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TECHNICAL REPORT

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Advanced particle measurement equipment at SINTEF Energy Research

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RESULT (summary)

Particle concentration and size distribution, in addition to their composition, are essential information not only because of environmental and health concerns but also in order to be able to avoid deposition on heat transfer surfaces which may result in corrosion, fouling and subsequent efficiency reduction and cost increase in biomass combustion plants. For the study of particle concentration and size distribution an ELPI can be used. In this device, already well-developed impactor technology is combined with particle charging and electrical detection allowing accurate real-time measurements and automatic operation. The collection of the size classified particles in the different impactor stages also makes subsequent chemical analysis possible.

Gravimetric particle and tar sampling is a relative simple method of measuring particle and tar concentrations against a limit value. Hot filter sampling enable the collection of solid particles by avoiding the condensation of organic compounds, i.e. tars. The STL dust sampler is provided with a heated filter system for temperatures up to 350°C. This temperature is sufficient for the separation of solid particles and tars. The heated filter system is connected to a tar condensation unit. Tars are the main barrier so far for attractive gasification of biomass because they often cause operational problems such as blocking of gas cooler, filter elements and engine suction channels.

The ELPI and STL setups described in this report can be used in connection with different experimental setups where particle concentration, size distribution and properties are in focus. This includes the multi-fuel reactor setup at SINTEF Energy Research.

KEYWORDS

SELECTED BY AUTHOR(S)	Biomass combustion	Particle size distribution
	ELPI	Tar

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1   INTRODUCTION

Combustion of biomass is the best known biomass conversion process for energy purposes but has some disadvantages when it comes to conversion of fuels with low quality. This results in corrosion and fouling and increased emissions levels, which require more advanced flue gas cleaning. Combustion of biomass in large scale applications results in emissions mainly from complete conversion through automatic process regulation. Emissions are mainly particles, nitrogen oxides, sulfur oxides and hydrogen chloride. Particles are often carriers for harmful substances and often are deposited at boiler surfaces and cause fouling and corrosion. Sampling of particles can be done by using a filter which collects particles above the filter pore size. This method is mainly used when particles are measured against an emission limit value and for chemical analysis.

Gasification of biomass gains more and more attention and seems to be a promising conversion process for biomass. However, there are still some problems which need to be overcome. The main barrier so far is impurities in the product gas, and in particular tars. Depending on the application of the producer gas the impurities cause operational problems in the downstream equipment such as blocking of gas cooler, filter elements and engine suction channels. Sampling of particles requires high filter temperatures to avoid the condensation of tars. Tars are collected after removal of the particles by condensation, absorption or adsorption.

The sampling and analysis of particles and tars is a basis for the research on fuel properties and process conditions to reduce the amount or change the composition of particles and tars, which can make combustion and gasification of biomass more attractive.

2   PARTICLE AND TAR EMISSIONS FROM BIOMASS CONVERSION PROCESSES

2.1   Particle emissions

Particle emissions are one of the main problems hindering the development of biomass as an alternative fuel for heat and electricity production. In addition to the environmental and health concerns, in industrial applications, particle emissions from biomass combustion often cause corrosion and fouling of heat exchange surfaces, reducing the efficiency and increasing the maintenance costs of the installation.

2.1.1   Origin

Particle emissions can originate from both complete and incomplete combustion. When the combustion is efficient, almost all organic material is converted to carbon dioxide and water, and few particles are formed (mostly ashes from the inorganic material in the fuel). In contrast, during poor combustion conditions, a lot of particles from incomplete combustion are formed, increasing drastically the total amount of emitted particles.

a. Particle emissions from complete combustion – Ashes

Ashes are formed during the combustion or gasification of the inorganic material in the fuel. They can leave the system as bottom ashes, fly-ashes or vapour.

Fly-ashes consist of:

- Coarse fly-ashes (particles with an aerodynamic diameter > 1 μm), which result from the entrainment in the flue gas of ash and fuel particles from the fuel bed.

- Aerosols (particles with an aerodynamic diameter < 1 μm), which are a result of chemical reactions mainly between K or Na and Cl or S, released from the fuel to the gas phase, and subsequent nucleation and condensation processes.[1]

b. Particle emissions from incomplete combustion

Particle emissions from incomplete combustion are unburnt carbon in the form of soot, char or condensed heavy hydrocarbons (tar droplets). Soot is an agglomeration of particles (often approximated by elemental carbon, but also containing hydrogen), which is a result of a local lack of oxygen in the flame zone and/or local flame extinction. Unburnt char fragments may be entrained in the flue gas due to their very low specific density, especially at high flue gas flow rates.[1] Condensed heavy hydrocarbons are usually the main contributor to particle emissions in applications with poor combustion conditions like residential wood combustion.

2.1.2   Health effects of particulate matter

Particulate Matter (PM) describes the sum of airborne solid particles and droplets. The US Environmental Protection Agency groups particle pollution into two categories:

- "Inhalable coarse Particles" ($PM_{10-2.5}$) such as those found near roadways and dusty industries range in diameter (Aerodynamic diameter. A particle of an arbitrary shape and density has an aerodynamic diameter corresponding to a spherical particle with a density equal to water and the same settling velocity as the arbitrary particle) from 2.5 to 10 μm.

- "Fine Particles" ($PM_{2.5}$) such as those found in smoke and haze have diameters less than 2.5 μm. $PM_{2.5}$ is referred to as "primary" if it is directly emitted into the air as solid or liquid particles, and is called "secondary" if it is formed by chemical reactions of gases (mainly sulphur dioxides and nitrogen oxides) in the atmosphere. Fine particles are mostly originating through combustion processes.

The sizes of particulate matter are illustrated in Figure 2-1:

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[1] International Energy Agency. The Handbook of Biomass Combustion & Co-firing, 2002.
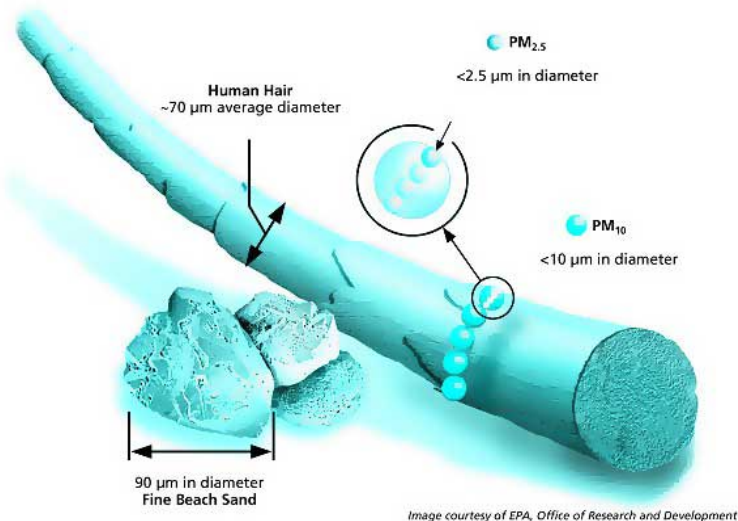
Figure 2-1: Comparison of particulate matter with human hair

Atmospheric particulate matter causes serious effects on human health. Several epidemiological studies show a relation between long-term exposure to particulate matter and increased hospitalization for respiratory and heart disease, lung cancer death rates, reduced lung function, exacerbation of asthma, etc. Particles are also carriers of toxic substances like benzene or PAH, with carcinogenic effects. Particle emission is characterised by its mass concentration and particulate matter regulations are mass based, however, the main determinant of health effects is the particle size. PM_{10} is considered to be the inhalable fraction, since larger particles are usually filtered in the nose or throat, but the smallest particles ($PM_{2.5}$) are of greatest concern, since they can penetrate deeply into the human respiratory system inflaming the lungs alveoli. Fine particles also remain longer in the atmosphere.

2.1.3 Other problems associated with particle emissions

The amount of coarse fly ashes emitted in industrial applications is usually larger than in small-scale applications like wood stoves. Most of the particles emitted from small-scale wood combustion are fine particles with a diameter smaller than 1 μm. The reason is that the flue gas flow in large-scale applications is considerably higher and therefore more ashes are withdrawn from the fuel bed to the flue gas. However, big particles are usually not an emission problem in these plants since they are removed by advanced cleaning equipment (such as cyclones, bag filters and electrostatic precipitators). In these plants the main problem related to particles is the corrosion and fouling they cause at heat exchange surfaces and in other downstream equipment.

Particle concentration and size distribution, in addition to their composition, are essential information not only because of environmental and health concerns but also in order to be able to avoid deposition on heat transfer surfaces which may result in corrosion, fouling and subsequent efficiency reduction and cost increase.

For the study of particle concentration and size distribution an ELPI can be used. In this device, already well-developed impactor technology is combined with particle charging and electrical

detection allowing accurate real-time measurements and automatic operation. The collection of the size classified particles in the different impactor stages also makes subsequent chemical analysis possible.

2.2 Tars

2.2.1 Definition and Composition of tars

The main components of the gasification product gas are CO, H_2, CO_2, CH_4, other light hydrocarbons, H_2O, N_2 and in addition organic (tar) and inorganic (H_2S, HCl, NH_3, alkali metals) impurities and particulates.

The tar fraction contains condensable hydrocarbons from relatively low molecular weight to high molecular weight polynuclear aromatics. The tars are difficult to remove from the product gas. Tars in addition tend to polymerize into more complex structures and cause fouling and lower e.g. the total efficiency in heat exchanger.[2]

At the moment no common definition of tars exists. Often an operational definition is given. Different operational conditions result in varying product gases for different applications. Therefore, tars have different composition depending on the conversion processes, with unlike characteristics and require appropriate collection methods.[2] Even though tars have no common definition; tars are considered as the condensable fraction of organic gasification products which are mainly aromatic hydrocarbons and benzene. The European project on standardization of measuring tar agreed in the Würzburg protocols on the definition that tars are all organic hydrocarbons higher than benzene.[3] The protocol was developed to support the standardization of tar sampling methods. So far it is not widely in use.

The formation of tar is a complex process including a series of reactions, and depends highly on the process condition and reactor design. The formation and temperature dependence of tars are summarized in Figure 2-2. Primary tars, mixed oxygenates, are formed at relatively low temperature. These primary products are converted to phenolic compounds, and further to aromatic hydrocarbons at higher temperatures.

Mixed Oxygenates		Phenolic Ethers		Alkyl Phenolics		Heterocyclic Ethers				
Mixed Oxygenates	→	Phenolic Ethers	→	Alkyl Phenolics	→	Heterocyclic Ethers	→ PAH	→	Larger PAH		
400°C		500°C		600°C		700°C	800°C		900°C		

Figure 2-2: One tar formation pathway proposed by Elliot[2]

Depending on the molecular weight, tars have been divided into five groups which are summarized in Table 2-1.

[2] Li, C., Suzuki, K., Tar property, analysis, reforming mechanism and model for biomass gasification - An overview, Renew Sustain Energy Rev, 2008.

[3] H.A.M. Knoef, Handbook Biomass Gasification, Biomass Technology Group, 2005.

Table 2-1: Overview of different considered tar classes[2]

Tar class	Class name	Property	Representative compounds
1	GC-undetectable	Very heavy tars, cannot be detected by GC	Determined by subtracting the GC-detectable tar fraction from the total gravimetric tar
2	Heterocyclic aromatics	Tars containing hetero atoms; highly water soluble compounds	Pyridine, phenol, cresols, quinoline, isoquinoline, dibenzophenol
3	Light aromatic (1ring)	Usually light hydrocarbons with single ring; do not pose a problem regarding condensability and solubility	Toluene, ethylbenzene, xylenes, styrene
4	Light PAH compounds (2–3rings)	2 and 3 rings compounds; condense at low temperature even at very low concentration	Indene, naphthalene, methylnaphthalene, biphenyl, acenaphthalene, fluorene, phenanthrene, anthracene
5	Heavy PAH compounds (4–7rings)	Larger than 3-ring, these components condensate at high-temperatures at low concentrations	Fluoranthene, pyrene, chrysene, perylene

Once tars leave the gas phase and condense in downstream equipment problems occur. It is considered that not only the concentration of the tars in the flue gas is decisive for condensation but also the composition of the tars. Condensation curves of different tar classes are illustrated in Figure 2-3. Since the dew point for a low concentration of class five tars lies above the dew point for class 2 and class 4 tars at a high concentration, it can be assumed that these tars are most important for the total dew point of the tars. The dew point for class 1 tars is not included since the composition is not known, however it is supposed that these tars start to condense at 300-350°C. As mentioned in the table above is class 3 tars unimportant in the context of tar condensation since the dew point even at high concentrations lies below 0°C.
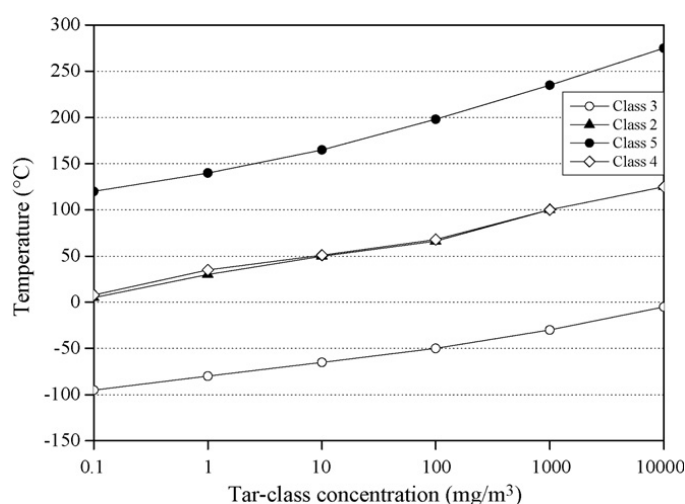
Figure 2-3: Dew points of different tar classes in relation to the concentration[2]

