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# Quick and Tubeless Suspension Analysis with Laser-diffraction

## 1 Introduction

Laser diffraction (LD), with reference to the analysis time, is highly advantageous for the automation of particle size analysis [1,2]. Up until now this advantage was only available for dry dispersing systems [3,4]. Former wet dispersing systems were optimised to robust design and maximum flexibility i.e. to cover a large variety of applications [5]. The system were difficult to automate and provided cycle times of minimum 5 minutes which made it uncomfortable to use.

The new attempt was optimised with respect to the main aims of automation and short cycle times. Automation demands complete remote control and long lifetimes of all components. Important border conditions were the improved performance of the system regarding maximum size and density of particles making the system also suitable for large focal distances.

Consequent optimisation of all necessary analysing and handling steps resulted in cycle times down to 30 s still keeping the reproducibility at the highest standard. The tubeless construction also makes the system ideal for aggressive media and gives long lifetimes. The possibility to attach external sensors, i.e. for liquid levels of feed, dispersant and waste supports automated measurements as well as series of standard measurements.

Additionally the developed system is also capable to high viscosity liquids such as oil and cocoa butter. Controlled heating is integrated as well as ultrasonics.

## 2 Concept of a tubeless wet dispersing system

The desired system was developed for industrial as well as research purposes. The performance of the system should meet the requirements listed in 2.1 in order of their priority.

## 2.1 Design rules and requirements

1. short cycle times which means especially fast

    - filling and
    - draining

2. highly automated

3. improved performance regarding

    - reproducibility
    - maximum particle size
    - maximum particle density

4. simple operation

5. high flexibility

    - high liquid viscosity
    - heatable

6. high chemical resistance e.g. no tubes.

## 2.2 First Approach

The first approach was a very simple system in which the flow was guided in a plane, see Fig. 1 and Fig. 2. A centrifugal pump was used for liquid and particle transport. The minimum channel width was 6 mm to allow transportation of large particles and fast draining at high viscosity. Fast draining is performed by a lowered bottom including the pump motor. The motor speed can be adjusted. The 1l basin is mixed up completely because the tangential inlet drives the rotation against that of the centrifugal pump which gives an optimal mixing of particles and liquid. No sample splitting is taking place, because the complete suspension passes through the measuring zone e.g. the laser beam.

