

# PARTICLE WORLD

Technical Papers of QUANTACHROME

Edition 2 • April 2009

**Characterization of particle size distribution by laser diffraction according to the recommendations of the standard ISO 13320**

**The Laboratory for Scientific Particle Analysis (LabSPA)**

**Gas adsorption with the QUADRASORB SI:  
one sample - one dewar ... four samples - four dewars!**

**Determination of particle size, zeta potential,  
rheological and other parameters in concentrated  
dispersions**

**The next generation: CILAS 990, CILAS 1090  
and CILAS 1190 for excellent particle sizing  
and optional particle shape analysis**



Dear Reader,



with our second PARTICLE WORLD we want to inform you about latest developments in the field of characterization of powders, dispersions and porous solids. QUANTACHROME offers sales, service and application support for various product lines of innovative analysis instruments around the world of particles. There are interesting news in particle sizing and particle shape analysis, in chemisorption for characterization of catalysts, with the QUADRASORB SI in highthroughput surface and pore size analysis, in acoustics and electroacoustics for determination of particle size, zeta potential and rheological parameters in concentrated dispersions and in density measurement by gas pycnometry.

Please read in PARTICLE WORLD 2 about the new generation of CILAS laser particle sizers in combination with a new option for parallel particle shape characterization. The new CILAS line combines the traditional strengths of highest reproducibility in particle sizing with new features and modern design. Reported are also experiences with laser diffraction and the application of the Fraunhofer and Mie calculation method for particle size distributions according norm ISO 13320.

Further news about particle characterization presented in the PARTICLE WORLD 2 are

- **ULTRAPYCNOMETER 1200e** and
- **PENTAPYCNOMETER 5200e** for density measurements,
- **CHEMBET Pulsar** for automated chemisorption and temperature programmed reactions for characterization of catalysts,
- **QUADRASORB SI** with 4 and alternatively 3 and 2 analysis ports,
- **DT-1200** as the tool for the characterization of concentrated dispersions by measuring particle size (5 nm – 1 mm) and zeta potential has an option now to measure additionally rheological parameters
- **Unique cryostat option in gas adsorption for the AUTOSORB-1-MP** for the measurement of gas adsorption isotherms from 77 K to 200 K for micropore analysis without liquid argon and for calculation of thermodynamic parameters for additional characterization of porous solids.

All these and other instruments are available in our **LabSPA (Laboratory for Scientific Particle Analysis)**. A complete analysis programme is available for your test and contract analysis respectively for method development in the field of characterizing powders, dispersions and porous solids! Within a few days you get not only the printed results but a professional report with explanation of the method, the run conditions and interpretation of the results.

Last but not least we invite you to our particle seminar 2009 in Delft. By use of the fax answer you can get more information about the seminar, but you can find the topics also in that PARTICLE WORLD. Do not hesitate to contact us for further information.

All the best for you, yours sincerely

Ton Goverde  
QUANTACHROME Office Benelux

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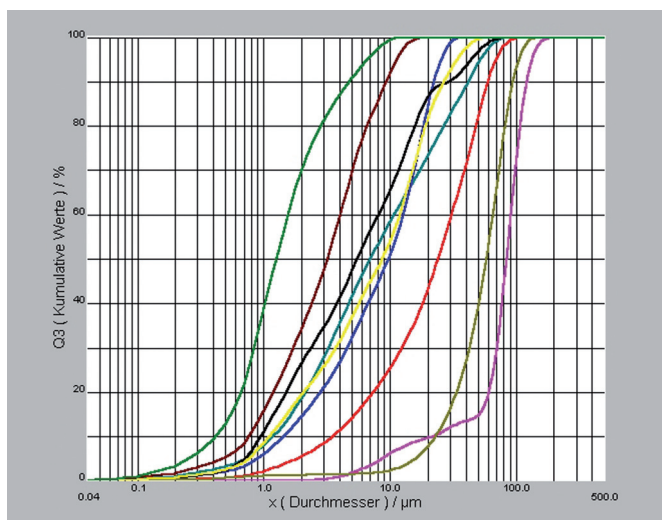
## Characterization of particle size distribution by laser diffraction according to the recommendations of the norm ISO 13320

With standard ISO 13320-1 for the laser diffraction method there is given a basis for comparable particle size analyses by use of this method. The laser diffraction method was converted into a commercial laser diffraction analytical instrument, for the first time worldwide, by the French hightech-company CILAS in the year 1968. Since 1968 the term Lasergranulometry is a synonym for a laser diffraction particle size analyzer, and particularly in the industry an appropriate analyzer is often generally called CILAS-Lasergranulometer.



**Figure 1**  
International standard ISO 13320-1 for particle size analysis, by means of laser diffraction method

Innovative CILAS laser technology is being used world-wide for the characterization of limestone and building materials (gypsum, cements, finery), of oxides (alumina, ferric oxide or SiO<sub>2</sub> in form of glass, sand, quartz) and metals (tungsten, iron, aluminum), of soils and sediments, of ceramical powders and slurries (high performance or dental ceramics), pigments (metallic or organic), carbon materials (graphite, coals or activated charcoal) or emulsions in cosmetics and the food technology as well as many further fine dispersed materials. Mill manufacturers check the grinding and separation processes, hundreds of other users in other quality control laboratories are using the CILAS technology for particle size distribution determination and the same in many research departments in industry or at research institutes of many universities.

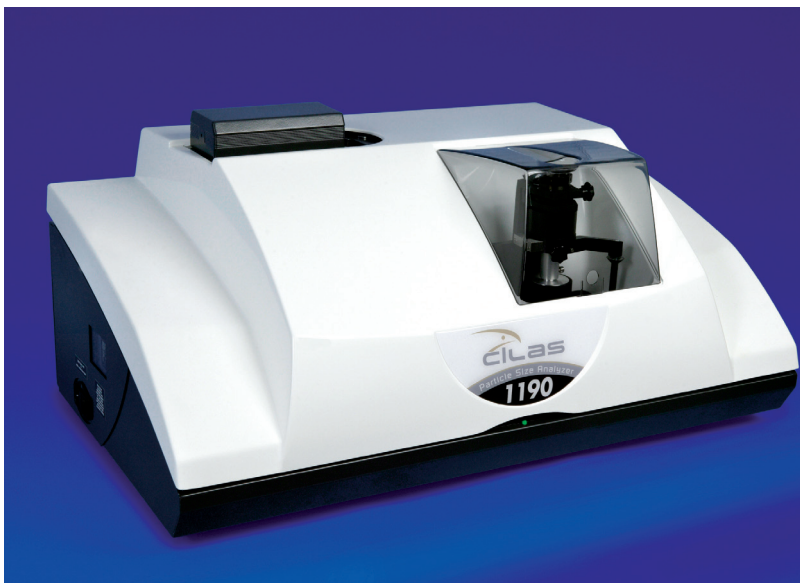


**Figure 2**  
Particle size distributions of different kinds of materials, measured with the CILAS 1064L, in the order of ascending particle diameters (D<sub>50</sub>-values): Zinc oxide, titanium dioxide, limestone, filling material, cement, graphite, lactose, milk powder, silicagel

With the innovative step of the introduction of commercial laser diffraction instruments by CILAS at the end of the 60's, the basis for the practical application of a measuring procedure was put to the particle size analysis, which is now described in ISO 13320. Laser diffraction technique is distinguished by the facts that

- it can be applied to different kinds of individual systems,
- it is fast,
- it can be fully automated,
- different commercial measuring systems are available.

Many CILAS instrument users usually unconsciously appreciate the latter point, if they decide for an analyzer that can solve a certain measuring task in an inexpensive way. So for example the CILAS analyzers CILAS 930e and CILAS 1064 do not offer certain possibilities of the CILAS 1180. The CILAS 1180 has additional capabilities which are relevant for some applications, e.g. to measure also coarser particles up to diameters of 2,5 mm. An optimal price performance ratio regarding the special measurement problem is rightfully an important criterion for an equipment purchase decision.



**Figure 3**  
The CILAS 1190LD is the successor model of the used CILAS 1180: See the new generation of the CILAS laser granulometers on page 8 of this PARTICLE WORLD. The CILAS 1190 is designed for complex particle size analysis, wet and dry, over a large measuring range with additional information about particle shape.

