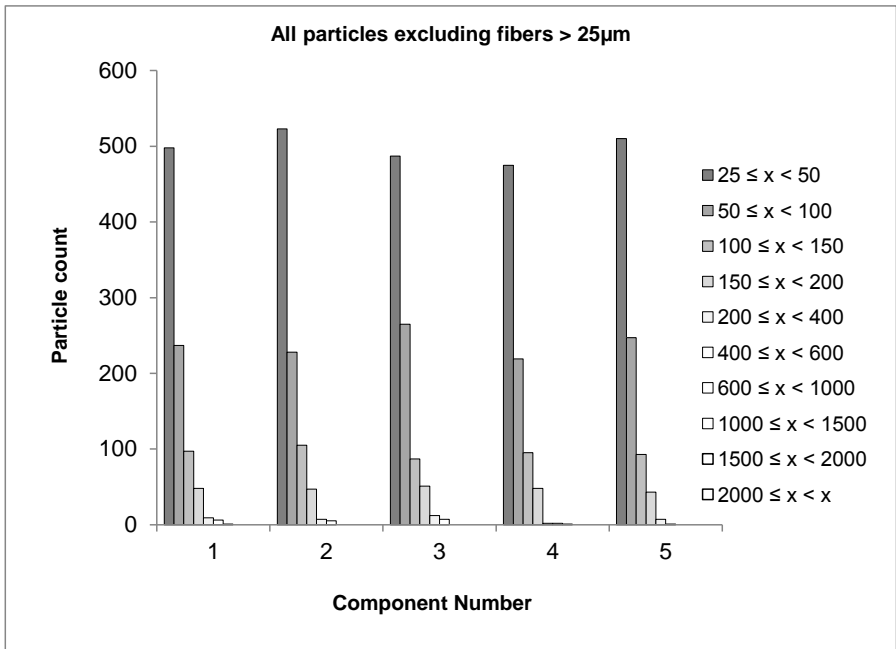
Quantity of residue particles excluding fibers 5.6 Functional area

Results of the routine inspections – functional area:

Metallic shiny particles

Particle count per size class										
Size Class	D	E	F	G	H	I	J	K	L	M
Width Ferret$_{min}$ in µm	25 ≤ x < 50	50 ≤ x < 100	100 ≤ x < 150	150 ≤ x < 200	200 ≤ x < 400	400 ≤ x < 600	600 ≤ x < 1000	1000 ≤ x < 1500	1500 ≤ x < 2000	2000 ≤ x
Component #1	263	87	48	6	1	0	0	0	0	0
Component #2	247	98	52	9	6	0	0	0	0	0
Component #3	216	57	37	3	4	1	0	0	0	0
Component #4	257	78	40	5	2	1	0	0	0	0
Component #5	229	83	28	1	3	0	0	0	0	0
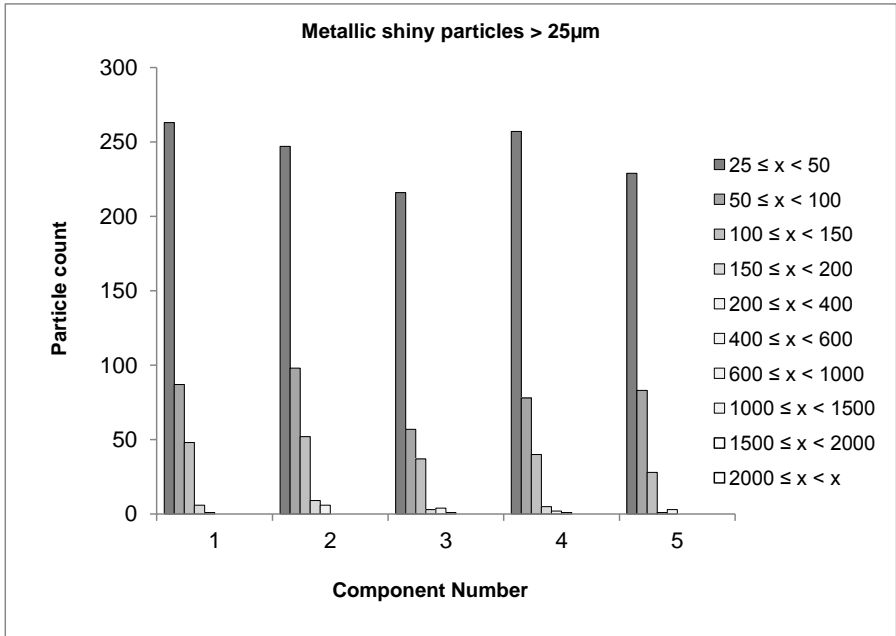
Quantity of metallic shiny residue particles 5.6 Functional area

