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In-line Particle Size Analysis in the Fines Outlet of an Air Classifier

1 Introduction

Laser diffraction (LD), with reference to the analysis time, is highly advantageous for the automation of particle size analysis. The high measuring frequencies allow a quasi continuous measuring operation. The fineness values obtained from the particle size distribution (PSD) can be transformed into electrical signals for automatic process control.

1.1 Start Situation

As product fineness is monitored continuously in-stream and virtually without delay, any changes in the fineness during classifier operation can be detected immediately. Although deflector wheel classifiers are well known for their stable operation over long periods, in-line particle size analysis enables detection of short-term fluctuations in the product fineness. On the one hand, this opens up a new field for studies of classifier performance. On the other hand, immediate quality control of even very small batches is possible during the micronisation process. The results demonstrate very clearly, for example, the importance of controlling the feed rate to the classifier. Therefore, fineness control by means of this in-line technique gives superior advantages for direct adjustment of the classifier speed.

In addition a main advantage of in-line measurements if done as described here is that no sample splitting is necessary.

1.2 Aims of in-line measurement

- better product quality within more narrow specification limits and higher product homogeneity.
- better yield because of direct control during start-up and shut-down periods.
- elimination of influences by means of direct intervention (manually or automatically). Influences caused by changes of process parameters, changes of quality and grinding behaviour of the feed material.
- process optimisation during test runs, quick and easy finding of optimal parameter settings for micronisation saves time and product (=costs) during process development.

The ultimate aim of the development is a closed loop control of product fineness in terms of a selected point of the PSD such as x_{50}.

2 Development of the in-line measuring cell

The first step was to check whether the concentration of particles leaving the mill via the classifier meets the requirements of the laser diffraction analyser. For this feasibility study, an open measuring zone was used. The first measurements showed immediately that nearly every configuration of process parameters produced concentrations within the normal measuring range of laser diffraction analysers. The relevant sorts of concentration for these kind of instruments are optical concentrations C_{opt} that are related to the transmission T, which is the ratio of intensities of the undiffracted beam with, I, and without, I_o, particles:

$$T = I/I_o \qquad \text{equation 1}$$

$$C_{opt} = 1 - T = 1 - I/I_o \qquad \text{equation 2}$$

C_{opt} depends strongly on the particle size distribution and its content of fines. For laboratory use, the optical concentration should not exceed 25%, but for in-line use, the region can be extended up to 80%, because the main interest of in-line measurements is the detection of changes in PSD. The shift of PSD to fines at high concentrations caused by multiple scattering is negligible compared to changes of the process.

2.1 Performance of in-line systems

2.1.1 Measuring apparatus

Important prerequisites that make a measuring instrument suitable for in-line operation are (see especially Leschonski /1/2/ and Polke /3/4/):

- high speed
- high reproducibility
- accuracy
- complete automisation

Modern laser diffraction instruments such as the HELOS fulfil these requirements in all points very well.

2.1.2 Measuring zone

The measuring zone is the area where the process and the instrument interact. The laser beam penetrates the dispersed, flowing product stream coming from the classifier. For both process and instrument certain requirements have to be fulfilled:

- complete encapsulation, with the consequence of using glass windows, suitable for
  - pharmaceutical products in terms of toxicity
  - process pressure control, and
  - explosion-proof systems (special design)
- continuous processing over more than 8 hours without interruption.
- all features automatically driven (remote control)

2.2 Design and construction of the measuring zone

The construction of the measuring zone was a compromise between

1. on the one hand keeping the windows as far away as possible from the particles to protect them from contamination, and
2. on the other hand keeping the flow rate of the rinsing air as low as possible, because this has an influence on the size of all downstream installations such as cyclone, filter and blower. Because mostly compressed air or gas will be used for rinsing (operational costs), it was important to find a good compromise.

The first approach was a redesign of the well-proven set-up for dry measurements: The particle-laden air/gas stream is about 60mm away from lens and window, which are protected by a sheath flow induced by the injector and the suction air flow. Both the particle laden air flow and the sheath flow are trapped by a exhaust-nozzle. As a consequence, this set-up, which was used during the feasibility study, had to be encapsulated. This new arrangement, therefore, was characterised by bi-directional sheath flow.

First measurements with this bi-directional sheath flow zone showed good PSD results but the time to window contamination was unsatisfactory. This is in opposition to the experiences of measuring with the dry dispersing system, where the windows are in the same position. Further development was necessary.

2.2.1 Flow-field calculations

We started with calculations of flow fields using the method of finite elements. The 1-phase flow calculations were conducted by the R&D department of HOSOKAWA Alpine.

The results showed the reason for the short time to window contamination, see Figure 1: High turbulence and broad turbulence zones in the measuring region combined with slow air velocities near the windows. A closer look at the flow-field situation discloses decisive differences to the off-line set-up. The situation behind a mill is significantly different. Especially the amount of particle laden air flow is more than ten times that of the standard dry disperser. This also means that the flow ratio to the sheath flow is ten times higher. The expected influence of the exhaust-nozzle on the flow contraction only occurs very close to the nozzle. The flow within the measuring zone is governed by the stream bursting open.

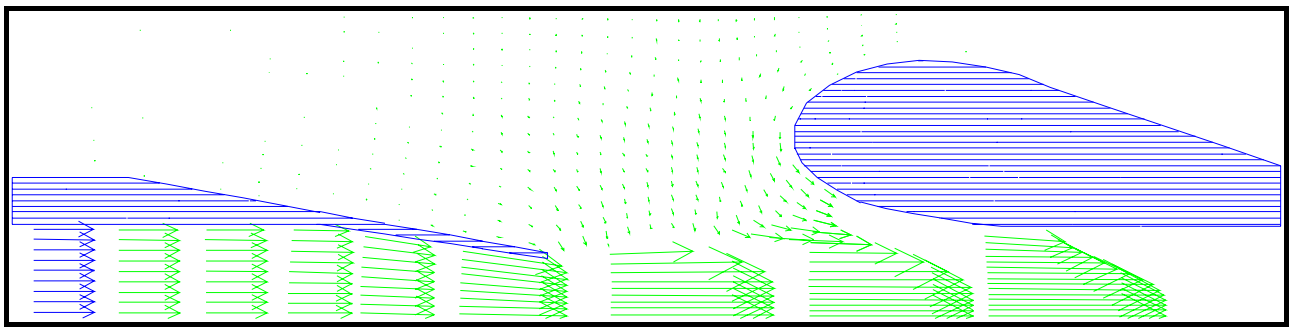


Figure 1: Calculated flow field within a measuring cell with bi-directional sheath flow

This knowledge led to a different construction: A cell with mono-directional sheath flow , which also has the advantage being easier to control and easier to build. The results of flow field calculations immediately showed the superiority of mono-directional flows as shown in Figure 2 and Figure 3. High rectified velocities near the windows and small turbulence zones around the particle-laden stream gave reasonable expectations for long intervals before the windows need cleaning.

