agitation unit. To keep particles in standing samples better in suspension, a stirrer should be utilized. Magnetic stirrers are unsuitable because they bind ferromagnetic contaminant particles and exclude them from the analysis (falsification of results).

9.4.2.3 Procedure

The sample is analysed in compliance with standards such as USP 788 or ISO 11500. When preparing and configuring the analysis set-up, it is essential that no air bubbles are present in the sample.

Standing liquid samples:

Before being analysed, all standing samples shall be agitated to homogenize particle distribution. A subsequent resting phase is then required to enable any air bubbles to rise and dissipate; at the same time, the sample should be kept homogeneous by stirring it at an appropriate speed.

NOTE Floating particles (e.g. lightweight fragments of plastic) cannot be detected with this analysis technique.

Use with pressurized tubing:

This method is generally used in conjunction with a functional test bench or apparatus for rinsing the interior of components. The apparatus and procedure shall prevent the presence of any air bubbles in the system. To ensure this, where possible the test component (fuel line, etc.) is "flooded" with liquid before commencing the actual internal rinsing step. To enable accurate analysis results, a specified constant flow rate is usually necessary.

9.4.2.4 Documentation

The following information shall be included:

— general description of the analysis device and sample feed system;

— date and type of the last calibration (polystyrene or ISO-MTD);

— sampling technique: sample container or direct connection to rinsing line;

— explicit remark to make it clear that the quoted particle sizes refer to equivalent sizes and not dimensions like e.g. $Feret_{max}$;

— when listing absolute quantities (not concentrations), the volume of liquid actually analysed shall be stated;

— when giving information about concentrations, the original volume of liquid used in the extraction step shall be stated;

— volume analysed expressed in percent in cases where the sensor effectively only detects/analyses a fraction of the through-flow liquid;

— dilution factor for highly-concentrated samples;

— total volume of liquid analysed;

— coincidence concentration.

9.4.3 Filter-blocking (optical)

9.4.3.1 Principle

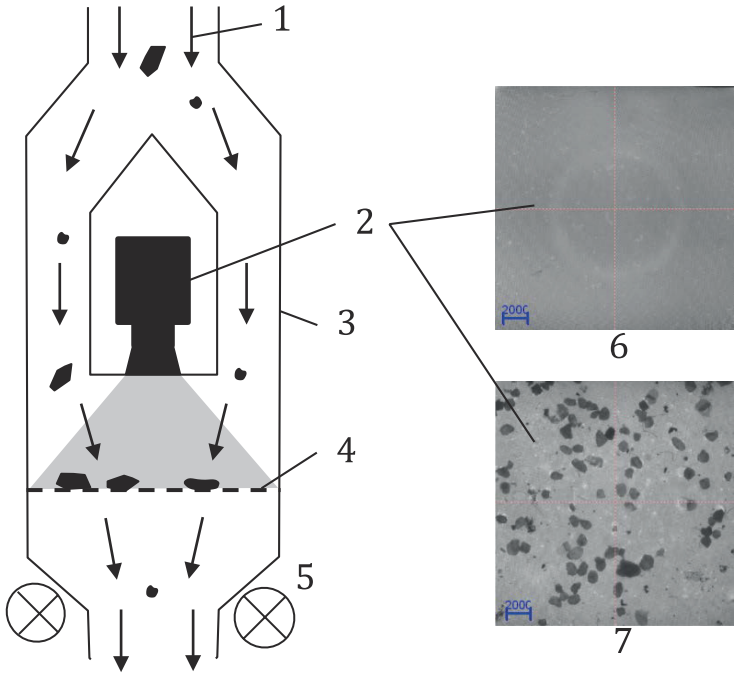
With this analysis method, particles from the flow of liquid (extraction liquid containing particles) are deposited directly on an analysis filter that is situated in the field of view of a camera (see Figure 23).

