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Nanomaterials UV-Vis measurement

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1 Works for me This protocol is published without a DOI.

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ABSTRACT

This SOP describes a sample preparation procedure for particle size measurements in gold NP suspensions. The procedure involves quantification of the extinction of light that is measured from the spectral pattern using absorbance. UV-Vis refers to the ultraviolet to visible spectral region of light, the absorption of which is size dependent at the nanoscale. UV-Vis is therefore an ideal technique for the size characterisation of NP suspensions through absorbance at an appropriate wavelength. The settings defined below will be refined to optimise results during the subsequent runs.

THIS PROTOCOL ACCOMPANIES THE FOLLOWING PUBLICATION

ISO/TS 17466:2015Preview. Use of UV-Vis absorption spectroscopy in the characterization of cadmium chalcogenide colloidal quantum dots. https://www.iso.org/standard/59853.html, Determination of Size and Concentration of Gold Nanoparticles from UV–Vis Spectra. https://pubs.acs.org/doi/abs/10.1021/ac0702084

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KEYWORDS

UV-Vis, Au, Gold, Nanoparticles, Nanomaterials

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GUIDELINES

1.1 Essential equipment

1. UV-Vis Spectrophotometer.
2. Calibrated Volume Pipettors of 1 and 5 mL with disposable tips.
3. Disposable 3mL cuvettes (Suggested: polystyrene 10 x 10 x 45mm, SARSTEDT, Catalogue number: 67.742).

1.2 Chemicals

1. Ultrapure water 18.2 MΩcm.

SAFETY WARNINGS

1.3 HSE issues

- All laboratory personnel must comply with local safety regulations when working in the laboratory.
- All personnel must consult Material Safety Data Sheets (MSDS) to be aware of known hazards relevant to all chemical substances used in this SOP.
- Personnel should utilise all necessary precautions to avoid exposure to chemical and nanomaterials (Lab-coats, nitrile gloves, protective glasses and masks).
- All residues and waste materials must be disposed of according to local environmental and safety regulations.

1 Sample Preparation

Vortex each AuNP stock suspension for 2 minutes.
Dilute the Au suspensions 1:2. Pipette 0.5 mL of the Au stock suspension and 0.5 mL UPW 18.2 MΩcm to obtain a final volume of 1 mL and a mass concentration of 25 mg/L.

2 Sample Analysis

1. Switch the UV-Vis Spectrometer (Jenway 6800 Double Beam Spectrometer Figure 3) on and leave for 20 minutes to allow the lamp to heat up.
2. Use 18.2 MΩcm UPW as the reference sample.
3. Before starting the measurements, the parameter settings shown in Table 1 should be set in the software (Flight Deck 1.0 or higher).

Parameter	Settings
Measurement Mode	Spectrum Scan
Data Mode	ABS
Start Wavelength	680 nm
End Wavelength	380 nm
Scan Speed	400 nm/min
Sampling Interval	0.5
Slit Width	1.5
Path Length	10

Table 1. UV-Vis Parameter settings

4. Baseline correction should be obtained by running a baseline using two cuvettes filled with 1 mL of UPW each, placed in the sample holders (Figure 4).
5. The reference cuvette with 1 mL UPW should then be left untouched and the other cuvette should be replaced with a new cuvette containing 1 mL of one of the diluted AuNP suspensions. A new cuvette should be used for each different sample analysed.
6. Three spectrum scan runs for each known BBI AuNP diluted suspension (5, 20, 40, 60 and 100 nm) should be obtained. Therefore a total of 15 scans should be collected. The results obtained should be reported as explained in Section 6 below and a calibration curve should be plotted.
7. Following this three spectrum scan runs for the unknown monodispersed AuNP suspension containing a monodispersed suspension of NPs of an unknown size.

3 Reporting results

1. Note the average maximum absorption wavelength (λ_{max}) of each if the UV-Vis readings (n=15) in the results Table 1.

2. Note the maximum absorption (AU_{max}) of each of the UV-Vis readings (n=15) in the results Table 2.
3. To add the results click the pencil button on the right hand-side of the results table.
4. The average λ, AU and and standard deviations for each set of measurements will be automatically calculated.
5. Plot the spectra of absorbance vs wavelength (nm) for each measurement (n=15).
6. Save the plots as a .jpg image and upload it using the File button above the results tables.

4 Data upload

1. Extract the raw experimental data for each measurement (n=18) as a .CSV or excel compatible file.
2. Upload the files using the File button above the results table.
3. The raw data will be used to calculate the Energy Band Gap (EBG) for each nanomaterials sample using Tauc plots and shared with partners.
4. (Optional) Add any comments you like in the comments section at the bottom of the results page.