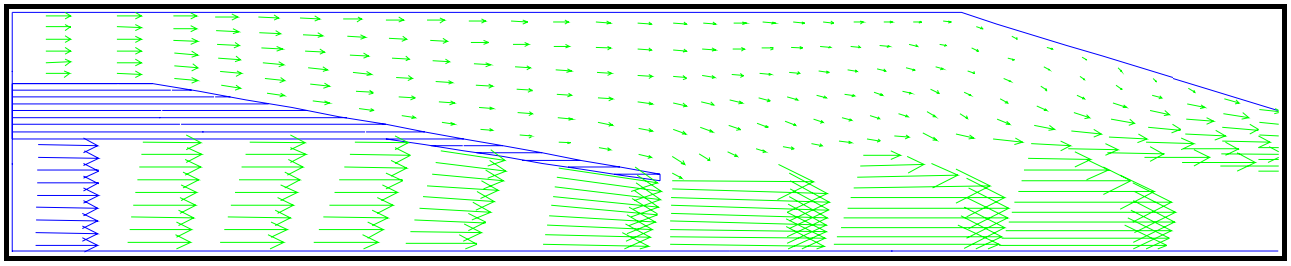


Figure 2: Calculated flow field within a measuring cell with mono-directional sheath flow with ∅ = 80mm: broad geometry.

With respect to the importance of the Magnitude of volume flow for all following unit operations as mentioned above, smaller cell diameters, which means smaller volume sheath flow, had to be tested. Figure 3 shows the flow field within the 60mm cell.

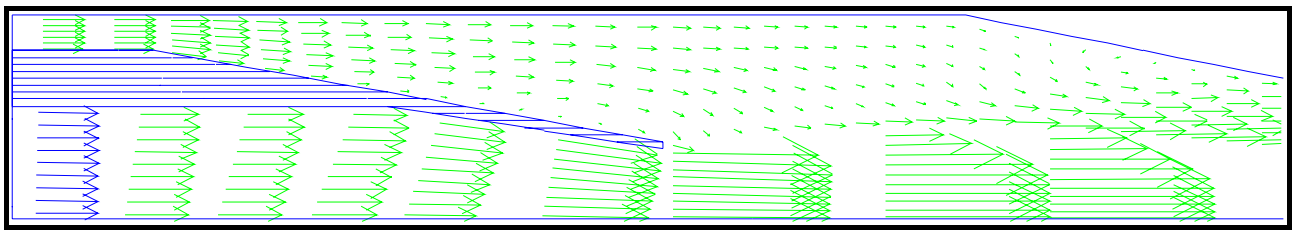


Figure 3: Calculated flow field within a measuring cell with mono-directional sheath flow with ∅ = 60mm: smaller geometry.

The situation within the smaller cell is not principally different, but it halves the volume of sheath flow. The calculation of both showed the flow-contraction and the low-level bursting of the particle-laden stream, which is even better at the smaller cell.

2.2.2 Technical realisation

The calculations were only able to give an initial idea. The different geometries had to be tested in action. Many tests with different geometries were performed. The criterion of judging the function was always the time to window contamination.

It turned out that a diameter of 60mm extended the time to window contamination considerably. This seemed to be a good compromise, and complete measuring cells with this geometry have been built.

The technical realisation as the standard type is shown in Figure 4, which is a very simple set-up constructed for easy dismantling, e.g. for cleaning. The cell for pharmaceutical use, shown in Figure 5 is prepared for CIP precleaning (CIP = cleaning-in-place), i.e. the complete cell can be flooded with a cleaning liquid. Two

draining pipes are connected at the lowest points of the cell, in Figure 5 drawn twisted through 90°. The inside geometry is exactly the same as in the standard cell.

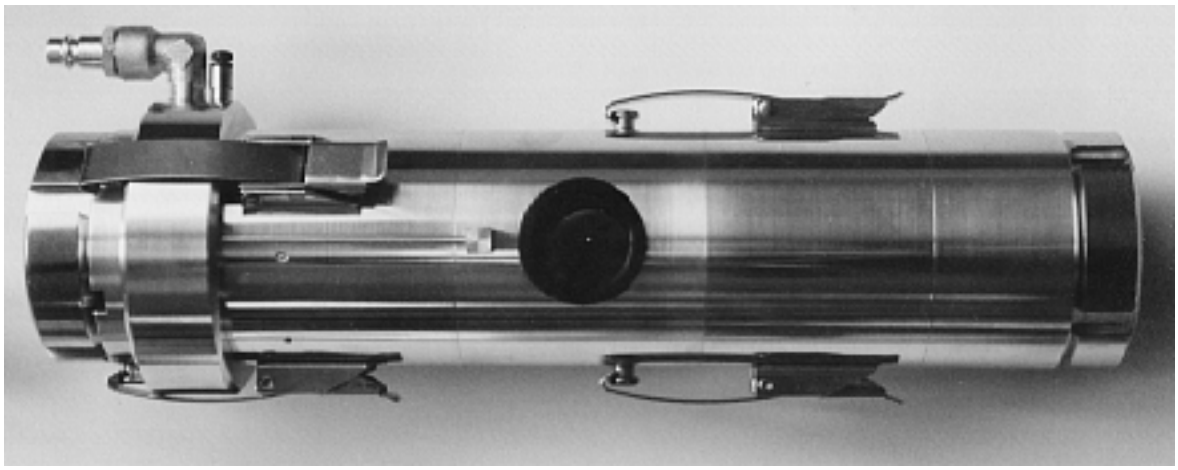
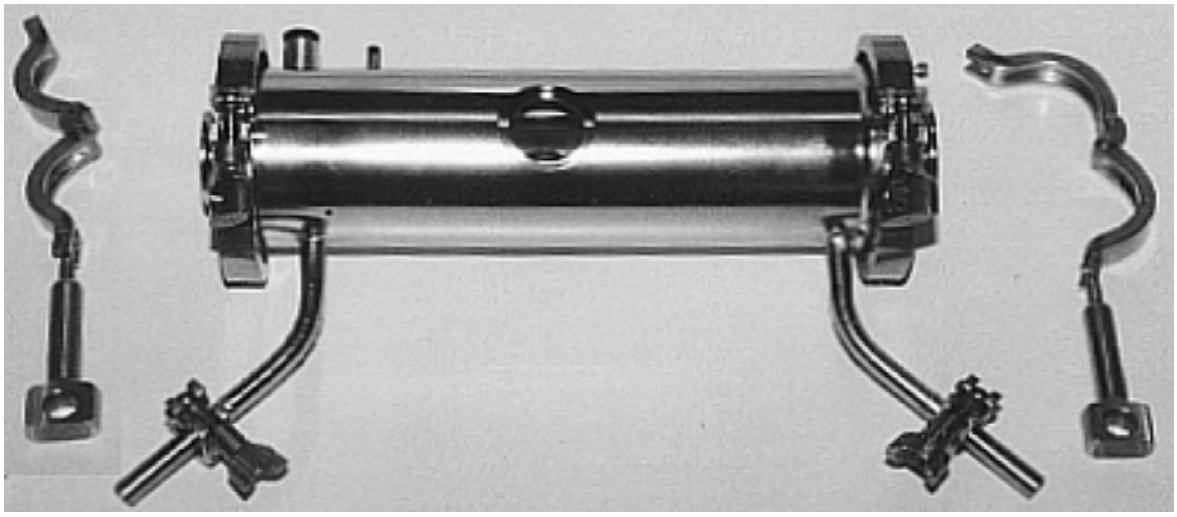