The pore size of the analysis filter is selected to enable only relevant particle sizes to be retained and smaller particles to pass through the filter. The measuring cell, which is located for example at the outlet of an extraction setup where the filter is normally clamped, is fitted with a (transmitted light) illumination source. This provides a high degree of contrast between the particles deposited on the filter and the filter background and assures their detection by the camera.

Further image processing and analysis steps are carried out conform to the light-optical analysis method (binarization and particle measurement). As opposed to automated microscope systems, instead of several image fields being combined to form an overall image, in this case only one image field is analysed. As a result, this system does not offer the optical resolution of microscope systems but is quite suitable for detecting largish single particles.

In each analysis, two images are evaluated — one before the extraction step and one after deposition of the contaminant particles on the filter. The difference between the two corresponds with the particle load extracted from the component.

If particle occupancy on the analysis filter is too high and analysis errors occur due to particles touching or overlapping one another, the analysis filter is backwashed to remove (the majority of) the particle load. Backwashing is performed by reversing the flow of liquid with the aid of suitable pumps and valves or by putting the analysis filter in the other way round.



Key

1	direction of flow of liquid from extraction apparatus	5	illumination
2	camera	6	start of the measurement
3	measuring cell	7	end of the measurement
4	analysis filter		

Figure 23 — Cell for the optical analysis of the filter blockade

9.4.3.2 Material and equipment

a) Measuring cell with:

— camera,

— analysis filter,

— (transmitted light) illumination,

— system for backwashing the analysis filter, and

— image processing and analysis.

b) Adapter to extraction setup and return feed of test liquid.

9.4.3.3 Procedure

The following procedure shall be adapted to the features of the respective extraction device and analysis system.

a) Record and analyse an image of the analysis filter to assess its initial state.

b) Perform the extraction procedure, including the final rinsing step. The complete extraction liquid and final rinsing liquid is then guided through the measuring cell and analysis filter.

c) Record and analyse an image of the analysis filter after the extraction step.

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

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

9.4.3.4 Documentation

Particles are analysed and documented in accordance with the procedure for light-optical analysis and grouped into the particle size classes stated in Clause 10.

10 Documentation

10.1 Overview

This clause specifies the content of the inspection documents, not the format.

Different documents are generated in the course of a cleanliness inspection (see Figure 24). Depending on the type of cleanliness inspection performed, either a qualification report or an inspection report is written.

The qualification report documents the test conditions, extraction parameters and the results of the qualification tests (declining tests), with the latter being used to determine the routine extraction procedure and thus the related inspection specification.

The inspection report briefly lists the extraction parameters and test conditions as well as the results of routine inspections. In the inspection report, only information related to the inspection performed is required.

The inspection specification contains details of the extraction and analysis parameters together with a clear and easily understandable description of the inspection procedure. The description can be written in a summarized or full version with photos or illustrations being added if desired. The inspection specification may include proof of the qualification or refer to a separate document, the qualification report (for example see Annex G). Further references (e.g. supplementary agreements) are optional.

The cleanliness inspection document(s) shall contain all the information mentioned in 10.2 to 10.8 that is necessary to reproduce the cleanliness inspection and assess the results. Further information shall be added as required.

