

# UV/Vis/IR SPECTROSCOPY ANALYSIS OF NANOPARTICLES

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4878 RONSON CT STE K  
SAN DIEGO, CA 92111  
858 - 565 - 4227  
NANOCOMPOSIX.COM

**Note to the Reader:** We at nanoComposix have published this document for public use in order to educate and encourage best practices within the nanomaterials community. The content is based on our experience with the topics addressed herein, and is accurate to the best of our knowledge. We eagerly welcome any feedback the reader may have so that we can improve the content in future versions.

Please contact us at [info@nanocomposix.com](mailto:info@nanocomposix.com) or 858-565-4227 with any questions or suggestions.

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# Introduction

Ultraviolet/Visible/Infrared (UV/Vis/IR) spectroscopy is a technique used to quantify the light that is absorbed and scattered by a sample (a quantity known as the extinction, which is defined as the sum of absorbed and scattered light). In its simplest form, a sample is placed between a light source and a photodetector, and the intensity of a beam of light is measured before and after passing through the sample. These measurements are compared at each wavelength to quantify the sample's wavelength dependent extinction spectrum. The data is typically plotted as extinction as a function of wavelength. Each spectrum is background corrected using a "blank" – a cuvette filled with only the dispersing medium – to guarantee that spectral features from the solvent are not included in the sample extinction spectrum.

Nanoparticles have optical properties that are sensitive to size, shape, concentration, agglomeration state, and refractive index near the nanoparticle surface, which makes UV/Vis/IR spectroscopy a valuable tool for identifying, characterizing, and studying these materials. Nanoparticles made from certain metals, such as gold and silver, strongly interact with specific wavelengths of light and the unique optical properties of these materials is the foundation for the field of plasmonics. At nanoComposix we have developed numerical modeling algorithms that can be used to predict the optical properties of various nanoparticles allowing for comparison between theoretical and measured properties.

Our standard UV-Vis analysis is performed with an Agilent 8453 single beam diode array spectrometer, which collects spectra from 200-1100 nm using a slit width of 1 nm. Deuterium and tungsten lamps are used to provide illumination across the ultraviolet, visible, and near-infrared electromagnetic spectrum. Spectra are typically collected from 1 mL of a sample dispersion, but we can test volumes as small as 100  $\mu$ L using a microcell with a path length of 1 cm.

Additionally, we have assembled a variety of light source/spectrometer custom setups for measuring optical properties of materials from the ultraviolet to the deep-infrared (200 nm to 20  $\mu$ m), and can customize analytical systems to measure scattering or absorption from both liquid and solid samples. We also have a highly instrumented chamber for aerosolizing nanoparticles and measuring the optical properties of the suspended particles. For more information on aerosol applications, please visit [www.nanocomposix.com/services/aerosol-facility](http://www.nanocomposix.com/services/aerosol-facility).

## Sample Preparation

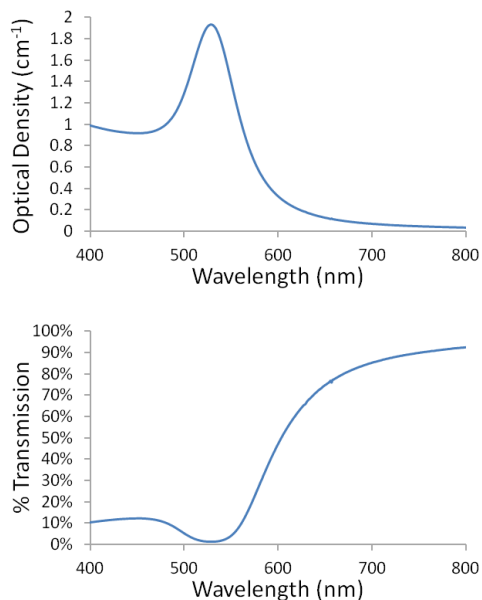
If samples are provided as a solution, we recommend sending at least 1 mL of your sample for measurement in a standard quartz cuvette. We can test volumes as small as 100  $\mu\text{L}$  using a microcell, though typically with slightly higher levels of noise, and in this case suggest sending at least 200  $\mu\text{L}$  of your sample. In addition, we suggest sending 2-5 mL of the solvent used to disperse your material, to be used to measure the solvent background and for any necessary dilution of your sample.