The concentration and composition of tars and particles depends on the reactor design and process conditions such as gasification agent, temperature and pressure. An updraft gasifier produces tars amount in the range of 50-150 g/m_n^3 and PM of 0.1-0.5 g/m_n^3.[4] In contrast, a downstream gasifier produces 0.5-2 g/m_n^3 tars and PM of 0.1-1 g/m_n^3. Higher temperatures decrease the tar yield. In a steam-blown fluid-bed gasifier the tar amounts to 15% at a temperature of 600°C and decrease

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[4] Sampling and analysis of tar and particles in biomass producer gases – Technical Report, Prepared under CEN BT/TF 143 "Organic contaminants ("tar") in biomass producer gases", July 2005.

down to 4% at 750°C. In an oxygen-blown fluid-bed gasifier the tar content decreases from 4.3% at 750°C to 1.5% at 810°C. An entrained flow gasifier produces very low concentration of tars with 1% at the very high temperature of 1000°C.[2]

2.2.2 Sampling tars

The most common sampling, treatment and analysis option are shown in Figure 2-4.
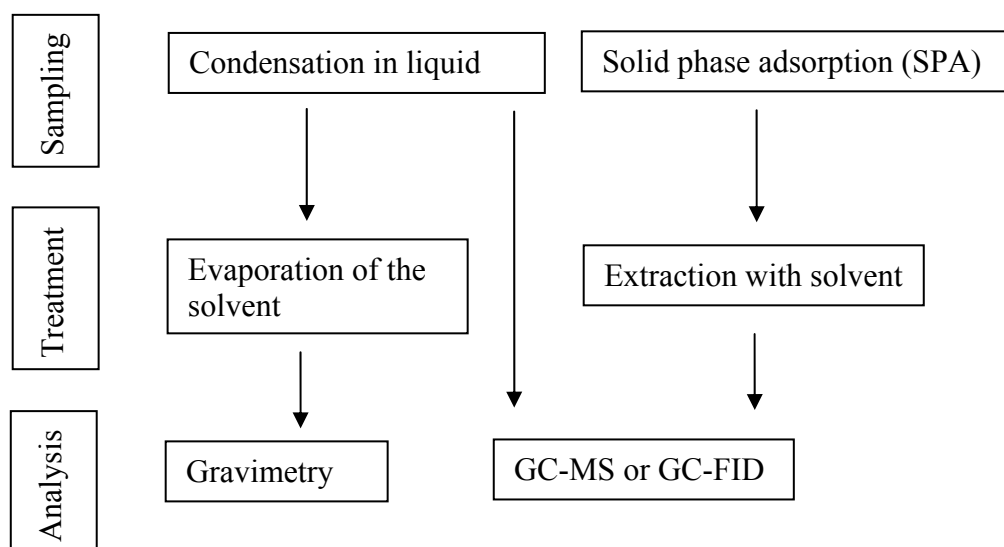

Figure 2-4: Conventional off-line method for tar analysis

Sampling
Sampling can be done by either condensation where the gas is cooled down below the dew point, adsorption by dissolving the tars in another bulk phase or absorption by which the tars solute on a surface of a liquid or solid.

Treatment
If the tars are condensed in a liquid the solvent needs to be evaporated. Tars adsorbed on a surface require the extraction with a solvent.

Analysis
The remaining fraction of tars can be determined gravimetrically, after carefully evaporating the solvent. The collected tars are weighed. This is a very well known and widely used method. The tars in the solvent can be analyzed by gas chromatography.[5]

The European project on standardization of measuring tar resulted in a draft of the standard and a technical background report. The method describes the four steps; gas preconditioning (atmospheric/pressurized gasification), particle sampling, tar collection and volume measuring, and after handling of the four parts. The tars, after removing the particles on a hot filter, are collected in isopropanol and finally analyzed by GC. A set of 6 impinger bottles or special designed equipment e.g. a "Peterson column" is used for trapping the tar.

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[5] http://www.btgworld.com/index.php?id=21&rid=8&r=rd

In opposite to the above mention off-line methods, also online methods were developed. A semi-online method was developed by IVD Stuttgart (Institut für Verfahrenstechnik, University Stuttgart). This method for high tars concentration compare the signals of a FID (flame ionization detector) in the raw gas and the gas after a tar filter.

BTG (Biomass technology group) and KTH (Royal University of Technology) developed a real online method based on the principle of photo-ionization with the help of PID (photo ionisation detector) for low tar concentrations.[5] The detector is more sensitive compared to conventional TCD (thermal conductivity detector) and FID, however, more expensive according to [2]. BTG however claims that this method is relatively cheap mainly because of reduced laborious sample treatment and analysis.[5]

3 ELECTRICAL LOW PRESSURE IMPACTOR

3.1 Introduction

An Electrical Low Pressure Impactor (ELPI) can be used to measure real-time particle size distribution and concentration in combustion flue gases. In this device, already well-developed impactor technology is combined with particle charging and electrical detection allowing accurate real-time measurements and automatic operation. The different impactor stages operate below the atmospheric pressure to be able to capture smaller particles than a normal cascade impactor. Due to its fast response the ELPI is commonly used in applications where rapidly changing particle concentrations or size distributions are expected. The instrument is also suitable for applications where wide particle size range measuring capability is needed. The collection of the size classified particles in the different impactor stages also makes possible subsequent chemical analysis and gravimetric measurements. An example of a particle size distribution measurement performed with the ELPI is shown in Figure 3-1.
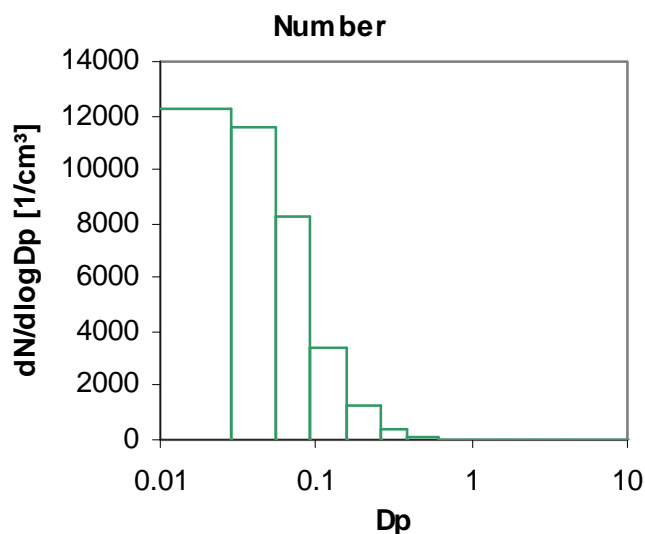

Figure 3-1: Example of particle size distribution measurement from ELPI

3.2 Equipment description and operating principle

In this section the Dekati Standard 10 l/min ELPI will be described. The gas needs to be conditioned/diluted before entering the sampling instrument. For this purpose a double diluter setup is used. Before describing the ELPI the dilution system will be explained in detail in the following section. The ELPI characteristics are presented in Table 3-1.

Table 3-1: Characteristics of Standard 10 l/min ELPI

Particle size range	30 nm – 10 μm*
Number of size classes	12
Nominal sample flow rate	10 l/min
Lowest stage pressure	100 mbar
Time resolution	2-3 s
Weight	35 kg
Maximum temperature	60°C

* With the filter stage accessory the range of the instrument is increased to 7 nm – 10 μm

3.2.1 The Double diluter system

The double diluter setup is shown in Figure 3-2. The system consists of two Dekati diluters. The first diluter incorporates a heating unit to avoid nucleation and condensation of volatile compounds. The flue gas is then cooled in the second diluter (which operates at ambient temperature) before entering the ELPI. When the flue gas reaches the second stage condensation is no longer an issue since the flue gas has already been diluted once.
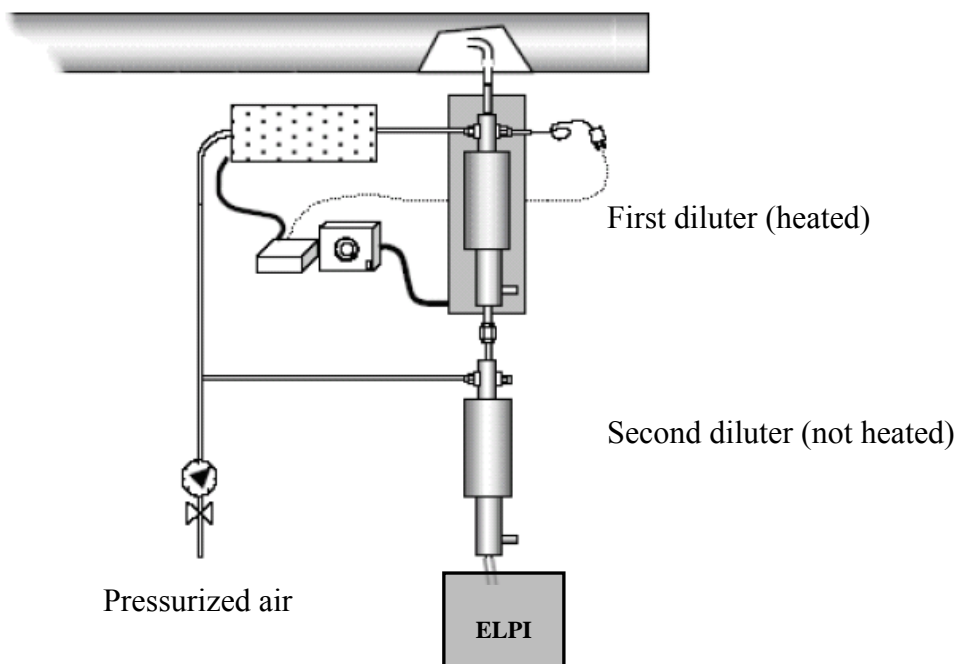

First diluter (heated)

Second diluter (not heated)

Pressurized air

ELPI

Figure 3-2: Double diluter system

The operation principle of the diluters is based on ejection dilution. The pressurized air enters the dilution chamber just above the nozzle, pulling out the sample gas from the ejector, thus instantly diluting the flue gas and eliminating the need to use any other expensive devices. The dilution chamber is illustrated in Figure 3-3.
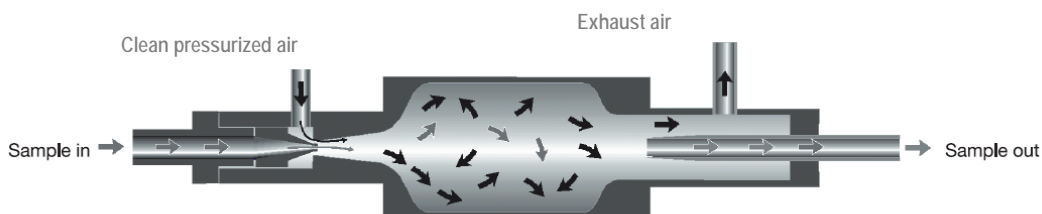

Figure 3-3: Dilution chamber

The maximum operating temperature for the Dekati diluters is 450°C but due to cooling effects in the probe and cooling by dilution air, sample gas temperatures up to 600°C can be handled. The outlet pipes of the diluters must be connected back to the stack, thus achieving a pressure balance in the diluters that avoids the influence of "background" pressure changes on the dilution ratio. The diluter specifications are detailed in Table 3-2. The real dilution ratio is calculated through calibration and its value is included in the instrument data sheet. The nominal dilution ratio for a Dekati diluter is specified at an operating temperature of 20°C and a diluter inlet pressure of 1013 mbar.

Table 3-2: Specifications of Dekati Diluter DI-1000

Sample air flow (inlet)	Approx. 7 l/min
Diluted sample flow (outlet)	60 l/min
Nominal dilution ratio	1:8
Dilution air pressure	2 bar
Operating temperature	0-450°C (600°C)

3.2.2 Operating principle of the ELPI

After being conditioned the flue gas enters the ELPI with the appropriate temperature. The ELPI operating principle is presented in Figure 3-4 and can be divided in three main functions: particle charging in a unipolar corona charger, size classification in a cascade impactor and electrical detection in a multichannel electrometer.
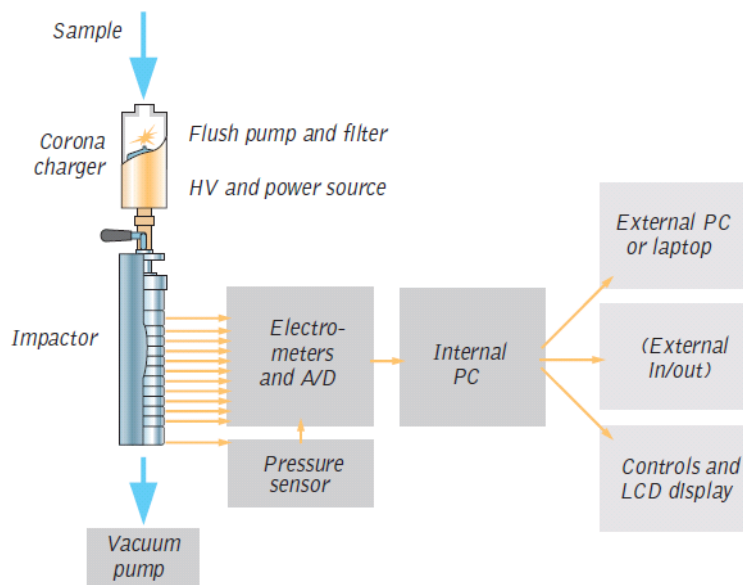
Figure 3-4: Operating principle of the ELPI

First, the sample gas enters a corona charger where the particles are charged to a well defined level establishing a relation between particle size and charge. The sample gas is then forced to flow through the cascade impactor where the particles are classified into the different impactor stages depending on their aerodynamic diameter. The different impactor stages are electrically insulated from each other with rings of dielectric material (PTFE). Finally, multichannel electrometers are used to measure the electric charge carried by the particles into each stage. The electric current measured by the electrometers is proportional to particle number concentration and size. The relation between size and charge established in the corona charger is then used to convert the electric current measured by the electrometers into real-time number concentrations for each stage.

