

# **NanoSpot<sup>TM</sup> Collector for Aerosol Sample Collection for Direct Microscopy and Spectroscopy Analysis**

## **Introductory Information**

We describe design and characterization of an aerosol NanoSpot<sup>TM</sup> collector, designed for collection of airborne particles on a microscopy substrate for direct electron microscopy, optical microscopy, and laser spectroscopy analysis. The collector implements a water-based, laminar-flow, condensation growth technique, followed by impaction onto an optical/electron microscopy substrate or a transmission electron microscopy grid for direct analysis. The compact design employs three parallel growth tubes allowing a sampling flow rate of 1.2 L min<sup>-1</sup>. Each of the three growth tubes consists of three-temperature regions, for controlling the water vapor saturation profile and exit dew point. Following the droplet growth, the three streams merge into one flow and a converging nozzle enhances focusing of the grown droplets into a tight beam, prior to their final impaction on the warm surface of the collection substrate. A miniscule sample deposition area is attained for effective coupling with microscopic and spectroscopic analysis. Experiments were conducted for the acquisition of the size-dependent collection efficiency, the uniformity of the spot deposit, the surface density distribution of the collected particles, and the aerosol concentration effect on the triple-tube NanoSpot<sup>TM</sup> Collector. Particles as small as 7 nm could be activated and collected on the electron microscopy stub. A spot deposit of approximately 0.7-mm diameter is formed for particles over a broad particle diameter range. The collected particle samples were analyzed using electron microscopy and Raman spectroscopy for the acquisition of the particle spatial distribution, the spot sample uniformity, and the analyte concentration. Finally, the NanoSpot<sup>TM</sup> collector's analytical measurement sensitivity for laser Raman analysis and the counting statistics for fiber count measurement using optical microscopy were calculated and were compared with those of the conventional aerosol sampling methods.

## **Experimental Methods**

Experiments were designed and conducted for the evaluation of the NSC based on the following key variables:

- (i) The particle diameter dependent collection efficiency,
- (ii) The number concentration dependent collection efficiency,
- (iii) The deposition spot diameter on the collection substrate,
- (iv) The spot sample deposit uniformity, and
- (v) The Raman Intensity calibration curves acquired when the NSC is employed.

The analytical measurement sensitivity and the counting statistics for fiber count measurement using optical microscopy were acquired for the NSC and compared with the calculated analytical measurement sensitivity and counting statistics promoted by the Sequential Spot Sampler, the aerosol focusing and collection through the implementation of aerodynamic lenses and the conventional technique of air filtration.

#### Particle generation:

Particles were generated by either a *nebulizer* or a *Fluidized Bed Generator*.

- ❖ *Nebulizer* (Salter 8900 Series Disposable Small Volume Jet Nebulizer; Salter Labs, Arvin, CA, USA):
  - Liquid solutions of sodium chloride (soluble) and respirable crystalline silica (insoluble) were used.
  - A diffusion dryer (model 3062, TSI Inc., Shoreview, MN, USA) was used downstream of the nebulizer for removal of water vapor.
- ❖ *Fluidized Bed Aerosol Generator* (model 3400A, TSI Inc., Shoreview, MN, USA):
  - Respirable crystalline silica was generated from dry powder. Filtered, lab air was implemented for the generation of the solid silica particles. The aerosol exiting the fluidized bed had a relative humidity of 60%.

#### Particle classification:

- The Aerodynamic Aerosol Classifier (AAC; Cambustion Ltd, Cambridge, United Kingdom) was used to obtain classified near-monodisperse (in aerodynamic size) test aerosol above 25 nm diameter.
- An Electrostatic Classifier (model 3080; TSI Inc., Shoreview, MN, USA) and a Nano-Differential Mobility Analyzer (model 3085; TSI Inc., Shoreview, MN, USA) were used to obtain smaller particle sizes.
- Only the AAC (Aerodynamic Aerosol Classifier; Cambustion Ltd, Cambridge, United Kingdom) was used for generation of near-monodisperse particle sizes for crystalline silica particles generated by the Fluidized Bed.
- Aerosols generated by the nebulizer, were classified through the Electrostatic Classifier and the nano-DMA.