### **Laser diffraction in the comparison**

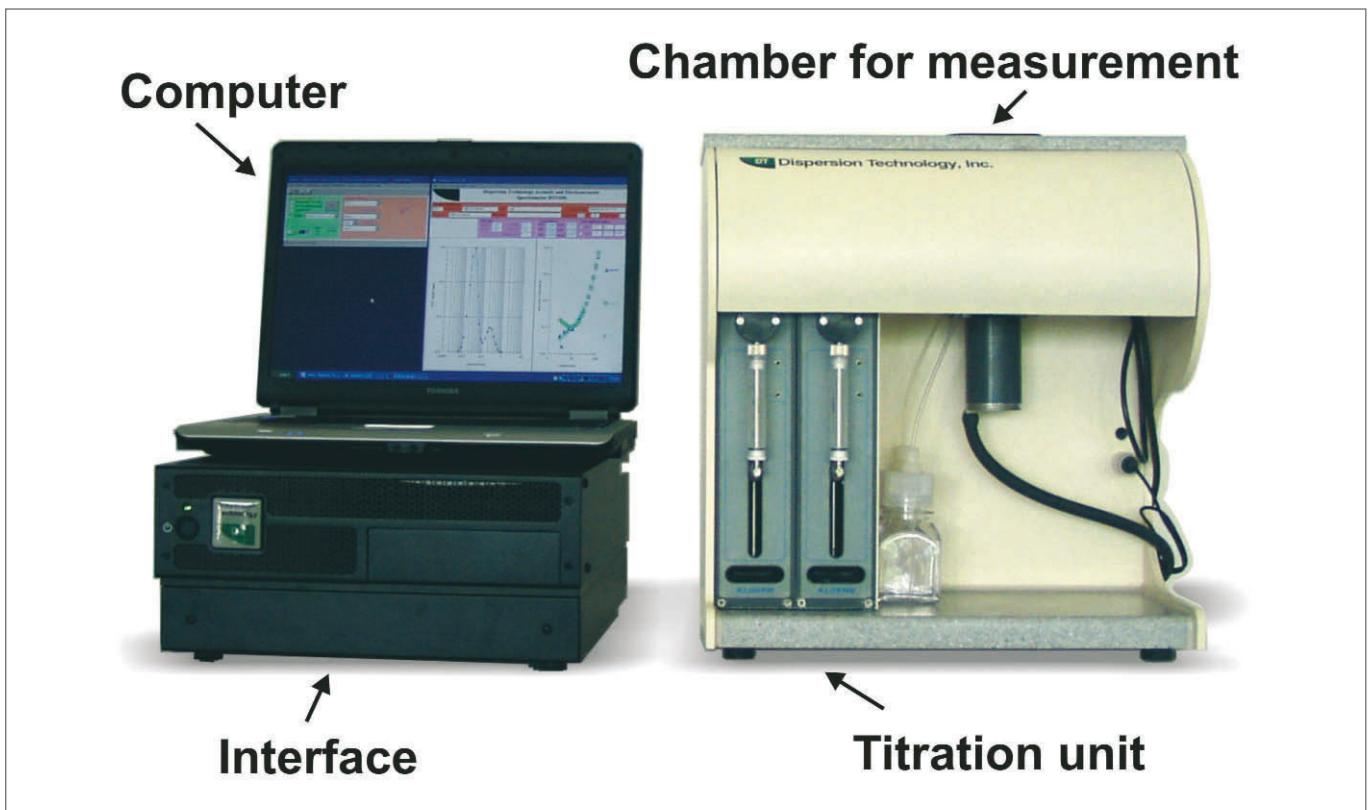
The particle size of powders and droplets determines relevant product properties of dispersed systems, which are developed for most different applications. A particle size analysis forms a substantial basis of the characterization of powders, suspensions and emulsions. If we reduce the term to the actual measurement principle, then we can enumerate and classify different measuring procedures according to different criteria. Traditional methods like the sieving and sedimentation are still used for comparison purposes, e.g. for the classification of soils in soil classes, or also because of the simple and/or partial inexpensive acquisition of the necessary tools (e.g. sieves, graduated cylinders). Commercial sedimentation lost enormous market shares in the field of commercial particle size analysis during the last few decades, since various advantages of the method cannot make up for their disadvantages, and because of the limited fields of application for this method. As example is mentioned the Fraunhofer calculation model used with laser diffraction, which does not request any material parameter, whereas sedimentation needs the density of the single particles and thus does not allow measurements of mixtures – but soils are normally mixtures!

In the lower nanometer range the laser diffraction method, according to ISO 13320, is not considered as the method that features the required strengths. Here the acoustic spectrometry has substantial advantages, also compared to various optical instruments, for many different tasks! The method of the attenuation of ultrasound can characterize dispersions in original concentration, including concentrated dispersions,

with particles in the nano and micrometer range very well. It can be combined with simultaneous measurement of other parameters like pH, conductivity, zeta potential and temperature. One of the main advantages in comparison to the photon correlation spectroscopy (PCS) is, that the sample has not to be motionless during the measurement, but can be stirred or pumped in a cycle, e.g. for online measurements too. Thus tendency to sedimentation does not disturb acoustic spectrometry results as for PCS! The samples will usually be measured, as mostly also with the laser diffraction technique, while a pump circulates the sample.

The Particle World 3 concerns itself completely, particularly and extensively, with the acoustic and electroacoustic method for the determination of the particle size distribution, the zeta potential as well as rheological parameters of dispersions in original concentration. This Particle World is to be requested at [ton.goverde@quantachrome.nl](mailto:ton.goverde@quantachrome.nl) or to be downloaded under Particle World of [www.quantachrome.eu.com](http://www.quantachrome.eu.com) or, by use of the enclosed fax answer, available as extensive and professional printed issue, free of charge at QUANTACHROME!

The laser granulometry shows its strength in micrometer and upper submicrometer range and is generally accepted as standard method that is described in ISO 13320. We follow here ISO 13320-1 in order to discuss the basic points of the possibilities and the execution of the laser diffraction method. However we also discuss some critical points and point out the method or the calculation models.



**Figure 4**  
The DT-1200 as combination equipment (DT-100 + DT-300 + DT-600) for the determination of the particle size distribution, the zeta potential as well as rheological parameters of concentrated dispersions



## Planning of particle size measurements

A substantial question to the analysis of particle size distributions concerns the definition of the goal. This cannot always be decided by the operator, but requires the knowledge of the processes, which have to be characterized. ISO 13320 refers to the fact that the laser diffraction method cannot differentiate between single particles and clusters of particles, which form agglomerates or aggregates. For most samples the primary particle size is the interesting parameter, so that all particle clusters must be dispersed to primary particles before the measurement. In the other cases, where the desired application is to characterize the particle agglomerates, this has to be indicated so that consciously no or a very mild dispersion is made before the measurement. Unnecessary misunderstandings can be avoided already here, if e.g. in the questionnaire of commercial analysis the measuring tasks are clearly described.

According to ISO 13320-1 (chapter 6.2.1) the investigation of samples begins with a sample inspection. This point is unfortunately often neglected and is the basis for various misunderstandings thereby. With a visual investigation of unknown samples and/or their investigation by means of a microscope one can often find some good information about the expected measuring range. Already with the sample inspection the CILAS technical advantages can play an important role. The CCD camera, integrated in the CILAS 1180, as well as the optional video microscope available for all CILAS analyzers, allows some extra possibilities. By use of the CILAS videomicroscope, different goals can be combined as the sample inspection with the particle shape determination and the calculation of the particle size distribution from microscopy by use of the Expert Shape software from CILAS. The ISO 13320-1 is excellently followed concerning sample inspection using the optional video microscope. Additionally to the result of the measurement of the laser diffraction, an inspection of the sample takes place by means of microscopy. By use of professional software one can calculate particle size distribution both from laser diffraction and image analysis for comparison and interpretation of the results. The appropriate CILAS software allows visual comparison between the laser diffraction results and the images from the microscope. The comparison and the interpretation can be accomplished in uniform software, even on the basis of an overlay of the particle size distributions from laser diffraction and image particle size analysis. Additionally with the CILAS 1180 the shadow images of the coarse particles from the standard CCD camera of the CILAS 1180 can be taken. By use of these options, the particle size ranges cannot only be estimated visually, but one receives at the same time, with moderate surcharge, information of the particle shapes. Differences of results e.g. to the sieve analysis can be understood, explained and predict substantially better on basis of parallel particle shape information.

The importance of sample splitting is also described in ISO 13320. QUANTACHROME offers for this the MICRORIFFLER, which can divide samples to small quantities, as they are used

for the laser diffraction measurements or others techniques. A MICRORIFFLER should have to be located in almost any laboratory, in which powder investigations are accomplished. The influence and therefore the importance of sample splitting to the results of the analytical methods is often not recognized - QUANTACHROME supplies the technology for this too!

## Theoretical background of the laser diffraction

In appendix A of the ISO 13320 the theoretical basics of interactions of laser light and particles is described. The four kinds of interactions are:

- The diffraction at the particle outline (Fraunhofer diffraction),
- Reflection at the particle surface (both at the outside and the internal surface of the particle),
- Diffraction at the surface from the medium to the particle and in reverse
- Absorption in the particle.

These overlaid phenomena lead to characteristic light patterns, which depend on the particle size and particle shape, as well as the optical characteristics of the particles. Examples of such diffraction patterns are schematically represented in figure 5. One recognizes the diffraction spectra of differently large single particles and the summarized spectrum of a particle size mixture from fine and coarser particles. The angle is in relation to the direction of the laser beam on the x-axis and the light intensity on the y-axis. Left it is recognizable that the intensity maxima of smaller particles shift toward larger angles. The summary spectrum of the particle size mixture, as it is received on the detectors, is seen right hand. The particle size distribution can be calculated by means of a matrix algorithm from this summary spectrum of the different particles.

It becomes clear that the highest intensity is in forward direction of the laser beam. However, there are clearly to be seen differences between the intensity maxima of the large and the small particles. Both spherical particles cause a symmetrical intensity pattern with specific particle diameter dependent maxima and minima of the light intensity. If one realizes

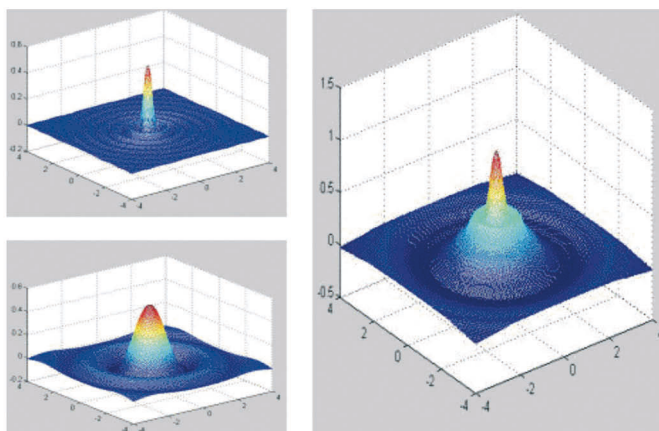


Figure 5  
Spectrum from different particles (left) and summarized spectrum of both particles (schematic)

by the analysis conditions, i.e. by work with sufficiently diluted dispersions, that multi scattering of the light is essentially excluded and a sufficient number of particles is statistically distributed measured, then the intensity spectrum of particle collectives can be evaluated.

The diffraction patterns of different particle diameters are well known. So it is necessary to use a mathematical algorithm to compute the portion of the theoretical spectra of all particle size classes from the experimentally determined total spectrum. The total spectrum is the experimental curve that means the light intensity as a function of the angle.

### Fraunhofer model

Historically seen, the Fraunhofer model was the base of the first optical model for particle size distribution calculations by laser diffraction. The following assumptions are:

- that the particles are spherical,
- that all particles are large enough, in relation to the wavelength of the light that only light scattering at the outline of the particles arises,
- that only light scattering in close forward direction is regarded, i.e. the angles are relatively small.

