PARTICLE SIZE ANALYZER CILAS 920,930e,1064 & 1180 USER MANUAL





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USER MANUAL UPDATES:

This page contains the history of the CILAS particle size analyzer user manual. The first version was published in 2001 under version number NT-1503192-31. The table below shows the changes in each version:

Creation Complete update

After each update, a new version number is give to the user manual and the following information will be noted in this section:

- the new version number,
- the modification date,
- a list of what has been modified.

This allows users to be informed of all the modifications to this manual made by CILAS.

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ABOUT THIS MANUAL:

PURPOSE:

This manual covers the utilization of your new particle size analyzer model manufactured by CILAS company.

ORGANIZATION:

The following is a guide to the organization of this manual.

- ✓ PART 1: IDENTIFICATION OF THE PARTICLE SIZE ANALYSER

 This part gives information on the manual, the particle size analyzer and the optional accessories.
- ✓ PART 2: SPECIFICATION OF THE PARTICLE SIZE ANALYSER

 In this part, we have all the information concerning the technical specifications.
- ✓ PART 3: DEFINITIONS

This part describes the theory and the vocabulary of laser diffraction.

- ✓ PART 4: INSTALLATION OF THE PARTICLE SIZE ANALYSER All advice for the set of the instrument is described.
- ✓ PART 5: MEASURES WITH THE PARTICLE SIZE ANALYSER

 All the advices for a good measurement with the instrument are given into that part.
- ✓ PART 6: MAINTENANCE

In this part, you will find all the maintenance operations which can be made by users.

✓ PART 7: SPARE PARTS

You will find at spare parts for the maintenance operations.

✓ PART 8: WITHDRAWING FROM SERVICE

Finally, the operations for the withdrawing of the instrument are described.

SUGGESTIONS:

To submit suggestions on this manual or on a product, please contact us at:

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SCHEMATIC SUMMARY:

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PART 1: INDENTIFICATION OF THE PARTICLE SIZE ANALYZER



1.1- SCOPE OF MANUAL:

This Particle Size Analyzer User Manual covers installation, use and maintenance of CILAS particle size analyzer models CILAS 920, CILAS 930e CILAS 1064 and CILAS 1180.

This manual is written for technicians who are thoroughly familiar with instruments used in laboratories for physicochemical characterization of materials. This user manual is available on the USER CD-ROM provided with your particle size analyzer in the directory "MANUALS". The manual is available into pdf format and can be print if necessary.

Keep in mind that this document can not copied, disclosed or transmitted without written permission of CILAS.

1.2- IDENTIFICATION OF THE EQUIPMENT AND DOCUMENTATION:

CILAS particle size analyzers are designed to measure the particle size of powder matter ranging from $0.04~\mu m$ to $2500~\mu m$ (depending of the model). Our systems allow the user to measure particles of many different sizes without changing the optics or the calibration of the equipment.

The particle analysis is based on diffraction of a light source by the samples under analysis. The system requires the use of a monitor connected to an IBM PC or compatible for processing the results. Optional pieces of equipment (sample feed, CCD camera) can extend the particle size analyzer's capabilities.

This particle size analyzer user manual explains how to use the particle size analyzer; it does not cover optional equipment. It is a part of a group of manuals which describe all the specific functions of the particle size analyzer. Installation or maintenance are covered in this manual and are also described in more detail in other manuals available on your USER CD-ROM. Keep in mind that users are only authorized to perform preventive maintenance.

1.3- ACCESSORIES:

There are a number of accessories available for your CILAS particle size analyzer. If you have a specific measurement need, an accessory can solve your problem and improve your measurements. Accessories available for your system include:

- autosampler: improves your productivity when performing routine analysis (controls, production, etc),
- small volume unit: a device for reducing the amount of sample and solvent necessary to perform a measurement,
- video camera: allows users to see the shape and the sample dispersion.
- alcohol regenerator: reduces the amount of solvent necessary for analysis, which results in less waste removal.

"The Particle Expert" software controls all of the CILAS accessories. If you need additional information on any of these accessories or would like a quotation, please speak to your local CILAS distributor or directly to CILAS. We will be happy to answer any of your questions. Information is also available on our website (www.particle-size-analyser.com).

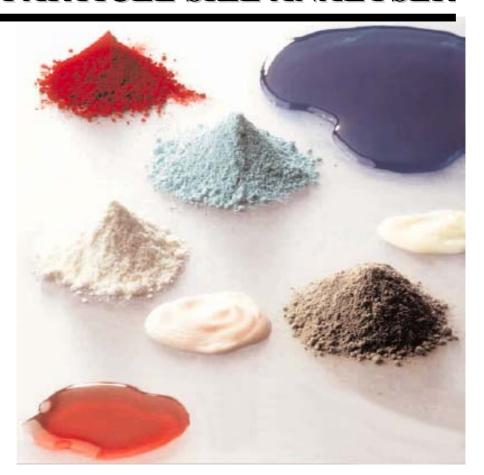
1.4- DECLARATION OF CONFORMITY TO THE NORMS:

Certificates of conformity for your CILAS particle size analyzer are provided in the appendix of your USER CD-ROM in the directory "CERTIFICATES". The following is a list of all the declaration of conformity, certificates of compliance, and material data sheets included on the CD ROM:

- Company certification:
 - ISO 9001 certificate,
- Particle size analyzer specific norms:
 - CFR 21 part 11 compliance,
- CE norms:
 - Electromagnetic compatibility certificate,
 - Low voltage conformity certificate,
- Certificates of security:
 - Laser security certificate,
 - Material Safety Data Sheet (MSDS),

If you need more information about any of these items, please contact CILAS.

PART 2: SPECIFICATIONS OF THE PARTICLE SIZE ANALYSER



2.1- GENERAL FUNCTIONS, RANGES AND UTILIZATION:

Particle size can be measured based on light diffraction. To analyze submicronics particles, CILAS uses laser diffraction. A particle size analyzer is used to determine the size distribution of a powder, a suspension or an emulsion. Using a particle size analyzer, you can very quickly visualize the shape of your particles and have an idea of the sample's dispersion. CILAS provides a complete solution with three products corresponding to three different measurement ranges.

CILAS offers a series of particle size analyzers with measurement ranges between 0.04 μm to 2500 μm . The figure below shows the CILAS model numbers and their corresponding measurement range:

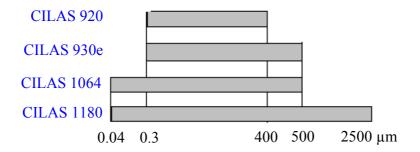


FIGURE 1: Range of CILAS particle size analyzers

Laser diffraction is now a very common and widely used method for particle characterization. With over 30 years of experience and knowledge, CILAS is the best choice to be your partner in particle analysis.

2.2- CHARACTERISTICS OF THE PARTICLE SIZE ANALYSER:

Compared to others systems or methods, CILAS particle size analyzers have many advantages. Our instruments are compact with a high level of performance. The most important characteristics of CILAS system are:

✓ A precise and reliable system:

CILAS uses a short bench concept which means users do not have to worry about optical alignment. The optical short bench is only available from CILAS and provides more reliable measurements. Its robust design ensures reliable operation, even in a hazardous industrial environment

✓ A fast and easy-to-use system:

The user simply has to introduce a sample into the particle size analyzer. A few seconds later, results will appears on the computer screen. CILAS particle size analyzers are designed to ensure proper dispersion (liquid or dry mode) and a reliable measurement without effecting the integrity of the particles.

✓ A user-friendly interface:

Making measurements with a CILAS particle size analyzers is made easy by using "The Particle Expert", a Windows[®] compatible software. "The Particle Expert" software is available in several languages and is available exclusively from CILAS. It allows users to measure, save and store data easily. Its interface is very easy because it uses a mimic screen, which shows pictures of each part of the particle size analyzer.

✓ A multi-level interface:

"The Particle Expert" provides three different password protected levels of security. The system is compliant with standard CFR 21 part 11. Depending on whether you are registered as an operator, an analyst or a controller, you can make measurements, write procedures or perform maintenance on the particle size analyzer.

All these characteristics ensure that CILAS particle size analyzers are reliable and precise laboratory instruments. They make CILAS particle size analyzers the best choice as your partner in particle analysis.

2.3- SYMBOLS AND SECURITY RULES:

In this chapter, the security symbols will be explained to the user and basic recommendations will be made in order to ensure the safety of users and maintenance technicians.



WARNING:

Anybody who will work with or on the CILAS particle size analyzer must respect all these safety recommendations. The operator in charge of maintenance must have the necessary skills and information to understand the operation of an electromechanical device. All users must follow the safety rules and policies of their company.

2.3.a)- Symbols:

Laser and electrical safety symbols are affixed on the outside and on the inside of the instrument. If one of the symbols is missing, it must be replaced with a new safety sticker.

✓ Laser symbols:

On the back of the instrument (figure 2), there are two labels in French and in English specifying the laser source class (1) and its certification of compliance with standard CEI 825 ADD 1 dated 17/09/1990.

Two yellow black edged labels (figure 3) located on the front of the optical block specify the laser radiation hazard, maximum laser diode power (3 mW), wavelength (830 nm) and device class (3B) when the cover's safety interlock is de-activated. A yellow black edged triangle (figure 3) with the laser danger symbol is affixed to the optical block tube.

✓ Electrical symbols:

There are six yellow black edged triangles with the electrical danger symbols affixed to elements presenting an electrocution hazard. The location of these symbols is as follows:

- transformer module (figure 5),
- ultrasound board (figure 4),
- distributor board (figure 4),
- power line filters (figure 4),
- board rack (figure 5),
- fan (figure 5).

If one of these symbols is missing, it must be replaced with another sticker which you can order from CILAS (see part 7).

