Quantitative Analysis of Nanoparticles Using Differential Centrifugal Sedimentation (DCS)

Differential Centrifugal Sedimentation (DCS) is a technique used mainly for the size distribution analysis of nanoparticles. Whereas Laser Diffraction (LD) and Dynamic Light Scattering (DLS) are lower resolution analysis methods, DCS can be used to detect and resolve peaks down to 2nm and differing in size by as little as 2%.

Introducing the UHR CPS Disc Centrifuge

The Ultra High Resolution CPS Disc Centrifuge (Figure 1) nanoparticle size analyser utilises the technique of Differential Centrifugal Sedimentation (DCS). A sample is injected into a hollow, optically clear disc (Figure 2) containing a compatible density gradient, driven by a variable speed motor. The sample strikes the back face of the disc and forms a thin film which spreads as it accelerates radially toward the gradient liquid. On reaching the fluid surface, sedimentation of individual particles begins, and as particles approach the outside edge of the rotating disc, they block the detector light beam that passes through the disc. The change in light intensity is continuously recorded, and converted into a particle size distribution.

Fig.1 - *The UHR CPS Disc Centrifuge*

While DCS has been used to characterise particles for many years, recent technological advancements in the CPS Disc Centrifuge 'UHR' now allow for routine analysis of nanoparticle distributions down to 2nm and varying in diameter by as little as 2% as stated above. This unique high resolution capability of DCS also enables the accurate quantitation of nanoparticles calculated as a weight and number distribution; and is described below.

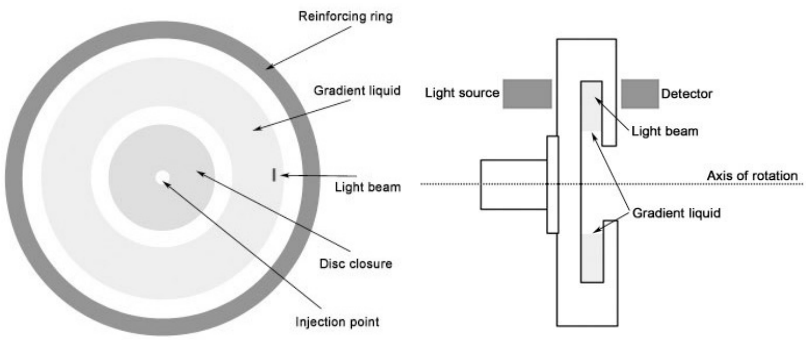

Fig.2 - *Schematic and cross-section of disc*

In the ideal case the analysis is entirely automated if:

1. The optical parameters for the particles and liquid are correct
2. The density values for particles and liquid are correct
3. The particles are spherical
4. The liquid viscosity is accurate

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For particles that are significantly smaller than the wavelength of light, they do not have to be perfectly spherical to get good results. Non-sphericity values up to ~1.5 still give reasonably good results with particles below about 150-200 nm. For larger non-spherical particles, it may be necessary to adjust one of the optical parameters a little to have the reported distribution match the known injected weight. This is because there are no perfect light scattering calculations possible for large non-spherical particles.

The general approach to quantitation by DCS is explained as follows:

At any moment during analysis, all the particles in the detector beam are essentially the same size. DCS relates the attenuation of the beam to the weight of particles in the beam by using Mie theory to calculate the theoretical turbidity for particles of known size.

Key to determining the absolute weight of particles is in knowing how far the particles have settled as they are detected. The sample 'spreads out' as the analysis proceeds; if the sedimentation path is longer, then the beam is at any moment seeing a smaller 'segment' (more correctly, a smaller differential) of the whole distribution, so the expected turbidity for the same injected weight of sample will be lower. In reality, the sample becomes more 'dilute' by the time it reaches the detector beam the longer the sedimentation path length. The sedimentation path length is calculated automatically based on the sedimentation time of the calibration standard and the viscosity of the liquid. The calibration standard run actually determines two independent parameters: the expected velocity of sedimentation of the sample particles as a function of diameter, and the path length through which the particles will settle to reach the detector.