3.2.3 The Corona Charger

The diode type corona charger consists of a cylindrical tube with a center corona tungsten electrode. The device creates a strong electric field that charges the particles when they pass through it. After the charging zone, a simple mobility analyzer is used as an electrical trap to remove residual molecular ions and the charged particles that have smaller size than the measuring range of the instrument. These small charged particles would produce error signals by getting collected inside the impactor stages due to diffusion[6]. The "charging efficiency" characterizes the charging process giving the electrical current carried by the charged particles leaving the charger, divided by the particle number concentration in the sampled gas. The charging efficiency, which increases with particle size, is used to convert the current measured by the electrometers into particle number concentration for each of the impactor stages. The design of the charger features simple construction and high charging efficiency.

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[6] Marjamäki, M., Keskinen, J., Chen, D.-R., and Pui, D. Y. H. Performance Evaluation of the Electrical Low Pressure Impactor (ELPI), *J. Aerosol Sci.* 31:249-261, 2000.

3.2.4 The Impactor

The ELPI main component is a 13-stage cascade low pressure impactor. An impactor has two co-linear plates; one acts as a collection surface for the particles, the other one has a small nozzle or nozzles in it to control the flow velocity. The classification of the airborne particles is achieved by forcing the sample gases through jet orifices of successively lesser diameter and increasing gas jet velocities. The jet orifices are drilled symmetrically in rings around the center of each stage. After the sample passes through the nozzles it is turned sharply before the collection plate; particles larger than the stage cut diameter cannot follow the flow stream lines and are inertially impacting on the collection plate. Particles smaller that the stage cut diameter continues to the following impactor stages where they are further size classified and collected. The particle cut size of each impactor stage is defined as the particle aerodynamic diameter which obtains a collection efficiency of 50% in that stage, also called $D_{50\%}$. A detailed sketch of the impactor and the impactor nominal stage cut diameters are shown in Figure 3-5. The real cut diameters are determined through calibration and are included in the impactor data sheet.

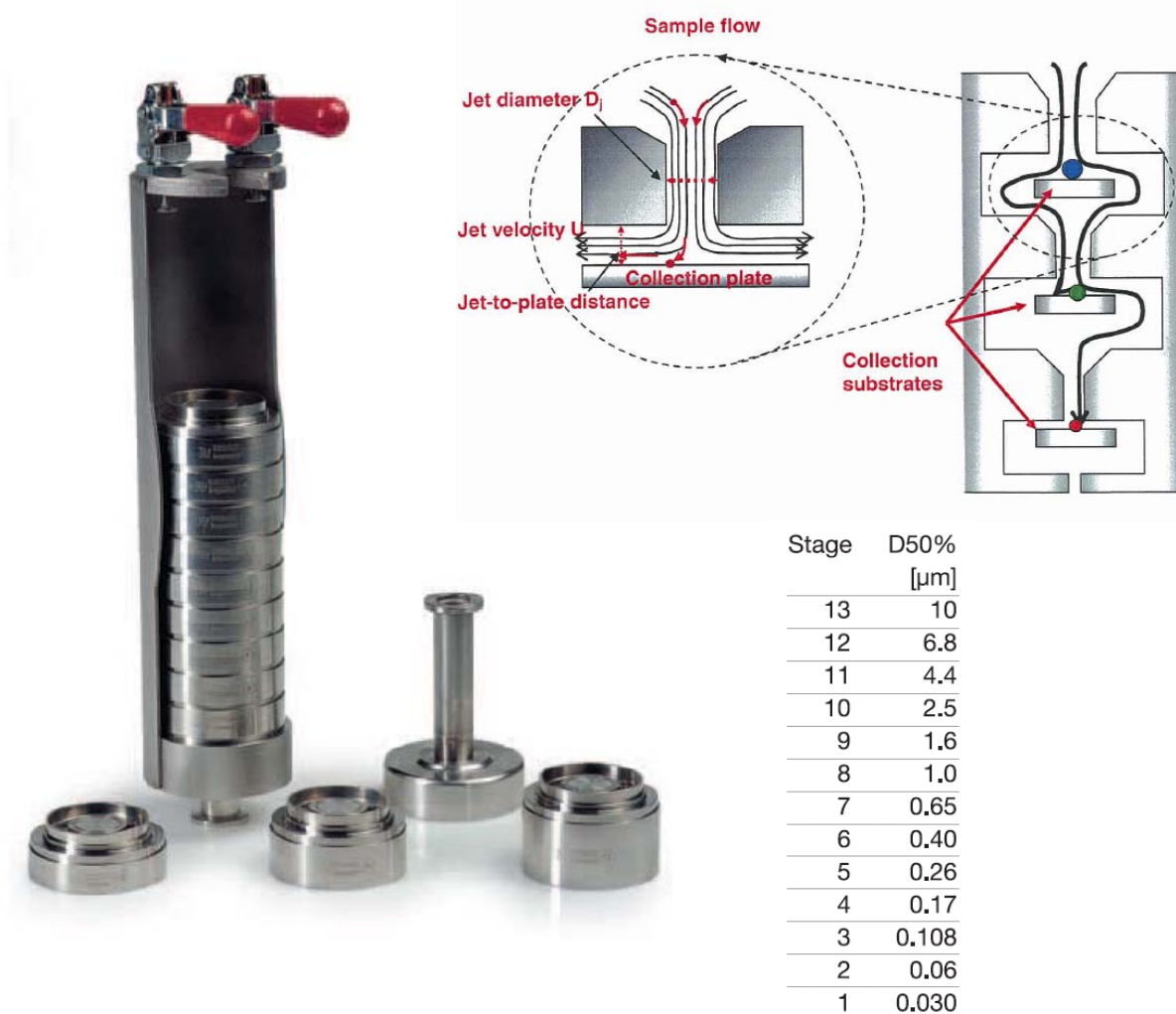| Stage	D50% [µm]
13	10
12	6.8
11	4.4
10	2.5
9	1.6
8	1.0
7	0.65
6	0.40
5	0.26
4	0.17
3	0.108
2	0.06
1	0.030

Figure 3-5: Impactor, detail of collection plates and nominal stage cut diameters

The substrates that will be used as particle collection surface are attached to the collection plates with a holder ring and have a diameter of 25 mm. The flow through the ELPI impactor stays constant as long as the impactor does not leak, the impactor nozzles are not clogged and 100 mbar is maintained entering the first impactor stage. When 100 mbar is maintained entering the first impactor stage, the first stage jet plate acts as critical orifice and controls the flow. Therefore, no additional flow control is required.

3.2.5   The Multichannel Electrometer

As shown in Figure 3-6 the outer surface of each impactor stage is electrically connected to an electrometer input cable by a spring-loaded contact on a removable connection box. The current signals are measured in parallel mode using a monolithic electrometer amplifier for each impactor stage[7]. A measured high current corresponds to a high particle population.
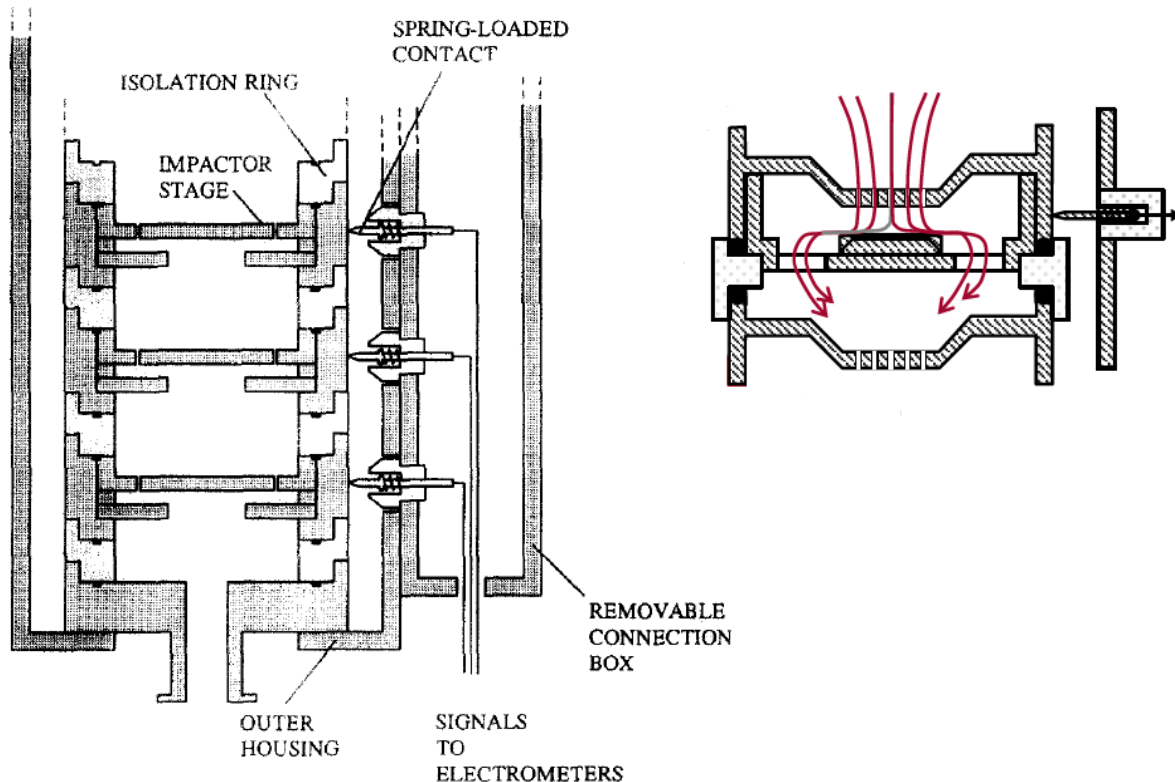
Figure 3-6: Detail of impactor stages

3.2.6   Other components

A vacuum pump is needed to sample the appropriate gas flow through the instrument and obtain the required operating pressure at the impactor outlet (which is controlled by an external pressure gauge). Furthermore, a flush pump and a high-voltage power supply for the charger are included.

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[7] Keskinen, J., Pietarinen, K., and Lehtimaki, M. Electrical Low Pressure Impactor, *J. Aerosol Sci.* 23:353-360, 1992.

The flush pump is used to adjust the offset levels of the electrometers by pumping HEPA-filtered air through the instrument (zeroing).

3.2.7 Control System and Software

During the test-run the ELPI is completely controlled. The instrument status and measurement parameters can be checked in the computer interface. The measured data is saved in the computer and the ELPIvi software is used to display useful information including real-time particle size distribution and total particle concentration. The appearance of the data display program window is shown in Figure 3-7. Other windows show configuration values and measurement information.

Figure 3-7: ELPIvi program window

3.3 Aerodynamic diameter and ELPI calculation

Particles are collected in the impactor according to their aerodynamic diameter. A particle of an arbitrary shape and density has an aerodynamic diameter corresponding to a spherical particle with a density equal to water and the same settling velocity as the arbitrary particle.

Figure 3-8: Aerodynamic diameter

After the particles are collected into the corresponding impactor stage a calculation is carried out by the ELPI software to convert the current values measured by the electrometers into selected size distributions. The first step of this procedure converts the current values measured in each impactor stage into number concentration of particles in that stage. This is done using the relationship between number concentration and induced current established in the charger:

$$n_i(D_m) = \frac{I_n}{E_{Ch}(D_m)}$$

Where:

$D_{m,i}$ is the geometric midpoint of the channel: $D_{m,i} = \sqrt{D_{p,i} * D_{p,i+1}}$

I_n is the current measured by the stage electrometer

E_{Ch} is the charger efficiency function, which is obtained during calibration and is proportional to the particle size

n_i is the number concentration of the particles collected in the stage

The software performs this calculation for each of the impactor stages obtaining the complete number to size distribution of particles. The calculation also provides several other distributions. The mass distribution is obtained using the following equation:

$$n_m(D_m) = \rho_p \frac{\pi}{6} D_m^{\,3} n_i(D_m)$$

Since the density of the particles (ρ_p) is an unknown value which is assumed to be equal to the density of water (1 kg/l), the calculated mass distributions are less accurate than the number distributions.

