CPS
CPS Instruments Europe
P.O. Box 180, NL-4900 AD Oosterhout, The Netherlands
T: +31 (0)162 472478 F: +31 (0)162 421944 E: info@cpsinstruments.eu

Speed Ramping with the CPS Disc Centrifuge

Introduction

Some types of samples contain a very wide range of particle sizes. Historically, these samples have been very difficult to measure using the differential sedimentation sizing method, because differential sedimentation has been limited to a dynamic range of about 70. This document explains how a modified disc design and ramping of centrifuge speed is used to greatly increase dynamic range with the CPS Disc Centrifuge, easily reaching a dynamic range of 1000.

Dynamic Range Limit for Fixed Speed

Differential sedimentation separates particles according to size, in accordance with Stokes' Law. This means that particles sediment at velocities that are proportional to the square of their diameters. A 10 micron particle sediments 100 times faster than a 1 micron particle. The large differences in sedimentation speed give differential sedimentation very high resolution compared to most other particle sizing methods, because relatively small differences in diameter translate to significantly greater differences in sedimentation velocity (see the document "*Ultra-High Resolution Analyses with the CPS Disc Centrifuge*" in the CPS Technical Library). Unfortunately, high resolution does not come without a price: the large differences in sedimentation velocity also limit dynamic range. (Dynamic range is the ratio of the largest measurable size to the smallest measurable size in a single analysis.)

Suppose that you had a sample where you wanted to measure particles between 20 microns and 0.05 microns. You must select a centrifuge speed so that the 20 micron particles (fastest moving) arrive at the detector beam no faster than ~0.75 second after injection, because it is not possible to accurately time the sample injection and collect distribution data with a sedimentation time below about ~0.75 second. The smallest particles will sediment at a much slower rate than the largest. The ratio of sedimentation speeds is: (D / D)^2. If the 20 micron particles reach the detector in 0.75 second, then the 0.05 micron particles reach the detector at 0.75 * (20 / 0.05)^2, or 120,000 seconds, or 33 hours and 20 minutes. This is clearly not a practical analysis time, and even if you were willing to wait 33 hours for results, Brownian motion of the smallest particles over 33 hours of sedimentation would cause substantial errors in the reported distribution.

The dynamic range depends in reality upon how long you can wait for results. If the longest run you can tolerate is 30 minutes, then the practical dynamic range is:

((30 * 60) / 0.75)^0.5 = 48.99

If you can wait only 15 minutes, the dynamic range falls to 34.64; if you can wait 60 minutes, the practical dynamic range is 69.28.

Many types of samples (if not most types) are easily measured with a dynamic range of 30 to 70, and so present no problem for the CPS Disc Centrifuge. However, there are some types of samples that really do have a broader dynamic size range, and these have been difficult or impossible to completely characterize using the CPS Disc Centrifuge.

Ramping the Disc Speed

The G-force inside the centrifuge is proportional to the square of the rotational speed, and so the sedimentation velocity of a particle is also proportional to the square of the rotational speed. If the rotational speed at the beginning of an analysis were low, and then gradually increased during the analysis, then the problem of limited dynamic range would be resolved: the lower initial speed allows analysis of the large end of the distribution, while the higher final speed allows you to measure the smallest particles in a practical run time.

CPS
CPS Instruments Europe
P.O. Box 180, NL-4900 AD Oosterhout, The Netherlands
T: +31 (0)162 472478   F: +31 (0)162 421944   E: info@cpsinstruments.eu

Consider the 20 microns to 0.05 micron sample discussed in the above section (dynamic range of 400). If the rotational speed were increased by a factor of 12 over the first 6 - 10 minutes of the analysis, then the whole size range could be measured in a run lasting only ~ 20 to 23 minutes, which is a reasonable run time for most particle sizing applications.

To make speed ramping practical, a couple of operational problems have to be resolved.

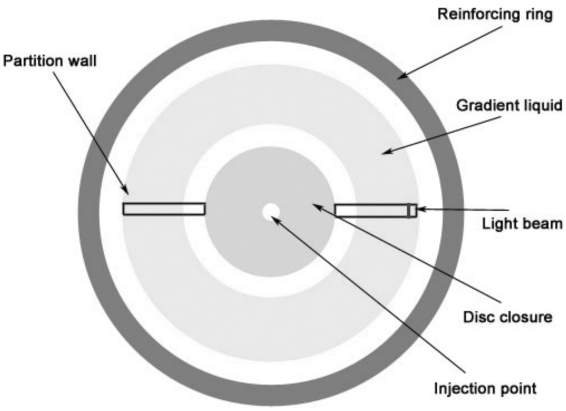
- Disrupting the Gradient

  Stable sedimentation (no "streaming") in the CPS Disc Centrifuge depends on having a density gradient inside the disc chamber, where the fluid at the outside edge of the disc is slightly higher in density than the fluid at the surface, and where the fluid density changes gradually between these two extremes. So long as the disc is turning at constant speed, the fluid inside the disc is "quiescent"; there is no reason for the gradient to be disrupted. If the disc speed is changed after the gradient is established, the change in speed will cause turbulent mixing of the fluids inside the disc, and total disruption of the density gradient. In other words, the fluid inside the disc will become uniform in composition and stable sedimentation will not be possible.

- Calculating the Particle Size

  If it were possible to avoid disruption of the density gradient, it would still be necessary to account for the changing speed so that an accurate particle size distribution can be calculated.

