



# Methods for Measuring Emissions of Particulate Matter from Solid Biomass Combustion



**Biomass  
energy use**





## **Biomass energy use**

Series of funding programme “Biomass energy use”  
**VOLUME 8**

# **Methods for Measuring Emissions of Particulate Matter from Solid Biomass Combustion**

**Published by**  
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March 2018

Funded by



Federal Ministry  
for Economic Affairs  
and Energy

Project management



Programme support



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**Layout & production**

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**Funding**

This publication was funded by the the Federal Ministry for Economy and Energy, Berlin (BMWi)

ISSN (online): 2364-897X

ISBN 978-3-946629-28-3

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## Table of abbreviations

| <b>Abbreviation</b>  | <b>Explanation</b>   |
|----------------------|--|
| <b>act.</b>          | Actual   |
| <b>CPC</b>           | Condensing Particle Counter  |
| <b>DMC</b>           | Differential Mobility Classifier   |
| <b>ELPI™</b>         | Electrical Low Pressure Impactor –<br>online measurement device for particle distributions |
| <b>FID</b>           | Flame-ionisation Detector  |
| <b>FMPS™</b>         | Fast Mobility Particle Sizer   |
| <b>FTIR</b>          | Fourier Transformation Infra-red Spectrometer  |
| <b>HEPA</b>          | High Efficiency Particulate Airfilter  |
| <b>H<sub>l</sub></b> | Lower heating value  |
| <b>IR</b>            | Infra-red range of the electro-magnetic spectrum of radiation                              |
| <b>min</b>           | Minimum  |
| <b>MW</b>            | Microwave radiation  |
| <b>NIR</b>           | Near infra-red   |
| <b>O<sub>2</sub></b> | Oxygen   |
| <b>OPC</b>           | Optical particle counter   |
| <b>PME</b>           | Particulate Matter Emission  |
| <b>sat</b>           | Saturated  |
| <b>SMPS</b>          | Scanning Mobility Particle Sizer   |
| <b>SaS</b>           | Service and support project of the funding programme                                       |
| <b>STP</b>           | Standard condition (273.15 K / 1.01325 bar)  |
| <b>TOC</b>           | Total organically-bound carbon   |
| <b>tot</b>           | Total  |
| <b>TVOC</b>          | Total volatile organically-bound carbon  |
| <b>UV</b>            | Ultraviolet range of the electro-magnetic spectrum of radiation                            |
| <b>VIS</b>           | Visible range of the electro-magnetic spectrum of radiation                                |
| <b>VOC</b>           | Volatile organic compounds   |
| <b>∅</b>             | Diameter   |



# 1 Preface

Long before the first debates on how to convert the modern energy sector towards more renewable energies, wood was a widespread means of heat generation. At present, almost three quarters of all heat provided by renewable energy technologies is produced from solid biofuels (Musiol et al., 2012). Currently the dominant technology for renewable heat production from solid biofuels are small-scale wood furnaces. The combustion of wood and other biogenic solid fuels contribute to a very large extent to the renewable energy consumed in the domestic sector. The climate protection objectives pursued by the German government would certainly not be achievable without the combustion of wood in such appliances.

Current research, however, shows that the combustion of wood in small-scale furnaces causes significant emissions, most notably emissions of particulate matter, and is a considerable source of air pollution (Birmili et al., 2008; Hausmann, 2010; Struschka et al., 2008). Particulate matter can have negative impacts as it is suspended in air due to its chemical/physical properties and can reach considerable concentrations. The exposure to components of particulate matter may increase the risk for respiratory tract diseases (Dockery et al., 1993).

Therefore, one of the objectives of the funding programme “Promoting projects to optimise biomass energy use” (short: “Biomass energy use”) is to solve the conflict between climate protection and/or greenhouse gas reduction on one hand and air quality management through the reduction of harmful emissions on the other. With recognition of the conflict between air quality management and a continued growth of the market for thermochemical biomass conversion technologies first approaches for emission reduction could be successfully implemented (Lenz et al., 2010). Within the funding programme, innovative approaches for the reduction of emissions are currently being developed and given special attention.

Common to all projects in this field is the need for emissions to be measured. In addition to the gaseous emissions, this applies in particular to emissions of particulate matter. In practice there are a series of different framework conditions for measurements of particulate matter which will have an influence on the result of the measurement and that may limit the comparability of the

results (type tests, laboratory measurements, in-situ measurements, chimney sweeper measurements, etc.). The measurement devices used and the measurement procedures utilised do, in part, considerably differ from one another. The ongoing debate shows that it is not an easy task to measure dust particles correctly and assess their potential impact on the human body and the environment (Mudgal & Turbé, 2009; Orthen et al., 2007; Rödelsperger et al., 2009; UBA, 2008; Wiedensohler et al., 2012).

With the amendment of Germany's First Federal Immission Control Ordinance in 2010 (1. BImSchV), the emissions requirements posed to small-scale wood furnaces became stricter. The investigation and implementation of newer and more precise measuring methods has continuously gained importance since then. Especially for the development of combustion technologies with extraordinarily low dust emissions the use of high resolution measuring techniques will become a priority. The well established but time-consuming gravimetric determination of dust concentrations may not always be sufficient for that purpose. The determination of dust concentrations over time, e.g. through counting of individual particles, has therefore gained attention. Different providers offer devices which seem to generate reliable values, but when compared with one another they actually have considerable deviations. In addition, insights gained in recent years show that especially aerosols from an incomplete combustion of solid biomass may change drastically, depending on the environmental conditions. Besides these rather academic/scientific challenges, the day-to-day dust measurements done in laboratories and in-situ feature their own challenges making a comparison between measured values from different projects difficult.

Therefore, it appears to be essential, especially as a support for the funding programme with its individual projects all aiming at emission reduction, to determine dust emissions under comparable, reproducible and generally accepted methods – also in the context of increasing European efforts to define standardised measuring methods.

### **The authors**

Leipzig, in march 2018



## 2 Introduction

### 2.1 Dust emissions during biomass combustion - Background

The emission of dust during combustion processes is being criticised as a potential health hazard. Airborne dust particles may enter the lungs causing diseases of the respiratory tract and/or of the respiratory organs and furthermore also diseases of the whole cardiovascular system (Dockery et al., 1993). Therefore it is advisable to limit such emissions. In the automotive sector, in large scale power plants and all other plants/systems which require an operation licence the risks originating from dust emissions have for decades been addressed by means of e.g. highly developed downstream filter systems and catalysts. More recently the public started perceiving the combustion of biomass as another source of potentially hazardous emissions. Especially the increasing share of biomass combustion in small-scale furnace for domestic heat production is a driver of local particulate matter emissions. However, through a targeted advancement of such firing systems towards a more controlled combustion behaviour and retrofitting dust emissions can be reduced to a minimum. Hence, it is necessary to develop and apply measuring techniques that are specifically tailored to small-scale combustion systems and/or their exhaust gas characteristics. Quite often, measuring techniques are used which have not been adjusted for measurements at biomass firing systems. This may lead to improper use and subsequently to imprecise or even incorrect results. It is imperative to minimise the errors from e.g. incorrect handling of non-adjusted measuring techniques by making suggestions on proper use and by outlining under which circumstances uncertainties can be expected. Thereby the numerous measurement techniques available today will become better interpretable and more comparable to one another.

### 2.2 Dust measurement - Areas of application

The measurement of dust emissions from small scale furnaces can be performed with very different objectives. Often measurements are conducted to assess whether legally binding minimum requirements on emissions are met when operating the furnace. Depending on the type of system, they may be performed e.g. on-site by the chimney sweeper as part of the mandatory inspection activities or in form of so-called suitability tests for series-produced products. Also, dust emission measurements are already performed during the development of new products at the test laboratories of the manufacturers or at research facilities. Furthermore, dust emissions can be determined for a purely scientific purpose, e.g. investigating effective emission reduction measures, the formation of dust and its components as well as chemical, physical or even toxicological characteristics. All these different backgrounds are currently addressed with different measuring techniques which have to be adjusted and optimized depending on the task at hand.

## 2.3 Programme participants

The diversity of the outlined application scenarios for dust measurements on biomass furnaces highlights the need for a harmonisation of the measuring methods. This will initially require a consolidation of all methods currently used. To be able to both describe the different approaches as well as discuss them in a larger context, recurring meetings and measuring workshops have been and are being organised with the affected programme participants as part of the funding programme "Biomass energy use" since 2010. To date, scientists of the following institutions have been involved in these meetings:

- Brandenburg University of Technology Cottbus (BTU),
- DBFZ Deutsches Biomasseforschungszentrum gemeinnützige GmbH (DBFZ),
- Fraunhofer Institute for Factory Operation and Automation (IFF),
- Fraunhofer Institute for Environmental, Safety, and Energy Technology UMSICHT (Fraunhofer UMSICHT),
- Helmholtz Zentrum Munich,
- Helmholtz Centre for Environmental Research (UFZ),
- Hamburg-Harburg University of Technology (TUHH),
- Technology and Support Centre within the Centre of Excellence for Renewable Resources (TFZ),
- University of Göttingen,
- University of Leipzig,
- Bavarian Centre for Applied Energy Research (ZAE Bayern).

These meetings were of great value for compiling the currently existing knowledge on the subject, promoting the dissemination within the scientific community and making it available to the users. As part of these meetings numerous manufacturers of measuring equipment were contacted and informed about the ongoing work.



## 2.4 Compiling dust measurement methods - Approach

The objectives of the “Biomass energy use”-projects, whether they are completed or still ongoing, are very diverse while the measuring of dust and emissions is common to most of them. As the background of the scientists and institutions involved is very multi-disciplinary the compilation and harmonisation of the different methods into a handbook of measuring methods is at times a tedious task. Therefore, in a series of workshops the different measurement options were screened and a comparison of the measured values was conducted. Communication between the partners was maintained to help compare organisational, material and staffing expenditures of each of the respective (dust) measuring methods. Individual measuring results were, in part, presented at international events and discussed with experts (Pfeiffer & Schröder, 2011; Schröder, 2011; Schröder et al., 2011; Schröder, 2012). During the conferences hosted by the funding programme the status of work was discussed and future targets were set. In addition, several conference calls were organised to include insights from institutions not participating in the funding programme. Thus it was possible to ultimately collect a wide range of information which was consolidated into the compilation of measuring methods at hand. The authors hope that the knowledge collected here will serve as a good introduction to the topic of "Dust Measurements on Biomass Furnaces".



## 3 Fundamentals

The fundamentals of solid biomass combustion processes and dust generation mechanisms occurring therein are well explained in standard literature and will not be explained here any further. However, there are some issues which were considered essential for a harmonisation of methods and needed to be addressed here. Most importantly, a standardised terminology had to be defined. Furthermore, a short overview of the options for dust characterisation is given, followed by a brief discussion of the essential variables influencing the recording of dust. Since these essential variables also need to be recorded during the measurements, the corresponding measuring methods are introduced and described with respect to their inherent range of error. At the end there is a standardisation and referencing of the measurement results, e.g. with respect to a certain amount of oxygen in the exhaust gas and/or a defined standard volume and/or a defined amount of fuel and its respective energy content.

### 3.1 Definitions

At the first workshop on measuring techniques by the sub-working group “Particulate matter” the work on a standardised terminology was initiated. The terminology was chosen so that a consistent use of terms for the duration of the funding programme and between the projects could be ensured. The overview is subject to continuous updates and amendments by all programme participants, under the direction of the DBFZ.

**Aerosol** is a mixture (suspension / dispersion) of liquid or solid particles in a gas. Examples of aerosols are fog, smoke and clouds. Rain and hail, however, are no aerosols.

**Reference oxygen content.** To account for a reduction in the concentration of certain exhaust gas components (in general, exhaust gas components subject to statutory limits) caused by the addition of air, the exhaust gas components are converted to a reference oxygen content. With a given fuel, the conversion to a reference oxygen content furthermore also corresponds to a conversion to fuel mass and/or fuel heating value.

**Single-room furnaces (SRF)** are furnaces that primarily serve as a source of heat in a single-room set up. This includes, for example, but also stoves (with or without indirectly heated baking facility), open fireplaces, and others (Lenz et al., 2010). In Germany, SRF make up for more than 90 % of all small-scale furnaces installed for the combustion of solid biomass. In Germany's First Pollution Control Regulation (1. BImSchV) as well as in the Ecodesign Directive (LOT 15) SRF are treated differently from furnaces that are equipped with a heating circuit.

**Emissions** are airborne pollutants originating from a furnace during the combustion of biomass [Federal Ministry for the Environment, First Regulation for the Implementation of the Federal Immission Control Act (Regulation for small-scale and medium-scale furnaces – First Pollution Control Regulation, January 2010)].

**Particulate matter** is a fraction of the suspended dust with an aerodynamic diameter of 2,5 µm or less. It is defined here as the totality of all solid or liquid components transported with the exhaust gas as a result of the combustion process. Both the total mass obtained by e.g. filtering as well as the quantity of calibrated aerosol equivalents may be referred to as particulate matter (EPA, 2014a), (DIN EN 481, 1993).

**Fine dust particle** (also, just "particle") is an object with a defined boundary surface and a very small mass in a condensed, i.e. solid or liquid, form. Particles often differ in their chemical and physical characteristics (composition, geometric dimensions).

**Calibration** is when a value displayed by the measuring device is checked against a corresponding value of a reference material (Hässelbarth et al. 2004).

**Lowest heating power** is specified by the manufacturer in accordance with the requirements of EN 303-5, at which the furnace can still be operated with a given fuel (DIN EN 303-5, 1999). In accordance with 4.4.6 of EN 303-5:2012, it should be lower than 30 % of the nominal heating power so that an adjustment of the furnace to less than one third of the nominal capacity is possible.

**Measuring uncertainty** is a value describing the variance in the measured results and is attributed to the respective parameter (Hässelbarth et al. 2004).

**Nominal heating power** is the heating power of the furnace, stated by the manufacturer, that is achieved upon combustion of the specified test fuel under test conditions (DIN EN 13240, 2010), (DIN EN 303-5, 1999).

**Sampling** refers to the targeted extraction of exhaust gas particle streams and/or (less often) exhaust gas components for their subsequent assessment with measuring equipment. It should be further described by information on the process and conditions.

**Sampling process** refers to the sequence of different steps taken to obtaining a defined amount of material necessary for the inspection with the respective measuring equipment.

**Partial load** is the ratio of heating power to nominal heating power, expressed in percent (DIN EN 303-5, 1999).

**Dilution** refers to a mixing of the exhaust gases with another gas and/or blend of gases (in most cases filtered air) to help determine the content of particulate matter in exhaust gases of biomass furnaces.

**Full load** corresponds to the state of operation of an automatic furnace with a heating power of 100 %. It, furthermore, corresponds to the state of operation with the highest achievable heating power of a manual furnace while adhering to the manufacturer's specifications. Under test conditions and in case of utilisation of the test fuel, the heating power produced should correspond to the nominal heating power (DIN EN 13240, 2010).

## 3.2 Options for the characterisation of particulate matter

The methodology used for characterising particulate matter emitted by small-scale furnaces depends to a large extent on the objective at hand. A chimney sweeper, for example, uses a very robust method for the regular mandatory dust measurements in order to check whether the required mass concentrations are met, producing a single required value from the less interesting raw data. Regarding acceptance tests, measuring methods described in standards and statutory requirements are used to check whether given thresholds are being met.

In research, on the other hand, significantly more elaborate methods are being used in respect to the scientific objectives. For instance, the investigation of particulate matter formation mechanisms or the impact of its emissions on the immission situation and/or on human health may be of interest. The measuring equipment used in these cases will then differ significantly from the methods used outside the laboratory and for acceptance tests. Thus, the degree of complexity can increase quickly and an in-depth understanding of the methods and their limitations is required. Many of the methods commonly used in research and development were specifically adjusted to the respective task. Often the methods are defined by the principles of chemistry and physics, while some of the principles may still be unknown or subject of current research. If particulate matter is fractioned into individual particles, it could be described with respect to its physical parameters and its chemical composition. However, during combustion typical quantities of approx. 100 million particles of particulate matter per cubic centimetre would make this selective description very costly. Thus, one possible attempt is to describe solid or liquid particles in their totality as aerosol (assuming that it is equally distributed in the gas). This can be achieved by various methods and a variety of indicators such as mass, particle size distribution, density distribution, chemical and physical characteristics, shape, colour, olfactory indicators, hygroscopic and volatility to describe the totality of the aerosol and/or dust. Furthermore, in addition to these purely descriptive parameters, in recent years research was started on evaluative indicators that describe the toxicological relevance of the aerosols as a whole or of their respective components.

The diversity of available characterisation options illustrates that many of the measuring methods are limited. Through the insights gained from in-depth description of aerosols, for example, it became apparent that the quantity of dust is not necessarily the most representative value, given that it can considerably differ depending on the composition and environmental conditions. The search for a more comprehensive description of aerosols is gaining more and more importance not only in the scientific realm but also in future regulatory classifications.

## 3.3 Variables influencing dust measurements

The current practice of setting dust threshold values based on mass, and in newer regulations also in respect to certain particle sizes, assumes a mass-related impact of the particles and droplets. However, current research shows that aerosols, in particular from the combustion of biomass solid fuels, undergo a considerable change over time, starting upon

exiting the furnace and during the entire time they are airborne. Exterior conditions considerably contribute to this change. Most important variable is temperature, which can rise to far above 800 °C in the combustion zone and may still be at 100 to 300 °C at the exit of the furnace. Furthermore, the prevailing partial pressures and the chemical constellations (exhaust gas composition) play a considerable role.

Similar to the *principle of mobile equilibrium* (LE CHATELIER) in chemical mixtures, the change that aerosols undergo over time will reach an equilibrium which is, to a large extent, dependent on exterior conditions. These, however, can be changed considerably through e.g. unintentional or intentional dilution.

In addition to variables mentioned above, the concentration of particles, their sizes and their electric charge are of great importance. Through the impact of other gaseous components as well as UV radiation further ageing processes of the aerosols may occur so that particles may change or new particles may be created.

Based on the multitude of influencing parameter, it becomes clear that a comparable statement regarding dust emissions is only possible if a minimum amount of those parameters is recorded via respective measuring techniques. The most common measuring methods for such parameters are presented below. In later chapters the main parameters that should be documented in existing methods and which are relevant to a harmonisation of methods will be covered in detail.

## 4 Measuring technology fundamentals

The determination of dust concentrations in the exhaust gas of biomass small-scale furnaces is meaningful only if they can be compared to other values. Therefore, it is not only required to measure the dust itself but also additional physical parameters such as the temperature and pressure as well as other gaseous exhaust gas components. Since many measuring instruments determine concentrations in cold and dry exhaust gas while others determine concentrations in hot exhaust gas, a complete characterisation of the exhaust gas composition may be necessary in order to specify absolute concentrations. Often, this is done via combustion calculation known from standard works (Kaltschmitt et al., 2016). In practice particular attention needs to be paid to the water vapor which is present in the exhaust gas from biomass combustion. If stable conditions can not be provided in the exhaust gas or the measuring line, a dropping of the gas temperature below the dew point may change the gas composition due to condensation while re-vaporisation of condensed water may occur when the temperature suddenly rise above the dew point again. In this chapter the relevant parameters which should be recorded during all dust measuring procedures are explained more closely.

### 4.1 Temperature measurement

As in virtually all technical processes and therefore in case of dust measurement from biomass furnaces too, the **temperature** is an important parameter to be determined. The physical parameter temperature is specified with the symbol  $T$  in case of Kelvin as unit and  $\vartheta$  and/or  $t$  when  $^{\circ}\text{C}$  is chosen from the International System of Units (SI). In accordance with the different measuring tasks presented in Figure 1, the temperature is determined for different reasons and in some cases with different methodologies depending on the objective. Also, the required measuring uncertainty and the process conditions may determine which equipment is used for recording the temperature. In addition, criteria such as durability, long-term stability, tendency to drift, but also the price may decide how the measurement is taken. The options for temperature measurements are described comprehensively in the technical literature (Bauer et al., 2007). Yet, in this section we will introduce a few examples to show the particularities of temperature measurements in biomass furnaces during dust measurements. To determine the dust concentration the temperature is measured at the following locations:

- A. near the dust probe, to determine the exhaust gas temperature,
- B. in direct proximity to the furnace, to determine the ambient temperature protected from radiation,
- C. in the partial volumetric flow at the gas meter.

The temperature measurement in the exhaust gas near the dust probe is necessary to put any parameter measured during operation in relation to standard conditions. The measurement of the ambient temperature is necessary for various reasons. Both the metering installation but also the furnace may cool down to a different extent in case of consider-

ably different ambient temperatures. In case of devices dependent on ambient air, this has an impact on the combustion and on the exhaust gas temperatures, and in case of devices independent of ambient air, it also has an impact on the radiation behaviour of the furnace. In general, temperature changes should be avoided. To document the ambient temperature during a measurement, the standard for the acceptance test of ovens (DIN EN 13240, 2010), for instance, requires that the room temperature is recorded at least once per minute, with an accuracy of at least 1.5 K. Additionally, it specifies where the sensor is to be located and how a radiation shielding should look like. Similarly, the temperature of the partial volumetric flow has to be determined.

Generally, temperature measurements are performed with contact thermometers. Resistance thermometers or thermocouples are used depending on the required accuracy, the temperature and the price. These two types of thermometers differ from one another in terms of their physical operating principle.

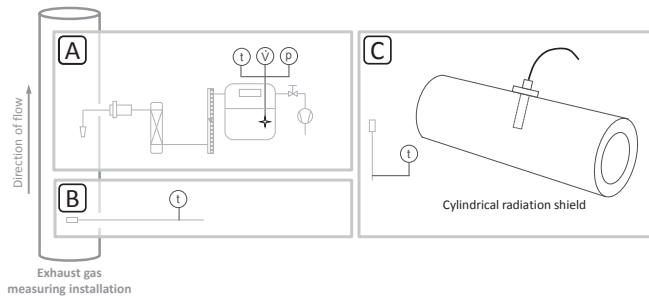


Figure 1: Sample depiction of the different temperature measurement sites. A) Temperature of the partial volume flow, B) temperature of the exhaust gas, C) ambient temperature with radiation shield in accordance with EN 13240

Resistance thermometers are constructed from metallic conductors whose electric conductivity and/or their resistance changes depending on the temperature to which the conductor is exposed. Thermometers made of nickel or platinum are common. The accuracies and areas of use are defined in the standards. To minimise the error caused by the impact of measuring line and electrical connections and to increase the precision of the measurement, resistance thermometers can be operated differently. Quite often 2, 3 and 4 conductor circuits are used while the type of coupling also depends on the transducer. Depending on the material, resistance thermometers can be used up to 850 °C. For higher temperatures thermocouples are better suited.

Thermocouples are also constructed from electric conductors. The measuring principle does, however, differ from resistance thermometers, relying on a change in voltage caused by a temperature difference. The thermocouple consists of two electric conductors made of different materials that are connected to one another at the point of measurement. A voltage difference occurs between the separated ends (opposite from the point of measurement) caused by the different material characteristics of the electric conductors. If the temperature at the point of measurement changes, the electric voltage between the open

ends will change too. Based on characteristic curves, the temperature can be read from the voltage difference. There are thermocouples made of different pairings of material such as NiCr-Ni or Fe-CuNi.

## 4.2 Pressure measurement

The measurement of dust contents in exhaust gases additionally requires the determination of pressure. In particular for the determination of flow rates and pressure differentials as well as for the conversion to standard condition, the pressure is another important physical parameter. According to the International System of Units SI, physical **pressure** ( $p$ ) is specified and measured in Pascal (Pa). It describes the specific force ( $F$ ) that bears down on an area ( $A$ ). Hence, one Pa is defined as the pressure of one Newton bearing down on an area of 1 square metre.

$$p = \frac{F}{A} \quad (1)$$

The following relationships between the units apply.

$$1 \text{ Pa} = 1 \frac{\text{N}}{\text{m}^2} = 1 \frac{\text{kg} \cdot \text{m}}{\text{s}^2 \cdot \text{m}^2} = 1 \frac{\text{kg}}{\text{m} \cdot \text{s}^2}$$

| <i>Symbol</i> | <b>Definition</b> | <b>Unit</b>    |
|---------------|-------------------|----------------|
| $p$           | Pressure          | Pa             |
| $F$           | Specific force    | N              |
| $A$           | Area              | m <sup>2</sup> |

In addition to the SI unit Pascal (Pa) other units are frequently used, such as (bar), (Torr), (at) or (psi). These units are occasionally listed on older measuring devices and regulations or may still be standard in other systems. The appropriate conversion of two of the more frequently used parameters into Pa is listed in the following Table 1. In the context of dust measurement, the units bar, millibar (mbar) and hectopascal (hPa) are often used. In particular, the specification of the atmospheric pressure is quite often stated in mbar.

Table 1: Conversion between common pressure units

|        |   |          |      |
|--------|---|----------|------|
| 1 Pa   | = | 0.00001  | bar  |
|        | = | 0.0075   | Torr |
| 100 Pa | = | 1        | mbar |
|        | = | 1        | hPa  |
| 1 bar  | = | 100,000  | Pa   |
|        | = | 750.0638 | Torr |
| 1 Torr | = | 133.3    | Pa   |
|        | = | 0.001333 | bar  |



During dust measurements in flowing gases, such as the exhaust gas of biomass furnaces, typically very small pressure differentials are measured. For example, for the operation of log wood burning stoves as required by acceptance tests, a pressure reduction by 12 Pa relative to the atmospheric ambient pressure is required. This is also referred to as drag and/or discharge pressure and must be determined with an accuracy of 2 Pa. This low pressure (drag) occurs in the oven due to the temperature difference between the exhaust gas temperature and the considerably lower ambient temperature, creating an upward flow in the exhaust gas pipe. This upward flow moves the hot exhaust gas through the chimney into the atmosphere and lets fresh room air flow into the furnace. Figure 2 exemplifies the pressure ratios typically occurring in a log wood oven and the respective calculation of discharge pressure from the static absolute pressure in the exhaust gas and the static ambient pressure.

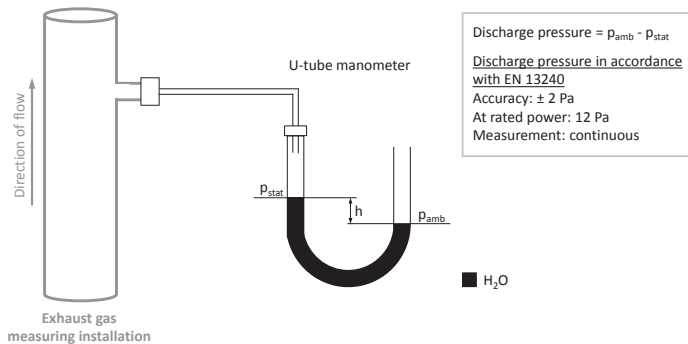


Figure 2: Simplified depiction of the static pressure conditions in the exhaust gas pipe of a small-scale furnace, at low pressure

Typically the pressure measurement is performed with an instrument capable of logging data over time so that after completion of the measurements a chronological sequence of the pressure differentials can be obtained. This data sequence can be helpful to identify unforeseen events during a longer measurement (e.g. on a test stand or an in-situ operation) and interpret the results accordingly. An unforeseen drop or increase in pressure can be caused e.g. by quickly changing environmental conditions (opening or closing the door of a room, or high wind speeds at the chimney outlet) or by events in the furnace itself (collapsing fuel, ember burn-up, etc.). In order to meet the required accuracy (required measuring uncertainty in accordance with DIN EN 13240 (2010)  $\leq 2$  Pa) not only the performance of the measuring device is crucial but the entire experimental set-up needs to operate at the required accuracies. For a conversion of emission data to standard conditions, the low pressure is furthermore recorded at the gas meter.

### 4.3 Standard conditions

For the comparison of measurements it is necessary to relate the results to standard conditions (STP). Standard conditions are characterised by a temperature of 273.15 K and a pressure of 101,325 Pa. A simplified expression of the relation between the two parameters in regards to an enclosed volume of gas (applicable to ideal gases) is used here.

$$p \cdot V = n \cdot R \cdot T \quad (2)$$

| Symbols | Definition                  | Unit of measure |
|---------|-----------------------------|-----------------|
| $p$     | Static pressure of the gas  | Pa              |
| $V$     | Volume of gas               | $\text{m}^3$    |
| $n$     | Substance quantity          | mol             |
| $R$     | Universal gas constant      | 8,314 J/(K·mol) |
| $T$     | Gas temperature (in Kelvin) | K               |

This equation highlights the linear dependency of the following parameters: pressure, temperature, volume, and substance quantity. If one of the parameters changes it will directly impact the others. Thus, the required measuring uncertainty of an exhaust gas component will determine the accuracy with which the temperature has to be measured.

An appropriate conversion is exemplified here.

A dust mass of 90 mg was determined, which is distributed in a gas volume of 1  $\text{m}^3$ . This gas volume was determined via a bellows-type gas flow meter. The gas had a temperature of 28 °C and a static high pressure of 20 Pa compared to the ambient pressure. The ambient pressure was at 101,500 Pa (the corresponding indicators are marked with the index "1"). The objective is to determine how much of a dust mass is contained in 1  $\text{m}^3$  of gas under standard conditions (index "STP"). The conversion is performed based on the aforementioned equation assuming that the substance quantity  $n$  is a constant parameter.

$$\frac{p_1 \cdot V_1}{T_1} = n \cdot R = \frac{p_N \cdot V_N}{T_N} \quad (3)$$

For  $V_N$  the following is true:

$$\frac{p_1 \cdot V_1 \cdot T_N}{T_1 \cdot p_N} = V_N, \quad \text{with figures:} \quad \frac{(20 \text{ Pa} + 101500 \text{ Pa}) \cdot (1 \text{ m}^3) \cdot 273.15 \text{ K}}{(273.15 \text{ K} + 28 \text{ K}) \cdot 101325 \text{ Pa}} = 1.36 \text{ m}^3 \quad (4)$$

| Symbols | Definition                                       | Unit of measure |
|---------|--|-----------------|
| $p_N$   | Static pressure of the gas (standard conditions) | Pa              |
| $V_N$   | Volume of gas (standard conditions)              | $\text{m}^3$    |
| $T_N$   | Gas temperature (standard conditions)            | K               |

The result is a standard volume of 1.36  $\text{m}^3$  in which the dust concentration of 90 mg was measured. Thus, the dust concentration is equal to 66  $\text{mg}/\text{m}^3$  (STP).

## 4.4 Flow rate

Another parameter that is relevant for the measurement of dust contents in flowing gases is the flow rate. The flow rate describes the actual amount of exhaust gas which is generated taking into account the corresponding profile factors (depending on the measuring device and pipe diameter). In addition, the flow rate within certain areas may have an impact on the homogeneous distribution of exhaust gas components across the pipe diameter. This is especially important when characteristic flow conditions (laminar and turbulent flow profile) change or measurements are performed in areas where flow profiles transition from one to the other. Therefore, a differentiation is always made between a turbulent and a laminar flow. A turbulent flow profile (higher speeds) is characterised by a flattened profile in the pipe diameter and causes a well mixed exhaust gas across the whole flow diameter. A laminar flow (low flow speeds) instead promotes a layering of the exhaust gas across the flow diameter. In a laminar flow no noticeable turbulences occur that could cause a mixing of the layers. For measuring the dust load a turbulent flow profile is therefore desirable. The type of flow (laminar or turbulent) is specified via the dimension-free parameter  $Re$  (Reynold's number). It is calculated for pipe flows via the following equation.

$$Re = \frac{w \cdot d}{\nu} \quad (5)$$

| <b>Symbols</b> | <b>Definition</b>                | <b>Unit of measure</b> |
|----------------|----------------------------------|------------------------|
| $d$            | Inner diameter of tube           | m                      |
| $w$            | Average flow rate                | m/s                    |
| $\nu$          | Kinematic viscosity of the fluid | m <sup>2</sup> /s      |

A  $Re$  of 2,300 indicates the transition between turbulent and laminar flow, it is referred to as  $Re_{crit}$  (critical Reynold's number). Flows with  $Re < 2300$  have a laminar flow profile while flows with  $Re \gg 2,300$  are predominantly turbulent. In case of a  $Re$  close to 2,300 a laminar or turbulent flow can never be completely determined and is therefore referred to as a transition area. The inner diameter  $d$  of a pipe does not only specify the original width of the pipe but may also include the roughness and surface characteristics. However, for an exhaust gas pipe coated with soot deposits this cannot always be determined. Thus, only the original width of the pipe is used in the calculations here. To achieve sufficiently high speeds in the exhaust gas, the inner diameter of the pipe can be reduced. This may ensure a more precise determination of the flow rate and is more likely to lead to a turbulent flow. The diameter, however, needs to be large enough so that a dust sampling probe can be inserted.

For an approximation that is usually sufficient when applied in practice, the following values can be used as a basis for identifying the type of flow in a pipe of typical width and roughness.

Table 2: Select physical characteristics as well as a respective calculation<sup>1</sup>

| Parameter            | Kinematic viscosity ( $\nu$ )           | Calculation <sup>2</sup> |   |
|----------------------|---|--------------------------|---|
|                      |   |                          |   |
| Water (20 °C)        | $1 \cdot 10^{-6} \text{ m}^2/\text{s}$  | $w$                      | 2 m/s                                   |
| Air (20 °C)          | $15 \cdot 10^{-6} \text{ m}^2/\text{s}$ | $d$                      | 0,15 m                                  |
| Air (200 °C)         | $47 \cdot 10^{-6} \text{ m}^2/\text{s}$ | $\nu$                    | $20 \cdot 10^{-6} \text{ m}^2/\text{s}$ |
| Exhaust gas (200 °C) | $31 \cdot 10^{-6} \text{ m}^2/\text{s}$ | $Re$                     | 15,000                                  |

Based on the calculation in Table 2, it is apparent that the flow profile is a question of order of magnitude. As long as the flow rate in a pipe with a original width of 150 is significantly above 1 m/s a turbulent flow may be assumed. If the speed is reduced to one tenth only a laminar flow would be achieved.

For flow measurements, as they are performed in the context of this handbook, only very robust measuring equipment is used. The measuring equipment should be able to withstand exhaust gas conditions (high dust load, high temperatures) over a longer period of time. Furthermore, in the case of very small biomass furnaces the flow rates are so low that the selection of suitable measuring equipment is limited to only few devices.

Typically the measurement is done via Prandtl's tube (Prandtl's sensor). It is able to convert the back pressure caused by the exhaust gas into a measure of speed. In addition other methods exist which, however, can be more expensive to purchase than the Prandtl's sensor and may also be more sensitive to the exhaust gas conditions.

The following list shows a typical selection of sensors which are often used for flow measurements in the exhaust gas of biomass furnaces:

- Prandtl's tube (Prandtl's sensor)
- vane anemometer
- vortex sensor
- orifice flowmeter
- hot wire anemometer

Under certain conditions (exact pressure sensor, minimal exterior influences, short measuring line), measurements can still be performed with Prandtl's sensors even at flow rates below 1 m/s. Because of that, and also due to its robustness, it is well suited for measurements in flowing exhaust gases. In the following paragraphs the measurement with the Prandtl's sensor is introduced and the calculation of the flow rate from the exhaust gas back pressure is explained.

<sup>1</sup> The values are tabulated values.

<sup>2</sup> The values assumed for this example are fictitious; they could apply to a furnace with a heating power in the double-digit kW range.

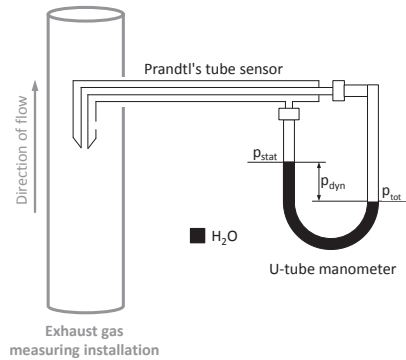


Figure 3: Simplified depiction of the measurement of the exhaust gas back pressure via Prandtl's sensor

Figure 3 shows how a Prandtl's sensor is operated in order to determine the dynamic pressure  $p_{dyn}$  caused by the flowing exhaust gas. This dynamic pressure is the result of the total pressure in the exhaust pipe ( $p_{tot}$ ) minus the static pressure in the exhaust pipe ( $p_{stat}$ ). The dynamic pressure  $p_{dyn}$  therefore corresponds to the pressure that is exclusively caused by the flowing exhaust gas. The dynamic pressure  $p_{dyn}$  is influenced by the flow rate of the exhaust gas as well as by the density of the flowing gas. How the density of the exhaust gas is determined is described in section 4.7 and no further details are mentioned during the following explanations. For the calculation of a flow rate from the dynamic pressure  $p_{dyn}$  the abbreviated and simplified Bernoulli's equation is applied. The most important term of the equation describes the relationship between the speed and the density of the gas as well as the dynamic pressure resulting therefrom.

$$\frac{\rho_1}{2} \cdot w^2 = p_{dyn} \quad (6)$$

$w$  is defined as:

$$w = \sqrt{\frac{2 \cdot p_{dyn}}{\rho_1}} \quad (7)$$

| Symbols   | Definition                                    | Unit of measure   |
|-----------|---|-------------------|
| $\rho_1$  | Density of the gas under operating conditions | kg/m <sup>3</sup> |
| $w$       | Flow rate                                     | m/s               |
| $p_{dyn}$ | Dynamic pressure measured                     | Pa                |

In the complete Bernoulli's equation the geodetic height, the pressure differential and the compressibility of the gas are also taken into consideration. However, for clarity and for an understanding of the fundamental relationships this is neglected here also considering that using the complete equation would only lead to a marginal increases in accuracy for the

task at hand (measuring the speed of flowing exhaust gases). With the exemplary density of the exhaust gas of  $0.76 \text{ kg/m}^3$  determined in section 4.7 the exhaust gas speeds and/or volumetric flows can now be calculated e.g. via the relationships described for various measured back pressures.

