APPLICATION NOTE

MICROTRAC MRB

PARTICLE CHARACTERIZATION

part of VERDER scientific

Guidelines for successful dry measurements with TurboSync

Introduction

For many applications in particle measurement technology, it is advantageous to analyze powders in a dry state:

- If the sample material contains water-soluble components and cannot be measured in an organic solvent for cost or safety reasons.
- If the sample swells in liquid.
- If no suitable liquid can be found as dispersion medium.
- If structures to be measured, e.g., granules, disintegrate in liquids.
- If the sample has a wide size distribution and the small sample quantity used in wet mode does not allow a repeatable result.
- If the material is further processed in dry condition and the results of a wet measurement would not be meaningful.

In dry measurement, a powder sample is dispersed in an air stream and fed to the measuring instrument, in this case the SYNC, laser diffraction analyzer. Good separation of the particles and uniform delivery of not too much and not too little material are critical for a correct result. To achieve this, particle analyzers have dry dispersion modules that use suction and/or compressed air to disperse particles and move them through the measurement zone.

Dry measurements with laser diffraction can usually be performed with particles > 1 µm. Smaller particles can only be efficiently dispersed in an air stream in exceptional cases and agglomerates are often still measured. Even with coarser particles, dispersion (singulation of the particles before measurement) can be difficult. The patented Typhoon accessory for the TurboSync can be used to optimize dispersion conditions for very fine powders. For free-flowing materials such as glass beads or metal powder, setting good measurement parameters is easier than for cohesive materials such as coffee powder. The suitability of a sample for dry measurement therefore depends on the particle size AND the material properties. In any case, various parameters such as sample quantity and sample supply as well as compressed air settings must be checked and determined during the application development. This document is intended to provide a guide on how to successfully find suitable conditions for dry measurement with the TurboSync.

How the TurboSync works

For dry measurements with TurboSync, the specimen is placed on a holder which is inserted into the measuring instrument. Two holders of different sizes exist, which are suitable for different sample quantities. With both sample holders, the powder is evenly distributed in an elongated groove. During the measurement, the sample holder moves at a constant speed under an aspiration tube (Figure 1). A vacuum generated by a connected industrial vacuum cleaner draws the powder into the TurboSync and transports it into the measuring zone. Shortly before it enters the detection zone, additional shear forces can be applied to the sample stream via compressed air, which causes the agglomerates to break up (Figure 2).

The following parameters are within the user's control and must be set up correctly for a successful measurement:

- Compressed air setting "Dispersion"
- Compressed air setting "Suction"
- Sample quantity
- Measuring time (speed of movement of the specimen holder)
- Vacuum (strength of suction)

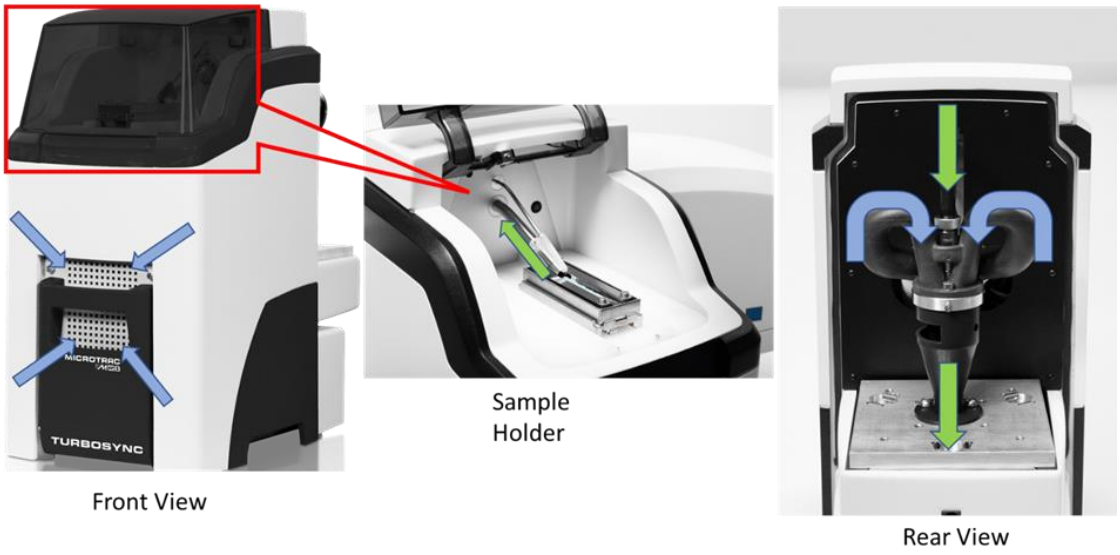


Figure 1: Mode of operation of the TurboSync. Blue arrows indicate the movement of the aspirated ambient air, green arrows show the movement of the sample particles.

Vacuum

The vacuum is generated by an industrial vacuum cleaner connected to the SYNC analyzer. The task of the vacuum cleaner is to generate a constant air flow that transfers the samples from the sample holder to the measuring device. After the sample has passed the measuring zone, it ends up in the vacuum cleaner. Ambient air is supplied through the intake tube and through the square holes on the front of the TurboSync. The air flows through the curved black tubes and creates an enveloping flow around the sample, which prevents particles from leaving the measurement zone and contaminating the inside of the instrument.

Sample Quantity

Sample quantity influences the transmission (loading factor) during the measurement and, to a lesser extent, the quality of the dispersion. With too much sample, multiple scattering can occur or overlapping particles are measured as large particles, producing an unrealistic oversize component in the result. If there is too little sample, the signal-to-noise ratio is poor,

resulting in either very narrow, "truncated" looking distributions, or "ghost peaks" outside the expected size distribution.