Results of the SEM-EDX analysis

Particle count per size class										
Size Class	D	E	F	G	H	I	J	K	L	M
Width Ferret$_{min}$ in µm	25 ≤ x < 50	50 ≤ x < 100	100 ≤ x < 150	150 ≤ x < 200	200 ≤ x < 400	400 ≤ x < 600	600 ≤ x < 1000	1000 ≤ x < 1500	1500 ≤ x < 2000	2000 ≤ x
Steel										
Component #1	174	78	15	3	0	0	0	0	0	0
Component #2	136	65	12	5	4	0	0	0	0	0
Component #3	116	47	8	1	2	1	0	0	0	0
Component #4	145	61	21	1	0	1	0	0	0	0
Component #5	129	39	6	0	1	0	0	0	0	0
Corundum										
Component #1	78	24	2	0	0	0	0	0	0	0
Component #2	89	32	0	0	0	0	0	0	0	0
Component #3	65	18	3	0	0	0	0	0	0	0
Component #4	72	9	1	0	0	0	0	0	0	0
Component #5	69	28	0	0	0	0	0	0	0	0

Conclusion:

The five analyses show comparable results. This not only indicates a uniform level of cleanliness of the tested components but also a stable test procedure.

However, although the cleanliness level of the components is close to the cleanliness specification requirements, these are not fully upheld.

13 DEFINITIONS, ABBREVIATIONS AND SYMBOLS

13.1 Terms and definitions

A

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

Agglomerate: collection of solid matter to form a larger structure

Agitation: extraction method implemented for internal surfaces; its cleaning effect is based on the turbulent change in direction of the extraction liquid inside the component

Air cleanliness class: specification of air quality based on the concentration of particles in a defined volume of air (according to ISO 14644-1)

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

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

Analysis liquid: liquid containing the particle load to be analyzed

Analysis parameters: settings on an analysis system that are used in the analysis step

Analysis system: device to measure and/or characterize particles

B

Blank value criterion: maximum value that may not be exceeded when determining the blank value, i.e. max. 10% of the required or anticipated cleanliness value

Blank value: particle load accruing during a cleanliness inspection that does not originate from the test component but rather from the extraction

apparatus and/or utensils used, the environment and possibly also from members of staff

C

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

Cleaning: process to make a liquid, object or extraction apparatus as clean as required

Cleanliness Level: a number (coded) assigned to a specific particle count that is numerically incorporated into the CCC

Cleanliness inspection: inspection of technical cleanliness including documentation

Cleanliness specification: documentation of permissible particle features and quantities for a component

Cleanliness state (also component cleanliness): cleanliness value or values of a component that may change over time due to external influences

Cleanliness value: a single value specifying the cleanliness of a component, e.g. residue weight, longest particle or particle count

Coincidence: particles grouped together, which can therefore not be analyzed as single particles, e.g. by an optical particle counter or microscope

Collecting container (also: collecting vessel): container to collect the extraction liquid for later filtration

Component Cleanliness Code (CCC): alphanumeric character sequence containing information about the particle size distribution in coded form

Conditioning: preparation step in which the analysis filter is pressure-rinsed, dried and dehumidified before its tare weight is weighed

Control area: general term for an inspection surface or control volume of a test component from which residues are to be extracted

Critical particle: a particle whose geometric dimensions or chemical-physical nature could cause component damage. A sub-category of critical particles is formed by so-called "killer" particles, which cause an immediate component failure.

D

Decline: on repeated extraction, a decrease in the contamination level of a component occurs

Declining criterion: parameter that shows whether a decline has occurred

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.

