



*Understanding, Measuring and Regulating Sub-23 nm Particle Emissions from Direct Injection Engines
Including Real Driving Conditions*

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| Deliverable No. 2.1 Super-Continuum Laser Multiwavelength Photoacoustic Aerosol Spectrometer (SCL-MPAS) | |
| Author(s): Nickolas Vlachos (APTL/CERTH), Anastasios Melas (APTL/CERTH) | |
| Quality control: Christos Softas (APTL/CERTH) | |
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| Project coordination and contact: Aerosol and Particle Technology Laboratory/CPERI/CERTH 6 th Km Charilaou-Thermi, 57001 Thermi – Thessaloniki, Greece Contact: helen@cperi.certh.gr | |

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Project Title: Understanding, Measuring and Regulating Sub-23 nm Particle Emissions from Direct Injection Engines Including Real Driving Conditions

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WP2 Scope and Objectives

The focus of SUREAL-23 is on the particulate emissions from contemporary light duty direct injection (DI) internal combustion (IC) engines (Diesel and gasoline) that will be addressed by homologation standards beyond Euro-6, especially for nanoparticles smaller than the current regulation cut-off diameter of 23 nm, with a threshold of at least 10 nm.

Within this context, the objectives of SUREAL-23 are:

- to complement existing standard instrumentation by introducing extensive size and composition characterisation of exhaust particles especially for sizes below 23 nm,
- to support future emissions compliance through technical development in real driving emissions measurement,
- to fully characterise the nature of the particulate emissions which potentially evade current emission control technology and regulations,
- to contribute to future definitions of particulate emissions limits of the “Super Low Emission Vehicles”.

The aim of WP2 is the development of technologies that:

- enable the understanding of PN sub-23 nm emissions with the introduction of in-situ, real-time characterization of particle composition,
- offer precise measurement of sub-23 nm particles number or size distribution allowing both ambient and hot temperature sample measurements with the potential for on-board use.

This report aims to document the delivered demonstrator “D2.1 Super-continuum laser multi-wavelength photoacoustic spectrometer setup”, a prototype instrument developed in the frame of WP2 of the SUREAL-23 project. It describes the principle of operation of the chosen concept, the main SCL-MPAS system components developed/selected as well as an initial conceptual/physical system layout. This prototype will continue to undergo improvements and will be available for sub-23 nm combustion aerosol characterisation in the SUREAL-23 project.



Abbreviations list

| | |
|----------|--|
| APTL | Aerosol and Particle Technology Laboratory |
| FHNW | University of Applied Sciences Northwestern Switzerland |
| NKT | NKT Photonics |
| PA | Photoacoustic |
| PN-PEMS | Particle Number-Portable Emissions Measurement Systems |
| PAH | Polycyclic Aromatic Hydrocarbon |
| RDE | Real Driving Emissions |
| SMPS | Scanning Mobility Particle Sizer |
| SCL | super-continuum laser |
| SCL-MPAS | super-continuum laser multiwavelength photoacoustic spectrometer |
| WP | Work Package |

Short Project Overview

There is mounting evidence that a large proportion (in terms of number) of the particles emitted from direct injection engines are below the currently stipulated 23 nm size cut-off and, although the EU aims to regulate those emissions, this is not yet possible to impose limits for new light-duty vehicles due to the absence of accurate sub-23 nm emission quantification methods, especially under real driving conditions. The main reason for this is the absence of adequate knowledge regarding the nature of sub-23 nm particles from different engine/fuel combinations under different operating conditions.

One of the ways the SUREAL-23 Project aims to overcome such barriers is by introducing novel measurement technology for concentration/size/composition measurements. The recently established supercontinuum laser technology will be integrated into a photoacoustic analysis system of tubale visible and near IR wavelength in an effort to obtain near real-time sensing composition of exhaust particles. Multi-wavelength light sources will also be applied – in the UV spectrum – for sub-23 nm exhaust particle composition detection via the strongly material-dependent photoelectric charging phenomenon. In parallel with the optical composition detection development, state of the art aerosol size/concentration measurement techniques will be advanced for better compatibility with sub-23 nm exhaust particles as well as with onboard use. The developed instrumentation will assess sub-23 nm particle emissions from both Diesel and GDI vehicles accounting for effects of the fuel, lubricants, after-treatment and driving conditions for existing and near-future vehicle configurations. The most suitable concepts will be developed for PN-PEMS applications and evaluated accordingly. The project will provide measurement technologies that will complement and extend established particle measurement protocols, sustaining the extensive investments that have already been made by industry and regulatory authorities. The project will deliver systematic characterization of sub 23-nm particles to facilitate future particle emission regulations as well as to assess any potential trade-off between advances in ICE technology towards increased efficiency and emissions. The consortium consists of European and US organizations, which are leaders in the field of aerosol and particle technology.



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1 Introduction to the proposed measurement concept

Aerosol particle composition is most commonly determined ex-situ, using various physical or chemical analysis methods on filter-collected particle samples. These methods include microscopy (i.e. SEM, TEM) techniques with Energy-Dispersive X-ray Spectroscopy (EDS), Thermogravimetric Analysis (TGA), chemical analysis for Soluble Organic Fraction (SOF) and Polycyclic Aromatic Hydrocarbons (PAHs), FTIR-Raman spectroscopy etc. The ex-situ particle composition characterization presents obstacles in the understanding of sub-23 nm engine/vehicle exhaust particles, due to the difficulties to study transient phenomena arising during cold start, filter regeneration or high-power bursts of the engine. Therefore, it is of interest to be able to detect exhaust particle composition in the time scale of up to a few seconds in order to understand such transient emission phenomena.