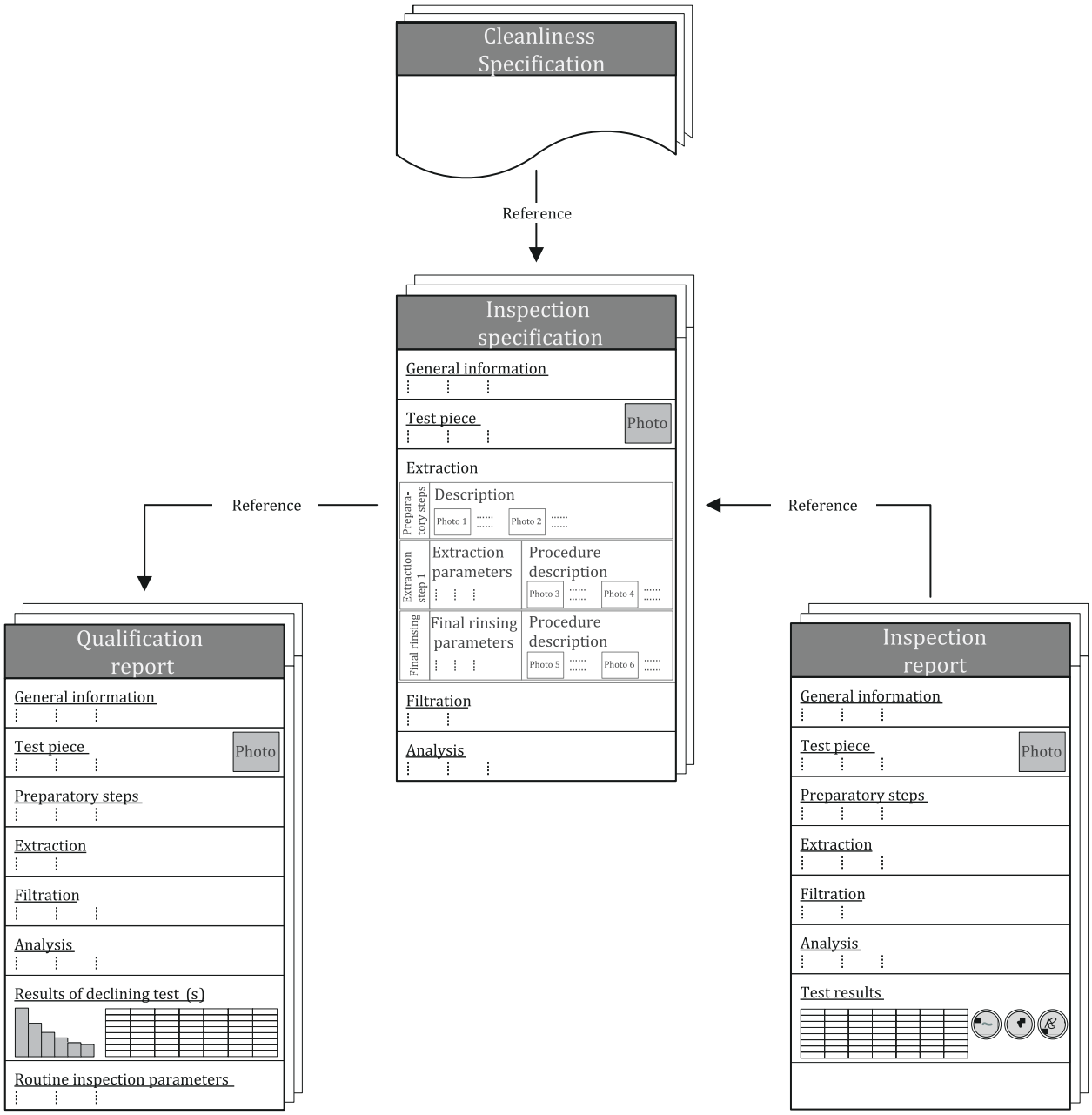


Figure 24 — Cleanliness inspection documents (overview and interrelations)

10.2 General information

"General information" (see Figure 25) covers higher level information to aid the classification and administration of the documents supplied.

General information

Order No.: ..... Report No.: .....
Customer: ..... Date of analysis: .....
Contact partner: .....

Test ordered: ☐ Routine inspection ☐ Double inspection ☐ Qualification test
Reason for test: ☐ Initial assessment ☐ Process control ☐ Modifications, constructional ☐ .....

Figure 25 — General Information

10.3 Information about the test component

The test component is described in more detail and identified clearly (see Figure 26). Additional information which may be useful could include:

— the material the test component is made from, and

— the presence of filmy residues on the test component.