Figure 4: In-line measuring cell: Standard type.



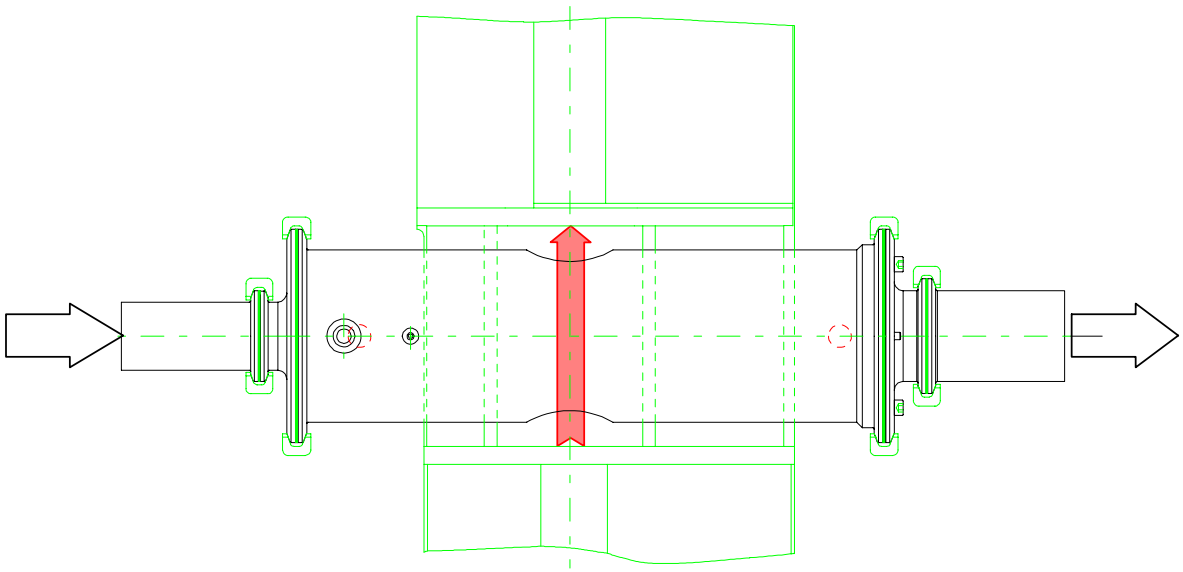


Figure 5: In-line measuring cell: CIP type for pharmaceutical and food applications.

All results presented in the following chapter were gained from experiments using this geometry of the measuring cell, in its standard design.

3 In-line results

Figure 6 shows the arrangement used, which in this case was completely prepared for CIP and included a fluidised bed jet mill 100 AFG, which implies a milling chamber of 100mm diameter and a classifier diameter of 50mm. From left to right, Figure 6 shows a computer driving the instrument, jet mill with feeder, laser diffraction analyser, pressure control box, tube switch, cyclone, process control panel and filter l