3.4   Post-analysis of collected samples

After running a test, the particles are classified according to their size in the different impactor stages, enabling chemical analysis of each size fraction individually. When chemical analysis is wanted, suitable substrates must be used. Polycarbonate substrates are suitable for most chemical analysis, however, it is important to check the compatibility of the substrates with the analysis laboratory. The grease used on the substrates to prevent bouncing (Apiezon-L) also needs to be compatible. The samples must be handled with special care (preferably in a clean room) to avoid contamination. The samples should be stored in a cold, dry and dark place. It is important that the amount of particles collected is sufficient for the analysis. The collection plates should be cleaned carefully before adding the collection substrates.

Using wet chemical analysis methods the inorganic composition of the particles collected in the different impactor stages can be analyzed. The samples are first leached in water over night. Then they are analyzed with ion chromatography and a conductivity detector (TCD) to determine the content of chlorine and sulphur. After this, the samples are dried in an oven. The remaining sample is then dissolved in nitric acid (HNO_3), fluorohydric acid (HF) and hydrogen peroxide (H_2O_2), followed by analysis with ICP-OES (Inductive Coupled Plasma with Optical Emission Spectroscopy) or Mass Spectrometry (ICP-MS) for determination of e.g. K, Na, Ca, Mg, P, Al, Fe, Ba, Mn and Si. The amount of sample needed to perform the analysis is at least 1 mg per collection substrate.

The determination of the compounds forming the particles can be performed using time-of-flight secondary ion mass spectrometry (TOF-SIMS).

While wet chemical analysis is applied to provide information about the chemical composition of particle collectives, scanning electron microscopy (SEM) has proven a suitable method for gaining information about the shape and morphology of single aerosol and fly-ash particles. Equipped with energy dispersive X-ray spectrometry (EDX) it can also give information on the elemental composition of the particles. When using polycarbonate substrates the peaks obtained in the element analysis for C and O are originated from the polycarbonate in the substrate. When SEM-EDX analysis is wanted the collection substrates should be used without grease, because the grease is not stable under the electron beam. The amount of sample needed to perform the analysis is about 1 mg per collection substrate. If polycarbonate foils are used and around 0.1 mg of sample is collected per collection substrate the analysis can be performed directly in the polycarbonate foils.

The mineralogy of the fly ash samples can be studied with X-Ray powder Diffraction (XRD) for the identification of the crystalline components.

Total carbon and organic carbon content in the samples can be also determined.

3.5 Possibilities and applications

The ELPI measures particle size distribution and concentration in a wide particle size range. It can be used in measurements of aerosols from different sources, including atmospheric, combustion and workplace aerosols. The parallel current measurement provides fast response capability which makes the ELPI suitable for applications where varying particle concentrations or size distributions are expected. The collection of the size classified particles in the different impactor stages also allows subsequent chemical analysis and gravimetric measurements. By switching off the charger, the ELPI can be also used as an electrometer for particle charge distribution measurements. Some common applications of the ELPI are:

- Automobile exhaust measurements

- Combustion studies

- In-stack measurements

- Indoor and outdoor air quality measurements

3.6 Limitations and restrictions

The ELPI has a limited particle size resolution. The maximum operating temperature for the Dekati diluters is 450°C but due to cooling effects in the probe and cooling by dilution air, sample gas temperatures up to 600°C can be handled.

When using the filter stage accessory, the measuring range of the instrument is increased to 7 nm – 10 μm. However, one of the stages has to be removed to make room for it. Therefore, one of the upper impactor stages is lost in this configuration. Furthermore, since the electrical trap fitted after the charger removes also small particles that could be collected by the filter stage in the new configuration, the trap voltage is set to zero when using the filter stage.[8]

Particle losses always occur in the ELPI charger when the charger has been switched on. These losses are taken into account in the ELPI calculation (in the charging efficiency curve) during a standard ELPI measurement. However, when gravimetric measurements are performed these losses are not considered in the calculation and they could influence the final result. To avoid this, the charger must be switched off when performing gravimetric measurements with the ELPI. This means that during gravimetric measurements the real-time measurement feature of the ELPI is sacrificed in order to obtain more accurate mass samples.

The detection of particles in the coarse size fraction may be problematic since the number concentration of these particles can be relatively low and thus their charge may not be enough to be properly measured by the electrometers. In [9] the charge accumulated in the ELPI stages with a

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[8] Maricq, M. Matti, Xu, Ning and Chase, Richard E. Measuring Particulate Mass Emissions with the Electrical Low Pressure Impactor, Aerosol Science and Technology, 40:1, 68-79, 2006.

[9] Hillamo, R., Mäkelä, T., and Kerminen, V.-M. Electrical Low Pressure Impactor (ELPI) in Atmospheric Aerosol Studies. Finnish Meteorological Institute, Helsinki, Finland, 1992.

cut diameter > 2 µm was often close to the noise level of the electrometers. This could influence an accurate total mass concentration measurement.

As mentioned before, the impactor stage cut diameters are defined as the size of the particles that would be collected with 50% efficiency. Ideally, the collection efficiency curves would be perfectly vertical, meaning that all particles bigger than the cut diameter of the stage would be entirely collected and that the smaller particles would continue to the lower stages. In reality, some of the bigger particles are collected in lower stages through particle bounce and blow-off (high collection efficiency in the lower stages) and some of the smaller particles are collected in the upper stages through diffusion, obtaining collection efficiency curves that look similar to the one shown in Figure 3-9. Particle bounce is reduced by a large extent by greasing the collection substrates.
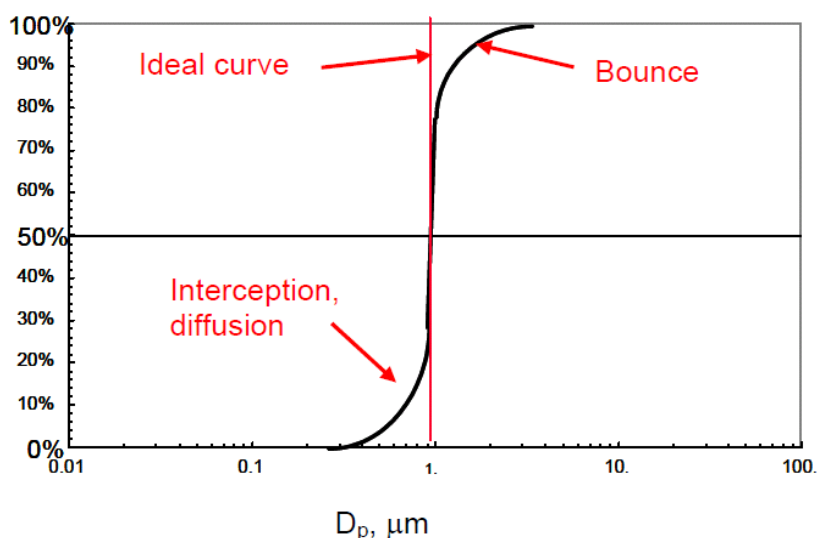
Figure 3-9: Collection efficiency curve

Diffusion deposition of fine particles causes particles smaller than the cut diameter of an impactor stage to be collected to some extent. As the concentration of particles in the gas in the vicinity of the collection plate approaches zero, diffusion creates a net flow of particles towards the collection plate. This phenomenon is characteristic to all impactors and does not depend on the detection method. In gravimetric impactors this phenomenon has negligible effect on the result since the mass of these small particles is often insignificant compared to the actual impacted mass. However, when the particles are detected by their electrical charge, the effect can be significant due to the better sensitivity of the charge measurement. Although the charging efficiency of these particles is low their number concentration is high. The error caused by the diffused particles is often insignificant when looking at number concentrations. However, the error in mass concentration is directly proportional to the third power of the diameter of the particle, meaning that even a small current signal can have big influence on mass results in the upper stages, obtaining high mass concentrations for aerosols without large particles.

Fine particle losses for the ELPI were studied in[10], where electrostatic particle losses are also investigated. The study found losses of less than 2% in the measuring range of the instrument (> 30 nm). To consider these losses an algorithm was developed to correct the ELPI reading. As shown in Figure 3-10, the correction algorithm basically removes from the upper stages of the impactor the particles that are considered to be fine particles wrongly collected through diffusion, reallocating them into the lower stages. The correction algorithm is implemented in the ELPIvi software and considers both diffusion and electrostatic particle losses. The accuracy of the algorithm increases when the filter stage accessory is used. The filter stage increases the range of the impactor making it possible to measure smaller particles and allowing the correction algorithm to consider them also in the diffusion losses calculation, thus increasing its efficiency.
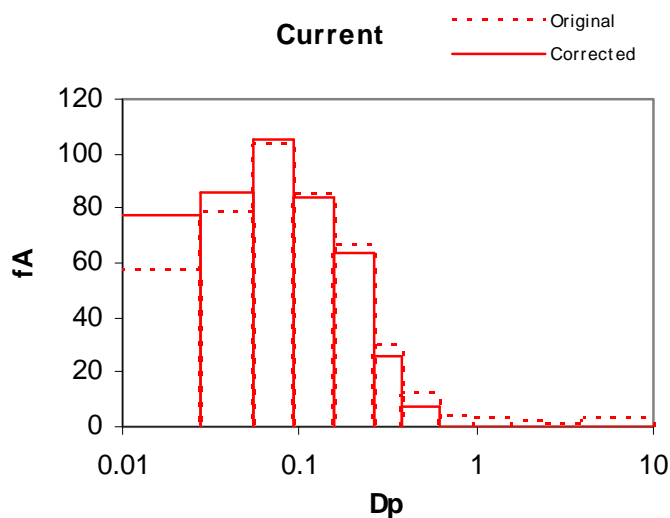
Figure 3-10: Correction algorithm effect

3.7 ELPI measuring accuracy

The measuring accuracy of the ELPI mainly depends on the size of the particles that are measured and on the range used in the measurements. The ELPI can be used in four measurement ranges (named according to the maximum current they can measure):

- 10 000 fA (femtoamperes (10^{-15} A))
- 40 000 fA
- 100 000 fA
- 400 000 fA

The lower measurement ranges have lower noise levels and are more adequate for measuring samples where low particle concentrations are expected. The higher measurement ranges have higher noise levels but are faster. These ranges should be used when sudden changes in particle concentration are expected during sampling and therefore fast-time response is needed.

[10] Virtanen, A., Marjamäki, M., Ristimäki, J., Keskinen, J. Fine particle losses in electrical low-pressure impactor, Journal of Aerosol Science, vol 32, pp 389-401, 2001.

Number concentration sensitivity is better with larger particles since the charge carried by these particles is higher. Mass sensitivity, on the other hand, is better with smaller particles. This is due to the fact that the ELPI is prepared for correct number concentration readings and mass values are calculated from this number concentration value. Since the mass of one large particle is much bigger than the mass of the small particles even a small error in number concentration can lead to big deviations in calculated mass results in the coarse size fraction. Figure 3-11 illustrates the sensitivity of the ELPI measurement.
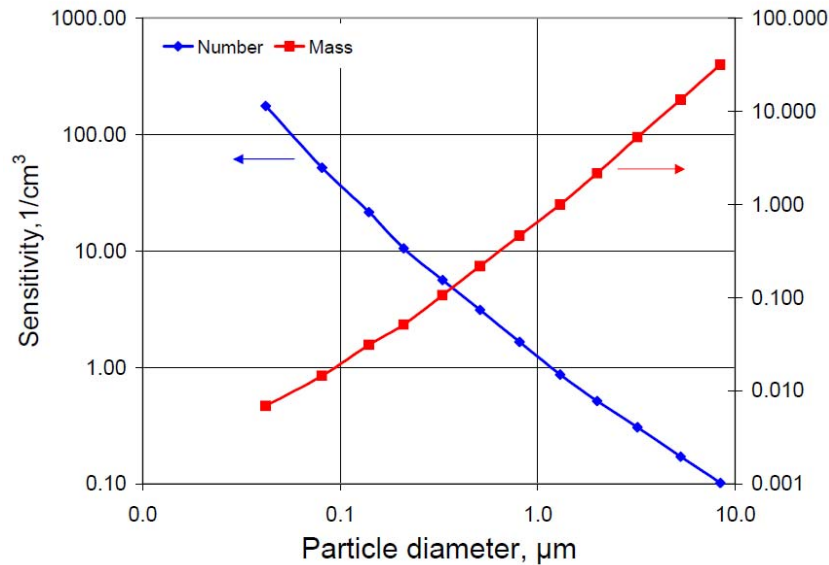
Figure 3-11: ELPI estimated measuring accuracy

3.8 Possible problems during operation

- Negative current values are measured during sampling. When corrected current values are displayed, if the amount of big particles in the aerosol is low and the amount of small particles is high, the correction algorithm used to account for the fine particle losses through diffusion could over-correct the current values in the upper stages of the impactor, obtaining negative current values for these stages. Negative current values could also appear in the raw data when rapidly changing particle concentrations are measured. Negative values in all other distributions are saved as zero in the data file to avoid erroneous readings.

- If not all stages show zero after calibration there might be some dirt between the stages.

3.9   Maintenance

Cleaning is the most important part of the ELPI maintenance. If the impactor is not clean the following problems could occur:

- The collection foils get overloaded affecting the impactor stage cut diameters.
- The jet plates nozzles get clogged, changing the sample flow rate through the impactor
- The Teflon insulators fitted between the impactors stages get dirty, reducing their insulating capability

The maximum recommended loading for one impactor stage is approximately 1 mg. The ELPI front panel displays the total estimated particle mass load collected to the impactor in percentage. When this value is close to 100% the impactor should be cleaned and the collection foils should be changed. After the cleaning process this value needs to be reset to zero.

