

Generating Emery Oil Aerosol for Calibration

Application Note EM-004

Counting particles in the exhaust emissions of vehicles is an important area of research within the automobile industry. Engine exhaust condensation particle counters, like the EECPC Model 3790 from TSI, are typically used adjacent to an appropriate aerosol dilution and conditioning system. This particular system is typically calibrated using Emery Oil aerosol, in order to elucidate the counting efficiency curve as well as the linearity of the response of the EECPC.

This paper describes a methodology for generating Emery Oil aerosol for calibration purposes.

Setup

TSI Electro Spray Aerosol Generator (EAG) Model 3480 is used to generate Emery Oil (EO) aerosol from a solution that contains emery oil in organic solvents with a buffer. The EAG samples from the vial containing the solution via a capillary (25 μm inner diameter). At the exit of the capillary, an electro spray process is used to generate primary droplets of the solution typically in the 100 nm size range. In the spray chamber the solute is evaporated and the remaining aerosol is neutralized and mixed with air as transport gas. The resulting aerosol diameter (D_D) depends on the droplet diameter (D_D) and the emery oil concentration (C) in the solution.

$$D_D = \frac{D_p}{\sqrt[3]{C}}$$

The EAG is coupled with a TSI Scanning Mobility Particle Sizer™ (SMPS™) spectrometer Model 3936N76, a scanning mobility particle sizer with nano-DMA and ultrafine particle counter, to verify the generated aerosol prior to the calibration process.

Operation

First, a buffer as carrier liquid to operate the electrospray is prepared by dissolving 67 mM ammonium acetate in Isopropanol and Ethanol (50/50).

Next, the droplet diameter is determined by spraying a 0.1% EO solution, using the equation above, and utilizing the mode of the distribution measured by a SMPS™ spectrometer (D_p).

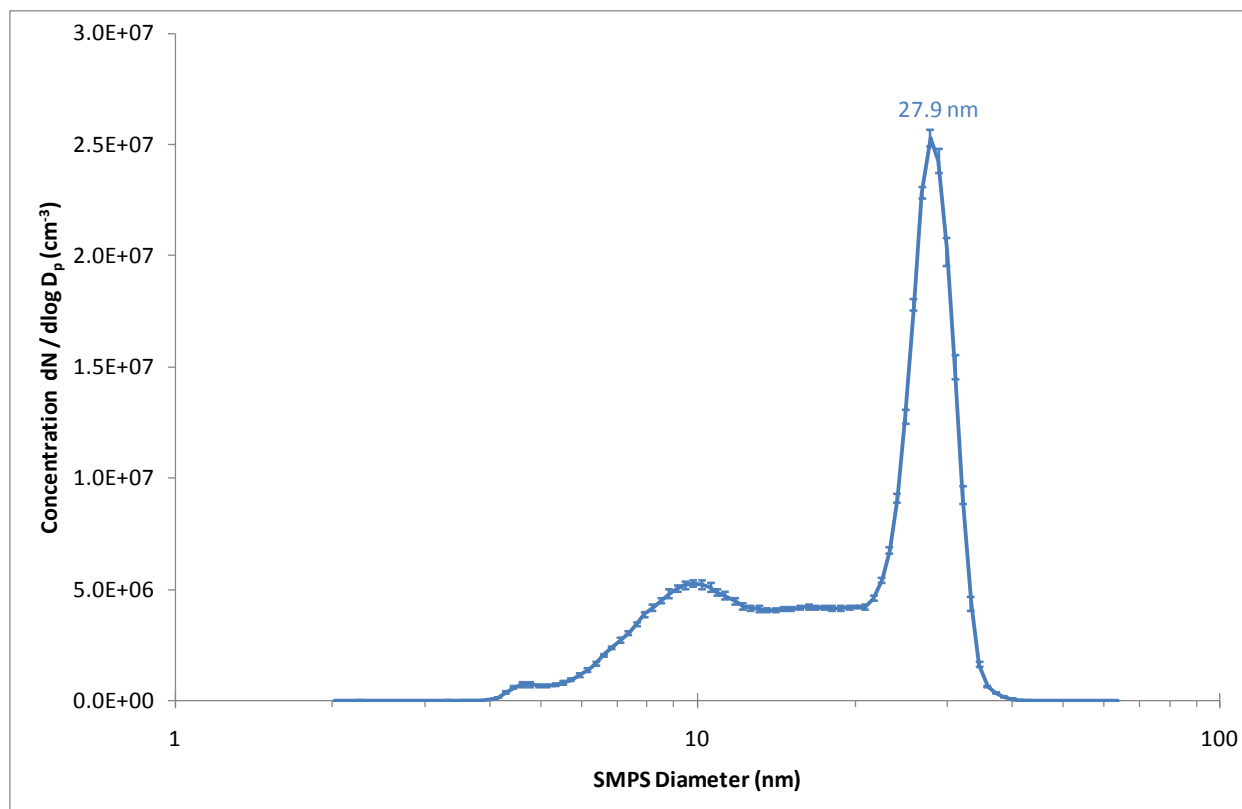
Finally, using the resulting droplet diameter, the EO concentrations needed to generate 23 nm, 41 nm, and 55 nm EO aerosol—which are used to determine the counting efficiency curve of the EECPC according to Reg83 and check linearity of the response of the EECPC respectively—can be calculated by solving the equation above for C .