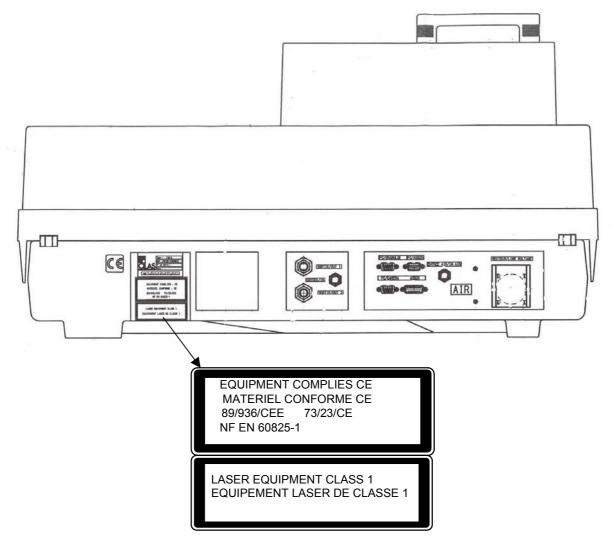


FIGURE 2: Back view

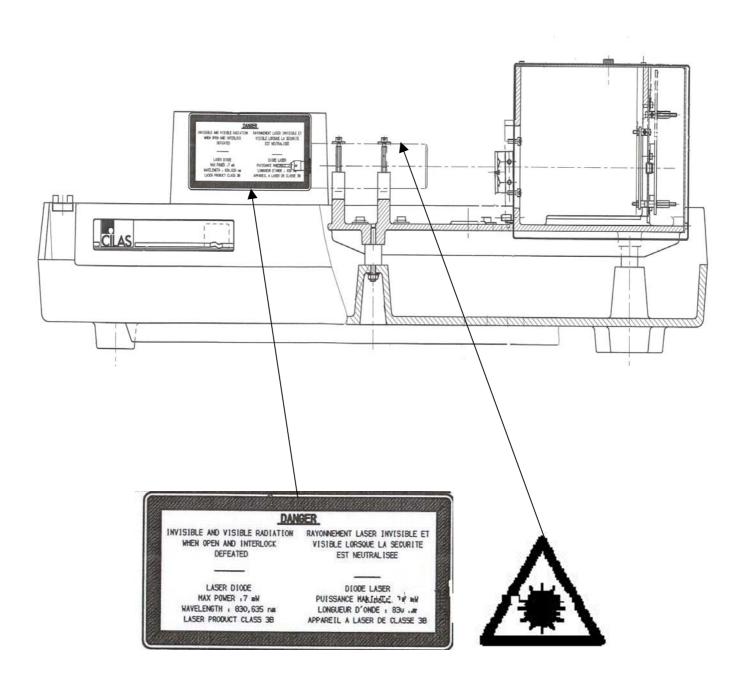


FIGURE 3: Front view

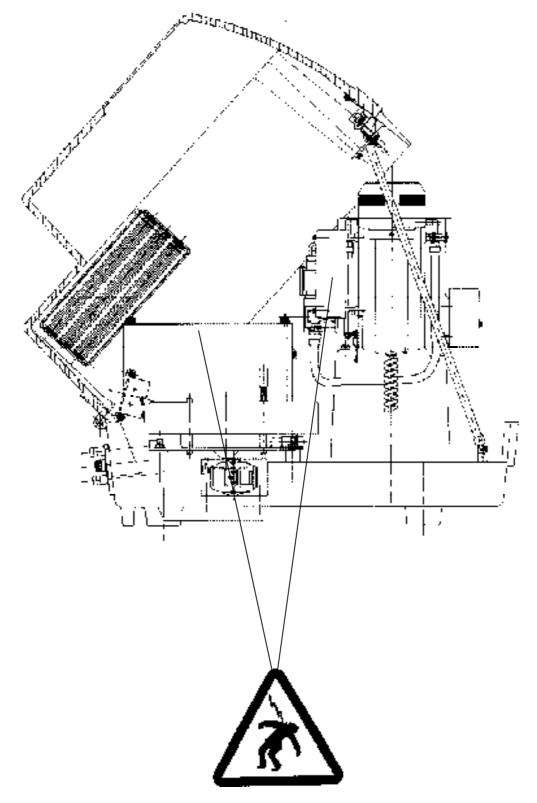


FIGURE 4: Side view

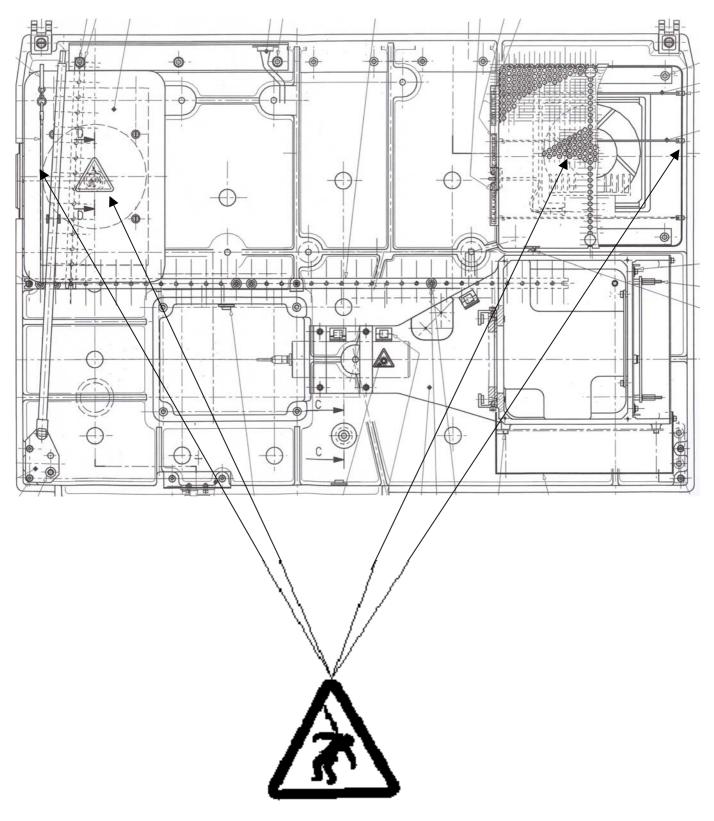


FIGURE 5: Top view

2.3.b)- Safety Rules:

The CILAS 920, 930e, 1064 and 1180 particle size analyzers comply with safety standard CEI 825 as per ADD1 dated 17/09/1990.

✓ Laser safety:

When the cover is closed, the operator is protected from the laser beam; therefore no additional protection is needed. This laser product belongs to Class 1 under standard CEI 825. If the cover is open, there is no accessible radiation because of safety interlock.

The general rules for laser safety are:

- Never look at the beam facing the source, either with the naked eye or with an optical instrument,
- Never place a reflective surface in the beam's path,
- The use of safety goggles is mandatory when any one of the above rules cannot be followed during maintenance operations.



WARNING:

Failure to the follow general rules could result in the emission of laser radiation, which can be harmful and caused permanent eye and skin damage. Safety goggles are required at all times when the cover is opened and the safety interlock is bypassed.

✓ Dangers presented by electricity:

The equipment described in this manual runs at high voltages that are potentially lethal. A voltage of 110 V can result in death. At all times, personnel must comply with safety provisions:

- Never perform any operations without first cutting off the source of high voltage,
- Be especially cautious with respect to safety systems such as circuit breakers, pull-out racks or doors. First make sure that the safety systems are working correctly. When carrying out any operation inside the equipment, cut off all the supply sources,
- Remember that safety systems are provided only for parts which are opened as part of routine maintenance and that dangerous circuits can still be encountered when the equipment is disassembled,
- In certain cases, dangerous voltages can still exist after the equipment has been shut down because of charged capacitors. These must be discharged before carrying out any work on the equipment. Wait several minutes in order to be sure the capacitors have fully discharged.

- YOU MUST NEVER BE ALONE WHEN OPENING A COMPARTMENT TO CARRY OUT INSPECTION OR REPAIRS ON EQUIPMENT PRESENTING A POTENTIAL HAZARD.



WARNING:

Failure to the follow general rules could result in electrocution.

2.4- GUARANTEE:

Like each product, the particle size analyzer has its own warranty. The start date of the warranty is the date that CILAS headquarters receives your end-user list.



NOTE:

The "end-user" list must be returned to CILAS. CILAS recommends you work with your dealer to sign and send the end-user list. Once the end user document is received, CILAS will inform you of the start of your warranty.

The warranty applies only if the product is used in standard conditions and does not include consumables like tubes, O-ring, etc. Parts are only guaranteed for defects and not for breakage due to non-standard conditions of use (like improper operating conditions, not respecting safety rules, etc). During system installation, use and maintenance, the guarantee would be invalid for reasons such as:

- connections at the user's factory are not standard (electric, water and air connections),
- installation is not done by a trained technician,
- use of parts not accredited by CILAS.
- improper operating conditions: to validate your operating method, please contact your local CILAS distributor,
- errors made by the users.



WARNING:

To ensure proper measurements after installation, you must carefully read this entire manual at least once. The warranty is conditional on only the use of parts certified by CILAS for maintenance.

PART 3: DEFINITIONS



3.1- THEORY ON LASER DIFFRACTION:

The operation of your particle size analyzer is based on the principle of light diffraction. If you want to know more about this physical phenomenon, please see the appendix on your USER CD-ROM. You will find a directory called "THEORY" which contains two files in .pdf format:

- "Laser diffraction in 5 minutes",
- "Theory and applications of laser diffraction".

The first file is a short presentation of the theory and its application to measurement of particle size. It is written for people who already have a basic understanding of laser diffraction. This document will review the most important points to help you understand the basic measurement principle.

The second document is more involved. It contains theorems, hypothesis (Fraunhöfer, Mie), equations (Maxwell), etc. You will also find information on the applications of laser diffraction. The second description is a bit more complete but is more complex. This document is designed for someone who wants to know more about laser diffraction and how we can measure a particle's size using this physical phenomenon. Please refer to the document that best fits your needs.

3.2- **VOCABULARY:**

In this section, we will define different terms that are used throughout the manual. This section is very important to help the user fully understanding of measurements provided by their CILAS particle size analyzer. These definitions are used for the designation and calculation of all measurement parameters. If you do not understand any terms used by "The Particle Expert" software, in an analysis report, or in a manual, please refer to this section for a definition. The definitions are given in alphabetical order. All the equations and the entire vocabulary section refers to standard ISO 9276-1.

✓ OPTIC CONCENTRATION / OBSCURATION:

Percentage or fraction of incident light attenuated due to the diffusion and the adsorption of light by the particles. This parameter is used to adjust the amount of sample necessary for an accurate measurement.

✓ DENSITY:

The density is the density of your sample in g/cm³. You will need to enter the density in the software to calculate parameters such as specific surface. If you do not know the density of your sample, you can measure it with a special instrument.

✓ DIAMETER AT 10% and 90%:

This is the diameter value for which the cumulative result is 10% or 90%. These two values give you approximations of the smallest and the largest diameter contained in your sample.

✓ DIAMETER IN VOLUME:

The results provided by laser diffraction are in volume. This is due to Maxwell's equation. By default, "The Particle Expert" software will give you results in volume. The standard results are shown in De Brouckere mean diameter (also called D[4,3], volume or mass moment mean).

CILAS recommends using this kind of results to improve the reliability of your measurements. The mathematical formula used to calculation the diameter in volume is:

$$D_{V} = \frac{\sum n_{i} \times d_{i}^{4}}{\sum n_{i} \times d_{i}^{3}}$$

Where:

n_i: number of particles of class "i",

d_i: diameter of class "i".

✓ DIAMETER IN NUMBER:

The diameter in number is an arithmetic average of the diameters. The mathematical formula is as follows:

$$D_{N} = \frac{\sum n_{i} \times d_{i}}{\sum n_{i}}$$

Where:

n_i: number of particles of class "i",

d_i: diameter of class "i".

In "The Particle Expert" software, diameter in number is calculated from the value of diameter in volume.

✓ DIAMETER IN SURFACE:

The diameter in surface is also called D[2,1] diameter. The mathematical formula is:

$$D_{S} = \frac{\sum n_{i} \times d_{i}^{2}}{\sum n_{i} \times d_{i}}$$

Where:

n_i: number of particles of class "i",

d_i: diameter of class "i".

In "The Particle Expert" software, diameter in surface is calculated from value of diameter in volume.

✓ DIAMETER WITH LOG NORMAL:

Log-Normal law is a good model for cumulative values of samples such as ceramics. It is called L and its mathematical equation is as follows:

$$L(LD_i) = \exp\left[-\frac{LD_i}{2\sigma^2}\right]$$

Where:

C_i: cumulative value for diameter class "i",

 $LD_i = ln(D_i)$ for class "i",

 $H_i = \frac{C_i - C_{i-1}}{LD_i - LD_{i-1}}$ histogram value for class "i", σ : sigma parameter of Log-Normal law

L(LD_i) must match with Hi. The integration of L gives the cumulative values of Log-Normal law similar to C_i.

✓ DIAMETER WITH ROSIN RAMMLER:

Rosin – Rammler law is a good model for cumulative values of samples such as cement or sand. It is called R and its mathematical equation is:

$$R(D_i) = 1 - \exp\left(-\left(\frac{D_i}{A}\right)^B\right)$$

Where:

C_i: cumulative value for diameter class "i",

D_i: diameter for class "i" (in μm),

A and B: parameters found, using mean square error minimization, to match the sample.

R(D_i) must match C_i.

✓ DISPERSING AGENT:

The dispersing agent is used only in wet mode. In some cases, to get good dispersion of your sample into the solvent, you have to introduce another product in a small quantity. That other product is called the dispersing agent and necessary for an accurate measurement. For the dispersing agent, like for the solvent, you need to know its refractive index if you want to use Mie's theory.

✓ FRAUNHOFER:

The Franuhöfer's theory is the most common theory used for light diffraction. This theory can be applied for large size particles. If your particles have diameter larger than the laser wavelength (λ_1 = 635 nm, λ_2 = 830 nm), you will get accurate results using Fraunhöfer's theory. If you want more information on Fraunhöfer theory, please see the documents about laser diffraction in the appendix of your USER CD-ROM.

✓ HISTOGRAM:

The histogram gives you the size distribution for different classes of sizes. The histogram H is the derivative function of cumulative values. If you note:

C_i: cumulative value for diameter of class i,

D_i: diameter value for class i (in μm),

MD_i: mean diameter value for class i (in μ m) where MD_i = $\frac{D_i + D_{i-1}}{2}$,

 LD_i : for class i $LD_i = ln (D_i)$,

Let X_i be the ordinate on X axis and H_i the ordinate on Y axis,

We have for the ordinates of the histogram:

$$X_i = Md_i$$

$$H_{i} = \frac{C_{i} - C_{i-1}}{LD_{i} - LD_{i-1}}$$

✓ LIQUID:

The liquid is the solvent used for a measurement in wet mode. Generally the solvent used is water but it can also be an organic solvent. The liquid is the solvent which carries the sample through the particle size analyzer.

