Laboratory

Nanofabrication, characterization and modeling of colloidal Au NPs

Aim:

Synthesis and characterization of colloidal spherical Au NPs.

Procedure:

1. **Synthesis** by the Turkevich method in aqueous solution
2. **Optical absorbance** in the VIS-NIR range
3. **Simulation** or fit with the Mie theory in the dipolar approx. (size-corrected) → *size, concentration, refractive index*
4. Grazing Incidence X-Ray Diffraction (**GI-XRD**) → *size*
5. Morphological/compositional analysis with scanning electron microscopy (**SEM**)

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Synthesis

Introduction to NanoPhysics

Turkevich method

- Au NPs with D about 10-20 nm
- Supersaturated solution from Au precursors
- T constant

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Turkevich Method for Gold Nanoparticle Synthesis Revisited

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The growth of gold nanoparticles by reduction by citrate and ascorbic acid has been examined in detail to explore the parameter space of reaction conditions. It is found that gold particles can be produced in a wide range of sizes, from 9 to 120 nm, with defined size distribution, following the earlier work of Turkevich and Frens. The reaction is initiated thermally or in comparison by UV irradiation, which results in similar final products. The kinetics of the extinction spectra show the multiple steps of primary and secondary clustering leading to polycrystallites.

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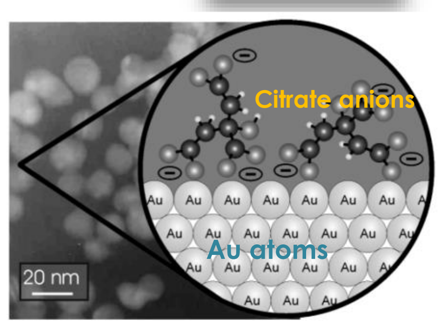
Synthesis

Introduction to NanoPhysics

Protocol:

1. Pour in the beaker 9.5 mL of the **$HAuCl_4$** solution (use the pipette)
2. Cover the beaker with the watch glass
3. Suspend the beaker in the crystallizer filled with normal water on the hot plate and rise the **temperature to 100 °C**
4. Activate the **stirrer**
5. Heat the **$Na_3C_6H_5O_7$** solution up to **100 °C**
6. When both solutions are at 100 °C, quickly add with the micropipette 0.5 mL of the $Na_3C_6H_5O_7$ solution to the beaker
7. Wait 15 minutes with the stirrer on and at 100 °C.



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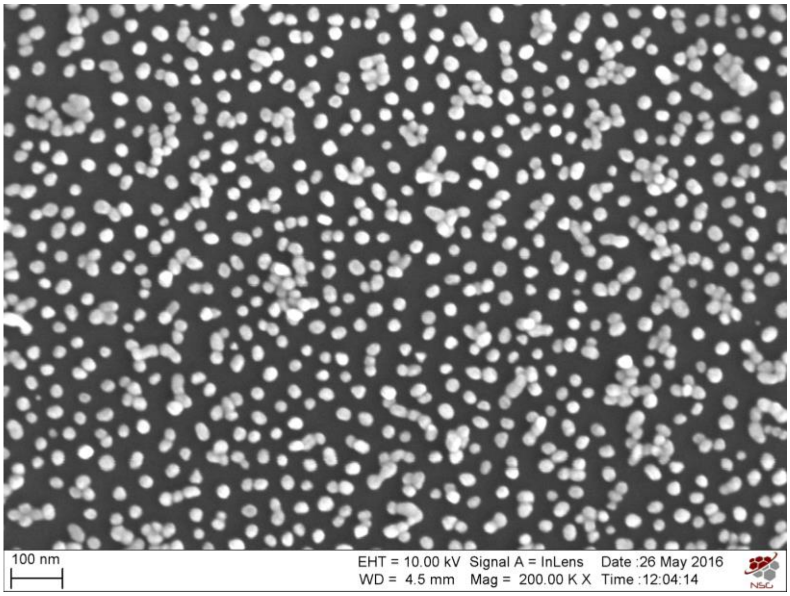
Synthesis



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Optical Characterization

Jasco V670 Spectrophotometer

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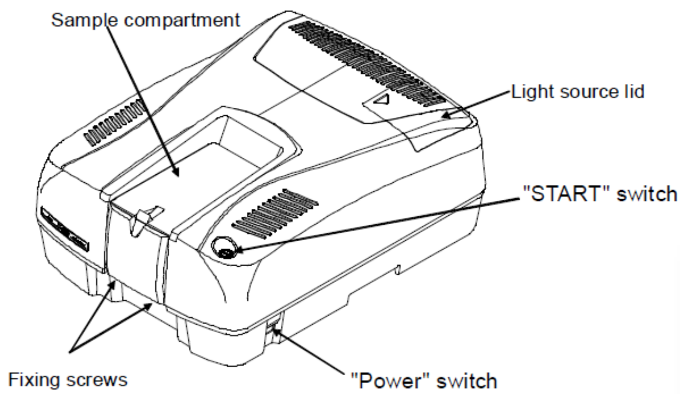
Optical Characterization

