APPLICATION NOTE

Laser Diffraction Round Robin Test: Three Perfect Results for MICROTRAC

Round robin test: objectives and implementation

The Microtrac laboratories in Haan (Germany), Osaka (Japan) and York (USA) successfully participated in an interlaboratory test conducted by BAM (German Federal Institute for Materials Research and Testing) in 2021. The focus of the test was "Measurement of the particle size distribution of ceramic powder by laser diffraction in accordance with ISO 13320". The aim of the round robin was to provide proof of performance and internal quality assurance for the participating laboratories. A total of 45 laboratories in 17 countries had signed up for the round robin. Measuring instruments from 7 different manufacturers were used. Five laboratories failed the test. 30 laboratories achieved a pass rate of 100%, including all three MICROTRAC sites!

This not only confirms excellent instrument to instrument comparability of MICROTRAC SYNC analyzers, but also the reliability of the results obtained. The SYNC devices used were instruments with the 3R configuration (three red lasers).

For the round robin test, 3 samples of ceramic powders were sent from BAM to the participating laboratories. Detailed specifications were made regarding sample preparation, dispersion, and evaluation to achieve the greatest possible comparability. The results in the form of the percentile values d10, d50 and d90 were transmitted to BAM in electronic form and evaluated there using statistical methods. Each test participant received a detailed final report, which also included the results of the other laboratories in anonymized form.

Sample materials and sample preparation

The following three powders were analyzed in the interlaboratory comparison:

- Sample 1: Al_2O_3 (corundum)
- Sample 2: SiC (silicon carbide)
- Sample 3: $BaTiO_3$ (barium titanate)

The analyses were performed in 3 mmol/l tetra-sodium diphosphate solution. Each sample was predispersed in a beaker and treated with an external ultrasonic probe according to BAM specifications:

- Sample 1: 5 minutes
- Sample 2: 1 minute
- Sample 3: 11 minutes

In each case, as much sample was added to the measuring instrument until a sufficient concentration for analysis was reached. Then 5 measurements (without changing the sample) were performed and the mean values from the individual analyses were determined for the d10, d50 and d90 percentiles. The entire procedure including sample preparation was repeated six times for each material, so that a total of 30 individual measurements were available from each sample.

The analysis was evaluated according to MIE theory. The refractive indices were specified by BAM.

	Real (λ = 663 nm)	**Imaginary (λ = 633 nm)**	**Shape**
Sample 1	1.766	0.001	Spherical
Sample 2	2.63	0.1	Irregular
Sample 3	2.40	0.01	Irregular

In addition, data were provided for blue light (λ = 470 nm). For the "Modified Mie" evaluation of the Microtrac analyzers, it is not necessary to specify the imaginary part, only the particle shape (spherical / irregular) must be defined.