✓ MEAN DIAMETER:

Mean diameter (also called mean size) is the statistical mean value of particles' diameters contained in the sample

$$MEAN SIZE = \sum (p_i \times MD_i)$$

Where:

C_i: cumulative value for diameter of class "i",

D_i: diameter value for class "i" (in μm),

$$MD_i = \frac{(D_i + D_{i-1})}{2}$$
: mean diameter value for class "i" (in μ m),

 $p_i = C_i - C_{i-1}$: probability for MD_i

✓ MEDIAN SIZE:

Median size is the diameter value for which the cumulative results are 50%. This means that half of the particles in the sample are above the median size and half of the particles are below the median size.

✓ MIE:

The Mie's theory is the most complete theory used to measure light diffraction. This theory needs to be applied for thin particles. If your particles have a diameter smaller than the laser wavelength (λ_1 = 635 nm, λ_2 = 830 nm), you will obtain accurate results using Mie's theory. If you want more information on Mie's theory, please see the documents about laser diffraction provided in the appendix of your USER CD-ROM.

✓ MONODISPERSED SAMPLE:

A monodispersed sample is a product that has a specific distribution. You will only have one kind of population in a monodispersed sample. Particles will be similar in size or will have a size distributed compatible with Normal law. The particle's size is a well-known value plus or minus a percentage. You can summarize the particle size by the following mathematical equation:

$$X = \overline{X} \pm 2\sigma$$

Where: σ : sigma parameter.

✓ O3 (cumulative curve):

The cumulative curve Q3(x) represents the proportion of sample that has a diameter equal or less than x. It is obtained with the following equation:

$$Q3(x_i) = \sum_{i} \frac{n_i}{n_T}$$

Where: n_i : number of particles with a diameter x_i n_T : total number of particles

✓ q3 (population density):

The histogram is called q3 in the software. The histogram is obtained using the following equation:

$$q3(x_i) = \frac{(Q3(x_i) - Q3(x_{i-1}))}{\log \left[\frac{x_i}{x_{i-1}}\right]}$$

Where:

Q3(x): value of the cumulative curve,

q3(x): histogram's value, x_i : diameter of class "i",

✓ SPECIFIC SURFACE:

The specific surface is the total surface area of the particles divided by their weight. To calculate this parameter, you need to know the density of your material. In Fraunhöfer or Mie theories, particles are assumed to be spherical. When you are working with particles that are non-spherical, you need to introduce a parameter called shape factor noted S. The shape factor will give you a better approximation for non-spherical particles. The mathematical equation for specific surface is:

Specific surface =
$$\frac{S \times C}{\rho} \times \left(\sum \left(\frac{Q3(x_i) - Q3(x_{i-1})}{\frac{1}{2} \times (x_i + x_{i-1})} \right) \right)$$

Where:

C: constant, S: shape factor (= 1 for spherical particles),

 ρ : sample density (g/cm³) x_i : diameter value for class i,

 $Q3(x_i)$: cumulative value for diameter of class i.

You should now have sufficient knowledge to understand how a particle size analyzer works. Like all instruments used for control and characterization, it is very important to understand how the particle size analyzer works before making measurements. Knowing how the system operates allows you to correctly analyze the results provided by the instruments and "The Particle Expert" software.

PART 4: INSTALLATION OF THE PARTICLE SIZE ANALYSER



4.1- TRANSPORT AND STOCK:

The particle size analyzer is transported in a shipment case that is specifically designed to ensure its safety during transport. When you receive the shipping case, the first thing you need to do is to check all the items in the case.

Using the "Packing list" document included in the shipping case, carefully follow all the steps described and make sure all parts are included. When you have completed the document, please return it to CILAS.

If a problem occurs when checking the system, please note the problem and contact your local CILAS dealer or the CILAS factory.

If everything is OK, you can unpack the particle size analyzer. A technician accredited by CILAS must perform installation of the instrument. Refer to the "Installation manual" included with the instrument for more information. The Installation manual is also available on your USER CD-ROM, in .pdf format.



WARNING:

Remember to keep the shipment box in case the system needs to be moved within your facility or returned to CILAS for maintenance. Only this shipping box ensures the integrity of the particle size analyzer during shipment.

If you want to store the particle size analyzer in its shipment case, be sure to choose a safe place. The temperature and humidity need to stay within normal operating conditions (temperature below 50° C and humidity < 90%). Protect the shipment box from all environment risks (water, gas, dust, etc)

4.2- SAFETY REQUIREMENTS FOR INSTALLATION:

When installing your new CILAS particle size analyzer, please be sure to follow these basic safety rules. First, never install the instrument alone. Make sure two people are present at all times during installation. To ensure proper installation, we recommend that a CILAS agent or a technician accredited by CILAS be presence during installation. This person will be in charge of the installation. Other requirements are:

- safety rules for users who manipulate products, solvents...
- proper ventilation in the laboratory,
- good aspiration of air near the particle size analyzer by an aspirator or a laboratory hood.

If you want more details on these items, please refer to part 3.4- of the installation manual provided with your particle size analyzer.

The user is required to read the entire installation manual before beginning system installation. They also must follow at all of the safety rules specified in part 2.3- of this manual (also available in part 1.5- of the installation manual).

CILAS is not responsible for damage or accidents that occur during installation. Keep in mind that the particle size analyzer has one or two laser and has many electronic parts. Be careful with electric and laser risks and completely read all safety advice given in the manuals.

4.3- UNPACKING:

Before beginning installation, after you have checked that you have received all the parts, please carefully read the entire installation manual (part 2.1- and 2.2-). This will help you when setting up the instrument. Be sure to unpack the particle size analyzer very carefully.

All components should be stored near the particle size analyzer. The shipment case should be kept. You will use it if you ever need to move the instrument. Using the shipping case is the only way to ensure good protection when transporting the instrument.

Unpacking is the first step during installation. This step is very important, please do it carefully. If you have scrap from unpacking (plastics, etc), please follow your company's instructions regarding removal of scrap materials. For more information, please see part 2.2- in your installation manual.

4.4- GLOBAL SET - UP:

Before unpacking the particle size analyzer, prepare a clean work area. The area where you will install the instrument needs to be equipped with:

- sufficient area for the particle size analyzer,
- ventilation system or system to aspirate the effluents,
- regulated temperature ($20^{\circ}\text{C} + /-5^{\circ}\text{C}$).
- humidity rate < 90%,
- clean water supply and/or compressed air supply (depending on our model),

When the work area is ready, you can install your new CILAS particle size analyzer. After installation, you will need to connect your instrument to:

- a clean water supply (for wet models),
- a compressed air supply (for dry models),
- an electrical plug,
- A personal computer (PC).



WARNING:

The CILAS particle size analyzer is controlled by a personal computer (PC). The PC must have the proper configuration to run "The Particle Expert" software correctly. The software runs in a Windows® environment. For more information, refer to part 3.1- of your installation manual.

For more information on set up, please refer to parts 2-6 of the installation manual. The installation manual is provided in the shipping case of your CILAS particle size analyzer and on the USER CD-ROM.

4.5- STORAGE AND PROTECTION DURING A NON-USE PERIOD:

The particle size analyzer is a laboratory instrument. It is designed for measuring the size of particles contained in a powder, suspension or emulsion. If you are not going to use the system for a short or a long time, you need to do the following:

- rinse the particle size analyzer (for wet model),
- close "The Particle Expert" software,
- turn off the PC.
- turn off the particle size analyzer using the switch on position 0,
- turn off the clean water supply and/or the compressed air supply (depending of your model),
- clean the optical parts (sample cell and lens barrel),
- protect the particle size analyzer from dust using a plastic cap.

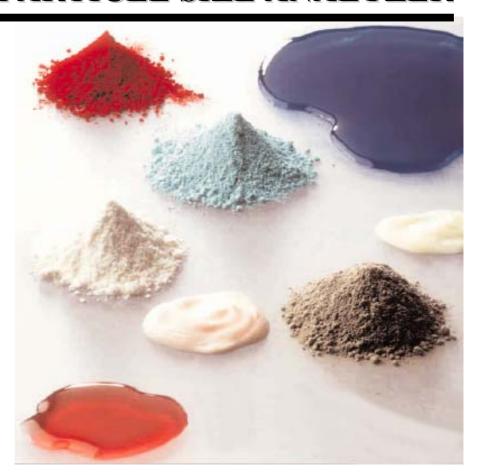
CILAS recommend you do not move the particle size analyzer during a non-use period. You can store the particle size analyzer if you are sure you have performed all the steps described above. If the non-use period is longer than 2 months, CILAS recommends that you disconnect the electrical connections, the water supply and/or the air supply.

If you want to move (to another place, plant...) the particle size analyzer, you will need to follow the procedure described in part 8 of this manual. Be sure that you have cleaned the entire device and that water is removed from the tubing system. If everything is OK, you can place the particle size analyzer in its shipment case. Be sure to also move all the documentation originally provided with the system (manuals, CD-ROM, parts, etc). CILAS recommends you move the PC with the instrument in order to save all measurements already in the database. If you are not moving the PC, you can zip the database and burn it on a CD-ROM.

4.6- ANSWERS TO FREQUENTLY ASKED INSTALLATION QUESTIONS:

If you have any question or want advice about the maintenance of the particle size analyzer, please see the file available on our website (download at www.particle-size-analyzer.com) or on the user CD-ROM. You can also contact your local CILAS dealer.

PART 5: MEASUREMENT WITH A CILAS PARTICLE SIZE ANALYZER



5.1- SAMPLE PREPARATION:

5.1.a)-Measurement errors:

The preparation of the sample is the most important step when making a measurement. As you can see on the following graph (see figure below), preparation for analysis (extraction and preparation of the sample) is the largest source of errors, much larger than errors introduced by the CILAS particle size analyzer.

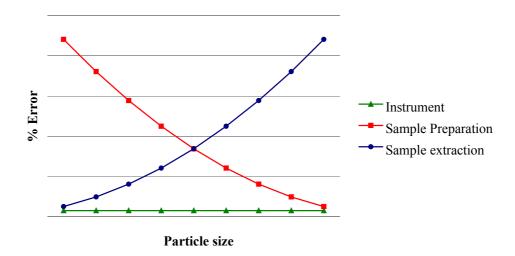


FIGURE 6: Sources of measurement errors

Measurement accuracy depends on sample preparation and extraction. You have to carefully define a method for extraction and sample preparation in order to obtain precise and reproducible results.



WARNING:

Your measurement results depend primarily on the way you have prepared and extracted your sample. Be very careful with this major step of the analysis if you want to obtain accurate and reproducible results. CILAS recommends defining a standard method of sample extraction and preparation for each specific product.

For thin size particles, the most important step is extraction. For larger size particles, sample preparation is the largest source of errors. If you have an idea of your size distribution, you can limit the sources of errors playing a major role.

The previous information shows you the user's importance to a measurement. Technicians in charge of making measurements with a CILAS particle size analyzer must be very knowledgeable. The company should educate them on the instrument. CILAS can not be responsible for measurement errors if a standard operating method is not defined and used (from preparation to the treatment of results) and if the operator is not well trained in making measurements with the particle size analyzer.

For preparing a sample, you have two different steps: extraction and preparation. Sample extraction is the first of the two steps. It consist of the extraction of an amount B from the original sample A. You want to know the size distribution of A. The extraction must be done correctly in order to have sample B identical to sample A in terms of size distribution. To do this, there are many different methods. CILAS recommends you choose one specific method for each particular product, considering the sample proprieties and the international and/or the national standards in place in your country.

Preparation consist of a division of the sample B (obtained by extraction) into a smaller amount C in order to be measured with the CILAS particle size analyzer. The amount of sample needed for a measurement in dry mode is between 1 mg and 100 mg. We have the same problem as before. The sample C must be representative of the sample B and therefore an accurate representative of sample A. If these conditions are met, then when you measure the properties of C, you measure the properties of your sample A in terms of size distribution. For sample preparation, there are many methods. CILAS recommend that you to choose one specific method for each particular product, considering the sample proprieties and the international and/or the national standards in place in your country.



WARNING:

For each specific product, you will need to define and use one procedure for sampling and extraction. CILAS can use its laboratory of applications to help you select the correct procedure.

CILAS will use its experience in particle analysis to help you in the development of your sampling method. With our laboratory of applications, we try to improve both the sampling (extraction and preparation of the sample) and the standard operating procedure (SOP) used for a measurement with "The Particle Expert" software.

