Silver Nanoparticle Synthesis in the Presence of Polyvinylpyrrolidone via Sodium Borohydride

Author(s): Jericho Mordasiewicz, Belle Shin, Alan Fang

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1.	Purpose of Standard Operating Procedure
Synthesize silver nanospheres of radius 15-30 nm	
2.	**Equipment and Chemical List**

Equipment List:	Chemical List:
- Fumehood - Centrifuge - Digital scale - Hotplate with magnetic stirring - Ice bath - Trifold clamp and stand - 15 mL centrifuge tubes - 150 mL beaker - 500 mL Erlenmeyer flask - 250 mL Erlenmeyer flask - Glass funnel - Quantitative filter paper, 8 µm - 1000µL pipette & pipette tips	- Silver Nitrate, $AgNO_3$ - Sodium Borohydride, $NaBH_4$ - Polyvinyl pyrrolidone, PVP - Deionized water, DI water

3.	**Hazard-Risk-Control Table**

Hazard	**Risk**	**Control**
Equipment/Chemical(s)	Potential risk description	Control measure(s) to be taken
$NaBH_4$	Strong reducing agent, reactions with the DI water and especially the $AgNO_3$ can be violent. Skin corrosion (Category 1B), Serious eye damage (Category 1)	- Use much larger beakers than the volume of solution & prepare the violent reactions in an ice bath - Work under hood. Do not inhale. Keep workplace dry - ALWAYS WEAR PPE
PVP	Avoid inhalation/ingestion and dermal contact of the compound	- Use nitrile gloves when

		handling material
$AgNO_3$	Classified as 1B oxidizer, can intensify fires, cause severe skin burns and severe eye irritation	- Handle $AgNO_3$ under a fume hood to avoid inhalation - Keep $AgNO_3$ from heat, hot surfaces, sparks, open flames and other ignition sources
Centrifuge	Damaging equipment	- Ensure the weight is balanced along each axis of the motor

4. Step-by-Step Methodology

1) Prepare necessary materials and ensure all equipment is in working condition

a) Set up ice bath for safer synthesis conditions
b) Prepare 8 mL of 10 mg/mL $NaBH_4$ (In the fume hood as this reaction produces hydrogen gas)
   i) Measure 80 mg of $NaBH_4$ and place it into a 150 mL beaker and add 7.92 mL of water at once
c) Set up simple filtration mechanism
   i) Set the glass funnel into the 250 mL Erlenmeyer flask and fold the filter paper along the inside of the funnel. Secure this with a trifold clamp or a small O-ring stand.

2) Carry out the procedure, ensuring proper documentation where necessary

a) Place the ice bath on the hotplate and the 500 mL Erlenmeyer flask in the ice bath
b) Gently set a large stir rod/bar in the 500 mL Erlenmeyer flask
c) Measure and place 500 mg each of $AgNO_3$ and PVP into a 500 mL Erlenmeyer flask
d) Dissolve the mixture of $AgNO_3$ and PVP in 100 mL of DI water, stirring at 300 RPM
e) Add the 8 mL of 10 mg/mL $NaBH_4$ to the 100 mL of DI water (The solution should immediately change from colourless to dark yellow)
f) Let the reaction proceed for 5 min then turn off the hotplate
g) Pipette 1 mL at a time into filtration station, being careful not to overfill the paper
h) Pipette into centrifuge tubes ensuring a balanced weight
i) Centrifuge at 5000 RPM for 1 h

3) Conduct any required measurements or observations
   a) After centrifuging the silver particles should precipitate and any excess PVP will be suspended in solution.

4) Record observations and results for further analysis

5.	**Cleanup and Waste Disposal**

Rinse glassware with DI water and pour into the waste container. Glassware can then be cleaned with Sparklean or an equivalent glassware detergent and regular water. Set on the drying rack once completed. The estimated volume of waste per synthesis is ~100 mL.
~98% DI water, 1% PVP, trace: BH_4^-,NO_3^-,Na^+, AgNPs <15 nm

6.	**Chemical Transport and Storage**

Chemicals will be transported by hand and segregated after use as per SO2070.