In the example shown below a mixture of gold standards (equal volumes of 10, 15, 20, 30 nm particles) was analysed using the CPS Disc Centrifuge. This analysis was complete in less than 4 minutes; typical analysis times are in the order of 3-15 minutes, with up to 40 samples analysed in a single run.

The results are shown graphically (Figure 3) as particle diameter in microns versus the absolute weight measurement in micrograms. The reported distribution is absolute, integration of the entire curve is the absolute weight of particles; integration of any portion of the curve yields the absolute weight of particles in the selected size range. So DCS provides not just total sample weight, but the weight of particles in any desired portion within that distribution (Figure 4). If an accurate sample injection syringe is used, even quantitative particle concentration can also be calculated.

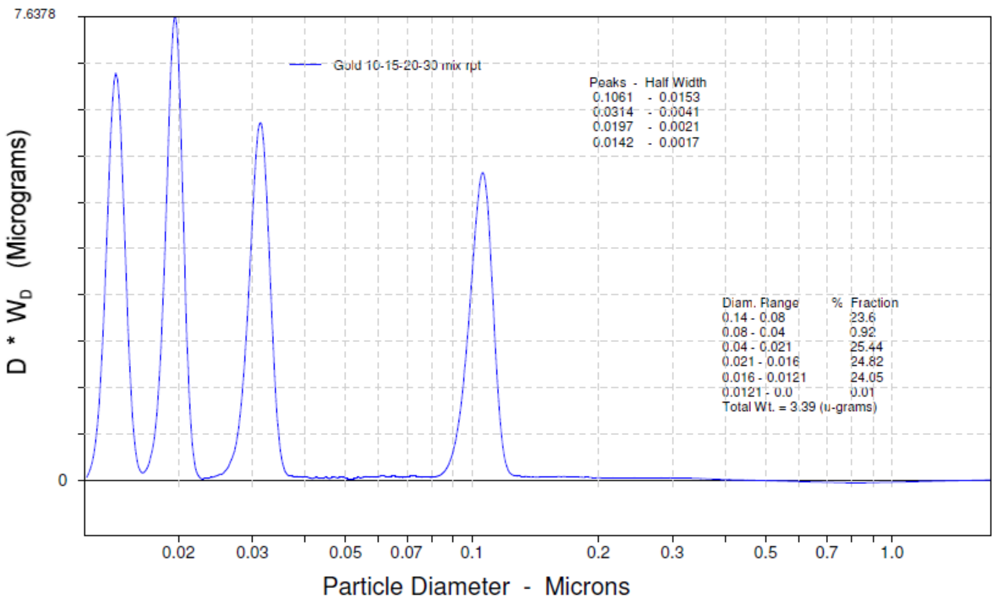

Fig.3 - *CPS DCS results of gold standards mixture*

Integration of each fraction (showing absolute weight in micrograms) for the mixture of gold particles is shown below (Figure 4):

Diameter range/nm	% Fraction	Weight/ug
0.14 - 0.08	3.6	0.8000
0.08 - 0.04	0.92	0.0312
0.04 - 0.021	25.44	0.8624
0.021 - 0.016	24.82	0.8414
0.016 - 0.0121	24.05	0.8153
0.0121 - 0	0.01	0.0003

Fig.4 - *Quantitative results from CPS Disc Centrifuge*

DCS is an extremely powerful technique for high resolution particle characterization, enabling very narrow distributions of particles differing in size by less than 2% to be resolved. Quantitative analysis as total sample weight or the weight of particles in any portion is available for this same high resolution distribution.

Want to find out more?

To learn more about high-resolution particle size characterisation using the UHR CPS Disc Centrifuge visit **analytik.co.uk/cps** (UK and Ireland) or visit **cpsinstruments.eu**.