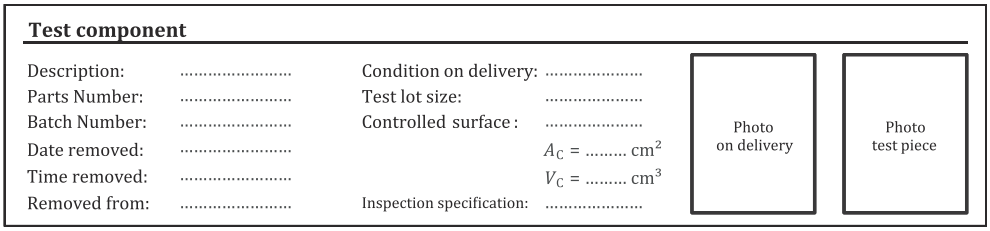


Figure 26 — Information about the test component

10.4 Information about preparatory steps

Information about preparatory steps indicates if and when supplementary work steps are required (see Figure 27). If no information about preparatory steps is mentioned, inspection results could be wrongly evaluated or misinterpreted.

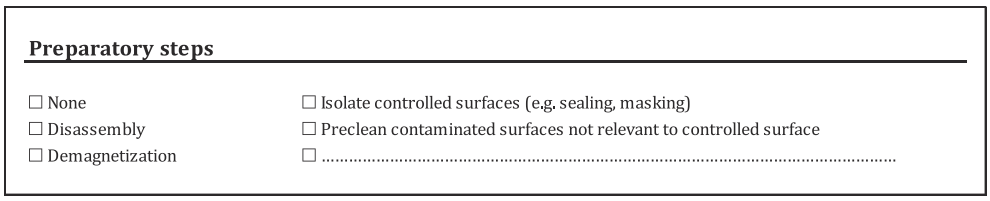


Figure 27 — Information about preparatory steps

10.5 Information about the extraction

10.5.1 General

The way the extraction step is performed may vary significantly according to the expertise of inspection staff involved. The degree of freedom regarding the extraction step is reduced by documenting the inspection procedure as clearly as possible (see Figure 28). This aims to make the extraction step reproducible and improve the comparability of inspection results. The number of possible answers should be restricted by the use of checkboxes or dropdown lists. Because the angle or feed rate of an open jet may fluctuate markedly during the extraction step, this information is optional.