The impactor must be cleaned regularly, wiping all parts with a paper tissue or a cloth impregnated with a solvent capable of dissolving the measured particles efficiently. Acetone is recommended for hydrocarbon particles. For a more exhaustive cleaning, all the parts (except the O-rings) should be placed in an ultrasonic bath with the appropriate solvent for about 20 minutes. The last wash is recommended to be made with isopropanol, since it eases drying of the parts. The Teflon insulators should be cleaned separately from other impactor parts using isopropanol and making sure that they are properly dried before using them again.

The charger should also be cleaned whenever the impactor is cleaned using the same method and making sure that the Teflon part is well dried before using the charger again. At least once a year it is recommended to dismantle the charger core and properly clean all charger parts. An increase in the charger voltage also indicates the need for cleaning. As particles deposit in the charger walls, the charger needs more voltage to obtain the same current (due to the insulation effect of the particle layer).

All Teflon parts should be handled with bare hands (do not use rubber gloves to prevent charging). To check that the impactor jet orifices are properly cleaned an external flow meter can be used to measure the inlet sample flow to the ELPI adjusting the pressure below the first stage of the impactor to 100 mbar.

The diluters also need to be cleaned regularly with acetone or another suitable solvent, putting special attention on the diluter ejection nozzle, which needs to be treated carefully and cleaned properly (follow the cleaning procedure included in the user manual). The flush pump filter needs to be changed approximately every two years. The main pump oil needs to be checked regularly. The silica gel for the pressurized air cleaning unit needs to be replaced.

4 STL-DUST SAMPLER COMBI DUST AND TAR SAMPLER

METLAB's STL Combi Dust Sampler is designed for isokinetic sampling of solid particles in stack emissions from e.g. industrial processes, power plants and incinerators with a maximum sample flow of 6 m^3/h. Another application can be stack emissions from small scale heating appliances such as wood stoves. The equipment fulfils the requirement for sampling according to the Swedish Standard SS 028426 which is replaced by the EN 13284-1 2002. The filter is provided with a heating device allowing collection of solid particles from hot flue gases which prevents condensation of aerosols. Zero pressure nozzles with an additional automatic motor valve simplify isokinetic sampling. In addition, the filter can be connected to a tar sampler if sampling of condensable hydrocarbons below 350°C is desired. The particles collected on the filter and condensed tars are appropriate for further chemical analysis.

4.1 Gravimetric sampling of particles

One method used for particle sampling is gravimetric analysis. It is a quantitative analytical method bases on the weight of the sample which is collected on a filter. The filter is conditioned and weighted before and after sampling giving the total mass of the collected material. Conditioning of the filter is important to avoid errors by water uptake. The main disadvantage of this method is the lacking time resolution. Though, it is a relatively simple method for measuring particles against an emission limit value. In the section below important aspects of gravimetric sampling are described.

Filter
The particle can be collected with an in-duct or out-duct filter holder. However the out-duct filter is most frequently used. The advantage of out-duct filter is that it allows sampling at high volumetric flow rates. Larger filter surfaces are used for higher particle concentrations. The filters are often designed for plane filtration and consist of quartz, glass fibre or Teflon. Quartz filter are mainly used for higher temperatures up to 950°C. If it is desired to sample just solid particles it is necessary to heat the filter above the dew-point temperature of the condensable compounds.

Isokinetic sampling
It is important that the extracted gas volume represents the flue gas flow in the duct. Inertial effects at the entrance of the nozzle occur mainly for large particles, and cause significant errors in the results. This can be avoided if the velocity in the nozzle is the same as the velocity in the duct, which is called isokinetic sampling. It ensures that neither more nor less particles are extracted from the flue gas. However isokinetic sampling is not always possible in practice. Therefore an isokinetic deviation (i.d.) of 10% or 95-115% is allowed. Over sampling, when the velocity in the nozzle is higher than in the duct, is less severe because fewer large particles are sampled.[4]

$$\text{i.d.} = \frac{(v_{nozzle} - v_{duct})}{v_{duct}} \times 100 \qquad \%$$

v_{nozzle} = velocity in the nozzle   m/s
v_{duct} = velocity in the duct   m/s

Flue gas velocity and gas flow measurement

The basis to achieve isokinetic sampling it is to identify the velocity in the duct.[4] The velocity in the duct can be measured with help of a pitot tube. Velocity measurement during the sampling helps in addition to calculate the isokinetic deviation. In combustion processes it can often be more accurate to calculate the flue gas flow from known fuel mass flow and flue gas analysis (O_2 or CO_2). The velocity determination is in addition required before sampling in order to choose the optimal nozzle size. It is recommended to measure the plane area even if earlier reported values are given to ensure the right calculation of the volume flow and the dust load.

Weighing procedure

Weighing is an important part of the determination of dust concentration to guarantee accurate measurement data. Parameters such as temperature, pressure and humidity influence the weighing result. Therefore this influence should be minimized by pre- and post-conditioning of the filter. It is recommended to use a blank control filter in all measurement steps to identify errors.

Dimension

Depending on the velocity in the duct the nozzle size shall be chosen so that the sample flow in the beginning of a sample run is approximately 70% of the sampling train pump capacity.[4]

Depending on the expected dust load the sampling volume and duration can be calculated.[11] The example below assumes a dust concentration of 5 mg/Nm^3 dry gas, a maximum sampling capacity of 5.7 Nm^3 dry gas/h, a relative weighing uncertainty of 4% and an absolute weighing uncertainty of ± 2 mg.

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[11] Lars Månsson. Dust sampling, Operator Instruction, Appendix D, METLAB, 2004.

Minimum amount of sampled dust

$$\Delta m_{min} = q_{ntg} \times t \times C_{dim} = \frac{\delta_{w,abs}}{\delta_{w,rel}} = (example) = \frac{2}{0,04} = 50 \qquad mg$$

Minimum sampling volume

$$V_{min} = \frac{\Delta m_{min}}{C_{dim}} = (example) = \frac{50}{5} = 10 \qquad m^3n \text{ dry gas}$$

Minimum sampling time

$$t_{min} = \frac{V_{min}}{q_{ntg,max} \times f} = (example) = \frac{10}{5,7 \times 0,7} = 2,5 \qquad h$$

where

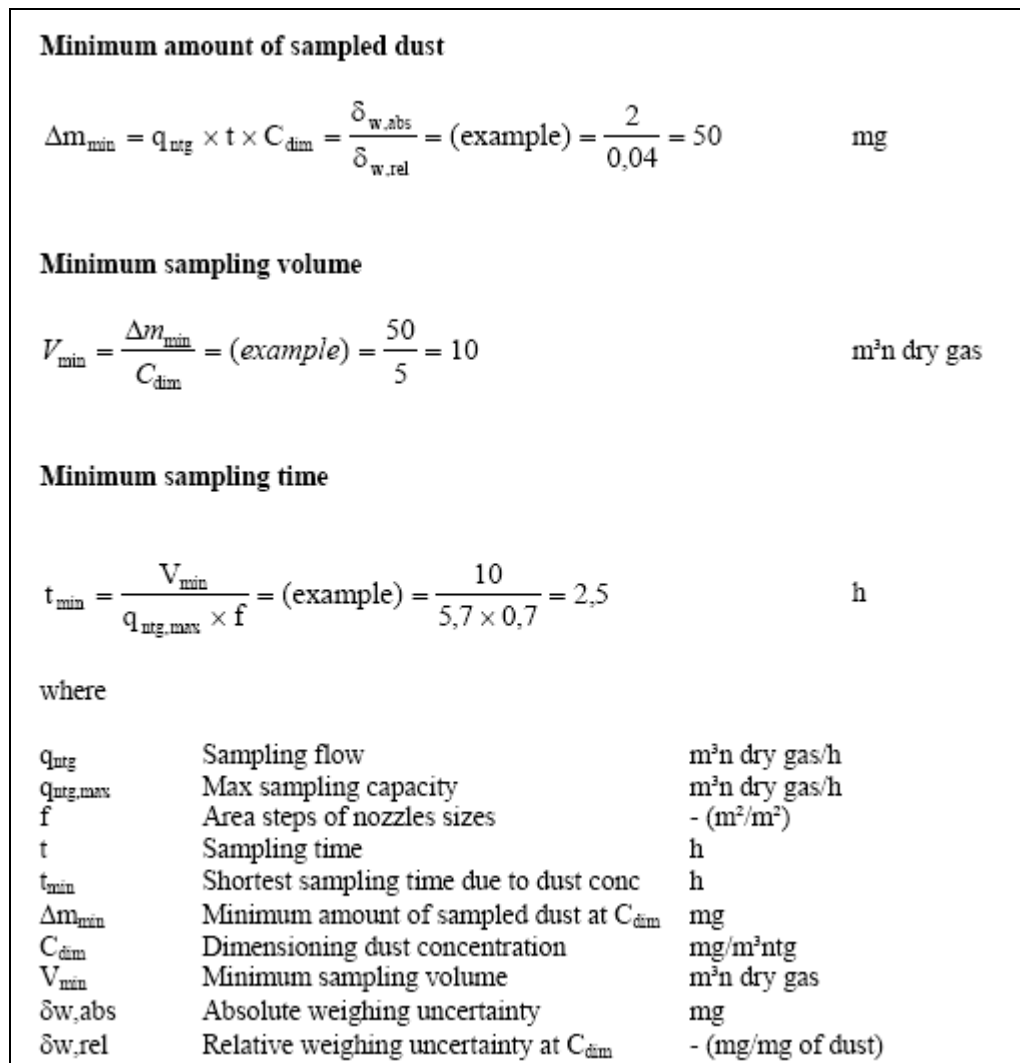|		
q_{ntg}	Sampling flow	m^3n dry gas/h
$q_{ntg,max}$	Max sampling capacity	m^3n dry gas/h
f	Area steps of nozzles sizes	- (m^2/m^2)
t	Sampling time	h
t_{min}	Shortest sampling time due to dust conc	h
Δm_{min}	Minimum amount of sampled dust at C_{dim}	mg
C_{dim}	Dimensioning dust concentration	mg/m^3ntg
V_{min}	Minimum sampling volume	m^3n dry gas
$\delta w,abs$	Absolute weighing uncertainty	mg
$\delta w,rel$	Relative weighing uncertainty at C_{dim}	- (mg/mg of dust)

Figure 4-1: Sampling dimensioning calculations[11]

Table 4-1 gives an overview of varying sampling conditions depending on the dust load.

Table 4-1: Different sampling volumes and sampling times for diverse dust loads[11]

C_{dim} (e.g. limit value) (mg/m^3n dry gas)	$\Delta m_{filter}/C_{dim}$		V_{min} (m^3n)	t (h)	q_{ntg} (m^3n/h)
100	50/100	=	0,5		
10	50/10	=	5	2	2,5
5	50/5	=	10	2,5	4
1	50/1	=	50	5	10
Normal condition: 273,15 K and 101,325 kPa					

A larger pump capacity is recommended for lower dust concentrations. 10 Nm^3/h is recommended for dust concentration below 5 mg/Nm^3 dry gas.

4.2   Equipment description and operating principle

The Combi Dust and Tar Sampler equipment can be divided into a probe unit, interconnections and a control unit.

The probe unit consists of the sample suction tube which is equipped with a heating device for hot flue gas sampling. The temperature can be controlled by a power regulator. Exchangeable zero pressure nozzles of the size 6, 8, 10, 12, 15 and 20 mm can be connected to the probe. The highest local velocity in the sampling plane needs to be known to choose the optimal nozzle size. Zero pressure nozzles are special designed nozzles which require an individually calibration due to non-ideal flow phenomena. Each nozzle shows a different deviation depending on the difference to the zero pressure and the velocity as Figure 4-2 shows. The deviation decreases with increasing velocity.
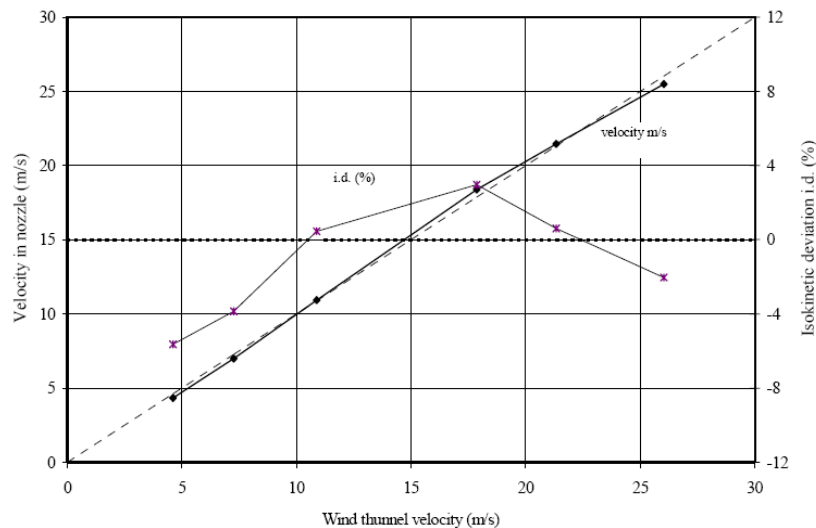

Figure 4-2: Calibration of zero-pressure nozzle Di = 10 mm, ID no 083 [11]

A zero pressure nozzle helps to control isokinetic sampling. Isokinetic sampling requires that the velocity in the duct and in the nozzle is equal. This can be done by comparing the corresponding static pressures. Therefore, the zero pressure nozzles contain two measurement chambers for the static pressure, inside the nozzle and outside the nozzle. Both chambers are connected to a pressure gauge which measures the differential pressure. If the pressure difference is adjusted to zero, isokinetic sampling is maintained. A manual flow valve is used to adjust the gas sample flow so that the differential pressure is zero or close to zero. The adjustment of the zero pressure can be done automatically if the equipment is connected to an automatic motor valve.
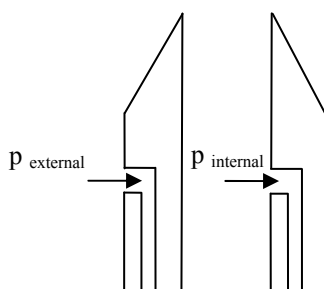

Figure 4-3: Zero pressure nozzle

The probe is equipped with one out-duct 90 mm plane filter. The probe has two connections for the pressure difference measurement in the control unit. In addition the filter holder can be assembled with a Swagelock connection which allows hot and wet gas sampling (HCl, Hg, SO_2, NH_3) after removal of solid particles.

