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**STANDARD OPERATION PROCEDURES (SOPS)**  
**DELIVERABLE NO. 2.2**

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## Abstract

This document contains draft versions of Standard Operation Procedures (SOPs) for particle measurements with various portable monitors (miniDiSC, DiSCmini, NanoTracer, Partector) and portable samplers (NB2013 sampler, ESPnano) including their application in personal field measurements.

## Keyword list

Standard Operations Procedures (SOPs), nanoparticles, particle measurement, portable monitor, portable sampler, miniDiSC, NanoTracer, Partector, NB2013 sampler, ESPnano

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## 1. Introduction

This document gives the final version of the Standard Operations Procedures (SOPs) for the several instruments that were identified in deliverable 2.1 “List of Candidate Instruments” and were available to be included in the investigations within nanoIndEx. As planned, “prototype instruments” were not described in an SOP.

Other identified instruments as of work package 2.1 could not be located within the consortium (examples: SidePak TSI, Nanocheck GRIMM, PAMS Kanomax) for experimental investigations and they were not provided by the manufacturers.

For the PGP FAP an SOP exists from IFA and cannot be modified within the consortium.

## 2. Standard Operation Procedures

As planned, the following SOPs constitute the harmonized SOPs at the state of the instruments as of May 2016 in all cases. As time moves on, modifications are likely, but will no longer be within the scope of the nanoIndEx project. In cases where SOPs were available from other research projects (example: NanoGEM) those versions are displayed here to document the continuous development of those procedures.

## 2.1 miniDiSC (portable monitor)



Standard Operation Procedure (SOP): WP2-1

# Procedure of particle measurements with the Miniature Diffusion Size Classifier (miniDiSC, FH Nordwestschweiz)

### Last Modification Date

05/2016

### Version

Final English (*Remark: Mostly identical with the corresponding SOP from the NanoGEM-project*)

### Number

2-1

### Responsible for the implementation of this SOP

Martin Fierz

Drafted within the project “Assessment of Individual Exposure to manufactured nanomaterials by means of personal monitors and samplers” (nanoIndEx).

This project is supported by the French National Funding Agency for Research (ANR), the German Federal Ministry of Education and Research (BMBF), the British Technology Strategy Board (TSB) and the Swiss TEMAS AG, under the frame of SIINN, the ERA-NET for a Safe Implementation of Innovative Nanoscience and Nanotechnology. IDÆA-CSIC received funding from FP7 Marie Curie ITN “HEXACOMM”

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## 1. Scope

The scope of this standard operation procedure is the procedure of the data collection and logging as well as the quality control of the determined and logged data from the miniDiSC (FH Nordwestschweiz) including the routine instrumentation maintenance. The scope of this standard operation procedure does extend to the application of the monitor in personal exposure measurements.

## 2. Notes

This standard operation procedure should be used in connection with the Guidance Document (“Assessment of Personal Exposure to Airborne Nanomaterials”).

## 3. Basics

The operating principle of the Miniature Diffusion Size Classifier (miniDiSC) is based on the deposition of charged particles in two consecutive stages and subsequent current measurement from both stages using Faraday cup electrometers. In the miniDiSC, particles get charged in a unipolar diffusion charger, before they are passed on to a dual stage particle deposition system. The first stage contains a diffusion screen where particles are deposited with a size-dependent efficiency. The second stage contains a high efficiency filter, on which all particles that penetrated the first stage are captured. Small particles have a larger diffusivity, and therefore are more likely to be collected in the diffusion stage, while larger particles are more likely to end up in the filter stage. Both stages are connected to electrometers that continuously measure the current, induced by the deposited particles. The average particle size is determined by comparing the currents in the diffusion and the filter stage. The total current and the particle size are then combined to determine the number concentration.

## 4. Instrumentation

The main components of the miniDiSC are:

- miniDiSC instrument
- Pre-separator (6 hole impactor)
- SD-Card for data collection and logging
- Charger/power supply unit

## 5. Measurements

### 5.1 Setting up

Set up the miniDiSC according to the manufacturer’s instructions.

- For long term measurements (e.g. monitoring) at a fixed location, connect the miniDiSC with the AC/DC power adapter and then plug in the power adapter to a 230 V power outlet.
- To use the instrument as a mobile measurement device, the miniDiSC is also equipped with rechargeable batteries (lifetime about 8 hours).
- For data logging, insert the SD card in the card slot on the left side of the instrument.
- Turn on the miniDiSC by pressing the Power Button for 2 seconds. The instrument immediately begins a warm-up following a 300 s countdown sequence. The manufacturer suggests further 10 minutes waiting time to fully warm-up before starting the measurements.