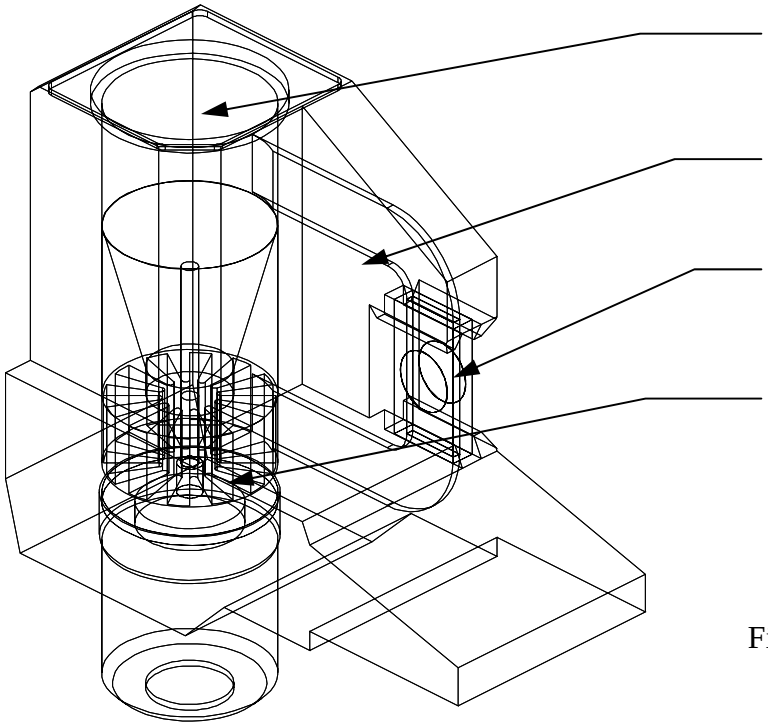


Fig. 1 First draft of an automated system

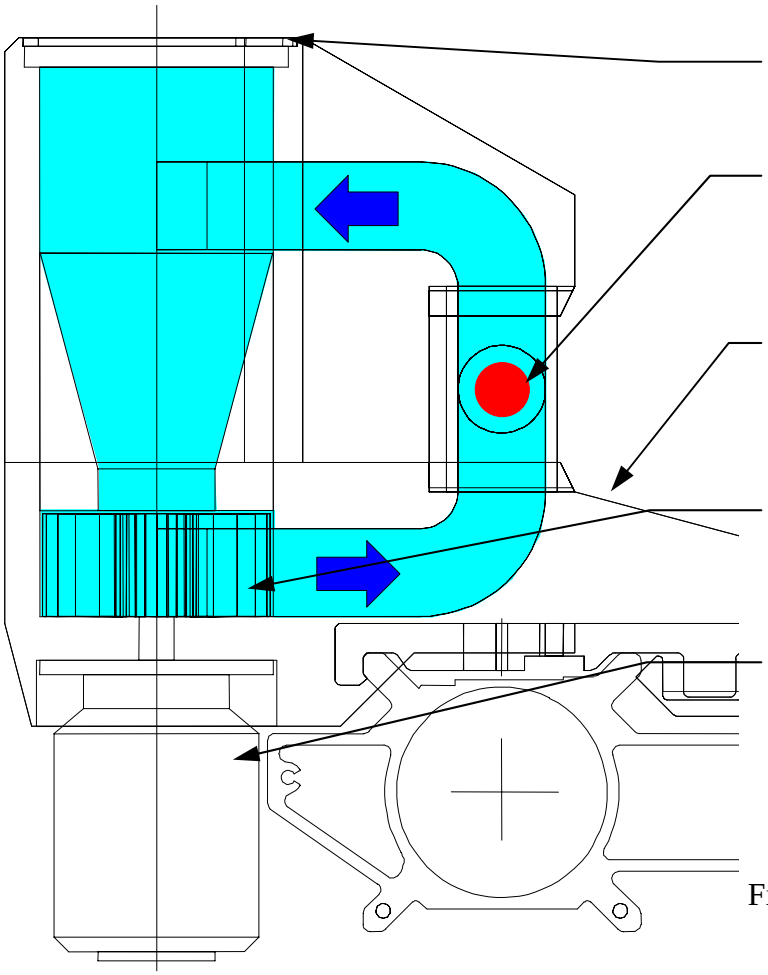


Fig. 2 Flow visualisation in the first conception of an automated system

## 2.3 Investigation of cuvette flow

Many experiments had to be made to find the best solution for particle transport to and through the cuvette. As indicator 3 mm glass beads were chosen as testing material, because the mixing condition could be seen very easily. For performance evaluation of the mixing condition the particle behaviour in the measuring zone was documented by taking pictures through the cuvette windows. The pictures were taken by a CCD-camera with a 20 mm-objective.

One of the very first results is demonstrated in Fig. 3A. It easily can be seen that the particles are transported by centrifugal forces by the former redirection to the wall.

That gave reason to build in many different flow guiding installations within the channel below the cuvette. The best result of that can be seen in Fig. 3B. The distribution of the glass beads is much better than without flow guiding, but the concentration near the left wall is still higher. For measurements this is certainly not good enough.

This gave rise to principle changes of flow geometry with the aim to get the centrifugal forces out and more mixing forces into the channel. From historic experiments it was known that a 90°-sudden redirection gives the best mixing. This was realised by a perpendicular redirection normal to the window which meant accepting the disadvantage of more complicated pipe guidance.

The big improvement could be seen immediately: Fig. 3. Segregation was no longer seen. The particles were transported ideally distributed. To prove this, a stronger test was performed adding 2 mm steel beads to the glass. But even these particles were transported properly, see Fig. 3.

This proved the suitability of the system for large particles of 3mm diameter and those with high density and large diameters. The settling velocities of the particles were 0.39 m/s for the glass beads and 0.61 m/s for the steel beads, which are very high settling velocities which the system was able to handle.