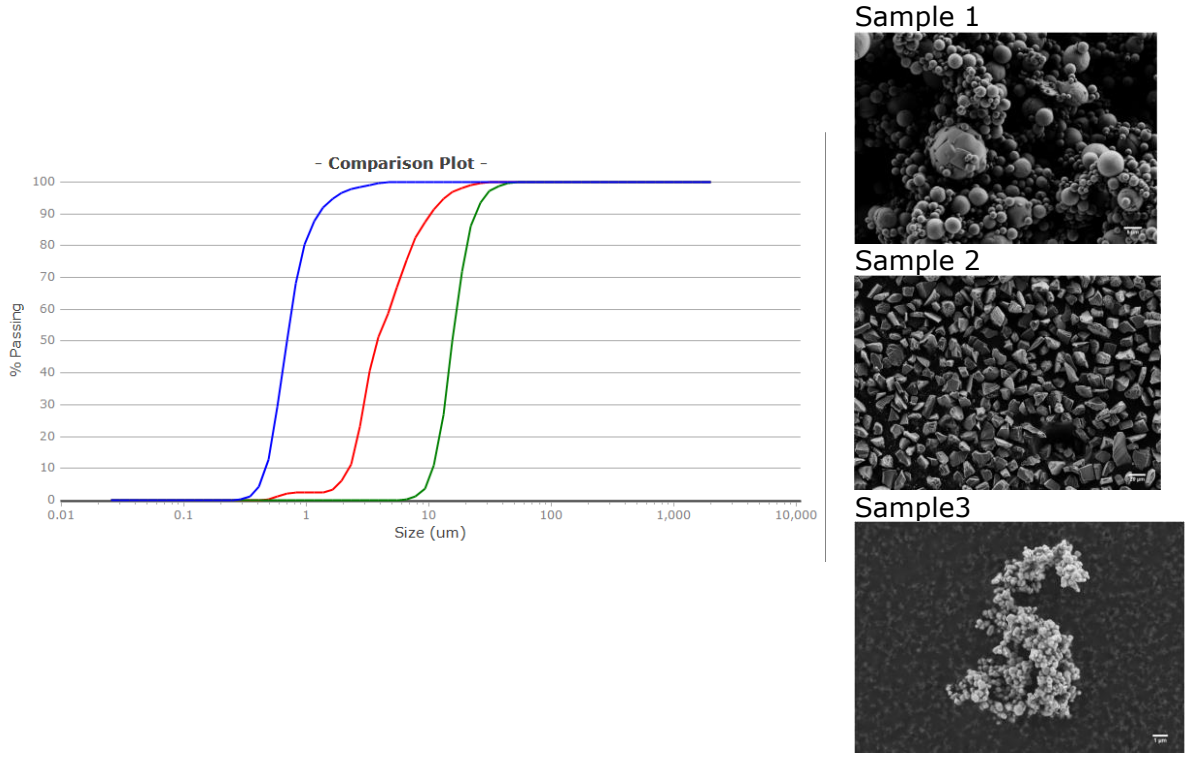

Figure 1: Particle size distributions of sample 1 (red), sample 2 (green) sample 3 (blue). Right side: SEM images of the samples.

Results of the MICROTRAC application laboratories

The figures below show the results of the three MICROTRAC sites for the proficiency test samples.

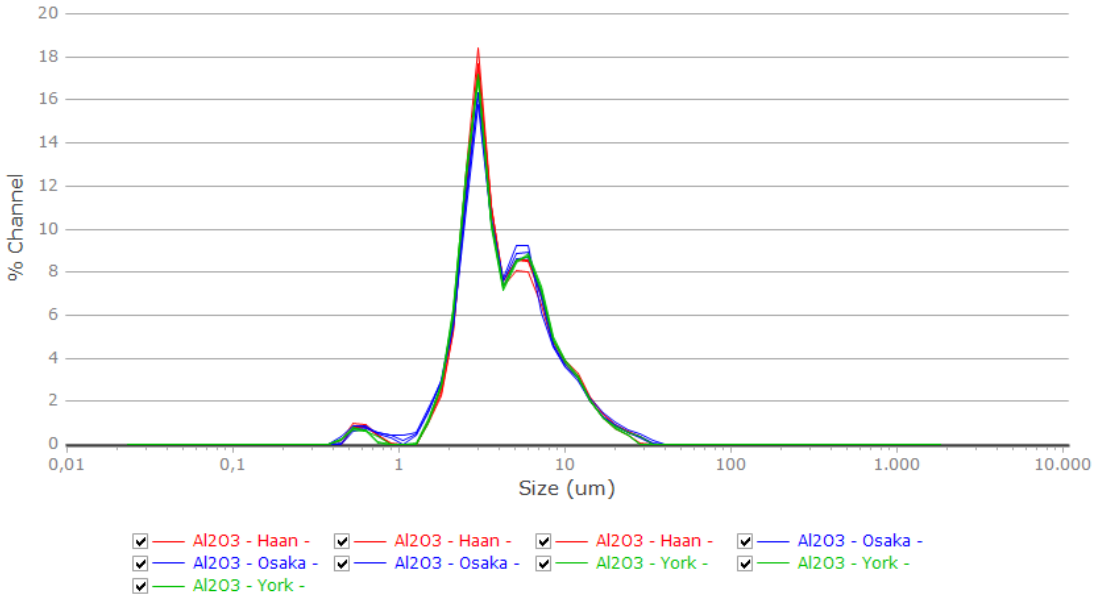

Figure 2: Size distribution of sample 1 (Aluminum Oxide). 3 results from each MICROTRAC lab in Germany (red), Japan (blue) and United States (green).