Measurement Duration

The measuring time refers to the time required for the specimen grip to pass completely under the aspiration tube once. Permissible values are 10-40 seconds. This setting influences the amount of sample that enters the measuring zone per unit of time. Measuring time is closely related to the "Amount of sample" parameter. Little sample and short measuring time results in similar particle concentration in the analysis as larger sample amount and slow feeding. It is recommended to keep the measuring duration constant and to change the sample quantity during the application development for unknown sample material. A long measuring duration of 40 seconds is advantageous, as homogeneous feeding is more likely to be achieved here. Furthermore, more images will be acquired with longer run time, which make the imaging part of the SYNC analysis more meaningful. If all other parameters are fixed, an attempt can optionally be made to realize a higher sample throughput by shortening the measuring time.

Compressed Air Setting „Dispersion“

The compressed air for dispersion is supplied via the upper of the two hoses above the measuring zone. The air flows at high speed through several holes and hits the sample stream. The holes either go downwards or are slightly inclined so that an air vortex is created. The velocity difference creates shear forces that break up agglomerates. The "dispersion" can be adjusted from 0 – 345 kPa in the SOP. Start with a low value (0 kPa or 20 kPa) and observe the effects on the result when you increase the pressure. If the pressure is too low, agglomerates may persist and show up as coarse particles in the size distribution. If the pressure is increased, these should gradually disappear. Above a certain value, the result remains constant even with increasing pressure. Increase the dispersion pressure in 20 kPa increments. Many samples that flow well can be measured excellently at 0-20 kPa. Most materials do not require more than 100 kPa pressure. Higher pressure is only required for extremely fine and/or cohesive materials. Note that at very high pressure settings, destruction of particles can also occur, e.g. when fragile granules are analyzed.
Attention: The higher the pressure, the more the particles are accelerated! You may have to increase the amount of sample. If the result suddenly changes abruptly, too little sample quantity could be the reason.

Compressed Air Setting „Suction“

The compressed air supply for the "suction pressure" is provided via the lower of the two hoses above the measuring zone. The compressed air passes through a circular gap in which it is compressed and accelerated (Venturi effect). This causes an increase in the suction effect of the vacuum cleaner. The "suction" setting is therefore not primarily used to break up agglomerates, but to improve sample feeding, especially for large particles with a high density. If you observe that the sample is not completely sucked in and the tube "plows" through the powder, increase the suction pressure. Again, small increments of 10 kPa are appropriate.
Caution: The higher the pressure, the more the particles are accelerated! You may have to increase the sample amount. With "suction pressure" this effect is even stronger than with "dispersion".
Higher particle velocity also dramatically reduces the number of particles in the image evaluation.