Furthermore, in this example it is assumed that the flow measurement was performed in the centre of the flow in an exhaust pipe with a diameter of 150 mm. The centre of flow is defined here as the location in the pipe which features the highest speeds. In general, the flow rate in round pipes increases from the sides to the centre. Since the measurements are taken in the centre of the flow the uneven distribution of the flow rates in the exhaust pipe still has to be considered when calculating the exhaust gas volume flow rate. In order to account for this effect reference is made here to the literature since correcting it in the calculations can be quite complex and it has to be carefully considered to how reasonable such a correction is regarding the task at hand. For the example given a profile factor of  $K = 0.9$  is assumed. Together with the other parameters an exhaust gas volume flow can now be calculated as shown in equation (8). The profile factor primarily depends on the measuring device, the inner diameter of the pipe and the upstream stretches. It should preferably be determined experimentally for each metering installation set up. The profile factors specified by the manufacturer should be applied for in-situ measurements.

$$\dot{V} = w \cdot A \cdot K = w \cdot \frac{\pi \cdot d^2}{4} \cdot K \quad (8)$$

| <i>Symbols</i> | <i>Definition</i>            | <i>Unit of measure</i> |
|----------------|------------------------------|------------------------|
| $\dot{V}$      | Exhaust gas volume flow rate | $\text{m}^3/\text{s}$  |
| $w$            | Flow rate                    | $\text{m}/\text{s}$    |
| $A$            | Pipe diameter flowed through | $\text{m}^2$           |
| $d$            | Inner diameter of the pipe   | $\text{m}$             |
| $\pi$          | Pi                           | 3.14                   |
| $K$            | Profile factor               | 0.9 (as an example)    |

Figure 4 shows flow rates and exhaust gas volume flows at different pressures according to the example above. The diagram depicts a realistic image regarding the relationships between exhaust gas back pressures (measured) and the flow rates and volume flows (both calculated) in the exhaust gas of biomass furnaces. Especially when looking at very low volume flows (less than  $50 \text{ m}^3/\text{h}$ , see box in Figure 4) it becomes apparent how low the flow rates in pipe diameters of 150 mm can be. The pressure sensors used, especially in case of furnaces with low nominal power, should have a high resolution and should also be able to measure accurately to a tenth of a Pascal.

With the example given above it becomes apparent that on furnaces with low volume flows (exhaust pipes with a diameter of 150 mm) flow measurements are not an easy task, due to the low flow rates. A thinning of the diameter can be a helpful tool as it moves the flow away from the transition area and generates back pressures that are necessary for a measurement with Prandtl's tube sensors or a vane anemometers.

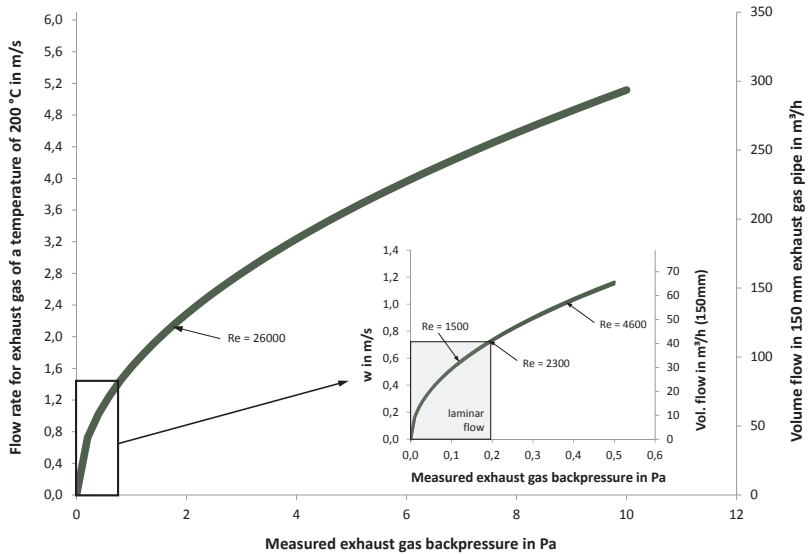


Figure 4: Flow rates and volume flows for differently measured back pressures of an exhaust gas flow with a temperature of 200 °C in an exhaust gas pipe with a diameter of 150 mm, determined via exhaust gas backpressure measurement

## 4.5 Determination of flow profile based on tangential method

For the calculation of an exhaust gas volume flow from a flow rate a profile factor is required. In case of an ideal plug flow the value is 1.0 and for actual flow profiles, under the assumption that the flow rate is measured at the point of its maximum, it is between 1.0 and 0.5 (for non-disturbed laminar flows). Knowing the flow profile, however, is important not only for calculating the volume flow but also required by various standards in order to ensure a representative sampling (1. BImSchV, 2010; BS ISO 9096, 2003; DIN EN 13284-1, 2004; VDI 2066-1, 2006), see Appendix 2.

The cross sections of exhaust gas ducts are typically circular or rectangular. Usually the number of possible measuring axes is one, in rare cases two and only in exceptional cases more than two. Therefore, the task is to determine a flow profile via measurements at spots along one or a maximum of two axes so that it is representative for the cross section.

Each measuring point should be assigned the same sub-area size, however, the geometry may not be trivial. Each sub-area is located in a different quarter of the "pie". Within these quarters the sub-areas are located radially inward in different distances to the center. As illustrated in Figure 5, the measuring points must be located in the centre of gravity of each sub-area. In the aforementioned standards the respective equations are mentioned so that these, with a little adaptation, can be applied to the determination of flow profiles.

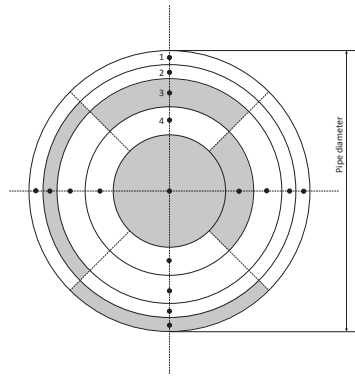


Figure 5: Positions of the measuring points (in accordance with DIN EN 13284). The filled areas feature the same area size

Initially the effective probe radius is determined. This value indicates how close the tip of the Prandtl's tube gets to the wall of the duct. Therefore, the Prandtl's tube is equipped with a ruler while being fully inserted and value  $a_i$  is read on the side facing away from the measuring nozzle. When the Prandtl's tube is fully pulled out the value  $a_o$  is read near the measuring nozzle. The effective sensor radius  $r_e$  (assuming the duct diameter  $D$  is known) amounts to:

$$r_e = \frac{D - (a_o - a_i)}{2} \quad (9)$$

The effective sensor radius should not be significantly larger than the actual radius of the Prandtl's tube (plausibility check).

In accordance with VDI 2066 (similarly also in EN 13284-1 and/or ISO 9096) the coordinates of the measuring points  $i$  for  $n$  measuring points are:

$$x_i = \frac{D}{2} \left[ 1 - \sqrt{\frac{n(n_D - 2i) + 1}{n(n_D - 1) + 1}} \right] \quad \text{for } i < \frac{n_D + 1}{2} \quad (10)$$

$$x_i = \frac{D}{2} \quad \text{for } i = \frac{n_D + 1}{2} \quad (11)$$

$$x_i = \frac{D}{2} \left[ 1 + \sqrt{\frac{n(2i - 2 - n_D) + 1}{n(n_D - 1) + 1}} \right] \quad \text{for } i > \frac{n_D + 1}{2} \quad (12)$$



| <i>Symbols</i> | <i>Definition</i>  | <i>Unit of measure</i> |
|----------------|--|------------------------|
| $i$            | Index of the measuring point on the diameter                               | –                      |
| $n_D$          | Number of measuring points on each measuring axis (including centre point) | –                      |
| $n$            | Either number of measuring axes or number of diameters                     | –                      |
| $x_i$          | Distance of point $i$ from the duct wall                                   | m                      |
| $D$            | Duct diameter  | m                      |

Equation (10) can also be transposed to determine  $n_D$  if for  $x_1$  the effective sensor radius is used and for  $i$  a value of 1. A meaningful value for  $n_D$ , and thereby an integer number of measuring points, is gained if resulting number is rounded up to the next odd number.

$$n_D = \frac{n - 1 + \left(1 - 2\frac{r_e}{D}\right)^2}{n - n\left(1 - 2\frac{r_e}{D}\right)^2} \quad (13)$$

Subsequently, the measuring points to be positioned are calculated via equation (13). The distance to be adjusted with the ruler is then calculated as follows:

$$x_{ruler,i} = a_o - r_e + x_i \quad (14)$$

## 4.6 Theoretical exhaust gas volume and exhaust gas volumetric flow rate

The combustion of solid biomass is a chemical reaction and in total an exothermic one. Through the reaction of elements and compounds contained in the fuel with oxygen from ambient air and oxygen bound in the fuel, energy is released in form of heat and light. Virtually the entire mass of the fuel is participating in this reaction. Assuming certain exhaust gas conditions, the predominant part of the fuel, except for a small part of the fuel which remains as ash, will enter the gaseous phase. These gaseous products including the solid particles (the dust) carried along are considered the total exhaust gas. In case of continuous combustion an exhaust gas volume flow is generated that can be measured. By applying the fundamental knowledge of its chemical and physical relationships, this exhaust gas volume flow can also be calculated (assuming ideal conditions). This can be helpful in order to adjust the measuring equipment appropriately and later assess the plausibility of the measuring results obtained or even while the measurement is ongoing.

In case of an approximated calculation of an exhaust gas volume first the composition of the fuel is important. In general, the composition is determined via an elementary analysis. With the purpose of simplification, in the following scenario an ideal fuel is assumed as the basis of further calculations. This ideal fuel is dry (water free) and (in this case, wood) essentially composed of carbon (C), oxygen (O), and hydrogen (H). In addition, however, there is a significant amount of water bound in the fuel which will evaporate during com-

bustion. Therefore, the water also constitutes a certain share of the exhaust gas volume flow. However, the fuel composition is most often specified referring to its dry state. In the following overview, a differentiation is therefore being made between "dry wood" and "moist wood".

For further theoretical elaboration the following ratios are assumed.

| Element  | Symbol | Average fuel composition in kg/kg <sub>fuel</sub> |            |
|----------|--------|---|------------|
|          |        | Dry wood  | Moist wood |
| Carbon   | C      | 0.49  | 0.45       |
| Hydrogen | H      | 0.06  | 0.05       |
| Oxygen   | O      | 0.44  | 0.40       |
| Nitrogen | N      | <0.01   | <0.01      |
| Water    | W      | 0.00  | 0.09       |

Typically a fuel is also composed of a multitude of other elements which also may play a part in the reaction. However, for the determination of the exhaust gas volume flow their contribution can in this case be neglected. For the following elaborations it is assumed that the fuel consists exclusively of the above mentioned elements.

Under typical conditions, if one kilogramme of such an ideal fuel is combusted completely a certain exhaust gas volume can be expected. If the combustion is continuous, as it is typical for a pellet firing system with automatic fuel transport, a continuous exhaust gas volume flow can be assumed. If the combustion takes place in batches, as it is the case in a wood log burning stove, then different momentary exhaust gas volume flows will occur over time. In order to be able to determine the dust concentration from a log wood burning oven it is therefore recommended to observe the complete combustion of the respective batch while in most cases shorter durations are sufficient in an automatic furnace.

For a better understanding of the determination of the exhaust gas volume an example is given. The following basic information is assumed for combustion in an automatic furnace with a continuous provision of fuel.

| Symbols   | Definition                           | Sample values              |
|-----------|--------------------------------------|----------------------------|
| $P_{nom}$ | Nominal heating power of the furnace | 15 kW                      |
| $\lambda$ | Air ratio                            | 2.2                        |
| $\eta$    | Efficiency of the furnace            | 80 %                       |
| $H_i$     | Heating value of wood (moist)        | 5 kWh/kg (and/or 18 MJ/kg) |

From the nominal heating power and the efficiency of the furnace the necessary fuel heating power ( $P_{fu}$ ) can be determined according to equation (15). It specifies which amount of energy, chemically bound in the fuel, has to be provided to the furnace per unit of time.

$$\frac{P_{nom}}{\eta} = P_{fu} \quad (15)$$

With the heating value  $H_i$  of wood the hourly fuel demand can be calculated using equation (16).

$$\frac{P_{fu}}{H_i} = \dot{m}_{fu} \quad (16)$$

The following values will apply for the example given.

| Symbols        | Definition                  | Sample values |
|----------------|-----------------------------|---------------|
| $P_{fu}$       | Fuel heating power          | 19 kW         |
| $\dot{m}_{fu}$ | Fuel mass flow rate (moist) | 3.8 kg/h      |

It is furthermore assumed that the most important oxidisable components of the fuel (hydrogen, carbon) are completely oxidised to water and carbon dioxide ( $\text{CO}_2$ ) by means of the oxygen present in the ambient air and the fuel. Additional energetic contributions such as the reaction of nitrogen or sulphur with oxygen are neglected here.

The minimum amount of dry air (ambient air)  $L_{min}$  required for the complete combustion of one kg of fuel is calculated via the minimum amount of oxygen necessary for the reaction. For the oxidation of 1 kg C to  $\text{CO}_2$  1.87 m<sup>3</sup> of  $\text{O}_2$  is necessary and for the oxidation of 1 kg  $\text{H}_2$  to  $\text{H}_2\text{O}$  5.6 m<sup>3</sup> of  $\text{O}_2$  is required.<sup>3</sup> If the values for the elementary analysis listed further above are entered in equation (17) (Kaltschmitt et al., 2016) a minimum ambient air demand of 4.5 m<sup>3</sup>/kg fuel (standard condition) can be assumed.

$$L_{min} = \frac{1,87 c + 5,6 h - 0,7 o}{0,21} \quad (17)$$

Via the elementary composition of the fuel, the product gases (carbon dioxide, steam) resulting from the reaction with oxygen, the air ratio and the minimum air demand the actual theoretical exhaust gas amount in m<sup>3</sup> per kg of fuel, relative to standard conditions (101,325 Pa, 0 °C), can be determined. The minimum volume of dry exhaust gas  $V_{exh,dr,min}$  occurring in case of complete stoichiometric combustion is calculated (when sulphur and nitrogen in the fuel are neglected) according to equation (18).

$$V_{exh,dr,min} = 1,87 \cdot c + 0,79 \cdot L_{min} \quad (18)$$

The actual volume of dry exhaust gas  $V_{exh,dr}$  is in turn calculated according to equation (19). At  $\lambda = 1$ , it is identical to  $V_{exh,dr,min}$ .

<sup>3</sup> However, for reasons of clarity, an explanation of these reactions is omitted, here. These are sufficiently described in the literature (Kaltschmitt et al., 2016).

$$V_{exh, dr} = V_{exh, dr, min} + (\lambda - 1) \cdot L_{min} \quad (19)$$

$$V_{exh, dr} = 1,87 \cdot c + (\lambda - 0,21) \cdot L_{min} \quad (20)$$

A dry exhaust gas volume of 4.5 m<sup>3</sup>/kg of fuel will result in  $\lambda = 1$ .

Calculating the moist volume of exhaust gas  $V_{exh, m}$  according to equation (21) takes the water into account which will be present in the exhaust gas due to the combustion of the hydrogen as well as the vaporisation of water contained in the fuel.

$$V_{exh, m} = 1,87 \cdot c + (\lambda - 0,21) \cdot L_{min} + 11,2 \cdot h + 1,24 \cdot w \quad (21)$$

Entering the respective values will result in a specific exhaust gas volume of 8.7 m<sup>3</sup> (at standard conditions) per kg of fuel when assuming the aforementioned fuel composition (moist wood). In case of the hourly combustion of 3.8 kg of this fuel with an air ratio of 2, an average exhaust gas volume flow of approx. 33 m<sup>3</sup> per hour at standard conditions (101,325 Pa, 0 °C) can be assumed. Converted to an exhaust gas temperature of 130 °C this will result in an actual volume flow of 49 m<sup>3</sup>/h for the example given.

Using the methodology described here shows that the approximate volumes of exhaust gas which are to be expect during a measuments can be calculated. If the cross section of the exhaust gas pipe is known the expected average flow rate can be calculated and from that the dust sensor cross sections for an isokinetic dust sampling can be derived.

## 4.7 Density of the exhaust gas

The density of the exhaust gas is especially important for calculating flow rates from the exhaust gas backpressure measured via Prandtl's sensor. However, the exhaust gas is a mixture of known and unknown substances rather than a pure gas and there will be a significant percentage of solid and liquid components (dust and liquid droplets) which have to be neglected at this point. Determining the density of the exhaust gas for dust measuring purposes does not need to be done to the highest accuracy possible. It mainly serves as a value that will help to decide how to operate and adjust the measuring devices. The gas mixture of the exhaust gas can therefore be assumed to be an ideal gas.

For an exact determination of the density of the exhaust gas the composition of the different exhaust gas components, the pressure and the temperature of the exhaust gas and other factors e.g. real gas factor would be required, which are neglected here.

The essential steps for determining the density of the exhaust gas are described in more detail below and the most important physical relationships are covered in detail.

The physical parameter density with the Greek symbol  $\rho$  (Rho) is a substance-specific parameter. It specifies the mass (in kg) which a respective substance with the volume of one m<sup>3</sup> will have. Particularly in case of gases the density is highly dependent on parameters such as temperature and pressure. A large share of known gases are referred to as ideal gases. For ideal gases, the temperature-dependent and pressure-dependent change of density can be described mathematically via equation (22).

$$\rho_N = \frac{M}{V_{m,N}} \quad (22)$$

The molar standard volume  $V_{m,N}$  can be calculated via equation (23).

$$V_{m,N} = \frac{R}{p_N} \cdot T_N \quad (23)$$

The density of a gas with the molar mass  $M$ , the universal gas constant  $R$ , at standard pressure  $p_N$  and the standard temperature  $T_N$  is described by the equation (24).

$$\rho_N = \frac{M \cdot p_N}{R \cdot T_N} \quad (24)$$

| <b>Symbols</b> | <b>Definition</b>                  | <b>Value and/or unit of measure</b>        |
|----------------|------------------------------------|--|
| $\rho_N$       | Density under standard conditions  | kg/m <sup>3</sup>                          |
| $M$            | Molar mass (substance-specific)    | kg/mol                                     |
| $V_{m,N}$      | Molar standard volume              | 0.022414 m <sup>3</sup> /mol               |
| $p_N$          | Pressure under standard conditions | 101,325 Pa and/or kg(m·s <sup>-2</sup> )   |
| $T_N$          | Standard temperature               | 273.15 K                                   |
| $R$            | Real gas constant                  | 8.314 J(K·mol)=kg·m(s <sup>2</sup> ·K·mol) |

The molar standard volume  $V_{m,N}$  corresponds exactly to the volume that is occupied by one mol of an ideal gas under standard conditions (273.15 K and 101,325 Pa). Once the conditions differ from the standard conditions the density has to be corrected. This correction is performed taking into account the temperature and pressure change in accordance with the calculation already performed in Section 4.3.

Equation (26) describes the density under operating conditions ( $\rho_{exh}$ ):

$$\frac{p_N}{\rho_N \cdot T_N} = \frac{p_{exh}}{\rho_{exh} \cdot T_{exh}} \quad (25)$$

$$\rho_{exh} = \frac{p_{exh} \cdot \rho_N \cdot T_N}{p_N \cdot T_{exh}} = \frac{p_{exh} \cdot M_{exh}}{T_{exh} \cdot R} \quad (26)$$

| <b>Symbol</b> | <b>Definition</b>                      | <b>Unit</b>                      |
|---------------|--|----------------------------------|
| $\rho_{exh}$  | Density under operating conditions     | kg/m <sup>3</sup>                |
| $p_{exh}$     | Pressure under operating conditions    | Pa and/or kg(m·s <sup>-2</sup> ) |
| $T_{exh}$     | Temperature under operating conditions | K                                |
| $M_{exh}$     | Molar mass of the exhaust gas          | kg/mol                           |

The density of a gas, or as in this case an exhaust gas, can thus be described via the above mentioned physical relationships and if the following parameters are known:

- The temperature of the exhaust gas
- The molar mass and/or the chemical/physical composition of the exhaust gas
- The absolute pressure of the exhaust gas

Since the exhaust gas is a mixture of different gases, the molar mass of the exhaust gas has to be calculated via the gas composition. Therefore, the concentrations of the exhaust gas components need to be measured (e.g. via CO sensor, FID, FTIR, O<sub>2</sub> sensor, etc.).

For better understandability the following elaborations are based on a simplified example. An emission measurement may result in the following exhaust gas composition:

| Chem. formula    | Definition                       | Concentration $c_i$ in $\text{m}^3/\text{m}^3_{\text{exhaust gas}}$ |
|------------------|----------------------------------|---|
| O <sub>2</sub>   | Oxygen                           | 0.1   |
| CO <sub>2</sub>  | Carbon dioxide                   | 0.09  |
| H <sub>2</sub> O | Gaseous water                    | 0.02  |
| CO               | Carbon monoxide                  | 0.001   |
| HCO              | Other gases, summarised as (HCO) | 0.009   |
| N <sub>2</sub>   | Nitrogen                         | 0.78  |
|                  | Sum                              | 1.00  |

The measurement was performed with an exhaust gas temperature  $T_{\text{exh}}$  of 200 °C (473.15 K) and a discharge pressure of 20 Pa relative to ambient pressure (101,500 Pa) which corresponds to a static pressure in the exhaust gas  $p_{\text{exh}}$  of 101,520 Pa. With the chemical and physical composition, the temperature and the pressure, all information necessary to determine the density of the exhaust gas for these exact operating conditions are available. At first, the molar masses of the respective gases have to be determined with the help of e.g. the periodic table of elements.

| Chem. formula    | Calculation of $M$                | $M_i$ (molar mass) |
|------------------|-----------------------------------|--------------------|
| O <sub>2</sub>   | $2 \cdot 16 \text{ kg/kmol}$      | 32 kg/kmol         |
| CO <sub>2</sub>  | $12 + 2 \cdot 16 \text{ kg/kmol}$ | 44 kg/kmol         |
| H <sub>2</sub> O | $1 \cdot 2 + 16 \text{ kg/kmol}$  | 18 kg/kmol         |
| CO               | $12 + 16 \text{ kg/kmol}$         | 28 kg/kmol         |
| HCO              | $1 + 12 + 16 \text{ kg/kmol}$     | 29 kg/kmol         |
| N <sub>2</sub>   | $2 \cdot 14 \text{ kg/kmol}$      | 28 kg/kmol         |

The density of the exhaust gas can now be calculated as follows:

$$\rho_{exh} = \frac{\sum(c_i \cdot M_i)}{V_{exh}} = \frac{p_{exh} \cdot \sum(c_i \cdot M_i)}{T_{exh} \cdot R} \quad (27)$$

| <i>Symbol</i> | <b>Definition</b>  | <b>Unit</b>                                       |
|---------------|--|---|
| $\rho_{exh}$  | Density of the exhaust gas                                   | kg/m <sup>3</sup>                                 |
| $c_i$         | Concentration of the respective component in the exhaust gas | m <sup>3</sup> /m <sup>3</sup> <sub>exhaust</sub> |
| $M_i$         | Molar mass of the respective component in the exhaust gas    | kg/kmol   |
| $T_{exh}$     | Waste gas temperature  | K   |
| $p_{exh}$     | Pressure under operating conditions                          | Pa and/or kg/(ms <sup>2</sup> )                   |
| $V_{exh}$     | Molar volume under operating conditions                      | m <sup>3</sup> /mol                               |

For the example given, the density is calculated assuming the exhaust gas  $\rho_{exh}$  to be 0.76 kg/m<sup>3</sup>. In this case the density is only half as high as under standard conditions which is primarily caused by the doubling of the temperature from 273 K (at standard conditions) to 473 K (under operating conditions).

## 4.8 Oxygen measurement

The oxygen content in the exhaust is relevant to further comparisons of the dust contents since in accordance with Germanies First Pollution Control Regulation (1. BImSchV), the total dust concentrations always has to be put in relation to 13 Vol.-% of oxygen. The same applies to other gaseous pollutants.

For the determination of the content of molecular oxygen in the exhaust gas quite often its paramagnetic property is utilised. Due to the special spin of the oxygen molecule, it features a magnetic momentum in comparison to most other molecules which is why it is attracted by a magnetic field. This characteristic is being taken advantage of.

The classic paramagnetic method operates in accordance with the magneto-mechanic measuring principle. In this, a dumbbell body that is located in an inhomogeneous magnetic field is pushed out of this magnetic field by the oxygen molecules present in the measured gas. Located on the dumbbell itself is a conductor loop through which an electric current flows. This electric current moves the dumbbell against the direction in which the oxygen molecules are pushing it. The strength of the electric current which is necessary to bring the dumbbell back into its original position is proportional to the oxygen concentration in the measured gas. This method is one of the most precise ones for quantifying the oxygen content in the range of 0 – 100 %. However, due to its moving parts it is sensitive to mechanic stresses such as shock and vibration. In addition, its use is limited due to the complex setup and the sensitivity to corrosive components present in the measured gas.

In case of a paramagnetic oxygen sensors based on the thermomagnetic measuring principle, a portion of the gas stream is influenced by the presence of an inhomogeneous magnetic field (permanent magnet). Through the magnetic field the oxygen molecules are redirected and this change (deflection) of the gas stream can be observed by measuring

technology. The intensity of the deflection is a measure for the concentration of oxygen prevalent in the measured gas. However, flow-through, temperature, and air pressure have a stronger impact on the results obtained by the thermomagnetic measuring principle than with the magnetomechanical principle.

According to HOFFMANN (1996) measuring devices which operate this way have a high cross sensitivity, i.e. other (interfering) gases may influence the measurement.

To circumvent these disadvantages sensors based on **solid state electrolytes** were developed which generally display very low cross sensitivity. The solid state electrolyte is mainly composed of zircon dioxide  $ZrO_2$  which is doped with yttrium oxide  $Y_2O_3$  to increase oxygen ion conductivity. The oxygen ion conductivity is the property of the solid state electrolyte that is essential to this measuring principle. It becomes applicable at temperatures  $> 350\text{ }^\circ\text{C}$ . The necessary operating temperature of the sensor is set by an integrated electric heating facility.

For the determination of the oxygen partial pressure in a measured gas a reference gas with known oxygen partial pressure is required. This is typically done by using ambient air as reference. In the sensor the measured gas and the reference gas are separated by the heated solid state electrolyte.

The oxygen atoms of a gas in touch with the solid state electrolyte are in balance with the oxygen ions in the electrolyte (corresponding to the oxygen partial pressure) of the gas. If the electrolyte now acts as a separation between gases of different oxygen partial pressures a difference in potential  $\Delta U$  is developing between the two boundary layers (via the electrolytes).

By two platinum electrodes, which are located on both sides of the solid state electrolyte, the difference in potential can be measured (potentiometric measuring principle). The electrodes have to be porous in order to allow for contact of the gas phase with the solid state electrolyte.

In accordance with the Nernst equation, the unknown oxygen partial pressure ( $p_1$ ) in the measured gas can be calculated according to equation (28):

$$\Delta U = \frac{RT}{nF} \cdot \ln \frac{p_2}{p_1} \quad (\text{with } n = 4) \quad (28)$$

| Symbol     | Definition                               | Unit                  |
|------------|--|-----------------------|
| $\Delta U$ | Difference in potential                  | V                     |
| $F$        | Faraday constant                         | $\text{A s mol}^{-1}$ |
| $T$        | Temperature inside the sensor            | K                     |
| $p_1$      | Oxygen partial pressure in measured gas  | mbar                  |
| $p_2$      | Oxygen partial pressure in reference gas | mbar                  |
| $n$        | Number of electrons exchanged            |                       |
| $\ln$      | Natural logarithm                        |                       |

Solid state electrolyte oxygen sensor are primarily used to determine the air ratio in the



catalytic converter of vehicles with gasoline engine and for the control of automatic furnaces. They have a very high sensitivity particularly in the area of  $\lambda = 1$  and are also being utilised in oxygen trace analysis.

An **electrochemical oxygen sensor** is based on an arrangement of two electrodes. It consists of a platinum electrode (working electrode) acting as the cathode and an Ag/AgCl electrode (alternatively also a Pb electrode) (counter electrode) which acts as the anode. Both electrodes are located in an electrolyte solution which in most cases is alkaline. A defined potential is applied between the two electrodes.

Via a membrane (typically PTFE) oxygen can diffuse from the measured gas into the electrolyte solution and from there on to the platinum electrode. At the platinum electrode the reduction of the oxygen will take place (due to the applied potential) while at the counter electrode the oxidation of the respective metal is taking place. Due to the equalisation of charge an electric current (maximum diffusion current) will result between the two electrodes which can be measured. The strength of the maximum diffusion current is dependent on the oxygen partial pressure prevalent in the measured gas.

## 4.9 Firing capacity from exhaust gas measurements

The firing capacity can be calculated from an exhaust gas volume at standard conditions when the fuel composition and the oxygen content is known. In several countries it is common to specify emissions relative to a standard volume at a reference content in the exhaust gas. In Germany, for example, the following specification for small-scale furnaces with solid fuels is common:  $\text{m}^3$  (STP) at 13 %  $\text{O}_2$ . In other countries, such as in Austria or Switzerland, so-called emissions factors are used which refer to the fuel heating value. Here it is common to specify MJ heating value wf (water free). Both specifications are equally important. The conversion of these two parameters is going to be explained below. In Switzerland the conversion factors are binding by law (BAFU, 2009).

Since a standard volume of exhaust gas at a reference oxygen level corresponds to an energy value, the standard volume flow corresponds to a power value (the firing capacity). The standard volume flow can be calculated in accordance with Chapter 4.5 and Chapter 4.6 with knowledge of the flow profile and temperature profile while taking into consideration the exhaust gas density (for which the oxygen concentration is necessary). After conversion to reference oxygen and correction for the humidity of the exhaust gas the firing capacity for a biomass can be calculated according to equation (29):

$$\dot{Q} = \dot{V} \frac{p}{p_0} \frac{T_0}{T} \cdot \frac{21\% - 13\%}{21\% - O_2} \cdot 0.67 \frac{\text{MJ}}{\text{m}^3 (\text{STP}, d) 13\% O_2} \cdot (87\% + 58\% \cdot O_2) \quad (29)$$

The first term is to calculate the standard volume flow (moist), the second term relates it to the oxygen reference, the third term introduces an energy output and the last term corrects it for the exhaust gas moisture content.

## 4.10 Hydrocarbons

During the combustion of biomass hydrocarbons (HC) play an important role. Thus, HC is defined as the sum of all chemical compounds that are composed by various proportions of hydrogen and carbon and in some cases even other elements. In case of the combustion of solid biomass fuels with a complete reaction, as assumed in Chapter 4.6, all carbon atoms and all hydrogen atoms will be completely converted to carbon dioxide and water. Thus, no HC will occur. In reality this is not the case since products of incomplete combustion always occur. Of the hydrocarbons contained in the emissions of biomass furnaces some compounds pose a potential health risk due to their chemical/physical characteristics. They are therefore quite often the subject of scientific investigations. For the HC emitted from the combustions of biomass, there are no standardised conventions as to how these are to be determined, categorised and assessed. However, different indicators exist which are widely used for measuring and assessing HC emissions without them being legally binding or regulated in a standardised way (EPA, 2014b). For the combustion of biomass the following terms for hydrocarbon compounds are commonly used:

- VOC – volatile organic compound
- VVOC – very volatile organic compound
- SVOC – semi-volatile organic compound
- NMVOC – non-methane volatile organic compound
- TC – total carbon
- OC – organic carbon
- EC – elemental carbon
- TOC – total organic carbon
- OGC – organic gaseous carbon
- PAH – polycyclic aromatic hydrocarbons
- totalC – total carbon

These terms are frequently used when exhaust gases from biomass combustion are described and characterised while they may be combined (based on different criteria) as well as used separately. Depending on the degree of differentiation additional groups may be added or replaced.

In most cases the measuring method defines which group of hydrocarbon compounds is relevant.

Different measuring methods exist which can provide more or less detailed information about the actual chemical composition of the HCs. In principle, the state of matter (solid, liquid, or gaseous) which the HCs have while present in the exhaust gas stream should be indicated. The state of matter is crucial for selecting the type of measuring equipment to be used. In the following elaborations only the HCs which are in gaseous form in the exhaust gas will be considered. The exhaust gas temperature and the pressure under which the measurement is performed will influence the result of the measurement. Some hydrocarbon compounds will be in gaseous form in the hot exhaust gases while others are liquid or solid in the cooler and diluted exhaust gases (e.g. in the form of dust particles or on dust particles).

Hydrocarbons that are present in exhaust gas are quite often recorded with the help of a flame-ionisation detector (FID). In a flame-ionisation detector the thermal ionisation of the measured gas component is performed in an oxyhydrogen flame. Thereby the measured gas stream and hydrogen are mixed and burned in air via a nozzle. The nozzle for generating the flame and the flame tip itself serve as cathode. Located above the flame is a collecting electrode (grid electrode) which acts as anode. The electrons released upon ionisation of the HCs in the flame reach the anode and can be detected as an electric current.

The strength of the current is approximately proportional to the amount of oxidisable carbons in the measured gas. However, due to the different molecule structures, a FID displays a different responsiveness to different HC compounds. Therefore, the term "response factor" is introduced.

Here the response factor  $R_{f,x}$  represents the relationship between the concentration of a compound detected with the FID and the actual concentration of the compound in the measured gas and is specific to each measuring device. Typically the FID is thus calibrated to propane.

$$R_{f,x} = \frac{S_{FID}}{c_x} \quad (30)$$

| <b>Symbols</b> | <b>Definition</b>  | <b>Unit of measure</b> |
|----------------|--|------------------------|
| $R_{f,x}$      | Response factor of a compound X                                  |                        |
| $S_{FID}$      | Concentration of compound X displayed by the device              | ppm                    |
| $c_x$          | Concentration of compound X actually present in the measured gas | ppm                    |

Even though the exhaust gas from biomass combustion is a gas mixture of unknown composition, it is assumed here that the response factors of the majority of the HCs contained in an exhaust gas do not significantly deviate from each another. Therefore, as mentioned earlier, the measuring signal is proportional to the amount of oxidisable carbon atoms in the measured gas. The concentration of gaseous hydrocarbons (total carbon) contained in the exhaust gas is in this case either specified as methane equivalents or as propane equivalents (TESTA, 2012). Often the specification mg/m<sup>3</sup> of oxidisable carbon is used in a legal context.

With an FID compounds with C-H and C-C bonds can be detected particularly well. Compounds with C-O, C-N and C-S bonds are also detectable while compounds such as  $N_2$ ,  $O_2$ , CO,  $CO_2$ ,  $H_2$ ,  $NO_2$  and noble gases can not (or barely) be detected. This results in a low cross-sensitivity to these compounds and also to the carrier gas. HCs which have a high thermal instability are also difficult to detect since they may disintegrate before entering the flame (ChemgaPedia, 2012). Critical comments, quote from DIN EN 12619 (2011: 17):

*"The FID is equipped with a cleaning tool for the measured gas in order to prevent a contamination of the device by particles and condensation. Long-chained HCs which enter the measuring device as solids are filtered out and therefore not measured. Even though the guidelines require the measurement of TOC (total organic carbon), the FID only measures TVOC (total volatile organic carbon). This is generally accepted by the industry and the responsible authorities. [...] exhaust gases containing aerosols or the presence of high-molecular VOC may therefore cause indeterminable measuring uncertainties. This should be taken into consideration when planning the measurements."*

To prevent such processes (especially condensation issues) the temperature in the (heated) sampling line should be controlled.

Otherwise, aerosols with high-boiling hydrocarbons can lead to contamination of the sampling system and thereby to uncontrollable memory effects which are caused by adsorption and desorption processes. This may in turn result in e.g. a delayed zero-point setting when the calibration gas is fed. If aerosols from the exhaust gas were previously retained in the filter or accumulated in the sampling line and temperatures of the filter or sampling line rise above the evaporation temperature of these aerosols they transition into the gaseous phase and will be measured by the FID (DIN EN 12619, 2011).

In addition to the method described, a large multitude of methods exists that are based on spectroscopy. Spectroscopy is an important and indispensable tool of analytical chemistry (Lindner et al., 2006). Generally, a differentiation can be made between emission spectroscopy and absorption spectroscopy. The basis of spectroscopy is the interaction of gas molecules with electromagnetic radiation. Absorption spectroscopy takes advantage of the fact that upon being exposed to certain wavelengths of light discrete parts of radiation will be absorbed. Through this absorption the energy of the exterior electrons is changed which will result in so-called valence electron spectra (Skoog & Leary, 1996). The reduced radiation intensity as compared to the original intensity caused by the partial absorption is proportional to the concentration of particles of the respective material. Absorption spectroscopy is differentiated in respect to where the spectra of gases is located on the electromagnetic radiation spectrum. Thus, STAAB (1994) separated gas spectrometers into three groups:

- (1) ultraviolet (UV), visible (VIS) and near infra-red (NIR),
- (2) infra-red (IR) spectrometers and
- (3) microwave (MW) spectrometers.

For gas analysis IR spectrometers are used most frequently. Furthermore, a differentiation can be made between dispersive (DIR) and non-dispersive (NDIR) infra-red spectrometry. In infra-red absorption spectroscopy, the property of certain gases to absorb infra-red radiation is utilised. With IR spectrometry-devices usually gases consisting of i.a.  $\text{CO}_2$ ,  $\text{CO}$ ,  $\text{CH}_4$ ,  $\text{C}_3\text{H}_8$ -molecules can be detected.

## 4.11 Sampling for analysis of gaseous components

While setting up the measuring devices for a sampling campaign quite often an assessment of a representative partial gas volume flow and its transfer to the actual detector needs is necessary. The partial flow sampling is typically performed via a heated sampling probe as well as a heated sampling line. It is crucial that the temperatures inside the probe and the sampling line are above the saturation state of the condensable components.

Due to high concentrations of particulate matter the respective filter elements may be contaminated and need to be cleaned regularly. When using an unheated sampling line condensation of water may occur. In this case, corresponding condensate traps will be necessary which are often built into the measuring device.

Figure 6 features a selection of typical devices which are used for sampling of a partial gas flow from the hot exhaust gas of a biomass furnace. Figure 6 A shows an unheated probe which simultaneously measures the temperature at the sampling site and the low pressure in the exhaust gas pipe. If such a probe is used in hot exhaust gases attention has to be paid to the cooling of the measured gas on its way through the line and the subsequent condensation of the water still present in the hot exhaust gas. In the worst case it can deposit on the interior walls of the sampling line and cause contamination. Since the condensation in the sampling line is uncontrolled the measured gas will be relieved of suspended matter (dust) and water before being fed into the analyser via a condensate trap (here in combination with activated carbon and sodium permanganate) and a filtering unit (see Figure 6 B). This equipment has to be cleaned daily, especially in case of longer lasting measurements, in order to prevent damage to the sensor. Figure 6 C shows an electrically heated probe. Heated probes are always used in connection with a heated sampling line (Figure 6 D). Such equipment is used when the measuring device is capable of working at temperatures above the dew point of water. With some devices the amount of water present in the gas phase can also be determined this way. Filter units which need to be cleaned regularly are used here as well.