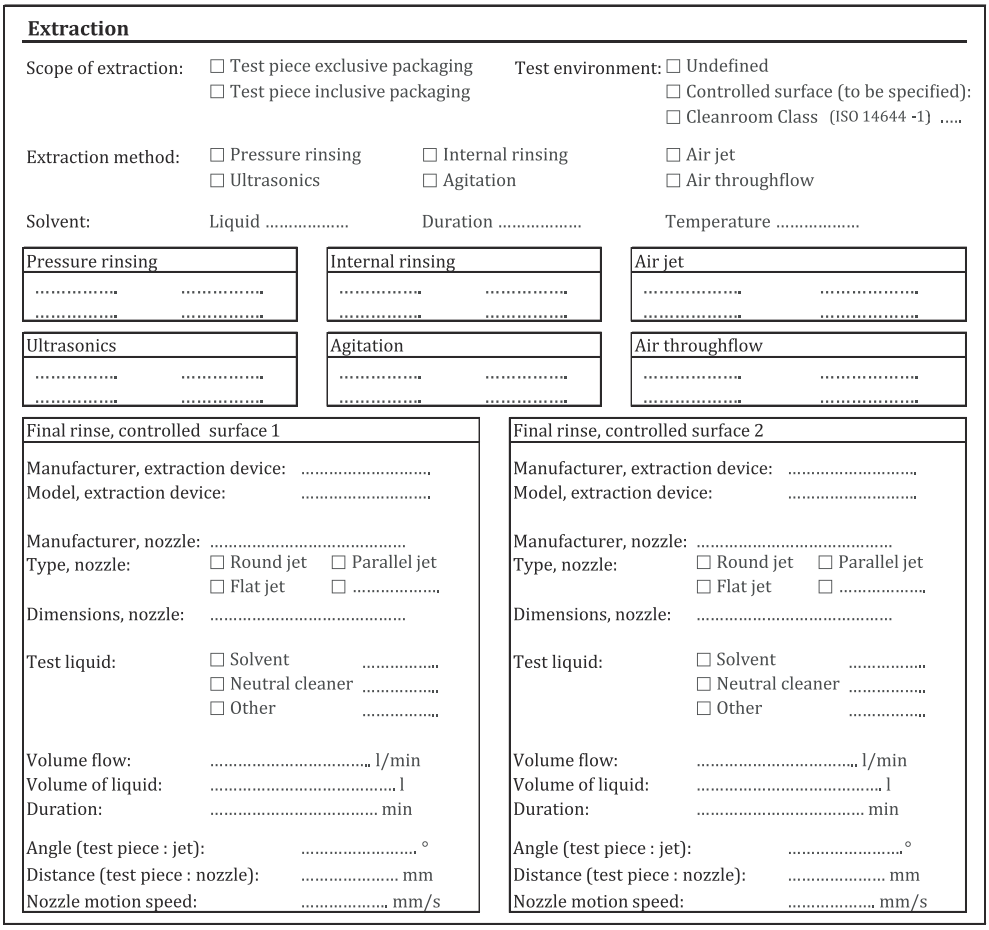


Figure 28 — Information about the extraction

10.5.2 Pressure-rinsing

Pressure-rinsing has the highest number of influencing parameters. Among other things, the cleaning effect of pressure-rinsing is influenced by the nozzle selected. Depending on the nozzle type, the following information should be noted in the section *Dimensions, nozzle*:

— *nozzle cross-section in mm* for a round jet (full jet) nozzle;

— *pressure-rinsing angle in* ° and *equivalent nozzle cross-section in mm* for a flat jet (fan-jet) nozzle;

— *nozzle cross-section in mm* and *number of bores* for a parallel jet nozzle.

Additional information can be documented as shown in Figure 29.

Pressure-rinsing

Controlled surface 1		Controlled surface 2	
Manufacturer, extraction device:	.....	Manufacturer, extraction device:	.....
Model, extraction device:	.....	Model, extraction device:	.....
Manufacturer, nozzle:	.....	Manufacturer, nozzle:	.....
Type, nozzle:	☐ Round jet ☐ Parallel jet ☐ Flat jet ☐	Type, nozzle:	☐ Round jet ☐ Parallel jet ☐ Flat jet ☐ .....
Dimensions, nozzle:	.....	Dimensions, nozzle:	.....
Test liquid:	☐ Solvent ..... ☐ Neutral cleaner ..... ☐ Other	Test liquid:	☐ Solvent ..... ☐ Neutral cleaner ..... ☐ Other .....
Volume flow:	..... l/min	Volume flow:	..... l/min
Volume of liquid:	 l	Volume of liquid:	 l
Duration:	..... min	Duration:	..... min
Angle (test piece : jet):	..... °	Angle (test piece : jet):	..... °
Distance (test piece : nozzle):	..... mm	Distance (test piece : nozzle):	..... mm
Nozzle motion speed:	..... mm/s	Nozzle motion speed:	..... mm/s

Figure 29 — Information about pressure-rinsing

10.5.3 Ultrasonic vibration

With ultrasonic vibration, more influencing parameters are known than are documented. As it is not easy to determine fluctuating sound pressures, pressure peaks and other variables, only details of the extraction parameters shown are documented (see Figure 30). It is not sufficient to note the ultrasonic output because this depends on the filling level of the ultrasonic device. Instead, information about the power density (output per filling volume) should be given.