Example—Three Step-Process

1. Determining the droplet diameter of the EAG.

A 0.1% emery oil solution in buffer was prepared, electro-sprayed, and the size spectrum recorded. The mode of the size spectrum was visible at 27.9 nm, resulting in a droplet diameter of 60 nm.

$$D_D = \frac{D_p}{\sqrt[3]{C(\%)}} = \frac{27.9 \text{ nm}}{\sqrt[3]{0.1}} = 60 \text{ nm}$$



2. Calculating the EO solutions to be prepared:

$$C(\%) = \left(\frac{D_p}{D_D}\right)^3$$

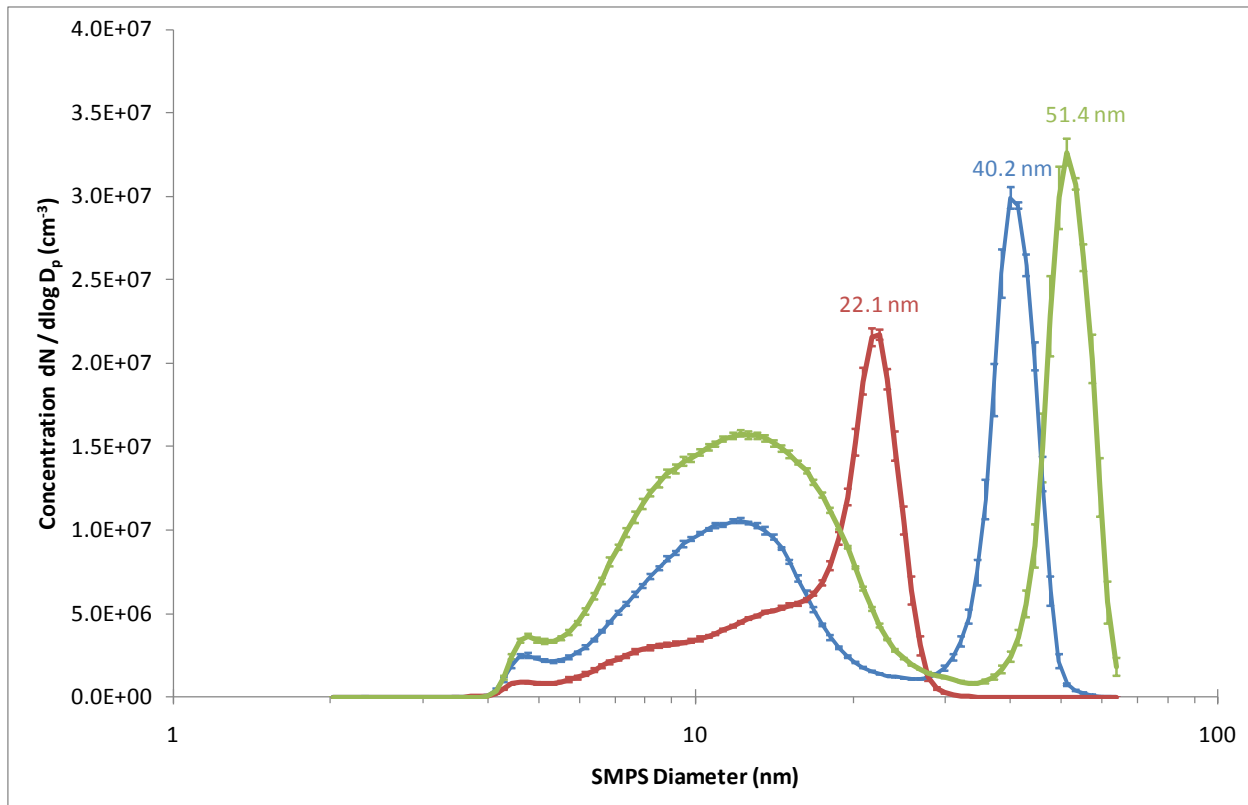
Calculating EO concentration for 23 nm: $C(\%) = 0.056\%$

Calculating EO concentration for 41 nm: $C(\%) = 0.317\%$

Calculating EO concentration for 55 nm: $C(\%) = 0.77\%$

3. Verifying the EO solutions prior to calibration process.

The prepared calibration solutions were subsequently sprayed and the resulting aerosol was analyzed for its size distribution using an SMPS™ system.