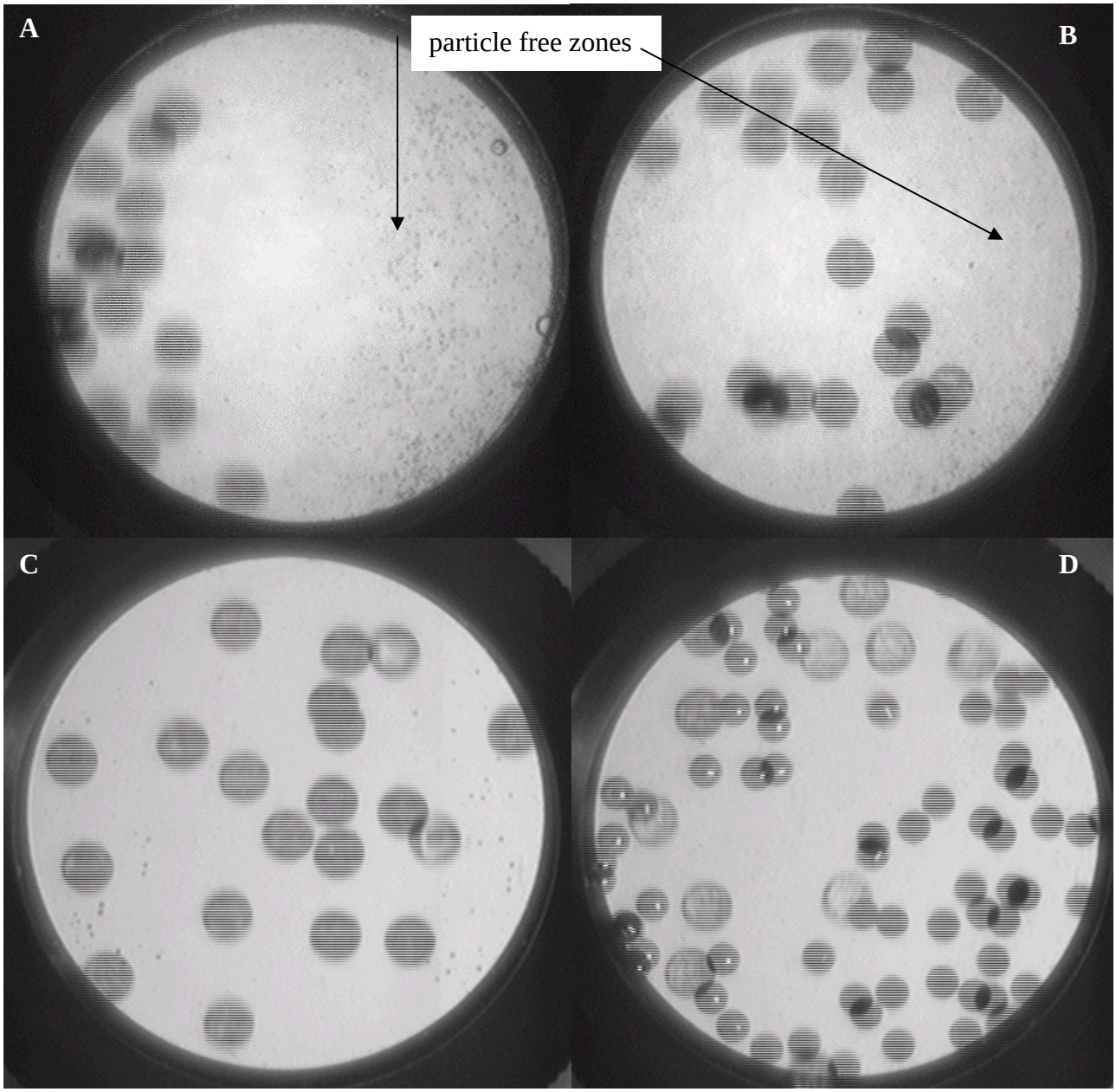


Fig. 3 3mm glass beads distribution in the window of the cuvette

A: Particles transported to the wall by centrifugal forces of the former redirection.

B Better mixed with flow guiding installations within the channel

C Good particle distribution after perpendicular redirection below.

D Perfect mixed transport of 3mm glass beads and 2mm steel beads.

## 3 Solution

The solution was a completely automated, compact, tubeless system where all parts in contact to the suspension are made out of stainless steel: Fig. 4. It fulfils all requirements mentioned in 2.1 including additionally an optimised handling, e.g. the quick and easy changing of the cuvette.


Fig. 4 Completely automated wet dispersing system.

# 4 Results

## 4.1 Cycle times

The cycle times of the system results out of the summation of steps:

| | | |
|---|---|---|
| filling; depends on pre pressure of the liquid e.g. 3 bar: | $t_f$ | 8s |
| removal of bubbles; typical | $t_b$ | 4s |
| reference measurement; adjustable | $t_{ref}$ | 5s |
| addition of material and mixing; typical | $t_{mix}$ | 5s |
| measurement; adjustable | $t_{mes}$ | 5s |
| drain | $t_d$ | 3s |
| cycle time: sum: | $t_{cycle}$ | 30s |

Table 4.1: Calculation of cycle times for water

Now for water cycle times of 30 s are possible with materials which do not need any ultrasonic and no rinsing of the disperser. But even with e.g. 20 s of ultrasonication and one rinsing step, cycle times of 1 min are possible, especially when the system is completely automated and fed by a robot.