Introduction to NanoPhysics

Jasco V670 Spectrophotometer



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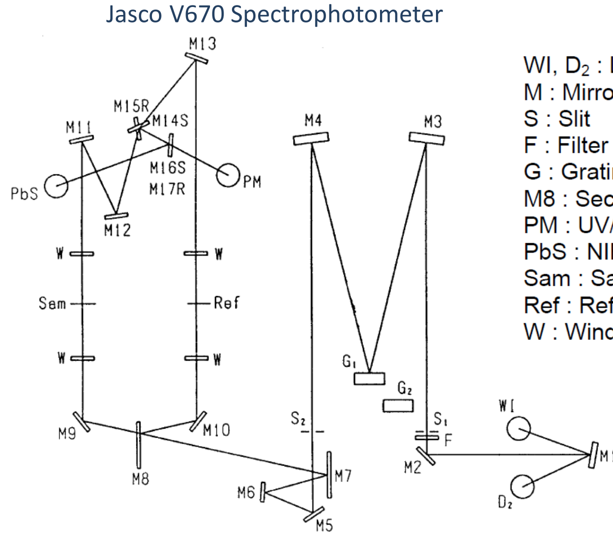
Optical Characterization

Introduction to NanoPhysics

Jasco V670 Spectrophotometer



WI, D_2 : Light source
M : Mirror
S : Slit
F : Filter
G : Grating
M8 : Sector mirror
PM : UV/VIS Detector
PbS : NIR detector
Sam : Sample beam
Ref : Reference beam
W : Window

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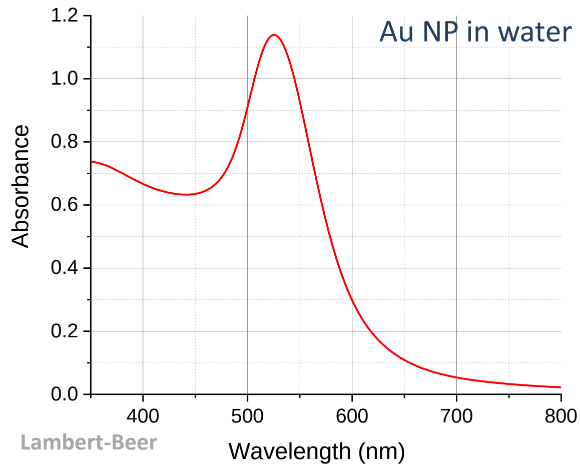
Optical Characterization

$z = 1\ cm$

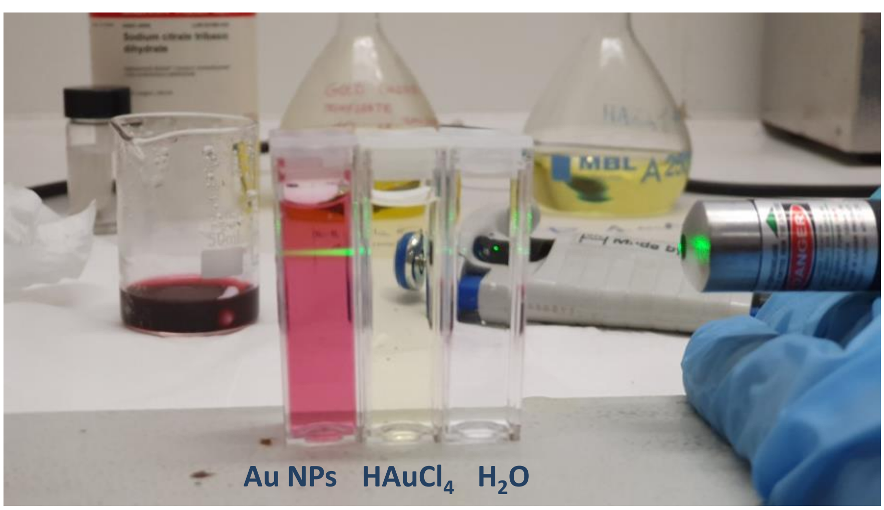


$I(z) = I_0 e^{-\gamma z}$ Lambert-Beer

$T \equiv \frac{I(z)}{I_0} = e^{-\gamma z}$ Transmittance

$A \equiv log_{10} \frac{1}{T} = \gamma\ z\ log_{10} e = log_{10}(e)\ z\ \varrho\ \sigma_{ext}$ Absorbance

Optical Characterization



Scattering from Au NP in water

Virtual Lab

Part 1: Synthesis

(NanoStructures Group Lab)

Virtual Lab

Part 2: Optical Characterization

(NanoStructures Group Lab)

Virtual Lab

HomeWork n.1

Analysis of the Absorbance

HW1: Aim

Simulation or **fit** with the Mie theory in the dipolar approx. (*size-corrected*)

→ *size, concentration, refractive index (R, ϱ, ε_m)*

HW1: Tools

Use any software you are familiar with

HW1: Activity

1. Simulate the Mie ext. cross-section using the experimental Au dielectric function of Johnson-Christy
2. Rescale it to match the experimental absorbance
3. Obtain the size-corrected Au dielectric functions (the bulk parameters can be taken from any textbook: e.g. Ashcroft-Mermin)
4. Check if it produces a better agreement
5. To obtain information on the stability of the best-fit parameters, calculate and plot the χ^2 values for different couples *(R, ϱ)* and *(R, ε_m)* and estimate the stability basin of your best fit result