

## **Size distribution analysis with the Nanoparticle Nebulizer Model 3485 in combination with an SMPS (spray characterisation)**

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### Version

1.0 English

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## 1 Scope

The scope of this standard operation procedure is the description of the procedure of the particle measurements with the Nanoparticle Nebulizer (Model 3485, TSI) in combination with a Scanning Mobility Particle Sizer (SMPS Model 3963, TSI).

## 2 Basics Nanoparticle Nebulizer

The Nanoparticle Nebulizer Model 3485 aerosolizes aqueous suspensions of particles for analysis by a TSI SMPS™ or other gas phase particle instrument. The liquid sample is metered into the instrument via a peristaltic pump, combined with a known ratio of pure water for online dilution, and atomized into ultrafine droplets using a Venturi effect at a controlled temperature. An impaction pin positioned within 0.1 mm of the nebulizer nozzle removes the largest liquid droplets. The remaining wet aerosol has a nominal droplet size of 300 nm. The aerosol is conditioned at an elevated temperature to evaporate liquid, leaving only the particles and dissolved residue within the sample. The particles are combined with dry, filtered, make-up air. Up to 2 l/min of dry aerosol flow can be drawn from the aerosol outlet.

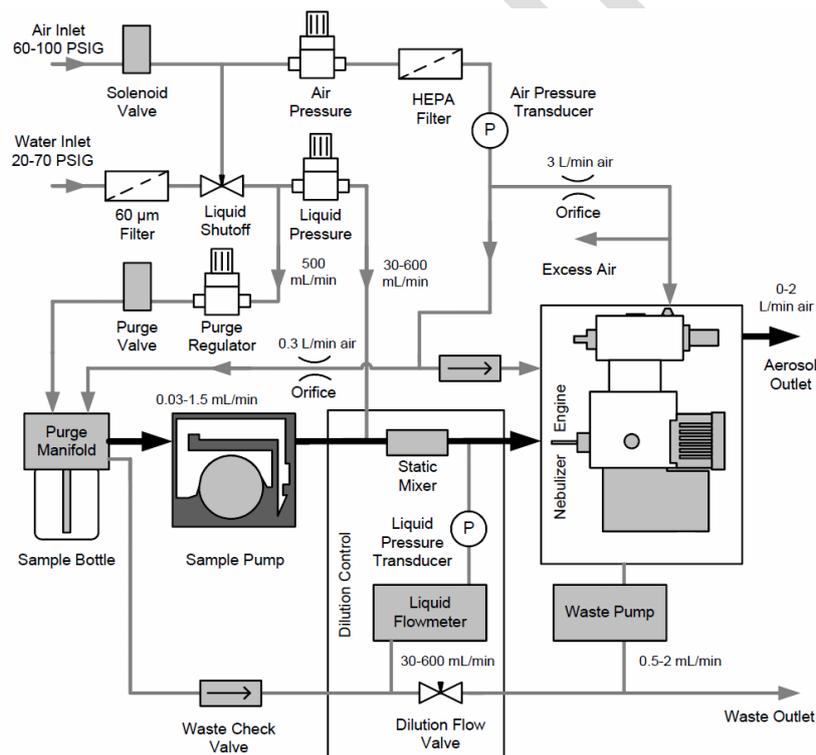


Figure 1: Flowsheet Nebulizer

### 3 Instrumentation

Instrumentation used for the particle measurements with particle number concentration and particle size distribution (suitable sampling carriers are to be used for the imaging processes (TEM, REM, EDX, etc.)):

- Nanoparticle Nebulizer Model 3485
- SMPS spectrometer Model 3936 with main components:
  - Aerosol inlet with impactor for pre-separation of larger particles
  - SMPS Classifier Model 3080 with neutralizer Model 3077 (Krypton 85) and DMA (Differential Mobility Analyzer) Model 3081 or NanoDMA Model 3085
  - CPC (Condensation Particle Counter) Model 3786 (here used, but other CPCs are also suitable)
  - Flow equalizer assembly to adjust the sample flow, depending on the CPC used
  - PC or laptop to data collection and logging
- NAS Nanometer Aerosol Sampler Model 3089
- Possibly Sensors for temperature, relative Humidity, pressure

### 4 Instrumentation preparation

#### 4.1 Nanoparticle Nebulizer

- Fill in the pressurised water container according manufacturers' instructions with distilled water. After closing, connect it with the water tube to the nebulizer.
- Connect the pressure water container to the compressed air supply and set it to at least 14.7 psi (~ 1 bar).
- Connect the nebulizer to the compressed air supply and set it to at least 60 psi (~4.1 bar).
- Connect the waste water tube to the nebulizer and connect it with a waste water container (with manually regular disposal) or the laboratory waste water system.
- Switch on the nebulizer after connection to the power supply. The warm-up period is approximately 15 minutes. After warm-up the status screen should display following temperatures:
  - Nebulizer Temp            15°C
  - Evaporator Temp        55°C
- Set the working pressures with the pressure regulators on the back panel of the nebulizer to:
  - Air Pressure            35 PSIG
  - Liquid Pressure        18 PSIG
- Setting of peristaltic pump cassette pressure
  - Unscrew the sample bottle and open the cassette lever, then press the cassette lever down until no longer any drips from the needle are seen.
  - Press the cassette lever up two more clicks.