Of course these cycle times depend strongly on the viscosity of the liquid. The system was proven to be suitable for very viscous liquids such as silicone oil and cocoa butter. With these kind of liquids the heating option is also very helpful.

## 4.2 Measurements of Particle size distributions

Only a small number of results can be presented here to show the strong performance of the developed system. First the results of measured 1.5 mm glass beads are presented in Fig. 5. The measured $x_{50}$ value only differs 0.7 % from the nominal one.

The comparison of results measured with different measuring ranges presented in Fig. 6 shows also the high performance of the system.

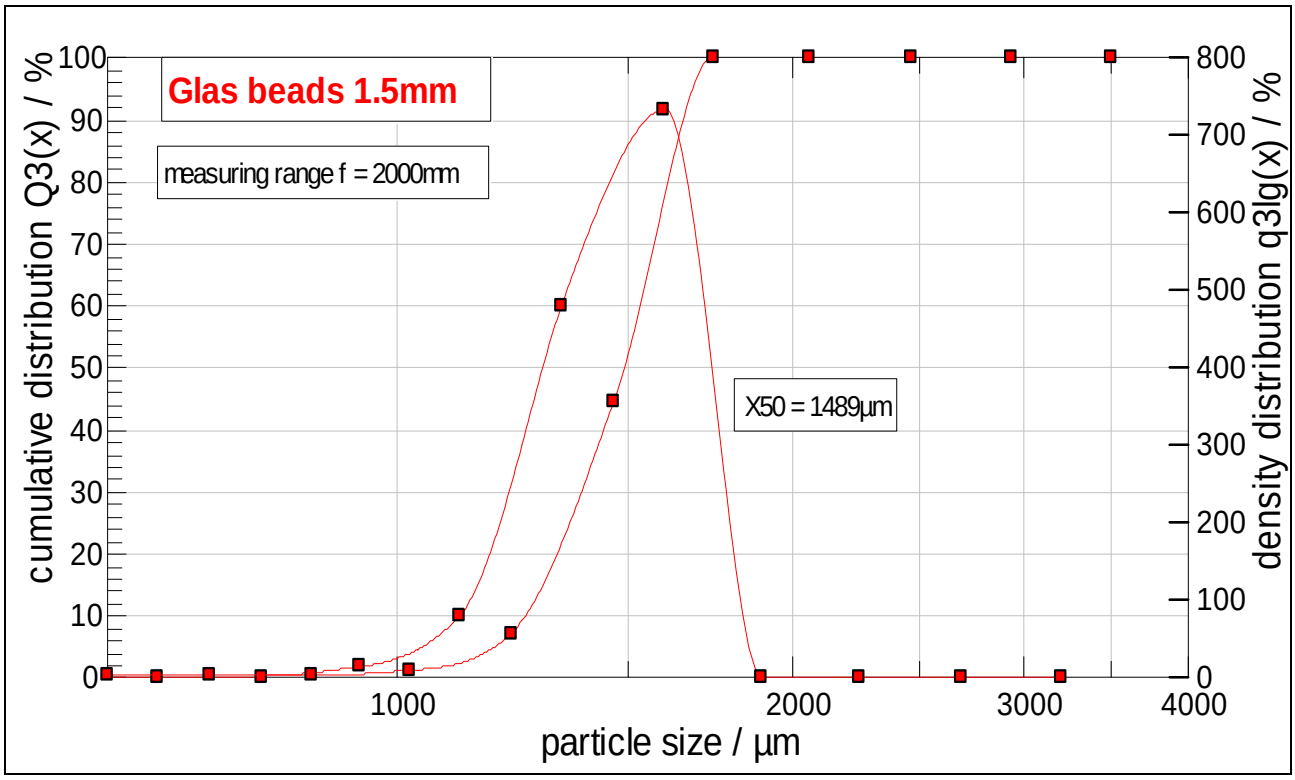


Fig. 5 1.5mm glass beads measured with a 2000mm focal length objective.