Sampling is the major source of errors and so this step of the measurement must follow a defined procedure. Users should be very careful because the accuracy of the measurement will depend on the user's ability to prepare a good sample that is representative of the material's properties.

5.1.b)-Choicing the dispersing mode:

There are two ways to disperse your sample: wet or dry mode. The choice depends on the product's proprieties (fluidity, wetability, density, heterogeneity, strength, and solubility...) and on its use (the application or environment...). It is best to analyze a product in the same dispersion mode as the one used for its application.

The dispersion mode also depends on the initial state of the product. On next page, you will see how to choose the dispersion mode depending on the initial state of the sample and its properties.



WARNING:

For each specific product, you will need to define and use one method of dispersion. CILAS can use its applications laboratory to help you figure out the best dispersion mode for each product.

✓ Initial state: liquid:

Wet mode must be used if your sample is a liquid, or if you want to measure particles contained in an emulsion or in a suspension. Dry mode cannot be used with these kinds of materials.

✓ Initial state: solid:

The choice of which mode to use for a solid is more complicated than for a liquid. The choice is made after considering all the characteristics of the product and the amount of sample needed for analysis. Many parameters enters in to the final decision. To find the best dispersion mode, the best way is to try making some measurements with your particle size analyzer. CILAS can use our experience with particle analysis to help you determine which method should be used.

The procedure for choosing wet or dry mode is shown in the following table:

Sample's proprieties	Level	Dispersion's mode
1/ Quantity of sample	low	WET
necessary for one analysis	high	WET / DRY
	low	WET / DRY
2/ Heterogeneity	high	WET / DRY with
	high	sample divider
3/ Density	low	WET / DRY
3/ Delisity	high	DRY with Free Fall
	low	WET / DRY
4/ Strength	high	DRY with Free Fall
	nign	DRY with low pressure
5/ Fluidity	low	WET
3/ Pluidity	high	WET / DRY
6/ Reactivity (to organic	low	WET / DRY
and inorganic solvents)	high	DRY
7/ Taviaity	low	WET / DRY
7/ Toxicity	high	WET

FIGURE 7: Choosing the dispersion mode for a solid sample

After revieing all these parameters, you will be able to determine the dispersion mode for your solid sample. Once the dispersion mode is chosen, please use the same dispersion mode each time you make a measurement on the same type of product. Keep in mind that a dry dispersion requires a bigger amount of sample than a wet dispersion.

The two steps described in this paragraph (sampling and dispersion choices) are some of the most important steps when performing analysis with a particle size analyzer. CILAS particle size analyzers will provide you exact and reproducible results only if you are careful with these two steps.

CILAS can help you optimize your operating conditions by using our laboratory of applications. Please do not hesitate to contact us if you need help.

5.2- USER SAFETY:

Any measurement made using a CILAS particle size analyzer is subject to safety recommendations. Each operator must respect his or her own company rules. All measurements made are the responsibility of the operator and his or her company.

The particle size analyzer must be in a ventilated area with the temperature regulated to room temperature (20°C +/- 5°C). The ventilation system should limit the inhalation of effluents in wet mode. The same system must limit the inhalation of small particles in dry mode. Users must wear standard protection equipment for operators who work in a laboratory (safety goggles, smock, rubber gloves, etc).

Before making a measurement, the operators need to read the safety data sheet of the sample in order to be informed of all possible product dangers. The operator must make sure all the products he will use (sample, solvent, etc) can be used with a CILAS particle size analyzer. The operator must do this before each analysis.

Finally, after each measurement, the operator needs to clean the particle size analyzer as explained in part 6.2-. It is especially necessary if you measure a lot of different products. These products can have properties that will react with each other.



WARNING:

Anybody who will work with the CILAS particle size analyzer must respect all these safety recommendations. All users make measurements under their company's rules and responsibility.

Before starting a measurement, operators must read the entire "User Manual", particularly section 2.3- which describes the symbols and safety rules. Now, you are ready to learn how to perform a measurement with your particle size analyzer. Depending on your model, you will refer to part 5.3- and/or to part 5.4-.

5.3- MEASUREMENT IN WET MODE:

The CILAS particle size analyzer allows you to measure a sample in a wet mode. A carrier liquid called "Liquid" carries the sample. Before measuring with the particle size analyzer, you need to define a carrier liquid for your product. Knowing the sample's physico-chemical properties will help you choose a good carrier liquid and a dispersing agent. In this section, we will explain the step by step process for making a measurement in wet mode.



WARNING:

The button allows you to stop a measurement if you have a problem during the procedure. If you click on it, the measurement in progress will be delete.

5.3.a)- Preliminary tests:

This step is only applicable for new products. It describes how to define the carrier liquid and the dispersing agent that will be used.



WARNING:

If the product has already been tested, please refer to the previous results and choose the standard carrier liquid and the standard dispersing agent. Note that a dispersing agent is not always necessary. CILAS can help you to define these technical points with our applications laboratory.

After reviewing section 5.1.b-, you have chosen a wet dispersion. This way of dispersion is the most common method used these days for particle characterization. You will need to control different parameters such as the sample preparation, operating conditions, carrier liquid and dispersing agent. To see how to prepare the sample, refer to the previous paragraph. The operating conditions will be described in a following section.

✓ Carrier liquid:

The carrier liquid must have the following properties:

- non reactive with the product you are measuring,
- non absorbent at the working wavelength,
- a refractive index different than the product,
- a low viscosity (to avoid bubbles) and a low evaporation point,
- homogeneous,
- compatible with the different parts of CILAS particle size analyzer (see tables given in the appendix of your USER CD-ROM).

If the liquid does not comply with one of the above points, you can not use it as a carrier liquid for your analysis. Once you have determined the carrier liquid for a specific product, do not change it. The main carrier liquids used are water, alcohol (methanol, ethanol, isopropanol, isobutanol, benzyl alcohol...), cyclohexane, ethylene glycol, acetone, oil (palm oil, parafin oil...), hexane, ammonium citrate, an ammonia solution, glycerol, xylene, diethyl ester, toluene, benzene, etc. The most commonly used solvents are water and alcohol. For all solvents, please check their compatibility with the tubing and the joints of CILAS particle size analyzer.

✓ Dispersing agent:

The use of a dispersing agent is recommended by CILAS in order to improve your results. Like the carrier liquid, the dispersing agent must have some basic properties:

- non reactive with the product and the carrier liquid,
- non absorbent at the working wavelength,
- must improve product's wetability (see figure below),
- homogenous and stable



No dispersing agent: wrong dispersion



Dispersing agent at good ratio: good dispersion



High concentration of dispersing agent: wrong dispersion

FIGURE 8: Dispersing agent

The amount of dispersing agent introduced in the tank must be not too low and not too high in order to have good dispersion of the product into the carrier liquid.

The main dispersing agents used are HMP (HexaMetaPhosphate), Tetra Sodium pyrophosphate, Igepal, Daxad, Dispex, Tween 80, Triton. The most common are Igepal, HMP, Tetra Sodium pyrophosphate and Daxad. Please always check that the dispersing agent has all the properties listed in the previous section.

Once the operating conditions are defined, the SOP should remain the same for each similar product. Before making measurements with a new type of sample, please use a microscope or similar device to make sure that you have good dispersion. When proper dispersion is obtained, you can begin making measurements in wet mode with your CILAS particle size analyzer.

5.3.b)- Making a measurement in wet mode:

This section describes how to make a measurement in wet mode. The measurement is described in several steps. Please carefully follow the procedure below:

- 1/ Turn on the CILAS particle size analyzer, the water (or solvent) supply and all the accessories if necessary,
- 2/ Turn on the PC and launch "The Particle Expert" software,
- 3/ Enter (as shown on the figure below) your user name, your level (defined by your company as operator, analyst or controller), and your password. Click on the Validate button.



FIGURE 9: Password & Security levels



NOTE:

Please do not forget your user name, your password or your security level. CILAS recommend that all companies keep these parameters in a database. This database can help when someone has forgot his parameters.

4/ The main window of the software will appear on screen. For a measurement in wet mode, you must have the following screen:

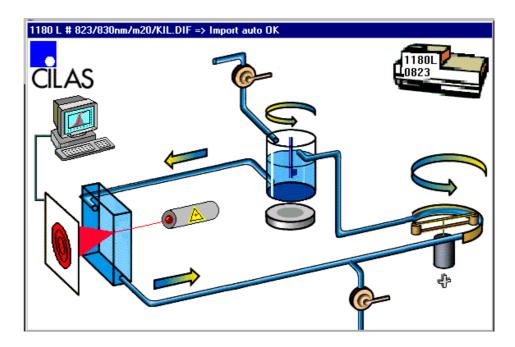


FIGURE 10: Wet mode's mimic screen

If this screen is shown, you can continue. If not, please click on the icon to change the configuration of the software.



NOTE:

If you have an instrument with only wet mode, the mimic screen will always be the correct one. If you have a "2 in 1" model, please check that the mimic screen corresponds to the measurement mode you want (dry or wet). Change it using the icon if it is not correct.

5/ On the mimic screen, check to make sure the model of CILAS particle size analyzer and its serial number are correct. They will appear in the upper right hand corner of the screen. If the numbers are correct, please continue to the next step.

If they are wrong, you will need to change them. To change them, go in the menu "Tools\New Serial Number" to change the serial number. If the model is not correct, please turn off the PC and the particle size analyzer and restart them. If the problem persists, contact your local CILAS dealer.

6/ Rinse the particle size analyzer once by clicking on the icon or by selecting from the menu "Commands\Liquid system\Rinsing".



NOTE:

All commands are available in the "Commands" menu. The most frequently used commands are available more rapidly using icons situated on the upper bar of "The Particle Expert" software.

7/ Fill the tank with the appropriated solvent (water, alcohol, etc). It must be the solvent you will use for the measurement. To fill the tank, use the valve button on the mimic screen.

8/ Click on the icon or select from the menu "Commands\Measuring". The following window will appear on screen:

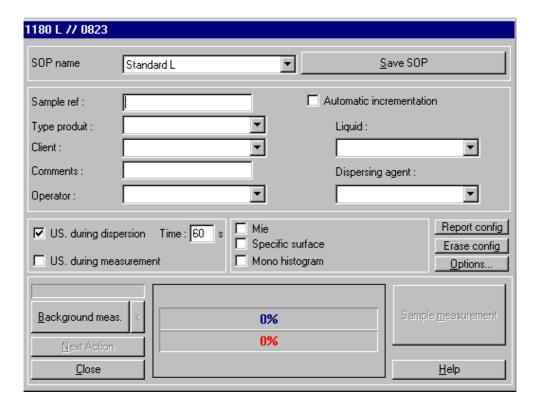


FIGURE 11: Selection of the measurement parameters in wet mode

This window allows you to define and to control the measurement. First, you have to define the parameters of the measurement.

✓ Definition of the measurement parameters:

9/ Before beginning a measurement, you will need to define some parameters in the measurement window. You will do this at the top of the window.

In the section called SOP name, select from a list of Standard Operating Parameters (SOP) already defined and saved. The list of SOP will appear if you click on the right side of the box. SOP contains parameters such as the theory used, operating conditions, etc. You can also create and save a new SOP using the button "Save SOP". The SOP is designed to provide good reproducibility in your results.



NOTE:

A SOP contains all the measurement parameters you have to set on the CILAS particle size analyzer. CILAS recommend that you define and save one SOP for each specific product. Please name the file with an explicit name containing, for example, the product's name or code.

In section 5.6-, the procedure for the creation of a SOP is explained. Please refer to that section for more information on how to create a SOP.

10/ Next, you will need to enter general information about the measurement, such as the sample's reference, the operator, the liquid and the dispersing agent used. If you select the box "Automatic incrementation", you will be able to make several measurements on the same sample. A new window will appear where you have to define the index of the first measurement, the total number of measurements, and the time between two measurements. This option is specially designed for companies which make multiple measurements using the same operating methods. This option can also be used with the "autosampler" accessory.

11/ The next screen has three little squares. In the first one (from left to right), you can define ultrasound parameters. You can choose whether or not to have:

- A dispersion using ultrasound for a defined time before the measurement,
- And/or a dispersion during the measurement.



WARNING:

Be careful using the ultrasound because it can disintegrate your particles and that would introduce an error in your results. The ultrasound is necessary to have single particles and not aggregates. You will need to define an appropriated amount of time for ultrasonic dispersion for each product.