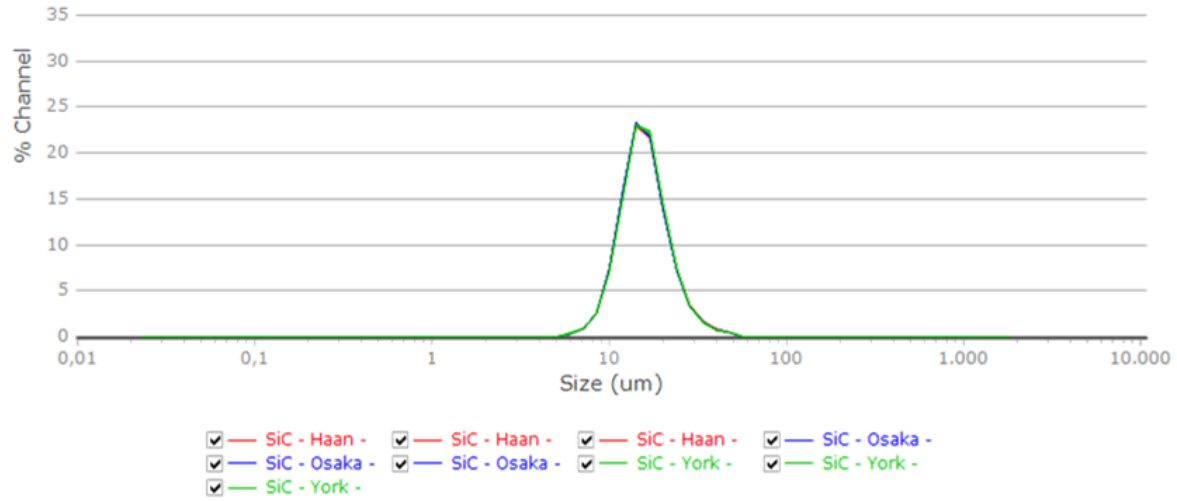

Figure 3: Size distribution of sample 2 (Silicon Carbide). 3 results from each MICROTRAC lab in Germany (red), Japan (blue) and United States (green).

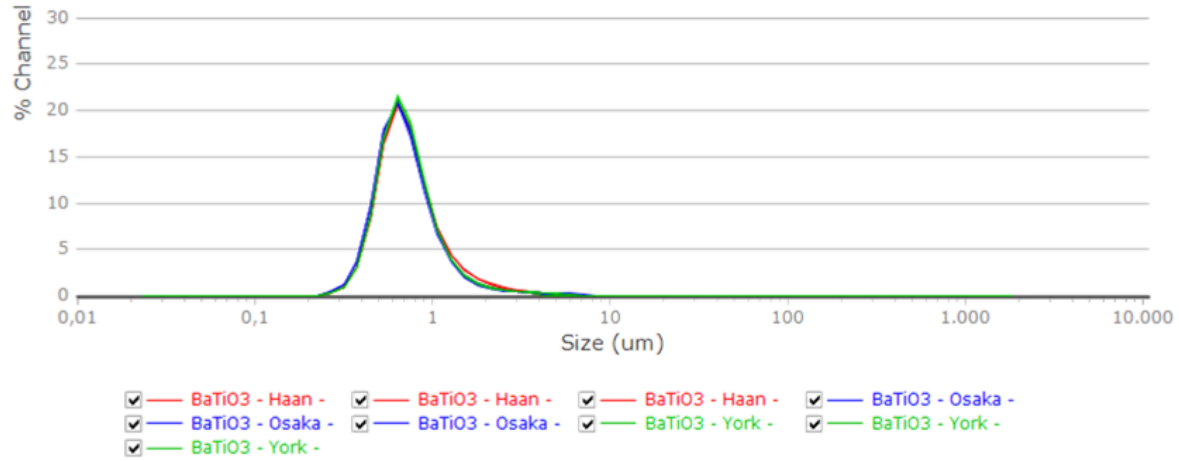

Figure 4: Size distribution of sample 3 (Barium Titanate). 3 results from each MICROTRAC lab in Germany (red), Japan (blue) and United States (green).