- After warm-up the miniDiSC display shows the main screen containing the particle concentration (N XXXX pt) and average particle diameter (Size XXX nm) on the left, the control signals depending on the status of the miniDiSC: corona voltage (lightning bolt), Flow ( $\Phi$ ), Heating (H), and Recording (blinking circle or number 1 for not re-cording) top right, and the remaining battery power (XX%) bottom right.
- Before start with recording measurements, set the internal clock of the miniDiSC: Press the menu button (middle button) for 3 seconds to move into advanced mode with the auxiliary screens. Press the menu button again to flip through the auxiliary screens. When the date/time screen appears, the year will be blinking for a few seconds. During this time, the year can be changed by pressing the left or right arrows. After a couple of seconds without action, the month starts blinking and can be changed with the arrow keys. This procedure continues until every value is set. Then the "Set Clock?" screen appears. By pressing the right arrow key located below the YES you will save the adjusted date and time. In order to have the clock set to precise timing (i.e. correct seconds) use the following procedure: Set the time in the future, e.g. to the next approaching minute (if it's 14:23:14 now, then set clock to 14:24), then wait until it's really 14:24:00 and then set the clock - in that way, the seconds will also be correct.
- Important: miniDiSC data recording may fail if one starts the measurement but presses the button for a long time (~3s) so that the recording stops again immediately. This status may be identified by a "beep" indicating that the instrument stopped the recording. In that case, the blinking circle in the display would no longer be visible and instead number 1 will be displayed.

## 5.2 Zero check

Before beginning to sample with the miniDiSC, it is important to verify that the instrument is operating normally. This zero check should be performed at least before you start the measurement, and also after you finish:

- After the miniDiSC's warm-up, attach a particle filter (HEPA) to the aerosol inlet.
- The particle concentration should go  $N < 100$  pt in a few seconds. The average particle diameter should show Size --- nm.
- By pressing the right arrow button (▶) the display shows the raw data screen with the two electrometer currents (upper value diffusion stage, lower value filter stage). The stage signals should indeed very close to zero fA.
- Remove the particle filter and press the left arrow button (◀) to go back to the main screen. The instrument is now ready for operation.

## 5.3 Sampling

- If required, connect a sampling tube to the aerosol inlet. Preferably PVC (e.g. Tygon®) tube should be used. Do not use silicone tubing, as these can cause drastic measurement errors.
- The main screen shows the number concentration und mean particle size.
- By pressing the left arrow button (◀) the display shows the surface area screen with a quantity that can be described as "alveolar lung-deposited surface area, A-LDSA" (SA [ $\mu\text{m}^2/\text{cm}^3$ ]).



- To save the measurements to the SD card, start recording by pressing the red record button. Recording is indicated by a blinking closed circle in the top right corner of the display in the main screen.
- By pressing the red record button again for 2 seconds, the recording stops and a beep sound will acknowledge the stop.

#### **5.4 Measurement data**

The recorded measurement data are saved as text files on the SD card. With a card reader, the tab-delimited text files can easily be imported into all common data processing programs. The use of the miniDiSC data conversion tool (version 1.18) is recommended. With this tool, it is easy to load and analyse raw miniDiSC data files, view the data, and export it for further use in other programs.

### **6. Maintenance**

- Clean the impaction surface before use (unscrew the impactor cap, and wipe the flat surface below the thread). Make sure to screw the impactor cap firmly into the impactor to ensure proper functioning of the impactor. Clean the impactor orifices regularly in an ultrasonic bath; place the impactor with the solid side down and the thread facing up into a beaker and fill the beaker with an alcoholic cleaning fluid up to the level of the orifices; make sure not to cover the internal thread of the impactor to avoid degreasing. Place the beaker in an ultrasonic bath and start the cleaning process.
- The wire in the corona charger can become contaminated with a silicon oxide coating, leading to an increase of the corona voltage to 5kV or higher. The instrument will produce an error message ("corona voltage out of range"), telling the user to clean the wire when this is due. The user can clean the corona wire according the procedure described in the corresponding application note: <http://fierz.ch/minidisc/pdf/miniDiSCApplication-Note7.pdf>. The instrument has to be serviced and calibrated once per year. Alternatively, the miniDiSC reading should regularly be crosschecked against a CPC in regular intervals. Remember: the miniDiSC is not a high precision instrument. Deviations of up to  $\pm 30\%$  compared to a CPC are tolerable.