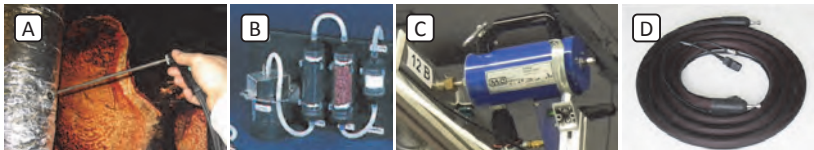


Figure 6: Depictions regarding partial gas flow retrieval, A: unheated probe for gas sampling, B: condensate trap for removal of water from the gas phase as well as filter cartridges (activated carbon and sodium permanganate) (photo: Eheim Messtechnik), C: heated probe for gas sampling, D: heated sampling line (© Eismann & Ströbe GbR)

The gas concentrations determined with a "hot-measuring" device differ from those determined with a "cold-measuring" device. This must be considered in further analysis. When defining the concentrations the specification "dry" is often added. Thus, it is clear that the concentration refers to an exhaust gas composition that does not contain gaseous water. If the measurement is performed above the dew point of water the specification "moist" should be added. If concentrations which were measured in dry exhaust gas are compared with those from moist exhaust gas the moisture content needs to be accounted for via equation (31).

$$c_{i,dr} = \frac{c_{i,m}}{1 - c_{H_2O}} \quad (31)$$

| Symbol     | Definition   | Unit |
|------------|--|------|
| $c_{i,m}$  | Concentration of an exhaust gas component "i" in the moist exhaust gas |      |
| $c_{i,dr}$ | Concentration of an exhaust gas component "i" in the dry exhaust gas   |      |
| $H_2O$     | Moisture concentration in the gas phase                                |      |

## 4.12 Referring to standard oxygen concentration

Depending on the type and setup of the biomass furnace the concentrations of the various exhaust gas components may have temporal fluctuations and can generally be different between systems. While differences in concentration during combustion are typical for furnaces operated in batches (e.g. ovens and firewood boilers) automatically operated furnaces (e.g. pellet and woodchip furnaces) have more consistent concentrations. In both the amount of the concentrations can depend on the quality of the combustion, the fuel itself as well as the settings of the dampers. Biomass furnaces are typically operated so that more combustion air is fed than is stoichiometrically necessary for the conversion of the fuel (so-called excess air). Therefore, the exhaust gas will always be partly containing some externally fed air. This can be recognized when oxygen is detected in the exhaust gas in concentrations still worth mentioning.

Biomass furnaces are quite often operated with an excess air coefficient between 1 and below 3. The excess air coefficient  $\lambda$  is determined according to equation (32) via the oxygen concentration in the ambient air and the oxygen concentration in the exhaust gas.

$$\lambda = \frac{0,21}{0,21 - c_{O_2,exh}} \quad (32)$$

| Symbols       | Description                             | Unit of measure |
|---------------|---|-----------------|
| $\lambda$     | Excess air coefficient lambda           | -               |
| $c_{O_2,exh}$ | Oxygen concentration in the exhaust gas |                 |

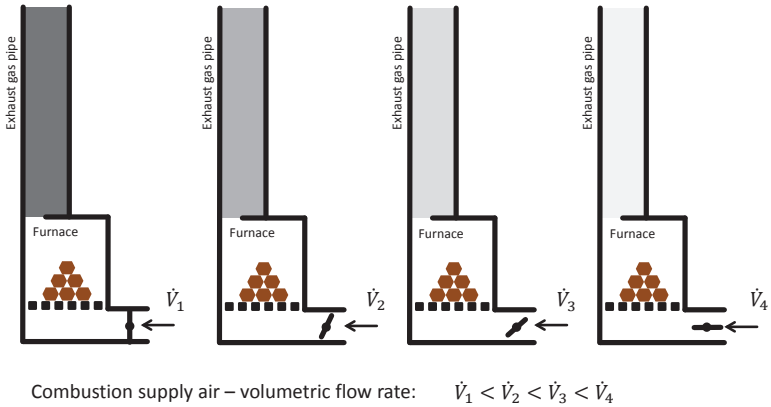


Figure 7: Schematic depiction of a furnace at different combustion air intake volume flows and change of the pollutant concentrations in the exhaust gas associated therewith (reduction of the blackening symbolises the decreasing concentration due to the dilution)

Thus, an oxygen concentration of 10.5 % in the exhaust gas corresponds to a combustion air ratio of 2 and a concentration of 14 % to a ratio of 3. A combustion air ratio of 1 would exist if exactly 0 % oxygen were to be measured in the exhaust gas. Figure 7 visualises the impact of different excess air coefficients (combustion air ratios) and the corresponding reduction of the pollutant concentrations in the cross section of the exhaust gas pipe. The different shades of grey symbolise the dilution of the dust concentration in the exhaust gas of the furnace generated by the different combustion air supply volumes.

An exhaust gas measurement (e.g. for dust or CO), as it is typically performed on biomass furnaces, never detects the absolute amount of the respective component emitted upon combustion of a specific amount of fuel. The current concentration can be determined but not the absolute amount. The measurement is therefore always a subset (several minutes) of a longer process (several hours). It is important to be aware of the phase of the overall process which it is representative of. Therefore, respective sampling periods are defined for different combustion technologies. However, results will only be comparable to one another if oxygen content is measured in addition to the temperature while conducting the actual emissions measurement. Since different air settings may have been applied the oxygen in the exhaust gas will indicate the different dilution ratios.

The average oxygen concentration determined over the duration of the dust measurement will be used to standardise the respective concentrations of exhaust gas components (of e.g. dust or CO) and put it in relation to a common reference oxygen content via equation (33).

$$c_{i,ref} = \frac{0.21 - c_{O_2,ref}}{0.21 - c_{O_2,real}} \cdot c_{i,m} \quad (33)$$

| Symbols        | Definition   | Unit of measure         |
|----------------|--|-------------------------|
| $c_{i,ref}$    | Concentration converted to reference oxygen content                          | mg/m <sup>3</sup> (STP) |
| $c_{i,m}$      | Measured average pollutant concentration                                     | mg/m <sup>3</sup> (STP) |
| $c_{O_2,ref}$  | Reference oxygen content (13 % Germanies First Pollution Control Regulation) |                         |
| $c_{O_2,real}$ | Average oxygen content measured in dry exhaust gas                           |                         |

The reference oxygen content  $c_{O_2,ref}$  is specified in Germanies First Pollution Control Regulation (1. BImSchV, 2010) to be 13 %. For systems which are not regulated by 1. BImSchV other reference values may be assumed.  $c_{O_2,real}$  is the placeholder for the measured oxygen concentration (in %) in the dry exhaust gas. The average measured concentration of an exhaust gas component  $c_{i,m}$  is included in the calculation in milligrams per cubic metre of exhaust gas at standard condition (mg/m<sup>3</sup> [STP]), (101,325 Pa, 273.15 K).

When standardising the measurements to the reference oxygen content, attention has to be paid to the fluctuation of the oxygen concentration in the exhaust gas and its impact on the accuracy of the component  $c_{i,m}$  which is to be standardised. This is most obvious when high oxygen concentrations (> 13 %) are observed while the concentrations of the components are severely fluctuating (variance  $s > 2$  %). The impact of variance ( $s$ ) increases with increasing oxygen concentrations in the exhaust gas, as illustrated by Figure 8 in combi-

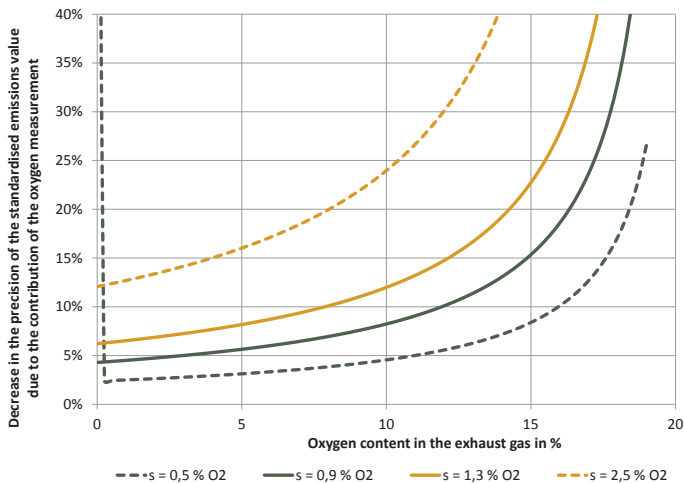


Figure 8: Impact of oxygen concentrations on the precision of an exhaust gas component to be standardised to the reference oxygen content. The variable  $s$  describes the variance of the oxygen concentration in the exhaust gas.



nation with Table 3. In other words, the higher the average measured oxygen content is relative to the reference oxygen content, the more pronounced the impact of the variance of the oxygen measurement will be on the precision of the value to be standardised. The measuring accuracy of the oxygen measurement also drops at high oxygen contents since the measuring devices are calibrated to a maximum of 21 Vol.-% of oxygen.

According to Figure 8, an oxygen concentration in the exhaust gas of 8 % (which is subject to an assumed variance (s) of 2.5 %) will decrease the accuracy of the standardised emission value by 20 % (c.f. Table 3). If a dust sampling gave a concentration of 61 mg/m<sup>3</sup> standardised to 13 % O<sub>2</sub> it would have an accuracy of only ± 12 mg/m<sup>3</sup>. Depending on the mode of operation of the furnace, the fluctuation of the oxygen concentration will be of different severity. In case of a batch combustion the oxygen content has a noticeable peak when the flames have gone out and only the residual carbon is still glowing. This will be reflected in significantly increased CO concentrations in the exhaust gas during that time. The fluctuations in the exhaust gas concentration of continuously operated furnaces, however, can in general be considered lower than those of furnaces operated in batches.

Table 3: Comparison of dust concentrations of 100 mg/m<sup>3</sup>, standardised to 13 % O<sub>2</sub>. Average oxygen contents in the exhaust gas of 2 to 18 % are assumed that were determined at differing variance (s), which results in differing precisions. In the fields marked in red, the variance exceeds 15 % of the standardised dust value.

| Average O <sub>2</sub><br>in exhaust<br>gas in % | 100 mg/m <sup>3</sup> ,<br>standardised to<br>13 % O <sub>2</sub> | Decrease in the precision of a standardised dust value due to average oxygen<br>concentrations with differing variance in mg/m <sup>3</sup> (13 % O <sub>2</sub> ) |            |            |            |
|--|---|--|------------|------------|------------|
|  |   | S = ± 0.5 %  | s = ±0.9 % | s = ±1.3 % | s = ±2.5 % |
| 2  | 42.1  | ±1.1   | ±2.0       | ±2.9       | ±5.6       |
| 4  | 47.1  | ±1.4   | ±2.5       | ±3.6       | ±7.1       |
| 6  | 53.3  | ±1.8   | ±3.2       | ±4.66      | ±9.1       |
| 8  | 61.5  | ±2.4   | ±4.3       | ±6.22      | ±12.3      |
| 10   | 72.7  | ±3.3   | ±6.0       | ±8.7       | ±17.4      |
| 12   | 88.9  | ±5.0   | ±9.0       | ±13.1      | ±26.8      |
| 14   | 114.3   | ±8.2   | ±14.9      | ±22.0      | ±46.8      |
| 16   | 160   | ±16.2  | ±29.8      | ±44.6      | ±106.7     |
| 18   | 266.7   | ±45.7  | ±87.9      | ±142.3     | ±727.3     |

The relationships described above illustrate that the measurement and comparison of average concentrations of individual exhaust gas components is subject to other parameters and their respective level.

### 4.13 Exhaust gas dilution

Exhaust gas dilution is beneficial for several measuring applications and may be used intentionally or even occur unintentionally depending on the technology and setup. In case of type-testing (e.g. required as part of 1. BlmSchV) or measurements done by chimney sweepers, the exhaust gas is usually not diluted and the measurements of total dust are performed directly in the hot exhaust gas. However, once the hot exhaust gas leaves the

chimney opening a dilution will occur which makes measurements in intentionally diluted exhaust gas be more similar to an immission measurement than the measurement in the hot exhaust gas of the furnace.

Hereby the dust concentration determined in the diluted exhaust gas is not standardised to a certain oxygen concentration, as is done during the measurement of crude gas, but put in relation to the amount of energy generated and/or consumed. This convention makes it easier to compare concentrations determined by different methods.

However, in Germany the threshold values for air pollutants emitted are, most of the time, specified in  $\text{g}/\text{m}^3$  (relative to a specific residual oxygen content in the exhaust gas). Since the oxygen concentration in diluted exhaust gas is very high (close to 21 %) a value determined in diluted exhaust gas cannot be converted to  $\text{mg}/\text{m}^3$  (dry, [STP], 13 %  $\text{O}_2$ ) without further adjustment (c.f. preceding section). For this particular case, a dilution rate or a dilution ratio has to be determined in a suitable manner.

In general, cleaned or untreated ambient air (indoor/outdoor) or processed compressed air is used as dilution agent.

An exhaust gas dilution leads to the following effects:

- The temperature of the diluted exhaust gas is lower than the exhaust gas temperature.
- The dew point of the diluted exhaust gas is lower than the dew point of the regular exhaust gas; a dew point below room temperature is desirable.
- Due to the dilution the concentration of the respective pollutant drops.
- Phase transitions occur.
- The flow rate increases.
- In case of high contents of organic gaseous hydrocarbons, these may cool down to a certain extent and condense during the dilution process, whereby dust concentrations in the exhaust gas may be increased.

While the dilution ratio may commonly be defined as the relationship of one component in relation to all other components of a mixture, in science the dilution ratio is defined as the ratio of the respective component to the total entity (mass, volume, quantity) (United Nations, 1997). The inherent balance of all components therefore adheres to the law of mass conservation. As such, the dilution ratio  $DR$  is defined by the ratio of the masses and mass flows according to equation (34):

$$DR = \frac{m_{tot}}{m_{exh}} = \frac{m_{da} + m_{exh}}{m_{exh}} \quad \text{respectively} \quad DR = \frac{\dot{m}_{tot}}{\dot{m}_{exh}} = \frac{\dot{m}_{da} + \dot{m}_{exh}}{\dot{m}_{exh}} = \frac{m_{tot}}{m_{tot} - m_{da}} \in [1, \infty[ \quad (34)$$

| <b>Symbols</b>            | <b>Definition</b>  | <b>Unit of measure</b> |
|---------------------------|--|------------------------|
| $DR$                      | Dilution ratio   | -                      |
| $m_{tot} (\dot{m}_{tot})$ | Total mass (flow) consisting of dilution air and exhaust gas | g (g/s)                |
| $m_{exh} (\dot{m}_{exh})$ | Mass (flow) of the undiluted exhaust gas                     | g (g/s)                |
| $m_{da} (\dot{m}_{da})$   | Mass (flow) of the dilution air                              | g (g/s)                |

The total mass  $m_{tot}$  is composed of the mass of the dilution air  $m_{da}$  and the mass of the undiluted exhaust gas  $m_{exh}$ . This applies to mass flows in the same way. Since the total mass (the total mass flow) cannot become smaller than any partial mass (any partial mass flow) a value range of  $\geq 1$  and  $< \infty$  results. A dilution ratio of 1 is therefore equivalent to the undiluted exhaust gas.

#### 4.13.1 Calculations with the dilution ratio

If the measurement is performed in diluted exhaust gas the concentration of the respective component under undiluted exhaust gas conditions is usually of interest in order to be able to compare results from different measurements. An appropriate way of conversion is described below.

Provided that the component to be diluted (index i) is not contained in the dilution air the concentration of the respective component in the diluted exhaust air can be calculated according to equation (35).

$$c_{i,exh} = c_{i,tot} \cdot DR \quad (35)$$

| <b>Symbols</b> | <b>Definition</b>   | <b>Unit of measure</b> |
|----------------|---|------------------------|
| $c_{i,exh}$    | Concentration of a component i in the undiluted exhaust gas | e.g. ppm               |
| $c_{i,tot}$    | Concentration of a component i in the diluted exhaust gas   | e.g. ppm               |
| $c_{i,da}$     | Concentration of a component i in the dilution air          | e.g. ppm               |

However, in case the component to be diluted (index i) is also contained in the dilution air equation (36) will apply:

$$\dot{m}_{i,tot} = c_{i,tot} \cdot \dot{m}_{tot} = c_{i,exh} \cdot \dot{m}_{exh} + c_{i,da} \cdot \dot{m}_{da} \quad (36)$$

Shortened by the exhaust gas mass flow rate, this results in equation (37):

$$c_{i,tot} \cdot DR = c_{i,exh} + c_{i,da} \cdot (DR - 1) \quad (37)$$

#### 4.13.2 Determination of the dilution ratio

The dilution ratio is a parameter that is composed of several linearly correlations. The question therefore arises how this parameter can be determined as accurately as possible with as little effort as possible. The following approaches may be considered:

- Determination of the mass flows
- Determination via concentrations of indicator substances
- Temperature and/or differences in temperature

These approaches as well as their limitations and accuracies are discussed in more detail below.

The dilution ratio, as was explained above, can be calculated from the mass flows of the incoming air and the exhaust gas, the exhaust gas and the dilution air, or the diluted air and the dilution air. There are no known inexpensive methods for a direct mass flow determination of dust-laden gases with changing composition.

However, determining flow rate and mass flow via the density of the exhaust gas can be a useful approach (c.f. section 4.7). The flow rate, for example, can be measured with the Prandtl's sensor (c.f. section 4.4) if the profile factor of the flow in the respective section of the pipe is known (c.f. section 4.5). Optionally, the average speed can also be performed with the vane anemometer and/or a vortex flow sensor. At the same time, knowledge of the corresponding temperature and negative pressure is assumed in order to be able to standardise the volume flows.

Another option for determining the dilution ratio is by identifying so-called indicator substances instead of mass flows. Suitable indicator substances are e.g.  $O_2$ ,  $CO_2$  or CO. These are in most cases contained in detectable amounts in the dilution air, the exhaust gas, or the diluted exhaust gas. In general, the composition of the ambient air can be obtained from literature. The measurement of the concentrations in diluted and undiluted exhaust gas should be performed with comparable measuring devices. The calculation is then performed in accordance with the relationship described in section 4.13.1.

The dilution can also be determined via the temperature and/or via temperature differences. Instead of the conservation of mass, the calculations are then based on the conservation of energy. By setting the zero-point at  $0^\circ\text{C}$  an enthalpy balance can be created analogous to the conservation of mass. Instead of mass concentrations the product of average caloric heat capacity and temperature at the respective measuring point is used according to equation (38).

$$\bar{c}_{p,tot} \cdot \vartheta_{tot} \cdot \dot{m}_{tot} = \bar{c}_{p,exh} \cdot \vartheta_{exh} \cdot \dot{m}_{exh} + \bar{c}_{p,da} \cdot \vartheta_{da} \cdot \dot{m}_{da} \quad (38)$$

| Symbols           | Definition  | Unit of measure |
|-------------------|---|-----------------|
| $\bar{c}_{p,tot}$ | Average isobar heat capacity of the diluted exhaust gas | J/(kg · K)      |
| $\bar{c}_{p,exh}$ | Average isobar heat capacity of the exhaust gas         | J/(kg · K)      |
| $\bar{c}_{p,da}$  | Average isobar heat capacity of the dilution air        | J/(kg · K)      |
| $\vartheta_{tot}$ | Temperature of the diluted exhaust gas                  | K               |
| $\vartheta_{exh}$ | Temperature of the exhaust gas                          | K               |
| $\vartheta_{da}$  | Temperature of the dilution air                         | K               |

In a first approximation the differences in average heat capacities can be neglected where equation (38) is reduced to equation (39).

$$DR = \frac{\vartheta_{exh} - \vartheta_{da}}{\vartheta_{tot} - \vartheta_{da}} \quad (39)$$

If thermocouples are utilised which are not specifically calibrated for this purpose their error may lie above the error assumed for the approximation. If resistance thermosensors are used a correction in accordance with Seidler (2011) is recommended.

#### 4.13.3 Error analysis

In the following, various options for determining the dilution ratio are compared with each other: determination via the indicator substances O<sub>2</sub> and CO<sub>2</sub> as well as the determination based on temperature via thermocouple and resistance thermometer. The measurements are each subject to different flaws which, in turn, results in different tolerances for the dilution ratio. For Figure 9 the following error margins were assumed:

- O<sub>2</sub>: ± 0.3 Vol-%. This error is typical for electrochemical cells. If two high-quality measuring devices operating with paramagnetism are available an error of ± 0.1 Vol-% may be assumed.
- CO<sub>2</sub>: ± 0.2 Vol-%. This error can be assumed if two well calibrated measuring systems are present and are verified regularly.
- Temperature: ± 1 K for thermocouples (type K), however the value may be higher as observed during a joint data collection. ± 0.15 K for resistance thermometers. Some customised resistance thermometers may achieve even smaller deviations.

Figure 9 features a comparison of the four options to determine the dilution ratio. The variants feature different error margins which affect the accuracy of the calculated dilution ratios differently, depending on the actual dilution ratio. Due to the high accuracy of the temperature sensors, measuring the temperatures seems to be the most accurate in comparison to the measurement of indicator substances. To achieve a high accuracy, also in practice, it is important to provide a proper installation, a good mixing of the gas and ideally no heat radiation.

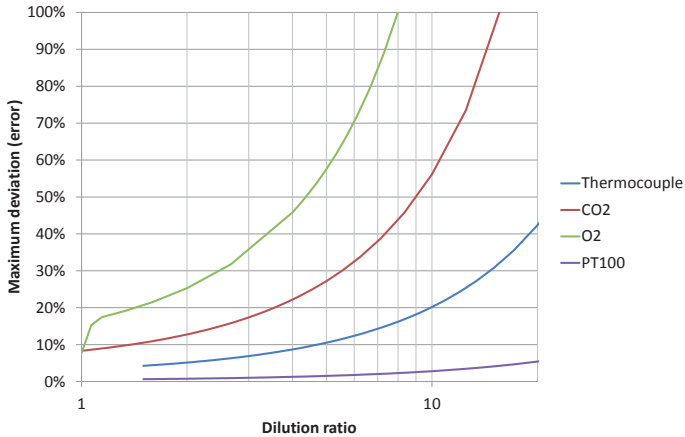


Figure 9: Comparison of deviations when determining the dilution ratio via different measuring methods. The determination via resistance thermometer is by far the most accurate method.

## 5 Gravimetric measurement of dust

### 5.1 General

Determining solid and/or liquid components and their concentration in the exhaust gas is often the main focus during emissions measurements. Here it is of great importance to detect the individual components, which are in most cases not visible to the human eye, in their frequency of occurrence. Also, it is important that the measured values can be related to common parameters and that the measurements are reproducible. For the gravimetric measurement of dust the primary kilogram is the basis on which concentrations are expressed. Gravimetric dust measurement still are state-of-the-art especially for measurements on small-scale furnaces. Despite the comparatively small masses typically emitted from these appliances, gravimetric dust measurement are often favored over measurements methods not based on the primary kilogram. In any case, the quality and/or reproducibility of dust measurements have to be ensured via clear definitions of the measuring conditions. For measurements on biomass furnaces standards such as VDI 2066 Sheet 1, DIN EN 13284 and the method in accordance with the certification programme DINplus (DIN CERTO, DIN SPEC 1101:2010-02) are, amongst others, applicable. In addition, several scientific studies focus on the development of such methods and their adjustment to the particularities of small-scale furnaces. In the following sections various issues are being discussed which have to be taken into consideration when dust measurements in flowing exhaust gases, particularly on biomass furnaces, are conducted.

### 5.2 Test set-ups

In this section various test set-ups are introduced which can be used for the determination of dust concentrations in exhaust gases. In addition to the measurement of dust often other gaseous components are also measured, therefore, the set-up of the respective equipment is explained as well. Here, the arrangement of both the devices and the accessories necessary for dust measurements on biomass furnaces and/or in the exhaust gas duct of the furnace is referred to as test set-up. Figure 10 illustrates an exemplary arrangement of measuring devices necessary for a gravimetric dust sampling. Therein a furnace with vertical exhaust gas system is depicted which is equipped with measuring nozzles for simultaneous measurement of different parameters in the exhaust gas flow. Ideally, exhaust gas temperature, flow rate, discharge pressure, dust concentration and gaseous exhaust gas components can be monitored and measured. The probes have to be placed so that the inlet and outlet sections are taken into consideration as well as the respective minimum distances between the measuring openings. It is of utmost importance that the installed measuring components in the exhaust gas system will not change or disturb the gas flow. Any disturbance of the flow may lead to a formation of turbulences which may in turn lead to a separation of the components and cause differences in concentration. In this regard even an elbow pipe may cause a disturbance of the flow as do sharp bends, branches

and sampling openings. The measuring points A to D indicated in Figure 10 represent the approximate position and the parameters to be recorded as part of a qualified dust measurement. For a regular dust measurement those parameters are: oxygen content, exhaust gas temperature, flow rate and discharge pressure. For further emission measurements additional measuring points may be added. Especially for a complex analysis of the emissions a gas measuring equipment that goes beyond the pure oxygen measurement is being utilised. For particle measurement another sampling point has to be added which would also be the case if e.g. toxicity is to be measured.

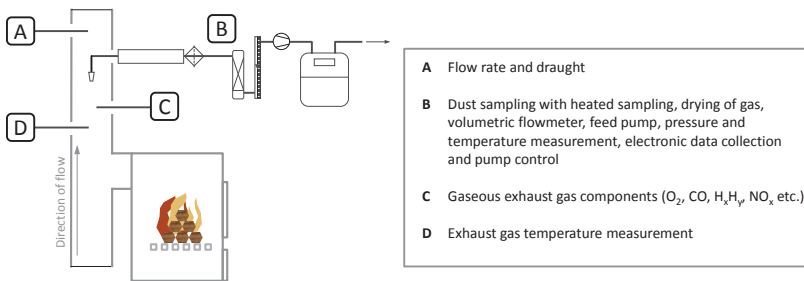


Figure 10: Sample depiction of the gravimetric total dust measurement on a furnace for solid fuels

Depending on the method the number of test probes used and their placement will differ. However, probes for oxygen measurement, for example, may already be combined with temperature sensors, just like the flow measurement may already have been integrated into the dust measurement equipment. That, in turn, will reduce the number of sampling points even further. However, this is largely dependent on the measuring equipment.

The following additional set-ups are utilised for the determination of emissions, especially dust emissions.

The emission measurement in accordance with HKI (2012) is, for instance, performed on a modified exhaust gas measuring set-up in accordance with EN 13240. Here dust is not part of the parameters to be investigated. Only gaseous components (mainly CO and  $O_2$ ) are measured. Figure 11 shows a comparison of different exhaust gas measuring set-ups. The set-ups A and B correspond to the requirements of EN 13240 (DIN EN 13240, 2010) with a defined pipe cross section of 150 mm. Measuring installation C is modelled after DIN SPEC 1101:2010-02, and measuring installation D is being utilised at the DBFZ. In most cases, the measuring installations have a diameter of 150 mm. The diameter specified in the standard, however, is comparatively large and may lead to very low flow rates in the exhaust gas pipe partly due to the low heating outputs of the furnaces. This makes an isokinetic dust sampling considerably more difficult since the equipment measuring the flow will no longer produce reliable results. Furthermore, the flow rates are inbetween turbulent and laminar which may promote an uneven distribution of the exhaust gas components across the pipe's cross section.



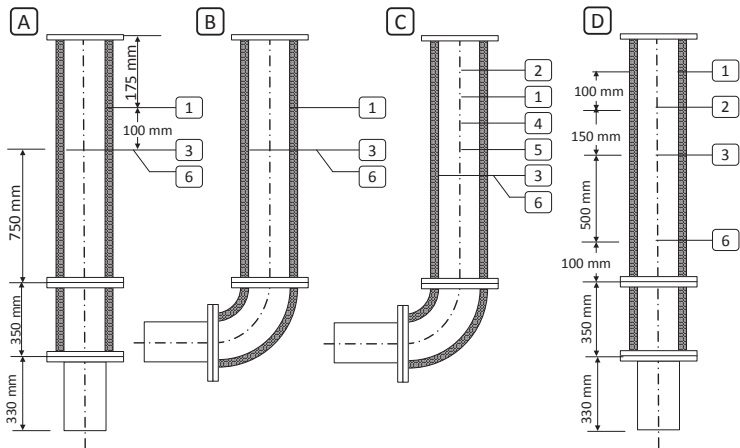


Figure 11: Various set-ups of exhaust gas measuring installations for the measurement on single-room furnaces; the inner pipe diameter is 150 mm in all set-ups

The dimensions used in Figure 11 are in accordance with the measuring equipment needed for the following exhaust gas analyses.

- |   |                                 |
|---|---------------------------------|
| 1 | Static discharge pressure       |
| 2 | Gravimetric measurement of dust |
| 3 | Temperature                     |
| 4 | Hydrocarbon compounds           |
| 5 | Nitrous oxides                  |
| 6 | Exhaust gas composition         |

For the sampling it is furthermore important to keep the required minimum distances between the sampling points and minimum distances to installed components which affect the gas flow. In EN 13240 (DIN EN 13240, 2010), for instance, the distances between the sampling points is specified as well as the exact length of the inlet pipe in relation to its diameter (Figure 11 A and B). The measuring set-up at the DBFZ has the exact dimensions listed in Figure 11.

Figure 12 shows a selection of different measuring set-ups described in the literature. To know the distribution of the flow rates across the pipe's diameter is therefore of importance for dust sampling and general emissions measurements. Investigations on the measurement taken at an exhaust gas flow pipe have shown that larger instrument nozzles can cause turbulences and therefore distort the flow pattern.

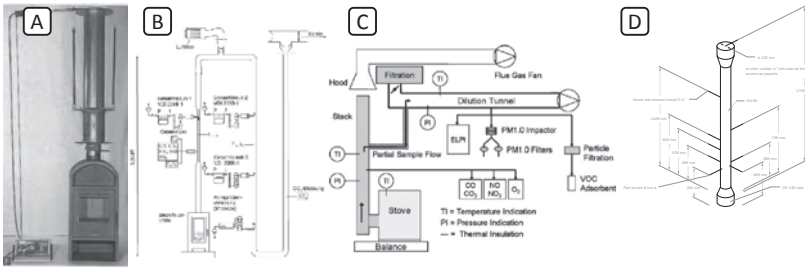


Figure 12: Examples of various measuring set-ups described in literature A: Figure from STIFTUNG WARENTEST (2011), B: Figure from ELLNER-SCHUBERTH et al. (2010), C: Figure from SIPPULA et al. (2006), D: Depiction of DBFZ standard metering installation

In addition to the insertion of probes into the exhaust gas pipe the alignment of the pipe can be of importance. To minimize errors in gravimetric dust measurement, the measurement should generally be performed in the vertical part of the exhaust gas pipe. A measurement in the horizontal sections is possible if an error in the dust concentration due to sedimentation, impaction, agglomeration and other processes can be excluded. Otherwise, the errors caused thereby have to be quantified and accounted for mathematically.

### 5.3 Dust sampling from the exhaust gas

The dust sampling can be performed in a variety of different ways. Different methods as well as different filter materials and filter types are available. The actual sampling can be performed outside of the exhaust gas pipe or directly in the exhaust gas pipe. Accordingly, the two methods are being distinguished as in-stack method (in the chimney) and out-stack method (outside the chimney). In addition, the extraction of the sample can be done via automatically controlled, manually controlled or non-regulating automatic suction. Often the exact error margin of the different processes remains unknown as, in the case of measurements on an exhaust system, it is often not possible to differentiate whether a deviation observed during multiple measurements was caused by the measuring device itself or is due to fluctuations in the furnace.

Albeit, a series of fundamental rules exists that can increase the accuracy of measuring. One of these fundamental rules is to perform sampling under isokinetic conditions. Isokinetic conditions are achieved when the extraction of an exhaust gas volume flow is performed with the same flow rate as the exhaust gas. For sampling under isokinetic conditions not only the flow rate is of importance but the vectors are as well.

Figure 13 highlights the fundamental concept of sampling under isokinetic conditions. Depicted is the head of a sampling probe that extracts a partial flow from a flowing exhaust gas. The variable  $c_1$  represents the flow vector of the flowing exhaust gas and  $c_2$  represents the partial flow extracted. In case of a non-isokinetic sampling the ratio of dust particles which are affected by the altered flow rate and those who are not may be changed. In case of hypokinetic sampling (Figure 13, Image A), in which the flow rate inside the

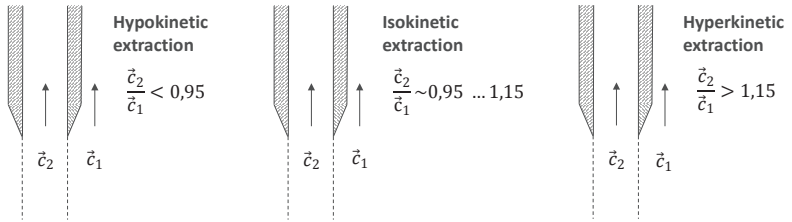


Figure 13: Schematic depiction of a sampling probe with the speed vectors of the extraction ( $\vec{c}_2$ ) as well as of the exhaust gas ( $\vec{c}_1$ ), for the three different conditions under which a partial flow will be retrieved from a flowing exhaust gas. In practice, the isokinetic sampling in accordance with VDI 2066 Sheet 1 constitutes the ideal case (Image B).

sampling probe is lower than that of the exhaust gas, dust concentrations may have been artificially increased. Whereas, in case of hyperkinetic sampling (Figure 13, Image C), in which the flow rate inside the sampling probe is higher than that of the exhaust gas, the opposite is true.

Depending on the objective of the investigation, the dust measurement may be performed according to the aforementioned conditions. A dust sampling can, for example, be performed to verify the adherence to emission thresholds, as is typically done by the chimney sweep. The way in which a measurement is performed is in most cases defined by the available time and equipment.

In some cases dust measurements are performed in order to investigate the different components and the composition of the dust. Here, time is less important and the sampling is more defined by the respective methodology.

Figure 14 shows a selection of steps typical for dust measurements performed under laboratory conditions. During the sampling, attention should be paid to accuracy and compliance with the defined methodology. A selection of different standard operation procedures is available in the Appendix and may serve as guidelines for proper sampling and handling of the respective equipment. Following these procedures will provide reproducible dust measurements with high accuracy.

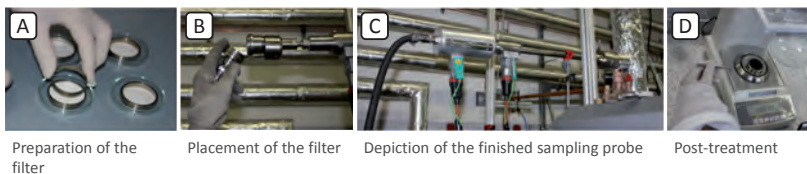


Figure 14: Sample sequence of a gravimetric total dust sampling in accordance with VDI 2066 Sheet 1, as it is performed in the laboratory for test purposes. A: Preparation of the planar filter, for this, c.f. SOP in the Appendix, B: Placement of the prepared filter into the sampling probe, C: Depiction of the finished sampling probe, D: Post-treatment of the used filter sample, in particular, the weighing-out to determine the load.

## 5.4 Error propagation

In order to calculate the mean error (and, if a confidence interval is specified, an extended error) of the calculation formula, the square root of the sum of the squares of the partial derivation of the respective formula is multiplied with the assumed error of each measured parameter. This calculation requires the individual parameters to be independent of one another e.g. a deviation in air pressure will not interfere with the determination of the temperature. Calculating the error propagation, a calculation guideline for the target parameter, in this case the dust concentration in the partial flow, needs to be developed. The dust concentration ( $c$  in  $\text{kg}/\text{m}^3$ ) is calculated according to equation (40):

$$c = \frac{m}{V_N} \quad (40)$$

The determination of the volume extracted is performed at ambient temperature and pressure. However, a conversion to standard volume according to equation (41) is required:

$$V_N = \frac{p}{1013 \text{ mbar}} \frac{273 \text{ K}}{\vartheta} V \quad (41)$$

In accordance with the German First Pollution Control Regulation, the respective equation from Section 4.12 must be used for the conversion to reference oxygen content. In combination the above mentioned will result in equation (42):

$$c_{13\%O_2} = \frac{21\% - 13\%}{21\% - O_2} \frac{m}{V} \frac{1013 \text{ mbar}}{p} \frac{\vartheta}{273 \text{ K}} \quad (42)$$

In general, the total error is calculated according to equation (43):

$$\Delta c_{13\%O_2} = \sqrt{\sum_{i=O_2}^{\vartheta} \left( \frac{\delta c_{13\%O_2}}{\delta i} \Delta i \right)^2} \quad (43)$$

The contributions of the individual errors (after respective transformations) need to be considered and are listed below:

$$\left( \frac{\Delta c_{13\%O_2}}{\delta O_2} \right) \Delta O_2 = \left( \frac{c_{13\%O_2}}{21\% - O_2} \right) \Delta O_2$$

$$\left( \frac{\Delta c_{13\%O_2}}{\delta m} \right) \Delta m = \left( \frac{c_{13\%O_2}}{m} \right) \Delta m$$

$$\left(\frac{\Delta c_{13\%O_2}}{\delta V}\right) \Delta V = \left(\frac{c_{13\%O_2}}{V}\right) \Delta V$$

$$\left(\frac{\Delta c_{13\%O_2}}{\delta p}\right) \Delta p = \left(\frac{c_{13\%O_2}}{p}\right) \Delta p$$

$$\left(\frac{\Delta c_{13\%O_2}}{\delta \vartheta}\right) \Delta \vartheta = \left(\frac{c_{13\%O_2}}{\vartheta}\right) \Delta \vartheta$$

The total error is then described according to equation (44):

$$\Delta c_{13\%O_2} = c_{13\%O_2} \sqrt{\left(\frac{\Delta O_2}{21\% - O_2}\right)^2 + \left(\frac{\Delta m}{m}\right)^2 + \left(\frac{\Delta V}{V}\right)^2 + \left(\frac{\Delta p}{p}\right)^2 + \left(\frac{\Delta \vartheta}{\vartheta}\right)^2} \quad (44)$$

The contribution of the individual measured parameters to the overall result will differ and depends on the selected measuring configuration.

## 5.5 Particularities of dust sampling during in-situ measurements

The information summarised in this section deals with the particularities that may occur when performing so-called in-situ measurements. In this context, in-situ measurements are measurements in which the measuring setup as well as the environmental conditions considerably differ from those in the laboratory. Referred to as in-situ measurement in this context often are measurements that are performed on actual systems under operating conditions. Typical in-situ measuring conditions are encountered by the chimney sweep when he performs emission measurements as part of his cleaning and inspection activities on domestic chimneys. Often measurements performed during research projects, in order to map the practical operating behaviour of e.g. boilers, are also considered in-situ measurements. The measuring equipment utilised here can be very similar to test stand measuring equipment.