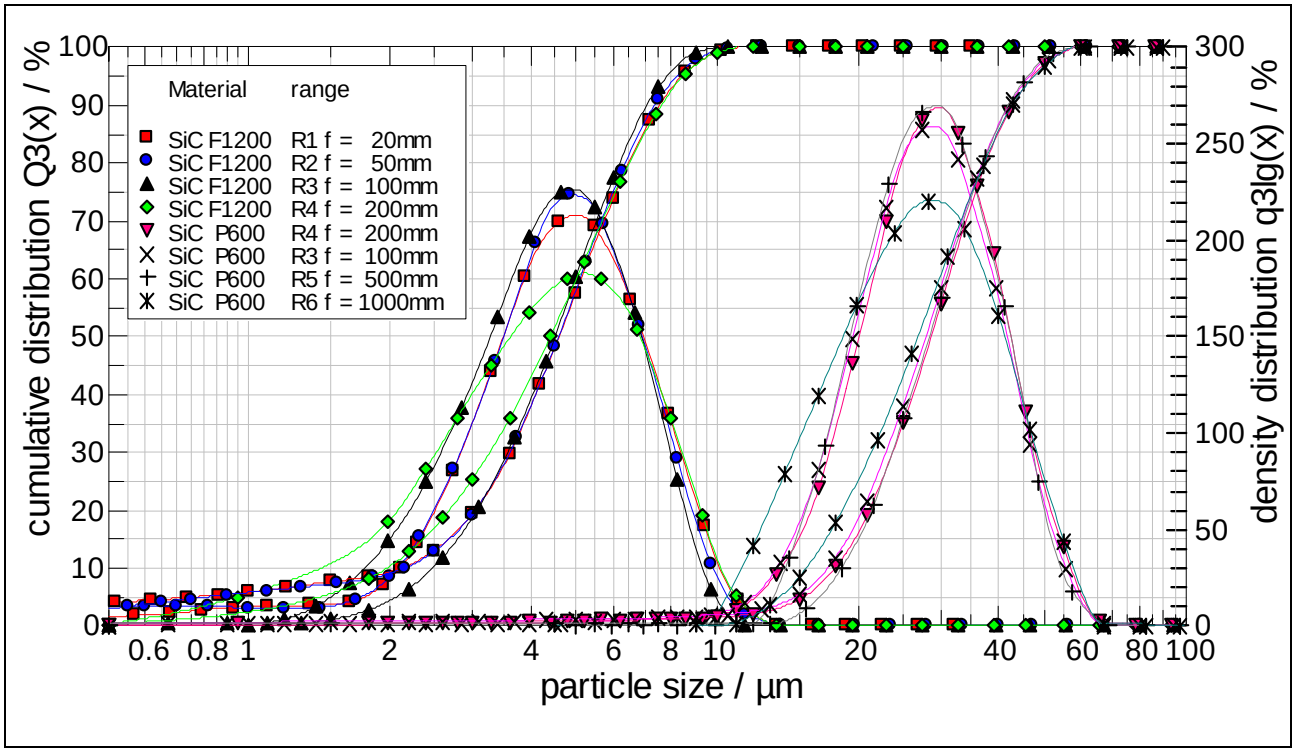


Fig. 6 Comparison of results measured with different measuring ranges.

An example for broad distributions is the mixture of separated distributions. This is presented in Fig. 7. The excellent reproducibility is demonstrated in parallel under the particle size distribution in the same figure.