### **7. Quality assurance**

Measures for quality assurance:

- Periodical testing for leaks of the miniDiSC
- Periodical check of the volumetric flow rate (Aerosol sample flow 1 lpm)
- At high humidity, the electrometer zero offsets can drift. The instrument is verified for up to 80% relative humidity at 20°C. Up to this point, drifts are small (at most a few fA). At higher humidity, proper operation of the instrument is not guaranteed. Please check the stability of the zero offsets when measuring at high humidity, and if possible, dry the aerosol upstream of the miniDiSC.
  - Periodically check the electrometer zero offsets.
  - Report anomalies during sampling or maintenance

## 2.2. NanoTracer (portable monitor)



Standard Operation Procedure (SOP): WP2-2

### Procedure of particle measurements with the Aerasense NanoTracer PNT 1000 (ANT, Philips-Aerasense)

Date

05/2016

Version

Final English

*(Remark 1: Mostly Identical with the corresponding SOP from the NanoGEM-project)*

*(Remark 2: Possibly a major revision of the software of the instrument is in preparation)*

Number

2-2

Responsible for the implementation of this SOP

Dirk Dahmann

Drafted within the project “Assessment of Individual Exposure to manufactured nanomaterials by means of personal monitors and samplers” (nanoIndEx).

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## 1. Scope

The scope of this standard operation procedure is the procedure of the data collection and logging as well as the quality control of the determined and logged data from the ANT including the routine instrumentation maintenance. The scope of this standard operation procedure does extend to the application of the monitor in personal exposure measurements.

## 2. Notes

This standard operation procedure should be used in connection with the Guidance Document (“Assessment of Personal Exposure to Airborne Nanomaterials”). This SOP is for the NanoTracer by Philips Aerasense. Meanwhile the instrument has been licensed to Oxility bv., Eindhoven, the Netherlands and upgraded under the name of NanoTracer XP. Its operation may differ from the one described here.

## 3. Basics

The Philips Aerasense NanoTracer (ANT) is a battery-powered handheld device for measuring the number concentration, mean particle size and (depending on the data evaluation software version) lung deposited surface area concentration of airborne particles in a particle size ranging from 10 to 300 nm (20 – 120 nm mean particle size) with an internal clock and internal permanent data storage. The operating principle of the NanoTracer is based on unipolar diffusion charging of the particles, followed by a low efficiency electrostatic precipitator (ESP) and a filter inside a Faraday cup electrometer. A square wave voltage is applied to the ESP, so that depending on the voltage level (high or low), a different particle fraction is removed inside the ESP and two independent currents are measured with the electrometer. The number concentration, mean particle size and lung deposited surface area concentration are derived from these currents.

## 4. Instrumentation

The main components of the ANT are:

- ANT
- Belt holster, belt, hose with clip for personal measurements
- Charger/power supply unit

In addition, for data preparation a suitable PC or laptop with installed software "NanoReporter" (supplied by Philips Aerasense) and a suitable USB cable is required.

The device operates in two modes, fast (FST) and advanced (AVD), which are selected using the on/off switch. In fast mode, the integration time is at least 3 seconds, a constant voltage is applied to the ESP and the lung deposited surface area concentration and average particle size are hence not determined. Instead of that, a mean particle size of 50 nm is assumed by the instrument. In advanced mode the integration time of 16 seconds, an average particle size is determined and stored. The improved version, NanoTracer XP, has a time resolution of up to 1s for the fast mode and of 10s for the advanced mode.

With the “Zero” button on the bottom of the display, a determination of the offset of the measuring sensor can be enforced during measurement. With the adjacent "Mark" button, the time stamp of the current record will be marked at that particular time. This feature is meant to be used to mark certain events that have to be documented on an appropriate form.

The operating principle of the ANT is based on measuring the electrical charges of the particles. A detailed description can be found in the manual.

## 5. Measurements

### 5.1 Setting up

To adjust the settings, the device has to be connected to the PC with a USB cable. When starting the "NanoReporter" software, the device will be detected automatically by the PC. The device does not have to record. In the software, all necessary adjustments are reachable in the sub-menu "Settings" of the menu "Devices". This menu can be selected only when a device is connected. The default settings are

- time with PC time sync (button "Synchronize with PC")
- Fast sampling - sampling period = "00:00:03"
- Intermittent mode = "Off"

All other settings are not relevant for the measurement. After changing any setting they have to be transferred to the device by activating the button "Transfer to device".

### 5.2 Sampling

- If required, connect a sampling tube to the aerosol inlet. Preferably PVC (e.g. Tygon®) tube should be used. Do not use silicone tubing, as these can cause drastic measurement errors. The desired mode is selected by the position of the on/off switch. After a warm-up phase, the offset of the measuring sensor is determined. Then the measurement starts automatically.
- By pressing the "Mark" button, the mark time and a mark letter will be stored in the current record. The mark time and letter is shown in the display. It has to be documented on an appropriate form.
- Sudden temperature changes may induce drifting measurement signals. In case this drift cannot be corrected quick enough by the periodically applied automatic zero-offset check, a manual zero-offset check can be initiated at any time by pressing the zero-check button
- Switching off the device ends the measurement.