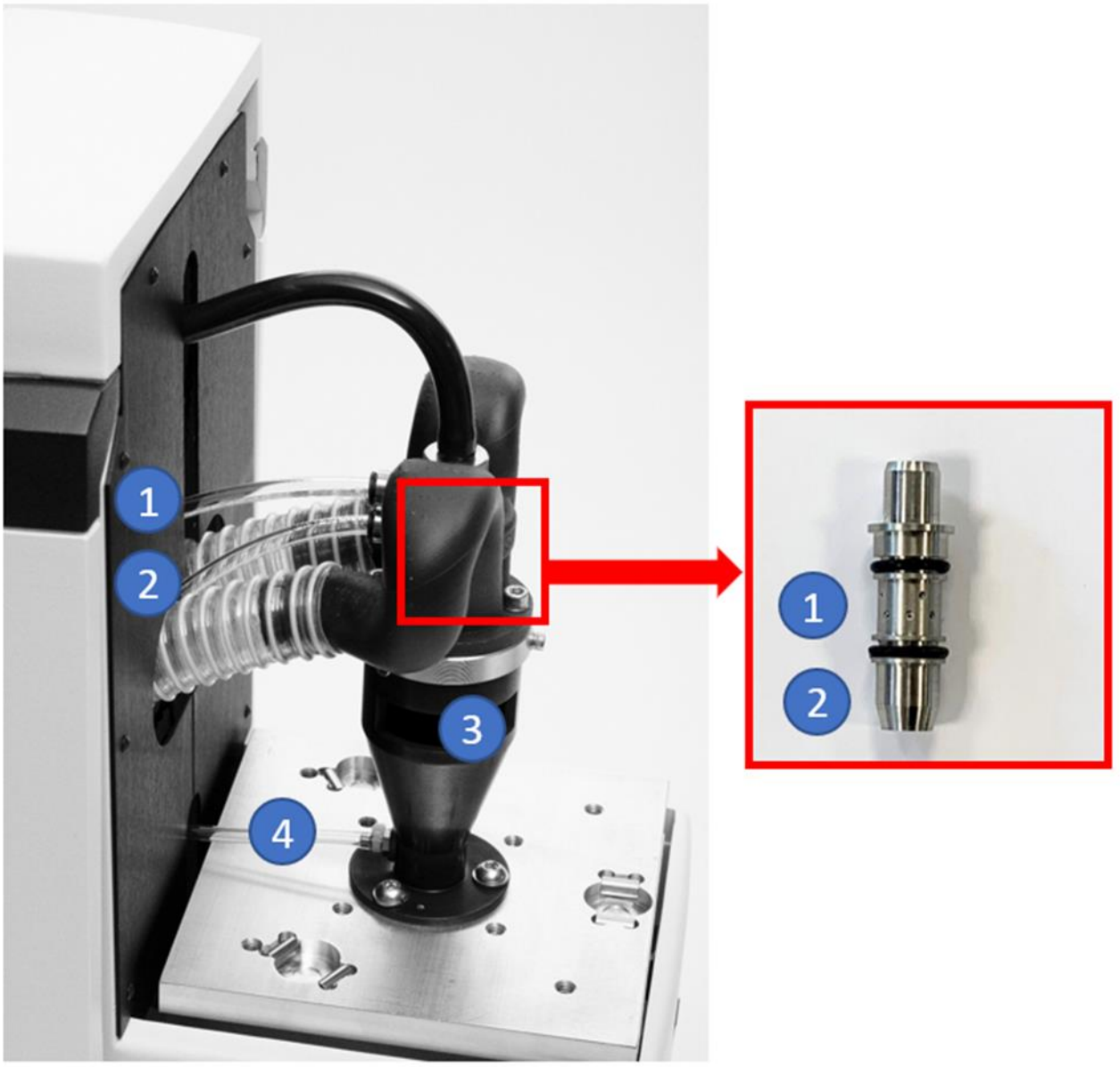


Figure 2: Compressed air supply. 1) dispersion; 2) suction; 3) measuring zone; 4) vacuum sensor. Right picture: Dispersing nozzle removed. The sample flows inside the tube from top to bottom.

Typhoon Accessory

The TYPHOON accessory is available for the TurboSync, which achieves better dispersion, more uniform sample feeding, and thus better results for many applications. This is a cylindrical chamber through which the particle stream is passed before measurement. The swirling motion within the Typhoon improves separation, breakup of agglomerates and creates a constant loading factor during the measurement run. The Typhoon is located between the aspiration tube and the dispersion nozzle.
The Typhoon has an inlet, that is connected to the aspiration tube and an outlet, that is connected to the dispersion nozzle. The cap of the Typhoon is designed for easy access having a bayonet fitting, e.g. for easy cleaning.

Figure 3: Typhoon accessory. Sample inlet (1), sample outlet (2).
Left: closed and locked
Right: shutter cover opened

Installation of the Typhoon accessory

1. Remove the sample feed tube connecting the aspiration tube to the dispersion nozzle. The tube is simply plugged on and can be easily pulled off.

Figure 4: Feed tube removed

2. Cut two pieces of the sample supply hose included in the accessory kit, 8 cm and 3 cm respectively. Supply hose inner diameter 5/16 inch (0.8 cm) / outer diameter 7/16 inch (1.1 cm).

Figure 5: 15 cm tube (for use without Typhoon)
8 cm tube (Typhoon inle)t
3 cm tube (Typhoon outlet)

3. Attach the 8 cm piece to the inlet of the Typhoon and the 3 cm piece to the sample outlet of the Typhoon. The hoses are simply plugged on and do not need any further fastening.

Figure 6: Hoses attached to typhoon

4. Make sure that the tube is correcly mounted and cover roughly 10 mm of the tubing it slides on.

Figure 7: Typhoon installed

Establishing Good Measurement Conditions

The settings required for dry measurement in the FLEX software are presented below.

Vacuum Pressure Monitor

Switch on the SYNC analyzer and install the TurboSync. Start FLEX and initialize the analyzer if necessary. Call up the vacuum pressure monitor via the "Tools | Service | Vacuum Pressure Monitor" menu. The vacuum cleaner switches on and the vacuum pressure is displayed. The value should be approx. 0.25 psi. Use the vacuum cleaner's vacuum pressure regulator to set the desired value. If the vacuum does not have a regulator, open or close the gap on the elbow piece on the back of the SYNC. Note that the quality of the vacuum depends on the condition of the aspirator (filter, bag filling) and should be checked regularly.

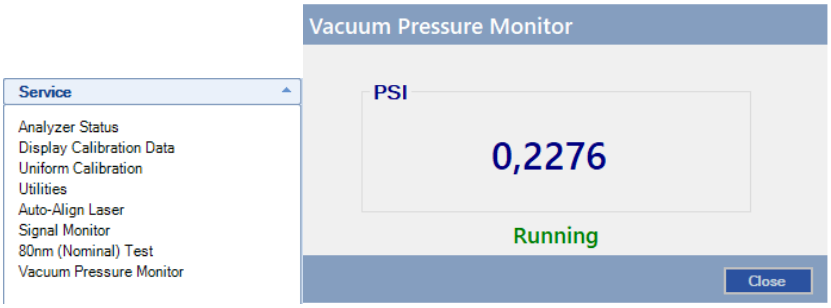


Figure 8: Vacuum Pressure Monitor

Timing

Open the "Sample Measurement | SOP | Measurement SOP" menu. In the "Measurement SOP" window, click the "Options" button to change the parameters for the next measurement.
In the "**Timing**" tab, set the time that the sample holder needs for one run. Start with the maximum value of 40 seconds. For the "SetZero" (background measurement), 15 seconds is sufficient.
If you want to perform several measurements of the same sample with the same settings in succession, increase the value at "Number of measurements". After completing the first measurement, you will be prompted directly to load the sample holder again. You will receive as many results as you have specified in "Number of measurements".
With the "**Dry Multi-Scan**" function, you can combine several runs of the specimen holder for one analysis. If the check box is deactivated, only one run is used at a time and one measurement result is generated. If the check box is activated, as many sample trays are measured as specified in "Scans". After each run, you will be prompted to reload the specimen holder.
Example:
Number of measurements = 3
Dry Multi-Scan = 2 and "activated".
The SYNC analyzer makes 3 measurements (3 results), each result is based on 2 runs of the sample holder. Thus, you have to load the holder 6x in total. While the measurement sequence is running, follow the instructions on the screen, you will be notified when you need to add new sample.