Over the last years on-line/in-situ optical composition detection techniques are being developed mainly based on advances achieved in incoherent light (lamp) sources as well as in coherent (laser) sources. The photoacoustic detection method has been known since the discovery of the optoacoustic effect in 1880 but active development of the method began to expand since the 1970's with the increasing availability of lasers. For gaseous species, laser powers of the 100 mW – 2 W range (continuous wave) have been used to achieve detection thresholds corresponding to concentrations in the ppb level.

For sub-23 nm aerosol particles, the absorption of visible and IR light occurs over the entire particle volume, since particle diameter is well below the light wavelength, and can be material dependent on the basis of the material absorption spectrum. Directly measuring the extinction of light passing through an exhaust aerosol involves the detection of a very weak absorption against a strong background (beam power) and is not applicable to the current scope. The photoacoustic technique is a method to measure the absorption of the light without the strong background of the excitation beam, thereby providing a means to measure particle mass concentration even for dilute / weakly absorbing aerosols such the engine exhausts of interest.

Photoacoustic signal generation involves a four-step process [1] : i) absorption of photons by the particle, ii) heat up of the particle, iii) particle thermal relaxation to to the surrounding gas, and iv) generation of an acoustic pulse by the gas local thermal expansion. In order to elicit a measurable acoustic signal from the above phenomena it is necessary to modulate the light input as pulses corresponding to the acoustic frequency that is required – the latter being typically determined by the resonant behaviour of the sample measurement chamber/cell.

Light sources that have been used for photoacoustic detection of gases and, in limited cases, particles, do include regular lamp sources but, for the most part, it has been the development of higher power lasers that has driven concurrent advances in photoacoustic methods.

To our knowledge, there are no commercially available photoacoustic aerosol instruments for vehicle/engine exhaust particles composition analysis. The MicroSoot Sensor, by AVL GmbH, relies on a fixed / single wavelength (in near IR spectrum) for the detection of soot (black carbon) mass concentration in appropriately sampled Diesel engine exhaust. For aerosol



composition, we are aware of a related development instrument also using a variably filtered supercontinuum laser for analysis of atmospheric aerosols – the different wavelengths facilitate the distinction of overall particle nature, i.e. black versus brown carbon or non-carbonaceous dust.¹

2 Description of the SuperContinuum Laser Multiwavelength Photoacoustic Aerosol Spectrometer (SCL-MPAS)

2.1 Related innovations in the project

In the scope of SUREAL-23 project requirements, the components of a variable wavelength photoacoustic setup were selected and/or implemented in order to demonstrate the feasibility of characterising the composition of sub-23 nm particulate emissions from contemporary light duty direct injection (DI) internal combustion (IC) engines (Diesel and gasoline). The proposed demonstrator requires the below developments within SUREAL-23:

- a sufficiently powerful light source providing a colimated beam with a selectable wavelength band within the 400 – 900 nm spectrum and with the capability for time-modulation of the output intensity compatible with photoacoustic excitation of an exhaust sample at frequencies below 20 kHz,
- a photoacoustic aerosol detection setup achieving detection thresholds compatible with total aerosol concentrations as low as 10^6 particles/cm³ for particle diameters in the range of 5 – 25 nm and in a carrier exhaust gas of variable composition,
- advanced combustion engine exhaust conditioning systems and instrumentation developed in SUREAL-23 project for sub-23 nm particle emissions investigations.

The advanced instrumentation being developed in SUREAL-23 project that may be employed in tandem with the SCL-MPAS include the advanced Half-Mini DMA (HM-DMA) instrument and a high flow catalytic stripper developed as part of the effort to advance exhaust sample conditioning techniques. The HM-DMA system is a supercritical differential mobility analyser, with a 2 cm working section, initially developed at Yale University and subsequently improved – within SUREAL-23 – by SEADM (Boecillo, Spain) able to classify combustion aerosol particles (including large molecular ions) in the size range 1-30 nm with high resolution and fast acquisition frequency [6]. The placement of the HM-DMA upstream of the photoacoustic spectrometer will permit demonstration of size-selective composition analysis of the exhaust aerosol. The high-flow catalytic stripper was developed by APTL and is able to remove heavy hydrocarbon and sulphur particles while the solid particle penetration is larger than 75% at particle diameters as low as 10 nm. The selective use of the catalytic stripper upstream of the SCL-MPAS will enhance understanding of the effect of standard and/or project-developed particle conditioning processes.

¹ Atmos. Meas. Tech., 6, 3501–3513, 2013



2.2 Principle of operation / system components

The SCL-MPAS is based on the use of appropriately modulated laser pulses in order to measure the wavelength-dependent absorption of an exhaust aerosol sample. The primary measurand is the photon energy absorbed by exhaust particles at a given wavelength. The absorbed energy is detected by microphones as the amplitude of the generated acoustic waves within a resonant measuring cell.

The prototype setups implemented within the project generally consist of the following parts:

- exhaust sampling subsystem
- tunable wavelength light source, consisting of:
 - a super-continuum white light laser source
 - a compatible monochromator (variable band pass filter)
- photoacoustic resonant cells
- microphones and signal amplification
- microphone signal data acquisition and lock-in amplifier
- computer and interface software for SCL source and monochromator control

Figure 1 shows a schematic representation of the photoacoustic measurement setup based on a super-continuum laser source and compatible optical band pass filter which include an electronic control interface i.e. it is not necessary to incorporate a mechanical/optical beam chopper in order to pulse the beam at the desired acoustic frequency.