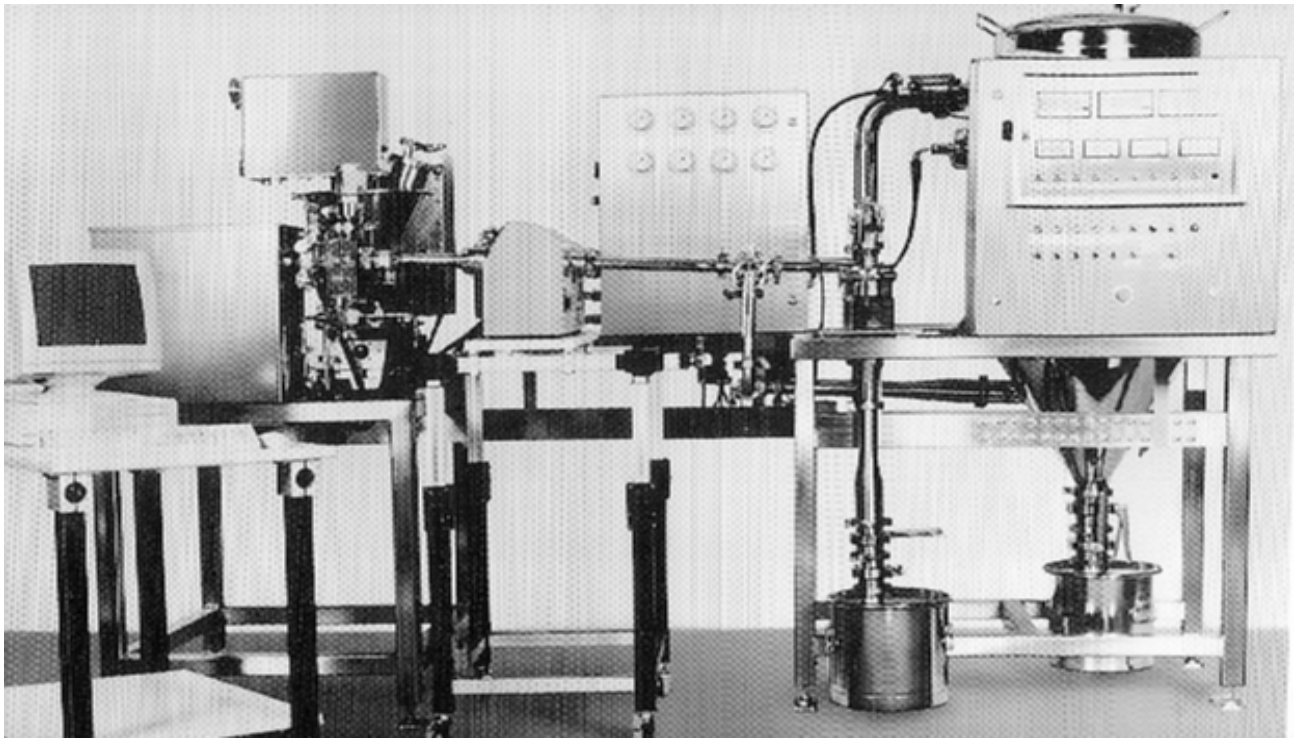
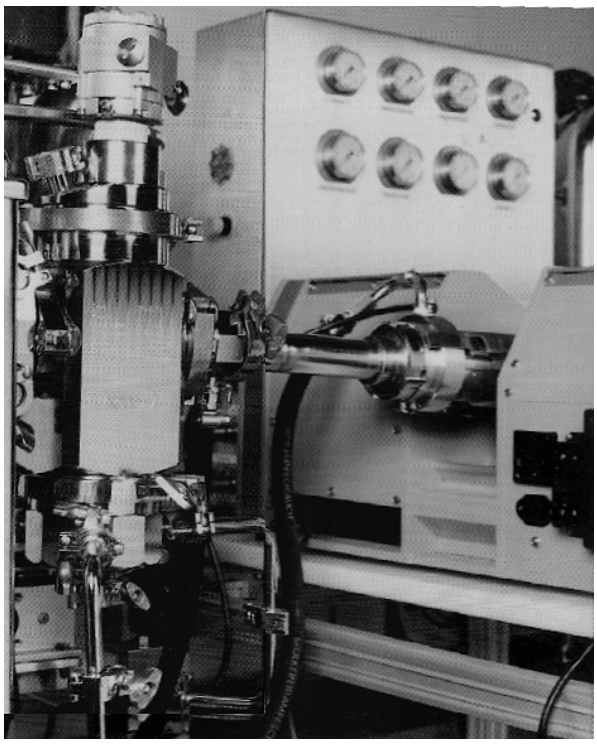


Figure 6: In-line 100 AFG system for CIP operation.



A closer view of the measuring cell and the instrument is shown in Figure 7. The plate on the left side covers the jet mill and the classifier. The centre of the figure contains the tube for sheath-flow compressed air supply. Measuring cell and instrument are completely separated, to avoid vibrations being transferred to the instrument.

Figure 7: Measuring cell and instrument installed in-line directly behind the classifier of a fluidised bed jet mill 100 AFG.

3.1 Results from monitoring the process

Many different materials have been tested so far. Some selected results are intended to demonstrate the capability of the system.

The results of quartz milling presented in Figure 8 were the very first ones to show fluctuations of the product PSD. The monitored change of the product fineness was mainly caused by small variations of the classifier speed due to fluctuations of its loading. The classifier motor- running at that time with an old model of variable speed drive, -was not stabilised properly. After the frequency converter was exchanged, the influence of the speed stabilisation was obvious, see Figure 11. In Figure 8, one can also see the start-up of the system during the first 4 measurements. Afterwards there is a fluctuation around a median value.

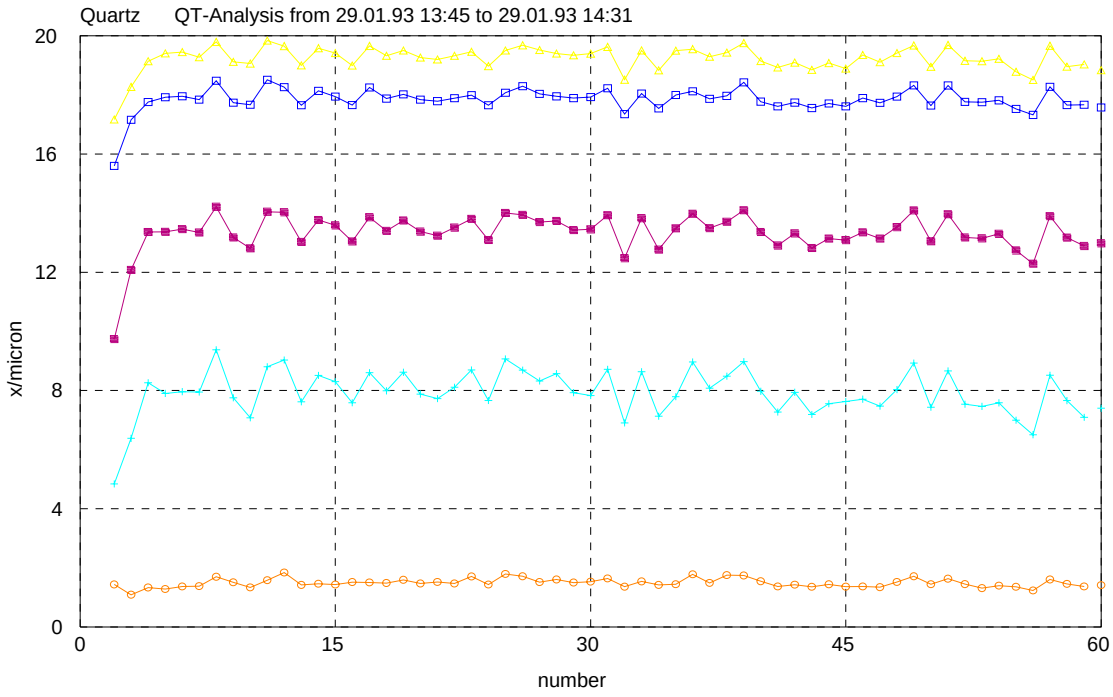


Figure 8: 60 results of quartz milling at n=4000rpm measuring time 20s, 2 measurements per min. Outlined parameters from top to bottom: x_{97}, x_{95}, x_{80}, x_{50} and x_{10}.