During the planning and performance of in-situ measurements practical aspects are the main focus. In general, a distinction needs to be made between measurements that take place under operating conditions (in-situ) and measurements that are performed on a test stand in a laboratory, especially with respect to the interpretation of the results. In case of in-situ measurements certain sampling points may not exist or may be maladjusted. In addition, it may not be possible to operate all measuring devices simultaneously due to insufficient space. Also the climatic conditions may affect the results as a measurement performed during winter (with ambient temperatures around 0 °C) cannot be compared to results from measurements during the summer (at >20 °C). All this needs to be considered when analysing and during interpretation of results. Since often during research projects extensive measuring campaigns are conducted the measurements need to be carefully planned. In the following section the methods practised by the sub-working group members are being presented together with their experiences from practical application.

### 5.5.1 Communication with plant operators and affected parties

Two typical situations occur when communicating with plant operators or other relevant parties. On the one hand, a plant/system operator may have the need to perform measurements on their plant due to a specific issue. In this case their support is granted. If, on the other hand, the measurements are not performed upon request of the plant operator but for the interest of a third party (e.g. research community) it is important to communicate that no disadvantage will arise for the operator. Possible negative effects for e.g. residents (during measurements in residential buildings) or the workflow at a plant (during measurement in industrial objects/industrial plants) should be outlined and the sampling be tailored to the respective circumstances. Thus, it should be communicated how much space is required for the sampling and that a corresponding area shall be kept available. The actual sampling time should be tailored to the processes and workflow on-site. The necessary drill holes in the flue gas path need to be announced and have to be approved by the operator accordingly. For the operator's understanding, it is helpful to emphasise that the system itself will not be modified and that the drilling will only be performed on the flue gas pipe. Advice and approval from the local chimney sweep regarding newly installed sampling points may be of advantage. To emphasise that the system will be left nearly unaltered various types of closings for the sampling points should be offered to the operator. The three most common ways to close holes in the flue gas path are: sampling caps, aluminium stickers and screws.

It is important to inform the people affected by the measurements ahead of time as the breakdown of warm water and heat, noise disturbance or the obstruction of paths may be considered unreasonable without sufficient notification. Providing notification to the affected parties will create tolerance and understanding for the upcoming measurements. Residents whose flats may be affected by intense noise due to the instruments used (e.g. compressor, vacuum pumps) should be informed personally about the schedule of measurements and the reason for it.

### 5.5.2 Selecting a plant and preparing the measurements

The furnace where the measurements will take place should be checked prior to the measurements as the environment in which the in-situ measurement will be conducted may differ. During this initial inspection the following factors should be paid attention to in particular:

- **Opportunities for access:** A lot of boilers feature more than a single access point. The safest, quickest and easiest path to the system should be identified taking into consideration the measuring instruments used. It needs to be clarified how access to the system will be provided the day of the measurement. In case of measurements at an industrial plant an employee should be available for support. For measurements in residential buildings the building manager or any person that can be present during the time of the measurement may be of help (pensioner, family with children).

- **Electricity:** For almost all measuring instruments an external supply of electricity is required. The availability of sufficient power outlets needs to be checked and whether the available sockets are fuse-protected. The maximum amount of power that can be drawn depends on the fuse(s) for the sockets. In most cases, these are 16 A fuses. It is recommended that the measuring instruments are split into different groups so that each group remains 10 % below the maximum available power (in case of 16 A and 230 V: 3.68 kW) at a single socket. If this is done before the actual setup on-site it will save time and eventually prevent the triggering of automatic circuit breakers. If a heavy current connection ( e.g. with 400 V, IEC 60309) is available on-site this can be used as well. In this case an adapter for lower current (230 V) should be brought.
- **Limited space:** The size of the boiler room is the crucial factor for the success of the measurements. Depending on the instruments used a certain amount of space is needed which needs to be assessed during the initial inspection. Often it is possible to place peripheral devices in adjacent storage rooms or hallways.
- **Flue gas path:** The flue gas path must fulfil certain requirements so that a measurement can be performed. Typical requirements are e.g. the overall length of the flue gas path or the presence of a long and straight segment for the sampling. These specific requirements depend on the number and type of the measurements which are to be conducted. Thus, during the inspection the height of the flue gas path exiting the boiler should be noted so that mounts for the flue gas probes can already be prepared prior to the setup of the instruments.
- **Heat storage:** A heat storage is very advantageous for in-situ measurements as it acts as a heat sink. Such a heat sink allows for a sufficient duration of the measurement. However, it needs to be coordinated with the operator to make the heat storage available and possibly empty it before the measurement.

In order to perform the measurement smoothly a checklist should be prepared ahead of time. This list can be a helpful tool to keep a check on the equipment brought along. Table 4 can serve as an example for such a list (based on the requirements in Germany). Additional utensils required can be added here (or certain items may be substituted with what is applicable in the respective country).

Table 4: Sample checklist with equipment for an in-situ measurement

| Tool / measuring device                     | Quantity                      | To                       | From                     |
|---|-------------------------------|--------------------------|--------------------------|
| Screwdrivers                                | 4 (2 Phillips, 2 plain slot)  | <input type="checkbox"/> | <input type="checkbox"/> |
| Open-ended spanners (various rated widths)  | 6 – 17 mm                     | <input type="checkbox"/> | <input type="checkbox"/> |
| Cable ties                                  | 1 set                         | <input type="checkbox"/> | <input type="checkbox"/> |
| Screws (to close openings)                  | 4 each of 8, 10, 12 and 14 mm | <input type="checkbox"/> | <input type="checkbox"/> |
| Cable reel                                  | 1                             | <input type="checkbox"/> | <input type="checkbox"/> |
| Adapter IEC 60309 to earthed safety socket  | 1                             | <input type="checkbox"/> | <input type="checkbox"/> |
| Electric drill with drill bits              | 2 (stepped drill & 4 mm)      | <input type="checkbox"/> | <input type="checkbox"/> |
| Maintenance equipment for measuring devices | 1                             | <input type="checkbox"/> | <input type="checkbox"/> |
| Extension lead                              | 2                             | <input type="checkbox"/> | <input type="checkbox"/> |
| Power distribution lead                     | 2                             | <input type="checkbox"/> | <input type="checkbox"/> |
| Notepad                                     | 1                             | <input type="checkbox"/> | <input type="checkbox"/> |
| Writing implements                          | 2                             | <input type="checkbox"/> | <input type="checkbox"/> |
| Folding rule                                | 1                             | <input type="checkbox"/> | <input type="checkbox"/> |
| Pocket torch                                | 1                             | <input type="checkbox"/> | <input type="checkbox"/> |
| Mirror (hidden openings)                    | 1                             | <input type="checkbox"/> | <input type="checkbox"/> |
| Steel wire for fixing in position           | 1 role                        | <input type="checkbox"/> | <input type="checkbox"/> |
| Folding stool/ladder                        | 1                             | <input type="checkbox"/> | <input type="checkbox"/> |
| Rag/ cleaner/cloth                          | 1, each                       | <input type="checkbox"/> | <input type="checkbox"/> |
| Signs for tenants/neighbours                | 3                             | <input type="checkbox"/> | <input type="checkbox"/> |
| Sellotape/adhesive tape                     | 1                             | <input type="checkbox"/> | <input type="checkbox"/> |
| Combination pliers                          | 1                             | <input type="checkbox"/> | <input type="checkbox"/> |
| Multigrip pliers                            | 1                             | <input type="checkbox"/> | <input type="checkbox"/> |
| Pincers                                     | 1                             | <input type="checkbox"/> | <input type="checkbox"/> |
| Gloves                                      | 1 pair                        | <input type="checkbox"/> | <input type="checkbox"/> |
| Anchor clamps (various styles)              | at least 3 pcs.               | <input type="checkbox"/> | <input type="checkbox"/> |
| Probe heads                                 | 3 (various diameters)         | <input type="checkbox"/> | <input type="checkbox"/> |
| Folding heater                              | 1                             | <input type="checkbox"/> | <input type="checkbox"/> |
| Suction extraction facility                 | 1                             | <input type="checkbox"/> | <input type="checkbox"/> |
| Xerogel                                     | 1                             | <input type="checkbox"/> | <input type="checkbox"/> |
| Prepared filters                            | 5                             | <input type="checkbox"/> | <input type="checkbox"/> |
| Clean transport containers                  | 5                             | <input type="checkbox"/> | <input type="checkbox"/> |
| Tweezers                                    | 2                             | <input type="checkbox"/> | <input type="checkbox"/> |
| Aluminium foil                              | 1 role                        | <input type="checkbox"/> | <input type="checkbox"/> |
| Holding devices                             | 4                             | <input type="checkbox"/> | <input type="checkbox"/> |
| Doorstops                                   | 3                             | <input type="checkbox"/> | <input type="checkbox"/> |
| Stepladder                                  | 1                             | <input type="checkbox"/> | <input type="checkbox"/> |



### 5.5.3 Performing the measurements, relevant parameters

The parameters to be measured are strongly dependent on the respective objective (e.g. legislative framework). For measurements according to the German First Pollution Control Regulation (1.BImSchV) all values need to be in relation to a reference oxygen content which therefore requires measurement of oxygen content ( $O_2$ ) in the flue gas. In furnaces smaller than 500 kW, according to the same legislation, the emissions of carbon monoxide (CO) and dust are required. Environmental parameters such as relative humidity  $\varphi$ , pressure  $p$  and temperature  $T$  can be useful for a detailed analysis. A log-book for parameters which are not recorded automatically should be created prior to the measurement so that their values can be logged by hand in regular intervals (5 minutes, better: 2 minutes). Any incidents or irregularities should also be documented in order to ensure a sufficient interpretation of the measurements. The flue gas temperature should be determined via a type K thermocouple. This parameter is helpful in assessing the loss of heat via the chimney. Also, via the flue gas temperature it can be assessed whether the boiler is operating under stationary conditions. However, the flue gas concentrations of  $O_2$  and CO have priority over temperature measurements for determining stationary operation. In flue gas pipes with 150 mm diameter flue gas velocities  $v$  between 1.5 and 3.5 m/s are typical. Thus, to determine the flue gas velocity a pressure differential sensor with an accuracy of 0,1 Pa in combination with a Prandtl's tube is recommended (accuracy of 1 Pa) in order to avoid imprecise measurements.

At the end of the measurement samples of the fuel and the residues (ash) can be collected for further analysis if needed. To achieve a representative sampling, the samples should be taken in accordance with DIN 51701-2. This is necessary since the composition of untreated biomass solid fuels (e.g. log wood) is very inhomogeneous and standardised biomass solid fuels (e.g. pellets) are often a combination of different batches (due to storage and transport). Representative sampling needs to account for situations found in e.g. regular indoor pellet storages (Figure 15, A) or biomass-fired systems (Figure 15 B).

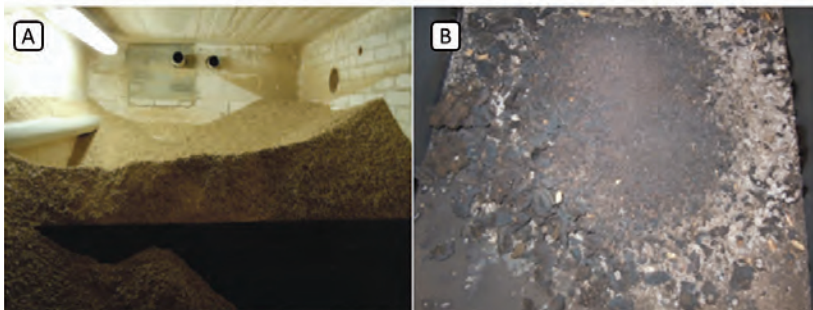


Figure 15: Pellet store of a real operational facility (Image A), ash and combustion residues from a pellet furnace (Image B).

**Arrangement of the measuring technology:** Site specific arrangement of the technology necessary for in-situ measurements is a common challenge. In this section general aspects regarding their installation will be covered and one successfully tested arrangements of the possible sampling spots will be explained. To prevent different dilution ratios while measuring the flue gas composition simultaneously at different points the measurements should always be performed upstream of the dilution damper. It may also be advantageous to seal off pipe connections with heat-resistant (aluminium) tape since a lot of flue gas paths are not sufficiently sealed. Otherwise the share of unaccounted air in the flue gas can increase along the flue gas path due to the negative pressure which is present most of the time. For the same reason the openings into which the probes are inserted should also be sealed off with heat-resistant tape. The flue gas temperature should be measured directly at the flue gas outlet of the boiler especially if a balancing of the boiler is planned. For the temperature measurement a comparatively small measuring opening is required which leaves enough space for other measuring devices in close proximity. If the flue gas temperature is recorded at this point it will not be representative for the entire cross section of the exhaust gas pipe. The temperature measured here can only be an approximation and the values will fluctuate by up to  $\pm 15$  °C across the diameter. If possible, the flue gas composition should be measured at the second sampling point after the flue gas outlet of the boiler. This will minimize the risk of unaccounted air entering the system before the measurement and leave the rest of flue gas pipe for total dust sampling.

The requirements of VDI 2066 for selection of the total dust sampling site are difficult to comply with in in-situ measurements and it is rare to find a vertical section of the exhaust system which can be used for the measurements. Additionally, in small systems it is difficult to provide the quintuple hydraulic diameter before and after the point of measurement (as stated in VDI 2066). Thus, a compromise needs to be made (c.f. Figure 16). One successful

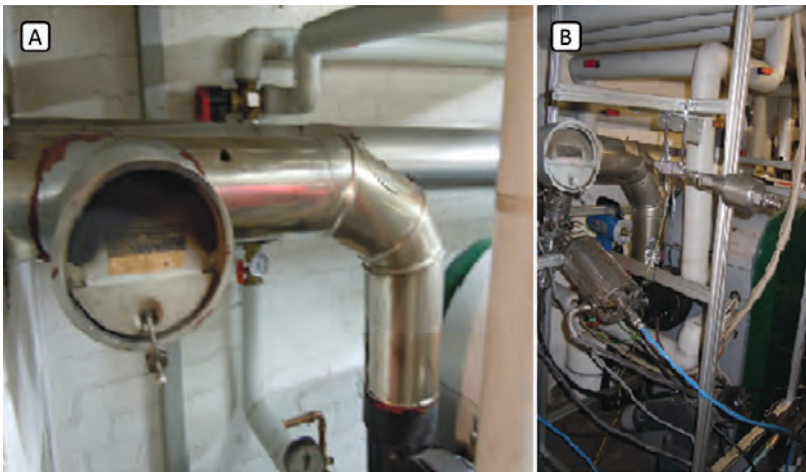


Figure 16: Example of exhaust gas pipe before (Image A) and after (Image B) installation of the measuring equipment

approach can be to separate the longest straight stretch of pipe into thirds and to utilise two thirds of the stretch as inlet. If the oven runs continuously ( $\text{CO}$ ,  $\text{O}_2$  constant) the flue gas velocity will barely change over time. The flow rate before and after the measuring point can be determined and the extracted amount of flue gas can be adjusted accordingly. According to the value determined before the measuring point the extraction volume flow will be adjusted. Based on the velocity measured after the measuring point it can be verified whether a constant flow rate was observed. If an additional measuring device is used (e.g. for online particle counting) it should be installed at the last measurement site (upstream from flue gas outlet of the boiler).

**Time management during measurements:** For a time efficient performance of the measurement the setup has to be planned carefully. Equipment with a long warm up phase should be turned on first while the rest of the setup is under way. Measurement sites should be identified next and the corresponding openings be drilled before further equipment is brought to the measurement sites and installed.

#### 5.5.4 Completion of the measurement

Once the measurement is completed all electronically stored data should be backed-up to a second storage device to prevent transport-related data loss. Similar to the setup, equipment with an extended cool-down phase or which needs to be flushed with air should be taken care of first. Once all probes have been removed from the flue gas path, all openings for sampling should be sealed again. Upon completion, it should be checked with the operator if the condition of the system/plant matches their expectations. The checklist of instruments and tools brought to the site should be checked once again before leaving.

## 5.6 Additional information regarding in-situ measurements

Regarding the issues mentioned in the preceding sections, additional helpful information was collected during expert discussions. This information is summarized below.

The results of in-situ measuring campaigns can not be compared to each other since the measuring and environmental conditions can differ considerably from one setup to another. Therefore, a representative and continuous state of operation via the utilisation of regular fuels is to be aimed for. One frequently occurring problem is the automatic adjustment of the furnace during the measurement. In most cases, the adjustment is performed as a result of the residual oxygen content in the exhaust gas of the furnace or via the respective current heat consumption in the heating grid. Such fluctuations in the operation of the furnace also have an impact on the measurement. Worst case is a shutdown of the heating installation due to a lack of heat consumption by the heating grid. Such incidents should be prevented through e.g. prior emptying of the heat storage or ensuring proper consumption of heat. The following bullets indicate circumstances which may be encountered as part of an in-situ measurement.

- An isokinetic sampling is difficult to realise in most cases. The measurement should, however, be performed at a speed proportional to the actual flue gas velocity in order to obtain representative values.
- The extraction of a constant volume flow is not recommended as a dust sampling at a proportional speed can not be guaranteed. The ratio of operational volume flow to constantly extracted volume flow is not always identical.
- The error generated by continuous extraction can be very high.
- Start/stop phases should be avoided at all times.
- Measurements should be performed when the CO concentration and the temperature are constant.
- A dust measurement over a complete combustion period is not recommended since differences in volume flow and concentration will limit comparability.
- A constant operation should be aimed for (approx. 25 min). However, it is challenging to achieve a constant operation for 25 min in-situ (the mode of operation of the furnace will depend on the respective heat demand). Small decentralized biomass furnaces often have a discontinuous mode of operation (with fluctuating fuel supply and/or movement in the combustion chamber).

- The measuring results are influenced by different conditions e.g. the position of the measuring equipment, their handling and installation and the periphery.
- The conclusions that can be drawn from the dust measurements and/or the measuring result only apply to the specific time of measurement and the specific circumstances. This has to be accepted as such. This is due to the following factors and their individual settings:
  - Time of day,
  - Temperature in the heat storage,
  - Heating grid,
  - Boiler control settings
  - The test stand values should be logged in addition.
  - It should be assessed prior to the measurement whether the system was sized correctly.
  - Often hypostoichiometric reactions occur in small-scale furnaces. Therefore, a hydrocarbon measurement (e.g. via FID) is advisable.

#### **Measurement location/site:**

- In case of in-situ measurements no test stand conditions should be assumed (often no upstream stretch and downstream stretch are available). This has to be accepted as such.
- The closing of the flue gas damper may in some cases be problematic.
- The draught limiter could be sealed off. However, a risks assessment should be made ahead of time.
- Pipes may be temporarily sealed with aluminium tape.
- Flow profiles are difficult to record.
- If possible, the core flow should be determined via the max. exhaust gas temperature across the pipe's cross section.
- The flue pipe should be cleaned prior to the measurements (with vacuum cleaner).
- For each measurement photos should be taken which may help with the analysis later on.

- Where applicable, measuring openings may have to be drilled. The owner should be asked for permission ahead of time. The necessary equipment needs to be brought to the site. Reasonable distances between the openings should be granted in accordance with the locality. If necessary the flue gas duct needs to be rearranged.
- The site of the dust probe should be specified first. Subsequently the position of the gas and temperature probes should be set
- If sections of the pipe have to be replaced with a metering installation (as a last resort) the owner should be asked for permission in advance.
- After completion of the measurements measuring openings have to be re-sealed properly and permanently (e.g. by screws).
- Work on system components located on-site should only be performed by the respective specialists or otherwise warranty claims may be lost.
- Furnaces may be fitted into small housing or may be located in the open. This may lead to space-related and climatic restrictions regarding the measuring equipment. In particular, low ambient temperatures may affect the measuring equipment negatively.
- The temperature on-site should correspond to the requirements of the measuring equipment (e.g. risk of freezing or of overheating).
- Access to the required electricity supply for the equipment needs to be clarified in advance. Extension cords, power outlet boxes and in some cases even electric supply (e.g. three-phase current 16A/400V via CEE plug, water-free and dust-free compressed air supply) needs to be brought to the site.

**Measuring duration:**

- The duration of the measurements are site dependent, two sites per day are possible (chimney sweep measurement).
- The more measuring devices are utilised (ELPI, diluter, etc.) the more time will be needed (max. one site per day).
- In case of extensive measurements, an extra day for repositioning or cleaning of the measuring equipment should be included in the planning.

- Before switching on the measuring devices the power supply should be checked in order to avoid triggering the earth fault circuit interrupter when e.g. turning on the pump for the automatic extraction.
  
- Exhaust gas volume flow
  - A vane anemometer is most practical in case of very low flow rates; disadvantage: a 28 mm drill hole is required.
  - The measurement of the flow rate by means of a Prandtl's tube requires a very accurate pressure measurement.
  - In case a vane anemometer is utilised, a regular cleaning (e.g. with acetone) needs to be scheduled.
  - The utilisation of a hot wire anemometer is possible, problems could be caused by the limited depth with which it can be installed
  
- Handling of samples
  - According to VDI 2066 with slight modifications (reduced bake out temperature, bake out of quartz filters for removal of manufacturing residues is done only once and put to dry in the desiccator) similar to the chimney sweep measurement.
  - Where applicable filter cartridges might be necessary.
  - Depending on the dust concentration the duration of the extraction has to be adjusted. Several mg should be present on the filter.

**Miscellaneous:**

- Do not connect gas meter to the intake side.
  
- Particle measurements via ELPI™ are performed with a Dekati diluter; applicability of the results needs to be discussed.
  
- Performing a gas premeasurement (temperature, pressure, speed, steam concentration) can help with the selection of suitable measuring equipment.

## 6 Particle counting methods

All methods known and utilised within the funding programme "Biomass for Energy", for measurements on biomass furnaces, which determine a count instead of a mass of dust particles were summarised under the topic "particle counting".

Depending on the measuring method, the dust-like exhaust gas components can be determined as a total amount per volume or as particle concentrations summarised in size groups. The results may vary depending on the measuring device and the sample preparation (sampling). The different particle measurements performed within the funding programme showed a clear variance in results obtained from different measuring devices. Furthermore, the measurement results obtained via different methods can not be linked to common parameters as is the case for the determination of mass. The mass can be put in relation to the primary kilogram. When detecting a particle count, a distinct and reproducible separation of all units is necessary. However, in case of the exhaust gases relevant here, this still requires considerable research mainly due to the small particle size, their large count and their tendency to agglomerate. Further research is also necessary regarding the sampling methodology. Yet, the methods presented here provide very good indications e.g. for the optimisation of the combustion process. In comparison to gravimetric dust measurement, the particle measurement provides a qualitative image of the current dust emissions without virtually any delay. This can be of significance for e.g. combustion-related optimisation processes. It is important, however, that the measurements are identical with respect to measuring equipment, setup and sampling. A before/after measurement with two different methods is not advisable. In case of the comparative measurements, which were performed as part of the scientific support to the funding programme, the following particle counting methods were utilised and are described in detail in the following sections.

### 6.1 Options for standardisation

The quantity of dust particles determined via particle-counting methods are standardised differently depending on the respective measuring method. The objective of a standardisation is to provide an indication of size that is both understandable and easy to comprehend for the user. The sphere is a very common shape with a clear geometry and easy to describe mathematically. Thus, the diameter of a spherical particle is often used as reference in particle-counting methods. The actual dust particles have a geometry which can be very different from a sphere, especially in case of agglomerated particles such as soot. This requires a different way of standardisation depending on the measuring method.

In Figure 17 the standardisation of a particle of arbitrary geometry is illustrated based on the volume displaced by such a particle.



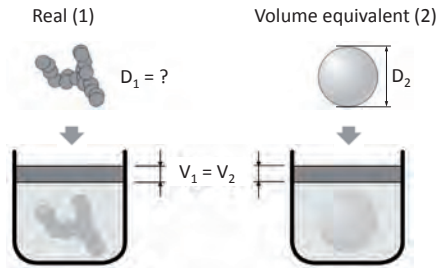


Figure 17: Standardisation of an actual dust particle to a volume-equivalent spherical particle

The volume-equivalent diameter replaces a particle with a complex geometry by a spherical particle of the same volume. Its diameter can then be determined based on equation (45).

$$D = \sqrt[3]{6 \cdot V / \pi} \quad (45)$$

| Symbols | Description  | Unit of measure |
|---------|--|-----------------|
| $D$     | Diameter of the volume-equivalent spherical particle | m               |
| $V$     | volume   | $\text{m}^3$    |

Another option for simplification of the complex geometry of a dust particle is via reference to a spherical particle with identical aerodynamic behaviour. Figure 18 illustrates the relation between a dust particle of complex geometry and an aerodynamically equivalent spherical particle.

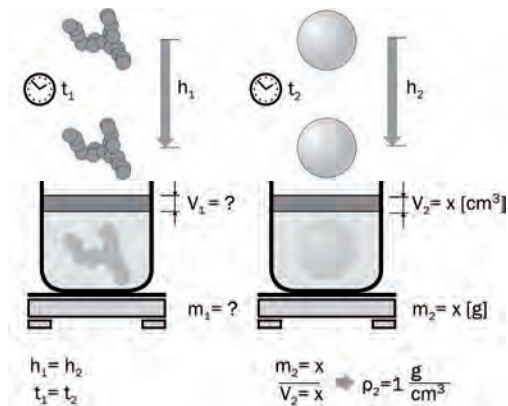


Figure 18: Standardisation of dust particles to spherical particles with aerodynamically equivalent diameter

The spherical particle has a defined density of  $1 \text{ g/cm}^3$  which is equivalent to the density of water under ambient conditions. This spherical particle is equivalent to the actual particle if it features the same settling velocity in still air. The settling velocity of the spherical particle is mathematically known and – among other things – depending on the diameter. The relation between settling velocity and diameter can be described in simplified form via equation (46). This mathematical equation applies under certain conditions, with certain limitations and is meant for a simplified consideration.

$$D = \sqrt[2]{\frac{18 \cdot v \cdot \eta}{\rho_p \cdot g \cdot C_c}} \quad (46)$$

| Symbols  | Definition   | Unit of measure   |
|----------|--|-------------------|
| $D$      | Diameter of the volume-equivalent spherical particle | m                 |
| $v$      | Settling velocity                                    | m/s               |
| $\eta$   | Dynamic viscosity of the medium                      | kg/(m · s)        |
| $\rho_p$ | Spherical density                                    | g/cm <sup>3</sup> |
| $g$      | Gravitational acceleration                           | m/s <sup>2</sup>  |
| $C_c$    | Correction factor                                    |                   |

This is referred to as an aerodynamic equivalent diameter by measuring methods utilising this form of standardisation.

The following standardisation of actual dust particles is very similar to the aerodynamic diameter. This equivalent diameter is in this case referred to as Stokes' equivalent diameter. Here a spherical particle is equivalent to an actual particle if it displays the same settling velocity in still air and also features the same density as the actual particle.

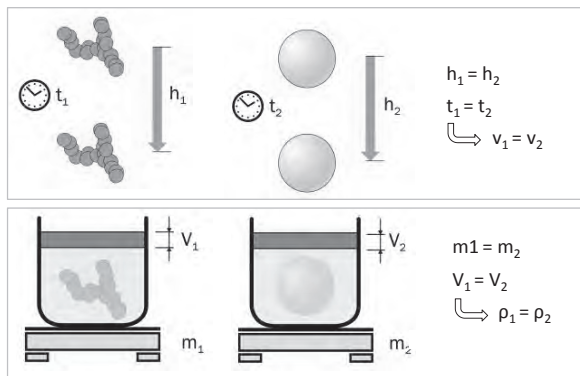


Figure 19: Standardisation of the dust particles to equivalent spherical particles with Stokes diameter

A mathematical relationship can be formulated which – however – requires a detailed description due to its complexity. For a comprehensive explanation the respective literature should be consulted.

Another way of standardising dust particles is by utilising the dust particles' capability to absorb different electrical charges depending on their size. The relationship has been investigated experimentally (Wiedensohler et al., 1986, Wiedensohler, 1988). Figure 20 illustrates this relationship.

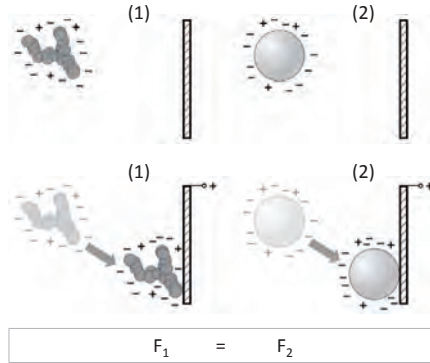


Figure 20: Dust particles with identical electrodynamic mobility

According to this, a dust particle with its actual geometry (1) displays the same electrodynamic mobility as a spherical dust particle (2) if it can be deflected equally severely in an electric field – due to a similar electric charge – as the spherical dust particle. Here, too, it was possible to determine a correlation between electrical mobility and the diameter for spherical particles within certain limits.

## 6.2 Particle mobility spectrometer (SMPS, DMPS)<sup>4</sup>

A particle mobility spectrometer is a measuring device in which particles are classified based on their electrodynamic mobility (c.f. section explaining Figure 20) and subsequently counted. Through the additional detection of the volume flow, the quantity of particles with the same respective electric mobility per unit of volume can be determined.

Particle mobility spectrometers have been available commercially for more than 30 years (Wiedensohler et al., 2012). The measuring devices are mainly used for the measurement of atmospheric aerosols. In the recent past these measuring devices were further developed. They have become more robust with respect to handling and transport and are now being used for measurements which were previously not possible with such devices.

<sup>4</sup> SMPS: Scanning Mobility Particle Sizer, DMPS: Differential Mobility Particle Sizer

Despite significantly simplified handling, the physical and mathematical context as well as the accuracy of the measurement is often not available to the operator. In the following section the experiences gained from measurements with mobility spectrometers as part of the funding programme are presented. This is meant to help future users of such technologies to avoid potential errors resulting from a lack of knowledge and experience.

Particle mobility spectrometers are often a complex unit comprised of various individual components which are also used individually or in other measuring devices. The following four elements are essential for a particle mobility spectrometer.

- PM1 prescreener
- Neutraliser
- Electrostatic classifier (different terms may be used e.g. electrostatic dust filter)
- Optical particle counter

Figure 21 shows a simplified sketch of a Scanning Mobility Particle Sizer (SMPS) like the one used by the DBFZ for comparative measurements as part of the funding programme. The operating principle is described in more detail below, based on the figure.

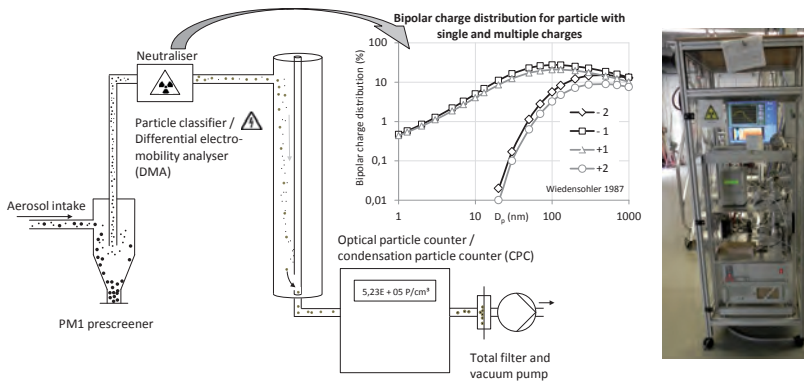


Figure 21: Simplified depiction of a Scanning Mobility Particle Sizer with diagram for the depiction of the charge balance of particles with single and multiple electrical charges (left), SMPS in the laboratory of the DBFZ (right)

The aerosol is directed to the aerosol intake of the measuring device (left) and passes a prescreeener which removes particles that are too large for an accurate determination. This is done via an impactor and/or separator removing particles with an aerodynamic diameter  $> 1 \times 10^6 \text{ m}$ . The reason for removing the fractions  $> \text{PM}_{10}$  is to prevent soiling of the device and to reduce the error generated by larger particles especially when they occur in large quantities. Subsequently, the aerosol passes a neutraliser. The neutraliser establishes a known (typically a universal) electric charge distribution. This can be done in different ways e.g. with a radioactive radiator or with electrically operated devices.

It has been proven with a certain statistical certainty that particles of identical size always feature the same number of electric charge carriers. The relation between electrical charge and size of a particle makes it possible to extract particles of identical electrical mobility from the aerosol. In Figure 21 this is illustrated by a diagram. The diagram shows the frequency with which particles in the range between 1 and 1,000 nm feature a single or double positive or negative electric charge. Spherical particles with diameters in the range between 10 and 100 nm, for example, feature only a single charge significantly more often. Once the aerosol has passed the neutraliser, a separation into different size fractions is performed by a differential mobility classifier (DMGC). Similar to an electrostatic dust filter, particles of differing electrical mobility are extracted from the aerosol by applying different electrical voltages. The particles of each size fraction are then passed through a saturated atmosphere, enlarged through condensation, and counted via an optical particle counter as part of the condensation nucleus counter. The larger the DMGC the more size groups can be determined. Since the SMPS measures one size fraction at a time the determination of a count/size distributions can take several minutes. For aerosols with quickly changing particle count/size distribution over time ( $< 5$  minutes) the measurement via the SMPS utilised here is not suitable. For those cases suitable measures should be taken e.g. the homogenisation of the aerosol or the utilisation of measuring devices with multiple DMGCs and/or CPCs. In the comparative measurements presented in WIEDENSOHLER et al. (2012) a measuring uncertainty of 10 % was determined for mobility spectrometers of different types under controlled laboratory conditions (e.g. standard aerosol), with a size range between 20 nm to 200 nm and after correction for internal losses. In the range of larger particles the uncertainty increased to 30 % while the reason for this is still being investigated.

An SMPS allows for the continuous determination of particle count/size distribution via extractive sampling. Depending on the type of device particles with an equivalent diameter ranging from a few nanometres to several 100 nm can be detected. The SMPS measurement provides the count/size distribution of the measured aerosol as electrodynamic mobility diameter.

## 6.3 Low pressure impactor with electric detector

Another measuring device that is used for measurements on biomass furnaces in the funding programme is the ELPITM (Electrical Low Pressure Impactor), a low pressure impactor with an electrical detector by Dekati®. The operating mode is depicted schematically in Figure 22. The particles are in stages, based on their size, separated and hit the respective plate of the impactor. Before hitting the impactor the particles receive an electrical charge. Once a particle hits an impactor plates a charge exchange takes place. This charge exchange is detected electrically.

The separation of the particles onto the respective impactor plate is in accordance with the natural laws formulated by Stokes. Thus, the separation of the particles depends on the speed of the aerosols in the nozzle of the impactor stage, the nozzle diameter, the particle density, the particle diameter, the viscosity of the air and a correction factor.

Very small particles can be separated and hit the impactor plates if the viscosity of the air is very low. This is achieved via a strong negative pressure (low pressure) that is produced by a vacuum pump inside the ELPITM. Due to the underlying physical principle of this method, when describing the particle separation onto the impactor plates reference is often made to the Stokes diameter (c.f. explanations regarding Figure 19).

The classification made by ELPITM is based on the impactor principle. Depending on the configuration and type of ELPITM up to 14 fractions of 0.006–10 µm can be determined and measurements are performed in real-time. A real-time measurement is possible since the particles contained in the aerosol are electrically charge by a corona. The particles release their charge on the impactor plates in separate stages. The electric impulse is processed by the ELPITM and logged into a .dat file by the software. Additional information is available on the manufacturer's homepage (Dekati, 2012).

### 6.3.1 Structure, measuring principle and functionality of the ELPITM

For various reasons the ELPITM cannot be operated without further equipment. The additional equipment depends on the specific case of application. A vacuum pump has to be utilised in all measurements in order to provide the negative pressure required for the operation of the ELPITM. The negative pressure is manually adjusted via a throttle valve between the ELPITM and the vacuum pump. In case of an older model a computer is required as an additional device for recording data. With the EPLIVI software provided by Dekati® all important functions can be controlled from the computer. The newer model ELPITM+ is equipped with a USB port through which the data can be stored directly onto a USB stick. A dilution system is necessary if the maximum permitted concentration of particles in the aerosol or the permitted aerosol temperature (60 °C) are exceeded (ELPI manual, / pg. 2-1) (DEKATI, 2012). If one of the two conditions is not fulfilled a dilution system needs to be utilised upstream of the ELPITM inlet. The collection plates can only be utilised up to a maximum load (rule of thumb: 1 mg of particles per stage, but depending on the type and shape of the particles) before they have to be cleaned (ELPI-Manual, A1.5.; A-6) (DEKATI, 2012). Therefore, the utilisation of a dilution system makes sense when measurements are performed over a longer period of time. If a dilution system is used this should be done with air of high purity in order to not falsify the result of the measurement.

Figure 22 illustrates the structure and the underlying principle of the ELPI™.

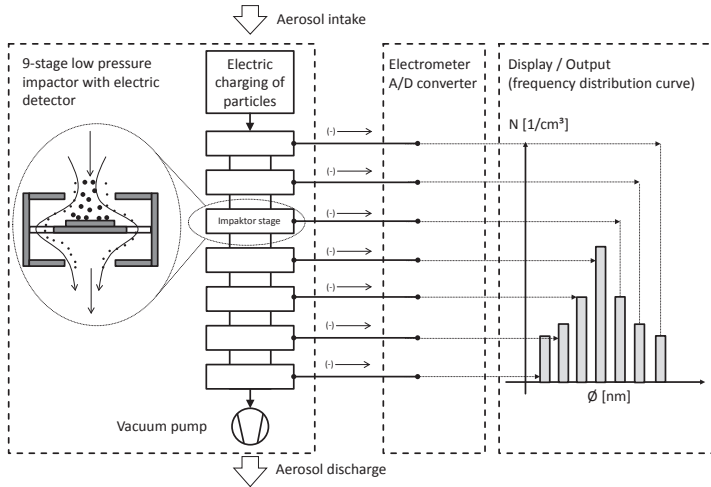


Figure 22: Schematic structure of the ELPI™, source: DBFZ

The sample entering the ELPI™ is guided past a single-pole, positively charged needle in the charger. The particles are electrically charged via a coronary charge. Once the particles in the aerosol have been charged the aerosol passes through a small electrostatic precipitator (ESP) which extracts particles smaller than the effective separating size of the bottom stage via an applied voltage. Larger particles may also be precipitated in the ESP which is compensated for by the ELPIVI software through adjustment of the charger efficiency. Subsequently, the aerosol enters the multi-stage impactor. Here, the particles are classified by their aerodynamic diameter. Depending on the type of construction and configuration of the ELPI™ up to 14 impactor stages connected in series are utilised (ELPI+™). Each stage consists of a gas distributor plate and a collection plate located downstream. The stages are isolated against one another by Teflon rings. At each stage the sample is accelerated through a gas distributor plate and subsequently redirected around the collection plates. Due to the different characteristics of the different particles, individual forces of inertia and friction have an effect on each particle. If the friction generated by the surrounding fluid is of sufficient strength, relative to the inertia forces affecting the particle, the respective particle will follow the flow of the gas around the collection plate. If the force of inertia is too strong, it will be precipitated onto the respective collection plate (c.f. Figure 22, detail at left). The absolute pressure at the ELPI™ outlet is set to be constant at 100 mbar (+/- 5 mbar). Thereby, reproducible flow conditions and particle collection curves are achieved at all stages. The lower the stage number (stage 1 is located at the bottom), the smaller the nozzle opening at the gas distributor plate. Thus, the acceleration increases and the size of the particles which are separated decreases. The increasing negative pressure from stage to stage also ensures that the friction on the fluid is reduced and that the ratio of inertia to friction increases further. This further supports the precipitation of very

fine particles in the bottom stages. Electrometers that are connected to the respective isolated stage register the electric charge dissipating to the collection plate of the stage. The electric charge of each stage is proportional to the particles precipitated and the count of particles can be calculated from the flow of electricity. This value is transmitted via an A/D converter to a computer and is assessed by the provided Dekati® software.