The Fraunhofer method is mathematically relatively simple to use and with the further advantage that no material parameters of the sample are needed. This is recognized also expressly in ISO 13320. Because Fraunhofer does not need any material parameter, this theory is recommended especially for sample mixtures. For mixtures it is almost impossible to get usefull material parameters, which are necessary when using the Mie-theory. This theory will be discussed below.

From a scientific point of view the Fraunhofer theory does not consider that the diffraction intensity of smaller becoming particles depends not only on the particle size but also on other effects. The extinction efficiency relative to the projec-

tion surface of the particle is accepted as constant. Thus when using the Fraunhofer approximation it is neglected that with small particles the diffraction intensity of the laser light decreases faster, than will be expected by geometrical projection surfaces of the small particle.

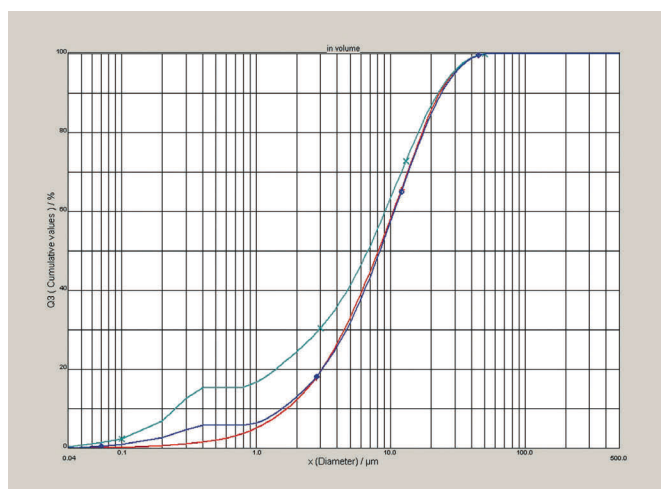
### Mie model

The Mie theory takes into account that other effects are influencing the diffraction intensity. Scientifically seen, the Mie theory is better to use for small particles as long as the material parameters are available. This is not the case for material mixtures and so we have a first restriction for the application of the Mie theory here.

The complex refractive index of the particle is needed for the Mie theory and this index consists of a real part and an imaginary part. The imaginary part of the refractive index describes the absorption of the light by the particle. Also for pure materials the ISO 13320 points out expressly that the knowledge of these material data is often not given. Particularly the imaginary part of the refractive index of the light in form of the absorption coefficient is usually not well known. It will not be argued here against one theory - all CILAS particle size analyzers offer both, the Fraunhofer and the Mie model, for a long time. The different evaluation models can be used in each case with a CILAS Granulometer, also after a measurement, and the results of both models can be compared. The raw data of a measurement are stored in a database. So, without further measurement, the appropriate evaluation model can be chosen. The raw data will be used for further calculations. In this article we will not use the ISO 13320 to overestimate the problems when using the Mie theory. However there is a substantial reason for critical use of the theories and there are reasons why the Fraunhofer theory is still the most used one. Additional to that, the ISO 13320 tells us that especially the imaginary part of the refractive index is strongly dependent from the wavelength of the laser light. Moreover the imaginary part is also influenced by crystalline inhomogeneity of most of the materials. The refractive index may change due to different crystal orientations and is also influenced by surface structures! Therefore no user of laser diffraction can consider all these phenomena as corrective for the particle size distributions.

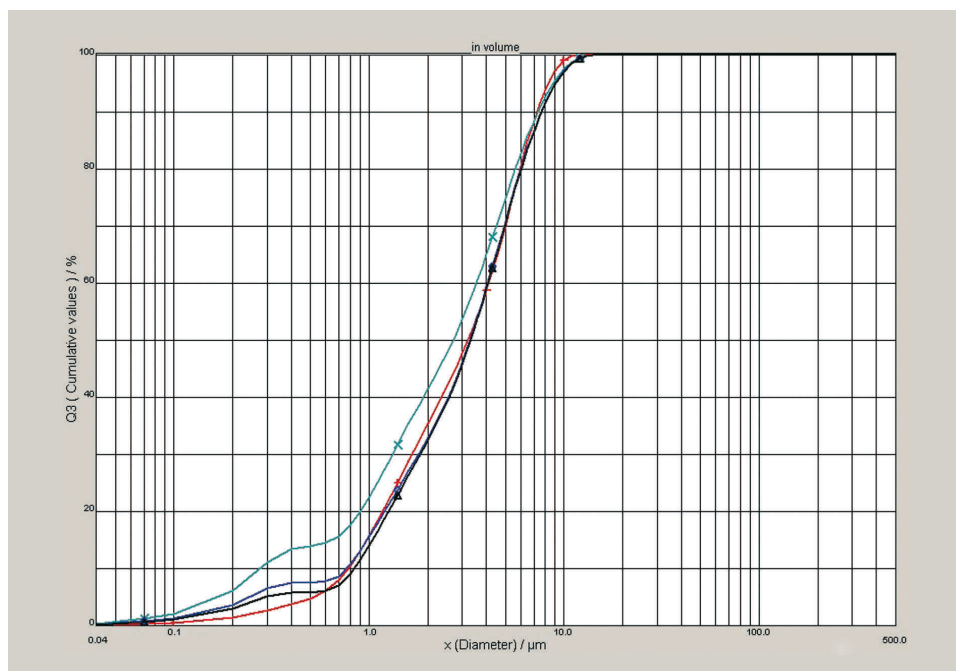
The described problem is clearly shown in the appendix D.2 of the ISO 13320: for approx. 75% of the solids listed, there is a range for the refractive index (real part) instead of a fixed value. More clearly it is shown for the imaginary part: for approx. 90% of the specified solids, no value is indicated, since for these materials no imaginary part is reported or known by the literature!

Unless it is theoretically correct to use the Mie theory, one can conclude from the ISO 13320, that this will not lead to a 100 % clear or well-defined particle size measurement. We usually decide on basis of the practical circumstances which evaluation model to choose. Not only information from the samples is being considered, but also traditions for comparison



**Figure 6**  
Determination of the particle size of a lime sample using Fraunhofer (red) and Mie: The ISO 13320 indicates a refractive index from 1,51 to 1,65, blue (1,65) and purple (1,51), note the significant differences.





**Figure 7**

Determination of the particle size of carbon black: The ISO 13320 recommends for the refractive index: 1,6 - 2,0 (real part) and 0,3 - 0,8i (imaginary part). The curves show the results using Fraunhofer (red) as well as Mie: 1,6-0,3i (türkis), 2,0-0,3i (blue) as well as 2,0-0,8i (black)

of results. Who does not have the material parameters of products available, for whatever of the reasons specified above (material mixtures, large range of the refractive index, non-availability of the imaginary part, different crystal orientations or completely different particle roughness), may incline to the classical Fraunhofer approximation. In addition, if data sheets of raw materials for quality control show results using the Fraunhofer approach (e.g. when using the CILAS technique for decennia) one would not change the calculation method unfounded.

With materially pure and very small particles, in particular within the nanometer range, the equipment user will decide rather for the use of the Mie theory, particularly if the refractive index of its products is close to the refractive index of the liquid. From the ISO 13320 and own comparative investigations with a CILAS Lasergranulometer one can conclude: The differences between results of the Fraunhofer and Mie evaluation are larger the closer the refractive index of the particle is to the refractive index of the medium.

Thus some tradition for the application of the Fraunhofer method becomes also scientifically understandable: Many mineral particles possess a clearly larger refractive index than the water, in which they are measured. A larger difference in refractive index between the particles and the liquid, gives very good results when applying the Fraunhofer theory. No miracle thus that fine oxides, carbide, carbonates or sulfates, as well as other materials with a high refractive index, traditionally and also in the future will be characterized with the Fraunhofer method. CILAS analyzers are, according to standard, adjusted to Fraunhofer for such applications. With only one mouse click in the software however, one can re-calculate the raw data using the Mie theory by use of the refractive

indices from the database of the instrument or values from the literature implemented in that database. This is standard for all CILAS instrument versions!

## Conclusion

With the introduction of the world's first commercial laser diffraction instruments by CILAS, almost 40 years ago, a measuring method became generally accepted, which can characterize fine dispersed material systems in the form of emulsions, suspensions or as dry powders, fast and over a large particle size range. A standard is given with the ISO 13320, which does not contain an instruction for the measurement of each kind of sample, but the theoretical and practical level of knowledge is very well clarified. With the Fraunhofer and the Mie theory, two fundamental evaluation models are presented, which are selectable with a simple mouse click in the CILAS software and are used according to ISO 13320 with consideration of the questions and requirements explained above.

**Our Laboratory for Scientific Particle Analysis (LabSPA) has the measuring technique available with all options as wet and dry modules, integrated video microscopes, internal and external ultrasonic devices of different strengths, DT-300 for zeta potential measurements for the investigation of the stable ranges of a dispersion, small volume unit, dispersing agents, among other things. With our experiences for many kinds of materials, we can perform test analysis as well as commercial analysis and method development. Please check the list of possible analysis of LabSPA in this Particle World and do not hesitate to contact us to get help in comprehensive particle characterization.**

**New**

**The next generation: CILAS 990, CILAS 1090 and CILAS 1190 for excellent particle sizing and optional particle shape analysis**



**CILAS 990**

for the particle size range 0,2 - 500 µm

**CILAS 1090**

for the particle size range 0,04 - 500 µm

**CILAS 1190**

for the particle size range 0,04 - 2500 µm

Figure 1 a-c  
The new CILAS laser particle sizer generation: CILAS 990LD, CILAS 1064L and CILAS 1180LD with optional videomicroscope for additional particle shape analysis

CILAS state-of-the-art laser particle size analyzers include features such as the patented short optical bench, intuitive software interface and a 2-in-1 design, which effortlessly integrates wet and dry modes in the same system. With the new generation of particle sizers from CILAS there are additional reasons to decide for the products of the inventor of commercial laser diffraction instruments. The new instrument generation implements the traditional strengths of CILAS technology, as superior reproducibility and stability, with new design and features to further improve the possibilities for particle characterization.