Declining value: quotient of the cleanliness value under consideration and the sum of all preceding cleanliness values (including that under consideration), expressed in percent

Desiccator: laboratory device for cooling the analysis filter in a completely dry atmosphere

Direct inspection: analysis method capable of detecting contamination directly on the component surface without the need for an extraction or filtration step

Double inspection: procedure in which two identical extraction steps are performed in order to confirm the efficacy and suitability of qualified extraction parameters; here the declining criterion is set at 30% instead of the 10% that applies for declining tests

E

Effective filter surface area: area of the analysis filter membrane through which analysis liquid flows during the filtration step

Element analysis (EDX): Energy Dispersive X-ray spectroscopy capable of analyzing elements based on their characteristic x-ray spectra

Extraction (also: sampling): procedure used to detach particulate residues from a test component with the aid of an extraction fluid

Extraction apparatus: equipment used to perform the extraction

Extraction liquid: see inspection medium

Extraction method: technique to detach the particle load from the test component

Extraction parameters: totality of all physical parameters influencing the extraction which can be set on or calculated from the extraction apparatus

Extraction procedure: complete sequence of all extraction steps performed

Extraction step (also: sampling step): single work step forming part of the extraction procedure that is performed either in a declining test or when sampling several control areas

F

Fiber: long, thin structure which is defined by the following: Ratio between stretched length and maximum inner circle diameter is greater than 20; the width measured via maximum inner circle diameter is lower or equal to 50 µm

It is generally used to characterize a textile fiber (flexible, pliable, made from organic materials) and differentiate it from compact particles. Warning: hard fiber-shaped structures such as glass fibers are also detected as fibers

Filter background: color of the analysis filter

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

Filter membrane: see analysis filter

Filter occupancy: surface area of the analysis filter covered by particles, expressed in percent

Filter pore size: for mesh filters, this equates to the nominal mesh width; for foamed analysis filters, it corresponds with the equivalent mesh width measured by means of the bubble point test.

Filtration: process in which particles are deposited on the analysis filter

Final rinsing liquid: liquid used after the extraction step to remove any particulate residues from the surfaces of the extraction apparatus and deposit them on the analysis filter

Fixative: liquid used to fix particles on the analysis filter to prevent their loss though electrostatic charging

Flat jet nozzle (also: fan nozzle): nozzle that generates a linear jet on impaction on a smooth surface. Parameters include jet width and equivalent bore diameter.

G

Gravimetry: analysis method used to determine the mass of all residues present on the analysis filter by measuring differences in weight

I

Inspection medium: general term for an extraction liquid or air utilized to detach contamination from the test component

Inspection procedure: term used to describe the full sequence of all work steps (preparatory steps, extraction, filtration, analysis) carried out in the course of a cleanliness inspection.

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

Inspection specification (also: inspection guideline): detailed description of the test procedure (where appropriate, also with the inclusion of illustrations, etc., to provide assistance)

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

Internal rinsing apparatus: technical construction consisting of a media supply and set-up to integrate the test component. It is used exclusively to perform an internal rinsing extraction step, e.g. an adapted test bench for a hydraulic component

Isolation: screening of areas of the test component that do not form part of the control area and which should not come into contact with the extraction fluid; sealing or masking are examples of isolation methods

L

Limit value: maximum permissible value for the residue weight, particle dimension (length, width, height) or particle quantity, which may not be exceeded

M

Material analysis: general term for analyses that accurately characterize a material by means of chemical, spectroscopic, mechanical or metallographic methods

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

Microscope: device used to magnify and visualize small objects, often containing an integrated camera system with image-processing. A differentiation is made between material, stereo and zoom microscopes

Monitoring: inspection of cleanliness at regular intervals with the purpose of evaluating processes relevant to cleanliness

O

Optical particle counter: inspection device used to count particles in clear liquids and measure their size; function based on the principle of scattered light or extinction

P

Particle load: totality of all particles present on a surface or in a liquid

Particle overlap: particles overlying one another, which prevents them from being optically recorded as single particles

Particle size: geometric feature of a particle that is stated in the cleanliness specification. If this is not stated, the particle size corresponds with the longest dimension (Feret$_{max}$)

Particle size class (also: size class): particle size range with an upper and lower class limit

Particle size distribution: particle count grouped into particle size classes

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

Particle: tiny structure made from solid organic or inorganic matter

Post-treatment liquid: liquid used after the filtration step to remove any filmy contaminants from the analysis filter that were not dissolved by the extraction fluid, e.g. anti-corrosives, oils or cooling lubricants