7.	**Training Requirements**

1) WHMIS, SO1017
2) Laboratory Safety, SO1010
3) Safe Chemical Handling, SO2032
4) Workplace Violence and Harassment, SO1081
5) Worker Health & Safety Awareness in 4 Steps, SO3000
6) Chemical Waste Segregation, SO2070

8.	**Attachments**

Any images or reference flow charts. *** Complete after first replication

9.	**References**

Papers

1) Preparation of Conductive Silver Films at Mild Temperatures for Printable Organic Electronics Lakshminarayana Polavarapu, Kiran Kumar Manga, Hanh Duyen Cao, Kian Ping Loh, and Qing-Hua Xu *Chemistry of Materials* 2011 *23* (14), 3273-3276
DOI: 10.1021/cm200471s

2) Fernandes, I.J., Aroche, A.F., Schuck, A. *et al.* Silver nanoparticle conductive inks: synthesis, characterization, and fabrication of inkjet-printed flexible electrodes. *Sci Rep* 10, 8878 (2020). https://doi.org/10.1038/s41598-020-65698-3

MSDSs

1) Silver Nitrate, $AgNO_3$
https://www.sigmaaldrich.com/CA/en/sds/aldrich/204390?userType=undefined
2) Sodium Borohydride, $NaBH_4$
https://www.sigmaaldrich.com/CA/en/sds/aldrich/452882?userType=undefined
3) Polyvinyl pyrrolidone, PVP
https://www.sigmaaldrich.com/CA/en/sds/sial/pvp40?userType=undefined

Sodium Borohydride ($NaBH_4$) Standard Operating Procedure

Hazards

- Water reactivity: Reacts violently with water, releasing flammable hydrogen gas (H_2) that may ignite spontaneously
- Toxicity: Toxic if swallowed, causes severe skin/eye burns, and respiratory irritation
- Exothermic reactions: Rapid decomposition occurs at elevated temperatures (>40°C)

PPE Requirements

- Always wear: Nitrile gloves, lab coat, safety goggles, and closed-toe shoes.

Handling Procedures

1. Engineering controls:
    a. Use a fume hood for all procedures involving $NaBH_4$
    b. Maintain lab relative humidity <30%
2. Synthesis precautions:
    a. Cool reactions in an ice bath (0–4°C) to control exothermic heat
    b. Add $NaBH_4$ solutions dropwise to avoid pressure buildup
3. Storage:
    a. Keep in airtight containers within a desiccator or dry box
    b. Label containers with “Water Reactive” warnings

Waste Disposal

- Liquid waste: Neutralize with isopropanol or water *slowly* in a fume hood before transferring to labeled waste containers.
- Solid waste: Collect contaminated materials (gloves, filter paper) in double bagged nanoparticle waste

Emergency Response

Skin contact	Remove contaminated clothing → rinse with water for 15 min → seek medical attention
Eye exposure	Flush eyes at eyewash station for 15 min → transport to hospital
Inhalation	Move to fresh air → if breathing stops, administer artificial respiration → call emergency services
Spills	Evacuate area → don PPE → contain spill with damp cloths → neutralize with isopropanol → dispose as hazardous waste

Silver Nitrate ($AgNO_3$) Standard Operating Procedure

Hazards

- Oxidizing agent: Reacts violently with flammable materials, ammonia, and reducing agents, potentially causing fires or explosions.
- Corrosivity: Causes severe skin burns, eye damage, and respiratory irritation.
- Photosensitivity: Decomposes under light to form toxic nitrogen oxides (NO_x) and elemental silver.
- Environmental toxicity: Highly toxic to aquatic life.

PPE Requirements

- Always wear: Nitrile gloves, lab coat, safety goggles, and closed-toe shoes.

Handling Procedures

1. Engineering controls:
    a. Use a fume hood for all procedures involving $AgNO_3$.
    b. Shield containers from light using amber glass or foil wrapping.
2. Synthesis precautions:
    a. Avoid contact with ammonia, halides, metals (e.g., copper), and organic compounds.
    b. Use non-metallic tools (e.g., plastic spatulas) to prevent displacement reactions.
3. Storage:
    a. Keep in airtight, light-resistant containers.
    b. Store separately from acids, bases, and flammable materials.

Waste Disposal

- Liquid waste: Collect in labeled containers for heavy metal waste treatment.
- Solid waste: Seal contaminated materials (gloves, filter paper) in double bagged nanoparticle waste.

Emergency Response

Skin contact	Remove contaminated clothing → rinse with water for 15 min → seek medical attention.
Eye exposure	Flush eyes at eyewash station for 15 min → transport to hospital.
Spills	Evacuate area → don PPE → contain spill with inert absorbents (e.g., vermiculite) → dispose as hazardous waste.
Fire	Use dry chemical extinguishers (never water).