Gefahrstoffe
Reinhaltung der Luft
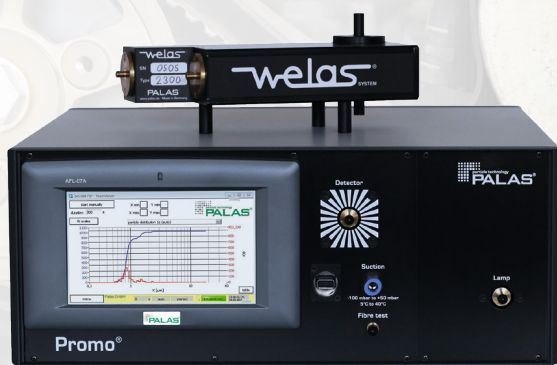
Air Quality Control

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Read inside:

**Dilution systems for test aerosols:
Fundamentals and potential applications**

L. Mölter, M. Schmidt

Dilution systems for test aerosols: Fundamentals and potential applications

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Summary Commercially available dilutions systems have proven their abilities to dilute solid aerosols in the particle size range from approx. 2 nm to 20 μm in practice. However, if aerosols with droplet diameters above approx. 3 μm are diluted, dilution systems often act as a separator. Highly concentrated droplet aerosols, such as those arising in the determination of the separation efficiency of oil mist separators (e.g. aerosols, compressed air aerosols after the compressor or with blowby mist in the engine), contain droplets in the diameter range up to approx. 8 μm, which have to be reproducibly diluted to reliably assess the performance of the separators. The working principles of dilution systems and a current new development, specifically for droplet aerosols, are presented.

1 Introduction

Aerosols are multi-phase systems in which the disperse phase consists of solid and/or liquid particles in a size range from approx. 2 nm to 100 μm and the continuous phase consists of a gaseous medium.
Examples of natural aerosols include:
- Salt particles from the sea,
- Volcanic eruptions,
- Desert dust,
- Smoke from forest fires,
- Pollen, spores,
- Viruses and bacteria,
- Fog.

Examples of anthropogenic aerosols include emissions from:
- Domestic heating systems,
- Agriculture,
- Traffic, e.g. from vehicles, ships and aircraft,
- Industry, e.g. from combustion processes, mechanical processes and chemical processes.

Aerosols can have a negative impact both on human health and on the operation of technical products. The particle concentration can, depending on the emission source, be more than 10^{18} m^{-3} in the case of particles with a diameter below 1 μm. Separation systems like filtration systems are used to remove aerosols. With special filter stages it is possible to achieve particle concentrations e.g. in a clean room of less than 100 m^{-3}. The particle concentration to be measured thus extends over 16 orders of magnitude, and the particle size range to be measured extends over 5 orders of magnitude.

1.1 Aerosol dilution for aerosol measurement devices

The quality of separators and filters is tested and compared with the aid of test aerosols in accordance with various norms and standards. These test aerosols are preferably produced with aerosol generators, which can typically pro-

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duce particle concentrations up to more than 10^{14} m^{-3}. Device parameters that characterize the performance capability of test aerosol generators are described in the standard VDI 3491, Blatt 1 [1].

Counting measurement methods, in particular aerosol spectrometers, are used for the metrological characterization of filters, separators and environmental aerosols, i.e. the particle size is determined on an individual particle and the particle concentration is calculated from the volumetric flow rate, which is accurately measured at the same time.

Aerosol spectrometers and other measurement systems are described in the series of standards VDI 3867, Blatt 1 bis Blatt 6 [2]. Depending on the measurement device, these measurement devices can be used to measure particles in a size range from approx. 2 nm to 100 μm even in high particle concentrations, without coincidence errors. ISO 21501–1 [3] lists the requirements for optical aerosol spectrometers (OAS), including the device parameters, and defines the calibration of these devices. Optical aerosol spectrometers come in many size classes, which means that they can characterize particle size distributions reliably and with a high resolution. In contrast to optical particle counters (OPCs), OAS devices can perform measurements in high particle concentrations, with some capable of performing measurements at concentrations as high as 10^{12} m^{-3} in counting mode.

OPCs are described in ISO 21501–4 [4]. They only come in a few size classes and can only measure low particle concentrations. They are used for the monitoring and characterization of clean rooms. With different clean room counters, it is possible to detect particle sizes from 0.1 μm, 0.2 μm, 0.3 μm or 0.5 μm up to approx. 20 μm in a concentration range of up to approx. 35 x 10^6 m^{-3}. These particle counters are operated with a high aspiration volume flow rate of up to 50 l/min, so that the measurement times can be kept short for very low concentrations.