The new CILAS line combines the traditional strengths of highest reproducibility in particle sizing with new features. The new CILAS generation of laser diffraction particle sizer will be presented at AICHEM 2009 exhibition (29. International Exhibition-Congress on Chemical Engineering, Environmental Protection and Biotechnology, Frankfurt/Germany, May 11-15, 2009) where QUANTACHROME presents a complete choice of analytical instruments for comprehensive particle analysis.

- There are three models available which continuously offer the best price-cost-relation according to your application:
- CILAS 990**  
for the particle size range 0,2 - 500 µm
- CILAS 1090**  
for the particle size range 0,04 - 500 µm
- CILAS 1190**  
for the particle size range 0,04 - 2500 µm

CILAS laser particle size analyzers meet the needs of many and various fields like cement, ceramics, manufacturing industries, pharmaceuticals, cosmetics, biology, food or environmental industries. With the new optional videomicroscope there are many ways for additional information about the particles (Fig. 2). In combination of a CILAS particle sizer with videomicroscope one can compare particle size diameter distribution from laser diffraction and from image analysis, but also particle surface and particle number distribution from both methods. This gives much more information e.g. for R&D applications.

Figure 2 shows the results and distribution curve of image analysis of about 5000 particles. With the optional videomicroscope of the new CILAS 990, CILAS 1090 and CILAS 1190 there are many possibilities for additional information to the laser diffraction method to determine particle size distributions according to ISO 13320. QUANTACHROME does not only offer the instruments to use this combination of particle size and particle shape analysis but offers also contract analysis from the Laboratory for Scientific Particle Analysis (LabSPA). Please check the list of possible particle size measurements from the LabSPA and contact us for use of our experience and technique.

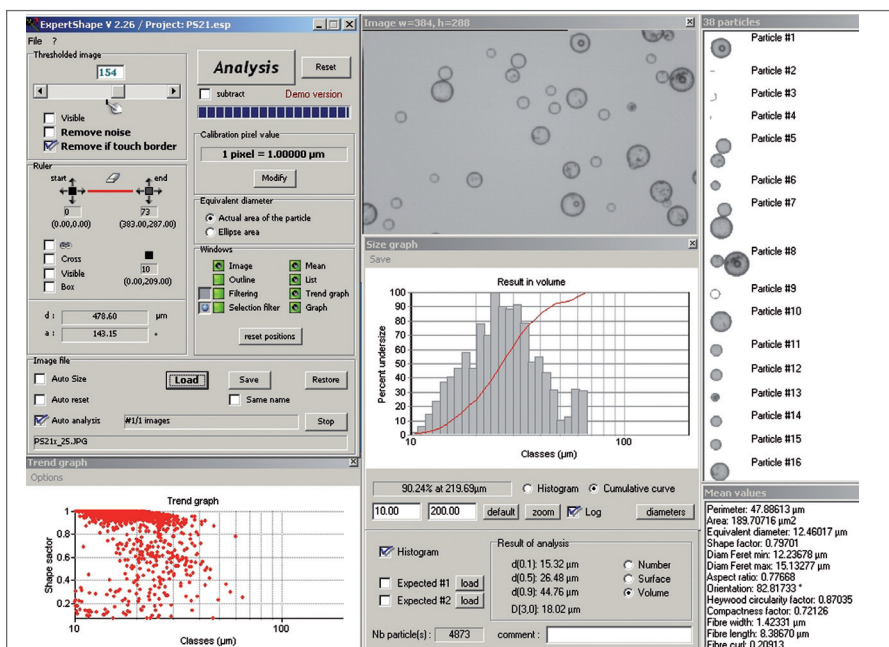


Figure 2  
CILAS EXPERT SHAPE Software for image analysis: Single Particle Analysis, calculation of median particle shape parameter and of trend graphs of shape parameters give additional information to the results of laser diffraction method according ISO 13320.



## About the advantages of water vapor adsorption using the HYDROSORB 1000 HT

The measurement of water adsorption of food, packaging materials, pharmaceutical products, adsorbents and other porous materials is an important method to carry out practical processes and surface chemistry of such substances. Figure 1 shows the water adsorption isotherm of sugar as an example in the food industry.

The measurement is carried out with the fully automated sorption analyzer HYDROSORB 1000 HT. The measurement principle is similar to the volumetric (manometric) gas adsorption. The essential differences of the HYDROSORB 1000 HT to other instruments and to the gas adsorption principle in general are described as followed.

- The HYDROSORB 1000 is a fast fully automated water vapor adsorption analyzer. "Fast" is in relation to the dosing algorithm and to the volumetric principle. In contrast to the classical gravimetric methods, there is no adsorption in a balance system that can cause additional equilibration time.
- The complete dosing system (manifold) inclusive all valves, lines and transducers is heated up to 100°C which prevents condensation and allows the determination of the water vapor adsorption. The higher the manifold temperature the better is the prevention of possibly condensation. With a working temperature of 100°C the HYDROSORB has substantial advantages in comparison to other instruments.
- The HYDROSORB is working according the patented NOVA sorption principle. Not only the void volume and pressure characteristics of the measurement cells are carried out by use of calibrated measurement cells, but the sorption behavior of each measurement cell is determined as well and subtracted as a zero measurement from the sample measurement.
- The HYDROSORB 1000 is especially useful for the determination of water vapor adsorption and desorption at different temperatures. It is available in two versions, Hydrosorb 1000 and HYDROSORB 1000 HT (Figure 2).

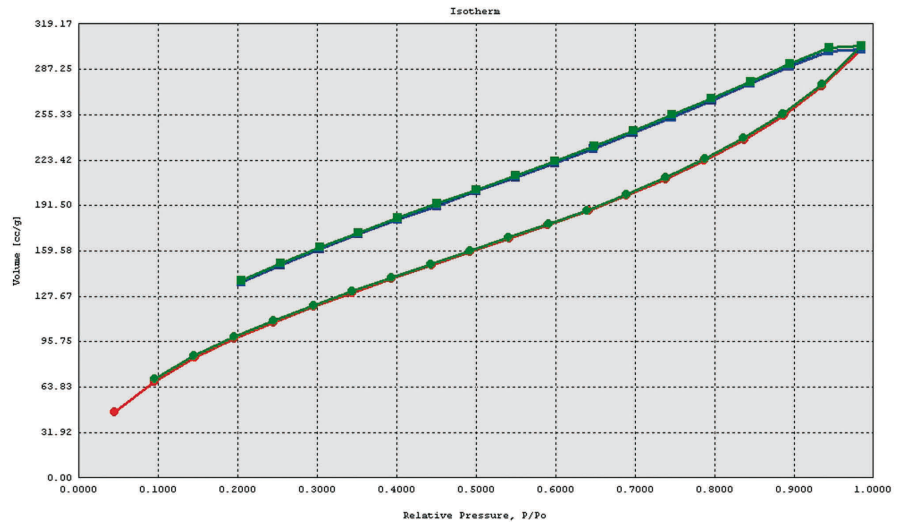


Figure 1 Water adsorption isotherms of sugar at 25°C (double measurement)



Figure 2  
HYDROSORB 1000 HT with a measurement range from 12°C to 85°C up to highest relative humidity

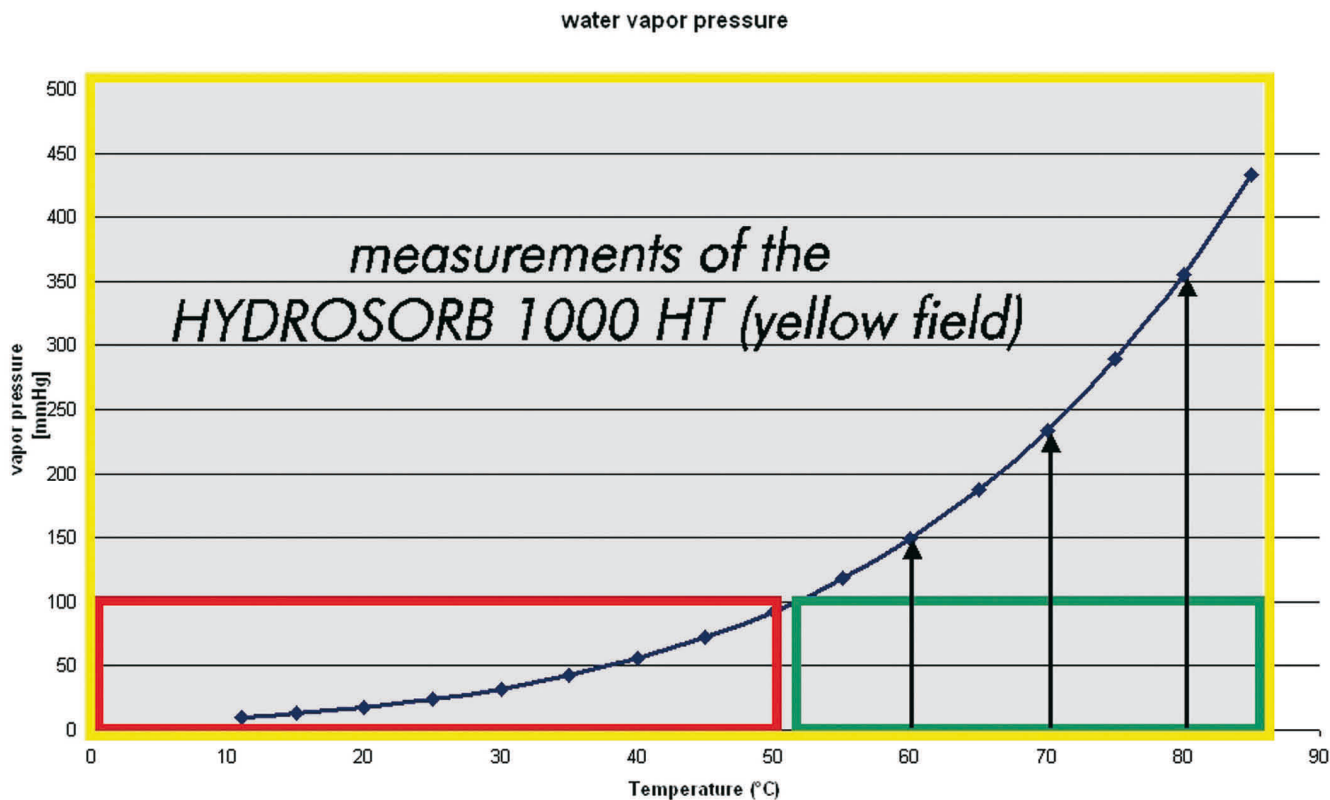


Figure 3  
Dependency of vapor pressure of water against temperature with limited measurement range of a 100 mmHg transducer (red and green area) and the additional possibility by use of HT option (yellow area)

Whereas the HYDROSORB 1000 is using a 100 mmHg transducer like other water sorption analyzers, the HT option (High Temperature) includes an additional 1000 mmHg transducer and several high temperature accessories for the measurement of complete isotherms up to 85°C. The requested relative pressure for a full isotherm measurement close to the saturation pressure  $P_0$  is above 100 mmHg, when the temperature increases over approximately 50 °C. Figure 3 shows the dependence of water vapor pressure against the measurement temperature.