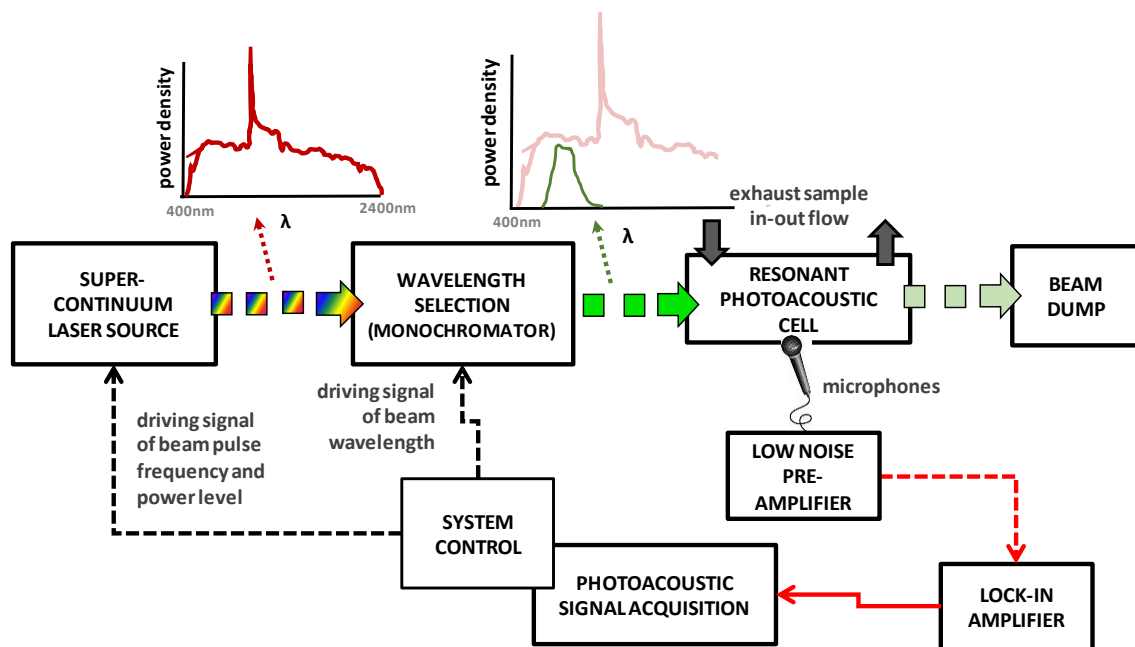


Figure 1. Concept schematic of the SCL-MPAS system that is implemented for demonstration in the SUREAL-23 Project.

The physical layout of the fully-integrated prototype SCL-MPAS system, shown in simplified form in Figure 2 below, is the result of a long series of exploratory experiments involving prototype components arranged in temporary / easily re-configurable bench-top setups where

basic photoacoustic operation with the SCL laser was achieved and numerous basic operation parameters were explored and/or discovered, e.g. optimum pulse modulation scheme of the SCL, variability of sample cell resonant frequency, acoustic coupling between beam path components, etc. An example of the temporary bench-top setups used during the initial exploratory experiments can be seen in Figure 3.

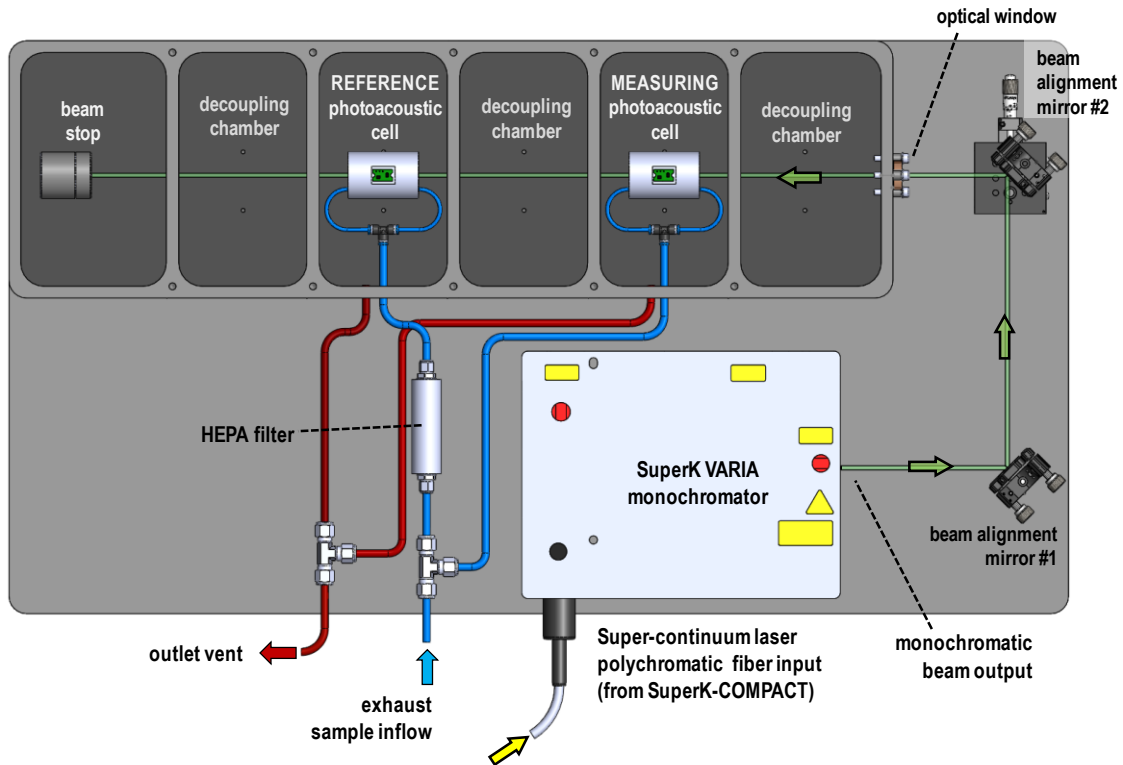


Figure 2: Layout of the main components of the SCL-MPAS system (without microphone pre-amplifiers and data-acquisition electronics).