Counting measurement methods offer the major advantage that they can determine the particle size distribution and the particle concentration at the same time. The necessary device parameters for the comparison of counting measurement methods, such as the particle size resolution capacity, the time resolution capacity, the classification accuracy, the upper and lower counting efficiency and the zero counting rate are defined in the standards mentioned above.

With all counting measurement methods, care must be taken to ensure that only one particle is available in each case for analysis in the measurement volume under analysis. If two or more particles are in the measurement volume at the same time, this is referred to as coincidence. Coincidence leads to so-called coincidence errors, which are a combination of two different errors: the counting error (the determined count is too low) and the size error (the determined particle size is too large).

In order to ideally avoid coincidence errors, the maximum number concentration approved by the manufacturer ($C_{n\,max}$), e.g. $C_{n\,max} = 10^{12}$ m^{-3} or $C_{n\,max} = 35$ x 10^6 m^{-3}, must be

Table 1. Dilution factor and volumetric flow rates.

K_v	$\dot{V}_A + \dot{V}_R$ [l/min]	$\dot{V}_A$ [l/min]
10	2 + 18	2
100	2 + 198	2
1.000	2 + 1.998	2
10.000	2 + 19.998	2
100.000	2 + 199.998	2

taken into account for every aerosol spectrometer used. Depending on the aerosol concentration C_n to be measured and the limit $C_{n\,max}$, significant dilution is required in some cases.

The challenge for a dilution system is to ensure that the properties of the aerosol are not altered in terms of the size distribution, and that the dilution factor is known across the particle size range (transfer function). In addition, the dilution system must offer long-term stability and must be capable of delivering reproducible results.

1.2 Aerosol dilution for aerosol generators

Test aerosols can be generated following different principles (refer to the series of standards VDI 3491 [5]) from solids, liquids, suspensions or solutions by using condensation methods, combustion processes or chemical reactions. During the generation of test aerosols, particularly depending on the dwell time between the aerosol generation source and the aerosol measurement, dilution needs to be performed at higher concentrations in excess of 10^{13} m^{-5} in order to avoid changes to the aerosol due to coagulation. The term ,coagulation' is used to describe the process of particles colliding and sticking together in the aerosol. In the case of small particles, these collisions are primarily caused by Brownian diffusion motion. As a result of coagulation, the particle size increases and the particle number concentration is reduced – but the mass concentration stays unchanged.

In coagulation processes, the period of time in which the particle number concentration is reduced by half on account of coagulation effects is referred to as the half-life $t_{1/2}$. At ambient conditions with p = 1013 hPa and T = 20°C, the number concentration at 10^{18} m^{-5} will e.g. drop to half its original value within 2 ms.

2 Aerosol dilution systems

On the one hand, one of the tasks of a dilution system can be to make the concentration of an aerosol measurable for a selected measurement device. On the other hand, a dilution system can also ensure that the generated concentration of a test aerosol is reduced far enough to ensure that no further changes due to coagulation can take place.

Standard VDI 3491, Blatt 15 [6] describes the most commonly used dilution principles. Depending on the dilution principle, these dilution systems or dilution stages are suitable for a particle size range from approx. 2 nm to 20 µm depending on the disperse phase of the aerosols (solid or liquid). In this way, a dilution system may work well enough to dilute solid aerosols in a size range up to 20 µm, but by contrast it may be a 100% separator for droplet aerosols up to 7 µm.

Commercially available dilution systems or dilution stages are supplied with a dilution factor ranging from 5 to 300, but usually with a dilution factor of 10 or 100. With the aid of cascadable dilution systems, dilution factors of up to 100,000 can be reliably achieved.

The principles and method of operation of dilution systems should be described unambiguously, and the device parameters should be determined and stated.

2.1 Method of operation and operating principles

The dilution systems that are described here are based on the standard VDI 3491, Blatt 15 [6]. In order to reliably and reproducibly reduce the particle concentration, the aerosol that needs to be diluted is mixed in a defined process with a particle-free gas, e.g. air. The dilution factor is defined as the ratio of the concentrations in the undiluted and diluted aerosol (see equation (1)).

$$K_v = C_{n,u}/C_{n,v} \tag{1}$$

K_v = dilution factor
$C_{n,u}$ = number concentration of the undiluted aerosol
$C_{n,v}$ = number concentration of the

If we assume that the aerosol transport through the dilution system is loss-free and that the clean air or mixed air are free of particles, then the dilution ratio can also be determined from the volumetric flow rates (see equation (2)).

$$K_V = (\dot{V}_A + \dot{V}_R) / \dot{V}_A \tag{2}$$

$\dot{V}_A$ = aspirated volumetric flow rate of the aerosol
$\dot{V}_R$ = volumetric flow rate of the clean air

Equation (2) shows clearly what volumetric flow rates of clean air are required to dilute a volumetric flow rate of just 2 l/min of aerosol in order to achieve dilution factors of 10, 100, 1000, 10,000 or 100,000 (see table 1).