Suction and Dispersion

Open the "Sample Measurement | SOP | Measurement SOP" menu. In the "Measurement SOP" window, click the "Options" button to change the parameters for the next measurement.
In the "**TurboSync**" tab, you set the values for "Dispersion" and "Suction pressure". The setting is in psi, a conversion to kPa is displayed. Start with low values as described above and increase in small steps as the sample material requires.
SUCTION:
When working **without Typhoon**, the value for Suction can usually be left at 0 kPa. Only if very large and/or heavy particles are analyzed, it may be that the aspiration of the vacuum cleaner is not sufficient. If you observe that the aspirating tube plows through the powder rather than lifting it from the holder and sucking it in, you should increase the Suction gradually in 20 kPa steps.
If you are working **with Typhoon**, there should always be a minimum of 20 kPa set for the Suction.
DISPERSION:
Free flowable samples like glass beads and many metal powders do not require dispersion in many cases and you can leave the setting at 0 kPa. For more agglomerating samples, start with low dispersion pressure and gradually increase in 20 kPa steps. If the agglomerates are broken up with increasing pressure, you will observe that the result is getting finer. At some point you will see no further change in the result when you increase pressure, which means that you have achieved the maximum possible sample dispersion and you have found the optimum pressure settings for your sample.

AutoClean

We strongly recommend to activate the AutoClean function after EVERY dry measurement. During the AutoClean process, pressurized air is blown into the aspiration tube, removing all sample residues. This avoids cross-contamination and is especially important when you are using the Typhoon accessory!

Making a Dry Measurement

Once the settings have been defined in the SOP, you only need to apply the sample to the holder, distribute it as evenly as possible, insert the holder into the TurboSync and close the cover flap. The measurement will not start if the flap is open.
In general, the larger the particles and the wider the distribution, the more sample should be analyzed to improve measurement statistics and repeatability. It is recommended to start rather with a small amount of sample and increase as needed. Some experience in dry measurement is an advantage here, Table 1 is intended to give an approximate indication of how much sample to start with (The example is for glass powder, for high density samples, use correspondingly more). The examples later in the document can also be helpful here.

Note that if the **Typhoon** accessory is used, you may be able to apply more sample without overloading the system. Without the Typhoon accessory sample amount needs to be adjusted more carefully.

Particle Size	Sample Quantity	Number of Scans	Holder
< 10 µm	50 - 100 mg	1 Scan	Small
< 100 µm	100 - 500 mg	1 Scan	Small
< 1000 µm	1000 mg	Multi-Scan (2-3 x)	Large

Table 1: Recommended sample quantity

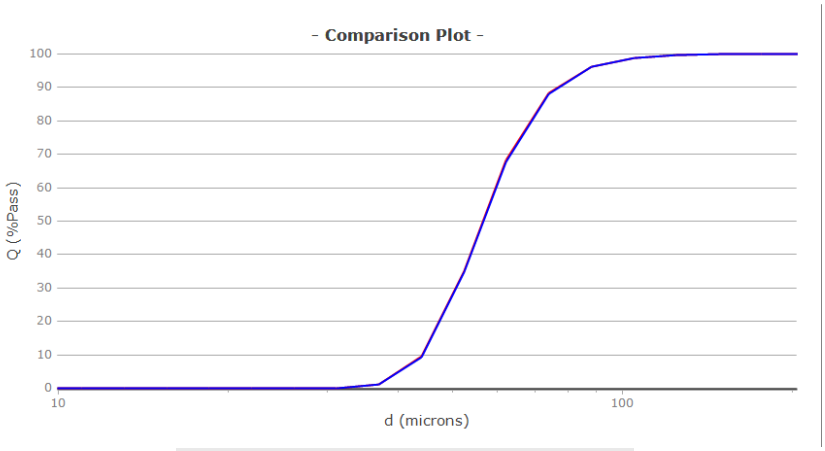
Now click on "Start Auto Sequence" in the live measurement window of Flex. The vacuum and compressed air are starting first and a Setzero measurement is performed. Then the sample holder moves at the set speed. When the run is complete, the measurement result is presented, or a prompt appears to load the sample tray again if working with multi-scan or multiple measurement.

Examples

In the following, some measurement examples of samples with different particle sizes are discussed. As a reference for the evaluation of the dry measurement, wet measurements of the same material with the SYNC are used or dry measurements with the CAMSIZER X2 image analyzer.

Glass 1

Glass 1 is a standard recommended by Microtrac for evaluating the condition and performance of the gauge. It is a very uniform material (narrow distribution) with a median of 56 - 60 µm. Figure 4 shows two dry measurements of Glass1, once with 100 mg and once 500 mg sample. The material has very good flowability and does not require compressed air for dispersion. The d10/d50/d90 values are identical for both sample quantities and are within the size ranges given for the standard. The analysis can be made with or without Typhoon.



Data Item	1	2
MV (um)	58.76	58.91
CS	1.07e-1	1.07e-1
SD	11.94	12.04
d(10)(%)	44.22	44.23
d(50)(%)	56.43	56.54
d(90)(%)	75.72	76.03

Figure 9: Results of dry measurements of "Glass1" with 100 mg and 500 mg sample.

Fine Glass Powder

Sample 410.6843 is a fine glass powder (Figure 5) with a size distribution of about 10-100 µm. The sample is not free-flowing and tends to form agglomerates. First, the sample was measured dry with the following settings:
Measuring time = 40 seconds
"Dispersion" = 0 kPa
"Suction pressure" = 0 kPa
Sample quantity = 500 mg

Figure 10: Fine glass powder on the sample holder (approx. 100 mg)

The result shows a coarse fraction > 100 µm, which is not present in the sample. In the next test, the sample quantity was reduced to 100 mg. The coarse fraction is still present, but significantly lower. In the next step, the "dispersion" was increased to 10 kPa, resulting in no particles >100 µm for the same sample amount of 100 mg. The result now fits very well with the result of the wet measurement (Figure 6).