For only dust sampling the filter holder is placed in an insulation cover for temperatures up to 160°C. The filter can be preheated by a hot air gun, to avoid condensation in the beginning of the sampling. For filtration at higher temperatures with tar sampling the filter is placed in a heating body for temperatures up to 350°C. The tar sampling unit in this case is connected to the heating body. The tar sampler contains a glass cooler, followed by a condensation bottle XAD column. The hot flue gas is cooled down in the glass cooler with circulating water. The tars condensate by the glass cooler and are collected in the glass bottle. The final XAD column adsorbs the remaining organic compounds in the gas flow. High temperature tar sampling does not require isokinetic sampling because the tars are extracted from the duct in the gas phase.[4] The nozzle and the probe can be chosen more freely to prevent mainly blocking of the nozzle and filter.

Interconnections contain vacuum hose for the sample flow, twin hose for differential pressure measurement, thermocouple wires for filter housing and stack temperature and an electrical cable for suction tube heating.

The control unit consists of in the flow direction inlet connector for vacuum hose, drying tower, needle valve to adjust the flow manually, outlet connector for the pumping unit (6 m^3/h), a return connector from the pump, a drying tower (silica gel), a rotameter and a gas volume meter. After the gas meter a tee-connector allows extraction of dry and filtered flue gas for gas analysis (O_2, CO_2, CO). The control unit contains further pressure gauges for zero pressure measurement, the static pressure in the sampling plane and a vacuum meter for indication of flow blockage. In addition temperature readings are installed for the temperature in the gas meter, at the inlet to the control unit, in the filter housing and in the sampling plane. A condensate unit containing a condensate separator with condensate bottle and a water cooled heat exchanger can be installed before the control unit for very wet flue gas sampling. An automatic motor valve can be connected between the vacuum pump and the control unit to regulate the isokinetic flow mechanically with help of the zero pressure nozzles and adjust the pressure difference to zero.

The filter holder can be replaced by an Anderson impactor either in the insulation cover or heating body.

4.3 Test procedure

A test run is performed by situating the probe in the duct with a sharp-edge nozzle facing the gas flow. A partial flow is extracted isokinetic over a certain period if isokinetic sampling is required. The out-duct filter separates the particles from the gas stream. The filter needs to be conditioned and pre-weighed before the sampling. The tars in the flue gas condense in the tar sampler and are determined gravimetrically by weighing. The extracted volume is determined by a gas flow meter and the collected dust and tar amount can be re-calculated from mg/Nm^3 to kg/h.

Preparation sampling point and nozzle choice

The highest velocity in the sampling plane can be either identified by a pitot tube or calculating the generated volume flow by a mass balance. Choose the right nozzle size either with help of the diagram or according to the calculations. Consider the risk of higher gas velocities. Identify the internal dimension of the duct and select the location of the sampling point.

Preparation filter

The filter can be transported, conditioned and weighed in one of the provided alumina transport boxes. The conditioning can be done by drying the filter in an oven for 2 hours at 105°C with following cooling to room temperature in a desiccator. Weighing of tempered transport boxes can be avoided by placing a thermocouple in the desiccator. Other temperatures can be chosen according to the influencing temperature. A laboratory blank filter should be used in addition as quality control routine and in case for corrections. In addition it helps to identify the influence of all steps of the filter handling. A scale with a resolution of 0.1 mg is recommended for weighing.

The filter shall be handled in a clean environment to prevent impurities. The use of the same balance reduces errors due to weighing. The weighing starts and ends with the blank filter. The filter weighing shall be repeated two times and written down in a log book just for filter weighing. Several filters can be prepared and stored in the provided transport boxes. It is recommended to keep them in a sealed plastic bag labelled with date of weighing, filter quality and signature.

Preparation at the sampling side

The instruments needs to be assembled closed at the sampling side. The hoses and interconnections shall be checked visually followed by leakage tests. A regular check of the calibration status of the equipment is recommended. Preheat the suction tube and filter. The silica gel can be weighed before and after sampling to determine the moisture content in the flue gas. Before the sampling start fill out the protocol (see Appendix) and check the filter temperature.

After sampling

After sampling the weighing of the used filter holder is carried out in the same manner as during pre-weighing. It is important that all tars are extracted from the equipment by washing the glass parts with an appropriate solvent. The amount of sampled tars is similar to the particles determined by weighing after evaporating carefully the solvent. The equipment shall be properly cleaned after each sampling, especially the probe and filter housing, to avoid uncertainties. In addition the total equipment shall be checked and necessary repairs and adjustments carried out. The composition of the sampled particles and tars can be further analysed chemically. Some information for filter preparation is found in the chapter of the ELPI. Gas chromatography can be applied to analyse the tar composition.

Calculation

It is recommended to a use a standard for calculation of emission levels. Available standards for gravimetric particle sampling are e.g. VDI 2066 Part 1, EN 13284 and EPA Method 5.

Problems during operation

Main problems during particles sampling are that the partial flow is not extracted isokinetic which causes uncertainties. If the temperature in the probe and filter drops below the desired sampling temperature also undesired aerosols might be collected.

4.4 Caution and Maintenance

The equipment requires careful handling. Especially damaging of the nozzles needs to be avoided when inserting the nozzle in the duct or withdrawing it from it. Dropping of the nozzle and probe must be prevented. Most of the equipment is provided with transport boxes to avoid damages of the equipment. The maintenance includes the appropriate cleaning after each experiment and leakage test. In the Appendix a detailed description of the leakage test procedure is included. The calibration status of the equipment shall be checked regularly. Isokinetic deviation is calculated as a routine to evaluate the results. It further helps to identify possible disfunction of the equipment. If the isokinetic deviation is greater than 10-20% it shall be followed up. The Appendix contains a brief list with potential dangers associated with particle and tar sampling to avoid exposure to hazards and accidents.

4.5 Limitations and restriction in use

The equipment is designed for filtration up to 350°C degrees with a maximum sampling flow of 6 m^3/h. At higher temperatures damages will occur. For only particle sampling the insulation cover tolerates temperature up to 160°C.

Isokinetic sampling can be easily achieved by the use of zero pressure nozzles. The recommended velocity is about 5 m/s because for lower velocities the isokinetic deviation is high for small variations from zero-pressure. The deviation from isokinetic sampling is 40% for a deviation of 12 Pa at a velocity of 5 m/s. The same pressure difference results for 15 m/s in 3% sampling error (Figure 4-1).[4] For lower velocities theoretic calculations are necessary depending on the velocity in the duct and the nozzle area. The flow needs then to be adjusted manually with help of the flow meter. Zero pressure nozzles are in addition not recommended for low velocities and where steady flow conditions frequently do not exist.
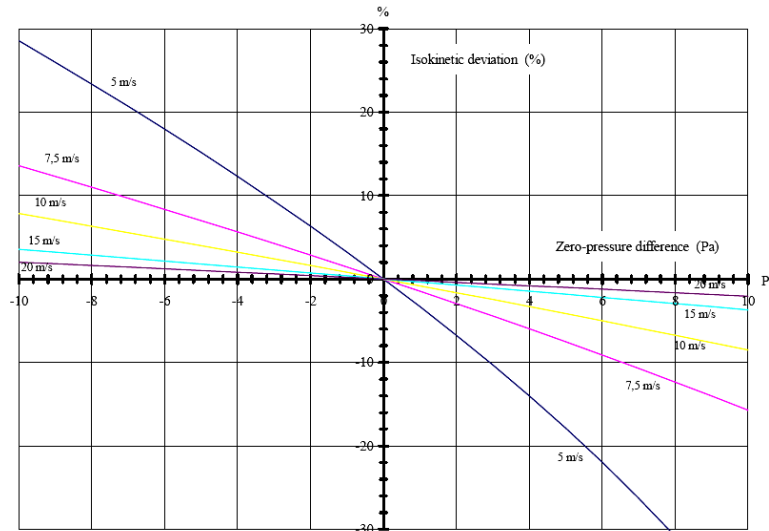
Figure 4-4: Isokinetic deviation as a function of zero-pressure deviation (gas T=25°C)[11]

Recommendation

If the flow is non-uniform sampling should be carried out at a number of pre-selected stated positions in the duct cross-section.

Uncertainties

Failures in the measurement data can occur due to wrong sampling (not isokinetic), leakages and low dust concentration which increase errors during weighing. The recommendation to reduce uncertainties is at least two samples at equal conditions, a filter increase of 50 mg and leakage tests before and after each test run to identify the amount of false air sampled. The final result will be calculated as a mean value. The sampling time shall be long enough to sample a sufficient amount of volume which is representative for the process and cover variations due to different process conditions.

Loss of sample dust

Clean carefully the inner surface of the probe and filter holder after each test at a calm and clean place. Brushes are included in the equipment. Acetone and water can be used for rinsing. Use the transport box for transportation.
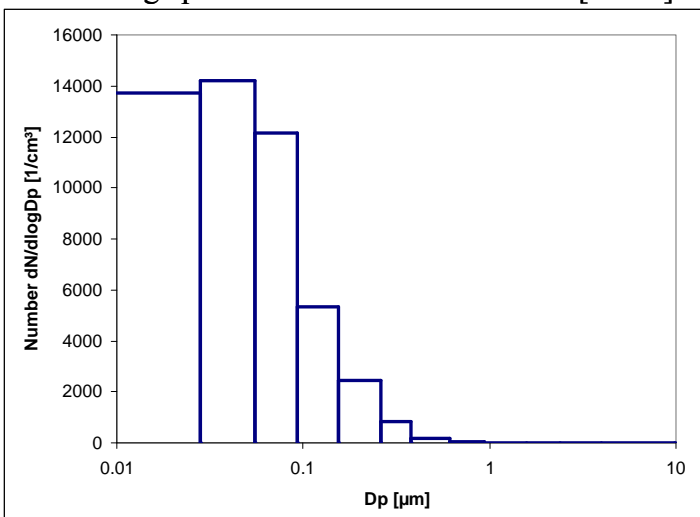
Contamination
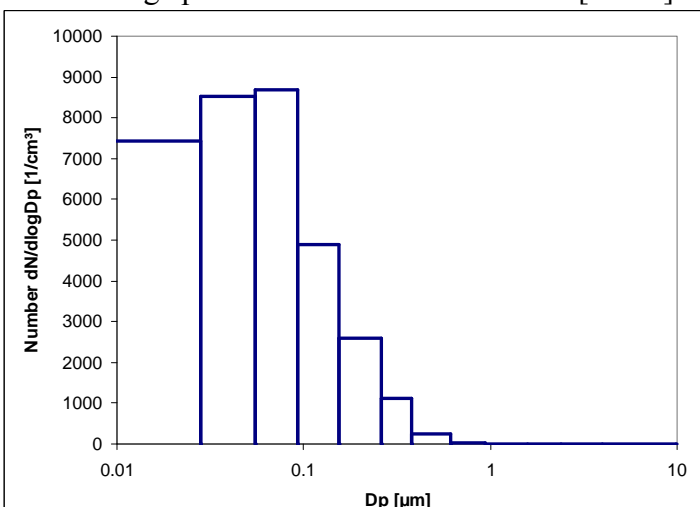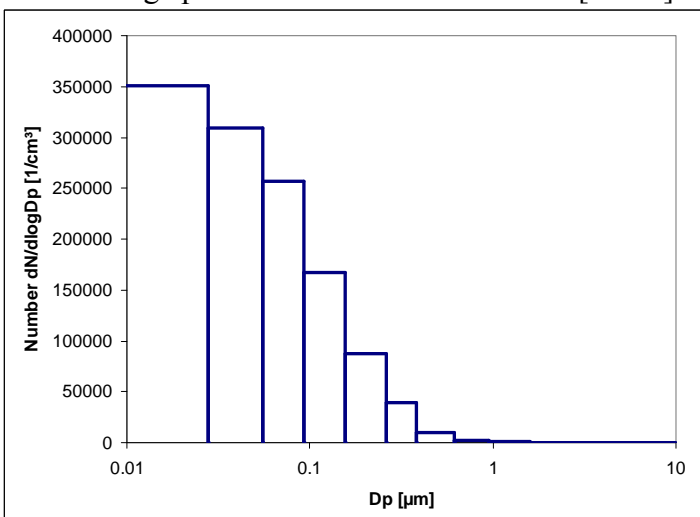
Deposits in the duct and on the access ports can cause contamination when the nozzle gets in contact. This shall be avoided.