### 6.3.2 Using the ELPI™

When using the ELPI™ it is essential to enter the calibration data determined by Dekati® correctly into the ELPI-VI software. The ELPI™ can be operated with four different settings and the calibration data is dependent on the setting. The type of collection plates can be chosen to either be plates with aluminium foil fixed to it or to be sintered plates. Aluminium foil has the advantage that particles collected at the individual stages can be inspected subsequently (by e.g. REM or EDX). Sintered plates have a ten to twenty times higher collection capacity which allows for measurements over a long period of time. Furthermore, the the ELPI™ can be operated with or without filter stage. The filter stage is installed as last stage below stage one and expands the smallest particle size detected from 0.03 µm to 0.007 µm. Depending on the setting of the ELPI™, the settings in the ELPI-VI software have to be adjusted accordingly. Since the classification of the aerosol in the ELPI™ relies on flow processes that are influenced by minimal changes, the average effective separating size D50% of each stage is individual for each ELPI™. These differences are taken into account by entering the calibration data into the ELPI-VI software. The calibration data can be saved in a .bin file and can be used for each coming application. Values not entered correctly can still be corrected via the software after the measurement. In Table 5, the ELPI-VI parameters are listed which need to be set and have to be modified in case of changes in configuration.

Table 5: Overview of changes of the ELPIVI setting depending on the ELPI™ configuration

| ELPIVI parameters  | To be changed depending on configuration                      |
|--|---|
| Pressure in impactor stages;<br>Retention time in stages | No changes  |
| Average effective separating size<br>D50%                | Different setting for aluminium or sintered collection plates |
| Charger setting  | Different setting for use with/without filter stage           |

The operating range of the electrometers of the ELPI™ can be set differently. The four different ranges are labelled based on the maximum current that can be measured per stage. The lowest current that can be set is 10,000 fA and the range goes up to 400,000 fA. The measuring ranges are distinguished by the following characteristics: The smaller measuring ranges have a lower background noise (they are more inert) while for the larger ranges the opposite is true. The optimal working range depends on the type of aerosol. The higher the concentration of particles in the aerosol, the higher the maximum measurable current should be. In case of lower concentrations, a lower current should be set. Right before each measurement the zero point should be set in order to achieve reproducible and



"clean" values. This procedure helps to compensate for deviations of the electrometers. The setting of the zero point should be performed in the same measuring range in which the subsequent measurement will be performed. Once the zero values measured by the ELPI™ have stabilised and a constant operating temperature has been achieved the zero point is set. Therefore, the ELPI™ should be operated with a lead time of approx. one hour. To flush the ELPI™ with clean air the flushing function can be used ('Flush Pump'). This will move gas through the ELPI™ via a HEPA filter. The sample gas inlet has to be open during the flushing since the flushing gas will exit here. If the inlet remains closed the pump may be damaged. For setting the zero point, the impactor pressure of the last stage has to be set – as for all measurements – to 100 mbar. If the flushing function is active and the continuously measured values of all stages (ELPIVI software) do not change, the setting of the zero point can be started.

## 6.4 Fast Mobility Particle Sizer™ (FMPS), model 3091, TSI company

The FMPS is an aerosol spectrometer that measures particles in the submicron range between 5.6 and 560 nm. The measuring device displays the particle count distribution in 16 channels per decade with a time resolution of one second. The FMPS uses the electric mobility of the particles as measuring principle.

The measuring principle is presented in Figure 23. Before the particles enter the measuring device the particles larger than the measuring range are removed via a cyclone with

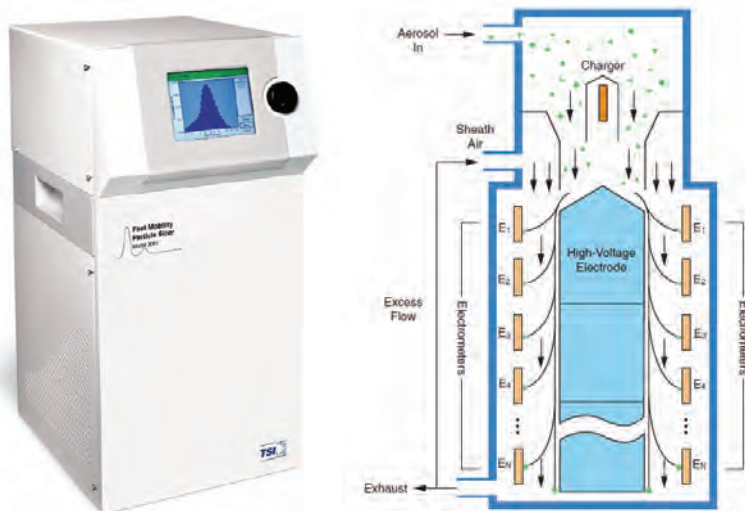


Figure 23: Fast Mobility Particle Sizer™, model 3091, TSI company, photo (to the left) and schematic depiction of the measuring principle (to the right) (TSI, 2006)

screen size (D50) of 1  $\mu\text{m}$ . The remaining particles are then at first brought to a defined charge level via a two stage uni-polar diffusion charger (electrical diffusion charger). Subsequently, the particle stream is guided from top to bottom through an annular space of two cylinders. The inner cylinder is subject to a positive high voltage, so that an electric field exists between the inner and the outer cylinder. Positively charged particles are repelled by the high voltage electrode, overcome the rinsing air stream depending on their electric mobility and eventually hit the exterior cylinder. The exterior cylinder consists of 22 electrically isolated electrode rings (measuring electrodes) which are each connected to a charge amplifier (a so-called electrometer). The charge dissipated by the hitting particles is amplified by the electrometers and subsequently digitalised and read by a microcontroller. The particle size distribution is determined via multiple nested computational algorithms (TSI, 2004). The measuring device is calibrated with monodisperse latex particles whose size has previously been determined with a SMPS. The particle size determines the measurable concentration range. In case of smaller particles, higher concentrations are necessary for a measurement due to the existing (disadvantageous) charge distribution and the stronger influence of the electrometer noise. The opposite is true for larger particles.

### Application

The device is to be operated at ambient temperatures between 0 and 40 °C and a relative humidity between 0 and 90 % (non-condensing). Strong vibrations (in the ground) will lead to faulty measurements and are to be avoided. In contrast to the SMPS, the device can be operated without a handling permit. Due to the maximum particle count concentration of e.g.  $1 \cdot 10^{+06} \text{ \#/cm}^3$ , for particle diameters of approx. 50 nm and maximum aerosol temperature of 52 °C, a gas dilution and gas cooling is mandatory in case of measurements in exhaust gas streams. The following requirements apply when a dilution unit is used:

- Sampling stream 10 L/min
- Aerosol temperature between 10 and 52 °C
- Pressure range between 700 and 1034 mbar
- Tolerable particle concentrations depend on the measuring channel (see handbook)

If it is not possible to set the required sampling stream at the dilution unit, particle-free air can be made available to the measuring device via a HEPA filter connected through a bypass (T-piece). It is recommended to determine the amount of air inserted via a flow meter in order to monitor the realised dilution ratio.

## Devices and materials

- FMPS model 3091
- Cyclone at the aerosol inlet to extract oversized particles
- HEPA filter for offset verification of the electrometers
- PC/laptop for data recording
- Aerosol dilution in accordance with requirements (see section above)
- Flowmeter (where applicable)
- Semi-conducting hose (should be as short as possible to avoid diffusion losses)

### 6.4.1 Measuring process

#### 1. Preparation

As with the other measuring devices described here, it is necessary to select a suitable measuring location and sampling system. Depending on the particle concentration a diluter may have to be installed prior to the measurement.

Before starting the FMPS, the upstream cyclone should be inspected for cleanliness and should be connected to the aerosol inlet of the FMPS. An improved efficiency of separation can be achieved by applying a thin layer of grease to the interior walls and the bottom plate of the cyclone. The FMPS is set up following the manufacturer's instructions, the main plug is connected to the electricity supply and the laptop is connected to the FMPS via data cable. The device must never be operated without cyclone or otherwise a massive soiling of the electrodes and the filters inside the device may occur. The FMPS has a warm-up phase of approx. 10 min. and its readiness for operation is indicated on the display. During the warm-up phase the device software on the laptop can be started. Prior to the actual measurement, a check for soiling of the electrometers (offset) is necessary as well as an adjustment of the settings in the software before data recording should be started. Without software it is not possible to record any data.

#### Check for soiling of electrometer

After the HEPA filter is connected to the aerosol inlet the histogram bars of the individual channels (on the LCD monitor) should, for the most part, be below the yellow line (indicating the lowest measuring range). The dialogue field "Instrument Status" has to be opened for a more accurate assesment of the electrometer offsets. "Instrument Status" can be accessed via the following path:

Menu → View → Instrument Status and/or via <Ctrl><I>

If in the dialogue field the "Electrometer Offsets and RMS..." button is clicked a tabular overview of all electrometer offsets will be displayed. According to manufacturer's specification these should not exceed 10 fA (Th. Krinke: 100 fA). The RMS (Root Mean Square) should be less than 2 fA. If this is the case, an improvement of the measurement result can be achieved by resetting the electrometers to zero (button "Zero the Electrometers..." in the dialogue field "Instrument Status"). In case the electrometer offsets exceed 10 fA and/or the histogram bars are significantly above the yellow line a cleaning is required (c.f. item "Maintenance") due to increased soiling of the measuring device. After a successful reset to zero the HEPA filter can be removed. If almost no particles are measured the reset of the electrometers can be skipped. In the dialogue field "Instrument Status" all other parameters can be controlled too (e.g. the current sampling stream at inlet, which represents the sum of the "Sample Flow" and the "Extraction Flow"). The reading of the electrometer offsets as well as the reset of the electrometers can also be performed via the measuring device itself. To do so, select the item "Menu" and then "User Settings" by turning and pushing the control button. From there, the option "Zero Electrometer" can be selected.

### Adjusting the measuring options prior to the data recording

Before the actual start of the measurement the display settings can be modified. For that a new file must be created via the path "file → new" or via the corresponding symbol in the toolbar. The software requests the storage location and name of the file to be created. Subsequently, various windows with information regarding the on-going measurement are displayed. This display is variable. In addition to the choice of windows to be shown other customisations can be done e.g. modifying the time resolution, the auto-scale function, or the units which are to be displayed (dN or dN/dlogDp).

It is recommended to warm up the FMPS over an extended period of time if the device has been stored at a different temperature prior to the measurement.

## 2. Measurement

To start the data recording select the path "Run → Start Data Collection" or push the button (red dot) in the toolbar. Subsequently, the window "Properties – New Run" appears. Here, the title of the measurement can be entered. Furthermore, duration and start time of the measurement, one-time or continuous measurement as well as the data type can be set. In case of continuous operation it is recommended to set the duration of the measurement to the maximum value of 12 hours and to select "Concentration" as data type. The recording of the measurement data is started when pushing the OK button.

If during the measurement the required operating parameters are not adhered to a warning message is displayed on the software's user interface and on the LCD monitor. It will also be logged in the recorded file. The parameters can once again be checked via "Instrument Status". It can also be checked whether the disruption still continues or was present only short-term, e.g. in case of a sampling stream that was too low or too high due to a disruption in the dilution system or sampling system.

To end the recording select the path "Run → Stop Data Collection" or push the button (black square) in the toolbar.

At the start of the next measurement the same settings will be displayed in the dialogue window. Now only the filename needs to be adjusted.

### 3. After the measurement

Handling the FMPS after the measurements are completed is unproblematic. The device can be turned off at the respective switch and then be disconnected from the power supply by simply pulling the plug.

#### 6.4.2 Analysis

A brief summary of the data can be performed by the program itself. For a detailed analysis, however, it is necessary to export the file to a spreadsheet program e.g. Microsoft Excel.

##### Data summary with the device software

For a data summary the respective file has to be opened with the FMPS measuring software. The entire measurement can either be played back in time-lapse or individual measuring points can be selected and the respective values can be read. The controlling of the time-lapse is performed via the toolbar with typical symbols from e.g. video/DVD players. The time-lapse setting is prompted when pressing the button.

##### Detailed analysis with Excel

For the detailed analysis in Excel, the file has to be exported from the software in TXT format and then be imported into Excel. While a direct export from the measuring software into an XLS file is possible it does not provide good results.

The export is performed via the path "File → Export" while the file is open. The dialogue window "Export Data Options" is displayed. Depending on the objective of the analysis the information to be exported as well as the display type must be selected. It is important to select "Text" under "Output File Type".

The saved file can be imported into Excel by following the path "Data → Import External Data → Get Data" (Excel 2003). The Text Import Wizard is displayed. In steps 1 to 3 (first window) the file origin can be set to Central European (if user is situated in Europe). In the second window, the delimiter selected during export (tab, or similar) has to be selected. In the third window, the delimiters used for the numeric data can be entered via the "More" button. Due to the US-American file origin, decimal separators are displayed as periods and the thousands separators not at all (this may result in incorrect numeric values if a computer with a different country setting than US is used). In the first dialogue window the corresponding number representations may be adjusted. The data is then imported via "Finish" and can be processed for analysis.

## Maintenance

The maintenance intervals of the measuring device components are stated in the respective manual:

- Cleaning the cyclone: 100 operating hours
- Inspection and/or cleaning of the charger needles: 100 operating hours
- Cleaning of the measuring electrodes as well as the high voltage electrode: 500 operating hours
- Replacing the internal filter cartridge: > 6,000 operating hours (or as needed)
- Replacing the pump filter: > 6,000 operating hours (or as needed)

How to perform the respective maintenance is described in detail in the manual. Furthermore, a semi-annual (or as needed) calibration with calibration particles is recommended. This requires a suitable measuring set-up. In case of a measurement with filtered air no or very few particles should be measured in clean condition. If this is not the case it might be an indication for a leakage. Furthermore, the required volume flow rates according to the manual should be checked regularly and any maintenance activity should be logged (BMBF, 2008).

Errors can have the following causes:

- Preheating of device
- Offset too high
- Hose too long

In case of particle concentrations above  $1 \cdot E5 \text{ \#/cm}^3$  (approx.  $1.5 \cdot E3 \text{ \#/channel}$ ), neutralisation will not be sufficient which in turn will increase the measuring error. Also, downstream of electrostatic separators an increased error is to be expected.

## 6.5 Additional measuring techniques

In addition to the methods described a number of other particle counting methods exist. These were, however, not used as part of the funding programme. The following methods

and/or measuring devices could also be used for particle measurements on small-scale biomass furnaces. The measuring methods are listed below and are not further described.

- Aerodynamic particle counter (APC)
- Tapered element oscillating microbalance (TEOM)
- Beta gauge
- Nephelometer
- Aethalometer
- Multi Angle Absorption Photometer (MAAP)
- Low pressure impactors
- Optical particle counter

## 6.6 Sampling, dilution and preparation

In case of particle measurements the sampling often requires more effort than for regular gravimetric dust measurements. The reason for that is the high dust particle concentration in the undiluted combustion exhaust gas which makes a detection with the existing measuring equipment rather unreliable. This can be overcome by reducing the particle concentration via e.g. partial flow dilution. Therefore, the dilution air needs to be free from dust particles and should cause no condensation. The dilution of a partial flow can be realised in different ways and a number of different partial flow dilution systems exist that are utilised for measurements on biomass furnaces. During the measuring workshops that took place within the funding programme different partial flow diluters were used. Depending on the measuring method and the particle size fraction that is to be determined it needs to be decided whether or not an isokinetic sampling can be omitted. The decision to omit isokinetic sampling for the measurement of fractions below an equivalent diameter of  $1\ \mu\text{m}$  at atmospheric conditions should be based on a literature review and on preliminary tests. A measurement of fractions with equivalent diameters larger than  $1\ \mu\text{m}$  should generally be done via isokinetic sampling. The cross section of the sampling probe is chosen according to the measured gas volume flow, the volume flow of the exhaust gas as well as the cross section of the exhaust gas pipe. The calculation is performed based on flow engineering principles in accordance with equation (47).

$$d_{sp} = \sqrt{\frac{\dot{V}_{mg}}{\dot{V}_{exh}} \cdot d_{exh}^2} \quad (47)$$

| <b>Symbols</b>  | <b>Definition</b>                |
|-----------------|----------------------------------|
| $d_{sp}$        | Diameter of the sampling probe   |
| $d_{exh}$       | Diameter of the exhaust gas pipe |
| $\dot{V}_{mg}$  | Measured gas volume flow         |
| $\dot{V}_{exh}$ | Exhaust gas volume flow rate     |

If the volume flow of the exhaust gas is unknown the cross section of the sampling probe can be calculated via the flow rate. If the flow rate is determined via the exhaust gas back-pressure, e.g. by means of the Prandtl's tube, a conversion in accordance with Bernoulli will apply accounting for the density of the exhaust gas. This can be determined based on equation (47) further above.

## 6.7 Challenges with analysis and comparability of results

The way in which the values determined by a particle-counting method are characterised has to be further specified. A suitable way of presenting the results of particle size related measurements is by means of particle count frequency distribution typical for such measurements. For better comparability, the count is standardised to the logarithmic distance of the specific size channels of the device as  $dN/d\log(D_p)$ . This enables the display of large measuring areas in a diagram as well as comparing results from measurements with a wide range of channel boundaries. Thus, this is a first step towards harmonisation of measuring values. Following are further issues to be discussed regarding the presentation of results:

- Presentation of the results a frequency distribution,
- Presentation as total count,
- Presentation as curve of the total count, as curve of the frequency distribution,
- Reference to a unit of volume, standardisation of the unit of volume,
- Reference to 13 % O<sub>2</sub>,
- Presentation as count per MJ with reference to:
  - The heating value of the fuel
  - The heat generated
  - Representation as amount of mass



The particle counting methods include optical particle counters (OPCs) and the devices SMPS, FMPS and ELPI. When comparing the results of differing particle counters the specific equivalent diameter should be considered as well as device-specific differences that may lead to measuring differences at the same specific equivalent diameter.

In case of an optical particle counter the scattered light equivalent diameter is determined, while in case of the FMPS or SMPS the equivalent diameter of electrical mobility is provided. In case of the ELPI the electrical mobility diameter and the aerodynamic diameter are considered. All measuring methods determine the particle count distribution ( $r = 0$ ). Since atmospheric particles and combustion aerosols are not absolutely spherical, but rather irregularly shaped, a comparison of the particle size distributions of differing measuring methods is permissible only if a functional connection  $y = f(x)$  between the differing characteristics is known. Therefore, a very accurate knowledge of the respective particle system is necessary. In ASBACH et al. (2009), HINDS (1999) und SCHMIDT (2001) the connections between different distribution functions as well as some sample calculations and sample diagrams are listed.

Both the SMPS and the FMPS determine the electrical mobility diameter. Therefore, the particle count distributions measured are comparable but comparability may be affected by:

- Higher resolution of the count distribution by SMPS (more channels)
- Higher temporal resolution by FMPS
- Different charger

Due to the higher resolution of the SMPS different channel widths exist. In addition, the measuring range of the SMPS is larger than that of the FMPS. Therefore, the log-standardised representation of the particle count distribution is necessary for a direct comparison. Where applicable, the measured values of the SMPS that are located outside the measurable range of the FMPS ( $d > 560$  nm) will have to be omitted.

Due to the higher temporal resolution of the FMPS changes in the particle spectrum can be determined in real-time and very accurately. The SMPS scans every channel whereas the FMPS utilises various calculation algorithms and distributes the information from 22 sensors to a total of 32 channels. In case of extreme particle size distributions, e.g. mono-disperse particle size distribution, distribution densities are calculate featuring a (near) log-standard distribution.

## 7 Harmonisation of methods

### 7.1 Motivation

The methods listed in the preceding chapters illustrate that the assessment of particulate matter emissions generated by the combustion of solid biofuels in small-scale furnaces can be performed in very different ways. However, as service and support to the projects of the funding programme “Biomass energy use” a minimum amount of standardisation was aimed for. Thus, for the three essential objectives:

- Dust mass control in practice (chimney sweep),
- gravimetric dust mass detection in science, and
- dust sampling for toxicological investigations,

typical sources of errors were identified. Thereafter, indications for standardised methods were provided, that list methods for error minimisation as well as provide assistance with the necessary documentation.

### 7.2 Measurement site/location

The bullets listed below indicate important aspects that can be relevant when selecting a suitable sampling site. As part of a future update to this compilation of measuring methods, the following issues will have to be further discussed, put in relation to literature and specified further based on selective measurements.

- Distance between sampling sites and from built-in components as well as bent pipes, etc.
- Measuring conditions
- Exhaust gas temperatures
- Flow profiles
- Conditions causing turbulence (Reynold's number)
- Typical flow rates and calculation of the optimal cross section in dependence on the combustion efficiency and the excess air value
- Determination of the standard volume flow (gas equation)

- Determination of the density of the exhaust gas via the concentration of the exhaust gas components and the molar mass of the respective component
- Conversion of concentrations in loads

Figure 24 shows the configuration of an exhaust gas measuring installation as it is used at the DBFZ which allows multiple sampling and multiple measurements simultaneously. This also allows measurements which can monitor the performance of the furnace. In particular, this can be the determination of the negative pressure in the chimney flue as well as the exhaust gas temperature. In addition, this measuring installation is suitable for the retrieval of gravimetric dust samples. When designing the installation and deciding its dimensions the flow rate played a key role. Especially for the dust measurements it is important to achieve flow rates of significantly more than one metre per second. To guarantee this even for very small furnaces, a diameter of only 80 mm in the area of the sampling opening was selected.

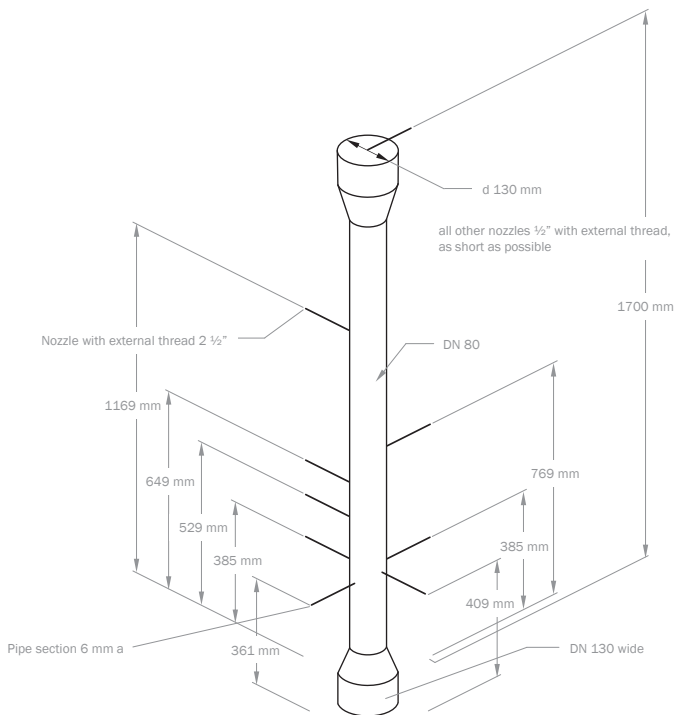


Figure 24: Standard exhaust gas section for boilers and individual fireplaces/stoves at the DBFZ

## References

1. BImSchV (2010): Erste Verordnung zur Durchführung des Bundes-Immissionsschutzgesetzes (Verordnung über kleine und mittlere Feuerungsanlagen (in Kraft getr. am 22.03.2010) §§ 1 ff).
- Asbach, C.; Kaminski, H.; Fissan, H.; Monz, C.; Dahmann, D.; Mülhopt, S.; Paur, H. R.; Kiesling, H. J.; Herrmann, F.; Voetz, M.; Kuhlbusch, T. A. J. (2009): Comparison of four mobility particle sizers with different time resolution for stationary exposure measurements. In: *J. Nanopart. Res.* 11 (2009), S. 1593–1609, doi: 10.1007/s11051-009-9679-x.
- Bauer, R.; Göllles, M.; Brunner, T.; Dourdoumas, N.; Obernberger, I. (2007): Was messen Temperatursensoren in einer Biomasse-Feuerung wirklich? (What is Really Measured by Temperature Sensors in a Biomass Furnace?). In: *Automatisierungstechnik* 55, p. 600–607.
- Bayerisches Landesamt für Umwelt (LFU Bayern) (2014): Effiziente Druckluftnutzung. URL: <http://www.lfu.bayern.de/energie/druckluftcheck/doc/druckluftnutzung.pdf> (04.02.2014).
- Birmili, W.; Brüggemann, E.; Gnauk, T.; Herrmann, H.; Iinuma, Y.; Müller, K.; Poulain, L.; Weinhold, K.; Wiedensohler, A.; Sächsisches Staatsministerium für Umwelt und Landwirtschaft (2008): Einfluss kleiner Holzfeuerungen auf die Immissionsituation – Teil Immissionsmessung. Leipzig.
- BS ISO 9096:2003-02-24 (2003): Emissionen aus stationären Quellen – Bestimmung der Konzentration und der Massenflussrate von Partikeln in geführten Quellen – Manuelle gravimetrische Methode.
- Bundesamt für Umwelt (BAFU) (2009): Information zum Inverkehrbringen von Kohle- und Holzfeuerungen nach Artikel 20 der Luftreinhalteverordnung.
- ChemgaPedia (2012): Detektoren in der Gaschromatographie – Flammionisationsdetektor (FID), URL: [http://www.chemgapedia.de/vsengine/vlu/vsc/de/ch/3/anc/croma/gc\\_detektoren.vlu/Page/vsc/de/ch/3/anc/croma/gc/detektoren/flamme/vid1m75te1100.vscml.html](http://www.chemgapedia.de/vsengine/vlu/vsc/de/ch/3/anc/croma/gc_detektoren.vlu/Page/vsc/de/ch/3/anc/croma/gc/detektoren/flamme/vid1m75te1100.vscml.html) (10.10.2012).
- Danielsen, P.H.; Møller, P.; Jensen, K.A.; Sharma, A.K.; Wallin, H.; Bossi, R.; Autrup, H.; Mølhave, L.; Ravanat, J.L.; Briedé, J.J.; de Kok, T.M.; Loft, S. (2011): Oxidative Stress, DNA Damage, and Inflammation Induced by Ambient Air and Wood Smoke Particulate Matter in Human A549 and THP-1 Cell Lines. In: *Chem. Res. Toxicol.* 24 (2011), p. 168–184.
- Dekati (2012): Dekati Ltd. Osuusmyllynkatu 13, FIN-33700 Tampere, Finland: <http://dekati.com>.
- DIN 1343:1990-01 (1990): Referenzzustand, Normzustand, Normvolumen; Begriffe und Werte.
- DIN 4702-1: 1990-03 (2003): Heizkessel: Begriffe, Anforderungen, Prüfung, Kennzeichnung.

- DIN EN 12619 (2011): Emissionen aus stationären Quellen – Bestimmung der Massenkonzentration des gesamten gasförmigen organisch gebundenen Kohlenstoffs – Kontinuierliches Verfahren mit dem Flammenionisationsdetektor; Deutsche Fassung prEN 12619:2011.
- DIN EN 13240: 2005-10 (2010): Raumheizer für feste Brennstoffe Anforderungen und Prüfungen.
- DIN EN 13284-1:2002-04 (2004): Emissionen aus stationären Quellen – Ermittlung der Staubmassenkonzentration bei geringen Staubkonzentrationen – Teil 1: Manuelles gravimetrisches Verfahren; Deutsche Fassung EN 13284-1:2001.
- DIN EN 14785: 2006-09 (2009): Raumheizer zur Verfeuerung von Holzpellets – Anforderungen und Prüfverfahren.
- DIN EN 303-3: 2004-10 (2010): Zentralheizkessel für gasförmige Brennstoffe – Zusammenbau aus Kessel und Gebläsebrenner.
- DIN EN 303-5:1999 (1999): Heizkessel – Teil 5: Heizkessel für feste Brennstoffe, hand- und automatisch beschickte Feuerungen, Nenn-Wärmeleistung bis 300 kW Begriffe, Anforderungen, Prüfungen und Kennzeichnung.
- DIN EN 481 (1993): Arbeitsplatzatmosphäre-Festlegung der Teilchengrößenverteilung zur Messungsluftgetragener Partikel. Berlin.
- Dockery, D.W.; Pope III, C.A.; Xu, X.; Spengler, J. D.; Ware, J. H.; Fay, M. E.; Ferris, B. G.; Speizer, F. E. (1993): An association between air pollution and mortality in six U.S. cities. In: The new England Journal of Medicine, Volume 329, Number 24.
- Ellner-Schuberth, F.; Hartmann, H.; Turowski, P.; Roßmann, P. (2010): Partikelemissionen aus Kleinf Feuerungen für Holz und Ansätze für Minderungsmaßnahmen. Berichte aus dem TFZ – 22, Straubing.
- EN 303-5 (2012): Heizkessel für feste Brennstoffe, manuell und automatisch beschickte Feuerungen, Nennwärmeleistung bis 500 kW - Begriffe, Anforderungen, Prüfungen und Kennzeichnungen; Deutsche Fassung EN 303-5:2012. Ed. by DIN Deutsches Institut für Normung. Berlin: Beuth Verlag.
- Europäische Kommission (2011): „Bericht der Kommission an das Europäische Parlament und den Rat über die Fortschritte bei der Erfüllung der Kyoto-Ziele“. 07-Okt-2011.
- Hasselbarth, Werner; Steiger, Thomas (ed.) (2004): Metrologie in der Chemie an der BAM – Aktivitäten im Rahmen der Meterkonvention (CCQM). Forschungsbericht 267. Berlin 2004, URL: [http://www.bam.de/de/service/publikationen/publikationen\\_medien/fb267\\_vt.pdf](http://www.bam.de/de/service/publikationen/publikationen_medien/fb267_vt.pdf) (10.09.2014)
- Hausmann, A.; Sächsisches Landesamt für Umwelt, Landwirtschaft und Geologie (Hrsg.) (2010): Holzkleinf Feuerungsanlagen in Sachsen, Schriftenreihe, Heft 17/2010. Dresden.
- Hinds, W. C. (1999): Aerosol technology: properties, behavior, and measurement of airborne particles. 2. Auflage. New York: John Wiley & Sons.
- Hoffmann, J. (1996): Messen nichtelektrischer Größen. Düsseldorf. VDI-Verlag GmbH.

- linuma, Y.; Böge, O.; Gräfe, R.; Herrmann, H. (2010): Methyl-Nitrocatechols: Atmospheric Tracer Compounds for Biomass Burning Secondary Organic Aerosols. In: *Environ. Sci. Technol.* 44 (2010), doi:10.1021/es102938a.
- Industrieverband Haus-, Heiz- und Küchentechnik e.V. (HKI) (2012): Feuerstättendatenbank zum Nachweis der gesetzlichen Anforderungen an häusliche Feuerstätten für feste Brennstoffe Grundlagen und Anforderungen für den Eintrag.
- Jalava, I. P.; Salonen, O. R.; Nuutinen, K.; Pennanen, S. A.; Happonen, S. A.; Tissari, J.; Frey, A.; Hillamo, R.; Jokiniemi, J.; Hirvonen, M.-R. (2010): Effect of combustion condition on cytotoxic and inflammatory activity of residential wood combustion particles. In: *Atmospheric Environment* 44 (2010), p. 1691–1698.
- Jokiniemi, J.; Hytöntö, K.; Tissari, J.; Obernberger, I.; Brunner, T.; Bärnthaler, G.; Friesenbichler, J.; Salonen, R.O.; Hirvonen, M.R.; Jalava, P.; Pennanen, A.; Happonen, M.; Vallius, M.; Markkanen, P.; Hartmann, H.; Turowski, P.; Roßmann, P.; Ellner-Schubert, F.; Boman, C.; Pettersson, E.; Wiinikka, H.; Hillamo, R.; Saarmio, K.; Frey, A.; Saarikoski, S.; Timonen, H.; Teinilä, K.; Aurela, M.; Sillanpää, M.; Bellmann, B.; Sandström, T.; Sehlstedt, M.; Sörberg, B. (2008): Biomass combustion in residential heating: particulate measurements, sampling, and physicochemical and toxicological characterization – ISSN 0786-4728.
- Kaltschmitt, M.; Hartmann, H.; Hofbauer, H. (ed.) (2016): *Energie aus Biomasse: Grundlagen, Techniken und Verfahren*. Berlin: Springer – ISBN 978-3-662-47437-2
- Konsortium des Projekts NanoCare, gefördert vom Bundesministerium für Bildung und Forschung (Hrsg) (2008): *Vorgehensweise bei Partikelmessungen mit dem Fast Mobility Particle Sizer (TSI Model 3091)*. Standardarbeitsanweisung SAA APM 06, Version 1.0. 14.07.2008.
- Lenz, V.; Thrän, D.; Hartmann, H.; Turowski, P.; Ellner-Schubert, F.; Gerth, J. (2010): *Bewertung und Minderung von Feinstaubemissionen aus häuslichen Holzfeuerungsanlagen*, DBFZ Report Nr. 1. Leipzig.
- Lindner, H.; Siebke, W.; Simon, G.; Wuttke, W. (2006): *Physik für Ingenieure*. Leipzig.
- Löffler, F. (1988): *Staubabscheiden*. Stuttgart: Thieme.
- Mudgal, S.; Turbé, A.; Bio Intelligence Service (ed.) (2009): *Preparatory Studies for Eco-design Requirements of EuPs (II) Lot 15 Solid fuel small combustion installations Task 1: Scope and Definition*. France.
- Musiol, F.; Nieder, T.; Rütger, T.; Memmler, M.; Rother, S.; Schneider, S.; Bundesministerium für Umwelt, Naturschutz und Reaktorsicherheit Referat KI III 1 (ed.) (2012): *Erneuerbare Energien 2011, Vorläufige Angaben*. (08.03.2012).
- Orthen, B.; Rappolder, M.; Zimmer, R. (2007); Bundesanstalt für Arbeitsschutz und Arbeitsmedizin (BAuA); Bundesinstitut für Risikobewertung (BfR); Umweltbundesamt (UBA): *Nanotechnologie: Gesundheits- und Umweltrisiken von Nanomaterialien – Forschungsstrategie*.
- Pfeiffer, D., Schröder, T. (2011): Optimization of biomass energy use – measurement and valuation methods for a purposeful development of methods for measuring at biomass combustions as an example, 4. IBC Leipzig, International Biomass Conference – 24./25.05.2011. Leipzig.

- Rödelsperger, K.; Brückel, B.; Podhorsky, S.; Schneider, J.; Bundesanstalt für Arbeitsschutz und Arbeitsmedizin (2009): Charakterisierung von ultrafeinen Partikeln für den Arbeitsschutz – Teil 2. Dortmund/Berlin/Dresden.
- Ruppelt, E. (ed.) (2003): Druckluft-Handbuch, 4. Auflage, (Vulkan-Verlag) Essen, ISBN 3-8027-2548-4.
- Schmidt, E. (2001): Kurz gefasste Grundlagen der Partikelcharakterisierung und der Partikelabscheidung. Aachen: Shaker.
- Schröder T., Lenz V. (2011): Aerosols from domestic biomass heating, characterization and toxicity – critical pathways towards sustainability of biomass based heating. Proceedings of the satellite workshop within the European Aerosol Conference, DBFZ Conference Report No. 1, Manchester.
- Schröder, T. (2011): Vergleichende Partikelmessung im Abgas von Kleinfeuerungsanlagen für Holzpellets, 9. 25. Aerosol Technologie Seminar – Grundlagen, Anwendung, Zukunft – 12.–13.09.2011, Karlsruhe.
- Schröder, T. (2012): Comparison and harmonization of dust measurement methods: outcomes of a national network project (SuB). Expert Workshop: Dust measuring procedures for small biomass furnaces. Berlin.
- Schröder, T., Helbig, K., Stahl, E., Groll, A. (2011): Measurement Procedures for Particulate Emissions from Residential Wood Combustions, 6. Aerosols from domestic biomass heating, characterization and toxicity - Critical pathways towards sustainability of biomass based heating, Satellite workshop within the European Aerosol Conference EAC 2011, September 3rd and 4th 2011, Manchester.
- Seidler, A. (2011): Entwicklung einer Prüfprozedur für das Ökolabeling von Kleinfeuerungsanlagen für biogene Festbrennstoffe an einem Versuchsstand mit Abgasvollstromverdünner. Leipzig, HTWK, Fak. ME, Masterarbeit.
- Sippula, O.; Hytönen, K.; Tissari, J.; Raunemaa, T.; Jokiniemi, J. (2006): Effect of Wood Fuel on the Emissions from a Top-Feed Pellet Stove. In: Energy & Fuels, 21, 2007, p. 1151–1160.
- Skoog, D.A.; Leary, J.J. (1996): Instrumentelle Analytik : Grundlagen, Geräte, Anwendungen. Heidelberg: Springer.
- Staab, J. (1994): Industrielle Gasanalyse. Wien: R. Oldenbourg Verlag München.
- Stiftung Warentest (ed.) (2011): Wenige feuern gut. In: Stiftung Warentest – test 11/2011, Berlin. p. 58–65.
- Struschka, M.; Kilgus, D.; Springmann, M.; Baumbach, G.; Umweltbundesamt (2008): Effiziente Bereitstellung aktueller Emissionsdaten für die Luftreinhaltung. Dessau, (Forschungsbericht 205 42 322, UBA-FB 001217 Texte 44/08, 11. 2008) – ISSN 1862-4804.
- TESTA – Analytische Messsysteme GmbH (2012): Kohlenwasserstoffe richtig messen mit dem FID; URL: <http://www.testa-gmbh.de/index.php?id=richtig> (10.10.2012).
- TSI Factsheet FMPS.