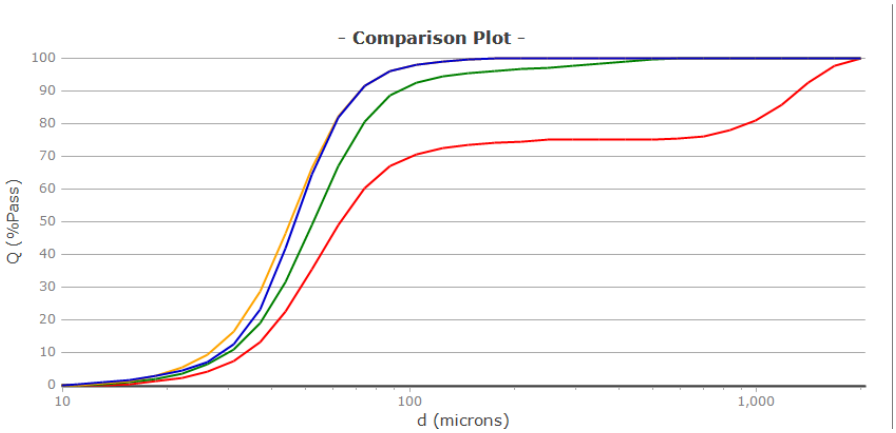


*Figure 11: Particle size distribution of glass powder 410.6843. Dry measurement 500mg, 0kPa (**red**), dry measurement 100 mg, 0kPa (**green**), dry measurement 100mg, 10kPa (**orange**), wet measurement (**blue**). The yellow and blue curves are almost on top of each other.*

A further increase of the pressure is not advisable, since this accelerates the particles, and the signal/noise ratio becomes worse. At 100 kPa, the result is still comparable to 10 kPa and wet measurement, but at 200 kPa ghost peaks appear in the coarse and fine ranges (Figure 7). Here, the noise is misinterpreted as a measurement signal (fit-to-noise). This can also be seen in the raw data (low intensity, discontinuous pattern, Figure 8). Another interpretation for suddenly occurring oversize is that the particle stream is compressed to a narrower space at high pressure and thus superpositions can occur in the measurement zone.

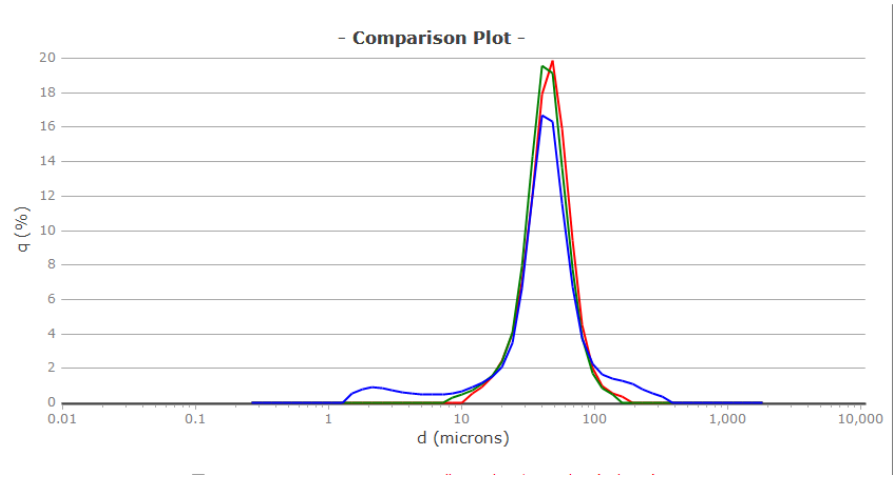


*Figure 12: Dry measurement of glass powder 410.6843, 100 mg each. The results at a dispersion pressure of 10 kPa (**red**) and 100 kPa (**green**) are still well comparable, at 200 kPa (**blue**) "ghost peaks" appear.*

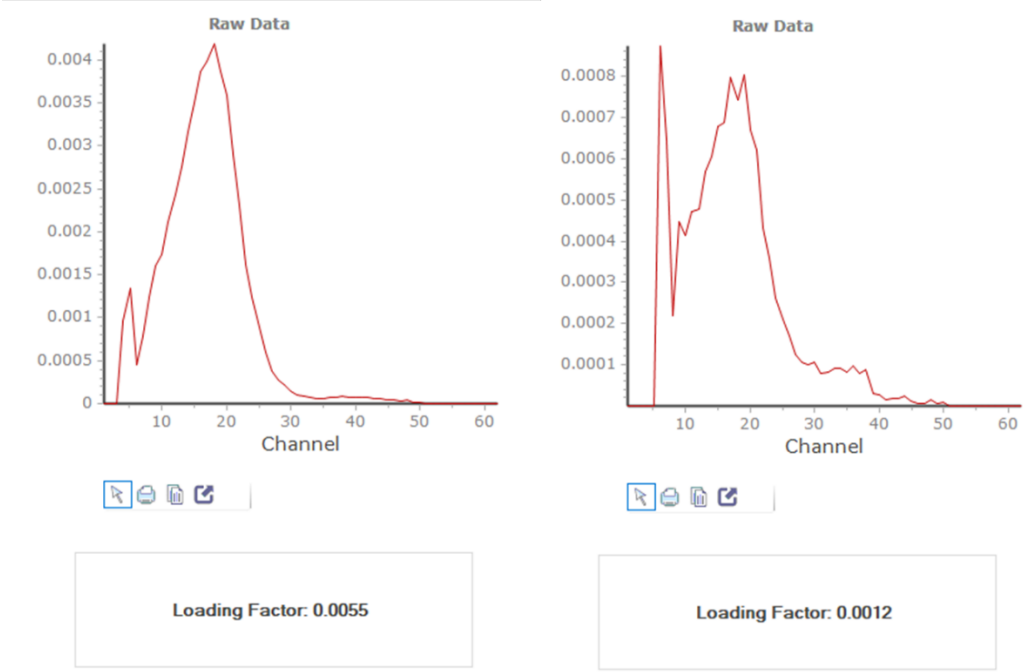


Figure 13 Comparison of the raw data of the measurements of glass powder 410.6843 at 10 kPa (left) and 200 kPa (right). The curve at 10 kPa is more uniform, the signal strength and the loading factor are higher.