12/ In another part, you will define the parameters used for the interpretation of results. You can select from three choices. The first selection allows you to choose Mie's theory instead of the standard Fraunhöfer theory. Mie's theory is used for thin (below 1 μ m) and/or transparent particles. Fraunhöfer is the most common used theory, corresponding to an approximation of Mie's theory. If you select Mie's theory, the following window appears:

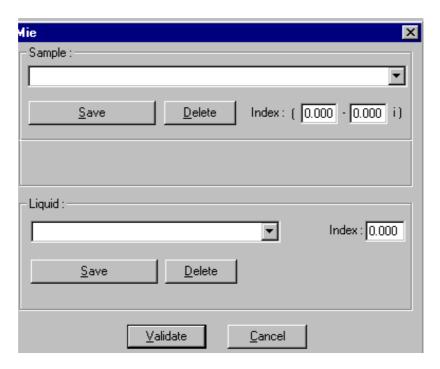


FIGURE 12: Mie's parameters

In the window, you will need to enter the refractive index of your product and of your carrier liquid. The refractive index is a complex number with a real and an imaginary part. A database is available for both if you click on the right side of the box. Its contains the most commonly used products. If you want to introduce a new value into the database, enter it in the "Index" boxes and save it using the "Save" button. Save it with an explicit name and/or comment in order to find it rapidly.

You may also want to measure the specific surface (see part 3.2-) by selecting the corresponding box. A window will appear in to which you need to enter the density of your product and its shape factor.



NOTE:

You can measure the shape factor using "ExpertShape" if you have an 1180 system or a video accessory. For density, you will need to measure it using standard methods.

You can also activate the "MonoHistogram" function, which allows you to eliminated noise from the signal and will only save one class of sizes. This option should only be used for monodispersed samples.

13/ Finally, the last square allows you to load a special report configuration using "Report config", or to erase the report configuration using the button "Erase config". The last button is named "Option". It allows you to set up all the other measurement parameters shown in the window below:

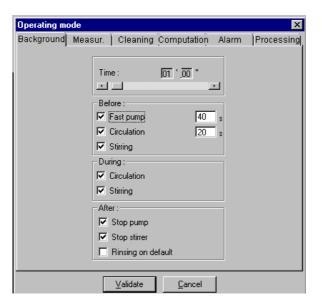


FIGURE 13: Operating method in wet mode

In the above window, you can set all the parameters from background measurement to results interpretation. Each step has its own section in the window. There are six specific parts:

- The first one is dedicated to the background measurement. You can set the amount time for the background measurement. You can also set parameters such as the speed of the pump, the use of ultrasound or the stirrer before, during and after the measurement. If you select the box "Rinsing on default", you will rinse the tank of the particle size analyzer after each measurement.
- The second part is designed for the settings during the measurement. You can set the measurement time and all the parameters before, during and after the measurement. Before the measurement, you can set the ultrasound, the pumping (fast and then normal speed) time, and the stirring. During the measurement, you can set the pump and/or the ultrasound. You can choose to dilute the sample for a defined time. Finally, after the measurement, you can set a default stop value for the pump and the stirrer.
- The "Cleaning" section is dedicated to the rinsing settings. You can choose the number of cleanings. By default, the number is 4. Next, you can choose the frequency of cleaning and the configuration after each cleaning (to stop or not stop the pump and/or the stirrer).



NOTE:

CILAS recommends cleaning the tank of the particle size analyzer after each measurement. If you do several measurements with the same sample, CILAS recommends you rinse the tank only at the end of the measurements.

- The next part is called "Computation". It is dedicated to the results of calculations done by the CILAS particle size analyzer. This window is important because the results you are shown depend on how it is set up.

You can choose the format of your results in the "Adjustments" box. You can choose between several result formats. The most commonly used is the default format called "Results in volume". For more information on these items, see section 3.2- of this manual. You can select a list of particular diameters you want to measure in the second part. If this list is already saved, you can load it by selecting it from the box. You can display the selected list of diameters by clicking on "Display". You can also create a new list using the "Create from selection" button.

In the "Sieve" part, you can compare results given by the particle size analyzer with those obtained by sizing. You will need to enter the sieve's size and the distribution of particles. This allows you to compare the two most common methods of particle characterization using "The Particle Expert".

You can set the shape parameters in the "Form" section. You will need to enter the width and the translation in the corresponding boxes. These values will be used for the calculations.

Finally, you can choose the values you want to measure for the cumulative curves. The most commonly selected values of 10%, 50% and 90% are used as default values. You can change these values or add other points on the curve.

- The "Alarm" section is dedicated to the alarm settings for the CILAS particle size analyzer. You can define the limits of concentration (recommended default values are: 0-300). You can define the number of attempts or you can select only one measurement by choosing the corresponding box. A "Quality control" option is available. This option is designed specially for a routine analysis. You can define size limits to validate or record your sample. Using the quality control option, the instrument can determine if the sample is accepted or not.
- In the last part called "Processing", you can select the kind of calibration you want from a list of suggestions. Be careful when selecting the calibration type because all of the results depend on the calibration being done correctly. CILAS recommends you use as a default the latest updated calibration.

To validate all the settings, click the "Validate" button in each specific window. If you do not validate the settings, the standard settings will be used for the measurement.

When all the settings are done, you can start the measurement using the last part of the window. The following paragraph describes how this is done.

✓ Making a measurement:

14/ Start the background measurement by clicking on the "Background meas." button. The particle size analyzer will start. In the window, you will see two gauges that show the measurement status. The blue one corresponds to the percentage of background time already passed. The red one corresponds to the percentage of the total measurement the instrument has completed. The blue gauge allows you to see the progress of the background measurement.

15/ When the background is finished, a message "Background OK» will appear in the window. "Introduce the sample" is also displayed. Please make sure that these two messages are displayed before beginning the next step. You can pass a step by clicking "Next action" and you can prevent the background measurement by clicking "Cancel background". CILAS does not recommend doing that. To ensure accurate results, you need to do a complete background measurement before each sample measurement.

16/ Introduce the sample in the ultrasound tank (see figure below). If your sample is an emulsion or a suspension, go on to the next step. If your sample is a powder, you have to also introduce the dispersing agent.



FIGURE 14: Sample introduction in wet mode



WARNING:

The amount of sample necessary to perform a measurement is about 100-500 mg. You can introduce it using laboratory tools such as a spatula. You must have a signal adsorption of about 15% between the blank measurement and the sample measurement. For the dispersing agent, you will need about 1-2 ml and you can introduce the dispersing agent using a pipette.

17/ Click on the "Sample measurement" button. The measurement starts and you can view the progress using the two indicators. During the measurement, you can view the time delay, the action in progress, the signal values, ... (see the following figure).

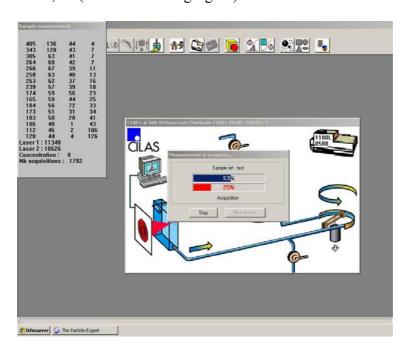


FIGURE 15: Measurement Window



NOTE:

Ø/

The button allows you to see the evolution of the histogram during the measurement. This option can help you to see the effects of parameters such as the ultrasound, etc.

18/ When the measurement is finished, the results window will appear. To see how to view and manage the results from the CILAS particle size analyzer, see section 5.5-.



NOTE:

When the measurement is finished, the results are automatically stored in the database. This lets you make several measurements before viewing the results

5.4- MEASUREMENT IN DRY MODE:

CILAS particle size analyzer allows you to measure a sample in a dry mode. The sample is carried by compressed air. Before measuring with the particle size analyzer, you need to set the pressure of the compressed air to ensure proper dispersion. The following section describes step by step how to perform a measurement in dry mode.

5.4.a)- Preliminary tests:

Before beginning measurements for each product, you will need to define the compressed air pressure needed to ensure proper dispersion. Follow the procedure below:

- 1/ Introduce the sample in to the dry feeder. The amount of sample needed is about 150 g,
- 2/ Start "The Particle Expert" software. Be sure the mimic screen for the particle size analyzer is set to dry mode,
- 3/ Begin a measurement (see the procedure in the following section),
- 4/ Check for proper dispersion by analyzing the results,
- 5/ If the dispersion is not correct, select a different air pressure value and take another measurement. If the results are good, keep this pressure as a standard one for this specific product.



NOTE:

Each product has a specific setting for the compressed air pressure. You will need to determine the right air pressure setting before making a measurement. The same air pressure must be used as part of the standard operating conditions. Good results are due to proper dispersion.

This procedure has to be performed each time you want to measure a new product. Dispersion is a very important parameter for every measurement. Be very careful with this step. Once the pressure defined, you can start measurements using the CILAS particle size analyzer.

5.4.c)- A measurement in dry mode:

This section explains how to perform a measurement in dry mode. The measurement is described in several steps. Please follow the procedure below carefully:

- 1/ Turn on the CILAS particle size analyzer, the air supply and the accessories if necessary,
- 2/ Turn on the PC and start "The Particle Expert" software,
- 3/ Enter (see figure 9) your user name and your security level (defined by your company as operator, analyst or controller),



NOTE:

Please do not forget your user name, your password and your security level. CILAS recommend that all companies store these parameters in a database. This database can help someone who has forgot his parameters.

4/ The main window of the software will appear on the screen. For a measurement in dry mode, you must have the following figure:

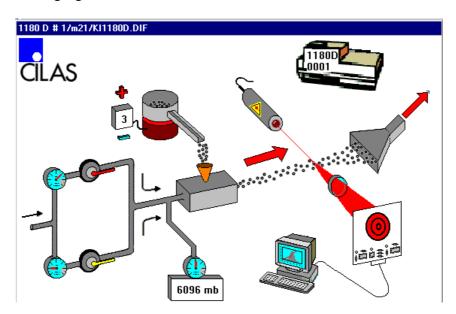


FIGURE 16: Dry mode mimic screen

If this screen comes up, you can continue. If not, please click on the configuration of the software.



NOTE:

If you have an instrument with only dry mode, the mimic screen will always be the correct one. If you have a "2 in 1" model, please check that the mimic screen corresponds to the measurement mode you want (dry or wet). Change it using the icon if it is not correct.

5/ On the mimic screen, make sure the model of CILAS particle size analyzer and its serial number are correct. They appear in the upper right-hand corner of the screen. If the numbers are correct, please continue to the next step.

If they are wrong, you will need to change them. Go to the "Tools\New Serial Number" menu to change the serial number. If the model is not correct, please turn off the PC and the particle size analyzer and restart at step 1/. If the problem persists, contact local your CILAS dealer.

6/ Clean the particle size analyzer once by clicking on the icon or by selecting "Commands\Liquid system\Rinsing" from the menu.



NOTE:

All the commands are available in the "Commands" menu. The most frequently used commands are available more rapidly using the icons situated on the upper bar of "The Particle Expert" software.

7/ Click on the icon or select the menu item "Commands\Measuring". The following window will appear on the screen:

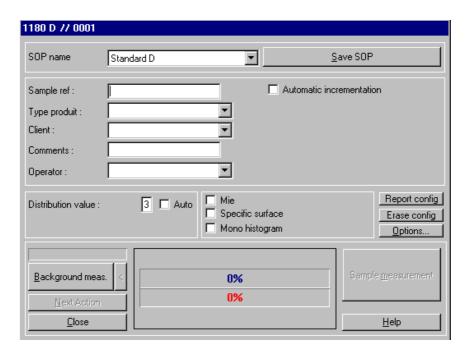


FIGURE 17: Selection of the measurement parameters in dry mode

This window allows you to define and to control the measurement. First, you have to define the parameters of the measurement.

✓ Definition of the measurement parameters:

8/ Before starting the measurement, you have to define some parameters at the top of the measurement window.

In the section called SOP name, select from Standard Operating Parameters (SOP) already defined and saved. A list of SOP appears when you click on the right side of the box. SOP contains parameters such as the theory's used, the operating conditions, etc. You can also save a new SOP using the "Save SOP" button. The SOP is designed to provide you good reproducibility of your results.