Since, in principle, it will never be possible to achieve loss-free aerosol transport in a mixing chamber, the transfer function of the dilution systems needs to be determined. The aim of a dilution is usually to achieve size-independent dilution of a highly-concentrated aerosol.

Among other things, the dilution systems differ in terms of how the clean air is generated and supplied. The clean air can e.g. be obtained by filtering a partial flow of the aerosol or by using a filtered, separate dilution air supply. In practice, three principles have proven themselves, and these are all described in the standard VDI 3491, Blatt 15 (edition 1996) [6].

2.1.1 External supply of clean air (ejector principle)

The externally generated, particle-free dilution air is supplied to the dilution system via a narrowing of the cross-section (ring gap), where it generates a vacuum. The aerosol that is to be diluted is aspirated by this vacuum. The ejector principle is shown in Figure 1. One particular advantage of these dilution systems is that the individual dilution stages with a factor of 10 or 100 can be cascaded. Thanks to this cascading, dilution factors of 10, 100, 1 000, 10 000 and 100 000 can be achieved easily, reliably and with low consumption of dilution air. For example, by cascading
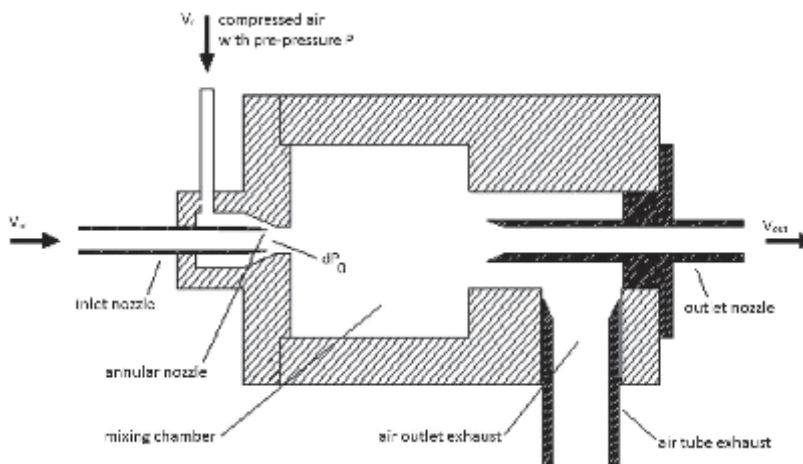
Figure 1. Sectional view of the Palas® dilution stage – ejector principle.

Figure 2. Principle of operation – dilution system with forced mixing.

1 Aerosol inlet
2 Mixing chamber
3 Flow measurement
4 Filter
5 Pressure regulator
6 Compressed air
7 Aerosol outlet

four ejector stages each with a dilution factor of $K_v = 10$, a volumetric flow rate of aerosol of 2 l/min can be diluted by a factor of 10,000 using just 72 l/min of dilution air instead of 19,998 l/min. Thanks to the exchangeable aspiration probes, these dilution stages can be used for particle counters with sampling volumetric flow rates in a range from 0.05 l/min to 50 l/min. Pressure-tight operation up to 10 bar and heated operation up to 200°C is also possible with these dilution stages.

2.1.2 External supply of clean air (forced mixing)

The basic layout principle of a dilution system with forced mixing is shown in Figure 2. At its heart is a mixing chamber or mixing line, which has proved itself to be particularly adept at diluting highly-concentrated aerosols from droplets with a diameter up to approx. 10 µm.

2.1.3 Internal clean air production (capillary tube)

In this principle, the generated particle-free dilution air is collected from a partial flow of the aspirated aerosol with the aid of a filter. The unfiltered part of the flow is directed through a capillary tube (Figure 3). Downstream of the capillary tube, the filtered partial flow is mixed back together with the unfiltered part of the flow. The dilution air is thus the carrier gas for the aerosol. Here, the dilution factor changes with the pressure difference across the filter and as a result of deposits in the capillary tube, which is why the capillary tube should also be monitored with a differential pressure sensor.