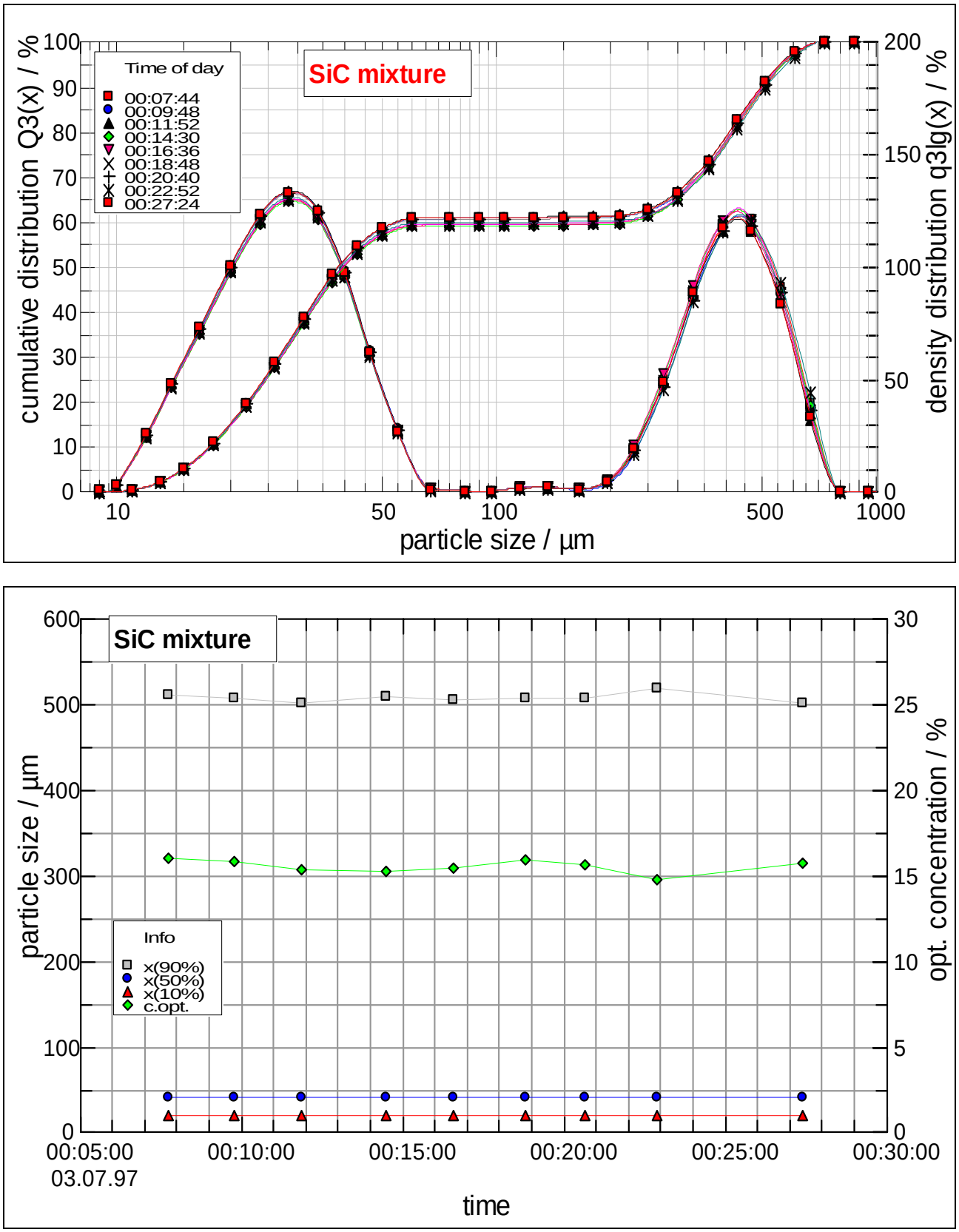


Fig. 7 Measured mixture of silicone carbide reference materials over time

## 5 Conclusions

The developed wet dispersing system for an LD instrument, fulfils all desired requirements. It incorporates nearly all fields of wet applications. Optimised cycle times allow high measuring frequencies up to a quasi continuous measuring operation.

The new suspension cell presently offers the possibility of completely automated fast particle size analysis, which up until now was only available for dry dispersing systems.

The possibility to attach external sensors, i.e. for liquid levels of feed, dispersant and waste supports automated measurements as well as series of standard measurements.

Optimised cycle times allow measuring frequencies down to 30 s. The variable filling level allows for dilution without loss of particles (and possible change of PSD) and working with small volumes down to 300 ml.

The tubeless construction makes the system also ideal for aggressive media. Large pipe diameters make it suitable for high viscosity liquids also. High liquid velocities allow application to size ranges of ca. 3 mm of glass and 2 mm for steel beads. The optimisation was made especially with respect to reproducibility, which could be improved.

## 6 References


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[5] M. Heuer, K. Leschonski — Erfahrungen mit einem neuen Gerät zur Messung von Partikelgrößenverteilungen aus Beugungsspektren, 3. Europ. Symp. Partikelmeßtechnik in Nürnberg, 1984

## 7 Nomenclature

| | |
|---|---|
| $C_{opt}$ | optical concentration |
| $t_f$ | filling time |
| $t_b$ | debubbeling time |
| $t_{ref}$ | reference measurement time; adjustable |
| $t_{mix}$ | mixing time |
| $t_{mes}$ | measurement time |
| $t_d$ | drain time |
| $t_{cycle}$ | cycle time |
| $x$ | particle diameter |
| $x_{10}$ | particle diameter to which 10 % of the cumulated undersize $Q_3(x)$ corresponds |
| $x_{50}$ | particle diameter to which 50 % of the cumulated undersize $Q_3(x)$ corresponds |
| $x_{90}$ | particle diameter to which 90 % of the cumulated undersize $Q_3(x)$ corresponds |