



Evaluation of data and recommendation on measurement technique and sampling protocol for determining concentrations of engineered and incidental nanoparticles



Date: 22 March 2012, ICHO2012

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Aim

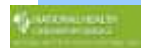
- To generate and evaluate relevant data to establish appropriate assessment and control methods and identify and prioritize exposure levels;
- To suggest control measures in line with observed exposures scenarios;
- To develop a simple risk assessment tool/checklist to assist managers in the process of health hazard identification and risk prioritization

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Establish appropriate assessment and control methods

- **Introduction**
 - Describes
 - Participants (& invitation to interested parties)
 - Material Selected For Studies
 - Approaches & measurement Methods for exposure assessment in work environment.
 - Planned research.



Participants

- **Main project participants**
 - Mintek, a South African State Owned Enterprise (SoE) synthesizes gold nano-particles
 - Eskom, a South African SoE mainly operate coal-powered power stations
 - CSIR, a South African SoE synthesizes various nano-particles
 - NIOH, a South African SoE conducting exposure assessment.
- Interested contributors are welcome to contact
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Material Selection

- To start with, the synthesis process of gold nano-particles is studied first



Material Selection: Au NPs

| Test Material | Mean Core Dia. | Capping Agent | By whom | Catalytic activity | Production method |
|---------------|----------------|---------------|---------|--------------------|--------------------|
| # 1 | 14 nm | Citrate | Mintek | CO oxidation | Chemical reduction |
| # 2 | 40 nm | Citrate | Mintek | | |



Au NPs: Major Commercial Uses

- Currently used for Lateral flow diagnostics and Catalysis; Expected to be also applied in Therapeutics (e.g., cancer) and Imaging



AU NPs' Physicochemical Properties Surface Chemistry

- Multilayer of citrate ions.

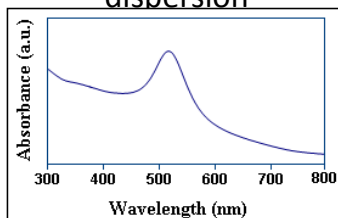


Physicochemical properties agglomeration/aggregation/dispersion

- UV/Vis
- TEM



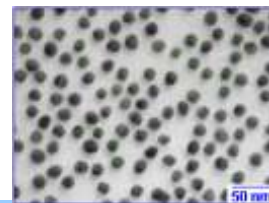
agglomeration/aggregation/dispersion



UV/Vis. spectrum of the 14 nm gold nanoparticles. The typical absorption peak of the 14 nm should be between 520- 524 nm.



14 nm Au TEM image



- The TEM image of the representative 14nm gold nanoparticles is given



AU NPs' behaviour in solutions

- The sharp UV/Vis. absorption spectrum and TEM image clearly indicate that the 14 nm gold nanoparticles do not aggregate in solution

Physicochemical Properties Surface Charge

- LM 20 Zetasiser used.
- Measuring -34.8mV.
- This value is sufficient to keep the particles from interacting with each other and therefore maintain a stable particle size

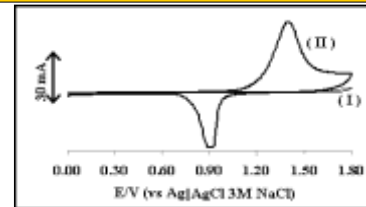
Physicochemical Properties Surface Chemistry



Figure 3. Agarose gel electrophoresis demonstration of different size gold nanoparticles.

- Gel Electrophoresis: separates Au nanoparticles based on size, shape and charge where particles migrate differently depending on molecular weight and charge.

Physicochemical Properties Redox Potential



- Cyclic voltammetry was used.
- CV generally responds to a variety of factors such as pH, solvent, ligand and even if the nanoparticle is bare – it still resembles the bulk gold patterns

Physicochemical Properties Octanol/Water Partition Coefficient

- An average of $\text{Log } P_{ow} = -2.0$ at about 25°C using the UV-vis Spectroscopy

Occupational Human Exposure

- Source Emission/Ambient
 - Inhalation.
 - At point of synthesis.
 - Area away from point of synthesis.