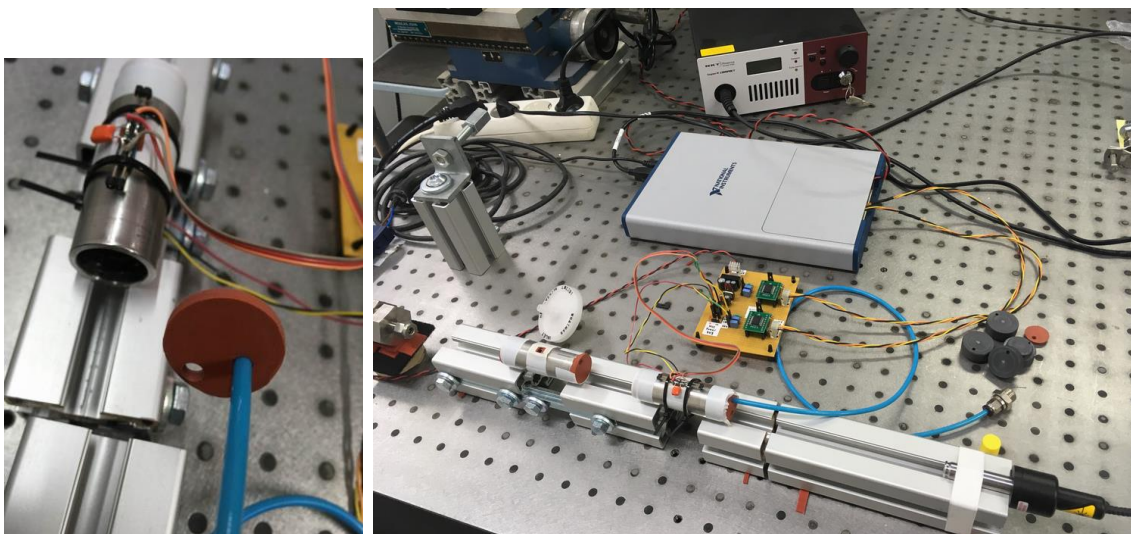


Figure 3: Close view of an experimental PA cell of 20.5 mm internal diameter, azimuthal resonance mode at approximately 9.8 kHz (left) and a snapshot of a bench-top test setup during early exploration of the PA system components.(right)

As would be expected, the easily re-configurable open setups were found to be strongly affected by ambient sound as well as parasitic sound from the supercontinuum laser system. Nonetheless, the main operational aspects of the photoacoustic method could be extensively explored in such open setups using a larger portion of the SCL spectrum (more optical power) than would be appropriate for actual spectroscopic measurements. Of course, transition of the experimental effort to spectrally resolved absorption measurements and dilute aerosols requires integration of the developed photoacoustic system components into an appropriate housing, designed to block ambient sound and to prevent acoustic coupling between system components interacting with the pulsed SCL beam. Such a housing and the associated routing of the SCL beam is illustrated in Figure 4 below.

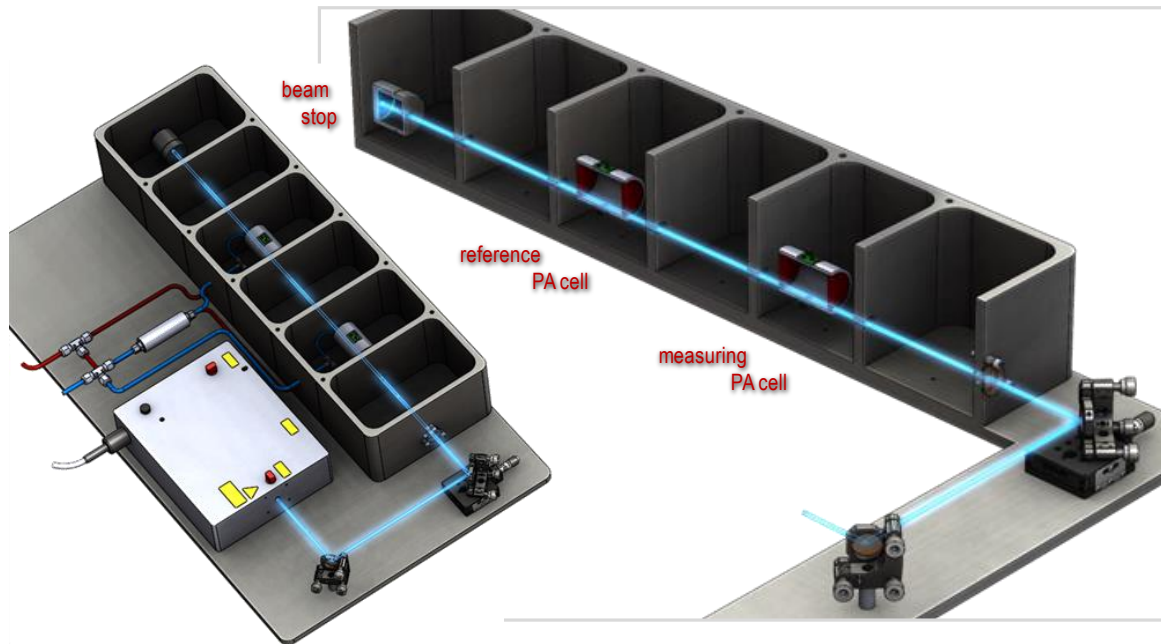


Figure 4: Rendering of the SCL-MPAS physical layout and cut-away view showing the main laser beam path through the system.

The primary sub-components of the SCL-MPAS system are described below.