If sending a powder, please provide 5-10 mL of the necessary solvent for suspending the material, along with any specialized instructions for suspension (e.g, bath sonication, vortexing, etc.).

## Interpreting UV/Vis/IR Spectroscopy Data

### TRANSMITTANCE AND ABSORBANCE

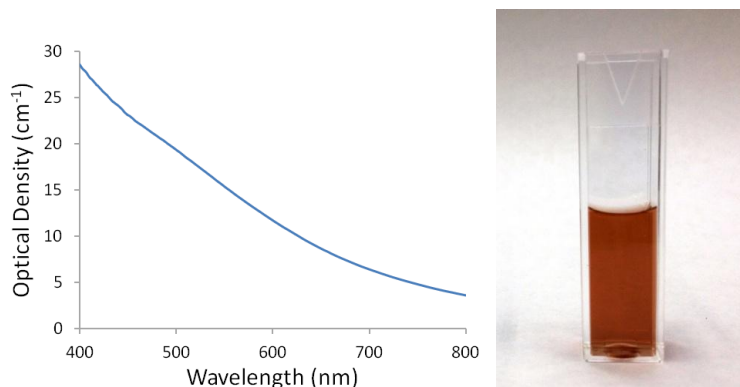
The transmittance of a sample ( $T$ ) is defined as the fraction of photons that pass through the sample over the incident number of photons, i.e.,  $T = I/I_0$ . In a typical UV/vis spectroscopy measurement, we are measuring those photons that are not absorbed or scattered by the sample. It is common to report the absorbance ( $A$ ) of the sample, which is related to the transmittance by  $A = -\log_{10}(T)$ . Figure 1 illustrates the relationship between transmittance and absorbance; the upper plot is the absorbance spectrum of 50 nm gold nanoparticles, and the lower plot shows the calculated transmittance. In the near IR, where the sample does not absorb strongly, the transmittance is close to 100%. In the UV portion of the spectrum, where the sample absorbs strongly, the transmittance drops to around 10% or less.



**Figure 1.** The absorbance (upper) and transmission (lower) spectra from a dispersion of 50 nm gold nanoparticles.

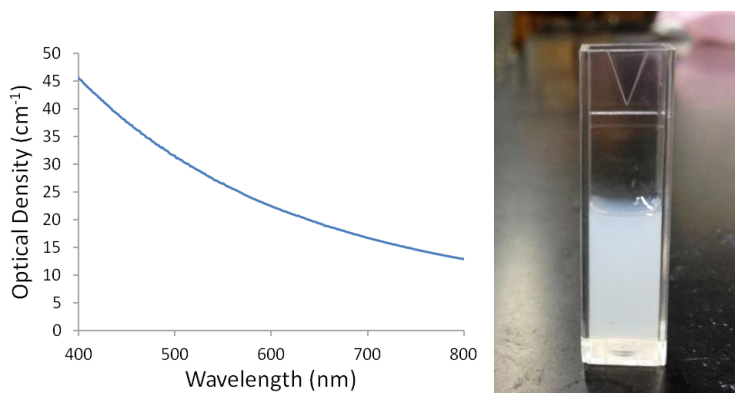
## SCATTERING AND ABSORPTION

The relative percentage of scatter or absorption from the measured extinction spectrum depends on the size, shape, composition and aggregation state of your sample. Your sample may absorb light, scatter light, or both. As a general rule, smaller particles will have a higher percentage of their extinction due to absorption. For example, light scattering by small (2 nm diameter) gold particles is negligible; the optical spectrum of the nanoparticles, shown below in Figure 2, is almost entirely due to photon absorption by the metal.



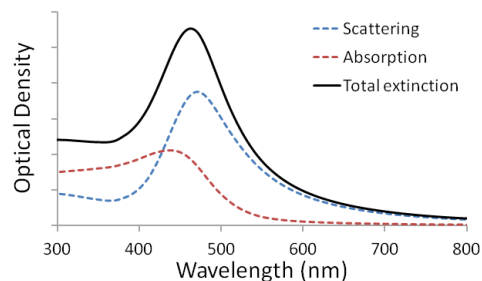
**Figure 2.** The experimental spectrum (left) from 2 nm gold nanoparticles is due almost entirely to light absorption with negligible scattering. The diluted solution (right) appears brownish-red since light is absorbed across the entire visible spectrum.