2.1.4 New: Internal clean air production (forced mixing)

Since droplet aerosols larger than 4 µm cannot be reliably diluted with the dilution systems that are currently known and commercially available, Palas® GmbH has developed a new dilution system. In this design, the carrier gas of the aerosol is used as the dilution air. The aerosol measured by the welas® digital aerosol spectrometer is filtered, and this clean air is then fed back into the dilution system as dilution air. A small portion of this clean air is disposed of as waste air (see Figure 4). As is shown in Figure 4, here again the heart of this dilution system is the mixing chamber, in which the droplet aerosols with a diameter of up to approx. 10 µm can be reliably and reproducibly diluted. The mixing chamber and the aerosol guide tube have a relatively large diameter, particularly in comparison to the dilution designs
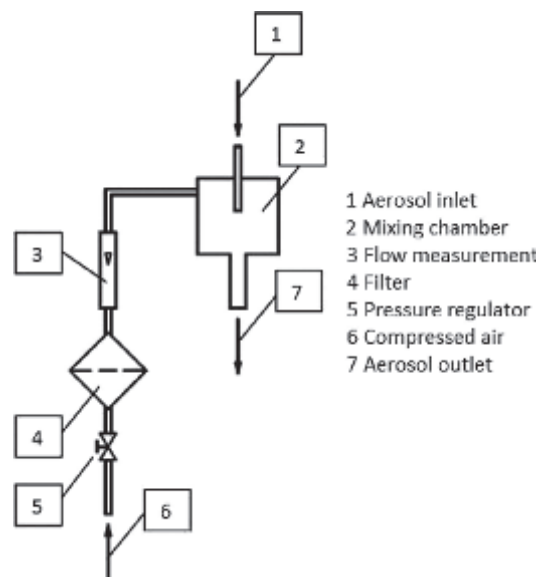

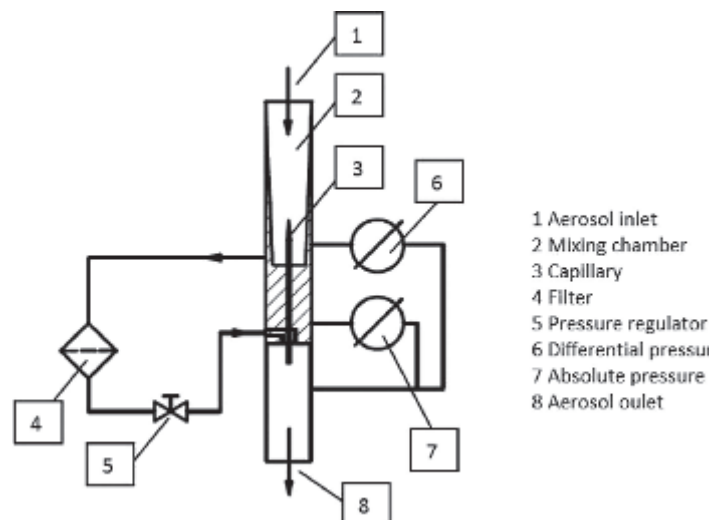**Figure 3. Principle of operation – dilution system with capillary tube.**

1 Aerosol inlet
2 Mixing chamber
3 Capillary
4 Filter
5 Pressure regulator
6 Differential pressure
7 Absolute pressure
8 Aerosol oulet

featuring capillary tubes. A second version of this new dilution principle with a dilution factor of 100 is shown in Figure 5.