Ultrasonic

Controlled surface 1		Controlled surface 2	
Manufacturer, extraction device:	.....	Manufacturer, extraction device:	.....
Model, extraction device:	.....	Model, extraction device:	.....
Test liquid:	☐ Solvent ..... ☐ Neutral cleaner ..... ☐ Other	Test liquid:	☐ Solvent ..... ☐ Neutral cleaner ..... ☐ 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

Figure 30 — Information about ultrasonic vibration

10.5.4 Internal rinsing

Internal rinsing may take different forms (see Figure 31). They range from simple internal rinsing, with the (pressure-rinsing) nozzle being applied hermetically to an opening, right up to complex internal rinsing systems, where parameters such as direction of flow, pulsation, etc. are all adjustable.

If the test liquid flows through the controlled surface in one direction with a periodically-changing flow rate, this is known as pulsation. The pulsation frequency is dependent on time and shall be stated.

Internal rinsing

Controlled surface 1		Controlled surface 2	
Manufacturer, extraction device:	.....	Manufacturer, extraction device:	.....
Model, extraction device:	.....	Model, extraction device:	.....
Manufacturer, nozzle:	.....	Manufacturer, nozzle:	.....
Type, nozzle:	☐ Round jet ☐ Parallel jet ☐ Flat jet ☐	Type, nozzle:	☐ Round jet ☐ Parallel jet ☐ Flat jet ☐ .....
Dimensions, nozzle:	.....	Dimensions, nozzle:	.....
Dimensions, adapter:	.....	Dimensions, adapter:	.....
Test liquid:	☐ Solvent ..... ☐ Neutral cleaner ..... ☐ Other	Test liquid:	☐ Solvent ..... ☐ Neutral cleaner ..... ☐ Other .....
Volume flow:	..... l/min	Volume flow:	..... l/min
Volume of liquid:	 l	Volume of liquid:	 l
Duration:	..... min	Duration:	..... min
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:	.....

Figure 31 — Information about internal rinsing

10.5.5 Agitation

Agitation is essentially a manual extraction method. The frequency and amplitude of the arm movements can neither be fixed nor measured but shall be approximated as best as possible in the documentation (see Figure 32).

The term frequency describes the up-and-down and side-to-side movement of the test component. The amplitude describes the distance of travel of the component during this movement, and the number of fillings indicates how often the test component was (re-)filled with clean liquid during the extraction step.

Agitation

Controlled surface 1		Controlled surface 2	
Manufacturer, extraction device:	.....	Manufacturer, extraction device:	.....
Model, extraction device:	.....	Model, extraction device:	.....
Test liquid:	☐ Solvent ..... ☐ Neutral cleaner ..... ☐ Other	Test 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:	.....

Figure 32 — Information about agitation

10.5.6 Air jet extraction

Air jet extraction is the same as pressure-rinsing except for the fact that air is used instead of a test liquid (see Figure 33).

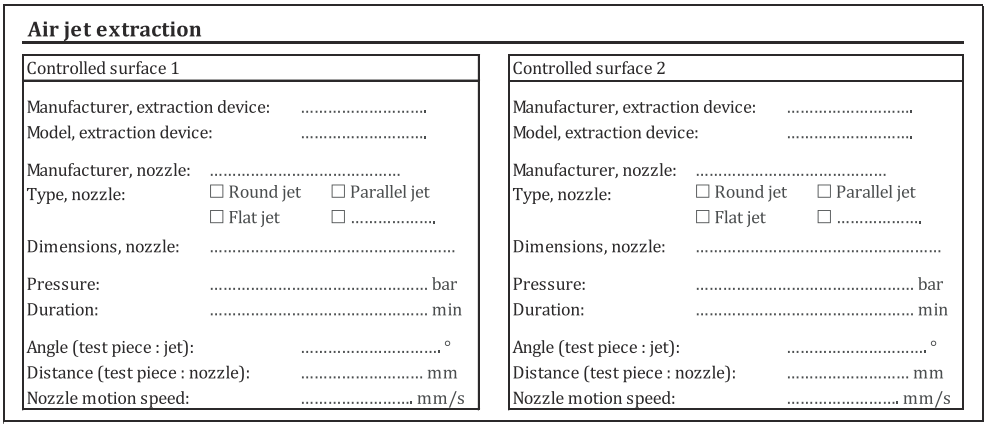


Figure 33 — Information about air jet extraction

10.5.7 Air through-flow extraction

Air through-flow extraction is the same as rinsing except for the fact that air is used instead of a test liquid (see Figure 34).