Conversely, amorphous silica does not absorb photons in the visible portion of the spectrum and large silica colloids will elastically scatter light (known as Rayleigh scattering). In the absorbance spectrum for 1  $\mu\text{m}$  diameter silica colloids, the spectrum shown in Figure 3 is typical of scattering from large particles. Even though the curves in Figure 2 and 3 are very similar the mechanism for extinction is different which is reflected in the strong differences in the visible appearance of the materials.



**Figure 3.** The experimental spectrum (left) of 1  $\mu\text{m}$  silica colloids is due almost entirely to light scattering with negligible absorption over the spectral range shown. The diluted solution of colloids (right) appears white since all wavelengths of light are scattered from the particles back toward the observer.

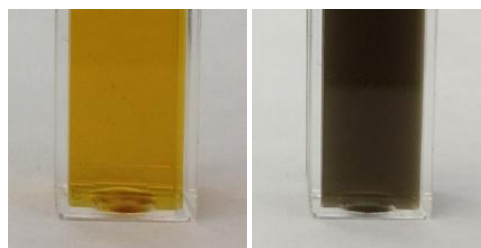
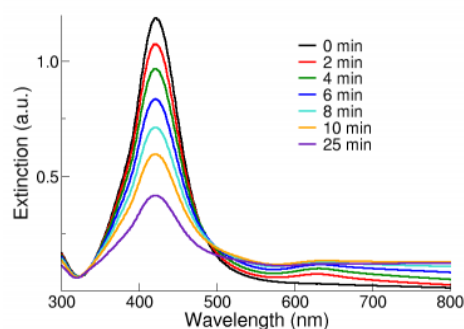
Most samples have contributions from both scattering and absorption. In some cases, it is possible to calculate the contribution of each effect using Mie Theory (see, for example, <http://nanocomposix.com/kb/general/plasmonics#modeling>, and our online Mie calculator at <http://nanocomposix.com/support/tools>). The calculated absorption spectrum for 50 nm gold nanoparticles is shown in Figure 4, along with the expected contributions from scattering and absorption.



**Figure 4.** The calculated spectrum for 50 nm gold nanoparticles in water (black) and the contributions from scattering (blue) and absorption (red) by the nanoparticles.

### EFFECTS OF AGGREGATION

Scattering from a sample is typically very sensitive to the aggregation state of the sample, with the scattering contribution increasing as the particles aggregate to a greater extent. For example, the optical properties of silver nanoparticles change when particles aggregate and the conduction electrons near each particle surface become delocalized and are shared amongst neighbouring particles. When this occurs, the surface plasmon resonance shifts to lower energies, causing the absorption and scattering peaks to red-shift to longer wavelengths. UV-Visible spectroscopy can be used as a simple and reliable method for monitoring the stability of nanoparticle solutions. As the particles destabilize, the original extinction peak will decrease in intensity (due to the depletion of stable nanoparticles), and often the peak will broaden or a secondary peak will form at longer wavelengths (due to the formation of aggregates). In the Figure 5, the extinction spectrum of 50 nm NanoXact silver is monitored as sodium carbonate is added to the solution (20 mM salt concentration). The rapid and irreversible change in



**Figure 5.** The absorption spectrum (upper) of silver nanoparticles changes as the particles transition from a well-dispersed state to an aggregated state following the addition of a concentrated salt solution; as aggregation proceeds, the plasmon peak at 420 nm decreases, a secondary peak at 620 nm emerges, and the baseline elevates due to scattering by aggregates. The final aggregated state is readily seen by eye, as the original dispersed particles form a yellow solution (lower, left) and the aggregated particles become grey (lower, right).

the extinction spectrum clearly demonstrates that the nanoparticles are agglomerating. UV/Visible spectroscopy can be used as a characterization technique that provides information on whether the nanoparticle solution has destabilized over time.