It is shown with the  $P_0$  axis that it is only possible to measure close to the  $P_0$  value the red area with a 100 mmHg transducer. The  $P_0$  value increases significantly with higher temperatures so that complete isotherms are only accessible with a 1000 mmHg transducer instrument. For example, the saturation pressure at 80°C is about 350 mmHg so that with a 100 mmHg transducer only isotherms up to a humidity by  $100/350 \times 100\% = 29\%$  are measurable (green area), whereas the HT option provided measurements close to  $P_0$ . Accordingly the temperature range of the HYDROSORB 1000 can be extended to 12°C – 85°C. The dewar is equipped with a 2-leveled magnetic stirrer and warrants through thermal isolation temperature stability in the

high temperature range. The measurement temperature is measured directly in the dewar and can be displayed on the instrument monitor.

The HYDROSORB 1000 provides different routines and display opportunities of results. Usual displays are as well the dependence of adsorbed amount against relative pressure  $P/P_0$  as the relative humidity.

**With the HYDROSORB 1000 and its HT option there is a powerful instrument available with essential improvements and advantages in comparison to alternative volumetric systems. You are welcome to check this with test or contract analysis in our LabSPA.**



## News

### Gas adsorption with QUADRASORB SI: one sample – one dewar ... four samples – four dewars!

Adsorption measurements for surface and pore analysis provides the application of gas sorption as a standard method in many fields like the characterization of nanomaterials, ceramic powders and building materials, polymers, adsorbents and catalysts. Multipoint analyzers have the advantage of higher sample throughput in BET surface area and pore analysis. However considering some tasks in more detail, additional flexibility is necessary! Typical needs are often in demand:

- high sample throughput,
- solution of flexible tasks,
- parallel measurements of different samples and different pore size ranges,
- good price/capacity relation.

But what are the differences of more station instruments with just one dewar and such with single dewars for each sample? We can consider this newest development with a comparison of the QUANTACHROME NOVAe series and the QUADRASORB SI analyzers (figure).

Whereas the types of NOVAe series and QUADRASORB SI look like similar in many capabilities, a decisive difference is due to the number of dewars:

	NOVA e-series	QUADRASORB SI-series
Number of analysis ports	1, 2, 3 or 4	2, 3, 4
Helium-free NOVA-Mode	Yes	Yes
Helium Mode possible	Yes	Yes
Thermistor for short cold zone	Yes	Yes
Low pressure option for micropore analysis	No	Yes
Krypton option for small surfaces	No	Yes
<b>Number of dewars</b>	<b>1</b>	<b>4</b>

The consequences of different instrument configurations can be easily extracted. With various sample cells in just one dewar, which is realized in NOVA and other adsorption instruments, one has to wait for the end of every measurement until a new measurement is possible. For example, if a 3-station instrument is working on e.g. two BET measurements and one isotherm measurement, the next two BET samples cannot be started until the

time intensive isotherm measurement is finished as well. Differently on QUADRASORB: Every sample cell has its own dewar and also its own reference  $P_0$  cell and pressure transducer and can be reloaded as soon as the former measurement is finished. The software introduces a new analysis request directly in the existing measurement routine! In this way the sample throughput can be increased significantly whereas the flexibility is given to start a measurement at every time a free station is available.

With the QUADRASORB SI an adsorption analyser is available that due to its configuration of one dewar and reference for each sample which superiors every other instrument concerning flexibility and throughput – test it before you use just one dewar only for all of your different samples!

### New

The QUADRASORB SI is now also available with 3 and 2 sample stations to optimize your needs. The new models retain the beneficial “each sample has its own dewar and transducer” features, but at a cost which is attractive to laboratories with smaller but flexible throughput requirements. And if your future throughput increases, you can upgrade your system to 4 stations!



Figure  
NOVAe in comparison to QUADRASORB SI:  
Various sample cells in one dewar (right)  
and one dewar for each sample cell (left)

## News

### Fully automated characterization of catalysts:

#### CHEMBET PULSAR TPR/TPD

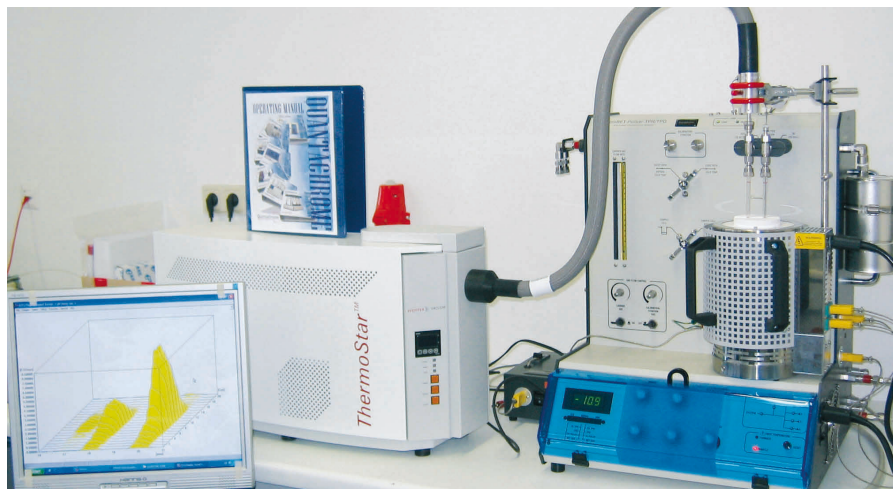
QUANTACHROME Instruments announces the launch of the new CHEMBET PULSAR TPR/TPD analyzer. The PULSAR combines affordability and automation and offers compact, bench-top catalyst characterization using automated flow methods of analysis.

The PULSAR builds on the reputation of the existing QUANTACHROME CHEMBET - combining its affordability with the automation of their renowned AUTOSORB-1C-TCD analyzer, and represents the very best value in catalyst characterization using automated flow methods of analysis, including pulse titration. Fully automated analysis sequences are programmed using the new TPRWin PC software. Titrations for metal area and dispersion determination are done now by use of a new automatic loop injector and automatic gas switching. Furnace temperature ramping provides for temperature programmed methods (TPR, TPD and TPO) and sample preparation, both

including rapid furnace cooling using forced air for higher throughput. The PULSAR retains the CHEMBET's proven TCD detector - both oxidation AND ammonia resistant, with stable current control for baseline stability and reproducible signals. Plumbed in stainless steel for maximum chemical compatibi-

lity, the PULSAR is ideal for use with a wide range of gases. High-temperature quartz sample cells are standard, as is the in-cell thermocouple providing accurate sample temperature measurements.

Options of the CHEMBET PULSAR TPR/TPD include a Quadrupole Mass Spectrometer for characterization of products of chemical temperature programmed reactions, and an external digital gas blender/mass flow controller.



**Figure**  
CHEMBET PULSAR TPR/TPD for fully automated catalyst characterization in combination with mass spectrometer for additional determination of the reaction products of temperature programmed reactions

## News

### Latest generation of automatic gas pycnometers:

#### ULTRAPYCNOMETER 1200e and PENTAPYCNOMETER 5200e

QUANTACHROME announces the launch of the upgraded and "connected" ULTRAPYCNOMETER 1200e. This latest version of the popular Ultrapycnometer now features ethernet connectivity and USB port.

New ethernet connectivity brings browser-based control and reporting capability including email. This density analyzer can even email you the report when the analysis is complete! The new on-board microprocessor enables not only the communication via web browser; it stores even the complete user manual as pdf-file. A new external power supply (similar to a power supply for notebooks) enables a better thermal stability even without the use of an external thermostat.

The ULTRAPYCNOMETER 1200e is available in the following versions: "standard" for density measurements on larger sample sizes (up to 135 cc), "micro" for smaller samples (less than 4.5 cc), "remote" for glove box operation, "T" for temperature controlled applications, and "Ultrafoam" for determination of open/closed cell content of rigid foams.

The five-station PENTAPYCNOMETER 5200e is also announced at the same time. It has the same ethernet and USB connection as the Ultrapycnometer and offers busy labs significantly increased sample throughput in only twice the footprint of the ULTRAPYCNOMETER.