2.2.1 Resonant photoacoustic cell

Although open space photoacoustic measurement of gases and suspended particles has been demonstrated, in the current context, the laser power available, the exhaust aerosol optical absorption properties as well as the detection requirements all mandate the exploitation of every compatible means for amplifying the photoacoustic response in order to obtain, in the end, a usable signal. In nearly all known applications, the primary means for photoacoustic response enhancement has been the use of a resonant photoacoustic cell, where the pulsed laser light is spatially deployed in such a way so as to excite one of the resonant modes of the air space within the measuring cell. It is important to maximise the spatial correlation between the beam absorption region and the mode shape of the resonant mode as well as to optimise the position of the microphones with respect to the pressure maxima/minima. One of the resonant cell schemes offering the above features is the cylindrical resonant cell, shown in Figure 5 below, excited in its first azimuthal resonance by passing the laser beam very near and parallel to (but not impinging upon) one side of the internal cylinder surface.

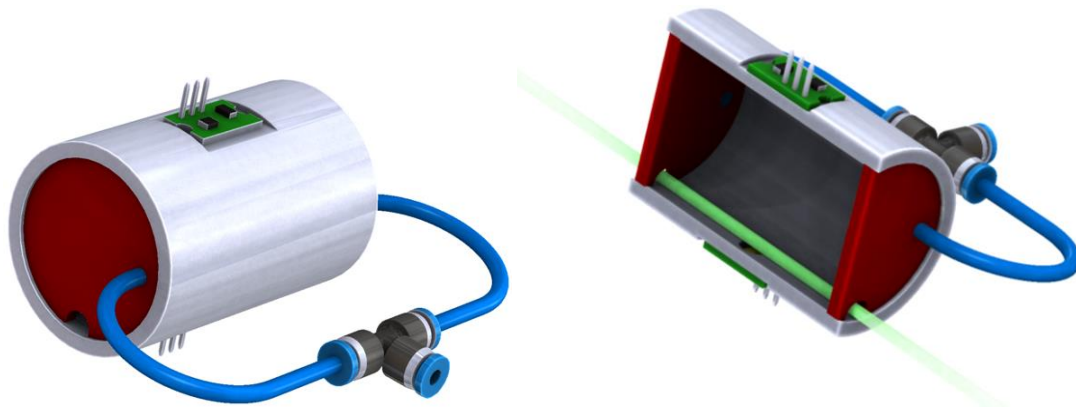


Figure 5: Visualisation of the cylindrical measurement cell design which will be the basis for the SCL-MPAS demonstrator.

In this scheme, the microphones can be alternately located in the pressure maxima/minima, thereby enhancing the detection of the PA cell resonance amplitude, as shown in Figure 6. Given that the pressures at each of the two microphones are always exactly 180° out of phase (antiphase) it is possible to amplify the differential output of the microphone pair, thereby obtaining a higher raw signal ($\times 2$ vs. single microphone) and significant cancellation of common mode parasitic sound especially at frequencies below the resonant frequency.

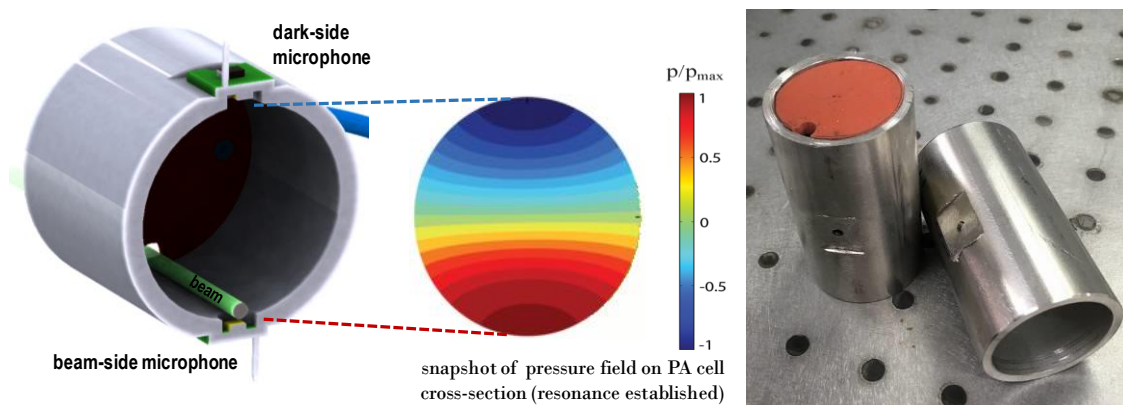


Figure 6: Illustration of the cylinder photoacoustic cell and its first azimuthal resonance mode - as would be seen on the cell cross section - excited by the pulsed laser beam. (left) Example of photoacoustic cells machined for integration in the SCL-MPAS test setups.

2.2.2 Super-continuum laser light source

The specifications of the light source have been chosen in collaboration between NKT and APTL. The tunable wavelength light source used in the developed setup consists of the supercontinuum laser unit *SuperK COMPACT*[®] and a compatible monochromator, the *SuperK VARIA*[®], manufactured for the project by NKT Photonics.

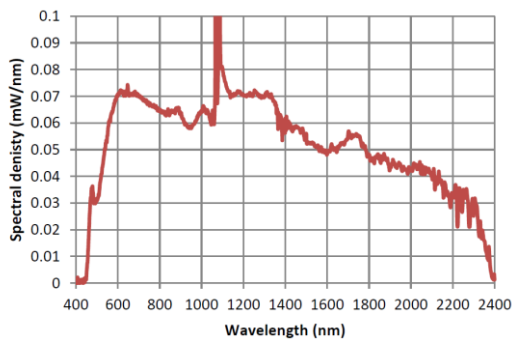
The SuperK COMPACT[®] laser is a low cost supercontinuum white light source delivering diffraction limited beam in the 450 – 2400 nm region with a brightness orders of magnitude larger than that of incandescent lamps and with far greater bandwidth than ASE (amplified spontaneous emission) sources or SLEDs (superluminescent emitting diodes). The supercontinuum light is delivered in a single mode fiber terminated with either a standard FC/PC connector or a compatible collimator.

