Particle Size Validation with Multi-Angle DLScat Instrument

Mohit Agarwal (Swabian Instruments GmbH)

The dynamic light scattering instrument developed by Swabian Instruments, also known as DLScat, measures particle size from the diffusion-driven fluctuations of scattered light, which are recorded simultaneously at five multiple-scattering angles. Simultaneous multi-angle acquisition improves robustness for polydisperse and larger samples, where a single angle can give an angle-dependent, and therefore biased, size [1]. This note reports a validation study in which certified and nominal size standards spanning roughly 30 nm to 10000 nm were measured with DLScat under standard aqueous conditions, assessing the reported size, polydispersity index (PDI), and repeatability against the manufacturer's values, using three analyses derived from the same measurement data.

METHOD

All samples were dispersed in 0.22 µm-filtered 10 mM NaCl (Art. No. 228G.1, Carl ROTH) and measured at 25 °C, with an acquisition time of 300s, repeated three times on the same loaded sample, extended to 600s for standard particles above 500 nm. Working suspensions were prepared by diluting the manufacturer's stock in the filtered medium until each angle reached 250 to 350 kcps at 5 mW laser power, and the measurement was repeated twice at that concentration. A subsequent dilution has been performed to minimize multiple scattering, which can result in smaller sizes. Table 1 lists the resulting number concentration for each standard. Each dataset was analyzed in three complementary ways. The primary size is obtained from the five multi-angle diffusion-coefficient extrapolations (D vs q^2), which fit the initial decay at each angle and extrapolate to scattering vector, q = 0, to remove any residual angular dependence. The decay-rate extrapolation (Γ vs q^2) provides a second multi-angle estimate by fitting all angles simultaneously, with greater weight on the larger angles. The 157° backscattering angle provides a single-angle estimate that, unlike the low-angle data that influence the other two methods, remains comparatively robust to sedimentation and number fluctuations at large sizes. D vs q^2 is reported as the default size, and the other two serve as cross-checks. A standard was verified when the D vs q^2 diameter fell within ±15% of the manufacturer's size. Within this size range, when the three modes agreed with one another to within ±15%, the resulting band was defined as acceptable for this study. ISO 22412 specifies a reproducibility of 2 to 5% for monodisperse samples between 50 and 200 nm, as shown by the errors in the measured diameters column of Table 1 [1].

RESULTS

Across the core range of roughly 30 to 500 nm, DLScat reported diameters within the ±15% acceptance limit and with excellent reproducibility (Figure 1a). The 30 nm silica standard measured 28.6 nm, and the polystyrene standards specified at 54, 100, 197, 239, 286, and 410 nm each fell close to their specified sizes, with PDIs consistent with near-monodisperse standards (Table 1). Whether independent analyses converge on the same size is a useful internal check, and across the core range, the three modes agree within ±15% limit, with none consistently closer to others (Figure 1b). D vs q^2 is reported as the default. It forms the apparent diffusion coefficient at each angle and extrapolates to q = 0, isolating the translational diffusion coefficient free of angular dependence, so it remains valid for polydisperse or unknown samples where a single fixed angle weights the distribution by its angle-dependent scattering. Γ vs q^2 recovers the same coefficient from the slope of Γ against q^2, which is equivalent when that coefficient is angle-independent, but only assuming strict linearity through the origin; both Γ vs q^2 and the 157° backscatter are reported as cross-checks. The one core-range difference is the 200 nm standard, where D vs q^2 gives 199 nm against 211 to 215 nm from the other two, all still within ±15%.