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

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 control area to be detached during the extraction step, e.g. disassembly, demagnetization, isolation, pre-cleaning

Q

Qualification report: document forming part of the inspection specification that only contains the results of the declining test, or an independent document containing general information together with details of the test component, information about the extraction, filtration and analysis steps as well as about the routine inspection conditions. Documentation of the qualification test (declining test and derivation of the routine inspection procedure)

Qualification test: test procedure for recurrent routine inspections that is determined by carrying out experiments

R

Residue weight: mass of the particle load determined as the weight difference using the method of gravimetric analysis

Routine inspection: cleanliness inspection performed with qualified extraction parameters (recurrent)

S

Sampling: see extraction

Spray nozzle geometry: geometric features affecting the shape of the jet and pressure-rinsing pattern

T

Test conditions: circumstances under which the cleanliness inspection is performed

Test lot size: number of test components comprising the test lot

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

Test object (also: test piece or component): single component, aggregate or system inspected for cleanliness

Turbulent flow: a flow of fluid with a cleaning effect generated by turbulences that is used to extract contaminants from internal component geometries. An effective turbulent flow prevails in a liquid if the following applies: Reynolds number ≥ 4000

U

Ultrasonics: extraction method with a cleaning effect based on the formation and subsequent collapse of vapor bubbles (cavitation) that is induced by high sound pressures. Frequencies ranging between 20 kHz and 400 kHz are used (in some cases up to 1 GHz)

Ultrasound bath: technical device used to clean components by means of ultrasonics

V

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

Wetted surface area/wetted volume: control area, expressed as surface area in cm²/volume in cm³, coming into contact with the extraction liquid during the extraction step. It is generally derived from the surface area or volume that is wetted when the component is in later operation.

13.2   Abbreviations and symbols

A:	Unit of reference "per 1000 cm²" when specifying the CCC
A_C:	Wetted surface area of all test objects in a test lot
ATR:	Attenuated Total Reflexion
BSE detector:	Back-Scattered Electron detector
C_i:	Cleanliness value
CCC:	Component Cleanliness Code
CCD:	Charge coupled device
CT:	Computer tomograph
d:	Internal diameter (e.g. of piping)
EDX:	Energy Dispersive X-ray spectroscopy
FT-IR:	Fourier transformation infrared spectrometer
G_C:	Particle load mass related to number of components tested
G_A:	Mass of particle load in relation to wetted surface area
G_V:	Mass of particle load in relation to wetted volume
h:	Absolute particle count
HC:	Hydrocarbon
H_C:	Particle count in relation to test lot size
H_A:	Particle count in relation to wetted surface area
H_V:	Particle count in relation to wetted volume
IR:	Infrared
ISO-MTD:	ISO mean test dust
λ:	Wavelength
LIBS:	Laser-induced breakdown spectroscopy
LM:	Light microscope
MAC:	Maximum allowable concentration
M1:	Tare weight of analysis filter
M2:	Total weight of occupied analysis filter
M:	Residue weight 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

ν:	Kinematic viscosity [m²/2]
OPC:	Optical Particle Counter
PET:	Polyethylene terephthalate
Re:	Reynolds Number, Re = w * d / ν
SEM:	Scanning Electron Microscope
SE detector:	Secondary electron detector
US:	Ultrasonics
VbF:	German directive on flammable liquids
V_C:	Wetted volume of all test objects in a test lot
w:	Velocity of the liquid [m/s]
x:	Variable for the particle size, e.g. 150 µm ≤ x < 200 µm
X.	Maximum permissible particle size
z:	Particle height

14   INDUSTRIAL ALLIANCE TECSA (INFORMATIVE)

Background

Since the anti-blocking system (ABS) came onto the market and especially since diesel direct injection systems became popular in the second half of the 90's, technical cleanliness has fully established itself as a quality criterion in the automotive and supplier industries. Not only major efforts on the part of large concerns in the cleaning technology industry but also generally in the field of clean manufacturing have led to a growing demand for a standardized method for inspecting particulate contamination. As a result, in 2001 the industrial alliance TecSa was founded through which, in the course of a two-year cooperation, the first VDA Volume 19 concerned with the inspection of the technical cleanliness of functionally-relevant automotive components was written. Under the expert guidance of Fraunhofer IPA and in collaboration with 25 companies predominantly active in the automotive and supplier industries, the world's first standard on this subject was created.