The SuperK VARIA® is a refraction grating based variable bandpass filter for NKT's SuperK EXTREME® and SuperK COMPACT® supercontinuum lasers that provides a coherent light beam within 400 – 900 nm range and 2-80 $\mu\text{W}/\text{nm}$ output power. The features of the SuperK VARIA® are:

- tunable center wavelength in the 400 - 900 nm range
- short pulses down to 20 ps (superK EXTREME source)
- arbitrary bandwidth (typically 20 – 200 nm)



SuperK COMPACT® typical output spectrum.



Examples of SuperK VARIA® output spectra.

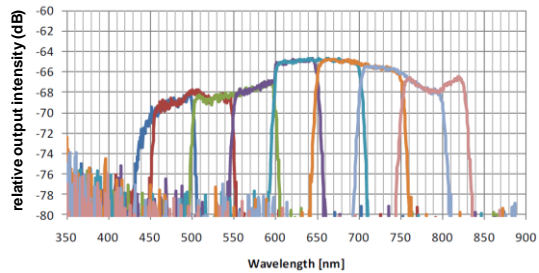


Figure 7: The multi-wavelength supercontinuum laser source system components and associated light spectra provided for the SCL-MPAS demonstrator application.

The SuperK COMPACT® and VARIA® units are connected by optical fiber. The output of the SuperK VARIA® is utilised as a free space beam in order to avoid further power loss. Appropriate mirrors, mounted on precision kinematic stages, will be used to align the beam with the photoacoustic system components, as previously shown in Figure 4. The pulse modulation of the driving beam from the SuperK COMPACT® is controlled by a digital output signal from the *National Instruments® NI 6351* module, itself controlled through the *Labview®* software running on a computer workstation.

The table below presents the technical specifications of the supercontinuum laser system components of the SCL-MPAS system:

NKT Photonics SuperK COMPACT light source

| | |
|--------------------|-------------------|
| Wavelength | 450-2400 nm |
| Total output power | > 110 mW |
| Dimensions | 93 x 221 x 332 mm |
| Weight | 3.8 kg |



SuperK VARIA variable bandpass optical filter (monochromator)

| | |
|-------------------|-------------------|
| Output wavelength | 380 – 880 nm |
| Dimensions | 272 × 212 × 68 mm |
| Weight | 7.9 kg |

2.2.3 Sample handling sub-system

The sample handling subsystem (with flow connection lines) can be seen in Figure 2 and Figure 4. The highly variable composition of the exhaust gas necessitates the continuous monitoring of the photoacoustic absorption spectrum due to the gas components in the exhaust sample. Therefore, in parallel with the main measuring photoacoustic cell (with sub-23 nm particles), a reference cell with a particle-free exhaust sample is also monitored. This necessitates the installation of a HEPA filter on the sample line feeding the reference cell. The sample flow can be driven in two ways, depending on the conditions available from the upstream sample conditioning process:

- a) by the overpressure from the exhaust sample source - most likely the sample conditioning unit, i.e. the sample gas is pushed to the photoacoustic cells.
- b) by suction applied to the outlet port, i.e. the exhaust sample (with / without particles) is pulled into the photoacoustic cells.

Sample driving scheme (b) necessitates a sealed photoacoustic housing and so the optical window at the beam entry port (indicated in Figure 2 and visible in Figure 4) becomes mandatory but the photoacoustic system is not dependent on the availability of overpressure at the outlet of the sample conditioning. Sample driving scheme (a) permits an open (windowless) laser beam entry port, potentially reducing parasitic sound generation that would be emitted by the pulsed heating of the window material.

2.2.4 Microphones and signal conditioning/acquisition components

The most widely employed and straightforward means for detecting the photoacoustic response of the measuring cell is by microphone elements which convert instantaneous pressure to an electronic voltage signal. Other means, such as quartz tuning fork elements in Quartz Enhanced Photo-Acoustic Spectroscopy² – "QEPAS" – have also been demonstrated in trace gas detection applications and offer exceptional immunity from ambient noise. However, QEPAS, which is entirely dependent on the resonant properties of the quartz element, is not readily adapted to the application at hand, namely, that of a dilute aerosol of sub-23 nm particles, due to the very small sample volume actually probed, typically of the order of 1 mm³.

² Review of Scientific Instruments, 76, pp.043105-1, 2005.



Miniature surface-mount MEMS type microphones have been identified as the most suitable type for the current demonstration. Three different models of MEMS microphones have been procured and suitably customised small printed circuit boards have been obtained for each microphone type, as can be seen in Figure 8 with the aim to facilitate their integration into the demonstration SCL-MPAS setups.

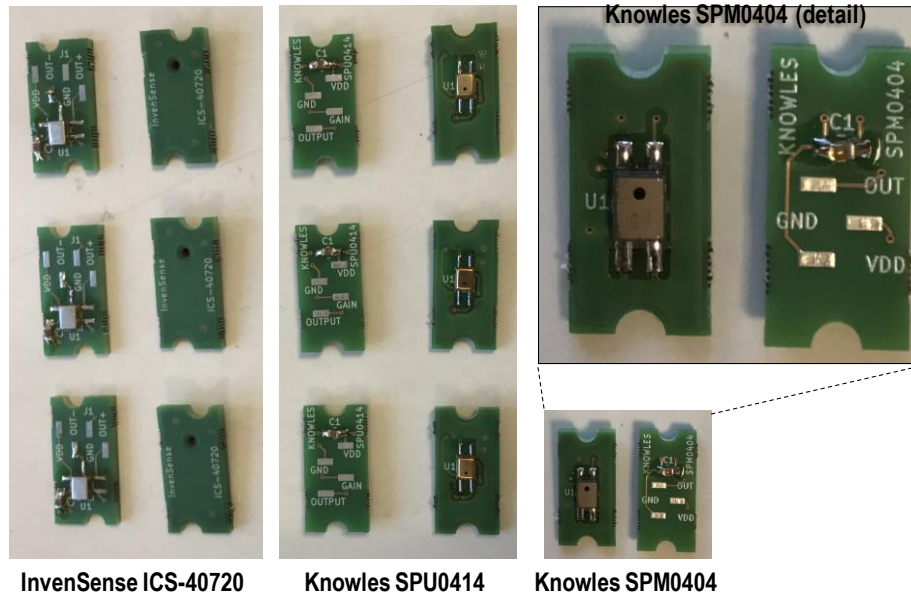


Figure 8: All MEMS microphones that were mounted on printed mini circuit boards for use in the photoacoustic particle spectrometer development and testing.