Fine SiC (Silicon Carbide) Powder

Figure 9 shows the comparison of a dry measurement and a wet measurement of a fine SiC sample "F800" with a median of 6 µm. The results are very well comparable. For all dry measurements 100 mg material was used.
At 0 kPa dispersion pressure, a coarse fraction in the range 10 - 100 µm is still measured. Already at 10 kPa, the dispersion effect is sufficient to achieve perfect comparability with the wet measurement.

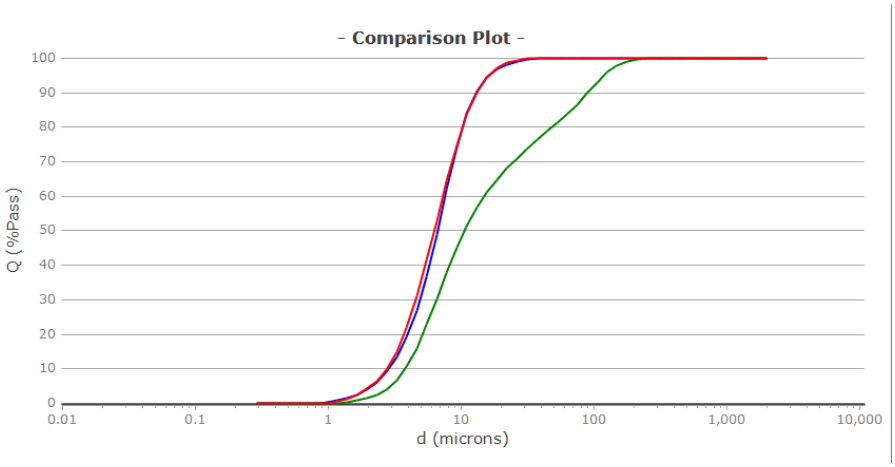


*Figure 14: Measuring results of the SiC specimen F800. Dry measurement 0 kPa (**green**), dry measurement 100 kPa (**red**) and wet measurement (**blue**).*

Very Fine SiC Powder

The SiC "WM" sample is even finer than the one in the previous example. The median is about 2.5 µm. This is one example where a dispersion pressure of 100 kPa is required. Agglomerates are still measured at 10 kPa and 50 kPa, and very good comparability with the wet measurement is obtained at 100 kPa (Figure 10). For the dry measurements, 40 mg of sample was used in each case.

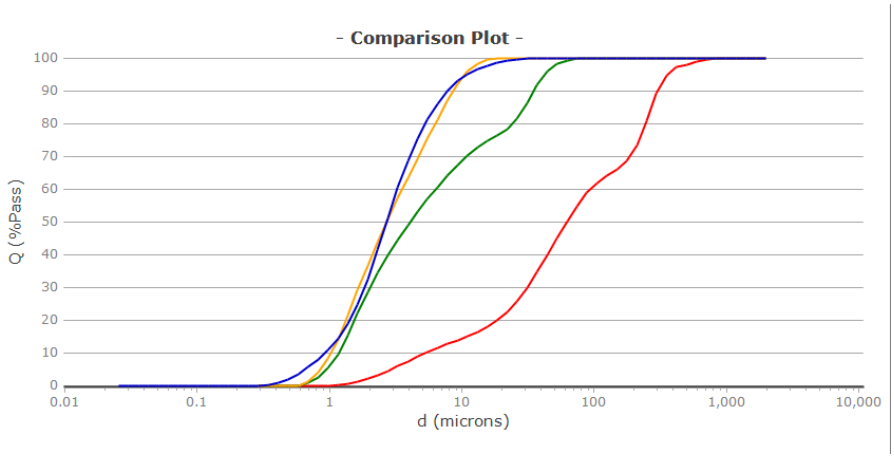


*Figure 15: Measurement results of the SiC sample "WM". Dispersion pressure 10 kPa (**red**). 50 kPa (**green**), 100 kPa (**orange**) and wet measurement (**blue**).*

Fine Plastic Powder

For the plastic powder in this example, the good reproducibility of the wet and dry measurements is shown (Figure 11). For the dry measurements, 100 mg of material and 20 kPa dispersion pressure was used. The wet measurements were performed in ethanol.

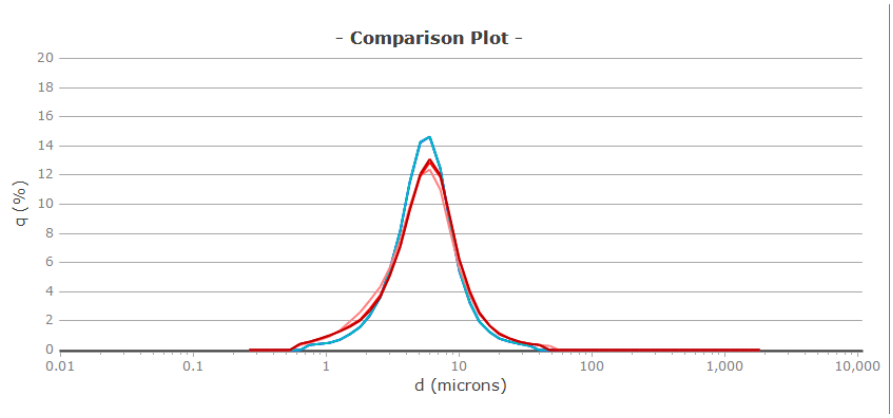


*Figure 16: Particle size distribution of a plastic powder. The median is 5.5 µm. Three wet measurements (**blue**) and three dry measurements are shown.*