For errors or failures in the measurement process address the following table for rescue:

Problem	Possible reason	Solution
The actual time in the display is 1:00:00	The backup battery for the clock is empty	The battery has to be changed by the manufacturer
The device switches itself off immediately	The battery is empty	Charge the battery or operate the unit with charger
The time is not updated every second	The „Mark“ button was pressed	Wait a few seconds
	The processor of the device is in an undefined status	Reset the processor as described in the manual

### **5.3 Measurement data**

The measurement data are saved as binary files in the internal memory of the device and can be read, saved on a PC and exported as text files only with the supplied software “NanoReporter”. They are listed in the sub-menu “Download” of the menu “Settings”. Further calculations and analyses will be made in appropriate spreadsheet software like “Microsoft Excel”.

## **6. Maintenance**

The Aerasense NanoTracer does not require maintenance by the user. At regular intervals, depending on the absolute running time, it must be calibrated by the manufacturer. In this case, "service required" is displayed.

## **7. Quality Assurance**

Measures for quality assurance:

- Periodical comparison with other particle counters and sizers like the CPC or SMPS
- Periodically check the electrometer zero offsets.
- Report anomalies during sampling or maintenance

## 2.3 Partector (portable monitor)



Standard Operation Procedure (SOP): WP2-3

# Procedure of particle measurements with the Partector (naneos particle solutions gmbh)

Date

05/2016

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2-3

Responsible for the implementation of this SOP

Martin Fierz

Drafted within the project “Assessment of Individual Exposure to manufactured nanomaterials by means of personal monitors and samplers” (nanoIndEx).

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## 1. Scope

The scope of this standard operation procedure is the procedure of the data collection and logging as well as the quality control of the determined and logged data from the Partector (naneos particle solutions) including routine instrumentation maintenance. The scope of this standard operation procedure does extend to the application of the monitor in personal exposure measurements.

## 2. Notes

This standard operation procedure should be used in connection with the Guidance Document (“Assessment of Personal Exposure to Airborne Nanomaterials”).

## 3. Basics

The operating principle of the Partector is based on the pulsed unipolar diffusion charging of nanoparticles, followed by subsequent non-contact detection with a Faraday cage electrometer. In the Partector, particles are charged with a unipolar diffusion charger that is operated in a pulsed mode. An ion trap removes excess ions, and then the charged aerosol pulses flow through a Faraday cage, which is grounded via an electrometer. In consequence, a periodic signal is measured at the electrometer, the amplitude of which is determined in the instrument. The amplitude of this signal is proportional to the charge carried by the aerosol pulses, i.e. it is proportional to the signal of standard diffusion charging sensors, and can therefore be calibrated to correspond to lung-deposited surface area concentration in the alveolar region of the lung of a reference worker, which is what the instrument reports.

## 4. Instrumentation

The main components of the Partector are:

- Partector instrument
- Micro-SD-card for data collection and logging
- USB Charger/power supply unit

## 5. Measurements

### 5.1 Setting up

Set up the Partector according to the manufacturer’s instructions.

- For long-term measurements (e.g. monitoring) at a fixed location, connect the Partector to the USB power adapter (or a USB port of a computer).
- To use the instrument as a mobile measurement device, the Partector is also equipped with a rechargeable battery (lifetime about 10 hours). Any commercial USB battery pack can be used to extend the battery lifetime.
- For data logging, insert the micro SD card in the card slot on the left side of the instrument.
- Turn on the Partector by pressing the power button. The Partector takes about 15 seconds to start up, and automatically begins logging data to the micro SD card. The manufacturer recommends waiting another minute before the actual measurement, as the signal is a bit less stable just after start up.
- The Partector display shows the main screen containing the alveolar lung-deposited particle surface area concentration (A-LDSA  $\mu\text{m}^2/\text{cm}^3$  XXXX.X) in the centre, a status bar at

the top of the screen containing date and time, charging symbol (lightning bolt), recording symbol (blinking red circle) and remaining battery power (battery symbol). Below the A-LDSA-value the maximal value of the current measurement is displayed (XXXX.X), followed by the date and time when this value occurred.

### **5.2 Zero check**

The Partector always logs data when it is turned on with a micro SD card inserted. To verify that the instrument is operating normally, a zero check at the start and end of the measurement should be performed.

- One minute after start up, attach a particle filter (HEPA) to the aerosol inlet.
- The A-LDSA concentration should go to  $< 2 \mu\text{m}^2/\text{cm}^3$  within a few seconds. Leave the HEPA filter on for 30 seconds to have a clear signal of the zero check in the data file.
- Repeat the zero check after the measurement

### **5.3. Sampling**

- If required, connect a sampling tube to the aerosol inlet. Preferably PVC (e.g. Tygon®) tube should be used. Do not use silicone tubing, as these can cause drastic measurement errors. The main screen shows the A-LDSA concentration
- To stop the measurement, turn off the instrument.
- Important: Never remove the micro SD card during recording, as the file will be corrupt if not switched off properly.