In all SCL-MPAS setups, the microphones are connected to the amplification circuit either for an absolute sound pressure signal (a single microphone per amplifier) or as a pair to the inverting and non-inverting amplifier inputs (differential signal – each microphone referenced to the other). Development testing was carried out using the perforated board prototype seen on the left in Figure 10, possessing two amplifier channels, while subsequent testing of the SCL-MPAS system will utilize a three channel amplifier circuit, the printed circuit board of which is also shown in Figure 10 (right).

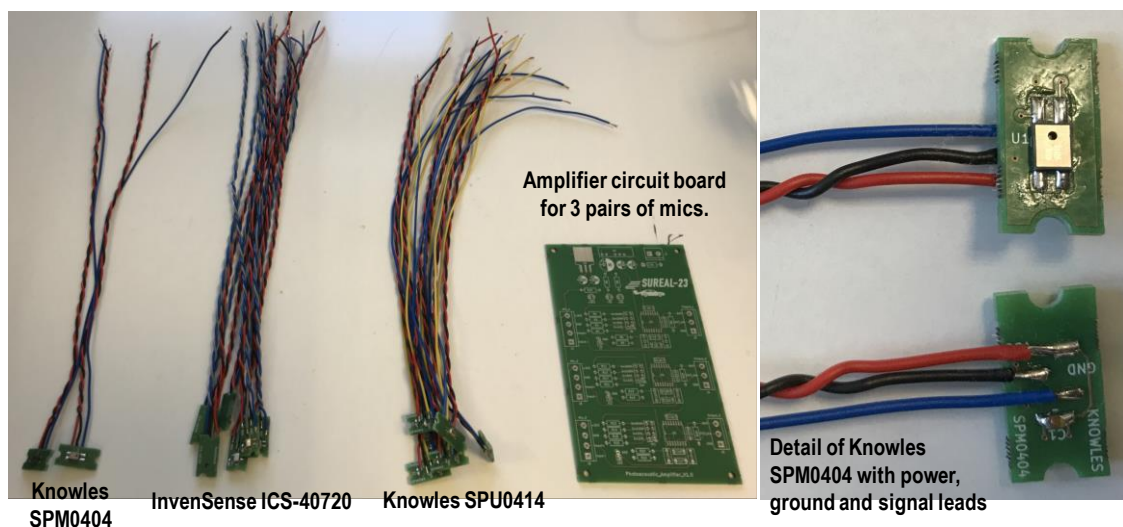


Figure 9: Connection of microphone leads and associated amplifier circuit board.

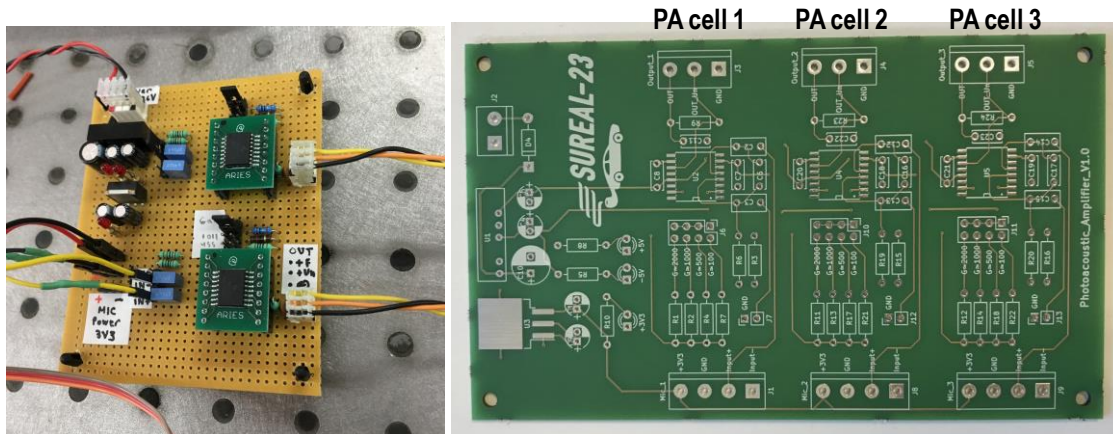


Figure 10: Prototype amplifier circuit for two differential pair PA cells.(left) Detail view of the microphone amplifier circuit board (version 1.0) configured for up to three PA cells, each with one differential pair of antiphase microphones.

The microphone models were chosen on the basis of sensitivity and signal-to-noise ratio (SNR). The main specifications of the MEMS microphones procured are given in the tables below.