- Before running a sample, or between running different samples, it is necessary to purge the sample needle and bottle.
  - Screw the sample bottle into the base of the purge manifold (over the needle)
  - On the display screen, press **SETUP**. Verify that the pump control is set to **BOTTLE** and that the bottle size is set appropriately (8 or 30 ml).
  - Press **HOME**, then press the purge ►► button at the top right of the screen. A purge valve opens, the sample needle is purged with 300-500 ml/min of dilution water, and the peristaltic pump tube runs forward at maximum speed. The screen displays the time remaining for the purge. Note: The purge for the 30 ml sample bottle takes 85 s and the purge for the 8 ml bottle takes 22 s.
- Following a purge, the nebulizer is in STANDBY mode. The instrument is ready to use.

## 4.2 SMPS

Connect the SMPS spectrometer according to the manufacturers' instructions with tubes and data cables. Install the impactor with the desired orifice (cut off) and grease the impactor plate slightly, and then connect the impactor with the aerosol inlet. Fill in the CPC Model 3786 according manufacturers' instructions with distilled water. For the measurement data logging, connect the CPC via Universal Serial Bus (Model 3786) and computer cable with the PC or laptop. Switch on all devices (Classifier, CPC, and PC) after connection to the power supply. The CPC Model 3786 warm-up period is approximately 15 minutes. After CPC warm-up the status screen should display following temperatures (in °C):

- Optc 75.0 (Optics)
- GT 75.0 (Growth Tube)
- SAT 12.0 (Saturator)

To check the status of these temperatures on the CPC display during the warm-up, select the **Status Screen** with the display button on the front panel. Once warm-up is complete, the CPC display shows the **Primary Concentration Screen**.

### Hardware adjustments

- On the classifier, the voltage control should be set to **Analog Ctrl** to allow the DMA voltage to be controlled by the CPC. To do so, rotate the black control knob until the **Ctrl-Pane** in the Classifier LCD Display is highlighted, and then press the knob to change from **Panel Ctrl** to **Analog Ctrl**.
- The volume flows have to be set depending on the particle size range used. According to the manufacturers' instructions, the ratio of the sheath air to aerosol flow rates is normally set to 10:1. For example, the covered particle size range is 13.5 to 750 nm mobility particle diameter for the settings of sheath flow 3.0 l/min and aerosol sample flow 0.3 l/min.

#### Sheath Flow adjustment

- Rotate the black control knob until the **Sheath Flow** pane in the Classifier LCD is highlighted.
- Click the control knob by pushing in.



- **Physical Properties**

- Gas Properties: Enter the Gas Viscosity and Mean Free Path (Default settings for Air at 293.15 K and 101.3 kPa)
- Particle Density: Enter the density, if known
- Correction Flags and Values:
  - Multiple Charge Correction: For the correction of multiple charged particles
  - Diffusion Correction: For the correction of particle losses caused by diffusion only within the SMPS. This does not account for the particle losses in sample tubes!
  - Nanoparticle Aggregate Mobility Analysis: For the correction from spheres to aggregates.

These corrections are mathematically based on the identified raw counts and can be switched on or off retrospectively, too, i. e. after sampling

By pressing the **OK** button the program retains the current settings.

## 5 Preparation for sampling

- Before sampling start, the CPC should be drained manually to reduce measurement data losses by draining the reservoir (pump will be turned off during draining). To do so, press the **Drain/Prime** button for one second to select the drain mode and press the **Drain/Prime** key again for 2 s to start draining, duration 1 min.
- Connect a sample tube from the aerosol outlet of the nebulizer to the aerosol inlet (impactor) of the SMPS.
- Screw the sampling bottle (8 or 30 ml) with the particle suspension to be measured into the base of the purge manifold (possibly pre-treatments are shaking, dispersion in an ultrasonic bath, dispersing with ultrasound finger, etc.).
- Set the sampling flow (0.03-1.5 ml/min) by pressing **SETUP** and ▼SAMPLE FLOW▲ depending on the dilution. The dilution depends of the dilution flow (30-600 ml/min) and is set later adjusted with the help of the dilution ratios. The Start Display appears by pressing **HOME**.
- To sample particles to be used for the imaging processes (REM, EDX) prepare the NAS with suitable sampling carriers (depending on the material to be examined) (see nanoGEM SOP NAS).