5 INITIAL TESTS

5.1 Initial tests for the ELPI and evaluation of the results

To test the ELPI at different conditions several experiments were carried out using lab air and smoke from different devices. The results are shown in Table 5-1.

Table 5-1: Size distribution of particles at different conditions

Total average particle concentration: 1.7E+4 [#/cm³]	Lab air
Total average particle concentration: 1.0E+4 [#/cm³]	Propane burner
Total average particle concentration: 4.2E+5 [#/cm³]	Candle

Total average particle concentration: 1.3E+6 [#/cm^3]	Smoke pen
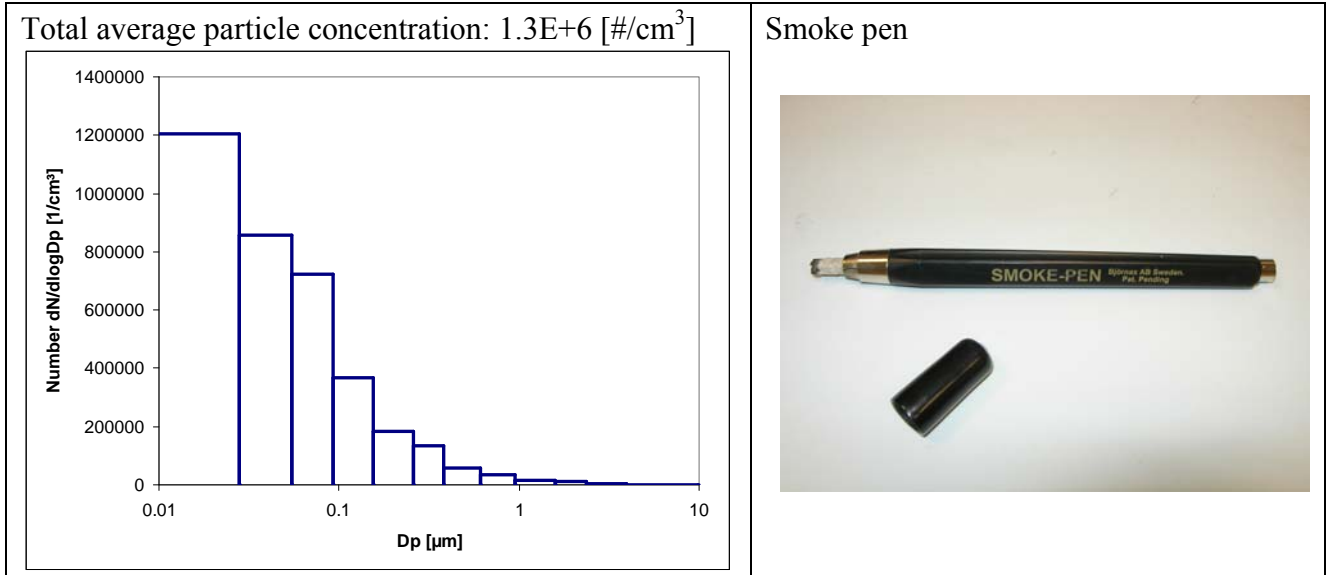
In addition, a test was performed with smoke from a wood-burning stove. For this purpose a flexible structure was built to fit the dilution system together with the ELPI and the pump. The experimental setup is shown in Figure 5-1. The dilution system was connected directly to the stack with a sampling probe. The sample was only collected during the char combustion phase (the last part of the combustion process) in order to prevent overloading of the instrument.
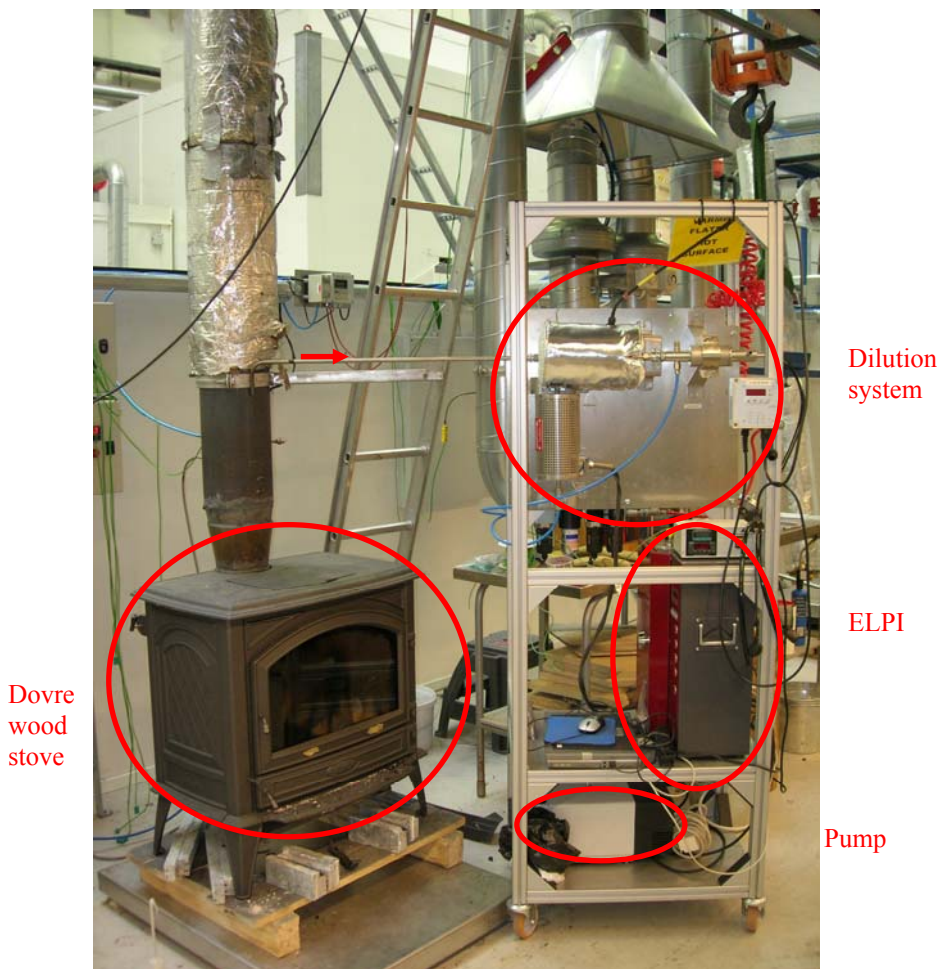
Figure 5-1: Wood stove experimental setup

The results of this experiment are shown in Table 5-2. The average particle concentration of the whole test run was 5.8E+8 [#/cm^3]. The experiments presented in this section show the capability of the ELPI for the study of particle concentration and size distribution in applications where rapidly changing conditions are expected and fast response is needed.

Table 5-2: ELPI results from wood stove experiment

A	B	C
Average particle concentration: 7.6E+8 [#/cm^3]	Average particle concentration: 1.2E+9 [#/cm^3]	Average particle concentration: 8.2E+7 [#/cm^3]
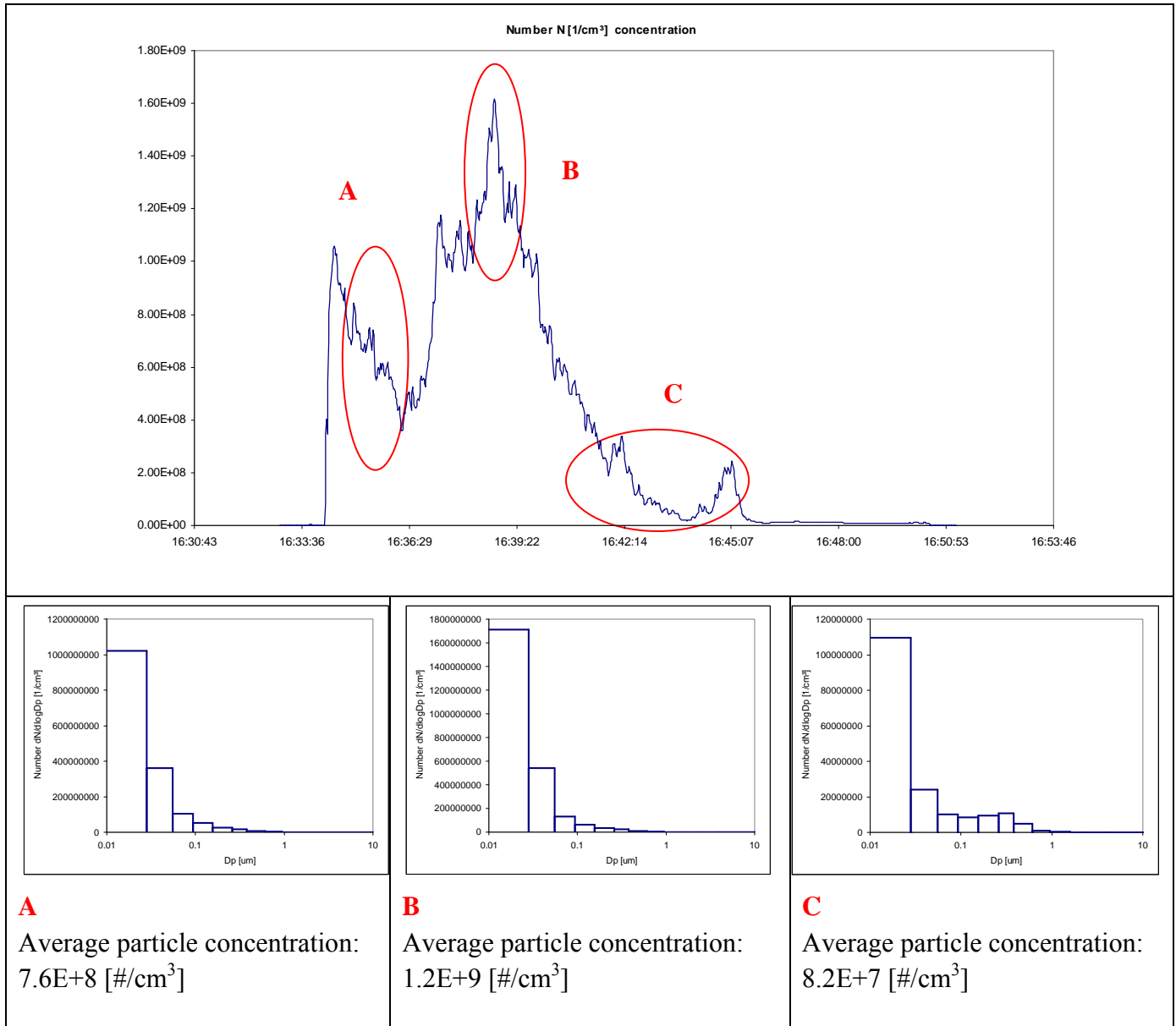
5.1.1 Conclusions and recommendations for further experiments

The presented initial tests prove the validity of the ELPI for the study of particle concentration and size distribution in applications where rapidly changing conditions are expected and fast response is needed. The use of the ELPI requires a well-planned and detailed procedure, especially in the maintenance part. Special care is needed when manipulating the impactor stages and the collection substrates. The collection substrates must be properly stored if subsequent

chemical analysis is wanted. The use of a clean room is strongly recommended when handling the samples.

Before running a test the impactor and diluters should be checked for cleaning (preferably the day before). The ELPI also needs one hour of warming up before the zeroing procedure can be carried out. It is also recommended to test the ELPI in each specific application to calculate the time needed to collect a particular sample amount. This is especially important if it is planned to perform a chemical analysis which requires a specific sample mass on the collection substrates.

The ELPI setup can be used in connection with different experimental setups where particle concentration, size distribution and properties are in focus. This includes the multi-fuel reactor setup at SINTEF Energy Research.

5.2 Initial tests for STL-Dust and Tar Sampler

The STL was used to sample particles from a wood stove stack, shown in Figure 5-2. It is difficult to measure the velocity in a wood stove stack with natural draught, but a low velocity in the stack was assumed and the largest nozzle with a diameter of 20 mm was chosen. The probe was preheated to 160°C. The sampling started by opening the manual valve and setting the sampling flow rate to 0.8 m^3/h so that the zero pressure manometer showed zero. The sampling velocity in the nozzle was 0.76 m/s.
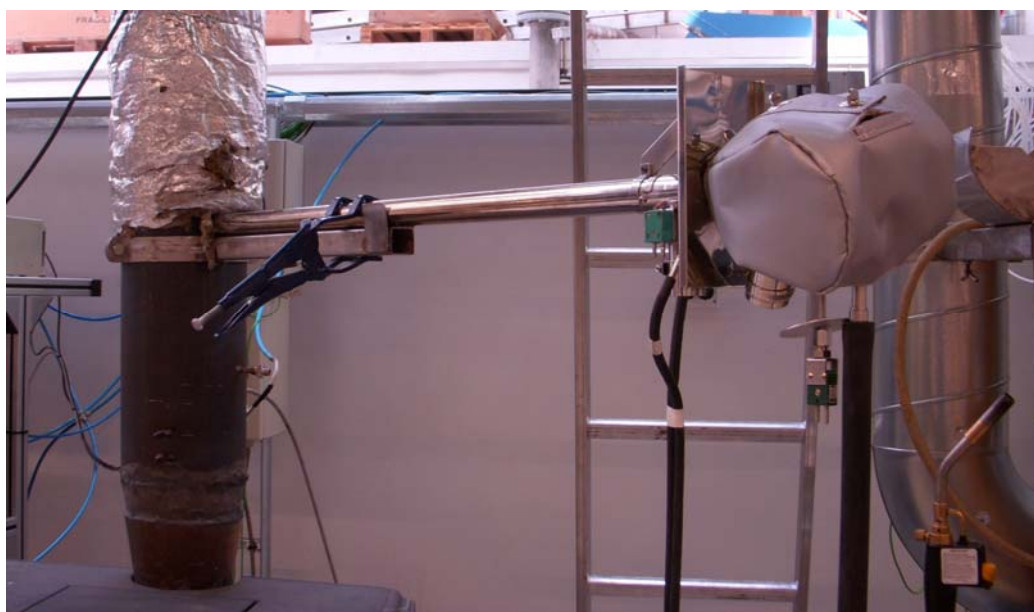

Figure 5-2: Heated filter inserted in the stack of a wood stove

Table 5-3: Sampling protocol

	Time min	Filter housing °C	Duct °C	Static pressure duct kPa
1	0	24	141	0.027
2	5	107	151	0.028

3	10	112	145	0.029
4	15	114	141	0.029

	Filter weight before g	48.2989	Gas meter start m³	18.145
	Filter weight after g	48.3918	Gas meter stop m³	18.364
	Dust amount g	0.0929	Gas meter m³-m³	0.215
	Dust concentration	550 mg/m³	V norm m³	0.177

The sampling conditions in the stack were almost constant over the test run. The filter temperature increased from 24 to 107°C in 5 minuets because it was forwent to preheat the filter. The total extracted volume was 215 l and the particles collected on the filter 0.093 g. The resulting dust concentration from this test run is 550 mg/m^3 in the flue gas. The filter after sampling is shown in Figure 5-3.