Figure 9 clearly shows the enormous influence of the screw feeder, which is dramatic for some special products, in this case lactose. The start of the screw feeder caused a direct breakthrough of coarse material through the classifier. This had the immediate consequence of changing the feeding mode from batch to very slow but continuous feeding for this particular product.

Figure 10 shows the results of NaCl milling. NaCl was used as a model substance for pharmaceutical products regarding milling behaviour. After the start-up of the process the classifier speed was changed. The steps in classifier speed resulted in clear steps of particle size, but small steps in concentration. The small instabilities of x values indicate instabilities of the process, which in this step were not yet controlled, but only monitored.

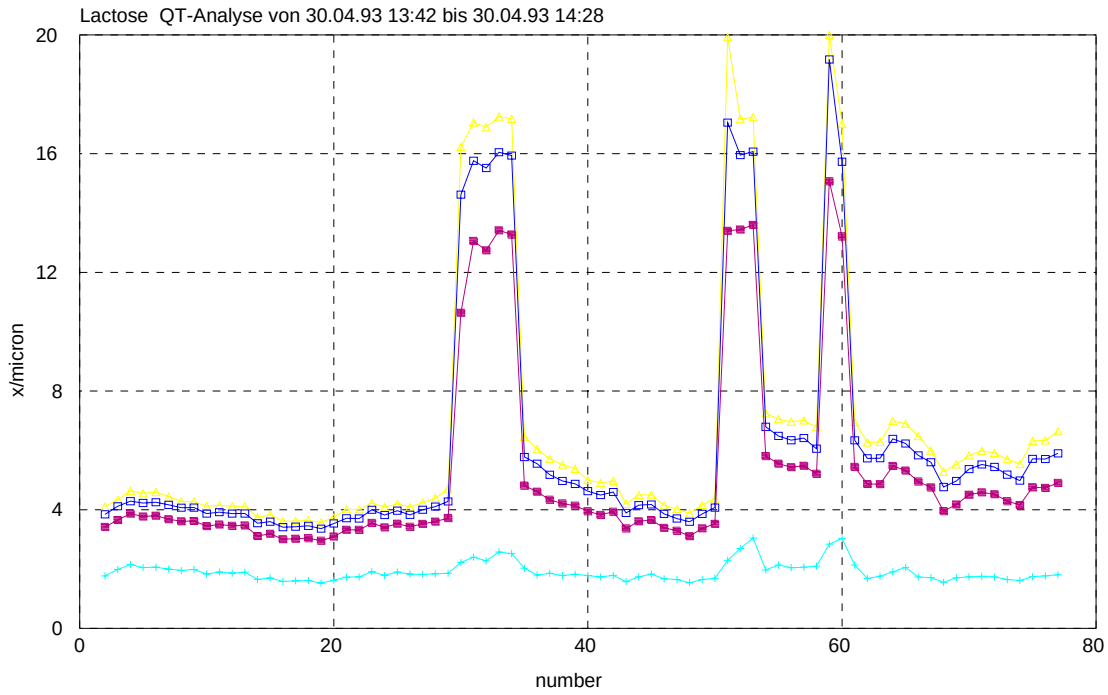


Figure 9: 75 results of lactose milling at n=17500rpm measuring time 20s 2 measurements per min. Outlined parameters from top to bottom: x_{97}, x_{95}, x_{90} and x_{50}.

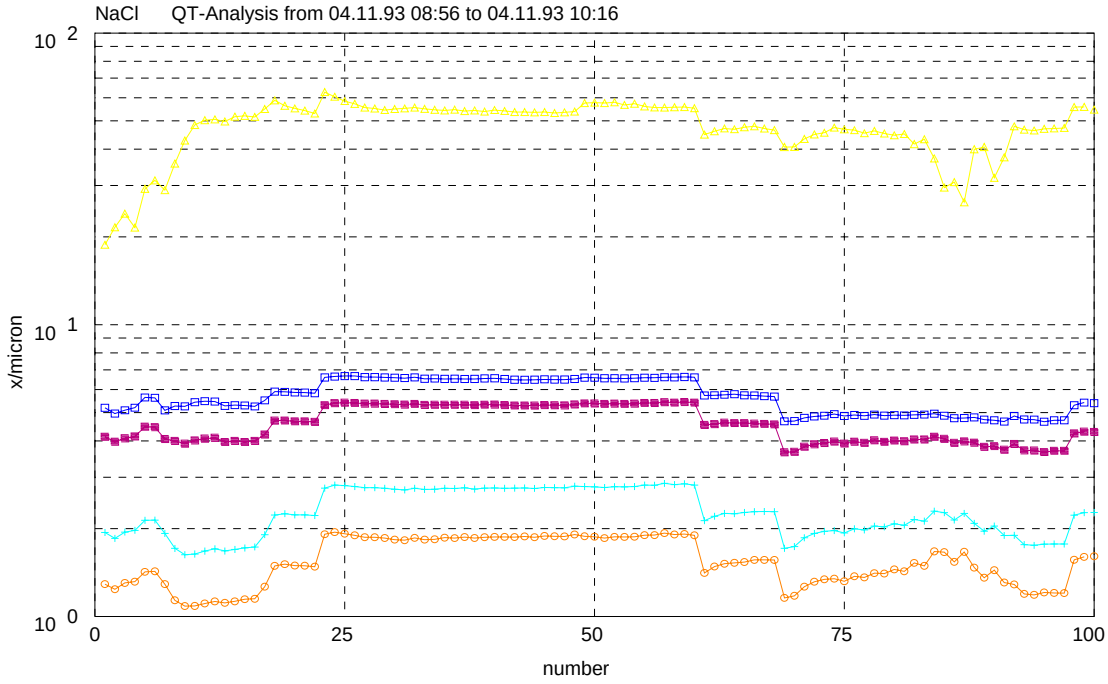


Figure 10: 100 results of NaCl milling at different rotational speeds: n=10000rpm, 9000rpm, 8000rpm, 9000rpm and 10000rpm again; measuring time 20s 2 measurements per min. Outlined parameters from top to bottom: C_{opt}, x_{97}, x_{90}, x_{50} and x_{30}.

