

## POINT OF VIEW

# The Challenges of Nanometrology for Metallic Nanoparticles

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The emergence of nanotechnology as the new industrial revolution following biotechnology has brought new challenges, new learning opportunities, and also the repetition of old mistakes. The discovery of new physical properties at the nanoscale and the ability to measure them significantly accelerated the amount of research related to this area. Furthermore, the possibility of direct manipulation at the nanoscale opened the door to the design of specific applications, resulting in a flood of new products on the market. All these advances go hand in hand with the need for methods and tools to accurately measure the dimensions, shapes, composition, and other characteristics of nanomaterials and nanostructures, among which nanoparticles play a prominent role. Without reliable measurements, the production of nanoparticles would be impossible, with a clear impact on their quality, the necessary government oversight, and, importantly, the public's understanding of this new industrial revolution.<sup>1</sup>

### What nanotechnology didn't learn from biotechnology?

Several decades ago, innovations in biotechnology followed a similar path. The market was flooded with biotechnological products, which simultaneously served as a "label" to distinguish and market them, and as a warning to those who distrusted them and their potential harm to health and the environment. This confrontation between the market and a segment of the population marked a learning point for the implementation of new developments.

Nanotechnology, as a new industrial revolution, had the opportunity to learn from this mistake, which could be summarized as empowering the public to take ownership of new advances. This implies information, education, but also extensive prior research, as well as disseminating this research to consumers and the general public.

Beyond the ethical obligation to keep the public informed, it is necessary to know what information is being shared. When dealing with new materials or structures, accurate measurements are necessary to establish precise toxicological studies and for proper quality control of commercial products containing them.

### Analytical challenges

In analytical terms, measuring the concentration of a substance in a given matrix is the ultimate goal of any metrological analysis. With nanotechnology products, the equivalent situation is not so simple, since the properties of nanoparticles depend not only on the chemical nature of what is being measured but also on their size and shape. The matrix plays a fundamental role that must be considered when validating the measurement, but in the case of nanoparticles, reference materials must be designed for a specific size and shape, making the number of required reference materials virtually infinite. The proposed analytical methods

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for discriminating the nature and size of nanoparticles include asymmetric flow field-flow fractionation (AF4), high-performance size-exclusion chromatography (HPSEC), and nanotracking analysis (NTA). These are complemented by specific characterization techniques, including UV/VIS spectroscopy (plasmonic properties), infrared/Raman spectroscopy (nature of the stabilizing agent), dynamic light scattering (DLS) (aggregate detection), Z-potential (colloidal stability), and transmission electron microscopy (TEM) (size and shape). In other words, a multiplicity of techniques is necessary to adequately describe the nanomaterial and the matrix in which it is embedded,<sup>2</sup> even allowing for the observation of changes resulting from interactions with the matrix.<sup>3</sup> Another layer of difficulty arises from the fact that there are no reference materials to validate the methodologies, so the techniques themselves do not have the metrological value to be used by official agencies in the control of products.

This is where analytical chemistry encounters its greatest challenges. Recent studies show that if UV/VIS spectroscopy and oxidation charge measurement analytical results are reported as a function of total atom concentration (mass or moles/L) instead of nanoparticles, it becomes possible to become independent of the size and chemical nature of the protecting agent. This greatly facilitates the generation of reference materials for technique validation. Future developments in analytical methodologies should aim to eliminate the influence of factors that alter analytical results, making them more general, so that more “universal” reference materials can be generated.

### Analytical decentralization

As I mentioned initially, commercial products containing nanoparticles have flooded the market, making it necessary to have rapid and decentralizable analytical methodologies to implement effective, rigorous, and quick control of these products. Many portable devices are available for their eventual use in the rapid control of commercial products, primarily liquids. For example, a simple laser pointer can determine the colloidal nature of a solution, allowing for an initial positive/negative analysis. Based on UV/VIS spectra measurements, cell phones can be used for rapid color measurement of a solution and its correlation with the optical properties of plasmonic nanoparticles (gold, silver, and copper). Oxidation charge measurements can be performed using disposable screen-printed electrodes and portable potentiostats.

In short, nanometrology presents an exciting challenge for the analytical community. Developing rigorous methodologies independent of particle size and stabilizing agents is essential for creating reference materials, which are crucial for properly controlling commercial products. Only through such efforts can the public have confidence in products emerging from this new industrial revolution.

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