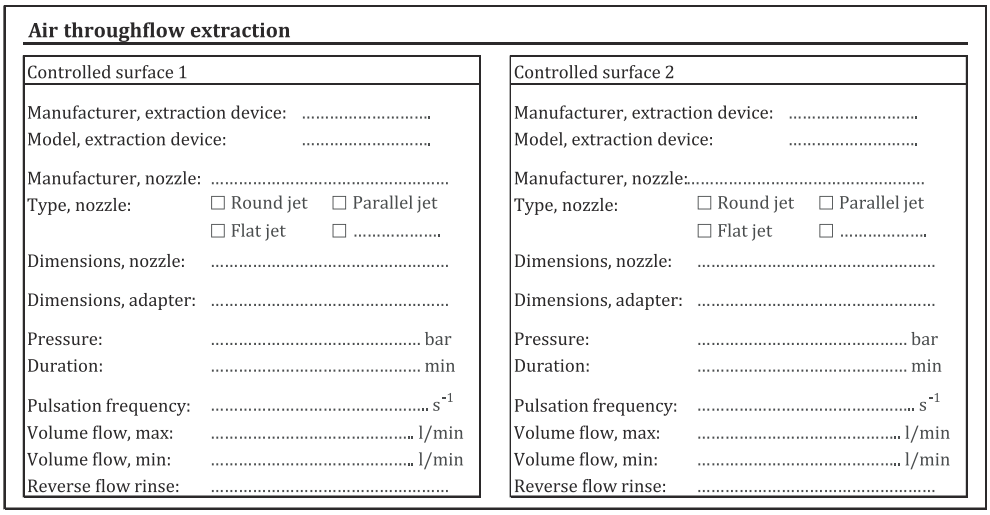


Figure 34 — Information about air through-flow extraction

10.6 Information about filtration

As well as documenting the actual filtration details, information about drying and any post-treatment steps should also be noted (see Figure 35).

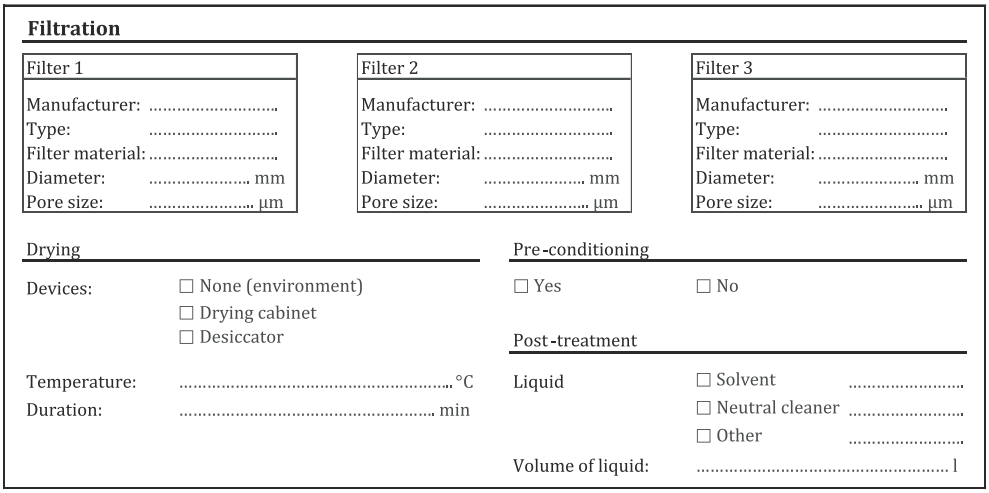


Figure 35 — Information about analysis filtration

10.7 Information about the analysis

10.7.1 General

Details and settings of the analysis system are documented. If the extraction step and analysis are performed in different places or different room qualities, the test environment shall be documented in the same way as shown in 10.5.