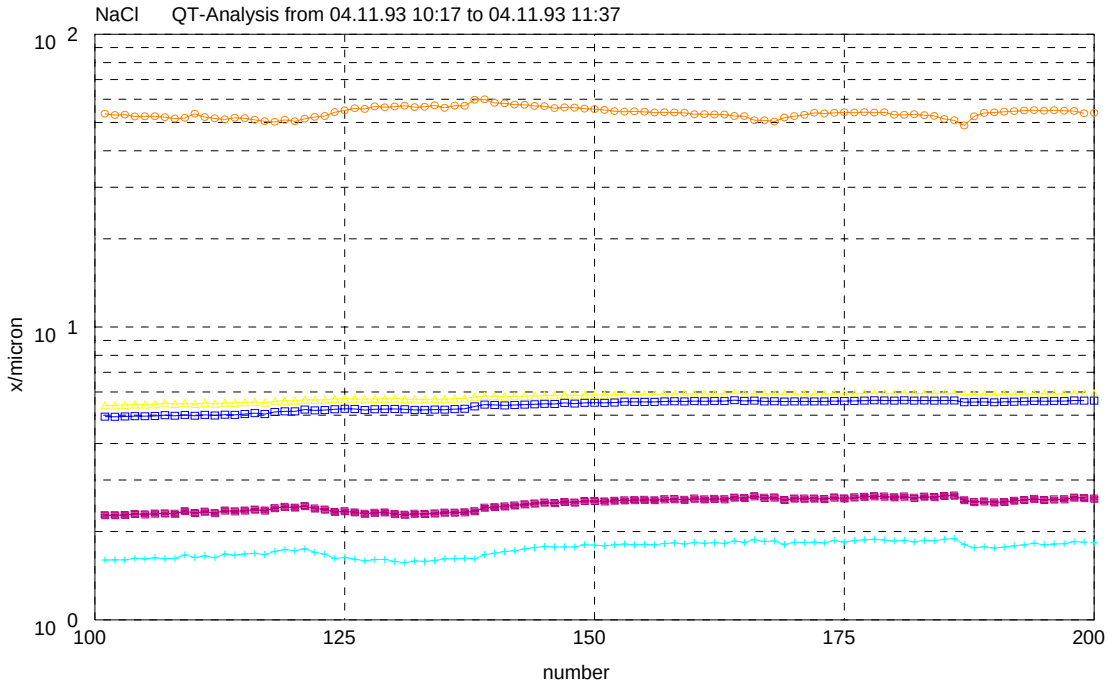


Figure 11: 100 results of NaCl milling without changing rotational speed: n=10000rpm; measuring time 20s, 2 measurements per min. Outlined parameters from top to bottom: C_{opt}, x_{97}, x_{90}, x_{50} and x_{30}.

Figure 11 shows the results of NaCl milling. It demonstrates impressively the stability of x_{97} and x_{90} results over a long measuring time, although there were fluctuations of optical concentration. The slow fluctuations are due to variations in the filling level of the mill chamber caused by the infrequent switching of the feeding screw. When the screw is in operation, the optical concentration tends to increase slowly, as more particles leave the mill via the classifier. Due to the higher loading of the air, its cut point is shifted slightly to the fine side. The contrasting curves for C_{opt} and x_{50} or x_{30} prove this well-known effect. But there are no more fast fluctuations due to the controlled classifier speed. Additionally, a small trend towards coarser product can be seen over the monitored time. There can be many reasons for this, such as temperature effects, changes in the amount of product inside the mill, changes of the filling level inside the mill, plugging of material to the inner walls and so on. An exact answer is difficult, but it is obvious that in-line analysis offers new possibilities as a tool to get a closer look at the behaviour of processes.

3.2 Results with closed loop

It was possible to overcome most of the detected influences on the product fineness mentioned above could be overcome by a stabilised closed-loop control of the classifier speed. As the classifier speed is the most important parameter in classifications, it is highly advantageous to use this parameter for a closed-loop fineness control with the in-line analyser. The speed can be changed easily by varying the frequency of the converter.

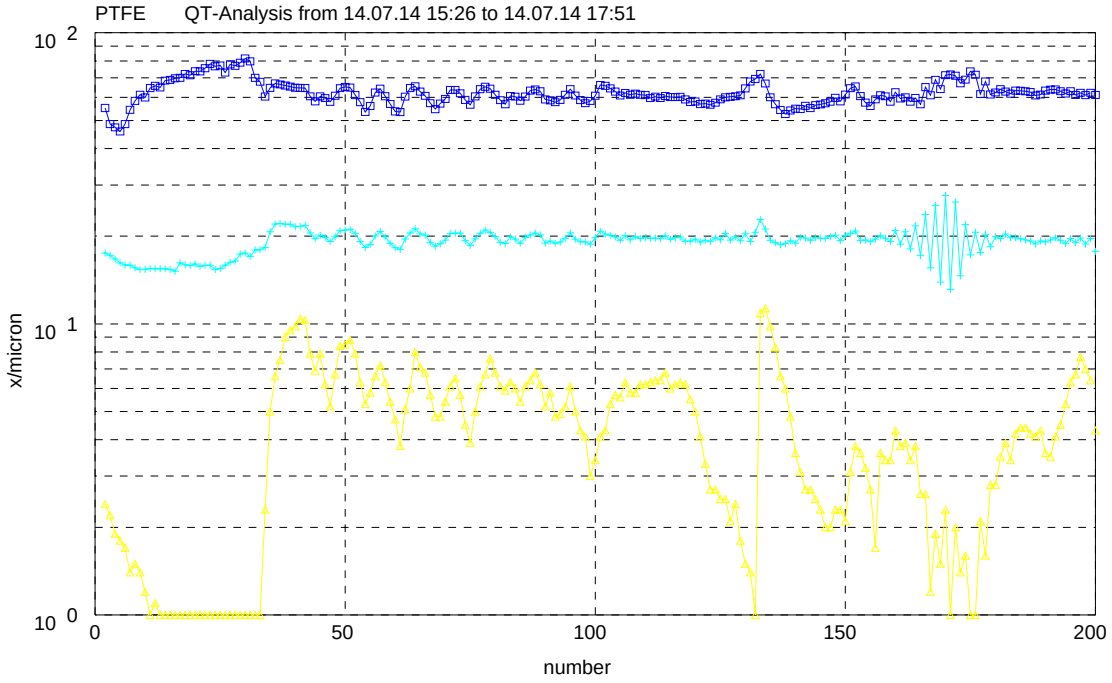


Figure 12: 200 results of PTFE milling with closed loop control of classifier speed without optimised control parameters; measuring time 10s, 2 measurements per min. Outlined parameters from top to bottom: x_{97}, x_{50} and C_{opt}. The settings of the proportional control parameter are listed in table 1.

number	setting Cp	system reaction
1 - 40	0,2	too slow, system start-up
41 - 49	0,2	too slow
50 - 75	0,4	too slow
76 - 97	0,8	too slow
98 - 157	1,6	OK
158 - 169	3,0	too fast
170 -183	1,6	OK
184 - 200	2,0	still too fast

Table 1: The settings of the proportional control parameter in Figure 12.