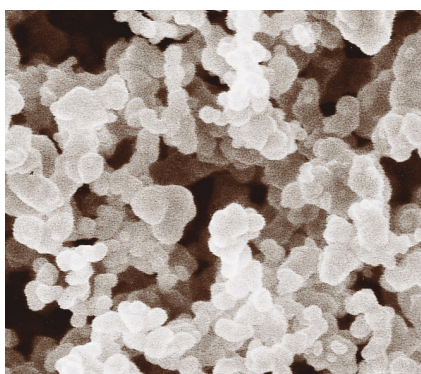
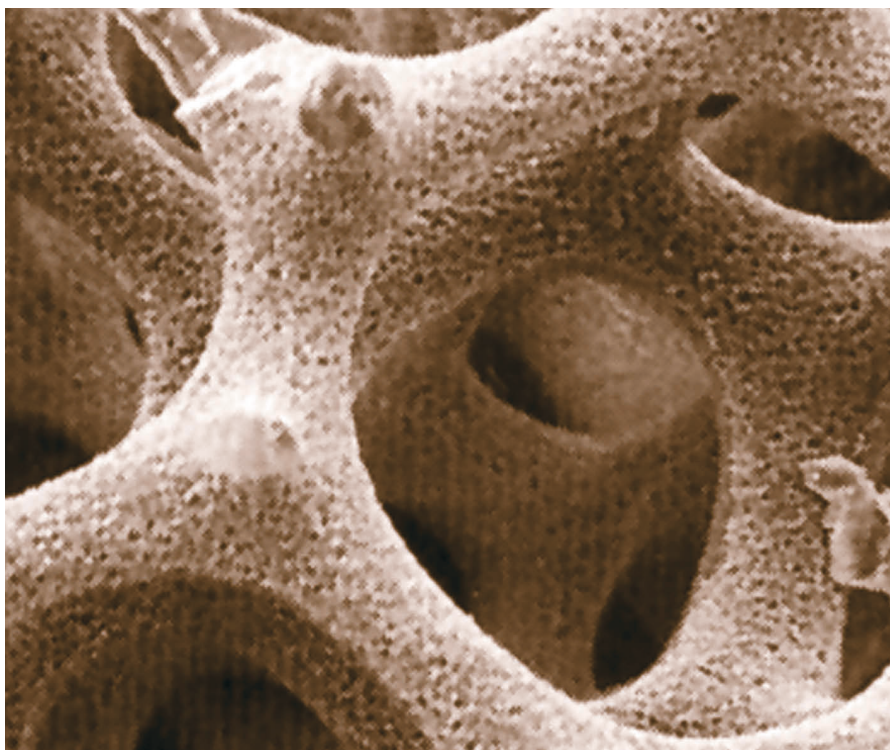


**Figure**  
For density measurement: the ULTRAPYCNOMETER 1200e (one port) and the PENTAPYCNOMETER 5200e (five ports)



## **The Laboratory for Scientific Particle Analysis (LabSPA)**

*Excellent results and reliable, fast reports as well as professional information*



**Figure 1**  
*Examples for sample types analysed in the Laboratory for Scientific Particle Analyses (LabSPA): Porous solids, colors and pigments, powders and high concentrated dispersions, nanoparticles*

The LabSPA is well known as reliable and experienced laboratory for scientific particle analysis. Many companies and institutes send a high number of samples to our LabSPA for test and contract analyses and comprehensive method development yearly. They use the long-term experience of our team to get professional results and reports.

The LabSPA provides test analyses of samples to search out the adequate analysis method to solve your application. However also the service for contract analysis, in case that your number of necessary measurements should not be substantial enough to make the use of own instruments economic, is a main field for the LabSPA. Our support is very valuable and appreciated in case that you have only single samples or regularly

batches of samples which we measure exactly by use of constant run conditions over the months and years. The Laboratory for Scientific Particle Analysis has all instruments of four product lines around the characterization of powders, dispersions and porous solids available and sends not only simple printed reports, but also a report with comments and explanations to understand the determined parameters and the methods.





Figure 2  
Partition of our Laboratory for Scientific Particle Analysis with the AUTOSORB series (right) for surface and pore analysis by gas adsorption and CILAS laser diffraction particle sizers

The Laboratory for Scientific Particle Analysis (LabSPA) is acknowledged for particle size measurements, surface and pore characterization methods and other analyses “around the particle”. We analyse your samples according to international and/or national standards and new developed innovative analytical methods. The LabSPA has the equipment available to divide your samples by use of the QUANTACHROME MICRORIFFLER to can follow the international norms not only regarding the analysis itself but also regarding the representative sample dividing.

You can load the necessary sample forms for the test and contract analysis respectively for the measurements for method development directly from our homepage [www.quantachrome.nl](http://www.quantachrome.nl), [www.quantachrome.dk](http://www.quantachrome.dk) or [www.quantachrome.eu.com](http://www.quantachrome.eu.com). You can also mention a necessary deadline to get the results according to your needs. If necessary you can clarify to get results overnight or as express service respectively. For more information about all the possible measurements of the LabSPA please use the fax answer sheet of this issue. Please check the following list for scientific particle analysis and do not hesitate to contact us ([ton.goverde@quantachrome.nl](mailto:ton.goverde@quantachrome.nl)).

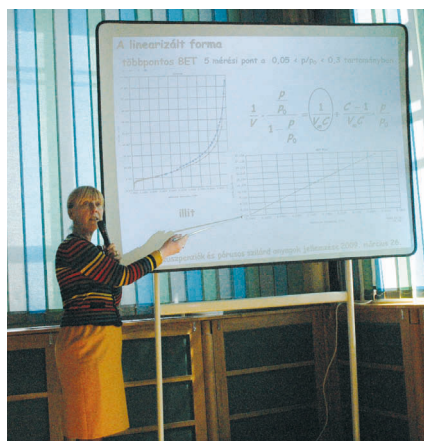


Figure 3 LabSPA experiences for seminars: Impressions from the QUANTACHROME Particle Seminar at University Budapest/Hungary in March 2009. Prof. Krisztina László and Heiko Sievers during their lectures about gas adsorption and particle size analysis; interesting exchange of ideas between the 60 participants also during the breaks.

## List of analyses for characterization of powders, porous solids and dispersions in the Laboratory for Scientific Particle Analysis (LabSPA)

No.	Method	Description
<b>Particle size analysis</b>		
90001	Particle size analysis / Laser diffraction method - wet	Measurement with CILAS granulometers according to ISO 13320-1 by means of water as dispersion medium. Particle size range 0,04 µm to 2500 µm. The exact dispersion (type of water, ultrasound etc.) according to customers requests.
96712	Particle size analysis / Laser diffraction method - dry	Measurement with CILAS granulometers according to ISO 13320-1 by means of dry dispersion. Particle size range 0,1 µm to 2500 µm.
90002	Particle size analysis / Laser diffraction method in isopropanol	Same as No. 90001 (ISO 13320-1), however measurement in isopropanol as dispersion medium.
90003	Particle size analysis / Laser diffraction method in other dispersion media	Same as No. 90001 (ISO 13320-1), however measurement in alternative liquids as dispersion media, use of Small Volume Cell (S.V.C.)
90004	Particle shape analysis / digital image analysis	Statistical evaluation of particle size and further geometric particle parameters like: aspect ratio, perimeter, surface area, equivalent diameter, etc., by CILAS granulometers. Evaluation range greater than approx. 1 µm.
90006	Particle size analysis with acoustic spectroscopy	Acoustic spectrometer (DT-1200 or DT-100), measurement of frequency dependend attenuation of ultrasound with calculation of particle size distribution, measurement in original sample concentration up to ca. 50 vol.-%, particle size range 5 nm to 1000 µm.
<b>Zeta potential</b>		
90007	Zeta potential measurement	Electroacoustic spectrometer (DT-1200 or DT-300), measurement of Colloidal Vibration Current (CVI) with calculation of zeta potential, measurement in original sample concentration up to ca. 50 vol.-%.
90008	Zeta potential measurement as function of pH (Titration)	Same as No. 90007 (DT-1200 or DT-300), however measurement at different pH values (pH range 0,5 to 13,5), titration and backtitration, determination of isoelectrical point.
90009	Measurement of dielectric permittivity	Measurement of dielectric permittivity as a degree of the electrical permeability of the dispersion
<b>Rheology and sound speed</b>		
96624	Extensional viscosity - acoustic	Calculation of the dynamic viscosity, of the compressibility and the sound speed
<b>Stability of liquid dispersions: Emulsions, Suspensions, Foams</b>		
90010	Stability (short-term)	Analysis stability of dispersion by optical scanning (TURBISCAN principle), short-term measurement
90145	Stability (short-term)	Analysis stability of dispersion by optical scanning (TURBISCAN principle), short-term measurement at 3 different temperatures
90011	Stability (long-term)	Analysis stability of dispersion by optical scanning (TURBISCAN principle), long-term measurement
90146	Stability (long-term)	Analysis stability of dispersion by optical scanning (TURBISCAN principle), long-term measurement at 3 different temperatures
90012	Heavy Fuel Characterisation	According to ASTM D7061