### **5.4. Measurement data**

The recorded measurement data are saved as text files on the micro SD card. With a card reader, the tab-delimited text files can easily be imported into all common data processing programs. The use of the Partector data conversion tool is recommended. With this tool, it is easy to load and analyse Partector data files, view the data, and export it for further use in other programs.

## **6. Maintenance**

- The instrument has to be serviced and calibrated once per year. The instrument will display a “calibration outdated” reminder during start up if the calibration is older than one year.
- The wire in the corona charger can become contaminated with a silicon oxide coating. The user can clean the corona wire by opening the corona service hatch at the back of the instrument, and cleaning the wire by wiping it with an artist’s brush. The instrument will produce an error message telling the user to clean the wire when this is due.
- If possible, the instrument should be checked against another A-LDSA instrument before and after cleaning the corona wire. This can be a second Partector, a TSI NSAM (or similar device), or a CPC if an aerosol of defined diameter is available (e.g. after a DMA).

## **7. Quality Assurance**

Measures for quality assurance

- The Partector checks its own status by monitoring several critical parameters, and displays warnings and errors if any of these are outside their tolerance values. These self-

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checks include the charging current in both on and off state of the charger, the electrometer zero offset, and the relative humidity in the instrument.

- The Partector has no internal flow sensor and relies on stable operation of its pump over time. A periodic check of the volumetric flow rate (sample flow 0.5 lpm) is therefore strongly recommended.
- The Partector monitors humidity in the instrument and will produce a warning on the display if it is higher than 75%. In this case, data should be checked carefully for plausibility, and the use of a small dryer upstream of the Partector is recommended.
- Report anomalies during sampling and maintenance.
- Use the java data analysis tool provided by the manufacturer to view the data, as this tool will also produce warnings based on the data file.

## 2.4 NB2013 sampler (portable sampler)



Standard Operation Procedure (SOP): WP2-4

# Procedure of particle measurements with the NB2013 sampler (CEA)

Date

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Responsible for the implementation of this SOP

Simon Clavaguera

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## 1. Scope

The scope of this standard operating procedure is the procedure for running sampling using the NB2013 aerosol sampler (NanoBadge V3). This SOP includes the routine instrumentation maintenance.

## 2. Notes

This standard operating procedure is used along with the standard operating procedures from NanoGEM and NanoIndEx projects related to the use of personal samplers and monitors.

This standard operation procedure should be used in connection with the Guidance Document (“Assessment of Personal Exposure to Airborne Nanomaterials”).

## 3. Basics

The operating principle of the NB2013 device is based on the sampling of the respirable particle size fraction using an impactor ( $d_{50}$  at 4  $\mu\text{m}$ , as defined by the ISO7708 document) followed by the collection of the particles on a filter. The NB2013 sampler can be used for stationary measurements or personal sampling in the breathing zone, powered by an internal battery allowing up to 8 hours of continuous operation. The filters are analysed offline using the state-of-the-art techniques listed in the nanoIndEx project, namely SEM, ICP-MS or TXRF.

## 4. Instrumentation

The main components of the NB2013 aerosol sampler are:

- NB2013 sampler.
- Filter units (single use) in individual zip bags. The unit is a sealed cassette containing a polycarbonate track-etched membrane to collect particles and equipped with a RFID chip to store data (sampling time, date, flow rate, errors, worker ID, sample ID, ...).
- Impactor (the sampler can be operated with or without impactor).
- ID badge (personal use, one for each person operating the sampler).
- USB cable and power supply unit.

## 5. Measurements

### 5.1 Setting up

Set up the NB2013 sampler according to the following instructions.

- The NB2013 sampler is equipped with a rechargeable battery (lifetime up to 16 hours). It can be charged using the provided USB charger. Both lights will blink when the sampler is connected to a power source and the battery is charging. The battery is fully loaded when the lights go out. The sampling will not start unless the charge remaining in the battery is sufficient for 8 hours of operation.
- Before starting the sampler, remove the plugs on the filter unit and connect it to the sampler by pushing it down onto the white inlet barb located at the top of the device. The filter must be pushed all the way to ensure that it sits tight or the sampler will not start. Keep the plugs and the zip bag of the filter unit as you will need them to seal the exposed filter unit after sampling. Connect the impactor on top of the filter unit if necessary.
- To turn on the sampler, press the button on the front of the device while holding the ID badge against the marked area on the back of the sampler until both lights on the device

turn green. A faint sound comes from the pump of the sampler when it is running properly.

- To stop the sampler, hold the button for a few seconds until the lights go out. The sampling time is automatically logged.