2.2 Process characteristics and device parameters for dilution systems

2.2.1 Process characteristics

The most important process characteristics of a dilution system are the aspirated volumetric flow rate of the aerosol, the necessary volumetric flow rate for the dilution air and the method how it is generated – from an additional dilution gas or from the aerosol itself. As a result of the particle size-dependent losses and demixing processes in the mixing chamber and/or in the capillary tubes and depending on the particle material (solids or droplets), the dilution factor is usually only constant within a limited particle size range. The dilution factor should thus be determined as a function of the particle size distribution and the particle material (Figure 6). The dilution factor should preferably be known and constant across the entire particle size range that is to be measured for the aerosol that is to be diluted.
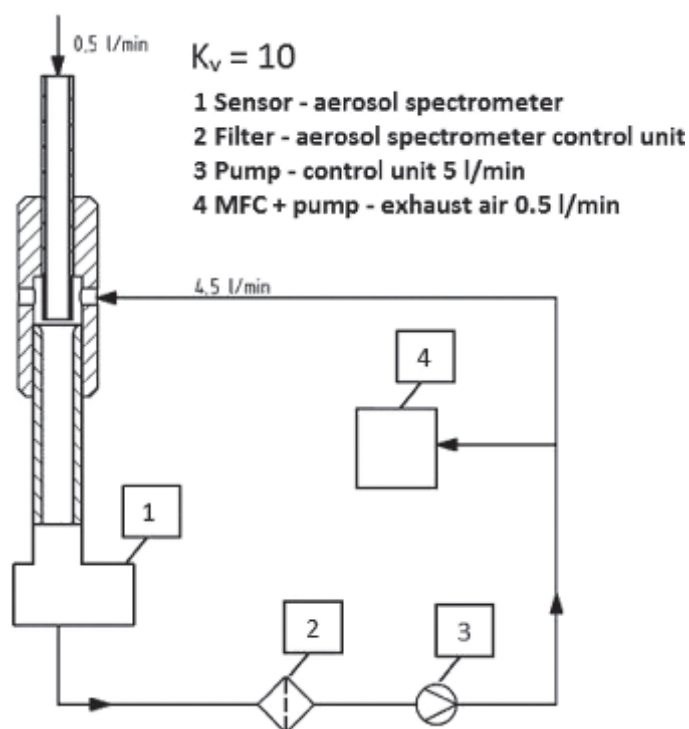
Figure 4. Principle of operation – dilution system (dilution factor = 10) with forced mixing and internal clean air production.
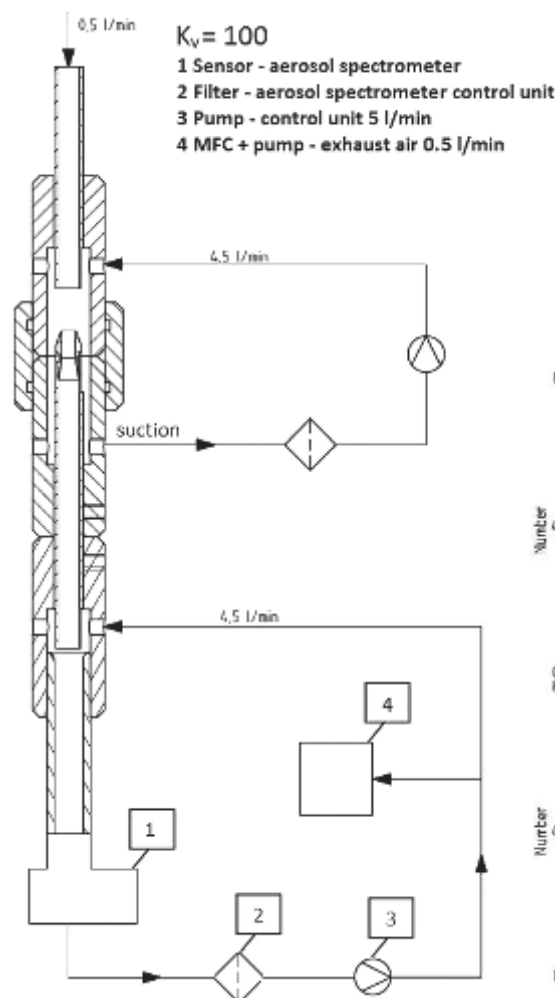

Figure 5. Principle of operation – dilution system (dilution factor = 100) with forced mixing, internal clean air production and cascaded dilution stage.

Equation (1) can therefore be expanded to include the particle diameter:

$$K_v(x) = C_{n,u}(x)/C_{n,v}(x) \qquad (3)$$

K_v = *dilution factor*
$C_{n,u}$ = *number concentration of the undiluted aerosol*
$C_{n,v}$ = *number concentration of the diluted aerosol*
x = *particle diameter*

2.2.2 Device parameters
The following device parameters are required in order to describe a dilution system: dilution factor, particle size range, particle material, total volumetric flow rate, number of cascadable systems, transfer function, temperature and pressure ranges of the aerosol to be diluted.

