

## Measurement of the particle diameters of dilute solutions by centrifugal concentration

CP-WX series preparative ultracentrifuge and P70AT fixed angle rotor

Given the considerable development of the nanotechnological industry in recent years, particle sizes of the nanometer order are now being controlled in materials used in various fields. Pretreatment is very important in measuring the grain size distribution of prepared samples with high reproducibility and accuracy. Among the methods of such pretreatment are separation and refinery that entail eliminating the effects of contamination and aggregate by using an ultracentrifuge, and the concentration of samples that are low in concentration or scattering light intensity.

This paper introduces a method of using an ultracentrifuge to concentrate the samples that are low in concentration and having scattering light intensity so weak that it cannot be measured in raw liquid, and using a laser diffraction/scattering-type particle characterization device to examine the particle size distribution of such samples.

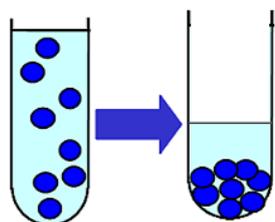


Fig. 1 Image of centrifugal concentration

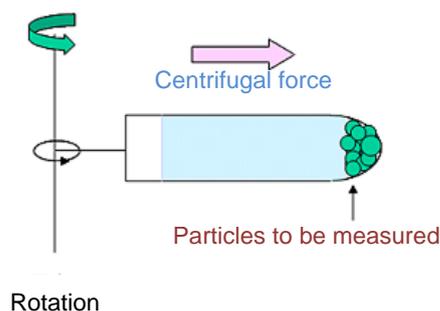


Fig. 2 Centrifugal concentrating system

### Description

#### 1. Samples

- ① 30-nm colloidal silica (Ludox TM-50, density: 1.40 g/cm<sup>3</sup>, manufactured by Grace), 0.25 mass%
- ② 80-nm PSL (NIST Standard 3080A, density: 1.050 g/cm<sup>3</sup>, manufactured by Thermo Fisher Scientific), 0.025 mass%

#### 2. Centrifugal and measuring conditions

Centrifuge: CP100WX ultracentrifuge

Rotor: P70AT fixed angle rotor (8 tubes)

Centrifuging tube: 30PC bottle (C3)

Rotating speed: 50,000 rpm

Maximum RCF: 257,000 Xg

Centrifugal time: 40 minutes

Particle characterization device: LA-950V2 laser diffraction (manufactured by Horiba, Ltd.)

Measuring temperature: 25°C

Particle size distribution standard: Volume

Sample refraction index: 1.45-0.00i (colloidal silica), 1.59-0.00i (PSL)

Dispersion medium refraction index: 1.33-0.00i

Measuring technique: Batch-type cell (for very small trace quantities: approx. 10 ml)

### 3. Results

The distribution of particle sizes without the pretreatment of colloidal silica (30 nm; 0.25 mass%) and PSL (80 nm; 0.025 mass%) samples cannot be effectively measured due to very low scattering light intensity. This prompted our use of an ultracentrifuge to concentrate the samples in order to double their concentrations, thereby allowing us to measure the distribution of particle sizes. Figures 3 and 4 show the results. Each sample was measured three times, resulting in high reproducibility.

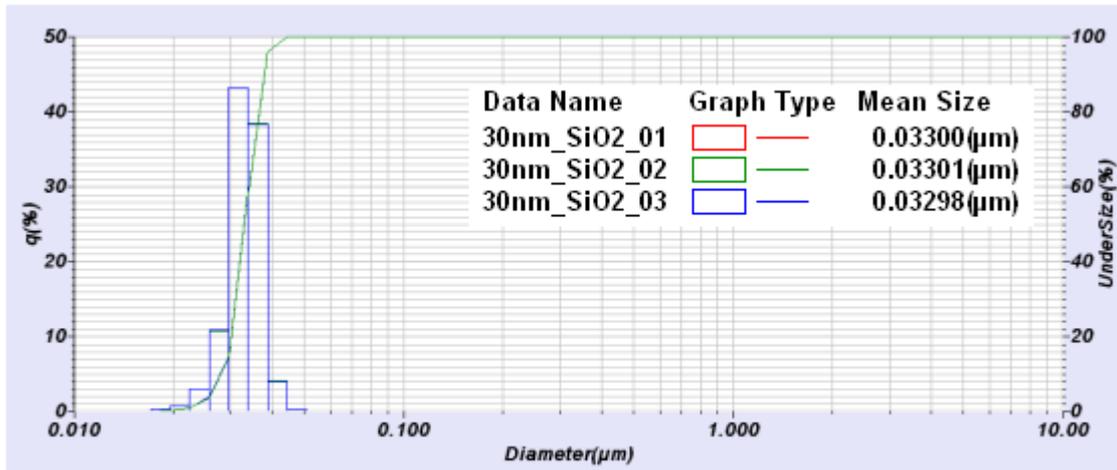


Fig. 3 Comparison of particle size distribution of colloidal silica (30 nm) after centrifugal concentration

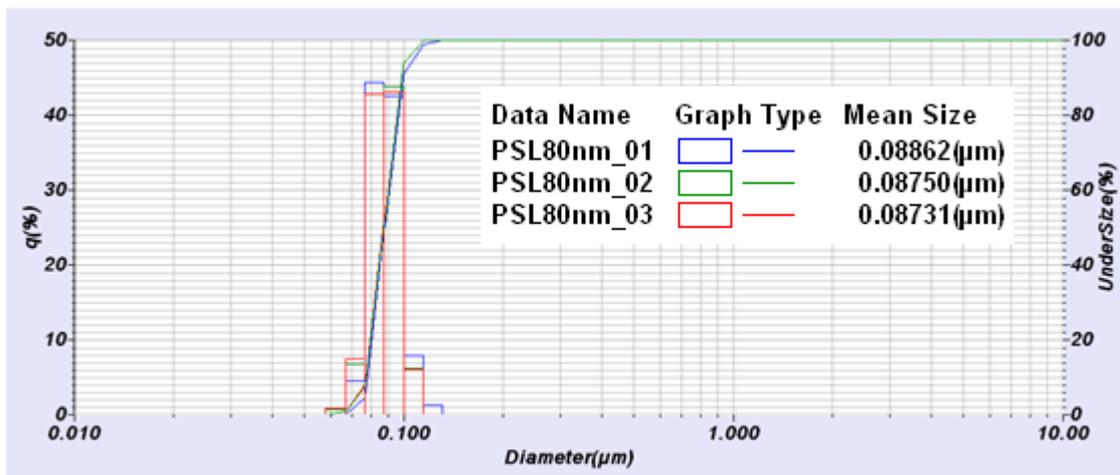


Fig. 4 Comparison of particle size distribution of PSL (80 nm) after centrifugal concentration

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