Figure 5-3: Filter after sampling

5.2.1   Evaluation of the results

This can not be assessed without the knowledge about accuracy. The velocity and volume flow in the stack are required to calculate the isokinetic deviation which in this case are not known. The uncertainty for so low velocities in the zero pressure nozzles is high. It is not recommended to use the zero pressure nozzles for isokinetic control. The sampling flow needs to be regulated manually according to the measured or calculated velocity.

5.2.2 Conclusions and recommendations for further experiments

It is important to reserve the appropriate time for preparing the sampling. The preparation includes the planning of the location of the sampling and the measurement of the velocity with a Pitot tube or calculation of the volume flow to choose the right nozzle size. Pre-sampling is recommended to check the equipment, the sampling conditions and to reduce errors during sampling.

The equipment is made for large scale applications. It is heavy, especially when the tar sampling unit is connected. Two persons should handle the equipment. If the preheating is done outside of the duct then the hot probe has to be handled with great care. The preheating can be carried out inside the duct when the bend nozzle head is used and the duct is large enough to turn the nozzle opening away from the gas flow.

The STL setup can be used in connection with different experimental setups where total particle and tar concentration and properties are in focus. This includes the multi-fuel reactor setup at SINTEF Energy Research.

6 FINAL CONCLUSIONS AND RECOMMENDATIONS

The presented initial tests prove the validity of the ELPI for the study of particle concentration and size distribution in applications where rapidly changing conditions are expected and fast response is needed. The collection of the size classified particles in the different impactor stages also makes possible subsequent chemical analysis. The STL setup has also been tested and it can be used in connection with different experimental setups where total particle and tar concentration and properties are in focus.

Both these particle measurement systems will be used in connection with the multi-fuel reactor setup at SINTEF Energy Research.

7 APPENDIX

7.1 Equipment Components

7.1.1 ELPI

ELPI-01	ELPI Measurement Device 10 l/min + carry case
IA-904	Vacuum Pump
IA-203	Vacuum hose (1.5 m) pressure valve + NW16 flange connectors
DI-2000	Double Diluter Package + carry case
DH-1523	Insulated diluter heater
DR-1623	Heater temperature controller
DH-1723	Pressurized air heater

DR-1823	Temperature controller for pressurized air heater
DI-1010b	Pressurized air regulator and dust filter
DI-1011	Exhaust valve for reversed flow
DI-1019	90 angle joint for exhaust valve set
IA-921	Pump Oil for Leybold Vacuum Pump (3 bottles)
DS-515	Dekati Collection Substrate Spray
	User Manual
Box 1	
ELA-443	USB - Serial adapter
IA-200	Collection plate set
IPR-509	Serial cable
IPR-110	NW 16 flange
IPR-524	Blind NW-16 flange for the impactor inlet
	Tube connector
	Mains cable
	Impactor stage 12
	Sample hose
Box 2	
CF-300	Aluminium foils
IPR-200	Polycarbonate collection foil
DS-125	Stencil
PMF-047	EMFAB filter
IA-206	Set of viton O-ring for impactor and filter stage
IPR-20-4013	BNC pin connector for electrometer contact
IA-321	Metal spring for impactor
IPR	Substrate holder ring
DI-1008	Spare copper-graphite seal set for Dekati Diluter
IPR-114	Corona needle
IPR-400	Impactor Tweezers
IPR-403	Tweezers

7.1.2 STL-Dust and Tar Sampler

Equipment components STL Dust and Tar sampler

Sample ESS Probe
-   entry nozzles of zero pressure: 6,8,10,12,15 and 20 with calibration protocol of deviation
-   two nozzle tools
-   anti-seize lubricant tape
-   straight nozzle head
-   nozzle head with 90° bend
-   transport box for nozzle and nozzle heads
-   heated suction tube of length 1060 mm
-   transport and probe protection box with foldable stand
-   power regulator for probe heating incl. transport box
-   filter holder for one out-duct plane filter 90 mm for temperatures up to 350°C

- filter cassette with transport container of aluminium 10x
- insulation cover for the filter holder FH90 for temperatures up to 160°C
- heating body for temperatures up to 350°C
- hot air gun up to 350°C
- cleaning brushes
- port hole adapters for a 2" sampling outlet

Tar sampler
- metal box for glass components: cooler, condense bottles, XAD2 column
- connection between filter holder and cooler (stainless steal)
- glass cooler (water based)
- connection tube between cooler and condensate bottles
- condense bottles 1 l
- connection tube between condenser and XAD2 column
- XAD2 column
- exit part for vacuum hose connection
- metal clips 1x19, 4x29, 1x35
- transport box for glass parts

Interconnections
- vacuum hose 15 m
- twin hose for differential pressure 15 m
- thermocouples wires 15m x 2
- electric cable for suction probe heating
- transport box for interconnections

Control unit
- control unit CU-6 up to 6 m^3/h sample flow
- pump unit installed in transport box (Q_{min} = 0.04 m^3/h; Q_{max} = 6 m^3/h)
- condensate separation unit with condense bottles
- heat exchanger out of stainless steel
- gas analysis port after dry gas meter
- difference pressure manometer
- calculation sheet for calculation of the differential pressure which corresponds to isokinetic sampling
- automatic motor valve
- transport box

Spare parts and accessory
- glass fibre filter 90 mm 4x50
- drying agent silica gel 5 kg (without colour indicator)
- 3x3 net bags for up to 2 kg silica gel
- high temperature o-ring for suction tube and nozzle head
- o-ring for filter holder 2x
- 2x service kit for vacuum pump

7.2   Test procedure checklist and protocols

7.2.1   ELPI Test procedure

1. Check that the impactor and charger are clean. Assemble the impactor using suitable collection substrates (e.g. greased Al-foils).
2. Place the impactor in the ELPI housing and connect the BNC connectors and the filter stage.
3. Switch on the ELPI power (the device has to be connected for one hour before starting the zeroing procedure).
4. Start the ELPI software.
5. Perform the leakage test.
6. Connect the impactor to the charger with the clamp and gasket.
7. Select "Measurement program" in the ELPIvi software, introduce the correct setup values (introduce dilution ratio and "date of experiment"; name of data file) and press OK.
8. Turn the vacuum pump ON. Adjust the pressure at the ELPI outlet to 100 mbar.
9. There will be a triboelectric effect when the pump starts. It causes spurious current readings; wait until the current readings are stabilized.
10. Put charger voltage ON.
11. Put the flush pump ON. Wait until the current values stabilize.

    - 2 min – 400 000 fA
    - 4 min – 100 000 fA
    - 10 min – 40 000 fA
    - 15 min – 10 000 fA

12. Perform the electrometer zeroing for the needed measurement ranges; press "zero" or "all zero"
13. After zeroing put the flush pump OFF (with "Zero with flush" ON just press "zero" or "all zero" and forget about steps 11, 12 and 13)
14. Check that the charger is ON and that the trap voltage is OFF (when the filter stage is used).
15. Check that the diluters are clean. Check the connections of the ejectors for leakage.
16. The first diluter heater and the pressurized air heater need to be connected before sampling to achieve the required set point temperature (250°C). Connect the ELPI to the diluter system and the sampling probe to the desired sampling point. Pay attention to the sampling conditions. Remember to save the desired data by changing the default "NOT saving" status.

After measuring use the flush pump to see that the offset currents of the electrometers have not changed during the measurement.

7.2.2   STL-Dust and Tar Sampler Test procedure

Preparation at the sampling side
1. Set up equipment
2. Open all access ports and clean them with a steel brush
3. Mount the access port adapter and close all other openings
4. Load the filter holder with a cassette (place the probe protection box as close as possible to the port; use the foldable stand with a clamp for assembling)
5. Place the insulation cover around the filter holder (even if the filter is not heated for protection)
6. Connect and check connections of vacuum hose, twin hose, thermocouples wires, electrical cable
7. Connect the nozzle
8. Replace/weigh silica gel if necessary (1-2 kg)
9. Check pump flow and vacuum capacity
10. Leakage test: check the vacuum side of the system
11. Leakage test: zero pressure system
12. Start preheating if sampling is required with a hot filter: adjust the power regulator and connect the hot air gun (10-30 min)

Sampling
1. Check filter temperature
2. Check logging of process data
3. Fill out protocol: filter weight, gas meter, silica gel weight, equipment and filter identification, barometric pressure, date, plant
4. Disconnect the hot air gun
5. Insert the probe with great care not to damage the nozzle
6. Lock the probe by help of the access port clamp
7. Start the pump with the flow valve closed
8. Note starting time, open the flow valve and reach isokinetic sampling either by adjusting the flow until the zero-pressure manometer shows zero or by adjusting a certain volume flow
9. Note temperatures and pressures

Sampling stop
1. Note the final values of temperature and pressure
2. Note the time, close the flow valve, note the gas meter
3. withdraw the probe and place on the stand

After sampling
1. Disconnect the vacuum hose from the filter housing and rinse the hose with a flow for 5 minutest
2. measure the condensate volume and weigh the silica gel
3. clean the probe coarse by knocking slightly along the with a hard plastic object
4. disconnect the filter housing
5. open it in a clean and calm place (might be hot) and remove the filter cassette
6. clean the filter housing with the probe's rinsing brush and added to the filter transport box

Post Sampling
1. Weighing of the used filter holder is carried out in the same manner as during pre-weighing. In addition with the laboratory blank filter as quality control.
2. Clean and check all parts of the sampling equipment. Make repairs and adjustments.
3. Regenerate the silica gel at 150-180°C

Dust sampling protocol

	Client Process Process condition		Date Signature	
Test No. Sampling No.		Dust sampler Id-No. Gas meter No. Gas meter deviation %		
No. of sampling points st Measuring plane Meas plane dimension mm		Sampling filter nr Filter size mm Filter quality Filter weight before g Filter weight after g Dust amount mg		
Probe/nozzle identity Id No. Nozzle size mm				
Barometric pressure kPa Static pressure in duct kPa				
Temperature instrument Id No.		Gas sampling throught probe	yes No	

Meas point No.	Time h - h	Gas meter m³-m³	Gas meter °C	Inlet gas temp °C	Filter housing °C	Duct °C	Static pressure kPa	Vacuum meter Bar
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								

Silica gel weight before g Silica gel weight after g		Gas meter volume in 1 min liter Corresponding flow m³/h Flow through rotameter m³/h Deviation rel rotameter %	
Absorbed water in silica gel g Condensate g			
Total amount of water g		Leakage test yeas No	

Leakage controls

Vacuum side
Turn on the pump with the flow valve closed, then adjust to a low flow (approx. 1 m^3/h) and block the nozzle with the special flat (and clean) nozzle plug. Increase the flow to reach maximum vacuum quickly. Read flow through the vacuum meter, rotameter and gas meter. Close the valve and remove the stopper slowly when leak check is performed with the filter.

Pressure side
Control the gas meter regularly with a calibrated gas (just used for this purpose and calibrated frequently). Check rotameter towards the gas meter and prove visual the connections after the pump.

Zero-pressure system
Connect the zero pressure hose to the probe. Disconnect the external pressure of the twin hose from the control unit. Suck or blow slightly while looking at the zero pressure manometer. Close the hose by folding it and check that the pressure does not fall. Repeat with internal pressure hose disconnected.

Health Safety and Environment

The operation of the tar and dust sampler may involve some hazards depending on the circumstances.

Hazards for the sampling operator are:
- working at heights
- exposure to toxic, corrosive and hot gases and dust
    → exhaust gas needs to be piped away
- electric hazards
- hot surfaces
- falling objects
- handling of heavy and bulky equipment
- trip hazards

Hazards to other personnel:
- inform by help of warning signs

Hazards to plant:
- ignition of flammable gases: equipment is not classified for the use in a potentially explosive atmosphere
- equipment dropped into the duct system
- risk of fire due to malfunction of the hot air gun