No.	Method	Description
<b>DRYING BEHAVIOR of coatings and films</b>		
90013	Drying behaviour: short-term characterization	Advanced drying analysis / Investigation of film formation by patent-pending optical A.S.I.I. technology (Adaptive speckle imaging interferometry), short-term measurement with HORUS
90014	Drying behaviour: long-term characterization	Advanced drying analysis / Investigation of film formation by patent-pending optical A.S.I.I. technology (Adaptive speckle imaging interferometry), long-term measurement with HORUS
<b>MERCURY POROSIMETRY - Pore volume / Pore size / Pore size distribution</b>		
90015	Pore size distribution / Pore volume	Mercury intrusion by use of POREMASTER: Pore size range ca. 1000 µm to 0,0036 µm pore diameter, pore volume and pore size distribution (DIN 66133), Mercury extrusion for additional analysis of pore structure.
90020	Expanded Porosimetry	Same as No. 90015, additional bulk density (raw density) (DIN 66137) and porosity in % from bulk density and density from helium pycnometry
<b>GAS ADSORPTION - Specific Surface Area</b>		
90021	Multipoint BET from nitrogen adsorption	Specific surface area measured with nitrogen at 77 K and calculated by use of multipoint BET equation, according to DIN ISO 9277 (Volumetric method). 5 point BET is standard, alternatives according to customer needs. Minimum 1 m <sup>2</sup> sample surface is necessary in sample cell, measurement by use of NOVA, QUADRASORB SI or AUTOSORB.
90022	1 point BET from nitrogen adsorption	Specific surface area according to 1 point BET method and measured with nitrogen at 77 K, according to DIN ISO 9277 (Dynamic method). Minimum 0,3 m <sup>2</sup> sample surface is necessary in sample cell, measurement by use of MONOSORB.
90023	Multipoint BET from krypton adsorption (to measure very small surface area)	Specific surface area measured with krypton at 77 K and calculated by use of multipoint BET equation, according to DIN ISO 9277 (Volumetric method). 5 point BET is standard, alternatives according to customer needs. Krypton adsorption is the BET method to determine surface areas smaller than 1 m <sup>2</sup> sample surface in sample cell, measurement by use of AUTOSORB or QUADRASORB SI Kr/MP.
90024	Micropore surface area / Micropore volume	Micropore surface area / Micropore volume measured with nitrogen at 77 K, analysis report contains BET surface area and micropore surface area and micropore volume calculated from t-method or Dubinin-Radushkevich-Equation according to DIN 66135-3 (Volumetric method), measurement by use of NOVA, QUADRASORB SI or AUTOSORB.
<b>GAS ADSORPTION - Pore volume / Pore size / Pore size distribution</b>		
90025	BET surface area / Pore volume / Average pore size	measured with nitrogen at 77 K, measurement by use of NOVA, QUADRASORB SI oder AUTOSORB.
90026	Standard mesopore size analysis (BJH method)	measured with nitrogen at 77 K, 20 points adsorption plus 19 points desorption. Maximum pore diameter is ca. 0,4 µm (limited by method of gas sorption). Analysis contains multipoint BET surface area (DIN ISO 9277), pore volume and BJH pore size distribution (DIN 66134), measurement by use of NOVA, QUADRASORB SI or AUTOSORB.
90027	High-resolution mesopore size distribution	measured with nitrogen at 77 K, 40 points adsorption plus 39 points desorption. Maximum pore diameter is ca. 0,4 µm (limited by method of gas sorption). Analysis contains multipoint BET surface area (DIN ISO 9277), pore volume and BJH pore size distribution (DIN 66134), measurement by use of NOVA, QUADRASORB SI or AUTOSORB.
90028	Micro pore size analysis	measured with nitrogen at 77 K with AUTOSORB-1-MP with 1 torr pressure transducer configuration, argon at 87 K or carbon dioxide at 273 K on request. Analysis report contains multipoint BET surface area (DIN ISO 9277), pore volume and micropore pore size distribution (basis is DIN 66135) according to the most appropriate calculation models (DR, DA, HK, SF, DFT, GCMC).



No.	Method	Description
90029	Micro pore and mesopore size analysis	measured with nitrogen at 77 K with AUTOSORB-1-MP with 1 torr pressure transducer configuration, ca. 70 points adsorption plus ca. 20 points desorption. Analysis report contains multipoint BET surface area pore volume, mesopore size distribution according to BJH (DIN 66134) and micropore pore size distribution (basis is DIN 66135) according to the most appropriate calculation models (DR, DA, HK, SF, DFT, GCMC).
96884	Micro pore analysis at carbon black	Determination by carbon dioxide-sorption (approx.40 measuring points adsorption) at 273 K (other temperatures possible on request); analysis of pore size distribution, pore volume and specific surface area by means of NLDFT, pore size range 0,35 nm to 1,5 nm.
96885	Isotherm measurement (non-corrosive gases)	Sorption measurement of any (non-corrosive) gases as adsorption isotherm. Pressure range and measurement temperature by arrangement; the result will be the physisorbed quantity vs. pressure .
90030	Calculations for gas adsorption measurements (data from other adsorption instruments) according to the most modern calculation methods	Calculations for gas adsorption measurements (data from other adsorption instruments) according to the most modern calculation methods, calculation of pore size distribution from ASCII-Dateien from other instruments, data analysis by use of QUANTACHROME-AUTOSORB software, calculations with Density Functional Theory (DFT) and/or Monte-Carlo-Simulation (GCMC).

### WATER VAPOR SORPTION

90081	Water vapor sorption isotherme	Determination of water vapor sorption up to 96 % humidity with 10 points of different humidity according to customer requests, temperature range from 12°C to 85°C, adsorption and desorption, measurement with HYDRSORB.
90082	Water vapor sorption - adsorption only	Same as No. 90081, but only adsorption isotherm
90083	High resolution water vapor sorption isotherme	Same as No. 90081, up to 40 isotherm points of different humidity.
90084	Water vapor sorption isotherme, behavior of hysteresis	Same as No. 90081, however with repeated adsorption and desorption, measurement with HYDRSORB.
90085	Water vapor sorption isotherme incl. Determination of heat of adsorption	Same as No. 90081, additional adsorption at different temperature according to the customer's request, additional calculation of water vapor sorption enthalpy according to Clausius-Clapeyron equation, measurement with HYDRSORB.

### CHEMISORPTION / TPD / TPR / TPO

90086	Chemisorption analysis	Determination of monolayer capacity, dispersion and active surface area (DIN 66136-1), adsorptive gases according to customers request: H <sub>2</sub> , NH <sub>3</sub> , CO, CO <sub>2</sub> , O <sub>2</sub> —> please specify
90087	TPD / TPR / TPO	Customer specified temperature programmed Desorption/Reduction/Oxidation, determination of energy of activation and/or desorption.

### DENSITY

90088	Density (true density)	Helium pycnometry at 25°C (DIN 66137-2), other temperatures on request, use of nitrogen for special applications, measurement with ULTRAPYCNOMETER.
90089	TAP density	Measurement with DUAL AUTOTAP (DIN ISO 787/11) to measure the sample volume (density) with 500 taps and/or until the volume of the powder becomes constant.
90114	Bulk Density	For powders and granulates by graduated cylinder
90090	Apparent Density	Gravimetric determination by mercury pycnometry at ambient pressure (open pore higher 15 µm neglected for bulk density).
90091	Foam characterization	Open and Closed cell content of rigid foams, foam compressibility in pressure range 2 - 20 psi (ca. 0,13 - 1,3 bar), DIN EN ISO 4590.

## **Determination of particle size, zeta potential and other parameters in concentrated dispersions**



Concentrated dispersions can be analyzed by acoustic and electroacoustic spectrometry. The main advantage is clear: Dilution can change your dispersion, especially the surface properties of particles and so the zeta potential. However both methods are also applicable in nanometer

range where e.g. laser diffraction has its limitations for determination of particle size. The following list of newsletters from DISPERSION TECHNOLOGY presents the many different applications for the acoustic and electroacoustic spectrometry to characterize very fine particles in original concentrations.

No.	Newsletter
1	Characterization of <b>CMP slurries</b> , Part 1. Resolution of Acoustics in determining large particles content
2	Electroacoustics for <b>Concentrated Dispersions</b>
3	Acoustics and Electroacoustics for <b>Ceramics</b>
4	Acoustics and Electroacoustics for <b>Emulsions</b>
5	Electroacoustics Phenomena in <b>Concentrated Dispersion</b>
6	Characterization of <b>Mixed Dispersions</b> by means of Acoustic Spectroscopy
7	Surfactant Titration of Kaolin Slurries using <b>Zeta Potential Probe</b>
8	Particle Size Distribution and <b>Micro-Rheological Properties</b> of the Structured Concentrated Dispersions
9	Influence of Chemical Composition on the Acoustic Properties of <b>Homogenous Liquids</b>
10	A New Way To Characterize Stability and Performance of <b>Cosmetic Emulsions and Suspensions</b>
11	Announcing Book Publication - <b>ULTRASOUND for CHARACTERIZING COLLOIDS - Particle sizing, Zeta Potential, Rheology</b>
12	Characterization of <b>CMP slurries</b> , Part 2. A new composite method comprised of Acoustic and Electroacoustic Spectroscopy and Sedimentation monitored with Ultrasound
13	Ultrasound for characterizing <b>liquid based food products</b> . Part 1. Acoustic spectroscopy
14	Ionic properties of so-called "non-ionic" surfactants in non-polar liquids
15	Evolution of <b>water-in-oil emulsion</b> controlled by droplet-bulk ion exchange. Acoustic, electroacoustic, conductivity and image analysis
16	Representative <b>On-line Measurement</b> of Comminution Results for <b>Nanogrinding in Stirred Media Mills</b>

Both the acoustic and electroacoustic method are described in the book "Ultrasound for Characterizing Colloids" from A. Dukhin and P. Goetz. However, the basics are published also in our Particle World 3 from C. Oetzel; you can get this Particle World free of charge from our homepage [www.quantachrome.eu.com](http://www.quantachrome.eu.com) or please send your request to [ton.goverde@quantachrome.nl](mailto:ton.goverde@quantachrome.nl), so that we send the printed issue Particle World 3 free of charge to you.

Three basic models of DT-instruments are available: DT-1200 for particle size and zeta potential, DT-300 for zeta potential and DT-100 for particle size. There are several options to implement e.g. in the DT-1200 to make it to the most powerful tool in characterization of high concentrated dispersions:

- pH and temperature
- non-aqueous option
- conductivity in aqueous solutions
- conductivity in non-aqueous solutions
- rheology
- electrical permittivity.

Key benefits of the DT-instruments for determination of particle size and zeta potential are

- no dilution needed, measure samples as is,
- wide particle size range,
- small sample volume,
- no particle size calibration required,
- measures particle size even for extreme cases of conductivity, e.g. Portland cement,
- works even with conducting particles,
- particle size measurement does not require charged particles,
- works with mixtures of dissimilar materials, e.g. alumina / zirconia ceramic slip,
- measures particle size in polymer solutions with extremely high macroscopic viscosity.

Sound pulses are transmitted through the sample for determination of **particle size**. The attenuation of these pulses is measured over a wide range of ultrasonic frequencies. The particle size is deduced from the measured spectra using software, which incorporates a detailed knowledge of the physical basis of acoustic attenuation in concentrated polydisperse systems. See Particle World 3 for more information.