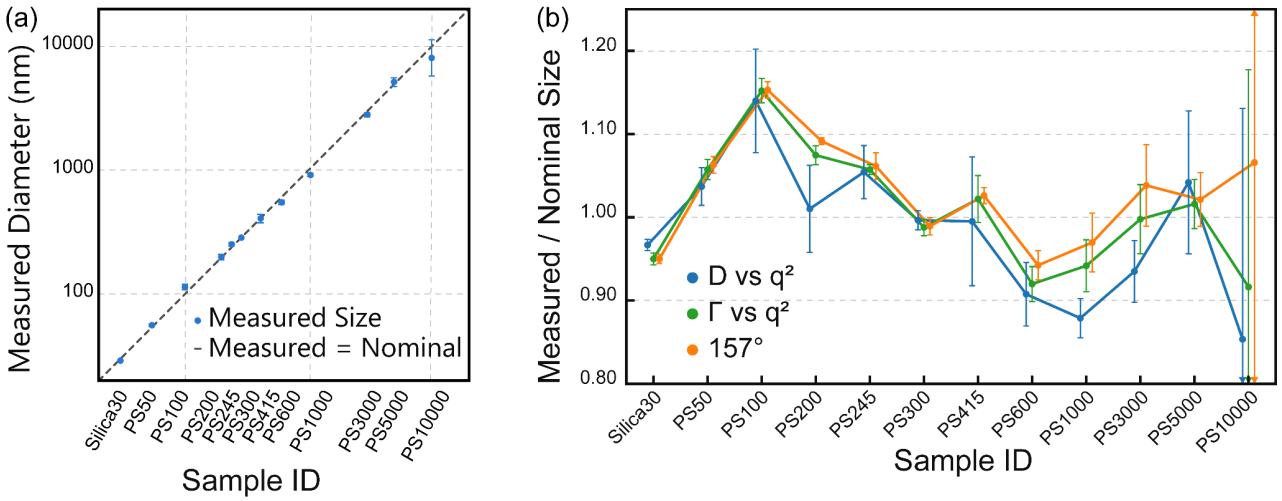

Figure 1. (a) DLScat measured diameter (D vs q^2, blue circles with repeatability error bars) against the manufacturer's specified size on logarithmic axes; the dashed line marks equality of measured and specified size. **(b)** The comparison of three analysis modes (D vs q^2, Γ vs q^2, and 157°) for several DLS size standards by dividing the DLScat measured diameter by the nominal sizes. A value of 1.0 indicates agreement with the nominal standard. The 10 µm error bars extend beyond the plotted range and are shown with arrows.

Above 600 nm, the modes diverged (Figure 1b), and the 157° backscatter became the more reliable estimator: as particles grow, sedimentation and number fluctuations distort the low-angle data underlying both extrapolations, so D vs q^2 and Γ vs q^2 read progressively low while the backscatter channel stays close to the specified size. The primary readout is therefore D vs q^2 up to about 600 nm and 157° above it, a change of estimator with size rather than a disagreement between modes.

Table 1: DLScat measured diameter (using D vs q^2 plot) and deviation from standard particle sizes.

Sample ID	*Manufacturer (Product No.)*	*Manufacturer Diameter (nm, PDI)*	*Number conc. (Particles/mL)*	*Measured Diameter, Ø (nm)*	*Deviation*
Silica30	micromod (43-00-301)	30 (< 0.20)	3.5×10^{13}	28.6 ± 0.2	−3.3%
PS50	Applied Microspheres (WS0130.21)	54 (< 0.05)	1.2×10^{14}	56 ± 1.2	+3.7%
PS100	micromod (01-00-102)	100 (< 0.20)	9.1×10^{10}	114 ± 6	+14.0%
PS200	microParticles (PS-R-0.2)	197 (< 0.03)	1.2×10^{10}	199 ± 10	+1.0%
PS245	microParticles (PS-R-0.25)	239 (< 0.03)	6.7×10^{9}	252 ± 8	+5.4%
PS300	Applied Microspheres (CH0330.231)	286 (< 0.01)	7.8×10^{11}	285 ± 3	−0.3%
PS415	microParticles (PS-R-0.4)	410 (< 0.03)	1.3×10^{9}	408 ± 32	−0.5%
PS600	Applied Microspheres (21600-20)	606 (< 0.03)	5.0×10^{5}	550 ± 23	−9.2%
PS1000	Applied Microspheres (22010-20)	1040 (< 0.03)	5.0×10^{5}	914 ± 25	−12.1%
PS3000	Merck (79166-5ML-F)	3000 (< 0.03)	1.0×10^{7}	2842 ± 127	−6.5%
PS5000	Applied Microspheres (22050-20)	4940 (< 0.02)	5.0×10^{5}	5147 ± 424	+4.2%
PS10000	Applied Microspheres (22100-20)	9990 (< 0.01)	5.0×10^{5}	8526 ± 2770	−14.7% *