Exposure Characterisation in Research Lab

- Walk-Through Survey
 - For possible emission ID
 - Determine frequency & duration of each operation
 - Determine ventilation: General & local exhaust
 - Determine containment breaches in process points

Sampling Strategy

- Background readings.
- Source specific area sampling
- Source specific personal sampling



P-TRAK Ultrafine Particle Counter 8525



Met One HHPC - 6 Airborne Particle Counter



| | Background Particle Counts - Met One HHPC - 6 Airborne Particle Counter | Background Particle Counts - TSK P TRAK, 8525 |
|--|---|---|
| Synthesis Areas | | |
| Location A: Fume Hood (1) | 44017 | 14649 |
| Location B: Bench (2) | 38329 | 12588 |
| Location C: Fume Hood (3) | 36224 | 11261 |
| Location D: Bench (4) | 31702 | 11349 |
| Non-synthesis Areas | | |
| Location E: Fume Hood (5) | 40212 | 11478 |
| Location F: Bench (6) | 34321 | 10435 |
| Location G: Bench (7) | 27163 | 9066 |
| Location H: Freezer (8) | 31323 | 9427 |
| Location I: Between Benches (9) | 23445 | 8031 |
| Location J: Between Bench and Far wall (10) | 26842 | 7813 |
| Location K: In corner of Lab next to "Binder" Machine (11) | 23303 | 7414 |
| Location L: Next to Balances (12) | 27061 | 7534 |
| Location M: Electrophoresis Area (13) | 27037 | 6644 |
| Location N: Unused Fumehood (14) | 27399 | 7431 |
| Applications Areas | | |
| Location O: Microwave (15) | 31503 | 8247 |
| Location P: Bio Imaging System Area (16) | 29929 | 8342 |
| Location Q: De-Ioniser Bench (17) | 27595 | 7664 |
| Location R: Entrance of Lab (18) | 29998 | 5996 |
| Location S: Electro Chemistry Room (19) | 2575 | 537 |
| Location T: Diagnostics Room (20) | 2897 | 528 |
| Location U: Tissue Culture Room (21) | 3801 | 709 |
| The Inter-leading corridor | | |
| Location V: Corridor Outside Lab (22) | 32290 | 8209 |

- These two instruments give the counts of the particles without their characterization.

Collection of particles for characterization

Nano ID

- Selects a wide-range sampler: 2 nm up to 20 μm .
- Particles are collected simultaneously and separated into 12 size channels.
- It can give:
 - The size-resolved structure,
 - Mass resolved size distributions,
 - Morphology,
 - Chemical composition of captured particles.
 - Determines the concentration of the Engineered Nanoparticles against a background of naturally occurring aerosols and other anthropogenic aerosols produced from processes such as combustion.
- o For example, engineered nano-silver has been detected at levels of 2 ng/m³ against a background aerosol concentration of > 40 $\mu\text{g}/\text{m}^3$



Physicochemical properties

- Aerodynamic diameter
- Particle number
- Size
- Surface area

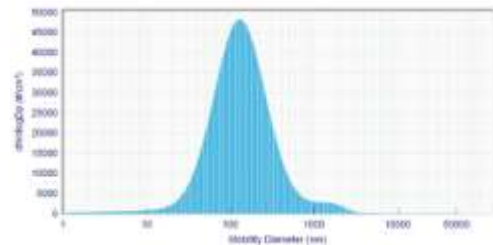


Instrument used

- TSI Scanning Mobility Particle Sizer (SMPS), MODEL 3080, for particle number size distribution:
 - Differential Mobility Analyzer (DMA) coupled in series with a CDC
 - DMA, MODEL 3081= selects particle size interval of sampled aerosol
 - Condensation Particle Counter (CPC), MODEL 3772 = counts particles exiting the classifier
 - * Particles neutralised @ DMA inlet with radioactive sources (Kr85, Am241) to reach charge equilibrium
 - * Electric Field Scanning to different particle sizes selected for particle size distribution
- APS 3321 (Aerodynamic Particle Sizer)



The histogram shows a combined data from a TSI APS (Model 3321) and TSI SMPS (consisting of an Electrostatic Classifier, Model 3080 and Condensation Particle Counter, Model 3372)



Physicochemical properties (cont.)

- Chemical analysis
 - ICP-MS
- Size and agglomeration stage
 - TEM
- Surface properties/Ligands
 - FT-IR
 - NMR
- Surface activity
 - ESR



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- Thank you for your attention