NOTE:

A SOP contains all the measurement parameters you have to set on the CILAS particle size analyzer. CILAS recommends that you define and save one SOP for each specific product. Please name the file with an explicit name containing, for example, the product's name or code. In section 5.6-, the procedure for the creation of a SOP is explained. Please refer to that section for more information on how to create a SOP.

9/ In the section below, you will need to enter general information about the measurement, such as the sample's reference, the operator... If you select the box "Automatic incrementation", you will be able to make several measurements on the same sample. A new window will appears where you have to define the number of the first measurement, the total number of measurements, and the time between two measurements. This option is specially designed for companies, which make multiple measurements using the same operating procedures. This option can also be used with the "autosampler" accessory.

10/ The next screen has three small sections. In the first section (from left to right), you can defined the distribution value. It can be set:

- automatically,
- manually by entering the number into the corresponding box.



WARNING:

The distribution value is a number between 1 and 9. It is the value of the intensity of the distributor, which is the vibrating system. This system causes the powder to fall in front of the laser beam. Like the ultrasound for the wet mode, the value has to be tested to make sure it is correct before taking a measurement.

11/ In another section, you can define the parameters used for the interpretation of results. You can select three options. The first selection lets you choose Mie's theory instead of the standard Fraunhöfer theory. Mie's theory is only for thin particles. If you select it, the window shown in figure 12 will appear. In this window, you have to enter the refractive index of your product. A database is available if you click on the right side of the box. Its contains the most commonly used products. If you want to introduce a new value into the database, enter it in the "Index" boxes and save it using the "Save" button. Save it with an explicit name and/or comment in order to find it rapidly.

You may also want to measure the specific surface (see part 3.2-) by selecting the corresponding box. A window will appear in to which you need to enter the density of your product and its shape factor.



NOTE:

You can measure the shape factor using "ExpertShape" if you have an 1180 system or a video accessory. For density, you will need to measure it using standard methods.

You can also active the "MonoHistogram" function, which allows you to eliminated noise from the signal and will only save one class of sizes. This option should only be used for monodispersed samples.

12/ Finally, the last part allows you to load a special report configuration using "Report config", or to erase the report configuration using the "Erase config" button. The last button is called "Option". It allows you to set up all the other measurement parameters in the window shown below:

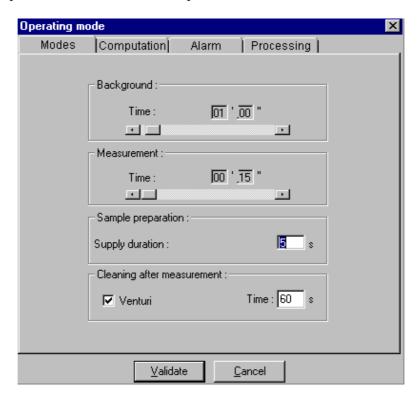


FIGURE 18: Operating method in dry mode

In the above window, you can set all the parameters from background measurement to results interpretation. Each step has its own section into the window. There are four specific parts:

- The first section is dedicated to the measurement. In the part called "Modes", you can select the measurement time for both the background and the sample. You can also enter the sample preparation time in the "Supply duration" box. You can also set the cleaning procedure. You can choose to clean the venturi system after each measurement and can set the cleaning time.



NOTE:

CILAS recommends cleaning the venturi system after each measurement. If you make several measurements on the same sample, CILAS recommends that you clean the particle size analyzer only after all the measurements.

- The next part is called "Computation". It is dedicated to the results of calculations done by the CILAS particle size analyzer. This window is important because the results you are shown depend on how it is set up.

You can choose the format of your results in the "Adjustments" box. You can choose between several result formats. The most commonly used is the default format called "Results in volume". For more information on these items, see section 3.2- of this manual. You can select a list of particular diameters you want to measure in the second part. If this list is already saved, you can load it by selecting it from in the box. You can display the selected list of diameters by clicking on "Display". You can also create a new list using the "Create from selection" button.

In the "Sieve" part you can compare results given by the particle size analyzer with those obtained by sizing. You will need to enter the sieve's size and the distribution of particles. This allows you to compare the two most common methods of particle characterization using "The Particle Expert".

You can set the shape parameters in the "Form" section. You will need to enter the width and the translation in the corresponding boxes. These values will be used for the calculations.

Finally, you can choose the values you want to measure for the cumulative curve. The most commonly selected values of 10%, 50% and 90% are used as default values. You can change these values or add other points on the curve.

- The "Alarm" section is dedicated to the alarm settings for the CILAS particle size analyzer. You can define the limits of concentration (recommended default values are 0-300). You can define the number of attempts or you can select only one measurement by choosing the corresponding box. A "Quality control" option is available. This option is designed specially for a routine analysis. You can define size limits to validate or record your sample. Using that the quality control option, the instrument can determine if the sample is accepted or not.
- In the last part called "Processing", you can select the kind of calibration you want from a list of suggestions. Be careful when selecting the calibration type because all of the results depend on the calibration being done correctly. CILAS recommends you use as a default the latest updated calibration.

To validate all the settings, click the "Validate" button in each specific window. If you do not validate the settings, the standard settings will be the one for the measurement.

When all the settings are done, you can start the measurement using the last part of the window. The following paragraph describes how this is done.

✓ Making a measurement:

14/ Start the background measurement by clicking on the "Background meas." button. The particle size analyzer will start. In the window, you will see two indicators that show the measurement status. The blue one corresponds to the percentage of background time already passed. The red one corresponds to the percentage of the total measurement the instrument has completed. The blue indicator allows you to see the progress of the background measurement.

15/ When the background is finished, a message "Background OK" will appear in the window. "Introduce the sample" is also displayed. Please make sure that these two messages are displayed before beginning the next step. You can pass a step by clicking "Next action" and you can prevent the background measurement by clicking "Cancel background". CILAS does not recommend doing that. To ensure accurate results, you need to do a complete background measurement before each sample measurement.

16/ Introduce the sample in to the tank (see figure below).



FIGURE 19: Sample introduction in dry mode



WARNING:

The amount of sample necessary to a measure is the amount necessary to fill the dry tank. It depends on product's density. You can introduce the sample using laboratory tools such as a spatula. You must have a signal adsorption of about 15% between the blank measurement and the sample measurement.

17/ Click on the button "Sample measurement". The measurement starts and you can visualize the progress with the two indicators. During the measurement, you can see the amount of time progress, the action in progress, the signal values...



NOTE:

0/

The button allows you to see at the evolution of the histogram during the measurement. This option can help you visualize the effects of parameters such as the ultrasound.

18/ When the measurement is finished, the results window appears. To see how to view and use the results given by the CILAS particle size analyzer, go on section 5.5-.



NOTE:

Once the measurement is finished, the results are automatically stored in the results database. So, you can take several measurements before viewing and analyzing the results.

Now, you have seen how to take a measurement in either dry or wet mode. The next section describes how to view and use the results. After this step, we will describe how to configure "The Particle Expert" software. If you have any problems during a measurement, please refer to section 5.6- and make sure that the software configuration is correct.

5.5- USING THE RESULTS:

The method for viewing and using the results is the same for a measurement made in wet or in dry mode. In that section, you will learn how to view and how to use the measurement files. All the measurements taken by the CILAS particle size analyzer are stored in a database. You will also learn how use the database.

5.5.a)- After a measurement:

When the measurement is finished, the results window will automatically appear on the screen. The results window for a single measurement is the following:

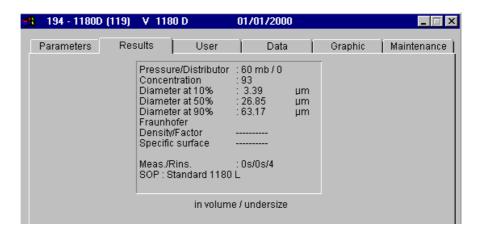


FIGURE 20: Results window

This window is composed of six different parts. The first part that appears on the screen is the part called "Results". In this part, you have a summary of the results and of the operating parameters. You can see the concentration, the results (diameters, specific surface...) and the operating parameters (theory used, model of particle size analyzer, etc). The information need for an accurate report is contained in this part.

In the "Parameters" part, you find all the information you have entered before the measurement and all the operating parameters. This part tells you the operating method used, the sample measured, etc. You can use this part to see if a comparison is possible between two measurements. For a comparison, the operating methods must be similar.

In the "User" part, you will find information about the user and in the "Data" part, you will find a table with the results. For each measured diameter x, the table will show the value of the population density q3 and of the cumulative curve Q3 (in percent). In the "Graphic" part, you can view the histogram of population density and the cumulative curve.

Finally, in the last part called "Maintenance", you will find different information reserved for the technicians in charge of the corrective maintenance for the CILAS particle size analyzers.

After a measurement, you have direct access to the results. Clicking on the button allows you to print the results. The results will be print by default in a standard report format.



NOTE:

If you want to customize your report, use the "ExpertReport" software designed by CILAS. You can design your own custom report using this software.

To launch "ExpertReport" from "The Particle Expert", click on the



icon.

5.5.b)- Loading results before the measurement:

Using the database, you can view any results that you want. This is a big advantage provided by your CILAS particle size analyzer. It allows you to use "The Particle Expert" software to compare results obtained today with those obtained one year ago.

✓ Searching for a file in the database:

To open a file already stored in the database, you need to click on the button or select the menu "File\Results". A window will appear on the screen.

Look in the box to see the files that are already loaded. If you find all the files you wanted in that list, you have already loaded the files.

If the files are not loaded, click on the "Modify selection" button. The following window will appear on the screen:

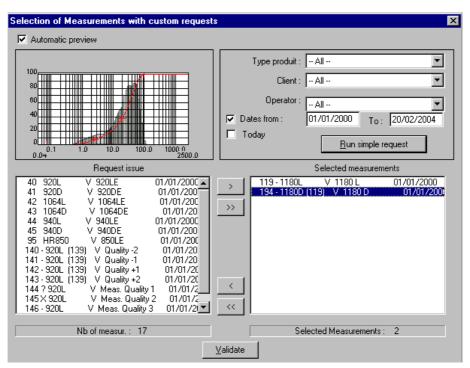


FIGURE 21: Searching the measurement files

The window contains four parts. In the upper part, you will need to enter the parameters used to search for the measurement file. You can search for files using the following parameters:

- the name of the product measured,
- the date of the measurement,
- the customer which requested the measurement,
- the operator who made the measurement.

You can also search just the most recent files by selecting the "Today" box. This allows you to search only the files with the selected parameters that were taken on the day selected.



NOTE:

The date is in your country's standard format. If you do not want to apply any criteria for your search, please select "All" in each box. Then all the files stored in the database will appear.

You can preview the histogram for the selected file in the left corner of the window. You can enable this option by selecting the "Automatic preview" box.

When you have found the file you want, select it by clicking on it. If you want to view it, you will need to move the file into the right list using the ">" button. If you want to select all the files, click on ">>". To unselect file(s), use the same procedure in the opposite direction using the "<" and "<<" buttons.

When you have chosen the file(s) you want, click on "Validate".

✓ Several measurements:

Once you have clicked on validate, the following window will appear on the screen:

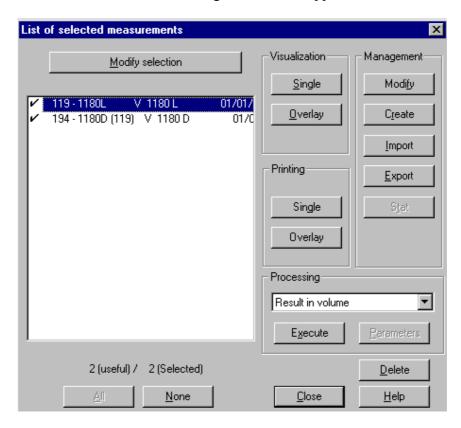


FIGURE 22: Loading a measurement file

In this window, you can view, modify, compare, and print the results obtained with your CILAS particle size analyzer.

To do something with a file, you have to select it. You can select all the files available in the list by clicking on "All". You can unselect all the files by clicking on "None". You can select the files one by one using your mouse or the space bar on your keyboard.

To view or print a single file, select a file with the mouse and click on the "Single" button in the "Visualization" or "Printing" section. If you select visualization, the results window will appear on the screen. If you select print, the report will be printed.

By default, the standard report format is printed. It is possible to change the report format. Please refer to the following section to learn more about the management of the result files.