Resolving the Speed Ramp Problems



CPS Instruments has resolved the problems of gradient disruption and calculation of an accurate size distribution with changing disc speed. Figure 1 shows how the disc is modified to avoid disruption of the gradient. Two "separator walls" are placed inside the disc chamber. During acceleration or deceleration of the disc, these separators keep the fluid inside the disc from moving at a speed different from the disc speed, and so eliminate virtually all mixing due to acceleration/deceleration.

Figure 1 - Disc Design for Speed Ramping

The problem of calculating an accurate size distribution with changing speed is solved by modifying the operating software to account for changing disc speed. The equation below is the differential form of Stokes' Law, modified to account for the G-forces inside a centrifuge. The equation shows the velocity of sedimentation of a particle:

$$dR/dt = R\,(D^2 (\rho_p - \rho_f)\,\omega_t^2) / (18\,\eta) \qquad \text{(Eq. 1)}$$

Where:
- η is the fluid viscosity
- ρ_p is the particle density
- ρ_f is the fluid density
- R is the radius of rotation
- ω_t is the rotational speed as a function of time
- t is time
- D is the particle diameter

Note that all of the terms on the right side of equation 1 except ω_t and R are virtually independent of time. Equation 1 can be rearranged to separate variables, and integrated:

$$ln\ (R/R_0) = (D^2\ (\rho_p - \rho_f))\ /\ (18\ \eta) \int \omega_t^2\ dt \qquad \text{(Eq. 2)}$$

Where: R_0 is the radius at the start of sedimentation (the surface)

$$D^2 = 18\ \eta\ ln\ (R/R_0)\ /\ ((\rho_p - \rho_f)) \int \omega_t^2\ dt) \qquad \text{(Eq. 3)}$$

Finally:

$$D = (18\ \eta\ ln\ (R/R_0)\ /\ ((\rho_p - \rho_f)) \int \omega_t^2\ dt))^{½} \qquad \text{(Eq. 4)}$$

Equation 4 shows that so long as we know how the rotational speed ω_t varies with time we can continuously integrate with respect to time during an analysis and generate an accurate distribution.

Example Analysis

Figure 2 shows a particle size distribution for a sample of poly vinylchloride (PVC) over a size range of 32 microns to 0.06 micron (dynamic range of 533). The distribution consists of 2,348 data points. The analysis started at 1,200 RPM, and the centrifuge speed increased over 6.375 minutes to 16,500 RPM, then was held constant for the remainder of the analysis. Total run time was 15.7 minutes. After the run was completed, an additional 6.375 minutes was used to return to 1,200 RPM for the next analysis, so total cycle time (run to run) was ~22 minutes. This sample could never be run at constant centrifuge speed, because analysis time would be more than 50 hours. The CPS Disc Centrifuge operating software allows speed ramping with any CPS Disc Centrifuge (12,000 o 24,000 RPM maximum speeds), so all that is required for speed ramping is the special disc described above. Please contact your local representative or CPS for additional information or a price quotation.

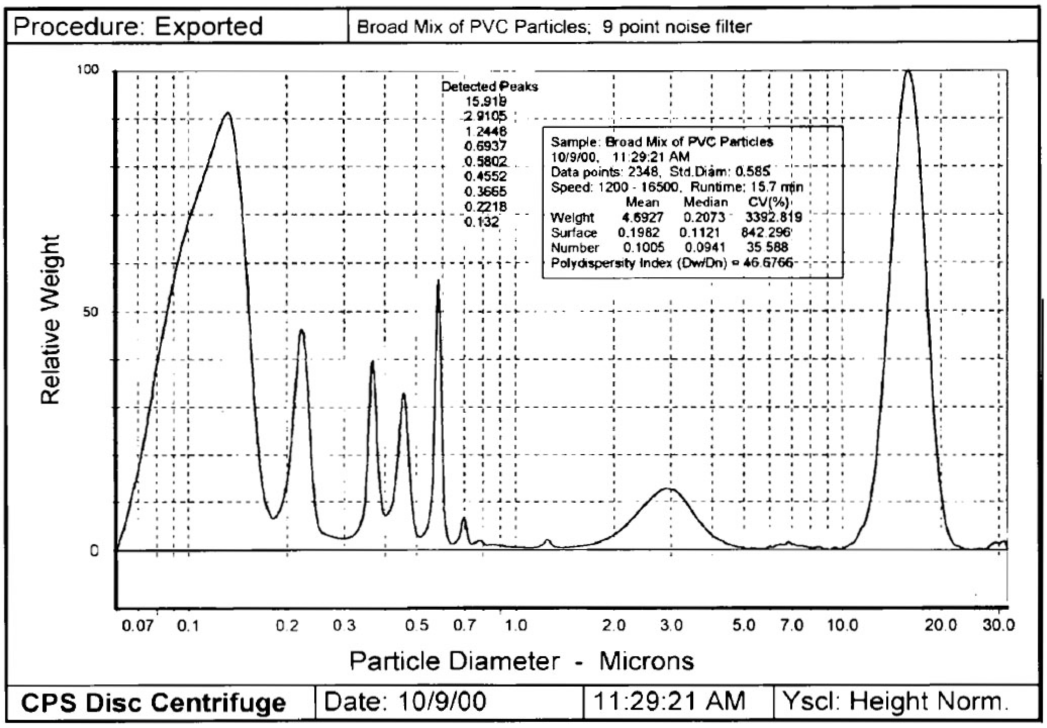


Figure 2