The double layer is disturbed by an ultrasonic wave for **zeta potential** measurement. The displacement of the ionic cloud with respect to the surface creates a dipole moment. The sum of these dipole moments over many particles creates an electrical field, which is sensed by a receiving antenna immersed in the sample. See Particle World 3 for more information.

Do not hesitate to contact [ton.goverde@quantachrome.nl](mailto:ton.goverde@quantachrome.nl) or [christian.oetzel@quantachrome.de](mailto:christian.oetzel@quantachrome.de) for more information and for questions regarding testing your application. We have the instruments available in the LabSPA to demonstrate the capabilities and to test your samples in original concentration.

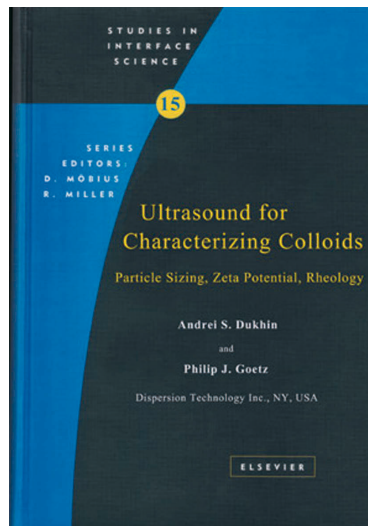


Figure 1  
"The Book" about Ultrasound for Characterizing Colloids by A. Dukhin and P. Goetz

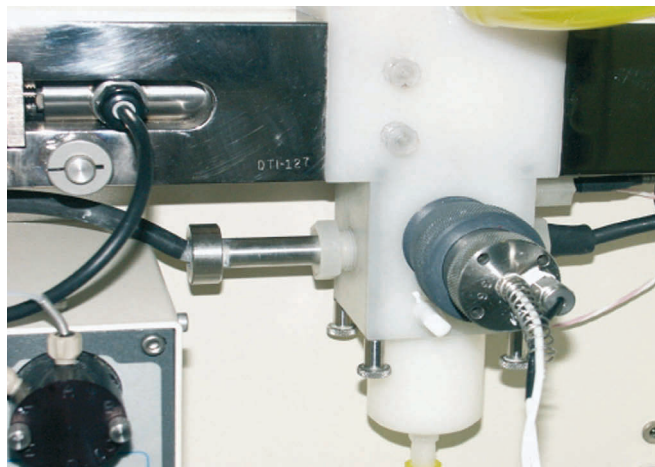


Figure 2 a-b  
The DT-1200 as the unique analytical instrument for characterization of concentrated dispersions and the zeta potential probe DT-300 as high quality zeta potential instrument



## **QUANTACHROME instruments in the food industry**

The food industry is surely not the key industry for the broad range of QUANTACHROME analytical instruments in the field of particle characterization, because a great many of our instruments are working in ceramic and building material R&D and quality control and many other different fields. However, if one think about the following short list of analytical methods it becomes clear that very different methods and instruments from QUANTACHROME are used in food industry too:

- Particle size analysis by use of laser diffraction according ISO 13320
- Particle size analysis according ISO 9276-6
- BET surface area determination according ISO 9277
- Pore size distribution by use of gas adsorption and mercury porosimetry from about 3 Nanometer up to 1 millimetre
- Determination of density and of tap density according ASTM and DIN norms
- Adsorption and desorption of water vapour

The LabSPA (Laboratory for Scientific Particle Analysis) has all these methods, the know-how and the analytical instruments, for the determination of the listed parameters available. In this way we can measure food samples beginning with professional sample dividing by use of the QUANTACHROME-MICRORIFFLER and finishing with the professional report including discussion of the results. Especially in economical situations complicated for many companies, it can be very helpful to can send some samples to the LabSPA for contract analysis or method developments, if the situation or the number of samples does not allow an own investment in analytical equipment.

One of the traditional analytical methods for various sample types in food industry is the determination of tap or tapped density. Tapped density is a routine measurement in all industries producing, handling and processing



*Figure*  
AUTOTAP for measurement of TAP density, CILAS with videomicroscope for particle size and shape, NOVA for determination of BET surface area and HYDROSORB for water vapor adsorption measurements



powders. The TAP-instruments from QUANTACHROME are working according many international norms, e.g.

- ASTM D4164 Catalyst and Catalyst Carriers
- ASTM B 527 Metallic Powders and Compounds
- ISO 787-11 pigments
- ISO 3953 Metallic powders
- ISO 8460 (Instant coffee)
- ISO 8967 (Milk powder)

In food industry, as a typical example for the importance to measure the tapped density, there are various applications for the AUTOTAP and DUAL AUTOTAP instruments from QUANTACHROME. Please see the following common examples for typical instrument users in food industry:

- Ground roasted coffee, instant coffee, instant tea
- Milk powder, infant formula
- Milled flour, cake mixes
- Ground spices, natural or artificial flavourings

- Corn, other meal grits (coarse granules)
- Sugars, sweeteners, cellulose fillers

Do not hesitate to contact us for further information or use the fax answer of this PARTICLE WORLD, e.g. to get more information about the instruments to measure Tap-Density, to divide samples for analytical measurements (Sample dividing by use of MICRORIFFLER) or particle sizing by use of the new generation of CILAS laser granulometer for particle sizing and particle shape analysis.

**Ground roasted coffee, instant coffee, instant tea**



**Corn, other meal grits (coarse granules)**



**Milk powder, infant formula**



**Sugars, sweeteners, cellulose fillers**



**Milled flour, cake mixes**



**Ground spices, natural or artificial flavourings**





## Invitation to the seminar

### "Characterization of powders, dispersions and porous solids"

Tuesday, 13<sup>th</sup> October, 2009 in Delft/NL



9.00	Greeting and overview about complex characterization of powders, dispersions and porous solids
9.15	Particle size and particle shape analysis with new generation of laser diffraction instruments and new videomicroscope option
9.45	Overview about new developments in the fields of density measurement
10.00	Coffee break
10.30	J. Groen (Delft Solid Solutions): Preparation and detailed characterization of combined micro- and mesoporous zeolites
11.00	Micro- and mesopore analysis by use of modern calculation methods and use of different adsorptives at different temperatures
12.00	Lunch break
13.30	Pore analysis with mercury porosimetry: Basics, model assumptions, traditional and state-of-the-art analysis and interpretations.
14.00	Colloidal dispersions in original concentration: a.) Analysis of nanoparticle size from attenuation of ultrasound in concentrated systems b.) Zeta potential in concentrated dispersions and its measurement by using electroacoustic method
14.45	Complex characterization of different systems on the basis of practical examples (pharma, catalysts, ceramics, adsorbents, geology, building materials, food etc.)
15:30	Coffee break
16.00	Demonstration of DT-1200 for particle size and zeta potential in concentrated dispersions, and Cilas 990 with videomicroscope for particle size and shape analysis
16:45	Final discussion
17.00	End



**P**owders, dispersions and porous solids represent important raw materials and intermediate and endproducts in many industrial fields. Depending on the application the knowledge of certain characteristics of these substances is indispensable to gain process control and optimization. Among the most important parameters are particle size and shape, zeta potential of the dispersion in original concentration, density, the specific surface area as well as pore size and pore volume.

Analysis of various types of particles of different fields will be discussed regarding the way to analyse size by means of laser diffraction method and acoustic spectrometry. Alongside with the theoretical approach especially the fields of application and advantages of both methods will be demonstrated based on practical examples. It will be showed that there are analysis methods available to can characterize dispersions in original concentration and up to down 5 Nanometer.

Gas adsorption and mercury porosimetry will be discussed in theory as well as demonstrated on the basis of practical experience out of different fields of application. Both traditional and state-of-the-art calculation methods will be reviewed. The practical part of the seminar will demonstrate gas adsorption instruments, as the QUADRASORB, combination of laser diffraction and particle shape analysis and the DT-1200, which can analyse concentrated dispersions by measuring particle size, zeta potential and other parameters.

The participation to the seminar is free of charge. Information material, lunch and catering throughout the seminar-breaks are organized and paid by QUANTACHROME. The number of participants is limited. The seminar will be held in English. We look forward to see you in Delft!

## Registration

Please use the enclosed fax answer for your registration to the seminar. For questions please contact Ing. Ton A.C.M. Goverde.

### QUANTACHROME

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Fax: +31 168 405113  
ton.goverde@quantachrome.nl

We kindly ask you to forward this information to colleagues if you yourself cannot participate or consider others more immediate concerned.



**Figure 1**  
Dr. D. Klank (right) during the award ceremony of the poster price, sponsored by QUANTACHROME, during Hungarian chemical conference in Siofok in 2008



**Figure 2**  
Dr. N. de Jaeger (International Fine Particle Research Institute) during his lecture at QUANTACHROME particle seminar in Zevenbergen/NL in 2007

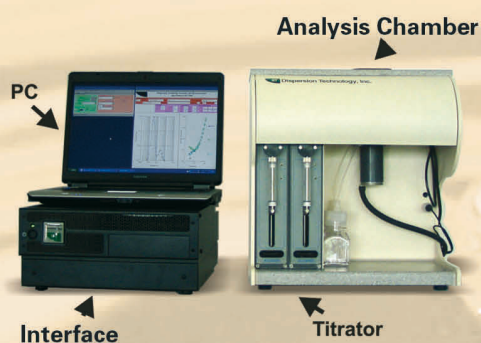
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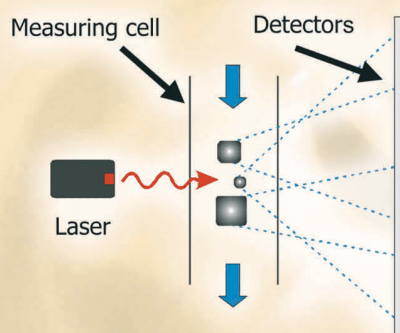
# Characterization of Dispersions



Particle size in concentrated Dispersions with the DT-1200



Zeta potential of concentrated Dispersions with the DT-300



Particle size in diluted Dispersions with the new CILAS series



Optional particle shape analysis with CILAS 990, CILAS 1090 and CILAS 1190

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