You can also view several files at the same time. Select all the files and click on the "Overlay" button. A window appears on the screen like the figure below:

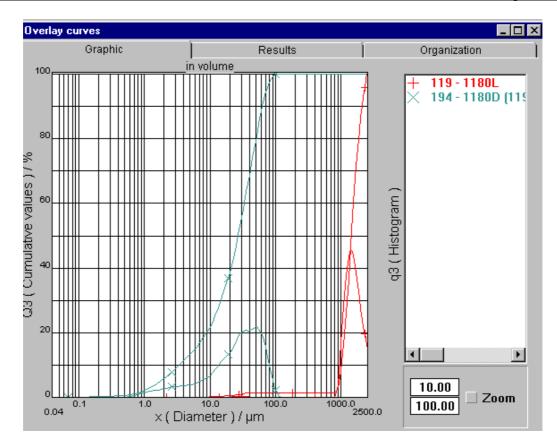


FIGURE 23: Overlay of results

In the "Graphic" part of the window, you can view the cumulative curves and the histograms. In the "Results" part, you can view a table containing the results and the differences between them.

For each result, you will see the minimal value, the maximal one and the standard deviation. It is possible to save this information as an Excel file. This option is specially designed for performing comparisons.

In the last part, called "Organization", you can choose the parameters you would like to select for comparison.

You can print an overlay report using the dedicated icon or clicking on the "Overlay" button in the "Print" section. You will have all the information shown on the screen in a standard report formal.

To customize your report, follow the procedure described above using "ExpertReport" software.

5.5.c)- Management of the results files:

In the "Management" part of the window (see figure 24), you can change several items in your results file(s). You can modify the file itself using the "Modify" button.

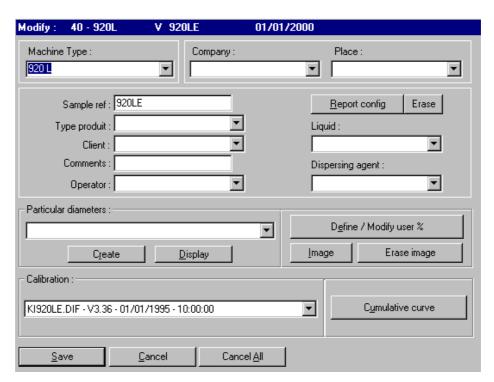


FIGURE 24: Management of result files

In this window, you can change any of the parameters you want. The modifications will be saved in the database but you will not lose the old configuration. The modifications will be stored in another file, which is a copy of the original. You can also add specific diameters into the histogram or customize the report using "Report config". You can also select a different calibration file. For the cumulative curve, you can add or delete values.

You can create a new measurement using the "Create" button. This option is described in more detail in section 5.6- of this user manual. You can also import a special type of results file that you have already saved in the database. Note that measurement files must have the extension ".mes". If it is not the case, you will not be able to import the file. You can also export the measurement files using the button "Export".

Finally, you can set up statistical testing using the "Stat" button. A new window will appear where you can choose the test you will use to compare several results. You can choose whether or not to display standard statistics (mean, min, max). You can also define your own name for each value. With the option "Mix", you can make a new measurement by mixing several results files. For example, if you have a sample containing two products, you can measure each product separately. Then you mix the results using the exact proportion of products in the sample. The software will ask for all the necessary information during the calculation.

This option is designed only for specific applications and is not selected as the default. The results file obtained using the mix is also stored in the database. Before mixing files, check to make sure that the operating method is the same. You can not mixed files with different operating methods or you will introduce errors in your results.

5.5.d)- Recalculation of the results:

It is possible to recalculate your results using a different method. The standard method gives you results in volume, which is the most commonly used method in laser diffraction. You can recalculate your results in surface, number, Log-Normal, Rossin-Ramler or for a monodispersed sample (to eliminate noise).



NOTE:

For more information about these items, please refer to section 3.2- of your user manual.

First, select the list of file(s) you want to recalculate. In the "Processing" part of the window shown in figure 22, choose the method you want to use for the calculation.

Click on "Parameters" to set parameters such as the particle shape (Width and Translation). You can choose to apply Mie's theory and to have results for the specific surface. If you activate the "MonoHistogram" option, you will use only the size that has the largest distribution. Other sizes will be removed and considered noise. Finally, you can add results that you have obtained by sieving the same sample.

Once the parameters are defined, begin the calculation by clicking on the "Execute" button. An indicator will show you the progress of the calculation. A new file will appear in the list. This new file corresponds to the results of the recalculation. This file is stored like the other files in the database and can be used like any other file.

5.6- CONFIGURATION OF THE SOFTWARE:

"The Particle Expert" is software specially designed for CILAS particle size analyzers. It has many options and is the best tool for particle characterization. This software has many functions. Those described above are the standard functions. This paragraph will explain how to set up other options in order to adapt the software to better meet your needs.



NOTE:

This paragraph is meant for technicians and/or operators who have a good working knowledge of the CILAS particle size analyzer. A person must be trained and must have a thorough understanding of the system in order to understand the different points described in this section.

The following section describes how to create a standard operating method (SOP), how to configure the software in order to control the hardware, how to use the database, how to define security levels, how to resolve problems, etc.

Be careful if you modify any of the settings. Once they are modified, some of the settings are considered standard and will be used for every measurement. If you are not sure of what you are going to do, please refer to the user manual. CILAS is not responsible for damages caused by an incorrect setting made by the user. CILAS has designed the software to avoid problems. Our default settings ensure there are no dangerous settings for the instrument or for the operator.

5.6.a)- Creating a SOP:

CILAS recommends creating a standard operating method (called SOP in "The Particle Expert" software) for each product. The SOP contains all the parameters for the measurement. When you have defined a SOP, you just have to load it before beginning the measurement. You will save time and improve your results if you use a SOP to make your measurements.



NOTE:

CILAS recommend you to name your standard operating methods (SOP) with explicit names. All new SOPs will be listed in a file that everybody can consult when they are making a measurement with the CILAS particle size analyzer.

To create a SOP, go on the "Files\Results" menu or click on the icon. A window will appear where you can click on the "Create" button in the management part. The following window is opened when you clicked on the "Create" button:

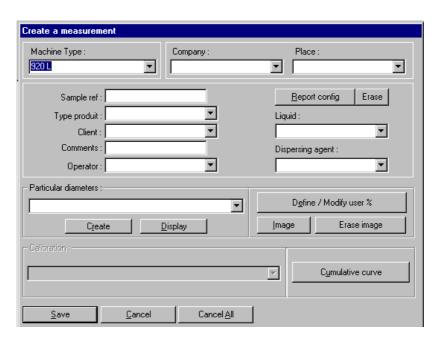


FIGURE 25: Creation of standard operating method (SOP)

To define the SOP, you will need to enter the following parameters:

- the model of particle size analyzer in the box "Machine Type",
- the company,
- the place,
- the sample reference in the box "Sample ref",
- the type of product,
- the name of the customer and the name of the operator,

- your comments,
- the carrier liquid and the dispersing agent used,

You can also:

- define the report configuration: you can load one using the "Report config"button. If you want to delete the report configuration already loaded, click on "Erase". The standard configuration will then be used for your reports,
- add measurements using the "Particular diameters" part. You can load a list of measurements already saved in the box. You can create a measurement list using the "Create" button. A specific window will be opened. Enter a name for the list of diameters and validate. Another window will appear where you can enter the diameters. When you have finished, click validate and load the list for the SOP's creation. You can view the selected list of diameters using "Display",
- define the calibration file to use for the measurement,
- add an image to the report using the "Image" button or erase the loaded image with "Erase image",
- define the diameters used for the cumulative curve with the "Cumulative curve" button. A list of diameters will appear on the screen. You can change the list in the right column and replace it with the list you want. To do that, you will need to enter the desired values and click on validate.

If you want to escape from the window without making any changes, please click on "Cancel All". If you want to erase just the last action performed, click on "Cancel".

Once you have finished, save the SOP by validating it using the "Save" button. You will have to enter a file name. You have now created a standard operating method (SOP) which you can use each time you want to make a similar measurement.



NOTE:

CILAS can help you to optimize your operating method. With our 30 years of experience, the application laboratory can help you improve your results by creating a SOP specific to your products. If you are interested, please contact us at info.particle@cilas.com or visit our website www.particle-size-analyzer.com.

5.6.b)- Configuration:

You may have to reconfigure the system (software and/or hardware) during the life of CILAS particle size analyzer. These operations are very delicate and can be the sources of errors in the instrument if not done properly. Be careful with all setting that you modify. There are some specific cases where you will have to reconfigure the system, such as when setting up a new accessory, alarms, etc.

To configure the system, go on the "Tools\Options" menu or click on the icon. A new window will appear (see figure on the next page). The window is composed of different parts. Each part is dedicated to a special configuration: interface, measurement, treatment of results, printer and hardware (alarm and configuration of the analyzer). The next section describes what you can change in each part.

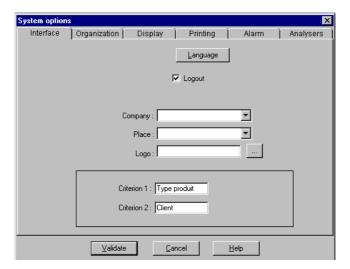


FIGURE 26: Options

✓ *Interface*:

In the interface part, you can set global parameters of "The Particle Expert" software. Using the "Language" button, you can choose the standard language of the software. If you select the "Logout" box, you will choose whether or not to active the security levels.

You can also set your company name and location, which will be set to the default. You can add your company's logo so you will see it on each report. To load the image, click on the "..." button. Then, set the two parameters "Criterion 1" and "Criterion 2". These parameters will be requested for each measurement and stored in the results file in the database. By default, the two parameters are the type of product and the customer's name.

✓ Organization:

In this section, you can set the parameters for the acquisition and display. On the left side, you have a section dedicated to acquisition.

You can activate or deactivate by selecting the box (from up to down):

- an automatic background for each measurement,
- reloading a background measurement already saved for each measurement,
- force all the measurements in the list of selected measurements in active state (you can no longer deactivate them) using the button "Forced selection",
- require criteria 1 and 2 to be filled in before executing a measurement with "Bounded Criteria",
- automatic export of the results after each measurement,
- choice of SOP between the standard SOP and a user-defined SOP,
- setting the acquisition time, if you select the box, you will have a standard time of acquisition for each measurement,
- automatic display of the results after the measurement with "Display measurement",
- automatic printing of the report after each measurement,
- automatic display of the last results with "Show last measurement",
- automatic display of the histogram and the cumulative curve for each measurement.

On the right side, you can set up the following:

- the acquisition window by choosing whether or not you want to see the value of the signal during the measurement,

- the calibration: if you want to use a calibration file from another particle size analyzer, active the "Different serial number" box,
- automatic save to the database. This option is highly recommended by CILAS.

To validate your settings, click on "Validate". You can also cancel the changes, quit the window or ask for help. Once your settings are validated, they are used by default as the standard parameters.

✓ *Display*:

In this section, you can set up all the parameters that affect how the results are displayed. To understand the definition of these parameters, please refer to section 3.2- in this manual. In the display set up window, you have the following options:

- "Display in": choose how the cumulative values & diameters are displayed in the Result window (tab Data), select either lines or columns. You can also choose between undersize or oversize. "Undersize" mode displays the amount of sample that is smaller than or equal to the related diameter. "Oversize" mode displays the amount of sample that is greater than or equal to the related diameter. Undersize or oversize mode will also apply to the cumulative curve if enabled in the display set up window.
- "Results display": the average diameter (statistical average of the results) will be displayed in the as "Mean diameter" results tab. "Hist bar" shows the histogram using bars. The line "Ref CILAS" displays additional information during selected measurements or when carrying out a query. This option is disabled by default because it slows down the loading of the measurement results. "Form parameters" allow you to show the shape parameters.
- "Particular diameters": "Q3" displays the cumulative value of a customer-defined diameter. "DQ3" displays the difference between the cumulative values of the customer-defined diameter and the previous diameter. (This allows the user to determine the percentage of a sample that is included between two specific diameters).
- "Diameters axis": you can choose to have the graph's diameter axis use either increasing values or decreasing values,
- "Diameters": you can set the diameters that appear in the histogram and the cumulative curve. In the "Diameter Nb" box, number of diameter measurements used to display the bar chart can be defined (it must be a number between 10 and 100). You can also set an equivalent distance between each diameter by selecting the "Equidistant" box.
- "Curve": you can set the parameters of the cumulative curve, such as its thickness, the number of points, the markers used, the color, etc
- "Overlay": you can set up the parameters for an overlay report in the graphic window. You can choose whether or not to view the histograms, the cumulative curves and the grid.
- "Single": you can set the parameters for a single report in the graphic window. You can choose whether or not to view the histogram, the value of parameters and the grid. Another option allows you to normalize all your histograms. If you check the "Const histo height" box, all your histograms will have use 80% of the cumulative curve as their maximum value. This display option allow the user to provide their customer with precise, standardized graphics.