10.7.2 Standard analysis

10.7.2.1 Gravimetric analysis

Among other things, the weighing result of a gravimetric analysis is dependent on the room temperature. If this is not documented anywhere else, the room temperature should be noted in the gravimetric analysis (see Figure 36).

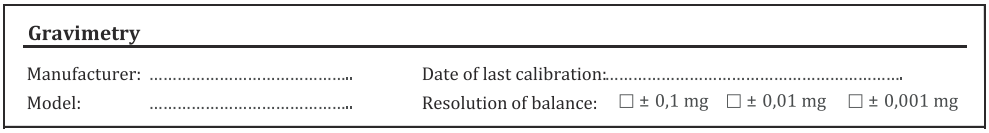


Figure 36 — Information about balance for gravimetric analysis

10.7.2.2 Light-optical analysis

Light-optical devices can be calibrated using particle standards. The date of the last calibration shall be documented. The scale shall be stated as the magnification per pixel in [µm/pixel] (see Figure 37).

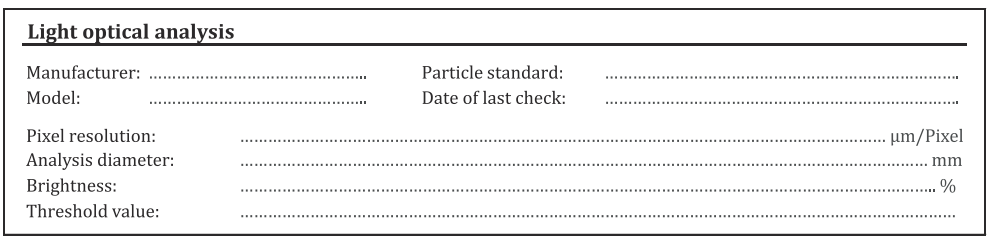


Figure 37 — Information about light optical analysis

10.7.3 Extended analysis (informative)

Information about extended analyses may vary depending on the analysis system utilized. An example of information about a scanning electron microscope with EDX detector is shown in Figure 38.

Scanning Electron Microscopy / Energy Dispersive X-ray spectroscopy (SEM / EDX)

Manufacturer: ..... Particle standard: .....
Model: ..... Date of last check: .....

Acceleration voltage: ..... keV
Analysis diameter: ..... mm
Working distance: ..... mm
Process: ☐ Automated ☐ Manual

Figure 38 — Information about SEM/EDX for extended analysis

10.7.4 Shortened analysis (informative)

The information shown in Figure 39 regarding a shortened analysis refers to an automatic particle counter.

Optical particle counter (OPC)

Manufacturer: ..... Coincidence limit: .....
Model: ..... Date of last calibration: .....
Calibration material: .....

Extraction liquid: ..... ml
Dilution factor: .....
Measuring interval: ..... min^{-1}
Nominal volume flow: ..... ml/min
Detection volume: ..... ml

Figure 39 — Information about optical particle counter for shortened analysis

10.8 Reporting of the inspection results

10.8.1 General

Information about cleanliness values shall be related to one of the following units of reference:

— number of components tested n = [1];

— wetted surface area of test component A_C = [cm^2];

— wetted volume of test component V_C = [cm^3].

10.8.2 Gravimetric analysis

The result of a gravimetric analysis is the residue mass $m = m_2 - m_1$, with the last digit being mathematically rounded. The masses m_2 and m_1 are read off using the resolving capacity of the weighing balance. The resulting particle mass m, which is calculated from the difference between the residue mass and the unit of reference, shall be documented.

Mass related to number of components tested

$$m_C = \frac{m}{n} = \left[\frac{\text{mg}}{1}\right]$$

The unit used is mg per (n) component(s).