The results of Figure 12 demonstrate impressively the influence of the controller parameters. The optimisation target for its parameter settings was to find a compromise between shortest reaction times on the one hand, and stable operation even after strong disturbances on the other hand. The controller was operating in PI mode. Over the period of time shown in Figure 12, the gain C_p (proportional value) was modified at constant Tn. Firstly, Cp was too small and had to be increased at measurement number 50 and 76. The set point of the controller was 20microns for the x_{50}. The result was constant and very stable operation. For testing the circuit's stability the feeding screw was switched off manually at measurement 116. The mill empties (decrease of C_{opt}), but the fineness remains constant. Only when the screw is switched on again at number 130, now at double speed, the x_{50} becomes slightly coarser for only 3 measurements (approx. 1,5 min.) and then stabilises again immediately.

When the gain C_p was increased excessively at number 158, the case of critical instability occurred. By applying the old controller parameters from no. 170 onwards, stability was reached again within 8 to 9 measurements. Although the process itself was not running very smoothly - demonstrated by fluctuations of C_{opt}, the fineness controller was able to hold the desired product quality. Micronisation of PTFE is not an easy job, because of plugging problems in the mill. When wall deposits break off, the filling level increases abruptly (see C_{opt}), and the feed rate has to be reduced immediately.

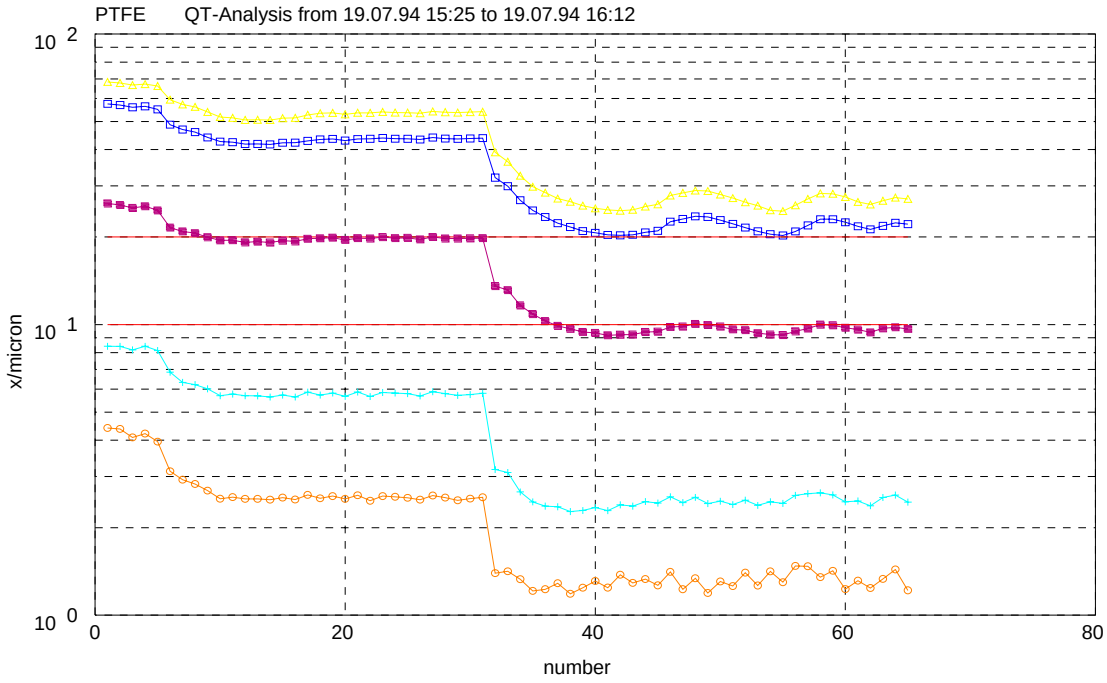


Figure 13: 100 results of PTFE milling with closed loop control of classifier speed with nearly optimised control parameters with two settings of x_{50} first to 20microns and after no. 30 to 10microns; measuring time 10s, 2 measurements per min. Outlined parameters from top to bottom: x_{95}, x_{90}, x_{50}, x_{10} and x_{05}.

Figure 13 shows the perfectly working closed loop control of the system. The chosen control parameter in this case was x_{50}, although the instrument software allows selection of every point of the PSD. The system immediately follows the change of the set point from x_{50}=20microns to x_{50}=10microns, and needs only 8 measurements, which is 4 minutes, to reach the new value. For testing of the system, a large step from x_{50}=20microns to x_{50}=10microns had been chosen. The large step results in smooth fluctuations around the set value, because the control parameters were not adapted at that time. It seems that for every material and every desired fineness, the settings for the controller have to be stored and changed.

3.3 Comparison of in-line and off-line results

For users of in-line systems, the characterisation of the gathered product is of major interest. The easiest way would be to use the in-line results directly, and calculate a mean product fineness of the total batch. To verify this, in-line and off-line results had to be compared. Two examples in Figure 14 and Figure 15 were selected to demonstrate the in-line system performance and the possibilities of using this system for permanent quality control of the powder.