### 5.2 Zero check

Starting the sampler initiates a self-test routine. The device emits an alarm sound and shuts itself down if an issue is detected. The colour of the LEDs indicates what kind of failure was diagnosed.

Left light (closest to the filter)	Right light (furthest from the filter)	Action required
Red	Red	Insufficient battery charge. Connect the sampler to a power source.
Green	Red	Sampling issue. Check that the filter unit sits tight on the sampler. If it does not solve the problem, try another filter unit.
Green	Green	The device is operating correctly.

### 5.3. Sampling

Once it has been turned on, the NB2013 device can be placed near a potential particle emission source, worn by a worker near the breathing zone in a chest pocket, or kept far from the release zone for background measurements. The sampler will turn down when the battery is exhausted, if it is manually switched off, or in case of sampling failure (e.g. leak in the filter unit or clogging of the collection filter). The sampling time is automatically logged in all cases. At the end of the sampling, remove the impactor and filter unit. Seal the filter unit with the plugs and keep in a closed zip bag for offline analysis.

### 5.4. Measurement data

The filters are analyzed offline at CEA premises. The measurement parameters (user ID, filter ID and sampling time) are contained in the filter unit which is sent for analysis. It should not be opened by the user and kept sealed in a dry and clean environment prior to analysis.

## 6. Maintenance

There is no maintenance required on the user side, apart from loading the battery and keeping track of the filter units. If the outside of the instrument is contaminated with dust, it can be cleaned using wet wipes. If the interior is contaminated it has to be shipped in a sealed zip bag to the manufacturer for a proper decontamination.

## 7. Quality Control

Measures for quality assurance:

- The NB2013 sampler runs a self-test at the beginning of the sampling and periodically during operation. The tests include the battery state and the pressure (detection of leaks or clogging of the filter unit).

- For each measurement, record the position of the sampler, the ID of the filter unit, the temperature and the relative humidity in a logbook. Keep a manual record of the sampling time to cross-check with the built-in time-logging system.
- Report anomalies during sampling and maintenance.
- Keep the filter units sealed in a dry and clean environment before and after sampling.



## 2.5 ESPnano (portable sampler)



Standard Operation Procedure (SOP): WP2-5

### Procedure of particle measurements with the ESPnano

Date

05/2016

Version

Final English

Number

2-5

Responsible for the implementation of this SOP

Ana Maria Todea

Drafted within the project “Assessment of Individual Exposure to manufactured nanomaterials by means of personal monitors and samplers” (nanoIndEx).

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## 1. Scope

The scope of this standard operation procedure is the procedure for sampling with the ESPnano aerosol sampler. This SOP includes the routine instrumentation maintenance.

## 2. Notes

This standard operation procedure should be used in connection with the Guidance Document (“Assessment of Personal Exposure to Airborne Nanomaterials”).

## 3. Basics

The ESPnano is a portable particle sampler designed to collect representative samples of airborne particles of all sizes. The operating principle of the ESPnano device is based on the use of a high-voltage electrical field to simultaneously charge and collect airborne particles onto TEM grids or metallic foil substrates, for analysis by electron microscopy or other microanalysis method.

## 4. Instrumentation

The main components of the ESPnano sampling kit are:

- ESPnano model 100 sampler
- White “sniffer tube” for directional sampling
- Protective caps for inlet and sampling ports
- Numbered sampling “keys” in storage holders, pre-loaded with TEM grids or metal disks

## 5. Measurements

### 5.1 Setting up

Set up the ESPnano according to the manufacturer’s instructions.

- Estimate or measure the particle concentration in the environment you wish to sample by using a particle counter.
- Insert sample “key” loaded with a fresh TEM grid or metallic media for SEM analysis into side port with the sample media facing upward and press firmly until it stops.
- Insert white “sniffer tube” for directional sampling into the inlet port.
- To turn on the ESPnano press any button on the control pad and the first of the menu screens will appear.
  - To set the sample duration for the test advance to the second menu “ENTER TEST TIME” by pressing the right arrow button. Use the down arrow key to select “time” and left/right arrow keys to adjust the time for the desired sampling duration that corresponds with the particle concentration of the test environment. For collecting samples longer than 500 s onto the same media (not recommended), once the first sample is complete push the directional buttons to return to “time” in second menu and reselect the desired sample time.

### 5.2. Sampling

Once the sampling time was selected the particle collection can be started by pressing the enter button with the cursor next to “time”. The pump will begin to operate to purge (default purge time 15 s) the ESPnano prior to sampling. The screen will advance to the third menu

and the letters “PRG” will appear on the screen. After the purging cycle, the corona will engage and particle collection will start. The display indicates a countdown of the sampling time. When it reaches zero the sample is complete. At the end of the sampling, remove the key and store in sealed protective holder for offline analysis.

