# Assessment of Microscopy Methods for Distinguishing Engineered Nanoparticles from Incidental Nanoparticles

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## Background

Exposure studies for engineered nanoparticles frequently utilize electron microscopy in order to obtain detailed particle information. Often occupational environments contain non-engineered or "incidental" nanoparticles that need to be considered separately.

## Objective

To assess the utility of electron microscopy for distinguishing engineered nanoparticles from incidental nanoparticles.

## Methods

Transmission electron microscopy (TEM) with energy dispersive spectroscopy (EDS) was used to analyze samples of known mixture of titanium dioxide ($TiO_2$, 15nm primary particle size) and Arizona road dust (ARD) or incense particles.

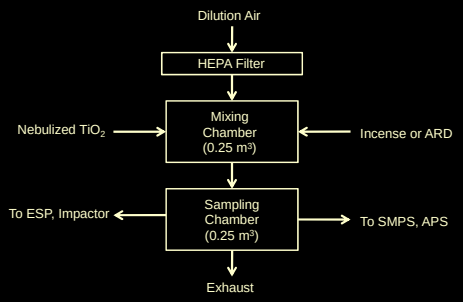
TEM collection methods included two different samplers: a Marple impactor and an electro-static precipitator (ESP). TEM grids were placed on the four lowest stages of the impactor. Concentration and size distribution data were collected using an SMPS and APS. Each mixture was analyzed twice producing a total of 10 grids per mixture.

Criteria for sampling time relative to ambient concentration were determined by the relationship below, which was developed using parameters that produced the cleanest grid images with evenly scattered particles.

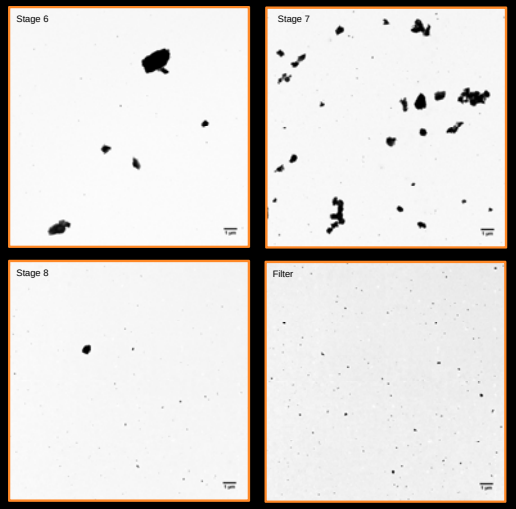
Particles were counted and analyzed with EDS to determine the proportion of $TiO_2$ particles on each stage. Aerosol mixture ratios were compared to particle ratios on grids from each stage.

$$\text{Sampling Time} = \frac{5 \times 10^{10}}{\text{Impactor Flow} \times \text{Total Concentration}}$$

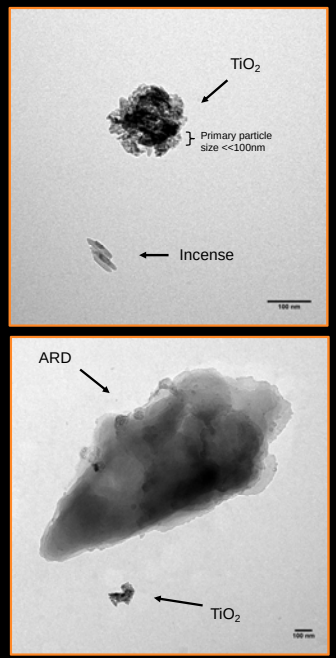
## Experimental Setup



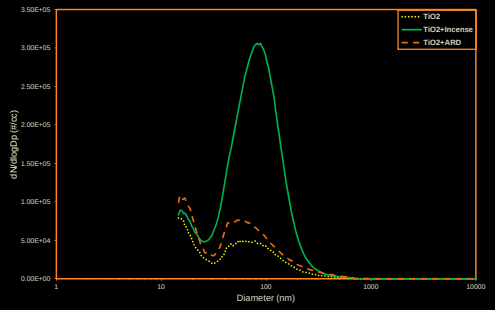

## Results



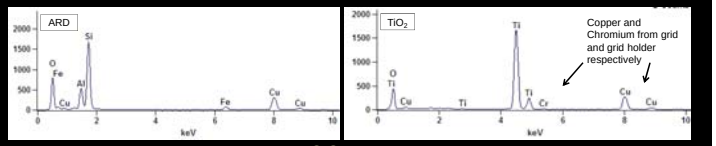

TEM images from each impactor stage




High magnification TEM images of particles on filter stage grids




Size Distribution Data for Mixtures and neat $TiO_2$




EDS Spectra

Impactor Distribution Data

| Impactor Stage | Aero. Cut Diam. (µm) | Expected $TiO_2$ Count % | Experimental $TiO_2$ Count % |
|---|---|---|---|
| 6 | 0.9 | 2.2 | 44.3 |
| 7 | 0.5 | 15.7 | 43.5 |
| 8 | 0.3 | 73.2 | 83.3 |
| Filter | < 0.3 | 90.9 | 90.0 |

## Discussion and Conclusions

$TiO_2$ was distinguishable from all other particles, but accurate particle proportions were not found on grids for either mixture. $TiO_2$'s density (4.3 g/cm$^3$) is greater than ARD's (2.7 g/cm$^3$) and 4 x incense's (1.1 g/cm$^3$). Collection favored $TiO_2$. Nanoparticles of $TiO_2$ were found on all stages and included in the counts.

Incense particles (carbon) were not identifiable using TEM/EDS. They were also not collected onto grids as readily as ARD and $TiO_2$ due to their much lower density.

The $TiO_2$/ARD mixture demonstrated that the impactor would remove most of the larger ARD particles so that grids on the filter stage could be used to analyze and image $TiO_2$ mostly alone.

Grids from the ESP generally did not produce images as clean as those from the impactor due to particle overloading and grid damage.

Provisions developed to save time involved with grid analysis (secure particle collection and distribution of particles on grid based on sampling time) were beneficial.

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