3 Interfering factors and sources of error

Highly-concentrated solid aerosols with special adhesion properties, such as carnauba wax and soot, tend to block the capillary tube and nozzles within a short space of time. Other solids, such as test dusts made of Arizona Fine Dust (A2 dust) that touch the walls of the mixing chamber usually fall off again. It is relatively easy to dilute this type of solid aerosol. Droplet aerosols up to 10 µm are less straightforward to dilute, since droplets that come into contact with the wall do not come off again. During extended operating times, e.g. when diluting a solid aerosol, deposited particles can become detached again and significantly alter the dilution factor. For this reason, the particle production rate of a dilution system, particularly one based on the application mentioned above, should be determined with an absolute filter positioned at the intake of the dilution system.
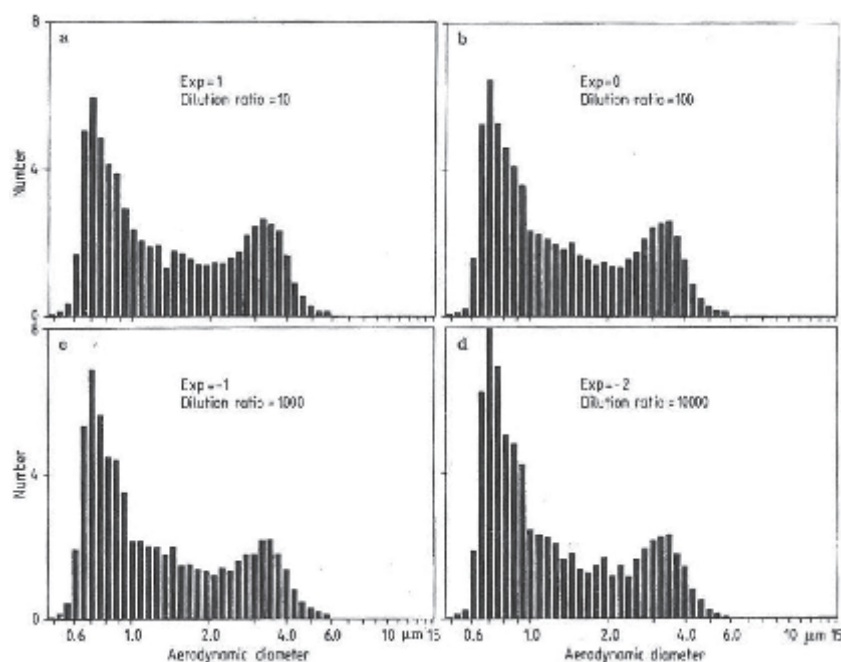

Figure 6. Size distribution of a test aerosol measured with the APS after 10x (a), 100x (b), 1,000x (c) and 10,000x (d) dilution [7]; aerosol: plastic particles.
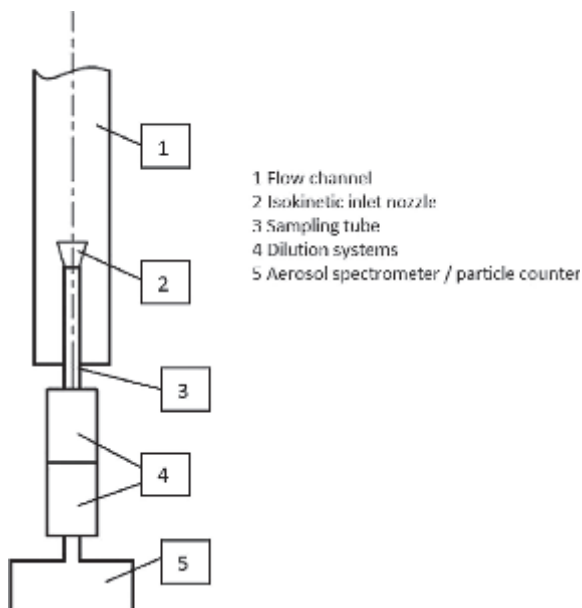
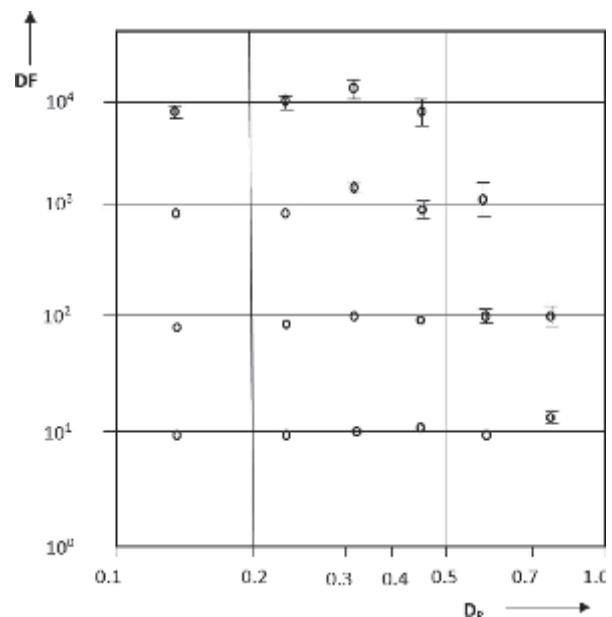Figure 7. Schematic representation of a test setup for testing aerosol measurement devices and dilution systems.