For troubleshooting or particles low collection efficiency in the measurement process address the following table for rescue:

<b>Problem</b>	<b>Possible reason</b>	<b>Action required</b>
The ESPnano does not turn ON	The batteries are empty	Change the batteries
ESPnano is not operating as expected/ collection efficiency less than 80-95%	Incorrect default settings	Set the proper default settings
	Battery voltage lower than 2.3 V	Check the battery voltage while the ESP is running; if needed change the battery
	Moist aerosol	Use a drying system before sampling the aerosol
Damaged TEM grids	Interaction with the accelerated particles	Choose grids with higher mesh count or a more robust film Reduce the corona voltage slightly

**5.3. Measurement data**

The loaded TEM grids or metallic foil substrates are analysed offline. The measurement parameters (type of sample substrate on the key, material collected, particle concentration, sampling time, etc.) should be recorded for each sample substrate used. After use, wheatear the sample substrate is left on the key or removed and stored elsewhere it is important to be protected from outside sources. For longer storage periods, the samples may be stored under vacuum.

**6. Maintenance**

There is minor maintenance required on the user side. To prevent contamination, the inlet and sampling ports have to be capped when not in use and the sniffer tube stored in a sealed container. If the sample keys are re-used or contaminated, they should be cleaned with alcohol or solvent and dried with lint free wipes. The batteries should be changed if their voltage drops below about 2.3 V and removed if the ESPnano is stored for longer than 2 months.

**7. Quality Control**

- Measures for quality assurance
- Check regularly if the default settings (pump voltage 2.9 V, corona voltage 5.0 kV) are set properly.
- Regular check of the battery voltage (at least 2.3 V) while collecting a sample

## 2.6 DiSCmini (portable monitor)



Standard Operation Procedure (SOP): WP2-6

### Procedure of particle measurements with the Miniature Diffusion Size Classifier (DiSCmini, Testo AG)

Date

05/2016

Version

Final English

*(Remark: The DiSCmini is the current commercially available version of the miniDiSC (see SOP 2.1))*

Number

2-6

Responsible for the implementation of this SOP

Martin Fierz

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## 1. Scope

The scope of this standard operation procedure is the procedure of the data collection and logging as well as the quality control of the determined and logged data from the DiSCmini (Testo AG) including the routine instrumentation maintenance. The scope of this standard operation procedure does extend to the application of the monitor in personal exposure measurements.

## 2. Notes

This standard operation procedure should be used in connection with the Guidance Document (“Assessment of Personal Exposure to Airborne Nanomaterials”).

## 3. Basics

The operating principle of the Miniature Diffusion Size Classifier (DiSCmini) is based on the deposition of charged particles in two consecutive stages and subsequent current measurement from both stages using Faraday cup electrometers. In the DiSCmini, particles get charged in a unipolar diffusion charger, before they are passed on to a dual stage particle deposition system. The first stage contains a diffusion screen where particles are deposited with a size-dependent efficiency. The second stage contains a high efficiency filter, on which all particles that penetrated the first stage are captured. Small particles have a larger diffusivity, and therefore are more likely to be collected in the diffusion stage, while larger particles are more likely to end up in the filter stage. Both stages are connected to electrometers that continuously measure the current, induced by the deposited particles. The average particle size is determined by comparing the currents in the diffusion and the filter stage. The total current and the particle size are then combined to determine the number concentration.

## 4. Instrumentation

The main components of the DiSCmini are:

- DiSCmini instrument
- Pre-separator (6 hole impactor with cut-off at 0.7  $\mu\text{m}$ )
- SD-Card for data collection and logging
- Charger/power supply unit

## 5. Measurements

### 5.1 Setting up

Set up the DiSCmini according to the manufacturer’s instructions.

- For long term measurements (e. g. monitoring) at a fixed location, connect the DiSCmini with the AC/DC power adapter and then plug in the power adapter to a 230 V power outlet.
- To use the instrument as a mobile measurement device, the DiSCmini is also equipped with rechargeable batteries (lifetime about 8 hours).
- For data logging, insert the SD card in the card slot on the left side of the instrument.