- TSI Incorporated (2004): Model 3091 Fast Mobility Particle Sizer™ Spectrometer, Preliminary information; Shoreview, USA, P/N 2980290.
- TSI Incorporated (2006): Model 3091 Fast Mobility Particle Sizer™ Spectrometer. Operation and Service Manual; Shoreview, USA, P/N 1980520, Revision B.
- Umweltbundesamt (ed.) (2008): Luftreinhaltung Leitfaden zur Emissionsüberwachung. Durchgeführt vom TÜV Süd Industrie Service GmbH, München, ISSN 1862-4804.
- United Nations (1997): Glossary of Environment Statistics, Studies in Methods. Series F 67, United Nations: New York.
- United States Environmental Protection Agency (US-EPA) (2014): Indoor Air – Volatile Organic Compounds (VOCs) – Technical Overview. URL: <http://www.epa.gov/iaq/voc2.html> (04.02.2014).
- United States Environmental Protection Agency (US-EPA) (2014): Particulate Matter – Basic Information. URL: <http://www.epa.gov/pm/basic.html> (04.02.2014).
- VDI 2066, Sheet 1 (1975): Particulate matter measurement - Dust measurement in flowing gases - Gravimetric determination of dust load - Overview. Pub.: Association of German Engineers. Berlin: Beuth Verlag [Beuth Publishers].
- VDI 2066, Sheet 1 (2006): Particulate matter measurement - Dust measurement in flowing gases - Gravimetric determination of dust load - Overview. Pub.: Association of German Engineers. Berlin: Beuth Verlag [Beuth Publishers].
- Wiedensohler, A. (1988): An approximation of the bipolar charge distribution for particles in the submicron size range. In: *Journal of Aerosol Science* Vol. 19, No. 3, p. 387–389.
- Wiedensohler, A.; Birmili, W.; Nowak, A.; Sonntag, A.; Weinhold, K.; Merkel, M.; Wehner, B.; Tuch, T.; Pfeifer, S.; Fiebig, M.; Fjåraa, A. M.; Asmi, E.; Sellegri, K.; Depuy, R.; Venzac, H.; Villani, P.; Laj, P.; Aalto, P.; Ogren, J. A.; Swietlicki, E.; Williams, P.; Roldin, P.; Quincey, P.; Hüglin, C.; Fierz-Schmidhauser, R.; Gysel, M.; Weingartner, E.; Riccobono, F.; Santos, S.; Grüning, C.; Faloon, K.; Beddows, D.; Harrison, R.; Monahan, C.; Jennings, S. G.; O'Dowd, C. D.; Marinoni, A.; Horn, H.-G.; Keck, L.; Jiang, J.; Scheckman, J.; McMurry, P. H.; Deng, Z.; Zhao, C. S.; Moerman, M.; Henzing, B.; de Leeuw, G.; Löschau, G.; Bastian, S. (2012): Particle mobility size spectrometers: harmonization of technical standards and data structure to facilitate high quality long-term observations of atmospheric particle number size distributions. In: *Atmospheric Measurement Techniques* 5, p. 657–685.
- Wiedensohler, A.; Lütkeemeier, E.; Feldpausch, M.; Helsper, C. (1986): Investigation of the bipolar charge distribution at various gas conditions. In: *Journal of aerosol Science* Vol. 17 No. 3, p. 413–416.



# Appendix

## 1

### **Instructions for the determination of dust content in exhaust gas as done by the TFZ**

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## 1.1 Procedure

The instructions provided in the following paragraphs describe the procedure for determining total dust concentrations in flowing exhaust gas. The procedure is based on the requirements of VDI 2066 Sheet 1. With respect to dust emissions the instructions are intended to increase the comparability of measurement results provided by different institutions. To state one example: So far the post-treatment is performed at different temperature levels leading to a higher or lower total dust emission. By means of these instructions a harmonisation of the determination methods for total dust emissions is intended.

## 1.2 Field of application

The here described methods can be used to determine the concentration of total dust in automatically or manually fed furnaces.

## 1.3 Measuring principle

A measurement consists of different steps. At first, an isokinetic (at identical speed) partial flow is retrieved from the centre of the exhaust gas flow. The partial flow passes through a setup of suitable filter media (typically quartz fibre filters, potentially with stuffed filter sleeve upstream) to separate the dust carried along. Afterwards the gas of the partial flow is dried in a drying tower and the gas volume retrieved is determined.

In accordance with the following equation, the dust mass located on the filter is put in relation to the partial flow volume extracted.

$$c_{total\ dust} = \frac{(m_{filter,out} - m_{filter,in})}{V_{partial\ flow,dr,N}} \quad (A1.1)$$

| <b>Symbols</b>           | <b>Definition</b>  | <b>Unit of measure</b>  |
|--------------------------|--|-------------------------|
| $c_{total\ dust}$        | Concentration of total dust at standard temperature and pressure | mg/m <sup>3</sup> (STP) |
| $m_{filter,out}$         | Mass of filter upon weighing-out                                 | mg                      |
| $m_{filter,in}$          | Mass of filter upon weighing-in                                  | mg                      |
| $V_{partial\ flow,dr,N}$ | Gas volume extracted at standard condition dry                   | m <sup>3</sup> (STP)    |

It is assumed that the average dust concentration in the partial flow is equivalent to the average dust concentration of the exhaust gas.

## 1.4 Measuring devices and aids

### 1.4.1 Measuring devices

The following measuring devices (table 1) are required for the measurement:

Table 1: Measuring devices for the determination of total dust

| Measuring device  | Intended use/comments   |
|---|---|
| Thermocouple (e.g. type K)  | To determine the exhaust gas temperature at the measurement site as well as to determine the ambient air temperature for a conversion of the extracted volume flow to a standard volume.  |
| Measuring device for the determination of moisture content in the exhaust gas | Suitable measuring methods have to be applied. Both continuously working measuring devices, e.g. FTIR, as well as a gravimetric determination of the moisture can be utilised. The share of water vapour can also be calculated from a material balance (combustion calculation).   |
| Gas analyser for the determination of oxygen content in the exhaust gas       | Necessary for the conversion of the dust load to a reference parameter at 13 % O <sub>2</sub>   |
| Gas analyser for the determination of CO <sub>2</sub> content                 | If measurements are performed in diluted exhaust gas, the dilution ratio in the undiluted and the diluted exhaust gas is required. The dilution ratio can be determined by means of CO <sub>2</sub> measurements.   |
| Measuring device for the determination of exhaust gas speed                   | The determination of exhaust gas backpressure is performed via a standardised Prandtl's tube. However, the latter tends to clog in case of high dust loads. Therefore, a vane flow sensor (temperatures up to 500 °C) or a vortex flow sensor (temperatures up to a maximum of 180 °C) can be utilised. In addition to the temperature at the measurement site, the measuring range of the device has to be taken into account. |
| Measuring device for the determination of static pressure                     | Necessary for the determination of the pressure at the gas meter for a conversion of the extracted volume to normal conditions  |
| Automatically controlled partial flow extraction device                       | With drying tower, pump, gas meter and dust sampling probe consisting of nozzle, bent tube, filter head device with heating sleeve and electrically heated extraction pipe  |
| Micro scale   | To determine the mass of the filters and/or stuffed filter sleeves before and after they have collected a load with an accuracy of 1 mg. The weighing should be performed at ambient conditions that are as constant as possible (for instance in a climatic chamber and/or in a climatic cabinet).   |

### 1.4.2 Materials

For a determination of total dust, the following materials are additionally required:

- Baked-out planar filters type Munktell MK 360 (preferably quartz fibre) with a suitable diameter (e.g. 45 mm for dust sampling holder of the Paul Gothe company),
- Filter sleeve stuffed with fine-fibrous quartz wadding, in case of high dust loads,
- Acetone for rinsing of the sampling probe,
- Distilled water for rinsing of the sampling probe,
- Aluminium foil for the packaging of filters for further analysis (not always necessary),
- Zip bag for shipping of sample for further analysis (not always necessary),
- Hygrometers, thermometers, barometers.

### 1.4.3 Tools

The following tools are needed for filter handling:

- Petri dishes,
- Tweezers,
- Brush/paintbrush,
- Gloves (heat-resistant) for installation and de-installation of the dust sampling probe,
- Laboratory gloves,
- Large beaker,
- Rinsing container,
- Water-proof pen for potential labelling of samples.

## 1.5 Selection of correct measuring location

Depending on the size of the furnace, a corresponding exhaust gas volume flow has to be extracted from the furnace. To ensure a reliable velocity measurement, velocities of at least 1 m/s are necessary. This can be achieved via selecting a suitable internal pipe diameter. For example the recommended inner diameter of a pipe should be approx. 60 mm. Compared to an oven, a boiler with a heat output of about 30 kW should feature an inner diameter of approx. 80 mm. If the hot exhaust gas is also diluted, a pipe diameter of 150 mm can be selected for the diluted exhaust gas.

For the measurement of total dust concentration an evenly developed flow at the measuring site has to be ensured. Therefore the inlet and outlet zones required by the speed measuring devices have to be adhered to as indicated by the manufacturer. The following inlet and outlet zones after a narrowing cross section are recommended (Table 2).

**Table 2: Recommended upstream and downstream stretches for selected measuring devices after a narrowing of the cross section**

| Measuring device    | Upstream stretch/downstream stretch  |
|---------------------|--|
| Dust sampling probe | 5 x inside pipe diameter, respectively   |
| Prandtl's tube      | 5 x inside pipe diameter, respectively   |
| Vane flow sensor    | Inlet zone with 15 x inside pipe diameter; outlet zone with 5 x inside pipe diameter |
| Vortex flow sensor  | Inlet zone with 15 x inside pipe diameter; outlet zone with 5 x inside pipe diameter |

The listed inlet zones have to be wider if the measuring device is to be installed after an enlargement of the cross section or downstream of a redirection. In addition to the inlet zones, the centric installation position of the respective measuring device is quite important. Due to its selective measurement, the Prandtl's tube can be placed at approx. half the inner diameter of the pipe. The vane flow sensor must not be pulled out by more than half the diameter after complete immersion in the exhaust gas pipe. Otherwise the actual vane will not be centred in the exhaust gas pipe.

Corresponding thermocouples have to be placed near the measuring devices in order to be able to make statements regarding the sampling temperature.

## 1.6 Performing the measurement

### 1.6.1 Preparation of the measurement

#### 1.6.1.1 Preparation of Petri dishes for storage of planar filters

Clean Petri dishes are to be used for the storage of planar filters without and with load. The Petri dishes have to be cleaned in a laboratory dish washer and subsequently dried. Ideally, the sample numbers are engraved in both the lid and the bottom. Otherwise, an unambiguous labelling of the samples must be ensured.

#### 1.6.1.2 Preparation of planar filters

Planar filters are retrieved from the package with tweezers and placed in a labelled Petri dishes. Afterwards, the planar filters are dried in the open Petri dishes at 120 °C for an hour before storing them for at least 8 hours in the desiccator. The desiccator has to be in the same room in which the weighing itself is going to be performed. At this point a deviation is made from VDI 2066-1 which mandates a temperature of 180 °C (VDI 2066-1, 2006). The climatic conditions (room temperature, humidity and air pressure) must be noted once during the weighing. After individual weighing, the planar filters are stored dust-protected in a closed Petri dishes until use.

#### 1.6.1.3 Preparation of stuffed filter sleeves

In case of high dust concentrations, especially if manually fed individual fireplaces/ stoves are tested (e.g. an oven) a planar filter is insufficient. Prior to each use, the numbered (engraved) filter sleeves must be cleaned in an ultrasound bath, rinsed with distilled water, and subsequently dried. For further preparation of filter sleeves, laboratory gloves must be worn. The filter sleeves are stuffed with approx. 1.8 g of fine-fibrous quartz wadding. The stuffing of the sleeves is performed under an extractor hood. Subsequently, the stuffed filter sleeves are to be heated for one hour at 120 °C. Prior to weighing stuffed filter sleeves without a load, they must be stored for at least 8 hours in a desiccator in the same room in which the weighing itself is going to take place. The climatic conditions (room temperature, humidity and air pressure) must be recorded once during the weighing. After the single weighing, the filter sleeves are sorted into instrument trays and stored until use. The instrument tray with lid is labelled with a note "weighed".

#### 1.6.1.4 Preparation of planar filter holders

The empty planar filter holders are to undergo a visual inspection prior to their use. If fibre residues and/or soiling are visible they are to be removed with a brush. In case of heavy

soiling, the planar filter holders are cleaned in the ultrasound bath (tap water). Subsequently, they are rinsed with distilled water and dried. The planar filter holders themselves are not weighed in.

#### 1.6.1.5 Preparation of the dust sampling probe



Figure 1: Filter holder for planar filters and filter sleeves by the Paul Gothe company; Image: TFZ

First, the filter holder, Figure 1 (to the right), must be equipped with a planar filter without load which has to be fixed in position with a clamping ring. The stuffed filter sleeve also has to be installed in case of high dust loads. Attention needs to be paid to the necessary sealing rings. Subsequently, the filter holder is screwed onto the clean probe. The nozzle diameter has to be adjusted to the speed in the measuring installation. In this, the volume flow extracted should amount to between 0.4 and 2.2 m<sup>3</sup> (STP)/h. In case of a low

dust load of the exhaust gas, a larger extraction volume flow and therefore a larger nozzle diameter can be selected for a higher accuracy of measurement.

#### 1.6.1.6 Preparation of the automatic isokinetic dust extraction

An extraction system is depicted, for example, in Figure 2. At the beginning of a measurement, the drying towers have to be filled with fresh desiccant beads. The first drying tower remains empty and serves as condensate trap. The communication between the extraction system and the measurement data acquisition system has to be checked.

After connecting the dust probe with the exhaust gas hose to the extraction unit, a leak test has to be performed. For this, the nozzle opening is sealed airtight, e.g. with a plug, and a negative pressure of up to 0.4 bar is generated via pump operation. The leakage test is considered successful if a pressure loss of less than 0.02 bar is observed over a period of 30 seconds. If the leakage test fails, the errors have to be remedied and the test has to be repeated. By means of the leakage test, the intake of false air is to be avoided which otherwise would lead to false measuring results.

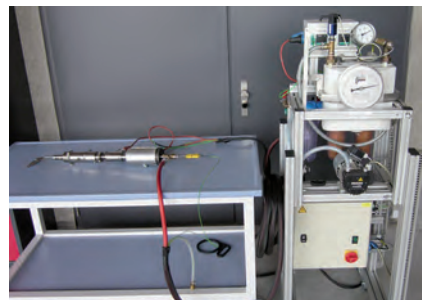


Figure 2: Dust sampling probe with controllable extraction unit consisting of a drying unit, controllable pump, gas meter and heating unit; Image: TFZ

### 1.6.1.7 Installation of dust sampling probe

Subsequent to a sufficient inlet zone, the dust sampling probe is to be installed in the centre of the exhaust gas measuring installation, at least 30 minutes before starting the measurement. The electrical trace heating of the extraction pipe as well as the heating sleeve has to be installed. The sampling temperature has to be adjusted to the prevalent exhaust gas temperature. The maximum filtration temperature is 180 °C, which is usually selected for ovens in the hot exhaust gas. If automatically stoked furnaces or e.g. tiled stoves with post-heater box are tested, a temperature of 100 °C in the undiluted exhaust gas may already be sufficient. The filtration temperature is to be checked.

If a dust sampling is to be performed in diluted exhaust gas, the filtration temperature is to be set to 50 °C. Temperatures above 50 °C have proven to be not practicable (Jokiniemi et al., 2008).

Given identical test conditions and measuring location, the dust sampling probe can be used multiple times. Afterwards the dust load determined via deposits in the sampling probe has to be split accordingly for the dust measurements.

### 1.6.2 Locating the centre of the flow

Before the measurements are started, the location of the centre of the flow needs to be determined via a suitable method. The following measuring instruments are typically used for this task:

- Prandtl's tube with differential pressure measurement device => Centre of the flow in the area of highest velocity
- Temperature sensor => Centre of the flow in the area of the highest exhaust gas temperature

If the flow profile is known, a search for the centre of the flow can be omitted. Considering the inlet zones given, a central installation of the measuring probe is usually possible.

### 1.6.3 Performing the measurement

The exhaust gas composition is determined and continuously captured via suitable gas analysis devices. Also, the speed measuring device with thermocouple has to be installed in the measuring installation. Ideally, two measuring devices for the determination of the speed are installed (e.g. Prandtl's tube and vane) in order to guarantee a reliable measurement. In this context, any narrowing of the cross-section (e.g. for the vane) has to be taken into consideration for the isokinetic dust sampling.

A suitable measuring file has to be created for the dust extraction. This includes information such as planar filter number, sleeve number, rinsing container number, nozzle diameter and exhaust gas pipe diameter at the measuring location. Regarding the purposes of standardisation the meter readings of the gas meter, the air pressure and the temperature at the gas meter have to be recorded, too.



The duration of the extraction has to be adjusted to the test conditions and the tasks at hand. In case of automatically stoked furnaces, the measuring duration regularly amounts to 30 minutes. In case of a batch combustion, for instance in an oven, the duration of the measurement has to be adjusted accordingly and the endpoint of the measurement has to be defined clearly. The measurement should be started directly after the door of the oven is closed. Measuring durations of less than 15 minutes should be avoided, due to higher measuring uncertainties. The filtration temperature during the dust sampling has to be checked and deviations from the pre-set temperature have to be logged. A premature ending of the sampling has to be logged as well, and the measurement has to be repeated, if necessary.

At the end of the measuring day, the dust sampling probe including the filter holder has to be de-installed and cooled off.

#### **1.6.4 Post-treatment of the measurement**

##### **1.6.4.1 Dismounting of the dust sampling probe**

After completing the measurement, the trace heating and the heating sleeve have to be disconnected and/or removed. Also, the extraction hose and the thermocouple for recording the filtration temperature have to be disconnected.

The filter holder is unscrewed and the probe is removed completely. Then the filter holder is taken apart and the stuffed filter sleeve is placed in the instrument tray. The clamping ring on the planar filter is removed and the planar filter is placed in the same Petri dish from which it had been removed. This way, the contents can be attributed unambiguously. Until the post-treatment, the filter media have to be stored dust-free.

##### **1.6.4.2 Post-treatment of the filter media**

The post-treatment is partly dependent on the desired material analyses. However, the method for the determination of the total dust should be pointed out, first.

For the post-treatment, the loaded planar filters and the loaded stuffed filter sleeves are dried for one hour at 120 °C. This drying temperature has to be added to the results, respectively. Subsequently, these filters and filter sleeves are stored for at least 8 hours in a desiccator located in the same room in which the weighing takes place. A single weighing is sufficient. During the weighing-in, the climatic conditions (room temperature, humidity, and air pressure) have to be recorded once.

If the temperature during the sampling is significantly below the drying temperature, any highly volatile components might get lost during the thermal post-treatment.

An important point is the consideration of possible fluctuations occurring in the weighing room. Therefore at least one reference of the weighed-in planar filters and stuffed filter sleeves per day has to undergo the same treatments as the loaded filter media and has to be weighed-out. This way, potential malfunctions of the micro-scale can be identified well.

In case of discrepancies, the filter medium has to be weighed again. A corresponding comment has to be added to the weighing file. In case of particular discolourations and/or an uneven dust load, photos have to be taken.

The filter media that were dried at 120 °C are suitable for material analyses with respect to anions, cations, and metals. However, if organic components are of interest, a different post-treatment of the filter media with load is recommended since a large share of the polycyclic aromatic hydrocarbons (PAH) are not thermally stable and thus the dust load determination will result in lower values.

#### 1.6.4.3 Post-treatment of the probe

The sampling probe including nozzle has to be rinsed after it has cooled down. A sequence of pictures can be found in Figure 3 and Figure 4. Here, in the first step, the probe is filled with distilled water and the liquid is distributed evenly throughout the probe before it is collected in a large beaker. This procedure is repeated two to three times with acetone, until the acetone is colourless. All liquids are initially collected in the same beaker. Then, the water/acetone mixture is transferred into an already weighed-in and numbered small rinsing container. Finally, the large beaker is rinsed once more with distilled water and the liquid is transferred into the rinsing container, too. A separate rinsing container is required for each dust probe. The rinsing container is subsequently dried at 105 °C in an explosion-proof drying cabinet. Once no liquid is visible in the rinsing container any more, it can be placed in the desiccator. It then has to be stored for at least 8 hours in the desiccator, in the room where the weighing takes place. A single weighing-in of the rinsing container is sufficient; the climatic conditions in the weighing room must be recorded once. As reference sample, a weighed-in rinsing container is partially filled with distilled water and acetone and subjected to the same treatment as the other rinsing containers.

For completion of the post-treatment of the dust sampling probe, it must be cleaned out with oil-free compressed air.



Figure 3: Rinsing of the sampling probe with distilled water and acetone; to the left: filling the liquid into the probe; to the right: distributing the liquid throughout the probe by shaking while sealing both openings off; Image: TFZ

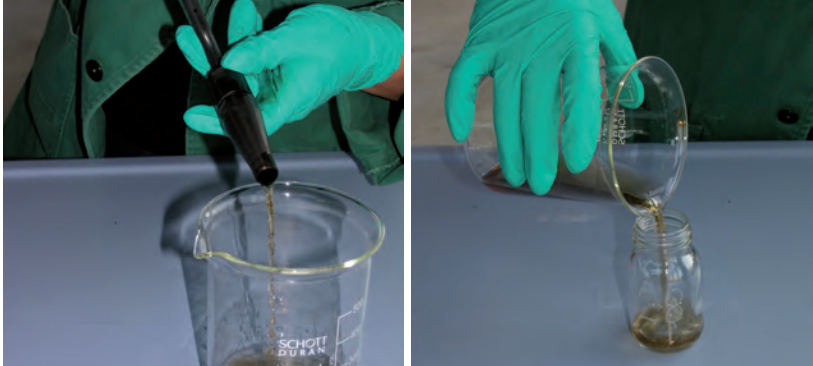


Figure 4: Rinsing of the sampling probe; to the left: emptying the sampling probe into a large beaker; to the right: pouring the entire amount of rinsing fluid (distilled water and acetone) into a rinsing container which is already weighed-in; Image: TFZ

## 1.7 Analysis

### 1.7.1 Determination of the absolute dust load per measurement

The load can be calculated via subtraction from the weighing-out and weighing-in of the filters and stuffed filter sleeves. The correction with the respective reference has to be taken into consideration.

$$m_{filter,load} = m_{filter,out} - m_{filter,in} - (m_{reference\ filter,out} - m_{reference\ filter,in}) \quad (A1.2)$$

$$m_{sleeve,load} = m_{sleeve,out} - m_{sleeve,in} - (m_{reference\ sleeve,out} - m_{reference\ sleeve,in}) \quad (A1.3)$$

$$m_{filter\ media,load} = m_{filter,load} + m_{sleeve,load} \quad (A1.4)$$

If the dust sampling probe is used multiple times, the amount of dust determined must be distributed to the respective dust samplings by percentage as follows.

$$m_{rinsing} = m_{rinsing\ container,out} + m_{rinsing\ container,in} - (m_{reference\ rinsing\ container,out} - m_{reference\ rinsing\ container,in}) \quad (A1.5)$$

$$m_{share\ of\ rinsing} = \frac{m_{filter\ media,load}}{\sum(m_{filter\ media,load})} \cdot m_{rinsing} \quad (A1.6)$$

The total load of the measurement is the result of:

$$m_{load} = m_{filter\ media,load} + m_{share\ of\ rinsing} \quad (A1.7)$$

### 1.7.2 Conversion of the values determined to $\text{mg}/\text{m}^3$ (STP) at 13 % $\text{O}_2$

The operating volume extracted through the filter media can be determined via the meter readings of the gas meter. The operating volume has to be converted to standard volume accordingly. Therefore, information regarding the temperature at the gas meter and the air pressure is needed.

$$V_N = \frac{(V_{\text{final gas meter read-out}} - V_{\text{initial gas meter read-out}}) \cdot p_{\text{gas meter}} \cdot T_N}{p_N \cdot T_{\text{gas meter}}} \quad (\text{A1.8})$$

Now a determination of the dust load under test conditions is possible.

$$c_{\text{total dust}} = \frac{m_{\text{load}}}{V_N} \quad (\text{A1.9})$$

To establish the reference to 13 %  $\text{O}_2$ , the average oxygen content over the course of the whole measurement is relevant. The following equation (A 1.10) shows the calculation of the conversion factor:

$$f_{\text{O}_2} = \frac{(21 - c_{\text{O}_2, \text{ref}})}{(21 - c_{\text{O}_2, \text{average}})} \quad (\text{A1.10})$$

Finally, the dust concentration has to be multiplied by the conversion factor.

$$c_{\text{total dust}@13\% \text{O}_2} = c_{\text{total dust}} \cdot f_{\text{O}_2} \quad (\text{A1.11})$$

## 1.8 General notes regarding dust sampling

If the amount of total dust is determined during the complete combustion phase or if there are very low dust levels in the exhaust gas (e.g. in automatic furnaces) the use of a planar filter is sufficient. If a stuffed filter sleeve is placed upstream, the measuring error due to the weighing may end up higher than the dust load itself.

In all other cases, a combination of stuffed filter sleeve and planar filter must be used. If a stuffed filter sleeve is required, the downstream placement of the planar filter is always necessary. If there are any doubts remaining, preliminary tests should be performed.

## 1.9 Summary

To provide reliable results regarding the dust concentration, a proper handling of the whole dust sampling is essential. Furthermore, the filter media have to be weighed-in and weighed-out with great diligence. The temperatures selected during the dust sampling and during the thermal post-treatment of the filter media are of great importance to the values determined. Therefore, these always have to be listed.

## 2

# **Determination of dust content (up to 50 mg/m<sup>3</sup>) in exhaust gas using small duct diameters - in accordance with VDI 2066 sheet 1**

## **SOP TK-01b**

### **Standard instructions**

Tobias Ulbricht

DBFZ - Deutsches Biomasseforschungszentrum gemeinnützige GmbH, Leipzig

## 2.1 Purpose / Principle

This SOP lays out a procedure for determining total dust concentrations (out stack) in flowing exhaust gases in accordance with VDI 2066 Sheet 1. It refers to VDI 2066 Sheet 1 "Particulate matter measurement / Dust measurement in flowing gases – Gravimetric determination of dust load" dated November 2006. In case of changes to this VDI guideline the SOP will have to be adjusted immediately. The SOP takes into consideration the sampling via an automatic isokinetic partial flow extraction unit (ITES) of the Paul Gothe GmbH company commonly used at the DBFZ.

## 2.2 Area of application

This procedure can be used under the following conditions:

- Internal pipe diameter less than 350 mm
- Anticipated dust concentration 20 – 150 mg/m<sup>3</sup> in exhaust gases without the presence of water drops
- Dynamic pressure of flue gas flow > 5 Pa
- Ratio of the highest to the lowest local gas velocity in the measuring cross section < 3:1
- Only tempered quartz filters, e.g. MK 360 by Munktell, may be used<sup>1</sup>

## 2.3 Measuring principle

During the measurement a partial flow is extracted from the centre of the exhaust gas stream under isokinetic conditions (at identical velocity). The partial flow passes through a stuffed filter sleeve and a downstream planar filter, separating the dust carried along. The gas of the partial flow is dried in a drying tower and the gas volume removed is determined. The dust mass accumulated on the filter is put in relation to the partial flow volume extracted.

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<sup>1</sup> The requirements for the filter material according to VDI 2066-1 have to be applied. The important article 9.1.7 states: The degree of separation of the planar filters must be greater than 99.5 % for a test aerosol with an average diameter of 0.3 µm or 99.9 % in case of a test aerosol with an average diameter of 0.6 µm. The degree of separation has to be certified. The filter material may neither react with gaseous components nor absorb them.

$$C_{total\ dust} = \frac{(m_{filter,\ out} + m_{sleeve,\ out} - m_{filter,\ in} - m_{sleeve,\ in})}{V_{partial\ flow,\ dr,\ N}} \quad (A2.1)$$

| Symbols                               | Definition   | Unit of measure         |
|---------------------------------------|--|-------------------------|
| $C_{total\ dust}$                     | Concentration of total dust at standard temperature and pressure | mg/m <sup>3</sup> (STP) |
| $m_{filter,\ out}$                    | Mass of filter upon weighing-out                                 | mg                      |
| $m_{filter,\ in}$                     | Mass of filter upon weighing-in                                  | mg                      |
| $m_{sleeve,\ out}$                    | Mass of filter sleeve upon weighing-out                          | mg                      |
| $m_{sleeve,\ in}$                     | Mass of filter sleeve upon weighing-in                           | mg                      |
| $V_{partial\ flow,\ dry,\ std,\ 24h}$ | Gas volume extracted at standard condition <sup>2</sup> dry      | m <sup>3</sup> (STP)    |

It is assumed that the average dust concentration in the partial flow is equivalent to the average dust concentration of the exhaust gas.

## 2.4 Devices and materials

### 2.4.1 Measuring set-up

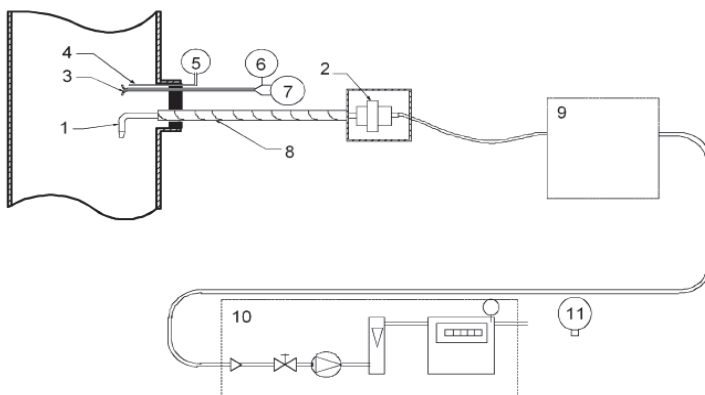


Figure 1: Example of an out-stack sampling facility from VDI 2066-1 (2006)

- |                                   |  |                            |
|-----------------------------------|--|----------------------------|
| 1. Sampling probe                 | 5. Temperature display                   | 9. Drying facility         |
| 2. Filter housing                 | 6. Measuring device for static pressure  | 10. Extraction unit (ITES) |
| 3. Exhaust gas backpressure probe | 7. Measuring device for dynamic pressure | 11. Manometer              |
| 4. Temperature probe              | 8. Extraction pipe                       |                            |

<sup>2</sup> Relative to 101.3 kPa and 273 K

For the measurement a filter head assembly for a plane filter is used:

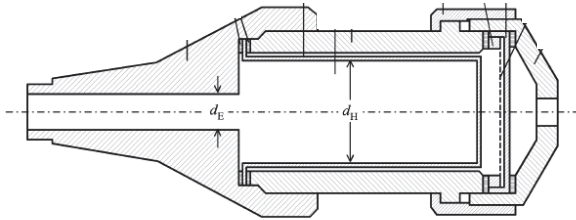


Figure 2: Filter head device for sleeve filter(s) and plane filter(s) from VDI 2066-1 (2006)

Table 1: Measuring devices required

| Device  | Designation / Test device number   | Comments  |
|---|--|---|
| Measuring device for the determination of the moisture content in the exhaust gas | FTIR 1 / FTIR 2 / HGMS   | Both continuously working measuring devices as well as a gravimetric determination of the moisture can be used. The share of moisture can also be calculated from a material balance (combustion calculation).  |
| Gas analyser for the determination of oxygen content in the exhaust gas           | PM B 7 EHEIM VISIT 02S<br>PM B 2 EHEIM VISIT 02S<br>PM V 4 EHEIM VISIT 02S<br>FTIR 1 / FTIR 2 / HGMS | -   |
| Measuring device for the determination of exhaust gas velocity                    |  | The determination of the exhaust gas backpressure is performed via a standardised Prandtl's tube.   |
| Automatic partial flow extraction unit ITES                                       | PM V 2<br>PM V 3<br>PM V 10<br>PM V 11   | With drying tower, pump, computer and dust probe consisting of nozzle, bent tube, filter head device with heating unit and electrically heated extraction pipe  |
| Scale   | PM L 2   | Resolution of the scale:<br>0.01 mg for reference value 0.2 mg/Nm <sup>3</sup><br>0.1 mg for reference value 2 mg/Nm <sup>3</sup><br>Uncertainty of the weighing: < 5 % of the reference value<br>The weighing should preferably be performed in an air-conditioned weighing cabinet. (A.2) |
| Drying cabinet  |  | Thermostat-controlled to +/- 5 K  |
| Desiccator or air-conditioned weighing cabinet / weighing room                    | DBFZ weighing cabinet  | With thermometer/hygrometer near the scale  |



### 2.4.3 Materials

- Plane filters type Munktell MK 360 with a diameter of 45 mm (tempered)
- Quartz filter wadding
- Filter sleeve (d=30 mm) made of titanium
- Acetone of "HPLC grade" purity
- Distilled water
- Parafilm (for sealing the rinsing liquid)
- Aluminium foil (for packaging of filters)
- Adhesive labels for the labelling of the samples (approx. 2 x 5 cm)
- Zip bags

### 2.4.4 Tools

- Tweezers
- Petri dishes
- Beaker, 150 ml
- Brush
- Gloves (heat-resistant)
- Laboratory gloves
- Water-proof pen
- Spatula
- Plug for nozzle

## 2.5 Performing the analysis

### 2.5.1 Preliminary considerations

#### 2.5.1.1 Measuring site

The measurement of the total dust concentration is conducted at a location with constant flow conditions. In general, these exist if a distance of at least five times the internal pipe diameter is adhered as inlet and outlet zone upstream of the measuring site and at least two times the downstream of it (five times the pipe's inner diameter is recommended). It is recommended to use the DBFZ standard measuring installations (Figure 3).

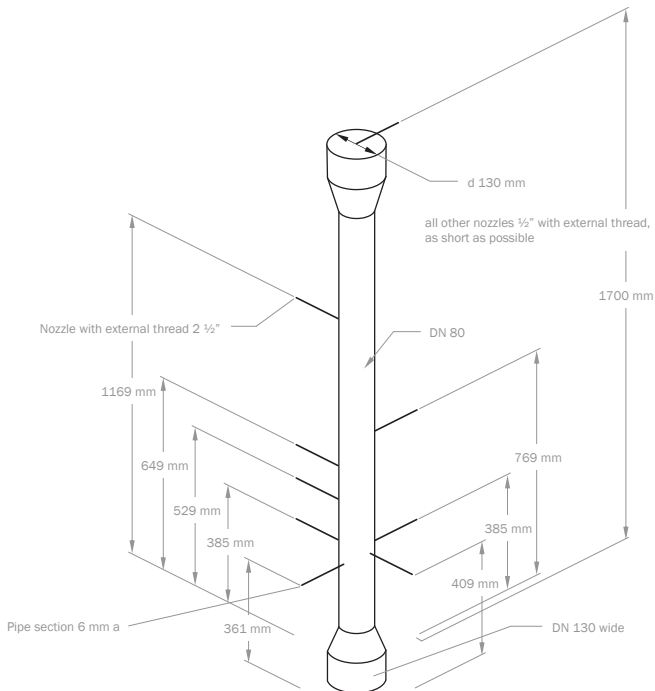


Figure 3: Dimensions of the standard measuring installation

### 2.5.1.2 Specifying the limit value and/or the reference value

Before the measurement is started, the statutory threshold that is the basis of the test should be known. If the objective of the measurement is not an adherence test given a specific threshold, a reference value has to be specified. The latter can, for instance, be a dust concentration specified by a manufacturer. Reference values below  $2 \text{ mg/m}^3_n$  are possible only if the resolution of the scale is at least 0.01 mg.

### 2.5.1.3 Specifying the minimum sampling volume and the sampling duration

The dust mass that is to be collected during the sampling, with dust concentrations near the specified threshold or reference value, has to be at least 20 times the weighing uncertainty<sup>3</sup>. Since the sleeve and the plane filter are weighed separately, twice the weighing uncertainty has to be assumed. If additional tests of dust concentrations are considered, the minimum concentrations should be used as basis for further uncertainties.

The minimum sampling volume necessary is determined based on the following equation:

$$V_{min} = \frac{\Delta m}{G} \quad (\text{A2.2})$$

| <i>Symbols</i> | <i>Definition</i>              | <i>Unit of measure</i> |
|----------------|--------------------------------|------------------------|
| $V_{min}$      | Minimum sampling volume        | $\text{m}^3$           |
| $\Delta m$     | Minimum dust mass              | mg                     |
| $G$            | Limit value or reference value | $\text{mg/m}^3$        |

Based on the required volume flow, either the sampling volume flow can be calculated with the help of the sampling duration, or the necessary sampling duration can be calculated from the sampling volume flow.

### 2.5.1.4 Specifying the nozzle diameter

The probe diameter required is the result of the highest gas velocity in the measuring cross section and the planned partial volume flow in the sampling probe.

$$d = \sqrt{\frac{4 \cdot \dot{V}}{\pi \cdot u_{max}}} \quad (\text{A2.3})$$

| <i>Symbols</i> | <i>Definition</i>           | <i>Unit of measure</i> |
|----------------|-----------------------------|------------------------|
| $d$            | Nozzle diameter             | $\text{m}^3$           |
| $\dot{V}$      | Planned partial volume flow | mg                     |
| $u_{max}$      | Maximum gas velocity        | $\text{m/s}$           |

<sup>3</sup> Under the conditions at the DBFZ, the weighing uncertainty currently amounts to 0.05 mg.

### 2.5.1.5 Specifying temperatures for sample treatment

The dust concentrations determined by means of this SOP do contain none or only a few condensable components. If necessary, these are measured and assessed as VOC during the measurements. This approach also corresponds to the draft standard DIN EN 303-5 (prEN 303-5:2010) "Heating boilers for solid fuels, hand and automatically stoked, nominal heat output of up to 300 kW – Terminology, requirements, testing and marking; EN 303-5:2012" (EN 303-5, 2012).

Therefore, VDI 2066-1 in general assumes a sampling temperature of 160 °C. In most cases, that temperature exceeds the water vapour dew point and the acid dew point of furnaces. The temperatures listed in this SOP refer to this value.

In case of exhaust gases with water drops, it may be necessary to adjust the temperatures. The temperature of the exhaust gas must not fall below dew point temperature. The temperatures to be used then can be found in Table 2.

Table 2: Temperatures

| Device                                   | Comments                                    |
|--|---|
| Temperature during sample pre-treatment  | higher/equal to sampling temperature + 20 K |
| Sampling temperature                     |   |
| Temperature during sample post-treatment | lower/equal to sampling temperature         |

Prior to the tests the treatment temperatures have to be coordinated with the employee performing the task. A sampling temperature of 160 °C is recommended. The filters to be used (filter sleeves and plane filters) must be marked and mentioned in the logs and in the measuring report.

