# Synthesis of silver nanoparticles in a continuous flow microreactor

## Background

Silver nanoparticles are used in a variety of applications, e.g. as antibacterial and antifungal agents in medical applications. The synthesis of these nano-sized metal particles through 'wet chemistry' serves as the most practical laboratory technique, as silver salts are easily reduced using common reducing agents in solution. While still being made in batch vessels most of the time, continuous flow can offer significant advantages since precise control of reaction parameters is often necessary to obtain high-quality nanoparticles with good size uniformity.

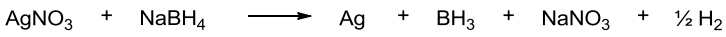


*Figure 1: Synthesis of silver nanoparticles scheme*

Using continuous flow chemistry, precise control over the size (diameter) of the formed nanoparticles is possible by varying the residence time, temperature or molar ratio of reagents. In continuous flow, a silver nitrate solution and a sodium borohydride solution are introduced into the microreactor, where they react to form the corresponding nanoparticles.

## Setup and method

### Material

- *Flow*Start B-200
- B-230 Pump Module
- B-242 Inlet Module
- Basic Quench Microreactor (internal volume $V_{\mu R}$ = 92 µL)

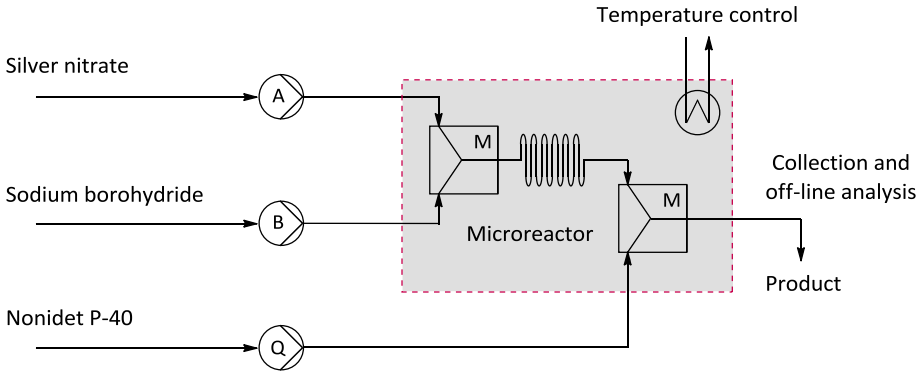


*Figure 2: FlowStart setup for the synthesis of silver nanoparticles*

### Chemicals

Recommended grade: *pro analysi* (p.a.) or *reagent grade*.

- Sodium borohydride
- Silver nitrate
- Sodium hydroxide
- Nonidet P-40

### Stock solutions

- **A.** Silver nitrate (5.7 mg, 310 µmol) dissolved to a total volume of 1.0 L with water (corresponding to 310 µM)
- **B.** Sodium borohydride (38 mg, 1.0 mmol) dissolved to a total volume of 100 mL with 10 mM NaOH (corresponding to 10 mM)
- **Q.** Nonidet P-40 (1% v/v in water)

Stock solutions are to be prepared at the beginning of the experiments, or can be prepared beforehand by the instructor (large volumes are stable for a long time when stored in the fridge).

### Analysis

Analysis of the reaction mixture is done using UV-vis. Analyse your samples and calculate the particles' diameter by using the response curve which can be found in the analysis section.

## Basic experiment

To get acquainted with the reaction and with flow chemistry in general, a so-called *basic experiment* is performed. This experiment is the synthesis of silver nanoparticles at fixed parameters – a reaction time ($t_R$) of 14 s, a temperature of 20°C and an sodium borohydride molar excess ratio ($ME_{B/A}$) of 30. The quenching flow is kept at roughly 5% of the sum of flow A and B (that is: $\phi_Q = 0.05 * (\phi_A + \phi_B)$). Roughly 100 µL of the microreactor outflow is collected and diluted to 1.0 mL (10 x dilution), which can be measured directly on a UV-vis spectrophotometer. The used setup can be seen in Figure 2.

The corresponding flow rates can be calculated according to the known equations. After preparation of this experiment, the instructor should check if the calculated flow rates and collection time are correct.

### Procedure

- Prepare solutions A, B and Q (or use the solutions provided by the instructor)
- Fill the three syringes with solutions A, B and Q
- Slide the microreactor into the holder and connect inlet and outlet tubing
- Connect the inlet tubing to the corresponding syringes, and place the syringes on the pumps
- Set the right flow rates and press start
- Stabilise for 1 minute
- Collect your sample for the calculated time
- Analyse your sample using UV-vis and calculate particle diameter from the calibration curve or relative response factor
- Rinse the *Flow*Start system by purging the tubing and microreactor with a soap solution (e.g. 5% hand soap in water) and then water
- Empty, clean and dry the syringes afterwards

## Questions

1. **Preparation of the experiment:**
    a. **Roughly calculate the cost of the experiment from the prices of the chemicals. In other words, calculate the price (e.g. per gram) of the product. For these calculations, assume that chemical yield of the silver nanoparticles with respect to silver nitrate is 100%.**
    b. **Find the safety aspects (including R/S values) of the used chemicals.**

2. **What advantages in performing the synthesis of silver nanoparticles in continuous flow can you think of? Also, can you think of any disadvantages?**