## 6 Measurements

- Press the ►Start button on the **HOME** screen and the sample will be injected into the instrument. The nebulized sample will begin exiting the aerosol outlet within 30 s. Press **STATUS** to show the status display.
- Set the dilution ratio by adjusting the **Dilution Flow Control** (silver dilution control knob next the Purge Manifold). Possibly a readjustment of Air Pressure and/or Liquid Pressure is required (indicated by the red VALUE error message in the STATUS display). Note values of STATUS display.

STATUS				HOME
MEASUREMENT	VALUE	SETPOINT	UNITS	
Dilution Ratio	96.9			
Dilution Flow	96.8		mL/min	
Sample Flow	0.000	0.100	mL/min	
Nebulizer Flow	1.30		mL/min	
Inspection Flow	0.46		µL/min	
Air Pressure	34.9	35.0	PSIG	
Liquid Pressure	17.7	18.0	PSIG	
Nebulizer Temp	15.2	15.0	°C	
Evaporator Temp	55.2	55.0	°C	
STATUS: OK				

- If the check box by **Properties – Scheduling – Start at Time** is marked and a clock time is entered, the sampling starts at the entered start time.
- Otherwise select **Run – Start Data Collection** to start the sampling immediately.
- At least 5 measurement scans should be measured after stabilization of the particle size distribution and particle concentration.
- Select **Run – Finish Current Sample** to stop the continuous monitoring after the current sample.
- Select **Run – Abort Current Sample** to stop the current sampling immediately and the data for the current sample is discarded.
- Note: Regularly appropriate dispose of the waste water during run of the peristaltic pump.
- After finishing the measurement series change the particle suspension dilution by turning the **Dilution Flow Control** and start a new measurement series. Otherwise stop particle generation by pressing **Stop** on the **HOME** screen.
- After stopping and finishing particle generation unscrew the sampling bottle of the Purge Manifold. Store the sampling bottle as a retained sample or dispose the sampling bottle and/or their content appropriate.
- Screw a sampling bottle with distilled water into the Purge Manifold and press the purge ►► button to purge the system (see also 4.1).
- Measure all particle suspensions according the described procedure.

## 7 Interpretation

- Simple data analysis with the **4-Pane-Layout**:
  - Select the sample in the **SMPS-Sample List** with mouse click
  - The window **Size Data Graph** displays the particle size distribution of the selected sample
    - The lower and upper size limit can be changed with the mouse pointer and pressing left mouse button (pointer becomes a hand).
    - Position the pointer on a bar (depending on type of graph that is active) and the pointer becomes a hand. By clicking the left mouse button the values diameter and concentration are displayed.
    - The displayed size distribution can be zoomed in by placing the pointer at one point of the area to be enlarged by pressing the left mouse button and dragging the mouse to the opposite point of the data. Release the left

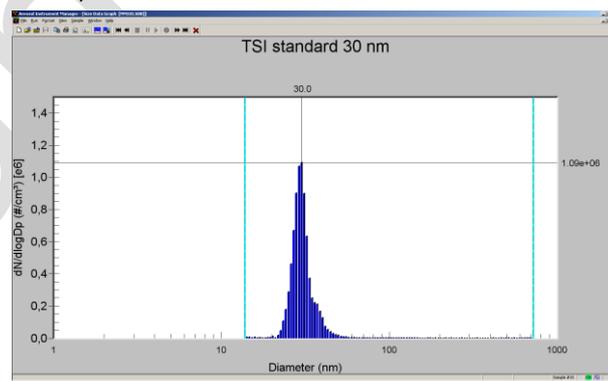
mouse button and the selected area is enlarged. To zoom out, select **Format – Undo Zoom** and the enlarged area is returned to normal view.

- The window **Size Data Table** displays the collected measurement data for each size class in table form.
- The window **Statistics Table** displays the different diameters, deviations, and the total concentrations.
- For further data interpretation:
  - Select the samples, which should be exported in the **SMPS-Sample List**. With **File – Export** the samples can be exported in a file. In the dialog box **Export Parameters**, select the data types. After selecting a filename, the data can be exported as a text file, and then imported into a spreadsheet program.

## 8 Quality assurance

Measures for quality assurance:

- Zero tests:
  - Periodical checking of adjusted volume flows (sheath and aerosol flow) with a suitable flow meter. By adjusting the voltage to zero V at the collector rod, the airstream flowing out of the monodisperse aerosol connector should be particle free and the CPC should not detect particles.
  - Periodical testing for leaks and sampling with filtered air (HEPA filter); the CPC should not detect particles.
- Routine checking of the calibration with particles of certified aerodynamic diameters (PSL)
- Periodical checking of the system nebulizer-SMPS with the TSI standard 30 nm (supplied by the manufacturer).



- Realization and reporting of the maintenance of nebulizer und SMPS according to manufacturer and manual.
- Reporting of anomalies during sampling or maintenance

**Title:** Size distribution analysis by spray characterisation

**This SOP is valid from:** 28.05.2016

**This SOP replaces the SOP from:** Primary version (1.0)

**Responsible for the implementation of this SOP:** Measurement personal

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**Signature/Date**

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