Knowles SPM0404HE5H-PB MEMS microphone

| | |
|---------------------------------|-------------------------------------|
| Sensitivity (@1kHz, 0dB=1 V/Pa) | -42 dB ($\approx 79\text{mV/Pa}$) |
| Supply / output signal level | 1.5 - 3.6 V |
| Signal to Noise ratio (@1kHz) | 59 dB |

Knowles SPU0414HR5H-SB MEMS microphone

| | |
|---------------------------------|--------------------------------------|
| Sensitivity (@1kHz, 0dB=1 V/Pa) | -22 dB ($\approx 790\text{mV/Pa}$) |
| Supply / output signal level | 1.5 - 3.6 V |
| Signal to Noise ratio (@1kHz) | 59 dB |

Invensens ICS-40720 ultra low noise MEMS microphone with differential output

| | |
|---|---------------------------------------|
| Sensitivity (@1kHz, 0dB=1 V/Pa), single ended | -38 dB ($\approx 158\text{ mV/Pa}$) |
| Sensitivity (@1kHz, 0dB=1 V/Pa), differential | -32 dB ($\approx 395\text{ mV/Pa}$) |
| Supply / output signal level | 1.5 - 3.6 V |
| Signal to Noise ratio (@1kHz), both modes | 70 dB |

2.2.5 Data acquisition and control sub-system and Lock-in amplifier

At a minimum, the operation of the SCL-MPAS system requires the following data acquisition and control functions:

- analogue voltage input signals acquisition:
 - microphone signals (acquisition frequency: at least $5\times$ the resonant frequency of the photoacoustic cell)
- digital (0 – 5 V) output signal generation:

- laser pulse burst initiation (trigger port of the SuperK COMPACT®)
- laser pulse burst duration (trigger port of the SuperK COMPACT®)
- sampling input valve open command (normally closed valve)
- sampling output (suction) port valve open command
- serial communication for control and status acquisition:
 - supercontinuum laser pulse repetition rate (SuperK COMPACT®)
 - supercontinuum laser number of pulses per programmed burst (SuperK COMPACT®)
 - monochromator low cut filter wavelength (SuperK VARIA®)
 - monochromator high cut filter wavelength (SuperK VARIA®)
 - SuperK COMPACT® and SuperK VARIA® status and error condition flags

In addition to handling signal acquisition and control, the Labview software system is used in order to provide a centralized user interface for the operation of all SCL-MPAS system components. The main panel of this interface is shown in Figure 11.

All photoacoustic detector systems require a lock-in amplifier in order to extract the amplitude of the photoacoustic cell response while ignoring the spurious sound signals that are not tuned in phase and in frequency to the photoacoustic excitation. Analogue and digital lock-in amplifier modules are available for this task. In the current application, the signal acquisition sample rate (1.2 giga-samples per sec.) of the NI 6351 data acquisition module is sufficient to permit – in combination with an intel i5® processor computer workstation – the implementation of a multi-channel lock-in amplifier function as a software module in the Labview virtual instrument that embodies the data acquisition and control operations. Two lock-in amplifier channels are implemented, allowing for the microphone signals of 2 photoacoustic cells to be monitored with a sample frequency approx. 500.000 samples/sec. This arrangement has been found to provide artifact-free acquisition of the photoacoustic sound signals for frequencies up to 12 kHz. For this reason, measurement cells have initially been constructed with internal diameter of 20 mm (as in Figure 3) resonant frequency 9.9kHz. In anticipation of the possibility of a third photoacoustic signal channel, for an additional zero-gas reference, measurement cells of 34 mm internal diameter (as in Figure 5, resonant frequency 6 kHz) have also been constructed.

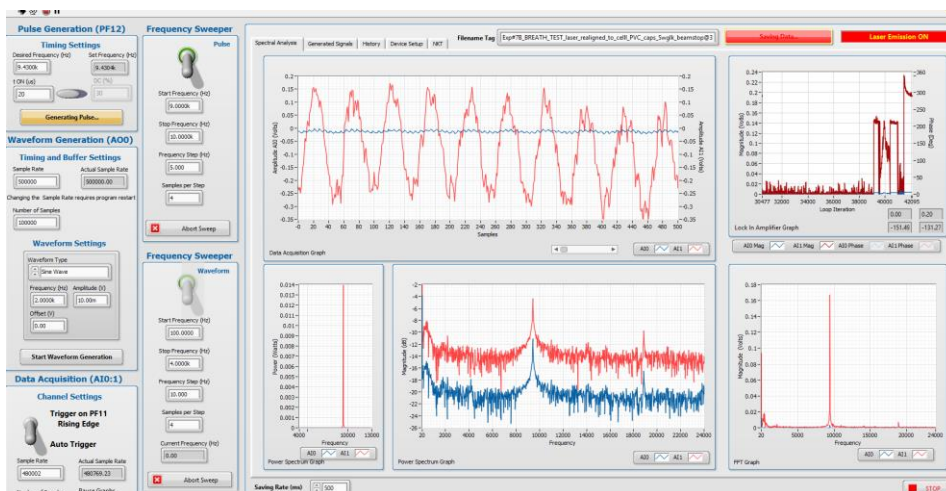


Figure 11: Main display and user interaction panel of the NI Labview® interface developed for the SCL-MPAS instrument.



3 Concluding summary

This report describes the main features of the SCL-MPAS system implemented in the SUREAL-23 Project for the purpose of demonstrating the feasibility of real-time composition analysis of sub-23 nm exhaust aerosols via photoacoustic spectroscopy. The description is focused on the specific sub-components of the SCL-MPAS system which have been constructed/programmed or procured and which will be retained more or less constant throughout the demonstration campaign. This report also describes an initial conceptual and physical system layout that provides for a main measurement cell – probing the multi-wavelength absorption of exhaust aerosol – as well as for a particle-free reference cell which will compensate for cross-sensitivity of the photoacoustic method to the highly variable gas composition of the exhaust sample. It is expected that the relative simplicity of the photoacoustic detection concept will permit numerous reconfigurations of the main components described in order to improve the compatibility of the concept for the application at hand. The SCL-MPAS system description provided herein pertains to the system components design/parameterization following a campaign of many preliminary experiments which were performed with simplified / temporary bench-top system layouts in order to understand the operation of the components and numerous particular aspects of the photoacoustic spectroscopy method.