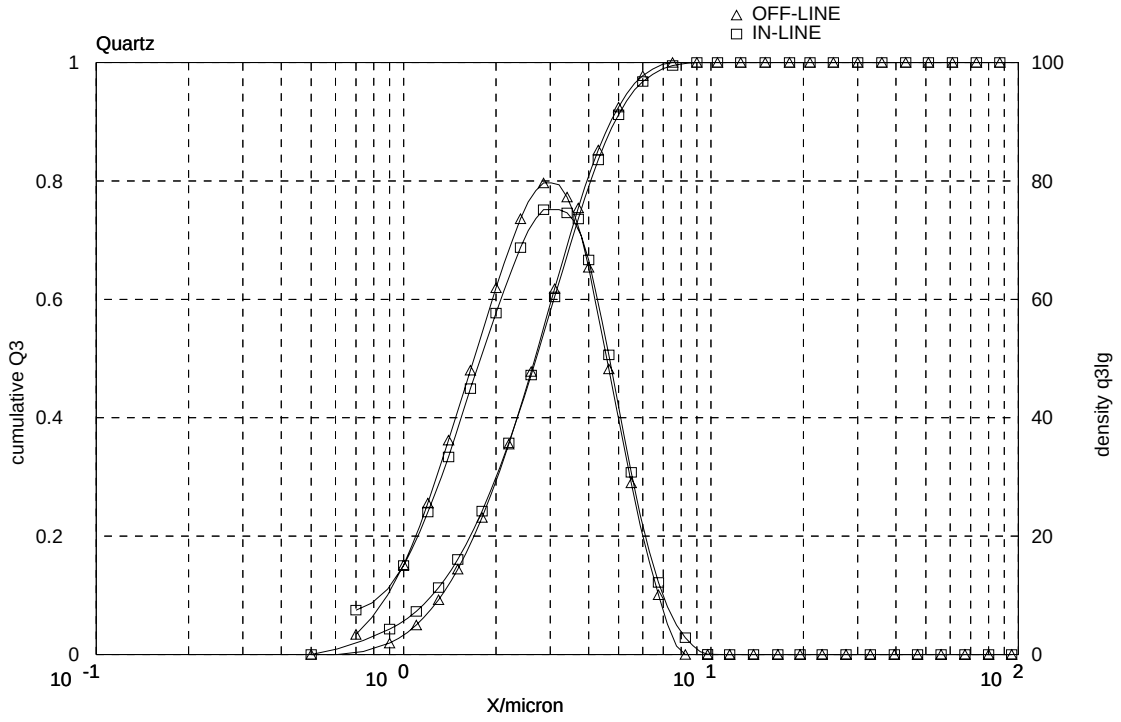


Figure 14: Comparison of the averages of in-line and off-line measurements of quartz milling; measuring time 20s, 2 measurements per min

The comparison of both products shows excellent concurrence between in-line and off-line measurements. No validation is necessary for comparison.

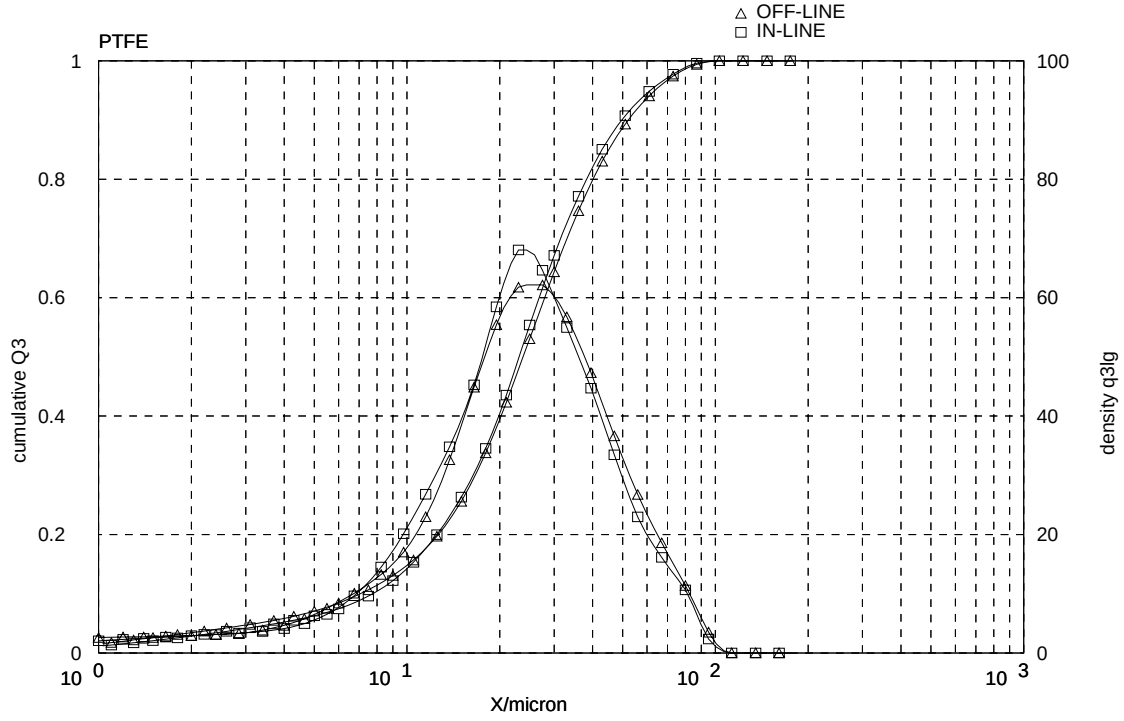


Figure 15: Comparison of the averages of in-line and off-line measurements of PTFE milling; measuring time 10s, 2 measurements per min.

4 Conclusions and outlook

The development of the measuring cell has been successfully completed. The expectations were more than fulfilled. Most important for the performance of the total system is keeping the windows free from dust contamination. Rinsing with secondary gas was found to be appropriate. Measurements with a laser diffraction analyser have shown, that it is possible to detect PSD directly in the undiluted 2-phase stream exiting the classifier. System operations over more than 10 hours without interruption were reached. The measuring frequency is in the range of 2 to 4, max. 6 per minute.

The system software is able not only to measure the sequence of PSDs, but also the trends of selected fineness values from the distribution. One of these can be transferred to an electrical signal of 0-10V/20mA. This is a basis to form a closed loop with a controller for a continuous fineness control of powders. Variations of the set point as well as various process disturbances are then automatically adjusted within a very short time.

The performance and possibilities for an in-line system in combination with a fluidised bed opposed jet mill 100 AFG were discussed. The incorporated classifier enables easy fineness adjustment by means of the rotor speed only. Thus, it is not only possible to keep the product fineness constant but the system also provides the possibility of validating the fineness in the general sense of quality control (ISO).

In principle it should also be possible to utilise the developed in-line technique for other types of classifier mills and ordinary impact mills without classifier. The

latter could be controlled by parameters such as grinding rotor speed or gas flow rate. But so far, this has not yet been tested.

The main target for further development is the application of this in-line analysis in larger production-scale plants. Here, the idea is to extract a partial stream out of the main stream in a big outlet pipe of the classifier. To ensure that it is representative, this partial stream has to be extracted under isokinetic conditions, see Röthele /5/.

5 Literature

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