** The 10000 nm point lies beyond the reliable range of DLS and is reported for completeness.*

DISCUSSION

Across 12 standards ranging from 30 nm to 9990 nm, supplied by four different manufacturers and covering silica and polystyrene, every D vs q^2 diameter fell within the ±15% acceptance limit of the specified size. Agreement was closest across the 30 to 500 nm core range, where the mean absolute deviation was about 4%, and the deviations changed sign from standard to standard, indicating scatter about the specified values rather than a calibration offset. Beyond the core range, the deviations become predominantly negative, consistent with the low-angle data being affected by sedimentation and number fluctuations. The three analysis modes

agreed within ±15% throughout the size range, so the reported size reflects the sample rather than the chosen readout; above about 600 nm, the modes diverge, and the 157° backscatter is preferred, a change of estimator with size. The 10 µm standard met the numerical limit but lies outside the technique's reliable range.
Reaching this agreement required setting the working concentration per sample rather than inferring it from size, since it varied roughly fiftyfold and not monotonically: 40 µL/mL for the low-contrast 30 nm silica; 1 to 2 µL/mL between 200 and 415 nm; and 50 µL/mL again above 600 nm, where too few particles occupy the scattering volume. These volumetric dilutions are stock-dependent, so a number concentration in particles per milliliter, as listed in Table 1, is the more transferable target, although the count rate remains the size-independent criterion because scattering per particle rises steeply with diameter. The upper bound is due to multiple scattering, which shortens the correlation decay and biases the diameter downward. Two steps located the window in every case: dilute until every angle reads 250 to 350 kcps, then repeat at twice the concentration, since a diameter that rises on dilution shows the first measurement was still multiply scattering.
Where no certified value exists, four internal criteria were applied: the three modes agree within ±15%; Γ varies linearly with q^2; the PDI matches the expected distribution width; and the diameter is independent of acquisition duration between 300 and 600s. Together, these identified an aged polystyrene stock as aggregated rather than mismeasured, based solely on its oversized diameter and elevated PDI. Above 5000 nm, sedimentation competes with diffusion, and the PDI rises to about 0.3, making that the practical upper limit in the standard 10 × 10 mm cuvette with 1 mL of sample.

CONCLUSION

DLScat sized standards from 30 nm to 5000 nm to within ±15% of their specified values across silica and polystyrene from four different suppliers, but reaching that agreement meant finding a workable concentration to be established per sample rather than inferred from size, since the requirement varied fiftyfold and not monotonically. The second observation is that simultaneous multi-angle detection is valuable beyond the extrapolation itself: because D vs q^2, Γ vs q^2 and 157° derive from a single measurement file, their mutual agreement, together with the linearity of Γ against q^2 and the PDI, judges a measurement where no specified value exists. D vs q^2 remains the primary readout, with backscattering the more reliable estimator above 600 nm, and 300s a sensible minimum acquisition time. For samples with diameters greater than 5000 nm, sedimentation competes with diffusion, making it difficult to obtain a stable size.

REFERENCES

[1] International Organization for Standardization, Particle size analysis — Dynamic light scattering (DLS), ISO 22412:2025, 3rd ed. (ISO, Geneva, 2025)

www.swabianinstruments.com | info@swabianinstruments.com | +49 711 40044790
Swabian Instruments GmbH | Stammheimer Str. 41 | 70435 Stuttgart | Germany