The industrial alliance

Ever since the publication of VDA Volume 19 in January 2005, the topic of technical cleanliness has constantly gained in importance. A new profession has also evolved – that of the "technical cleanliness inspector". In 2014, component cleanliness was inspected in approximately 1000 laboratories in the automotive and supplier industries: Around 40 laboratories and firms in Germany offer services related to technical cleanliness. In 2012, subject to the experiences made over recent years in this field together with the numerous changes taken place or improvements made in cleanliness inspection technology, it became clear that VDA Volume 19 needed to be revised. In the summer of 2012, during an open industrial workshop the following points were identified as primary revision aims:

- Improved comparability of analysis results

- Inclusion of additional parameters and analysis methods

- Standardized procedure for deriving limit values

- Procedures for dealing with cleanliness values in quality control (in the event that limit values are exceeded, escalation, etc.)

- Information about protection of the health of inspection staff.

Especially with today's advancing globalization, this issue has become highly relevant in places where technical cleanliness is inspected in world-wide supplier networks.

In December 2012, within the scope of revising VDA Volume 19, the industrial alliance TecSa 2.0 was founded and began its 18-month long project. With Fraunhofer IPA acting as a neutral expert coordinator, in this cooperation comprising 41 companies and three associations the second revised edition of VDA Volume 19 was written. The changes and additions were worked on in four sub work-groups, discussed in approx. 35 technical meetings and agreed on by consensus of opinion. The fundamental methods described in the first VDA 19, such as extraction, filtration and analysis, as well as the qualification of methods by means of declining tests, have proved in practice to be highly effective and have therefore not been altered in any way. The changes made in order to achieve the above-mentioned objectives are summarized briefly in Chapter 1.

We would like to thank all the people involved for their motivation, constructive advice, active support, fine teamwork and faith in the work carried out by Fraunhofer IPA.

Dr.-Ing. Markus Rochowicz

Fig. 13-1: Members of the industrial alliance TecSa 2.0 at the final meeting

Table 13-1: Companies and associations participating in the industrial alliance TecSa 2.0

Automotive manufacturers	Supplier companies	Service firms and technical suppliers
AGCO GmbH (FENDT)	Alupress	Brändle
Audi	Behr	CleanControlling
BMW	Bosch	Dürr Ecoclean
Daimler PKW	Continental	Gläser
Daimler Trucks	EJOT	HMT Häseler Metalltechnik
MAN	Freudenberg	HYDAC
Porsche	FTE	JOMESA
Volkswagen	GETRAG	Leica
	Hengst	Nerling Systemräume
	INA-Schaeffler	PALL
	Mahle	PAMAS
	MANN+HUMMEL	Quality Analysis
	Modine	Rap.ID
	SKF	RJL
	VOSS Automotive	Zeiss
	Walter Söhner	
	Willy Voit	
	Witzenmann	
	ZF	

Associations

FIT Fachverband Industrielle Teilereinigung e.V.

IBU Industrieverband Blechumformung

VDFI Verband der deutschen Federnindustrie

Coordination and expert management of the industrial alliance:

Fraunhofer IPA

Department of Ultraclean Technology and Micromanufacturing (www.technische-sauberkeit.de)

Nobelstraße 12

70569 Stuttgart

Germany

Dr.-Ing.	M. Rochowicz	rochowicz@ipa.fhg.de
Dipl.-Ing. (FH)	S. Boos	boos@ipa.fhg.de
Dipl.-Wirt.Ing.	P. Brag	brag@ipa.fhg.de
Dipl.-Ing.	C. Ernst	ernst@ipa.fhg. de
	Y. Holzapfel	holzapfel@ipa.fhg.de

Quality Management in the Automotive Industry

Current editions of the published VDA volumes on quality management in the automotive industry (QAI) can be found in the Internet under http://www.vda-qmc.de.

They can also be ordered directly via this homepage.

Reference:

Verband der Automobilindustrie e.V. (VDA)
Quality Management Center (QMC)
Behrenstr. 35
10117 Berlin
Germany

Tel +49 (0) 30 89 78 42-235

Fax +49 (0) 30 89 78 42-605

E-mail: info@vda-qmc.de

VDA QMC

Qualitäts Management Center im
Verband der Automobilindustrie