- Turn on the DiSCmini by pressing the Power Button for 2 seconds. The instrument immediately begins a warm-up following a 300 s countdown sequence. The manufacturer suggests further 10 minutes waiting time to fully warm-up before starting the measurements when starting the instrument from a cold state.
- After warm-up the DiSCmini display shows the main screen containing the particle concentration (N XXXX pt) and average particle diameter (Size XXX nm) on the left, the control signals depending on the status of the DiSCmini: corona voltage (lightning bolt), Flow ( $\Phi$ ), Heating (H), and Recording (blinking circle or number 1 for not recording) top right, and the remaining battery power (XX%) bottom right. Pressing the left arrow brings up the surface area screen (A-LDSA value). Pressing the right arrow from this screen brings the main display back.
- If using the impactor, unscrew it from the impactor cap from the impactor body and check that the impaction surface is clean. See the maintenance section if the impactor requires cleaning.
- Before start with recording measurements, set the internal clock of the DiSCmini: Press the menu button (middle button) for 3 seconds to move into advanced mode with the auxiliary screens. Press the menu button again to flip through the auxiliary screens. When the date/time screen appears, the year will be blinking for a few seconds. During this time, the year can be changed by pressing the left or right arrows. After a couple of seconds without action, the month starts blinking and can be changed with the arrow keys. This procedure continues until every value is set. Then the “Set Clock?” screen appears. By pressing the right arrow key located below the YES you will save the adjusted date and time. In order to have the clock set to precise timing (i.e. correct seconds) use the following procedure: Set the time in the future, e.g. to the next approaching minute (if it's 14:23:14 now, then set clock to 14:24), then wait until it's really 14:24:00 and then set the clock - in that way, the seconds will also be correct.
- Important: DiSCmini data recording may fail if one starts the measurement but presses the button for a long time (~3s) so that the recording stops again immediately. This status may be identified by a “beep” indicating that the instrument stopped the recording. In that case, the blinking circle in the display would no longer be visible and instead number 1 will be displayed.

## 5.2 Zero check

Before beginning to sample with the DiSCmini, it is important to verify that the instrument is operating normally. This zero check should be performed at least before you start the measurement, and also after you finish:

- After the DiSCmini's warm-up, attach a particle filter (HEPA) to the aerosol inlet. The zero check starts automatically at the end of the warm-up time.
- The particle concentration should go  $N < 100$  pt in a few seconds. The average particle diameter should show Size --- nm.
- By pressing the right arrow button (▶) the display shows the raw data screen with the two electrometer currents (upper value diffusion stage, lower value filter stage). The stage signals should be below 1 fA.



- Remove the particle filter and press the left arrow button (◀) to go back to the main screen. The instrument is now ready for operation.

### 5.3 Sampling

- If required, connect a sampling tube to the aerosol inlet. Preferably PVC (e.g. Tygon®) tube should be used. Do not use silicone tubing, as these can cause drastic measurement errors. The main screen shows the number concentration and mean particle size.
- By pressing the left arrow button (◀) the display shows the surface area screen with a quantity that can be described as "alveolar lung-deposited surface area, A-LDSA" (SA [ $\mu\text{m}^2/\text{cm}^3$ ]).
- To save the measurements to the SD card, start recording by pressing the red record button. Recording is indicated by a blinking closed circle in the top right corner of the display in the main screen.
- By pressing the red record button again for 2 seconds the recording stops and a beep sound will acknowledge the stop.

### 5.4 Measurement data

The recorded measurement data are saved as text files on the SD card. With a card reader, the tab-delimited text files can easily be imported into all common data processing programs. The use of the DiSCmini data conversion tool (version 1.18) is recommended. With this little tool, it is easy to load and analyse raw DiSCmini data files, view the data, and export it for further use in other programs.

## 6. Maintenance

- Clean the impaction surface before use (unscrew the impactor cap, and wipe the flat surface below the thread). Make sure to screw the impactor cap firmly into the impactor to ensure proper functioning of the impactor. Clean the impactor orifices regularly in an ultrasonic bath; place the impactor with the solid side down and the thread facing up into a beaker and fill the beaker with an alcoholic cleaning fluid up to the level of the orifices; make sure not to cover the internal thread of the impactor to avoid degreasing. Place the beaker in an ultrasonic bath and start the cleaning process.
- The instrument has to be serviced and calibrated once per year. Alternatively, the DiSCmini reading should regularly be cross-checked against a CPC in regular intervals. Remember: the DiSCmini is not a high precision instrument. Deviations of up to  $\pm 30\%$  compared to a CPC are tolerable.

## 7. Quality assurance

Measures for quality assurance:

- Periodical testing for leaks of the DiSCmini
- Periodical check of the volumetric flow rate (Aerosol sample flow 1 lpm)
- At high humidity, the electrometer zero offsets can drift. The instrument is verified for up to 80% relative humidity at 20°C. Up to this point, drifts are small (at most a few fA). At higher humidity, proper operation of the instrument is not guaranteed. Please check the stability of the zero offsets when measuring at high humidity, and if possible, dry the aerosol upstream of the DiSCmini.

- Connect the DiSCmini to an HEPA filter for one minute at the end of each sampling period. It will allow to observe the zero offsets and to see if the instrument has been drifting.
- Periodically check the electrometer zero offsets.
- Report anomalies during sampling or maintenance