## 2.5.2 Sample preparation

### 2.5.2.1 Preparation of the Petri dishes and storage containers for sleeves

The Petri dishes and containers intended to be used are subjected to a visual inspection. If any soiling is determined a cleaning in the laboratory dishwasher is performed. Since any labeling is typically removed during the process it has to be renewed.

### 2.5.2.2 Preparation of the filter sleeves

During laboratory activities laboratory gloves have to be worn. The empty filter sleeves are roughly cleaned and afterwards treated in the ultrasound bath (tap water) for 5 minutes. In the ultrasound bath the sleeves are placed in a suitable container. This is to prevent the rinsing water from mixing with the water in the ultrasound bath. A common laboratory cleaning agent (powder) is added to the rinsing water. Subsequently, the filter sleeve is rinsed with distilled water and dried.

Now the dry filter sleeves are stuffed with quartz wadding. The filters are filled up to approx. 2/3 of the sleeve's volume. The filling should consist of connected wads of wadding which should be free from short-fibred flakes and small particles. Subsequently, the wadding is compacted and therefore no channels throughout the material exist.

To dry the stuffed filter sleeve it is placed in the bottom part of a Petri dish and set in the drying cabinet at 180 °C for at least 1 h.<sup>4</sup> Prior to the weighing-out, the filter sleeve on the bottom part of the dish is stored in the humidity-controlled weighing cabinet for at least 8 h. Furthermore, the scale has to be tested with standard weights before the weighing or an automatic calibration could be performed. The result of the scale test as well as the temperature and humidity conditions in the weighing cabinet have to be recorded.

The weight of the filter sleeves has to be determined at least three times and logged in the respective file. If the standard deviation of the measurements is more than 0.05 mg the weighing has to be repeated.

Until further use, the weighed-out filter sleeves are stored in closed glass containers in the dust protection cabinet. Sleeves intended to be used at a sampling temperature of 160 °C are stored on the shelf labelled "VDI 160 °C only".

For each series of measurements, three weighed-out sleeves remain in the laboratory as control filters in order to determine any difference in weight.

### 2.5.2.3 Preparation of the plane filters

During laboratory activities laboratory gloves have to be worn. The empty plane filter holders are roughly cleaned and afterwards treated in the ultrasound bath (tap water) for 5 minutes. In the ultrasound bath the filter holders are placed in a suitable container so that mixing of the rinsing water with the water in the ultrasound bath is prevented. A common laboratory cleaning agent (powder) is added to the rinsing water. Subsequently, the filter holder is rinsed with distilled water.

The plane filters are placed into individually numbered plane filter holders using tweezers. From that point on the plane filter holders are stored or transported in Petri dishes labelled "VDI 160 °C". If other temperatures are used the labelling has to be adjusted.

To dry the plane filter holders with inserted plane filters they are to be placed in the drying cabinet at 180 °C for at least 1 h. Prior to the weighing-out, the equipped plane filter holders are stored in the humidity-controlled weighing cabinet for at least 8 h.

Furthermore, the scale has to be tested with standard weights before the weighing or an automatic calibration could be performed. The result of the scale test as well as the temperature and humidity conditions in the weighing cabinet have to be recorded.

The weight of the equipped plane filter holders has to be determined at least three times and logged in the respective file. If the standard deviation of the measurements is more than 0.05 mg, the weighing has to be repeated.

Until further use, the weighed-out plane filter holders are stored in the dust protection cabinet in closed Petri dishes. Filters intended to be used at a sampling temperature of 160 °C are stored on the shelf labelled "VDI 160 °C only".

For each series of measurements, three weighed-out plane filters remain in the laboratory as control filters in order to determine any difference in weight.

<sup>4</sup> For other temperatures, see chapter 2.5.1.5

#### 2.5.2.4 Preparation of the automatic isokinetic extraction and the probe

Prior to starting the measurements the measuring device has to be checked. Especially the following issues have to be considered:

- Check if cleaning of the measuring device is necessary
- Check the probe inlet for damage
- Thoroughly check the sealing surfaces; replace improper seals and hose connections (keep a stock of back-up material at hand)
- Check the drying towers and, if applicable, empty and refill them with prepared drying agent
- Inspect the electrical devices (e.g. replace defective cables, plugs and switches)
- Check the validity of the calibration

The software is started and has to be checked. The main focus lies on the serial number of the device which has to correspond to the configuration file loaded. The serial number is located on top of the housing of the ITES.

Before the measurement or the leakage test can be started, the ITES has to operate for a minimum of 20 min.

According to the software instruction, the leakage test is to be performed by closing the probe's opening with a plug. Therefore, the bypass has to be adjusted to the maximum negative pressure achievable during sampling. But always a negative pressure of at least 200 mbar has to be ensured.

The probe's heating unit has to be connected in time (a minimum of 60 min prior to the start of measurement) to heat up the filter heads. Before starting the extraction the required temperature of 160 °C<sup>4</sup> has to be reached.

#### 2.5.3 Locating the centre of the flow

Before the measurements are started, the centre of the flow needs to be determined via a suitable method. The following instruments may be used:

- Prandtl's tube with differential pressure measurement device => Centre of flow is in the area of highest velocity
- Temperature sensor => Centre of flow is in the area of the highest exhaust gas temperature

The ratio between the lowest and the highest velocity measured in the measuring cross section may not exceed 1:3. A negative velocity is not permitted.

If the flow profile is known, locating the centre of the flow can be omitted. This has to be noted in the log. The position of the probe has to be noted in the log as well.

#### 2.5.4 Performing the measurement

The composition of the exhaust gas is entered into the entry mask of the ITES using the data of the gas analysers or a combustion calculation.

The hoses of the Prandtl's tube must be connected to the sensor box. The connections are marked black/red/yellow. The desired extraction parameters (duration of measurement, nozzle size, humidity of the exhaust gas) must be entered. Furthermore, the thermocouple that is located in the exhaust gas duct is connected to the sensor box.

The file naming is performed according to the specifications of the technical centre.

The desired duration of the extraction must be entered. Storage intervals are 60 s. The actual duration of the extraction is entered into the log. If the automatic determination of some measuring parameters is waived (setting in the system), the data has to be checked before each measurement and entered manually.

The measuring duration must amount to at least 30 min.

The probes are introduced into the measuring installation either immediately prior to the measurement or they remain in the exhaust gas flow during the whole measuring cycle. Either way, the procedure has to be noted in the log. The probe has to be turned until the sampling probe is angled  $\pm 10^\circ$  upstream. The sampling site must be sealed.

The filter head is equipped with a filter sleeve and a plane filter holder and closed. The heating unit is placed and the extraction hose is connected.

The extraction is started.

Once the measuring cycle ends (acoustic signal) the extraction must be ended manually (in the program).

The sleeve, the filter and the filter housing have to be inspected visually for indications of cracking or discolouration due to pressure or humidity. Discolourations suggest a temperature drop below the dew point. In this case, the measurement is invalid.

The volume extracted (at standard condition) is entered into the log.

After each series of measurement or at least once a day, an empty sample must be drawn. For the empty sample a sleeve and a plane filter are pre-treated and post-treated like the measurement filter and inserted into the sampling facility. However, the pump is not started. Once the measurements at a measuring site have ended the probe must be removed and the leakage test repeated.

## **2.5.5 Post-treatment of the measurement**

### **2.5.5.1 Dismounting of the filters**

The extraction hose and the heater are removed and the filter head is opened with the corresponding key. The plane filter holder is retrieved with tweezers and placed in a covered Petri dish. The filter sleeve is retrieved with tweezers and stored in the storage glass.

### **2.5.5.2 Dismounting of the filter sleeve**

The filter sleeve is placed on a bottom part of a Petri dish and dried for at least 1 h in the drying cabinet at 160 °C<sup>d</sup>. Afterwards, the filter sleeve is put into the weighing cabinet for at least 8 h to cool down and acclimatise. Placed below the Petri dish is a note with an unambiguous labelling of the sample.

Furthermore, the scale has to be tested with standard weights before the weighing. The result of the scale test as well as the temperature and humidity conditions in the weighing cabinet have to be logged. Subsequently, the three control filters belonging to this series of measurements are to be weighed-out in accordance with the following paragraph.

The weight of the filter sleeves is determined at least three times at intervals of one minute and logged in the respective file. If a clear trend is determined, the weighing is to be repeated at the earliest after 4 h. The results are also to be entered into the result log. Upon request of the employer photos of the sleeves can be taken.

### **2.5.5.3 Post-treatment of the plane filter**

The plane filter holder with plane filter is placed on a bottom part of a Petri dish and dried for at least 1 h in the drying cabinet at 160 °C<sup>d</sup>. Afterwards, the filter sleeve is put into the weighing cabinet for at least 8 h to cool down and acclimatise. Placed below the Petri dish is a note with an unambiguous labelling of the sample.

Furthermore, the scale has to be tested with standard weights before the weighing. The result of the scale test as well as the temperature and humidity conditions in the weighing cabinet have to be recorded. Subsequently, the three control filters belonging to this series of measurements are to be weighed-out in accordance with the following paragraph.

The weight of the equipped planar filter holders is determined at least three times at intervals of one minute and entered into the respective file. If a clear trend is determined, the weighing is to be repeated at the earliest after 4 h. The results are also to be entered into the result log.

Upon request of the employer, photos of the planar filters can be taken.



#### 2.5.5.4 Post-treatment of the probe

The probe is rinsed in a beaker (150 ml) as follows:

Distilled water (approx. 30 ml) is run alongside the interior wall of the probe from the filter head device to the nozzle. The liquid must spread across the entire wall surface. Subsequently, this procedure is repeated with approx. 20 ml acetone HPLC grade and finally repeated with approx. 30 ml of distilled water. The beaker with the rinsing liquid is sealed with parafilm and given to the laboratory with a request for analysis. The further process is regulated by a separate SOP. The probe pipe is dried with compressed air (oil-free).

#### 2.5.5.5 Checking of the measuring device

After completing the investigations the following issues have to be considered carefully:

- Check if cleaning of the measuring device is necessary. The probe is cleaned with water and special brushes. In a final step the probe is rinsed with distilled water and dried.
- Check the probe inlet for damage
- Thoroughly check the sealing surfaces; replacement of improper seals and hose connections (keep a stock of back-up material at hand)
- Empty and refill the drying towers with prepared drying agent
- Inspect the electrical devices (e.g. replacement of defective cables, plugs and switches)

## 2.6 Analysis

### 2.6.1 Manual calculation method

The mass of the empty filter, the loaded filter, the empty filter sleeve, and the loaded filter sleeve is determined by means of the three weighing results each. The mass of dust from the rinsing water is added as a percentage to the respective dust loads of the filters with load (sleeve + planar filter). The amount of dust collected consist of the mass difference between loaded planar filter and sleeve as well as the percentage of rinsing water and the empty/unloaded filter and sleeve. The amount of dust collected is divided by the volume of gas extracted (at standard condition, dry).

$$c_{total\ dust} = \frac{\left( (m_{filter, out} + m_{sleeve, out} + m_{share\ of\ rinsing}) - m_{filter, in} - m_{sleeve, in} \right)}{V_{partial\ flow, dr, N}} \quad (A2.4)$$

| <b>Symbols</b>             | <b>Definition</b>   | <b>Unit of measure</b>  |
|----------------------------|---|-------------------------|
| $c_{total\ dust}$          | Concentration of total dust                                 | mg/m <sup>3</sup> (STP) |
| $m_{filter, out}$          | Mass of filter upon weighing-out                            | mg                      |
| $m_{share\ of\ rinsing}$   | Share of amount of dust from probe rinsing                  | mg                      |
| $m_{filter, in}$           | Mass of filter upon weighing-in                             | mg                      |
| $m_{sleeve, out}$          | Mass of filter sleeve upon weighing-out                     | mg                      |
| $m_{sleeve, in}$           | Mass of filter sleeve upon weighing-in                      | mg                      |
| $V_{partial\ flow, dr, N}$ | Gas volume extracted at standard condition <sup>5</sup> dry | m <sup>3</sup> (STP)    |

Subsequently, the measuring result still has to be put in relation to a reference oxygen value (e.g. 13 %).

$$c_{total\ dust, 13\%O_2} = c_{total\ dust, realO_2} \cdot \left( \frac{0.21 - c_{O_2, ref}}{0.21 - c_{O_2, real}} \right) \quad (A2.5)$$

| <b>Symbols</b>             | <b>Definition</b>                               | <b>Unit of measure</b>  |
|----------------------------|---|-------------------------|
| $c_{total\ dust, 13\%O_2}$ | Total dust concentration at 13 % O <sub>2</sub> | mg/m <sup>3</sup> (STP) |
| $c_{total\ dust, realO_2}$ | Total dust concentration at nat. O <sub>2</sub> | mg/m <sup>3</sup> (STP) |
| $c_{O_2, ref}$             | Reference oxygen concentration (e.g. 13 %)      | %                       |
| $c_{O_2, real}$            | Oxygen concentration in the exhaust gas         | %                       |

### 2.6.2 Measuring error

Due to the temporal change of the exhaust gas composition it is not possible to determine repeatability limits and reproducibility limits of this method. A statistical comparison is possible only via successive, simultaneous double measurements with two identical sampling devices.

After experimental tests, VDI 2066-1 states the following detection limits in case of a 30 min sampling duration:

- For dry gases, approx. 0.3 mg/m<sup>3</sup>
- For gases saturated with water, approx. 2 mg/m<sup>3</sup>

For these detection limits a weighing without systematic errors is assumed.

<sup>5</sup> Relative to 101.3 kPa and 273 K

These errors are created by:

- Insufficient temperature balance of the material to be weighed
- Different temperatures at weighing-in and weighing-out
- Insufficient moisture equilibrium
- Different humidity at weighing-in and weighing-out
- Influence of changes in air pressure

Assuming a sufficient acclimatisation period, if the scale is set up in the weighing cabinet, no errors are generated by insufficient balances or differing humidity at weighing-in and weighing-out. If the room the scale is set up in is air-conditioned, errors due to different temperatures at weighing-in and weighing-out can be neglected.

The influence of the change in air pressure can cause a systematic error if the test weight for the automatic calibration of the scale does not feature the same density as the material to be weighed. The systematic error occurring here can amount to up to 0.3 mg.

If an oxygen value is referred to, the main part of the measuring error will be based on the oxygen concentration measurement. In this method (gravimetric dust measurement with reference to an oxygen value) calculations are performed with a relative measuring error of 7 % is assumed.

### 2.6.3 Measures regarding quality assurance

To check whether the filters supplied are free from volatile organic compounds they are subjected to a random EC/OC analysis. If increased concentrations are suspected the filters may not be used for this process. If necessary the filters have to be tempered at the DBFZ and must subsequently be subjected to a renewed testing.

The empty sample may not exceed the limit value or reference value by more than 10 %. Otherwise, the measurement is invalid.

#### 2.6.3.1 Weighing cabinet

For a reliable weighing, a weighing under the same climatic conditions, a weighing cabinet has proven to be most practical.

The weighing cabinet is a hood made of transparent plastic surrounding the scale, preferably located on a weighing table. Inside the cabinet shelves for the filters are installed. By means of a small membrane pump and a drying tower, the weighing cabinet is constantly supplied with dry air. Therefore, in case of leaks, dry air may leave the weighing cabinet but no humid air will enter.

The weighing is performed under the same conditions as the acclimatising of the sample. Thus, measuring errors due to different temperatures and humidity levels can be ruled out.



Figure 4: Weighing cabinet with shelves

#### 2.6.4 Measuring report

In accordance with VDI 2066-1 the measuring report must contain the following information:

- Stating the employer and the employee responsible for the measurements
- Description of the purpose of the investigations, the plant/system and date of sampling
- Description of the operating conditions of the plant/system and of all changes that occurred during the measurement
- Description of the sampling site and characteristics of the gas in the exhaust gas duct:
  - Duct dimensions, number and location of the measuring axes and the measuring points
  - Speed and temperature profile
  - $O_2/CO_2$ , humidity content, density of the gas
  - Adherence to the requirements with respect to the flow in the measuring cross section

Measuring method:

- Speed measurement (calibration of the speed measuring devices, if no standardised exhaust gas backpressure probes or similar devices are being utilised)

- Details of the sampling facility:
  - Diameter of the sampling probe
  - Filter type (material, size, etc.)
  - Calibration of devices for measurements of volume or volume flow
  - Filtration temperature
  
- Weighing method:
  - Pretreatment temperature
  - Correction of weights

Test results:

- Number of measurements
- For each measurement:
  - Date, time and duration
  - Volume collected and average volume flow
  - Barometer reading
  - Mass of dust on the filters and in the rinsing liquid
  - Concentrations under actual conditions and under reference conditions
  - Special circumstances or incidents
- Raw data (e.g. gross weight, average temperature during sampling, average static pressure in the exhaust gas duct) must either be included with the measuring report or be available for further assessment

Quality assurance:

- Results of the leakage tests
- Reference value (empty)
- Adherence to the isokinetic conditions

Comments:

- Also to be stated in the measuring report are all circumstances that may have had an influence on the results as well as any information with respect to the uncertainty of the results.
- If it was necessary to modify the method, these changes must be mentioned in the measuring report and must be justified accordingly.

Summary:

- The report must contain a summary of the results.

# 3

## **Simplified determination of dust content in exhaust gas ("70 °C measurement")**

### **SOP TK-02**

#### **Standard instructions**

Tobias Ulbricht

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### 3.1 Purpose / Principle

This SOP regulates the simplified method for determining total dust concentrations in flowing exhaust gases. The operating procedure is modelled after VDI 2066 Sheet 1.

The procedure is a compromise between an exact gravimetric dust measurement according to VDI 2066 Sheet 1, a sampling for subsequent chemical tests and economic considerations.

### 3.2 Area of application

This method can be used under the following conditions:

- Internal pipe diameter less than 350 mm
- Exhaust gas backpressure greater 5 Pa
- Only tempered quartz filters MK 360 may be used

### 3.3 Measuring principle

During the measurement, a partial flow is extracted isokinetically (at identical velocity) from the centre flow stream of the exhaust gas. The partial flow passes through a filter, separating the dust carried along. The gas of the partial flow is dried in a drying tower and the gas volume retrieved is determined.

The dust mass located on the filter is put in relation to the partial flow volume extracted.

$$c_{total\ dust} = \frac{(m_{filter,\ out} - m_{filter,\ in})}{V_{partial\ flow,\ dr,\ N}} \quad (A3.1)$$

| <b>Symbols</b>               | <b>Definition</b>                                | <b>Unit of measure</b>  |
|------------------------------|--|-------------------------|
| $c_{total\ dust}$            | Concentration of total dust                      | mg/m <sup>3</sup> (STP) |
| $m_{filter,\ out}$           | Mass of filter upon weighing-out                 | mg                      |
| $m_{filter,\ in}$            | Mass of filter upon weighing-in                  | mg                      |
| $V_{partial\ flow,\ dr,\ N}$ | Gas volume extracted at standard conditions, dry | m <sup>3</sup> (STP)    |

It is assumed that the average dust concentration in the partial flow is equivalent to the average dust concentration of the exhaust gas.

## 3.4 Devices and materials

### 3.4.1 Devices

**For a measurement the following devices are required:**

Table 1: Measuring devices required

| Device  | Comments   |
|---|--|
| Measuring device for the determination of the exhaust gas temperature             | –  |
| Measuring device for the determination of the moisture content in the exhaust gas | Suitable measuring methods must be applied. Both continuously working measuring devices as well as a gravimetric determination of the moisture can be used. The share of moisture can also be calculated from a material balance (combustion calculation). |
| Gas analyser for the determination of the oxygen content in the exhaust gas       | --   |
| Measuring device for the determination of the exhaust gas speed                   | The determination of the exhaust gas backpressure is performed via a standardised Prandtl's tube.  |
| Measuring device for the determination of the static pressure                     | --   |
| Automatic partial flow extraction facility ITES                                   | With drying tower, pump, computer and dust probe consisting of nozzle, bent tube, filter head device with heating unit and electrically heated extraction pipe   |
| Scale   | The weighing is performed in the DBFZ lab on a scale type "ABT 220-5 DM" located in an air-conditioned weighing cabinet.   |

### 3.4.2 Materials

- Plane filters type Munktell MK 360 with a diameter of 150 mm (baked out for 6 h at 350 °C)
- Acetone of "HPLC grade" purity
- Distilled water
- Parafilm (for sealing the rinsing liquid)
- Aluminium foil (for packaging of filters)
- Prepared adhesive labels for labelling of the samples
- Zip bags



### 3.4.3 Tools

- Tweezers
- Petri dishes
- Beaker, 150 ml
- Brush
- Gloves (heat-resistant)
- Laboratory gloves
- Water-proof pen
- Watch glass
- Spatula

## 3.5 Measuring site

The measurement of total dust concentration is conducted at a location with consistent flow conditions. A distance of five times the pipe's inner diameter both upstream and downstream of the measuring site is recommended as inlet and outlet zone. Furthermore, a simultaneous introduction of both the exhaust gas backpressure probe and the probe of the exhaust gas analysis device has to be possible.

It is recommended to use the standard measuring installations that exist at the technical centre.

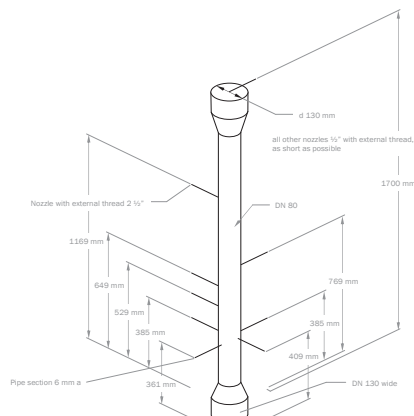


Figure 3: Dimensions of the standard measuring installation

## 3.6 Performing the analysis

### 3.6.1 Sample preparation

#### 3.6.1.1 Preparation of the Petri dishes

The Petri dishes and containers intended to be used are subjected to a visual inspection. If any soiling is determined a cleaning in the laboratory dishwasher is required. Since labelling is removed in the process it has to be renewed.

#### 3.6.1.2 Preparation of the planar filters

During laboratory activities laboratory gloves have to be worn. The empty plane filter holders are roughly cleaned and afterwards treated in the ultrasound bath (tap water) for 5 minutes. In the ultrasound bath the filter holders are placed in a suitable container so that mixing of rinsing water with water in the ultrasound bath is prevented. A common laboratory cleaning agent (powder) is added to the rinsing water. Subsequently, the filter holder is rinsed with distilled water.

The planar filter holders are dried in the drying cabinet (180 °C) for 20 min.

With the use of tweezers the planar filters are placed into individually numbered planar filter holders. The planar filter holders are then stored/transported in Petri dishes labelled "70 °C".

Prior to the weighing-out, the equipped planar filter holders are stored in the humidity-controlled weighing cabinet for 2 h (-0 h / +24 h).

The weight of the equipped planar filter holder is determined at least three times at intervals of one minute and logged in the respective file. If a clear trend is determined the weighing will not have to be repeated before 4 h. This has to be noted in the log. To assure a high quality standard the person responsible for quality management has to be informed.

Until the weighed-out planar filter holders are used, they are stored in the dust protection cabinet in closed Petri dishes on the shelf labelled "NOT BAKED OUT" .

#### 3.6.1.3 Preparation of the automatic isokinetic extraction

The software is started and has to be checked, mainly in respect to the serial number of the device which has to correspond to the configuration file loaded. The serial number is located on top of the housing of the ITES.

Before a measurement or the leakage test can be started, the ITES has to operate for a minimum of 20 min.

According to the software instruction, the leakage test is to be performed by closing the probe's opening with a plug. Therefore, the bypass has to be adjusted to the maximum negative pressure achievable during sampling. However, a negative pressure of at least 200 mbar has to be ensured at all times.

#### **3.6.1.4 Preparation of the probe**

The probes are introduced into the measuring installation either immediately prior to the measurement or remain in the exhaust gas flow stream during the whole measuring cycle. Either way, this has to be logged. The probe's heating unit has to be connected in time (a minimum of 30 minutes prior to the start of measurement) to heat up the filter heads to the temperature required. The temperature is adjusted to 65 °C.

The filter head is equipped with a planar filter and closed. The heating system is installed and the extraction hose is connected.

#### **3.6.2 Locating the centre of the flow**

Before the measurements are started the centre of the flow stream needs to be determined via a suitable method. The following instruments may be used:

- Prandtl's sensor with differential pressure measurement device => Centre of the flow is in the area of highest speed
- Temperature sensor => Centre of the flow is in the area of the highest exhaust gas temperature.

If the flow profile is known, identifying the centre of flow can be omitted. This has to be noted in the log. The position of the probe has to be noted in the log as well.

#### **3.6.3 Performing the measurement**

The exhaust gas composition data obtained from the FTIR or EHEIM and the calculatory values are entered into log as specified.

The hoses of the Prandtl's tube must be connected to the sensor box. The connections are marked black/red/yellow. Similarly, the thermocouple must also be connected to the sensor box.

The desired extraction parameters (duration of measurement, nozzle size, humidity of the exhaust gas) must be entered. The file naming is done in accordance with the specifications of the technical centre.

In the next stage of the process the exhaust gas temperature is entered if no thermocouple has been installed.

The desired duration of the extraction must be entered. Storage interval is 60 s. The actual duration of the extraction is entered into the event log.

In general, the measuring duration should be 30 min. After consultation with the person in charge of the experiment a shorter duration may also be possible. However, measuring durations of less than 15 min should be avoided.

Once the measuring cycle ends (acoustic signal) the extraction must be ended manually (in the program).

The volume extracted (at standard condition) is entered into the event log.

### **3.6.4 Post-treatment of the measurement**

#### **3.6.4.1 Dismounting of the planar filter**

The extraction hose is removed.

The folding heater is removed and the filter head is opened with the respective tool. The planar filter holders are retrieved with tweezers and stored in their covered Petri dish.

The planar filter holders are placed on the bottom part of the Petri dish and put into the weighing cabinet for at least 10 h to cool down and acclimatise. A note with unambiguous labelling of the sample is placed under the Petri dish.

The equipped planar filter holder is weighed at least three times at intervals of one minute and the values are logged in the respective file. If a clear trend is determined the weighing will not have to be repeated before 4 h. Those results are also to be logged in the results file. To assure a high quality standard the person responsible for quality management has to be informed.

In case of particular discolourations photos of the planar filter are to be taken.

The soot number is determined via comparison to a soot number card and entered into the weighing file and the results log.

The planar filters are removed with tweezers, placed on the aluminium foil, folded (in the middle) with the help of the spatula, and wrapped in the aluminium foil (druggist fold). The run, load and filter number are captured on an adhesive label and the label is stuck onto the packaged filter (do not write on the filter or the druggist fold!). The druggist fold is packaged in a bag labelled with the description of the test.

The type of storage (e.g. freezer or at room temperature) depends on the objective of the analysis.

If stored in the freezer this has to be noted in the corresponding storage list (file “Freezer-Contents”).

#### 3.6.4.2 Post-treatment of the probe

The probe is rinsed out into a beaker (150 ml) as follows: distilled water (approx. 30 ml), approx. 20 ml acetone HPLC grade, approx. 30 ml distilled water.

The rinsing liquid is sealed with parafilm and placed in the lab with a request for analysis. The further process is regulated by a separate SOP.

The probe pipe is blown dry with compressed air (oil-free).

### 3.7 Analysis

The mass of the empty filter, the loaded filter, the empty filter sleeve, and the loaded filter sleeve is determined by means of the three weighing results each. The mass of dust from the rinsing water is added as a percentage to the respective dust loads of the measuring filters with load (sleeve + planar filter). The amount of dust collected consists of the mass difference of the loaded planar filter and sleeve as well as the percentage of the rinsing water and the empty/unloaded filter and sleeve. The amount of dust collected is divided by the volume of gas extracted (at standard condition, dry).

$$c_{total\ dust} = \frac{((m_{filter, out} + m_{sleeve, out} + m_{share\ of\ rinsing}) - m_{filter, in} - m_{sleeve, in})}{V_{partial\ flow, dr, N}} \quad (A3.2)$$

| <b>Symbols</b>             | <b>Designation</b>   | <b>Unit of measure</b>  |
|----------------------------|--|-------------------------|
| $c_{total\ dust}$          | Concentration of total dust at standard temperature and pressure | mg/m <sup>3</sup> (STP) |
| $m_{filter, out}$          | Mass of filter upon weighing-out                                 | mg                      |
| $m_{share\ of\ rinsing}$   | Share of amount of dust from probe rinsing                       | mg                      |
| $m_{filter, in}$           | Mass of filter upon weighing-in                                  | mg                      |
| $m_{sleeve, out}$          | Mass of filter sleeve upon weighing-out                          | mg                      |
| $m_{sleeve, in}$           | Mass of filter sleeve upon weighing-in                           | mg                      |
| $V_{partial\ flow, dr, N}$ | Gas volume extracted at standard condition dry                   | m <sup>3</sup> (STP)    |

Subsequently, the measuring result still has to be put in relation to a reference oxygen value (e.g. 13 %).

$$c_{total\ dust,13\%O_2} = c_{total\ dust,realO_2} \cdot \left( \frac{0.21 - c_{O_2,ref}}{0.21 - c_{O_2,real}} \right) \quad (A3.3)$$

| <b>Symbols</b>             | <b>Definition</b>  | <b>Unit of measure</b>  |
|----------------------------|--|-------------------------|
| $c_{total\ dust, 13\%O_2}$ | Total dust concentration at 13 % O <sub>2</sub> at standard temperature and pressure | mg/m <sup>3</sup> (STP) |
| $c_{total\ dust, realO_2}$ | Total dust concentration at nat. O <sub>2</sub> at standard temperature and pressure | mg/m <sup>3</sup> (STP) |
| $c_{O_2, ref}$             | Reference oxygen concentration (e.g. 13 %)   |                         |
| $c_{O_2, real}$            | Oxygen concentration in the exhaust gas  |                         |

### 3.8 Measuring error

The described measuring procedure (gravimetric dust measurement with reference to an oxygen value) has a relative measuring error of 7 % (regarding calculations).

The respective measuring error has to be calculated individually for each application case.

The main source for measuring uncertainties originates from the oxygen concentration measurement (+/- 2% or 1 Vol-% absolute) and the determination of volume flow.

### 3.9 Quality assurance measures

To check whether the filters supplied are free from volatile organic compounds they are subjected to a random EC/OC analysis. In case of increased concentrations, the filters may not be used for this procedure. If necessary, the filters have to be tempered at the DBFZ and must subsequently be subjected to a renewed testing.

The weighing table is equipped with a function that displays a clearly recognizable trend in colour. Drying and humidification trends are captured and analysed as part of the quality assurance process. This way, it can be checked whether the storage durations in the weighing cabinet are sufficient.





For quality assurance, the following information should be noted in the log:

- A repetition of the weighing due to an observed trend in masses (3.6.1.2 and 3.6.4.2)
- Number of times the probe has been used (3.6.1.2)
- The position of the dust probe in the exhaust gas flow (3.6.2)
- Whether locating the centre of flow was omitted. (3.6.2)

### 3.10 Illustrated book

#### Total dust measurement with planar filter, 45 mm diameter, 70 °C

##### Pretreatment steps

|  |   |   |
|--|---|---|
| <b>01</b>  | <b>Cleaning planar filter holder</b>                                |    |
| <ul style="list-style-type: none"><li>• cleaning coarsely</li><li>• ultrasound bath (5 min), rinse with tap water</li><li>• rinse with distilled water</li></ul>   |   |   |
| <b>02</b>  | <b>Drying at 180°C for 20 min</b>                                   |    |
| <ul style="list-style-type: none"><li>• place filter holder in drying cabinet</li><li>• dry filter holder for 20 min at 180°C</li></ul>  |   |   |
| <b>03</b>  | <b>Place planar filter in holder</b>                                |   |
| <ul style="list-style-type: none"><li>• use rounded tweezers</li><li>• place planar filter in holder</li><li>• number Petri dish (same number as filter holder number)</li></ul>   |   |   |
| <b>04</b>  | <b>Filter and planar filter holder in weighing cabinet &gt; 2 h</b> |  |
| <ul style="list-style-type: none"><li>• transfer filter and holder into weighing cabinet and store for &gt; 2 h</li><li>• weighing-out within 24 h</li><li>• record weight; 3 measurements at one minute intervals</li><li>• subsequently store in dust protection cabinet</li></ul> |   |   |

## Preparation of the probe

### 05 Install probes

- determination of the centre of the flow
- insert probe at the beginning of the test
- set the temperature of probe heating unit at 65 °C before starting the measurement



### 06 Equip the probe

- equip, connect extraction hose
- carry out leak test (200 mbar)
- connect pressure and temperature sensors



## Carrying out the measurement

### 07 Even prior to the extraction

- obtain exhaust gas composition from gas measurement in parallel
- measuring durations from 15 - 30 min



## Post-treatment

### 08 Completing the measurement

- retrieve exhaust gas hose, folding heater, filter holder
- place filter holder in Petri dish, close Petri dish
- log entry





### 09 10 h weighing cabinet

- store filter and holder for at least 10 h in weighing cabinet
- if necessary, separate labelling of the samples
- weighing-out, 3 measurements at one minute intervals each





|  |                                    |   |
|--|------------------------------------|---|
| <b>10</b>  | <b>Packaging of planar filters</b> |  |
| <ul style="list-style-type: none"><li>• if analyses are necessary, retrieve filter and package it</li><li>• for this, wrap it in aluminum foil</li><li>• labeling -&gt; in case of PAH analyses: store in freezer<br/>-&gt; in case of inorganic analyses: instruct a laboratory</li></ul> |                                    |   |
| <b>11</b>  | <b>Rinse probe</b>                 |  |
| <ul style="list-style-type: none"><li>• with approx. 20 ml acetone and 30 ml H<sub>2</sub>O dest.</li><li>• fill out analysis order</li></ul>  |                                    |   |

# 4

## Gravimetric total dust measurement with 150 mm quartz fibre filter

### SOP TK-03

### Standard instructions

Torsten Schröder  
Tobias Ulbricht

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## 4.1 Purpose / Principle

This SOP lay out the process for determining total dust concentrations in flowing exhaust gases and the simultaneous generation of filter samples with a 150 mm quartz filter. This operating procedure is modelled after VDI 2066 Sheet 1.

## 4.2 Area of application

This method can be used under the following conditions:

- Internal pipe diameter less than 350 mm
- Exhaust gas backpressure greater 5 Pa
- Exhaust gas temperature < 70 °C (due to durability of material)
- Flow profile at the measuring site in the exhaust gas pipe is known

## 4.3 Measuring principle

For the measurement a partial flow is extracted isokinetically (at identical speed) from the centre of flow of the exhaust gas. The partial flow is passed through a filter on which all of the dust carried along will settle. The gas of the partial flow is dried in a drying tower and the gas volume removed is determined.

As per instruction, the filter is separated into three pieces (two small ones and one large one), the individual pieces are weighed and the percentage share of mass of the filter pieces is determined. Subsequently, the large piece is conditioned and weighed-out once again. To obtain the mass of the dust from the whole filter the mass of the whole filter is derived via the mass of the large piece.

The dust mass located on the filter is put in relation to the partial flow volume extracted.

$$c_{total\ dust} = \frac{(m_{filter,\ out} - m_{filter,\ in})}{V_{partial\ flow,\ dr,\ N}} \quad (A4.1)$$

| <b>Symbols</b>               | <b>Definition</b>  | <b>Unit of measure</b>  |
|------------------------------|--|-------------------------|
| $c_{total\ dust}$            | Concentration of total dust at standard temperature and pressure | mg/m <sup>3</sup> (STP) |
| $m_{filter,\ out}$           | Mass of filter upon weighing-out                                 | g                       |
| $m_{filter,\ in}$            | Mass of filter upon weighing-in                                  | g                       |
| $V_{partial\ flow,\ dr,\ N}$ | Gas volume extracted at standard condition, dry                  | m <sup>3</sup> (STP)    |

It is assumed that the average dust concentration in the partial flow is equivalent to the average dust concentration of the exhaust gas.

## 4.4 Devices and materials

### 4.4.1 Devices

For the measurement, the following devices are required:

Table 1: Measuring devices required

| Device  | Comments   |
|---|--|
| Measuring device for the determination of the exhaust gas temperature             | -  |
| Measuring device for the determination of the moisture content in the exhaust gas | Suitable measuring methods must be applied. Both continuously working measuring devices as well as a gravimetric determination of the moisture can be used. The share of moisture can also be calculated from a material balance (combustion calculation). |
| Gas analyser for the determination of the oxygen content in the exhaust gas       | -  |
| Measuring device for the determination of the exhaust gas speed                   | The determination of the exhaust gas backpressure is performed via a standardised Prandtl's tube. Alternatively, the use of a vane sensor is also possible. In that case, however, the current speed value has to be logged in the program manually.       |
| Measuring device for the determination of the static pressure                     | -  |
| Automatic partial flow extraction facility ITES                                   | With drying tower, pump, computer and dust probe consisting of nozzle, bent tube, filter head device with heating unit and electrically heated extraction pipe   |
| Scale without weighing cabinet  | Type AB 265-S  |
| Scale with weighing cabinet   | Scale type "ABT 220-5 DM" located in the air-conditioned weighing cabinet  |

### 4.4.2 Materials

- Planar filters type Munktell MK 360 with a diameter of 150 mm (baked out for 6h at 350 °C)
- Acetone of "HPLC grade" purity
- Distilled water

- Parafilm (for sealing the rinsing liquid)
- Aluminium foil (for packaging of filters)
- Prepared adhesive labels for labelling of the samples

#### 4.4.3 Tools

- Tweezers
- Petri dishes
- Beaker, 150 ml
- Brush
- Gloves (heat-resistant)
- Watch glass
- Laboratory gloves
- Water-proof pen
- Spatula

## 4.5 Measuring site

The measurement of total dust concentration is conducted at a location with consistent flow conditions. A distance of five times the internal pipe diameter both upstream and downstream of the measuring site is recommended as inlet and outflow zone. Furthermore, it must be possible to introduce both the exhaust gas backpressure probe and the probe of the exhaust gas analysis device simultaneously.

The process is only suitable for measurements at the dilution tunnel.

## 4.6 Performing the analysis

During the laboratory activities (preparation, weighing, etc.) attention has to be paid to an environment that is, for the most part, dust-free. Furthermore, laboratory gloves must be worn during these activities.

A short graphical overview is given in the Appendix.

#### 4.6.1 Sample preparation

##### 4.6.1.1 Preparation of the Petri dishes

The Petri dishes and containers intended to be used are subjected to a visual inspection. If any soiling is determined a cleaning in the laboratory dishwasher is required. Since the labelling may be removed in the process the numbers have to be transferred to cleaned watch-glasses.