Counting efficiency determination is done at 23 nm and 41 nm for 50% and 90% efficiency. The linearity response check is performed at 55 nm. The maxima of the EO aerosol distributions should be slightly below these sizes, as the aerosol is typically electrostatically classified for these calibration processes. Classifying right of the mode of the distribution significantly reduces the influence of multiple charged particles of larger diameters, as the number concentrations decreases sharply right of the mode.

Details of Operation

1. Prepare the buffer and EO concentrates.
 - a. Mix 0.51 g of ammonium acetate in 50 mL ethanol and 50 mL isopropanol. Dissolve it by ultrasonication for 6 minutes.
 - b. Prepare the 5% EO concentrate by adding 950 μL buffer and 50 μL pure emery oil in a 1.5 mL vial. Use a vortexer to mix the solution homogeneously. Dissolve the EO by ultrasonication for 6 minutes. Label the vial "5% EO".
 - c. Prepare the 1% EO concentrate by adding 800 μL buffer and 200 μL 5% EO concentrate in a 1.5 mL vial. Use a vortexer to mix the solution homogeneously. Label the vial "1% EO".
2. Determine your actual EAG's primary droplet diameter.
 - a. Prepare the test sample of 0.1% EO by adding 980 μL buffer and 20 μL 5% EO concentrate in a 1.5 mL vial. Use a vortexer to mix the solution homogeneously. Label the vial "0.1% EO". Insert the vial into the EAG sample chamber. Start the spray process. Use a mass flowmeter to adjust the EAG output flow to 1.5 L/min.
 - b. Record some Model 3936N76 scans (sheath flow 15 L/min, aerosol flow 1.5 L/min). Average the mode of a few stable samples. The mode should be around 25-30 nm. Use the result as D_p to calculate the primary droplet diameter D_D .
 - c. Clean the capillary by flushing it with buffer for a few minutes.
3. Prepare the EO solutions for calibration.

From the primary droplet diameter, calculate the concentrations of the emery oil calibration standards for 23 nm, 41 nm and 55 nm in separate 1.5 mL vials.

$$C(\%) = \left(\frac{D_p}{D_D}\right)^3$$

The volume of emery oil needed to create a solution with concentration C:

$$x = \frac{C(\%)}{100} * 1000$$

23 nm: Prepare the 0.056% calibration solution by mixing 56 μL of the 1% EO concentrate in 944 μL buffer.

41 nm: Prepare the 0.317% calibration solution by mixing 63 μL of the 5% EO concentrate in 937 μL buffer.

55 nm: Prepare the 0.77% calibration solution by mixing 154 μL of the 5% EO concentrate in 846 μL buffer.

4. Verify EO aerosol distribution from calibration standards.
 - a. Insert the "23 nm" solution into the EAG sample chamber. Start the spray process. Use a mass flowmeter to adjust the EAG output flow to 1.5 L/min. Record a few SMPS™ system scans to verify the mode of the distribution to be ≤ 23 nm.
 - b. Insert the "41 nm" solution into the EAG sample chamber. Start the spray process. Use a mass flowmeter to adjust the EAG output flow to 1.5 L/min. Record a few SMPS™ system scans to verify the mode of the distribution to be ≤ 41 nm.
 - c. Insert the "55 nm" solution into the EAG sample chamber. Start the spray process. Use a mass flowmeter to adjust the EAG output flow to 1.5 L/min. Record a few SMPS™ system scans to verify the mode of the distribution to be ≤ 55 nm.

Operating the EAG with Emery Oil Samples

- Set the pressurized air feed line connected to the backside of the EAG to 16 psi.
- When changing samples of the EAG, first decrease voltage (<1 kV) and capillary pressure drop (<2 psi), prior to opening the sample chamber.
- Start the spray process by setting the air flow to roughly 1.5 L/min, increasing the sample chamber pressure drop to the maximum (maximum opening of the valve), and setting the high voltage to 2.0 kV. The current reading should stabilize between -50 and -70 nA.

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