Coarse Plastic powder

The coarse plastic powder in this example is characterized by a broad size distribution from 10 µm to just over 1000 µm. The sample quantity required for a measurement is correspondingly larger. In this case, 800 mg was used for one run. It is recommended to use several runs for one analysis to improve reproducibility. This is easily done with the Dry Multi Scan function. The example shows three dry measurements and one wet measurement. For the dry measurements, the setting "Dispersion = 0 kPa" was used (Figure 12). The air flow generated by the vacuum cleaner is sufficient to separate the particles.

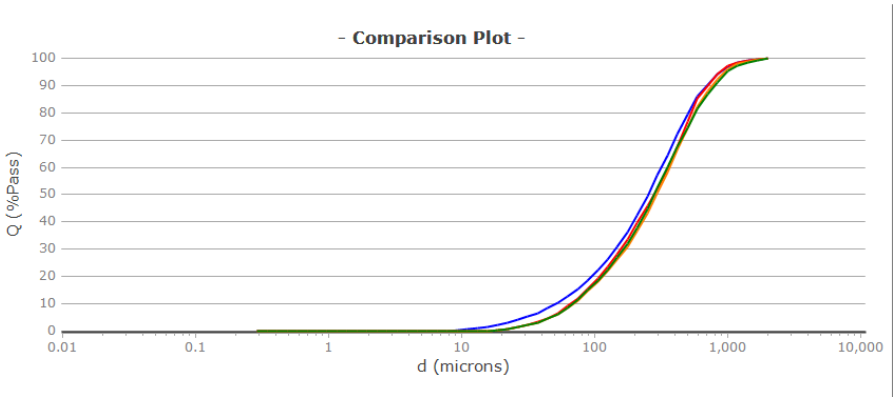


*Figure 17: Three dry measurements of the coarse plastic powder (red / **green** / **orange**) and comparison with the wet measurement (**blue**)*

Coffee Powder

Coffee powder presents a particular challenge. The particle size is measurable with usually 10-1000 µm with laser diffraction, but the resolution of the method gets worse for coarse particles. The biggest problems, however, are caused by the high fat content of the powder (about 15%), which makes the powder very sticky and cohesive and therefore makes it difficult to convey and to disperse. The coarse particles in the sample and the wide distribution require the analysis of relatively large amounts of powder and greater energy input during dispersion. For good measurements with sufficient signal quality and repeatability, the following settings were selected:

- 600 mg sample per measurement run
- Large sample holder
- Multi-scan=3 (alternatively 3 passes followed by calculation of an average)
- Dispersing pressure = 100 kPa, suction = 0 kPa
- Measuring time: 20 seconds

Note the shorter measurement time, which can be an advantage for coarser samples to generate enough signal. Figure 14 shows the mean value from three measurements of a relatively fine coffee powder sample. The percentile values were compared with the results of the CAMSIZER X2 image analysis system (Table 2). The data show good agreement. Thus, the analysis of coffee powder with laser diffraction is feasible. Nevertheless, pure dynamic image analysis with the CAMSIZER X2 has advantages here, since better reproducibility is achieved by analyzing larger quantities of powder. In addition, the resolution and accuracy of the method are significantly better for particles of several 100 micrometers.

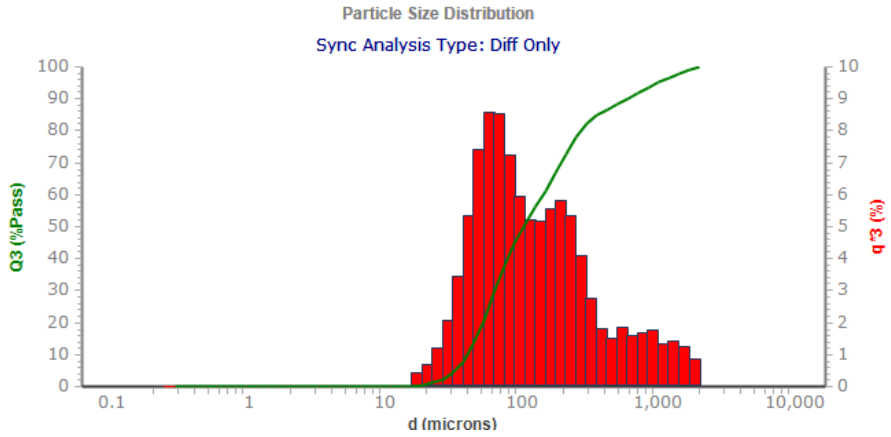


Figure 18: Particle size distribution of a coffee powder sample measured with the SYNC laser diffraction analyzer in dry mode.

	SYNC Laser Diffraction	CAMSIZER X2 Image Analysis
D10	40 µm	30 µm
D50	104 µm	117 µm
D90	660 µm	685 µm

Table 2: Comparison of percentile values of a coffee powder sample from SYNC and CAMSIZER X2

Measurements with Typhoon

Glass powder < 20 µm

A glass powder with a particle size < 20 µm was analyzed in dry mode with and without the Typhoon accessory. With 20 kPa dispersion pressure, the result is finer with the Typhoon, compared to the runs without Typhoon at both 20 and 345 kPa. This shows that the Typhoon can efficiently improve particle dispersion even at low pressure. The dry result with Typhoon has been confirmed by wet analysis.