Figure 8. Dilution factors as a function of particle size [8]; measurement devices: µLPC 101 and CPC 3020; DEHS aerosol; aerosol generator: AGF 2.0.

4 Functional testing of dilution systems

4.1 Functional testing with solid aerosols and droplet aerosols up to 2 µm

In a functional test with the test setup (Figure 7), several of the device parameters, such as the transfer function during cascading in terms of the overall dilution factor (Figure 6 and Figure 8), can be tested; see also [9] and [10].

4.2 Functional testing with clean air on-site

With the simple test setup (Figure 9), the function of the dilution systems with OPCs can also be reliably tested with the room air in the laboratory. It should be taken into account here that, when using an OPC in accordance with ISO 21501–4 [4], the clean air must always be diluted, as these particle counters can already encounter coincidence errors during measurements with normal laboratory air. As the first step, the particle concentration (Table 2: Measurement 1) is determined with the test setup (Figure 9, Measurement 1) free of coincidence errors using a functioning dilution system. Afterwards, the dilution stage under testing is connected between the pre-dilution and the OPC (Figure 9, Measurement 2) and measured again. In this way, the dilution factor can be reliably determined. The number across the size classes is determined. However, the dilution factor is determined from the total number of all particles, as the room air can still change slightly during the measurement period, see Table 2. If it is not possible to verify the expected dilution factor, then either the Measurement 1 is still affected by coincidence errors, or the dilution system is defective.

Table 2. Numerical values for the functional test (Figure 9).

Particle classes in µm	Number
0.2	151648
0.3	71604
0.5	4305
0.7	360
1	82
2	16
3	1
5	0
Total	228016

Measurement 1

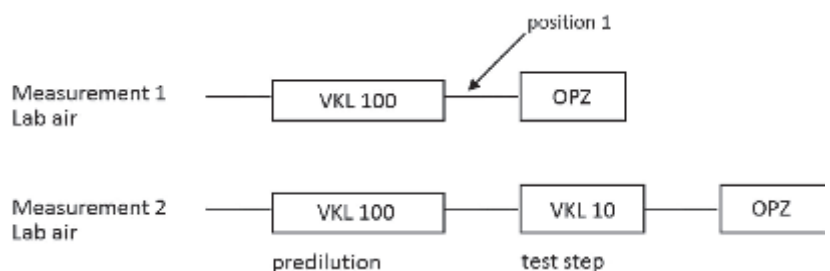| Particle classes in µm	Number
0.2	15166
0.3	7290
0.5	524
0.7	65
1	21
2	3
3	0
5	2
Total	23071

Measurement 2

Figure 9. Test setup with the ejector dilution stages VKL 10 / VKL 100 for functional testing of a dilution system with clean air.

Table 3. Verification of the dilution system with mixed air, dilution factor 10, monodisperse droplets 5 µm and 7 µm.

Particle diameter	Dilution factor K_v	Number / raw gas	Number / clean gas
5 µm	9.91	64475	6505
7 µm	10.59	32443	3063

Table 4. Verification of the dilution system with mixed air, dilution factor 100, monodisperse droplets 5 µm and 7 µm.

Particle diameter	Dilution factor K_v	Number / raw gas	Number / clean gas
5 µm	100.01	304322	3043
7 µm	99.87	236687	2370

Number density distribution dN/N/logd(dx)

Number density distribution dN/N/logd(dx)

Figure 10. Dilution factor 10 – particle size distributions of 5 µm (left) and 7 µm (right).

Number density distribution dN/N/logd(dx)