*Particle diameter and number concentration-dependent collection efficiency measurement:*

- A total flowrate of 1.2 L min<sup>-1</sup> was introduced into the NSC.
- The outlet of the collector was connected to an Ultrafine Condensation Particle Counter (model UCPC 3776, TSI Inc., Shoreview, MN, USA) to measure the number of particles that were transmitted throughout the collector.
- The UCPC required a total flowrate of 1.5 L min<sup>-1</sup>; thus, filtered, dilution air was added as make-up air to the aerosol stream exiting the collector.
- The same UCPC with the make-up air was also connected at the inlet of the NSC for measuring the total aerosol concentration upstream of the collector.
- The effect of particle diameter on the NSC performance was compared at two initiator temperatures, 40 or 45 °C.
- Three temperature combinations for the initiator and the moderator were tested for the number concentration effect: 40 and 12 °C, 45 and 12 °C, and 45 and 25 °C.

Spot sample characteristics:

- Polystyrene latex (PSL) nano (20 nm, 100 nm, 700 nm) and micro-spheres (1900nm; Thermo Fisher Scientific, Waltham MA, USA) were used.

- A Scanning Electron Microscopy (SEM) stub was positioned in the sample heater chamber of the collector, and an aluminum backed carbon tape (Ted Pella Inc., Redding CA, US) was placed on the surface of the stub, for the acquisition of superior SEM (Phenom XL Desktop SEM, Thermo Fisher Scientific, Waltham MA, USA) images.
- The ImageJ software was implemented for measuring the projected area of the accumulated particles depicted on the SEM images.
- The calculated spot deposit diameter ( $D_{90}$ ) corresponds to the diameter of the circle that contains 90% of the total projected area of the collected particles in the spot sample.

#### Particle Collection for Raman analysis:

- Dry crystalline silica powder (Min-U-Sil@5; US Silica, Katy TX, USA) was aerosolized through the fluidized bed generator.
- The SEM stub's surface was cleaned with isopropyl alcohol before mounting into the sample chamber of the collector.
- During the particle collection, a piezo balance dust monitor (model 3520; Kanomax, Andover NJ, USA) was used to simultaneously quantify the aerosol mass concentration entering the NSC.
- For the Raman signal intensity acquisition, the portable i-Raman Spectrometer (i-Raman<sup>®</sup>; B&W Tek, Newark, DE, USA) was implemented. The maximum laser power and the laser spot diameter incident on the sample was 420 mW, and about 105 micrometers, respectively. The excitation wavelength was at 785 nm. An integration time of 30 s was used for this study.
- The characteristic peak for crystalline silica was observed at a Raman shift of  $465\text{ cm}^{-1}$  for  $\alpha$ -quartz. The Raman calibration curves were constructed by using the area below the characteristic peaks and the particulate mass collected on the SEM stub.

#### Analytical measurement Sensitivity:

- The analytical measurement sensitivity of the NSC was calculated and then compared with that of prevalent particle collection methods, such as the Sequential Spot Sampler, Aerodynamic lenses, and filter-based collection method

#### Poisson Counting Statistics of Fiber Concentration Measurement

- Calculations were performed to compare Poisson counting statistics of fiber concentration measurement using the phase contrast microscopy (PCM; NIOSH Method 7400) for various collection methods, such as NSC, Sequential Spot Sampler and filter-based collection.

#### **Citations- Publications based on the dataset**

Zervaki, O., B. Stump, P. Keady, D. D. Dionysiou, and P. Kulkarni. n.d. “NanoSpot™ Collector for Aerosol Sample Collection for Direct Microscopy and Spectroscopy Analysis.” *Aerosol Science & Technology*, 57:4, 342-354, DOI: 10.1080/02786826.2023.216764.

#### **Acknowledgements**

Orthodoxia Zervaki, [nvr7@cdc.gov](mailto:nvr7@cdc.gov)

Braden Stump, [bstump@aerosoldevices.com](mailto:bstump@aerosoldevices.com)

Patricia Keady, [pkeady@aerosoldevices.com](mailto:pkeady@aerosoldevices.com)

Dionysios D. Dionysiou, [DIONYSDD@UCMAIL.UC.EDU](mailto:DIONYSDD@UCMAIL.UC.EDU)

Pramod Kulkarni, [ewg7@cdc.gov](mailto:ewg7@cdc.gov)

#### **Contact**

For further information contact:

Health Effects Laboratory Division (HELD), National Institute for Occupational Safety and Health  
(NIOSH), Cincinnati, OH

513-533-6800