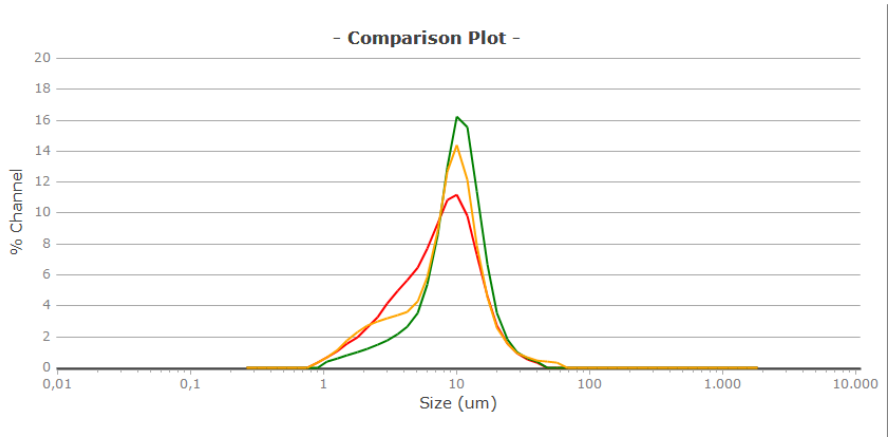


*Figure 19: Dry run of < 20 µm glass powder. With typhoon suction 20 kPa and dispersion 20 kpa (**red**)*
*Without Typhoon, 20 kPa suction and 20 kPa dispersion (**green**)*
*Without Typhoon 200 kPa suction and 350 kPa dispersion (**orange**)*

Coal dust

A coal dust sample was analyzed in wet mode (ethanol) and dry mode. If the Typhoon accessory is used, the result at 20 kPa is coarser than the wet measurement. At 200 kPa pressure, there is a good agreement between wet and dry analysis. Without the Tyhoon, the result at 200 kPa is still larger than in wet mode.

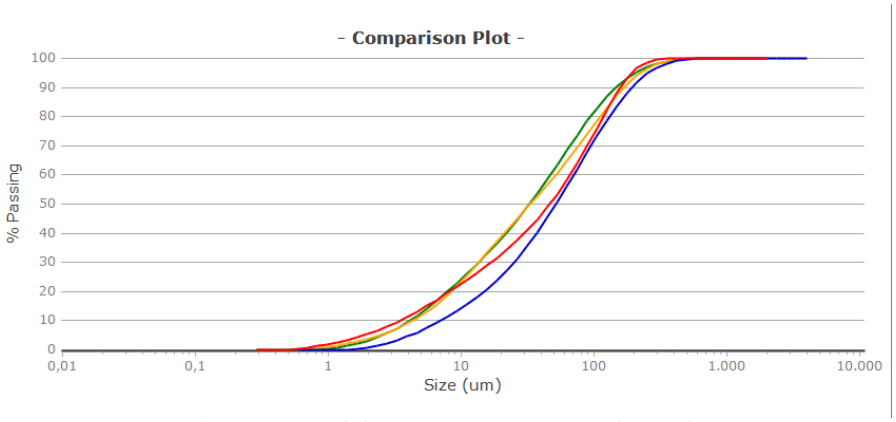


*Figure 20: Coal dust wet measurement (**green**)*
*Dry with Typhoon 20 kpa (**blue**)*
*Dry with Typhoon 200 kPa (**orange**)*
*Dry without Typhoon 200 kPa (**red**)*

Alumina

An alumina sample with a nominal size of 2 µm (median) was analyzed in dry mode with and without the Typhoon accessory. The results without Typhoon show large agglomerates. With the Typhoon accessory it is possible to achieve good dispersion and to obtain the correct result. This alumina sample can not be masured dry without Typhoon.

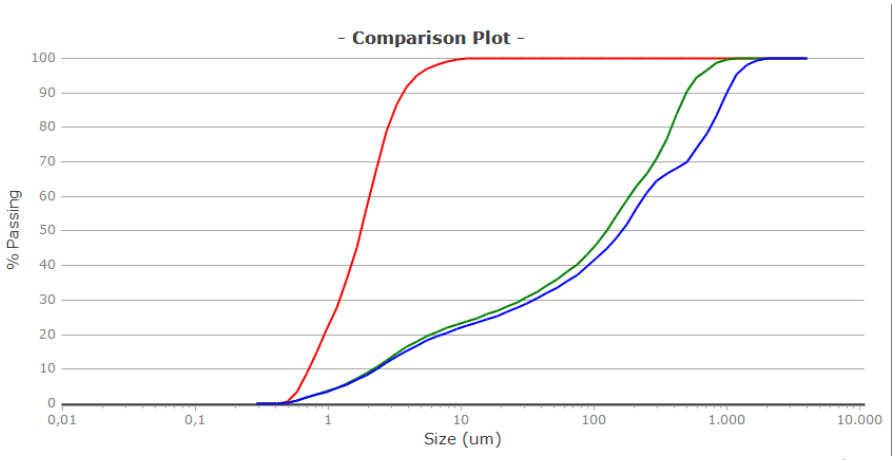


*Figure 21: Two failed attempt to analyze the alumina sample in dry mide (**blue** / **green**). With Typhoon, the result is correct and all agglomerates are broken up (**red**).*

Summary

With the SYNC laser diffraction analyzer and the TurboSync dispersion module, size distributions of powders and granulates can be determined quickly and easily. For unknown materials, some tests are necessary during the application development. Sample quantity, measuring speed and dispersion pressure are the most important parameters.
It is recommended to start with slow speed, low pressure and little sample and increase if necessary.
The Typhoon accessory simplifies handling and allows the analysis of difficult materials.

For more information visit us at www.microtrac.com