##### 4.6.1.2 Preparation of the planar filters

The filters are removed from the packaging and placed in numbered Petri dishes (dish bottoms only). The filters in the dish bottom are subsequently dried for 60 min in the pre-heated drying cabinet at 105 °C.

After the drying the filters in the lower shell are removed from the drying cabinet and cooled in the ambient air for approx. 10.

Prior to the weighing-out, the filters in the Petri dishes are stored in the humidity-controlled weighing cabinet for at least 6 h. For weighing the filter is removed with both hands from the Petri dish, using tweezers, and placed on a watch-glass on the scale in the weighing cabinet. If the watch-glass "wobbles" a sealing ring can be placed under it. The scale with the watch-glass must be tared first. If necessary, the air supply to the weighing cabinet has to be turned off (influence on the weighing due to the air flow). The weight is determined a minimum of three times and logged in the respective file.

Until they are used, the weighed-out filters are stored in the dust protection cabinet in Petri dishes closed by a lid.

##### 4.6.1.3 Preparation of the probe

The probes are introduced into the measuring installation either immediately prior to the measurement or remain in the exhaust gas flow during the whole measuring cycle. Either way, it has to be noted in the log. If the probes are to remain in the exhaust gas flow a leakage test (see below) must be performed prior to installation.

The clean filter holder is warmed for one hour in the drying cabinet at 50 °C immediately prior to use.

The filter is retrieved from the Petri dish with tweezers and spatula and placed on the cleaned filter holder (bottom part). Subsequently, the flat gasket (ring thickness 0.25 mm or 0.5 mm) is inserted. The flat gasket must rest on the filter evenly. The cleaned top portion of the filter holder is then put in place and the top and bottom portion are closed tightly with wing nuts.

The filter is carefully transported to the measurement site. The leakage test has to be performed in accordance with the software instructions. For this a probe opening is sealed with a plug and the bypass has to be adjusted to a negative pressure of approx. 200 mbar. After the leakage test the probe is installed by two people and positioned accordingly in the exhaust gas pipe (where applicable check via vertical pipe nozzle). The measurement is performed in the centre of the flow of the exhaust gas.

The probe's heating unit must be connected in a timely manner (approx. 30 min before the start of measuring). The temperature is adjusted to 60 °C.

The extraction hose is connected.

#### **4.6.1.4 Preparation of the automatic isokinetic extraction**

Start the software and check if the configuration file corresponds to the serial number of the device. The serial number is located on top of the housing of the ITES. Before the measurement the drying tower must be checked and the drying agent replaced if necessary.

The hoses of the Prandtl's tube must be connected to the sensor box. The connections are marked black/red/yellow. The desired extraction parameters (duration of measurement, nozzle size, humidity of the exhaust gas) must be entered. Furthermore, the thermocouple that is located in the exhaust gas duct has to be connected to the sensor box.

#### **4.6.2 Performing the measurement**

The exhaust gas composition is entered into the data entry masks of the ITES in accordance with the results log (current template) from the FTIR or EHEIM data and the calculated values.

The desired duration of the extraction must be entered. Storage interval is 60 s. The actual duration of the extraction is entered into the log. If the automatic determination of some measuring parameters is waived (setting in the system) these data must be checked before each measurement and will have to be entered manually.

In general, the measuring duration should be at least 30 min. Upon consultation with the person in charge of the experiment shorter durations are possible. However, measuring durations of less than 15 min should be avoided.

The extraction is started.

Once the measuring cycle ends (acoustic signal) the extraction must be ended manually (in the program).

The volume extracted (at standard condition) is entered into the event log.

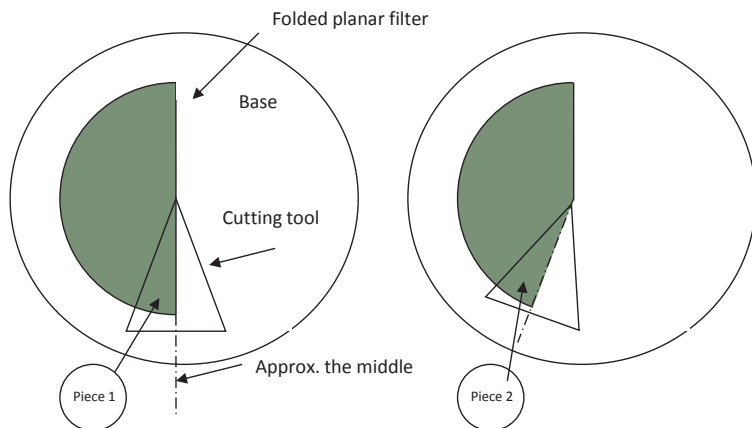


Figure 1: Creating the filter pieces 1, 2 and 3

### 4.6.3 Post-treatment of the measurement

#### 4.6.3.1 Dismounting of the planar filter

Caution! From the end of the measuring period until the freezing of pieces 1 and 2 and the weighing of piece 3 no more than 30 min should pass.

The extraction hose is taken off and the filter holder is unscrewed and transferred into the laboratory.

For the following work steps a dust-free base and clean tools are required!

In the laboratory the filter holder is placed on the stand (to avoid risk of tilting) and the screws on the filter holder are loosened. Subsequently, the upper part is lifted straight up and the sealing ring is removed with tweezers. The filter cake must not be damaged during this step.

With the help of the spatula and tweezers the filter is removed and placed on a dust-free base (tile).

The filter is folded in half while the side containing the load is facing inward. The filter is then weighed-out on a scale (three individual measurements each) and the results are recorded. For the weighing a watch-glass is placed on top of the scale outside the weighing cabinet and tared in.



Then 15° of the folded filter are punched out (Fig. 1). The smaller piece receives the number 1 and is weighed, too (three individual measurements).

Another partial piece of 15° is punched out and weighed (three individual measurements on the scale outside of the weighing cabinet). Attention has to be paid to the fact that the pieces are to be connected at the tip. They must, however, not be cut off from the opposite side of the large filter piece. This piece receives the number 2.

The pieces are wrapped in aluminium foil with the sides folded together and creased multiple times as a druggist fold. The number of the piece, the number of the Petri dish, the date and the number of the project are recorded on an adhesive label and the label is stuck onto the packaged filter (do not write on the filter!). Then, the druggist fold is packaged in a labelled bag and stored in the freezer at -21 °C and entered into the respective freezer list. The remaining piece of the filter (partial piece 3) is weighed once again (three individual measurements on the scale outside the weighing cabinet) and the result is recorded. Thereafter, the remaining piece is dried in a Petri dish (dish bottom) in the drying cabinet for one hour at 105 °C. After the drying the remaining piece is taken out of the Petri dish and cooled down in ambient air for approx. 10 min. For a complete cooling down and acclimitating the Petri dish with the remaining piece is stored in the weighing cabinet for 6 h. A note with the unambiguous labelling of the sample is placed under the Petri dish.

The weight of the conditioned remaining piece is determined a minimum of three times and entered into the respective file. For this purpose the scale in the weighing cabinet has to be used.

The remaining piece is then wrapped in aluminium foil, labelled as above, and added to the other folds in the labelled bag in the freezer.

#### **4.6.3.2 Post-treatment of the probe**

The probe is rinsed out into a 150 ml beaker as follows: distilled water (approx. 30 ml), approx. 20 ml acetone HPLC grade, approx. 30 ml distilled water. The flat gasket also must be rinsed and the rinsing liquid collected.

The rinsing liquid is sealed and analysed for dust load in the laboratory. The probe pipe is blown dry with compressed air (oil-free).

## 4.7 Analysis

The calculation is performed analogous to Appendix 3 Chapter 3.7, where the amount of dust to be used is calculated via the following steps:

The change of mass (percentage) through acclimitising of piece 3 (remaining piece) from the mass before and after the acclimitising is determined.

Calculating the "acclimitised amount of dust" from the weighing of the whole filter before the cutting and the impact of the acclimitising calculated from the weighing of piece 3.

For further calculations, the "acclimitised amount of dust" is used.

## 4.8 Measuring error

For the method of gravimetric dust measurement with reference to an oxygen value, calculations are performed assuming a relative measuring error of 7 %. Additionally, an even and concentric dust distribution on the filter is assumed. The main source of the measuring uncertainties originates from the oxygen concentration measurement (+/- 2 % or 1 Vol-% absolute) and the determination of the volume flow.

## 4.9 Quality assurance measures

To check whether the filters supplied are free from volatile organic compounds, these are subjected to a random EC/OC analysis. In case of increased concentrations the filters may not be used for this process. If necessary, the filters have to be tempered at the DBFZ and must subsequently be subjected to a renewed testing.

## 5

# Guidance for the gravimetric measurement of dust with 150 mm quartz fibre membrane filters

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## Pretreatment

### 01 Dry heated filter for 1 h at 105 °C



- Remove filter from packaging
- Place filter on numbered Petri dish (subshell only)
- Place filter on dish bottom in heated drying cabinet  
105 °C / 60 min

### 02 Retrieve dried filter and let it cool down



- Cool down the filter on numbered Petri dish (dish bottom only, approx. 10 min)

### 03 Place dried, cooled down filter in weighing cabinet for 6 h



- Transfer filter on numbered Petri dish (dish bottom only)
- into weighing cabinet for at least 6 h (or desiccator)

### 04 Determine empty weight of the filter



- Dried, cooled down filter in weighing cabinet
- For weighing, transfer filter with both hands from numbered Petri dish (dish bottom only) into weighing cabinet to watch glass (before tare scale with watch glass, if necessary, turn of extraction (triple weighing)

### 05 Storing filter for use



- Storing weighed-out, dried, cooled down filter for use
- Retrieve filter on bottom of Petri dish from weighing cabinet
- Cover with Petri dish top and put down for use

**06 Preheat filter holder for 1 h, 50 °C**

- Preheat filter holder in drying cabinet for 1 h at 50 °C
- Preheating directly before use

**Sampling****07 Using the filter**

- Filter is weighed-out, dried and cooled down
- Retrieve filter from Petri dish
- Place filter in cleaned filter holder (bottom part)

**08 Insert sealing ring**

- Selection of thickness: 0,25 mm oder 0,5 mm
- Sealing ring must rest flat on the filter

**09 Close the filter holder**

- Place the clean planar filter top construction on bottom part
- Tightly close top and bottom part with wing nuts
- Perform leak test
- Attach and switch on probe's heating unit (30 min before start of measuring)

**10 Insert filter holder**

- 2 people required
- Be carefully during transport (grab only onto the holder)

**11 Insert filter holder**

- 2 people required
- Be carefully during transport (grab only the holder)

**12 Start extraction**

- Set up ITES, values from parallel gas measurement
- Heat probe to 60 °C

**Post-treatment****13 Preparation**

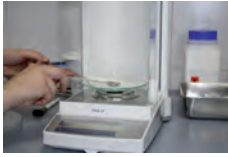
- Pre-heat drying cabinet to 105 °C
- Prepare aluminium foil for packaging
- Prepare adhesive labels, where applicable

**14 Complete extraction**

- Complete extraction
- Disconnect extraction hose from filter holder
- 25 min for the following procedures up to item 23
- Transfer filter holder to laboratory and take it apart

**15 Folding the filter**

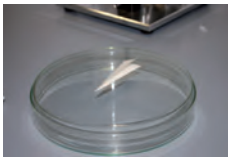
- Ensure a clean, dust-free base
- Use cleaned tools
- Place filter on aluminium foil
- Fold filter in half – load facing inwards

**16 Weigh out fouled filter**

- Use scale out of the weighing cabinet!
- Put watch-glass „wobble-free“ on scale (place a sealing ring underneath) and tare the scale
- Weigh out folded filter

**17 Cut the filter**

- Cut out 15° of the folded filter (estimating the centre)
- The small partial piece receives the No. 1

**18 Partial piece 1**

- Place on a clean surface after cutting
- The small partial piece receives the No. 1
- Weigh partial piece 1 on the watch glass (3x) & enter values in Excel-table
- Use scale out of the weighing cabinet!

**19 Partial piece 2 (work step may be omitted, where applicable)**

- Cut out an additional partial piece (15° of the large remaining piece) (pieces must be contiguous), not from the opposing side
- Weigh partial piece 2 on the watch glass (3x)
- Use scale out of the weighing cabinet!

**20 Packaging partial piece 1 and partial piece 2**

- Use small pieces of aluminium foil

**21 Packaging partial piece 1 and partial piece 2**

- Fold sides together and crease multiple times

**22 Label partial piece 1 and partial piece 2**

- Use labelled adhesive label
- Information: Piece no. 1 or 2
- Number of the Petri dish, date, project number, etc.
- Place both parts in a bag, label it and place it in the freezer (-21°C)

**23 Weigh out the remaining piece (piece 3) once more**

- Tare scale with watch glass and weigh folded remaining piece 3 times
- Use scale out of the weighing cabinet!

**24 Place remaining piece in drying cabinet for 1 h at 105 °C**

- Place folded remaining piece (piece 3)
- On Petri-dish bottom
- Place for 1 h at 105 °C in drying cabinet

**25 Let dried remaining piece cool down**

- Retrieve folded remaining piece (piece 3) from drying cabinet
- Let it cool down on numbered Petri dish (5 - 10 min)



**26 Transfer remaining piece into weighing cabinet for 6 h**

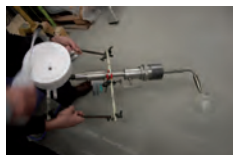
- Transfer cooled down remaining piece (piece 3) to weighing cabinet
- Leave it in there for 6 h
- Subsequently, weigh it out (3 x) and record the values

**27 Packagieng the remaining piece**

- Retrieve folded remaining piece (piece 3) from weighing cabinet
- Package remaining piece in aluminium foil

**28 Label remaining piece and send it**

- Label the remaining piece (piece 3) wrapped in aluminium foil
- Use labelled adhesive label
- Package remaining piece and store / send it
- or place it in the bag with the others (at -21 °C)

**29 Cleaning filter holder and probe**

- Rinse filter holder and probe with acetone and H<sub>2</sub>O dest.
- Evaporate eluviation and weigh-out residues
- Transfer the values to the log

## 6

# **Instructions for the gravimetric quantification of dust emissions in case of long measuring periods and transient emissions behaviour due to modification of the dust measuring method in accordance with VDI 2066-1**

Robert Kunde  
Mamun Adeili  
Florian Volz  
ZAE Bayern, Garching

## 6.1 Area of application

The following describes a procedure for gravimetric quantification of cumulated total dust emissions from small-scale furnaces. A wood pellet small-scale furnace which is operated under realistic operating conditions, while an unsteady emissions behaviour is occurring is used as an example. The procedure is modelled after the requirements of VDI Guideline 2066 Sheet 1 (VDI 2066-1, 2006). The dust measuring method described here is modified by suitable measures to allow high dust loads measurements. Depending on the dust concentrations, longer measuring durations (up to 120 h) can therefore be realised.

## 6.2 Measuring principle

In case of daily total dust measurement on a small-scale pellet furnace described here, the system is operated under controlled conditions, therefore the boiler provides a realistic load profile. Due to the variable heat load flow, however, diverse operating conditions of the small-scale pellet furnace may occur with corresponding variations in emission levels, for instance, automatic on and off settings of the furnace.

Due to an extended duration of measurement and unsteady emissions behaviour combined with frequently high dust concentrations, the requirements for the method had to be adapted in relation to VDI 2066-1. The increased dust load requires, on the one hand, an increase of the filter capacity, and also an adjustment of the continuous exhaust gas conditioning (refrigeration and dehumidification) given the longer measuring duration. On the other hand, the measuring equipment has to work without operating personnel, which requires an automatic control of the extraction flow volume, automatic data logging as well as appropriate safety facilities and turn-off mechanisms.

For the determination of daily total dust emissions only the phases of boiler operation during which dust is emitted are of interest. To increase the life-span of the exhaust gas desiccation via silica gel (orange gel) it is therefore practical to extract a partial flow only during emissions-relevant operating states. Thus, an automatic differentiation must be made between emissions-relevant and non emissions-relevant operating states. This is achieved by defining an emissions-relevant operating state as one with a significant exhaust gas volume flow. This is the case when the exhaust gas fan of the furnace is operating. For some of the pellet furnaces under investigation, this information could be derived from the electrical power consumption of the furnace.

During measurement, a partial flow is retrieved from the centre of the flow in accordance with VDI 2066-1. Due to the small particle size of the dust, isokinetic sampling is not required. The partial flow, however, has to be retrieved proportional to the volume flow which can be achieved through a regulated mass flow controller (MFC). It is recommended, nevertheless, to avoid a sub-isokinetic extraction by selecting a suitable diameter for the extraction nozzle.

To increase the filter capacity it is recommended to utilise pre-manufactured glass fibre filter sleeves (if necessary, additionally stuffed with quartz wadding). As a safeguard (e.g. in case of a torn filter, leaking, or anticipated potentially incomplete deposit of the dust in the

filter sleeve a planar filter should be placed downstream. Special attention has to be paid when selecting the filter medium in order to provide enough capacity for the anticipated dust load.

The conditioning of the exhaust gas to standard conditions is performed via a pre-desiccation by means of intensive cooling and subsequent drying in a bed of desiccant beads (silica gel, orange gel). The volume of the extracted partial flow is determined with a gas meter (e.g. bellows-type gas flow meter) while the temperature and residual moisture (standard conditions) are recorded.

The average dust concentration is determined by putting the amount of dust located on the filter in proportion to the partial flow volume extracted in accordance with the following equation (A6.1):

$$c_{total\ dust} = \frac{(m_{filter,\ out} - m_{filter,\ in})}{V_{partial\ flow,\ dr,\ N}} \quad (A6.1)$$

| <b>Symbols</b>               | <b>Definition</b>   | <b>Unit of measure</b>  |
|------------------------------|---|-------------------------|
| $c_{total\ dust}$            | Concentration of total dust at standard temperature and pressure  | mg/m <sup>3</sup> (STP) |
| $m_{filter,\ out}$           | Mass of filter upon weighing-out                                  | mg                      |
| $m_{filter,\ in}$            | Mass of filter upon weighing-in                                   | mg                      |
| $V_{partial\ flow,\ dr,\ N}$ | Gas volume extracted at standard condition (273 K, 1013 mbar) dry | m <sup>3</sup> (STP)    |

For a partial flow extraction proportional to the volume flow and the respective controlling (MFC), a continuous monitoring of the exhaust gas volume flow (e.g. via pipe cross section and flow rate) is necessary.

The total dust load emitted during the measuring period (here 24 h) is calculated in accordance with the following equation (A6.2):

$$m_{total\ dust,\ 24h} = c_{total\ dust} \cdot V_{total\ exhaust\ gas\ flow,\ dr,\ N} \quad (A6.2)$$

| <b>Symbols</b>                           | <b>Designation</b>   | <b>Unit of measure</b>   |
|--|--|--------------------------|
| $m_{total\ dust,\ 24h}$                  | Total dust load emitted during the measuring period; here 24 h   | mg/duration under review |
| $c_{total\ dust,\ 24h}$                  | Concentration of total dust at standard temperature and pressure   | mg/m <sup>3</sup> (STP)  |
| $V_{total\ exhaust\ gas\ flow,\ dr,\ N}$ | Measured (and/or calculated) total exhaust gas volume at standard conditions (dry), during the measuring period; here 24 h | m <sup>3</sup> (STP)     |

Thereby, it is assumed that the average dust concentration in the partial flow is equivalent to the average dust concentration of the exhaust gas.

Due to high dust concentrations, a filter's life-span may be shorter than the period of investigation (e.g. 24 h). In that case, it must be assessed whether an extrapolation of the

dust data up to this point to the full investigation period is appropriate. Also, it has to be assessed whether the emissions behaviour of the furnace during the actual measurement is similar to its normal behaviour.

## 6.3 Measuring devices and tools

### 6.3.1 Measuring devices

For a measurement according to the procedure the measuring devices in Table 1 are recommended.

Table 1: Measuring devices for the determination of total dust

| Measuring device   | Intended use/comments   |
|--|---|
| Thermocouple (e.g. type K)   | For the determination of the exhaust gas temperature at the measuring point..   |
| Gas analyser for determination of oxygen content in the exhaust gas  | Necessary for a conversion of the dust load according to the reference parameter at 13 % O <sub>2</sub>   |
| Device for measuring the exhaust gas speed   | A hot-wire anemometer is used to determine the exhaust gas speed. In addition to the temperature at the measurement point, the measuring range of the device has to be paid attention to.<br>A regular rinsing of the hot-wire with pressurised cold air helps to ensure measuring accuracy over longer periods of time.<br>The exhaust gas speed can also be utilised as a control parameter to check whether the exhaust gas fan is operating or not. |
| Device for measuring the pressure differential via the filter  | To determine the pressure differential via the filter, to detect the filter load, and for safety shut-off of the partial flow extraction, e.g. in case of a torn filter.  |
| Devices for measuring temperature, humidity and static pressure  | To determine the state of the partial flow as well as the ambient temperature and ambient pressure for a calculatory conversion of the partial volume flow to standard conditions. The humidity can also be used as a shut-off criterion for the automatic partial flow extraction in order to detect a saturation of the desiccation facility.   |
| Automatically controlled partial flow extraction unit;<br>Device for measuring electric power consumption of the furnace | Dust sampling probe consisting of nozzle, bent tube – where applicable –, electrically heated extraction pipe, and heated filter sleeve head device with downstream planar filter head device, drying tower, vacuum pump, gas meter; exhaust gas volume flow proportional controlling of the partial volume flow via mass flow controller (MFC).<br>Partial flow extraction that can be switched on and off, depending on electric power consumption.   |
| Micro scale  | To determine the mass of the filter sleeves (stuffed where applicable) before and after loading, with an accuracy of at least 1 mg. The two weightings should be performed at ambient conditions which are as similar and/or constant as possible (for instance in a climatic chamber and/or climatic cabinet).   |

### 6.3.2 Measuring set-up

In Figure 1, the basic design of the measuring apparatus is depicted schematically.

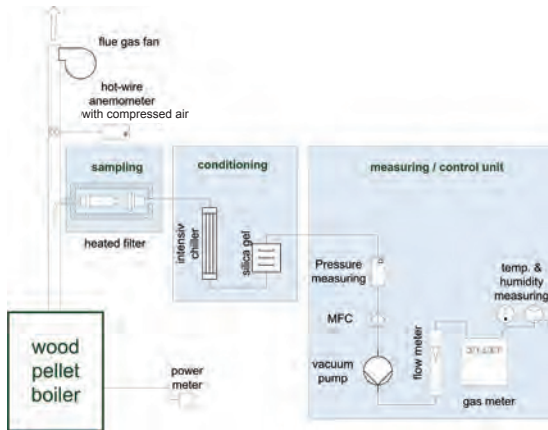


Figure 1: Schematic depiction of the measuring set-up for determination of total dust emissions

For the regulation of the partial volume flow a mass flow controller (MFC) is used. The planned partial volume flow is derived, e.g. by means of test stand software, from the exhaust gas speed / volume determined via a hot-wire anemometer.

### 6.3.3 Materials

For a determination of total dust, the following materials are additionally required:

- baked out filter sleeves made of glass fibre (e.g. type Munktell ET/MG 160) with corresponding filter sleeve holder and suitable filter holder (support cage),
- baked out planar filters made of quartz fibre (e.g. type Munktell MK 360) with a suitable diameter (e.g. 50 mm for filter holders made by Gothe),
- Isopropanol and distilled water for rinsing of the sampling probe,
- Aluminium foil – where applicable – for packaging of filters for further analysis (not always necessary),
- Zip bags – where applicable – for shipping of the samples for further analysis (not always necessary),
- Hygrometer, thermometer, barometer for determination of ambient conditions

Since the filter sleeves typically do not have a constant outer diameter (due to manufacturing), an individually adjustable support cage should be used as a filter holder.

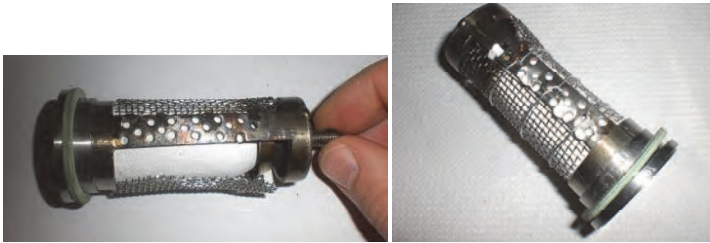


Figure 2: Filter sleeve support cage with variable adjustment of circumference and length; Image: ZAE Bayern



Figure 3: Filter sleeve holder and planar filter holder in the heating case (with temperature measurement); Image: ZAE Bayern

#### 6.3.4 Tools

The following additional tools are required for filter handling:

- Clean wide-neck glass vessels,
- Clean tweezers,
- Brush/paintbrush,
- Gloves (heat-resistant) for installation and removal of the probes,
- Laboratory gloves (powder-free),
- Large beaker,
- Rinsing container,
- Waterproof pen for labelling of samples.

## 6.4 Choice of appropriate measuring point

Depending on the size of the furnace, a corresponding exhaust gas volume flow has to be taken from the furnace. For a reliable speed measurement by means of hot-wire anemometer, speeds of at least 1 m/s are necessary. This can be achieved by choosing a suitable inner pipe diameter. For example, for a boiler with a heating power of approx. 15 kW, an inner diameter of approx. 100 mm is preferable.

For the measurement of total dust concentrations it has to be ensured that the flow at the measuring location is even. Thus, the upstream and downstream stretches of the speed measuring devices have to adhere to the manufacturer's specifications. For a narrowing in the cross section, upstream and downstream stretches in accordance with Table 2 are recommended.

Table 2: Recommended upstream and downstream stretches for some measuring devices after a narrowing of the cross section

| Measuring device    | Upstream stretch/downstream stretch  |
|---------------------|--|
| Dust sampling probe | 5 x pipe's inner diameter, respectively  |
| Hot-wire anemometer | Upstream stretch with approx. 5 x pipe's inner diameter; downstream stretch with approx. 5 x pipe's inner diameter |

The upstream stretches have to be longer if the measuring device is to be installed downstream of an expansion in cross section or of a redirection.

In addition to the upstream stretches, a centric installation position of the measuring devices has to be adhered to.

Corresponding thermocouples have to be placed near the measuring devices in order to be able to make statements regarding the sampling temperature.

## 6.5 Performing of measurement

### 6.5.1 Preparation of measurement

#### 6.5.1.1 Preparation of Petri dishes and wide-neck glass vessels

Clean wide-neck glass vessels and/or Petri dishes are to be used for the storage of filter sleeves and/or planar filters without and with load. These have to be cleaned and subsequently dried prior to use. Unambiguous labelling of the samples must be ensured.



#### **6.5.1.2 Preparation of planar filters**

The planar filters are retrieved from their packaging with tweezers and placed in the labelled Petri dishes. Subsequently, the planar filters are dried in the open Petri dishes at 120 °C for 90 minutes to remove any (highly) volatile organic matter. The planar filters are then dried for at least another 6 hours in the open Petri dish in a desiccator in the same room in which the weighing takes place. It must be ensured that the Petri dishes are covered with the respective lids so that no contamination of the planar filters may occur, yet a sufficient air exchange is guaranteed for drying. Prior to the weighing the planar filters have to acclimatise to the conditions of the weighing room for at least 6 hours outside of the desiccator. This is a deviation from VDI 2066-1. The climatic conditions (room temperature, humidity and air pressure) must be noted once during the weighing. After the single weighing the planar filters are stored, protected from dust in the closed Petri dishes until use.

#### **6.5.1.3 Preparation of filter sleeves**

If high dust loads are anticipated a planar filter is not sufficient as the filter medium. Additional, pre-manufactured filter sleeves stuffed with filter wadding (if necessary) will then have to be placed upstream. Filter sleeves are prepared analogously to the planar filters.

#### **6.5.1.4 Preparation of planar filter holder**

The empty planar filter holder must undergo visual inspection prior to use. If fibre residue and/or soiling are visible, the filter holder needs to be cleaned with a paintbrush. In case of coarse soiling the planar filter holder is cleaned in an ultrasound bath (tap water). Subsequently, it is rinsed with distilled water and dried. The planar filter holder itself is not weighed-in.

#### **6.5.1.5 Preparation of filter sleeve holder**

The preparation is performed analogously to the approach for the planar filter holder, however, filter sleeves should be weighed as they are installed into the support cage. Otherwise, the handling of the filter sleeves during installation and removal from the support cage may lead to damages to the filter sleeve with corresponding (significant) impact on its weight.

#### **6.5.1.6 Preparation of dust sampling probe**

Firstly, the filter holder must be equipped with an empty planar filter which is fixed in position with a clamping ring. Then, the equipped filter holder must be installed carefully in the planar filter head device. This must then be installed carefully in the filter sleeve head device. The devices have to be assembled properly; Attention needs to be paid to the necessary sealing rings. Subsequently, the filter holder is screwed onto the clean probe. The nozzle diameter needs to be selected so that a sufficient life-span in case of high dust concentration can be guaranteed.

### 6.5.1.7 Preparation of automatic dust extraction

At the beginning of a measurement the drying tower has to be filled with fresh desiccant beads (silica gel, orange gel). The condensate container at the intensive refrigerator has to be checked. The cryostat (intensive refrigerator) must be switched on. The data communication between the extraction apparatus and the measurement results capturing system has to be checked.

Upon connecting the filter holder to the exhaust gas hose a leakage test has to be performed. There to, the nozzle opening is sealed airtight, e.g. with a plug, and a negative pressure of up to 0.4 bar is generated via a pump. The leakage test is successful if a pressure change of less than 0.02 bar is observed over a period of 30 seconds. If the leakage test fails, the leaks have to be sealed and the test has to be repeated.

### 6.5.1.8 Installation of dust sampling probe

After a sufficient upstream stretch, the dust sampling probe is to be installed in the centre of the exhaust gas measuring installation at least 30 minutes before measurement starts. During installation it must be ensured that no dust deposits from the exhaust gas pipe enter the measuring opening. The electrical trace heating of the extraction pipe as well as the heating sleeve have to be installed and the thermostat must be switched on. The sampling temperature should be approx. 20 K above the exhaust gas dew point, typically at 75 °C.

## 6.5.2 Performing a measurement

The exhaust gas composition is determined and continuously captured via suitable gas analysis devices.

The speed measuring device with temperature sensor has to be installed and/or connected and the logging of the measured data must be started.

The automatic dust measuring system must be started via the respective software.

A suitable measuring file has to be created for the dust extraction. Such includes information like planar filter number, rinsing container number, nozzle diameter and exhaust gas pipe diameter at the measuring location. Furthermore, the meter readings of the gas meter, the air pressure, and the temperature at the gas meter have to be logged for standardisation purposes.

The dust measurement is performed automatically whenever the exhaust gas fan of the furnace is operating.

At the end of the measurement the dust sampling probe including filter holder has to be removed and stored to cool down.

### 6.5.3 Post-treatment of a measurement

#### 6.5.3.1 Dismounting of the dust sampling probe

After completion of the measurement, the trace heating units have to be switched off and disconnected and/or removed. The extraction hose and the thermocouple for recording the sampling temperature have to be disconnected.

The filter holder is unscrewed and the probe removed.

The filter holder is then disassembled and the filter sleeve is placed in the prepared numbered container. The clamping ring on the planar filter is removed and the planar filter is placed in the Petri dish from which it had been removed. Thereby the contents can be attributed unambiguously. Until the post-treatment of the filter media, they have to be stored dust-free and dry.

#### 6.5.3.2 Post-treatment of the filter media

The post-treatment of the filter media depends on the desired results. If the dust samples are to be analysed, no further post-treatment should be performed. Depending on the planned tests, it may be practical to freeze the sample immediately. However, the method for the determination of total dust will be presented here first, which also takes into account highly volatile dust components gravimetrically.

In contrast to the measurement specifications of VDI 2066-1 (for the gravimetric dust measurement in the exhaust gas of biomass furnaces) a baking-out and/or drying-out of the used filter at temperatures higher than the sampling temperature should be omitted. Otherwise, highly volatile components such as a portion of the polycyclic aromatic hydrocarbons (PAH), whose mass can play a significant role, may be lost for a complete assessment of the emissions from biomass furnaces.

The filters are stored for at least six hours in a desiccator located in the same room in which the weighing-out takes place. It has to be determined if a single weighing is sufficient. During the weighing-in, the climatic conditions (room temperature, humidity, and air pressure) have to be recorded.

In order to detect any climatic fluctuations potentially occurring in the weighing room, it is recommended that at least one weighed-in planar filter and filter sleeve per day be subjected to the same treatments as the filter media with loads. This can provide indications for handling or device errors during weighing. In case of discrepancies, the filter medium has to be weighed again. If necessary, photos should be taken.

Filter media post-treated in the above mentioned way are suitable for further material analyses after the weighing.

### 6.5.3.3 Post-treatment of the probe

The sampling probe and nozzle have to be rinsed after cooling down. Therefore, the probe is filled with distilled water and the liquid is distributed evenly in the probe before it is caught in a Petri dish. This procedure is repeated two to three times with isopropanol until the isopropanol is colourless. A separate rinsing container (e.g. Petri dish) is required for each dust probe. The rinsing container is subsequently dried at 105 °C in an explosion-proof drying cabinet. Once no more liquid is visible in the rinsing container, it can be placed in the desiccator. It then has to be stored for at least six hours in the desiccator and in the room of the weighing. A single weighing-in of the rinsing container is sufficient; the climatic conditions in the weighing room must be recorded once. For reference, a weighed-in rinsing container is partially filled with distilled water and acetone and subjected to the same treatment as the other rinsing containers.

Finally, the dust sampling probe is dried by blowing it out with particle-free and oil-free dry compressed air.

## 6.6 Analysis

### 6.6.1 Determination of absolute dust load and dust concentration

The determination of the dust deposited in the filter media and the dust sampling probe is performed according to VDI 2066-1.

Thus, the load is calculated via subtraction of the weighing-out and weighing-in of the filters and stuffed filter sleeves. A correction via the respective reference may have to be considered. The dust deposited in the dust sampling probe has to be rinsed out, evaporated, and taken into account for the calculation of total emissions (in case of multiple use of the probe, as a percentage share).

The output is the dust mass (mg) of the partial flow which was deposited during filtering. From the deposited dust mass, the dust concentration, with respect to the total sample volume of the partial flow sample, can be calculated (Equation A6.3).

$$c_{total\ dust} = \frac{m_{load}}{V_{partial\ flow, dr, N}} \quad (A6.3)$$

| <i>Symbols</i>             | <i>Definition</i>  | <i>Unit of measure</i>  |
|----------------------------|--|-------------------------|
| $c_{total\ dust}$          | Concentration of total dust at standard temperature and pressure | mg/m <sup>3</sup> (STP) |
| $m_{load}$                 | Mass of dust deposited in the filter media and in the probe      | mg                      |
| $V_{partial\ flow, dr, N}$ | Gas volume extracted at standard condition (273 K, 101.3 mbar)   | m <sup>3</sup> (STP)    |

The gas volume extracted at standard condition is determined by subtracting the gas meter reading after the measurement from the reading before the measurement and converting it to standard volume in accordance with (Equation 4). For the calculation, the temperature at the gas meter and the air pressure are needed.

$$V_N = \frac{(V_{final\ gas\ meter\ read-out} - V_{initial\ gas\ meter\ read-out}) \cdot p_{gas\ meter} \cdot T_N}{p_N \cdot T_{gas\ meter}} \quad (A6.4)$$

| Symbols                             | Definition  | Unit of measure      |
|-------------------------------------|---|----------------------|
| $V_N$                               | Volume extracted during measurement with the partial flow at standard condition, 273 K, 1013 mbar | m <sup>3</sup> (STP) |
| $V_{final\ gas\ meter\ read-out}$   | Value at gas meter after the measurement  | m <sup>3</sup> (STP) |
| $V_{initial\ gas\ meter\ read-out}$ | Value at gas meter before the measurement   | m <sup>3</sup> (STP) |
| $p_{gas\ meter}$ , $p_N$            | Pressure (mbar) at the gas meter (ambient pressure), standard pressure 1013 mbar                  |                      |
| $T_{gas\ meter}$ , $T_N$            | Temperature (K) at the gas meter (ambient temperature), standard temperature 273 K                |                      |

### 6.6.2 Determination of the total dust load

Calculating the total dust load for the respective investigation period (here 24 h) can be done via extrapolation from the average total dust concentration and the total exhaust gas volume during that period.

$$m_{total\ dust,\ 24h} = c_{total\ dust} \cdot V_{partial\ flow,\ dr,\ N,\ 24h} \quad (A6.5)$$

| Symbols                            | Definition   | Unit of measure          |
|------------------------------------|--|--------------------------|
| $m_{total\ dust,\ 24h}$            | The total dust load during the investigation period; here 24 h   | mg/duration under review |
| $c_{total\ dust}$                  | Total dust concentration at standard condition   | mg/m <sup>3</sup> (STP)  |
| $V_{partial\ flow,\ dr,\ N,\ 24h}$ | Measured (and/or calculated) total exhaust gas volume at standard condition dry during the investigation period; here 24 h | m <sup>3</sup> (STP)     |

### 6.6.3 Determination of the dust concentration in mg/m<sup>3</sup> (STP) @ 13% O<sub>2</sub>

If a specification of the average dust concentration at a reference oxygen content of 13 % O<sub>2</sub> is desired, the dust concentration at standard conditions must be put in relation to a residual oxygen content of 13 %.

For that the average oxygen concentration during the whole measurement is relevant. The conversion factor is calculated in accordance with equation (A6.6):

$$f_{O_2} = \frac{(0.21 - c_{O_2,ref})}{(0.21 - c_{O_2,average})} \quad (A6.6)$$

| <i>Symbols</i>    | <i>Definition</i>  | <i>Unit of measure</i> |
|-------------------|--|------------------------|
| $f_{O_2}$         | Dimension-free conversion factor from averaged oxygen content measured to reference oxygen content | -                      |
| $c_{O_2,ref}$     | Reference oxygen content; here 13 %  | -                      |
| $c_{O_2,average}$ | Average oxygen content during the entire measurement   | -                      |

Finally, the dust concentration must be multiplied with the conversion factor.

$$c_{total\ dust@13\%O_2} = c_{total\ dust} \cdot f_{O_2} \quad (A6.7)$$

## 6.7 Additional notes

For a reliable determination of the desired measurement results, proper operation of the entire dust sampling process is important. Similarly, the filter media have to be weighed-in and weighed-out with great diligence. The temperatures selected during the dust sampling and thermal post-treatment of the filter media are of critical importance for the values determined. Therefore, these values always have to be given.





[www.energetische-biomassenutzung.de](http://www.energetische-biomassenutzung.de)

ISSN (online): 2364-897X

ISBN 978-3-946629-28-3

Funded by



Federal Ministry  
for Economic Affairs  
and Energy

Project management



Programme support