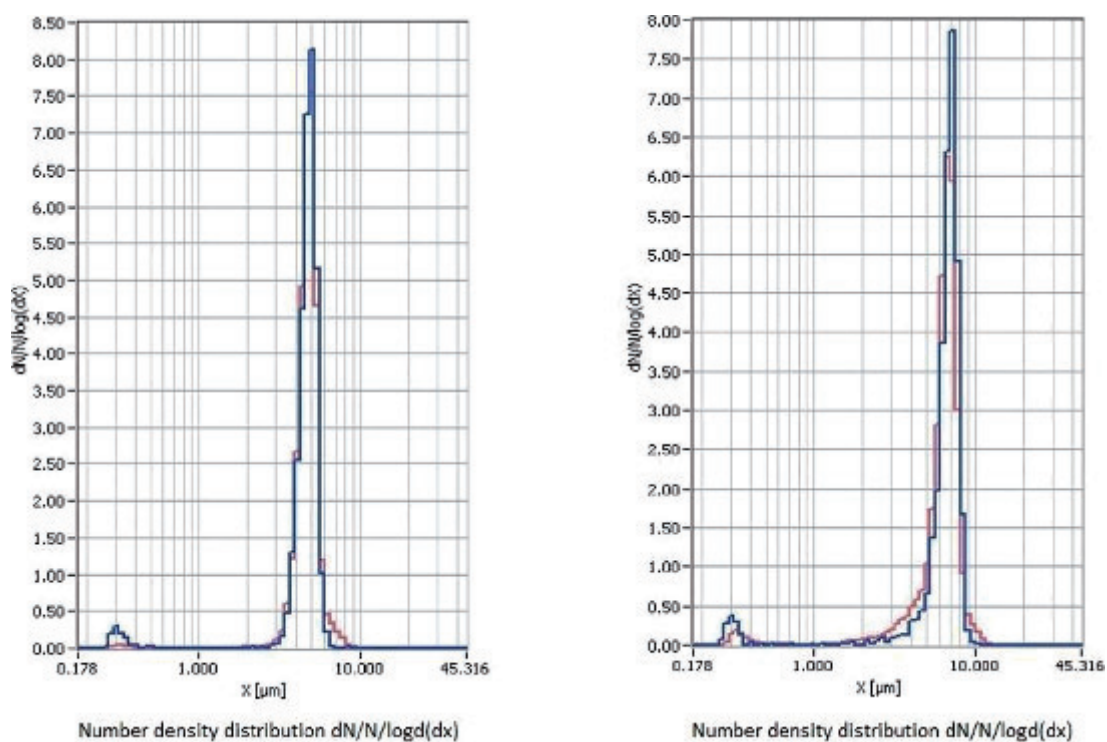Number density distribution dN/N/logd(dx)

Figure 11. Dilution factor 100 – particle size distributions of 5 µm (left) and 7 µm (right).

4.3 Functional testing with droplet aerosols up to 7 µm

These transfer functions or dilution functions must also be verified during the dilution of droplet aerosols. To do this, the test setup (Figure 7) and an aerosol generator in accordance with VDI 3491, Blatt 4 [11] for generating monodisperse aerosols (e.g. the MAG 3000 unit from Palas®) are used. The measurement results for the proof of functionality for the new dilution systems for the dilution factors 10 and 100 with the dilution principle of forced mixing are shown in Table 3 and Table 4. The welas® digital system was used as the aerosol spectrometer. In order to determine the dilution factor 10, the welas® sensor 2300 was used with a measurement time of 120 s and a volumetric flow rate for the mixed air of 50 m³/h. The measurement time for determining the dilution factor 100 was 180 s, the volumetric flow rate for the mixed air in the channel was set to 28 m³/h, and the welas® sensor 2500 was used. On both welas® sensors the measurement range was set to 0.3 µm – 17 µm. Table 3 shows the numerical values for the measured particles in the raw and clean gas for 5 µm and 7 µm with the dilution factor 10, and in Table 4 the measured particles in the raw and clean gas are shown for 5 µm and 7 µm with the dilution factor 100. Figure 10 and Figure 11 show the particle size distributions that belong to the tables.

4.4 Summary of the functional tests

It was shown that dilution systems can be characterized relatively simply, clearly and reliably. This includes the statement of the dilution factor K_v for different aerosol materials, particle sizes, temperatures and pressures. Only with clearly and unambiguously characterized dilution systems is it possible to achieve reliable and reproducible measurement results.

References

[1] VDI 3491 sheet 1 Particulate Matter Measurement – Generation of Test Aerosols – Fundamentals and Overview.

[2] VDI 3867 sheet 1 to sheet 6 Messen von Partikeln in der Außenluft – Bestimmung der Partikelanzahlkonzentration und Anzahlgrößenverteilung von Aerosolen – Grundlagen. Berlin: Beuth 2009.

[3] ISO 21501-1 Determination of particle size distribution – Single particle light interaction methods – Part 1: Light scattering aerosol spectrometer. Berlin: Beuth 2009.

[4] ISO 21501-4 Determination of particle size distribution – Single particle light interaction methods – Part 4: Light scattering airborne particle counter for clean spaces. Berlin: Beuth 2007.

[5] VDI 3491 (all sheets) Messen von Partikeln – Herstellungsverfahren für Prüfaerosole. Berlin: Beuth 2017.

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[11] VDI 3491 sheet 4 Messen von Partikeln – Herstellungsverfahren für Prüfaerosole – Kondensationsverfahren. Berlin: Beuth 2016.

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