Evaluation by BAM

Since the analyzed samples are not certified reference materials (CRM), the interlaboratory comparison corresponds to a comparison of the participants under "intermediate precision conditions" as described in ISO 13320:2020 in point 6.5. From the data of the participating laboratories, an assigned mean and (target) standard deviation was calculated for each characteristic (d10, d50 and d90 of each sample). This was used to compare and evaluate the results of the participating laboratories.

Sample	Characteristic	Assigned Mean Value [µm]	Standard dev. [µm]
P1 (Al_2O_3)	D10	2,103	0,229
P1 (Al_2O_3)	D50	4,296	0,391
P1 (Al_2O_3)	D90	9,561	1,180
P2 (SiC)	D10	10,321	0,467
P2 (SiC)	D50	15,545	0,231
P2 (SiC)	D90	23,160	0,832
P3 ($BaTiO_3$)	D10	0,468	0,085
P3 ($BaTiO_3$)	D50	0,805	0,169
P3 ($BaTiO_3$)	D90	1,536	0,431

From the six analyses of each sample, a mean value of the percentiles d10, d50, d90 was calculated for each participating laboratory. In the next step, a Z-score was determined, which is the difference between the laboratory mean and the assigned mean divided by the target standard deviation. If this Z-score is between -3 and +3, the measurement result is within the acceptable tolerances. Z-scores >+3 or <-3 represent impermissible deviations. Z-scores with a magnitude >2 represent a "warning" signal, but are still within the permissible tolerances.

$$Z - Score = \frac{laboratory\ MEAN - assigned\ MEAN}{Standard\ deviation}$$

The table shows the associated Z-scores for the laboratory mean values of all three sites.

Sample	Characteristic	Germany		Japan		USA	
		Result	Z-Score	Result	Z-Score	Result	Z-Score
P1 (Al_2O_3)	D10 [µm]	2,258	0,68	2,119	0,07	2,228	0,55
P1 (Al_2O_3)	D50 [µm]	3,772	-1,34	3,805	-1,26	3,745	-1,41
P1 (Al_2O_3)	D90 [µm]	10,203	0,54	10,163	0,51	9,895	0,28
P2 (SiC)	D10 [µm]	10,773	0,97	10,787	1,00	10,837	1,10
P2 (SiC)	D50 [µm]	15,557	0,05	15,497	-0,21	15,637	0,40
P2 (SiC)	D90 [µm]	23,703	0,65	23,507	0,42	23,615	0,55
P3 ($BaTiO_3$)	D10 [µm]	0,467	-0,01	0,451	-0,2	0,464	-0,04
P3 ($BaTiO_3$)	D50 [µm]	0,688	-0,69	0,667	-0,81	0,678	-0,75
P3 ($BaTiO_3$)	D90 [µm]	1,253	-0,66	1,131	-0,94	1,168	-0,85

Summary

All three MICROTRAC application laboratories passed the round robin test of the German Federal Institute for Materials Research and Testing with 100% success rate. For each feature considered, the magnitude of the Z-score was below 2, which speaks for an excellent result. This underlines the reliability and performance of MICROTRAC SYNC laser diffraction analyzers.

References:

1. P. Kuchenbecker, F. Lindemann: Bundesanstalt für Materialforschung und -prüfung, Fachbereich 5.5: "Ergebnisbericht zum Ringversuch PT LS BAM-5.5-2021 Bestimmung von Partikelgrößenverteilungen von keramischen Pulvern mittels Laserstreulichtverfahren"