To validate your settings, click on "Validate". You can also cancel any changes, quit the window, or ask the help. Once your settings have been validated, they become the default display settings.

✓ Printing:

In this section, you can set up the printing parameters. Results obtained with a CILAS particle size analyzer can be printed directly from "The Particle Expert" software. In the software, you have:

- "Print with" where you can choose to view the curves and/or the table of data
- Below "Print with", you can link an image to the report. To do that, select both the "Scale" and "Image" boxes. If you select "Scale", the image will automatically be the standard size. Using "Image", you can choose the size of your image. Enter the coefficient of size into the "*" box and the values for limits "dX" and "dY". This option is not used often because the "ExpertReport" software allows the same thing with optimized parameters.
- "Curve" allows you to define the size of the graphical part. The graphical part can be printed in a reduced format. The size of the graphic to be printed can range from 33% to 100% of the original size.
- "Print with ExpertReport" allows you to print the report using the optional "ExpertReport" software
- "Identification", if activated, will print the users name in the report. This option does not affect software compliant with CFR 21 part 11. When using 21 CFR part 11, the user name will automatically be included in each report print.

To validate your settings, click on "Validate". You can also cancel the changes, quit the window, or ask the help. Once your settings are validated, they become the default print settings.

✓ <u>Alarm:</u>

In this section, you define the alarms that warn you about a problem on CILAS particle size analyzer. By default, this part has been set to ensure proper functioning of the particle size analyzer for a majority of application. CILAS recommends modifying the settings only if it is necessary and only after carefully read all the manuals. Only technicians with a thorough knowledge of CILAS particle size analyzers should modify the alarm operations. You will be able to set:

- "Nr of std deviation for background" is where you set the deviation that is acceptable for a background measurement. By default, the value is 3. The standard deviation of the acquisitions that are made during a background measurement must be low enough to ensure a high quality measurement. This standard deviation depends mainly on the quality of the bearing liquid. A standard deviation of 0.5 would be ideal if it is very pure (free from particles and air bubbles) and a standard deviation up to 3 is acceptable in other cases.
 - If the predefined values are not suitable, click on "Another value" to specify a standard deviation between 0.1 and 9.9.
 - To obtain high quality measurement, we recommend that you do not specify a standard deviation above 5.
- "Laser value low limit": If the laser signal is too low (mainly caused by deposits in the glass cell), the background measurement will be rejected. The nominal value of the laser is 10.000. A low limit set to 7000 is normally enough to enable measurements in any conditions. It is highly recommended that you do not specify a lower value that could hide a problem of sizeable deposits and alter the measurements.
- "Signals too high": The sum of the signals on the 5 last silicon detectors is a good indicator of the cleanliness of the glass cell. If this sum is higher than a given limit, the background measurement is will be rejected. By default, this value is set at 2000.

It can be increased if very messy samples are used or lowered if the quality of the measurements requires complete cleanliness of the glass cell (we highly recommended the value remain in the range (1000-2500)).

- "Back measur OK during" is where you define the delay (in minutes) where the last background measurement is still effective. Beyond this delay, you will need to take a new background measurement.
- "Cancel background" is where you can activate an automatic reset of the software after each measurement.
- "Error message" is where you activate or deactivate a warning message from the instrument.
- "ExpertConcentration" is where you activate or deactivate the use of "ExpertConcentration" software. The software allows you to have a well-defined concentration of product for each measurement. This will provide better reproducibility for your results.

To validate your settings, click on "Validate". You can also cancel the changes, quit the window, or ask the help. Once your settings have been validated, they become the default alarm settings.

✓ Analyzers:

In the last section, you can configure the particle size analyzer and all your accessories using "The Particle Expert" software. The lower part is inactive. It is only accessible to CILAS certified technicians for corrective maintenance. Users can only modify the upper part, where you will find:

- "Options": in this part, you have to select the corresponding box for the accessory you will have installed on your CILAS particle size analyzer. For more details about accessories, please see their dedicated manuals available on your USER CD-ROM.
- "Starting": the checked box refers to the mode in which the particle size analyzer will run when the user boots up the software. "Wet method" configures the particle size analyzer for a use in wet mode. "Microcell" configures the particle size analyzer for a use in wet mode with the Microcell option actived. "Dry method" configures the particle size analyzer for a use with dry mode. "Automatic" means that the configuration will be pre-set on the Controller board. Models with wet mode only and 2 in 1 (wet and dry mode) are designed to start up using wet mode. Models with dry mode only will automatically start in dry mode.
- "System": If the "2 in 1" box is checked, the system allows the user to switch between wet mode and dry mode in software. This option is only available for wet and dry models of the 920, 1064 and 1180.



WARNING:

Switching to dry mode if the particle size analyzer is in wet mode only will cause an overflow and may damage it.

For 1180 models only, you have anther option. If the "DSP" box is checked, the particle size analyzer will carry out measurements with the DSP video camera. You must restart the software to validate the changes.



WARNING:

Do not switch to DSP mode unless your particle size analyzer is an 1180 model. If you switch to DSP mode with a different mode, you won't be able to perform any analysis

- "Remote control": there are two options in that section. If the box "Validate" is checked, the PC connected to the particle size analyzer becomes a slave PC and can be controlled by the master PC using a RS232 link. To validate the remote control module, you have to quit "The Particle Expert" and restart it. The communication rate of the master and the slave PC must be the same. The remote control serial port configuration file is located in the following folder "C:\Program files\Cilas\Gwin\exe\Tmrs232.ini". The particle size analyzer begins measurement when an order comes from either the slave or the master PC. If the "Top most" box is checked, the remote control state report window is in "Top most" mode.

To validate your settings, click on "Validate". You can also cancel your changes, quit the window or ask the help. Once your settings have been validated, they become the default analyzer settings.

5.6.c)- Database & security levels:

"The Particle Expert" is compliant with the ISO 13320 standard and with CFR 21 part 11. This is why the software has a database to store the results and security levels to identify the user. The following section describes how to manage these two options.

✓ Database:

The database of results from the CILAS particle size analyzers is available on the hard disk of your PC. The default directory for data storage is "C:\Cilas\Gwin32". The standard file extension is by default ".Db". A "PARADOX" compatible database is integrated into the software to easily sort and manage the results. The database is transparent to the user, however a number of operating settings have to be configured. Access to the tools of the database is through the "Tools" menu, sub-menu "Database" and contains the following items:

- "Secondary database management": this function manages the contents of the secondary tables, and allows the user to change, add or delete data. To delete an entry, it must not be used by any measurements. When attempting to delete data that's being used, the following error message will appear "BDE: Master has detail records: cannot delete or modify". First, delete the measurements using the affected data and then delete the data. The function "Modify" can be used even if the data is already used by some measurements. But they will all display the new text after the modification.
- "Save database": this function allows the user to save the entire database in a compressed file. Using the displayed dialog box, you need to provide a name for the backup file and specify it (For example: save the file 270498.ZIP in the directory C:\GWIN\EXE\SAVE). The default file name is C:\GWIN\EXE\SAVE. The "network" button can be used to specify a destination on your network. To save to a network, you need to have already defined a "Network drive" (contact your network administrator to set that up). Click on the "OK" button to confirm the backup or on the "Cancel" button if you do not want to go on. A dialog box will prompt you to input a comment which will be saved in the back-up file. This can be used to describe the backup. An automatic back up of the database will be carried out whenever the software is closed
- "Restore database": this function is used to recall a database previously saved using the Save database function.



NOTE:

The restore function will destroy the current database. We highly recommend that you save the current database before performing any restore.

The selection of the backup file is made through the same dialog box described in the previous paragraph. Once the file is selected, the comment that was saved during the back up will be displayed. In you are not sure you want to perform the restore function, click on the "Cancel" button.

- "Select database": it is possible to select a given database among several databases listed in the software. You may want to have several databases for distinct activities, for instance: a "laboratory" database and a "production control" database. Managing several databases is a delicate process and should be done only when necessary. In any other case, we advise that you use just the standard Database. By default, the standard database is the only database that can be selected. The default filename for this database is "GRANULOG" and it is stored in the folder C:\Cilas\Gwin32\Db". To create new databases, refer to the following item "creating the database". To select a different database, just click on the filename in the box which lists the file names and click on the "Validate" button.
- "Creating a database": First, we will explain how to create the directory and the files for the new database

The default database (filename "GRANULOG") is included in the file C:\Cilas\Gwin32\Db". To create a new database:

- 1. On the Workstation or in Windows® Explorer, open the folder in which the new directory will be created.
- 2. In the Files menu, select New, then click on File. The new file is displayed on the screen with a temporary name.
- 3. Type the desired name of the directory you are creating, then press ENTER.
- 4. Copy all of the files included in the folder C:\GWIN\DB0 into the new directory.

Be careful when creating a new database. No calibration information will be included in the new database so you should perform an import of the calibration values before the first measurement. The second item concerns the naming of the database. The database engine requires a configuration file containing the name and the place for each different database. You need to name the new database in this file. This operation can be done by running the "BDECFG" program in "The Particle Expert" group of programs.

The "The Particle Expert" group can be accessed from the "Start" menu in the sub-menu "Programs" from the task bar in Windows 95® or from the group of programs found in the office in Windows 3.11®. The program then displays a dialog box containing 6 tabs. Click on the "Aliases" tab and then on the "New Alias" button. An input box will be displayed. Input the name (alias) under which the new database will be named and click on the "O.K" button. In the topic "Aliases", click on the "Path" line and type the whole path containing the new database. For a network path, a network drive has to be defined previously (Contact to your network administrator to carry out this operation). Exit the application by clicking on "Exit" in the "File" menu. A confirmation of the changes is required to confirm the new database. To confirm the changes, click on the "Yes" button. If you are not sure you want to make the changes, click on the "No" button to cancel all changes.

The new database can be selected using the "Selection of the database" function. After the new base is selected, you have to import the calibration file for your particle size analyzer. To carry out this operation, select the database "GRANULOG", then in the "Tools" menu, submenu "Calibration", click on "export the calibration". (This operation must be repeated in wet mode and dry mode if your particle size analyzer has both wet and dry modes). Select the new database, import the calibration information included in the files KID.DIF and KIL.DIF. (Refer to the topic "importing the calibration" in the results dialog box). Enter the serial number of the current particle size analyzer in the menu "Tools"==>"Change serial number". This operation must be repeated in wet mode and dry mode if your particle size analyzer has both modes.



WARNING:

Be careful. Save your database regularly. CILAS can not held be responsible for the lost of data due to a problem with the database. Before performing any operations, save the database. CILAS recommends that you frequently burn a CD-ROM with the saved database.

You may want to move, change or zip the current database. Before zipping or moving your database, please be sure that you have saved all the results. Be careful because you can lose your data if you do not follow this advice. If you only want to change your database, go on the menu "Tools / Update database / Secondary database management". You can add a new folder, which will be your new database. If you want to zip and / or move your database, open the menu "Tools / Update database / Save database". To move your database, you should zip it. Select the directory you want and enter the name. Click on "Ok" to validate. You have zipped your database.

✓ Security levels:

The security levels are defined the first time you run "The Particle Expert" software after installation. The software will ask you if you want:

- security levels compliant to CFR 21 part 11,
- multi-level security,
- free access without security levels.

Please refer to the installation manual available on your USER CD-ROM. The security levels are very important to ensure an optimized functioning of the instrument. If you choose on of the first two security options, you will need to enter your user name and a password each time you start the software. In case of CFR 21 part 11 security levels, the user name will be saved with all the results and will appear in all the documents and reports. For each measurement, you will know who was the operator. You also have three levels of security:

- operator,
- analyst,
- controller.

You choose the level you want after considering what tasks each user will have to do on the particle size analyzer. To create an identity, please go to the menu "Tools\Identification\Create". The following window will appear:



FIGURE 27: Creation of identification

You will need to set up the user name, the level and the password. The password must have a minimum of 6 characters and a maximum of 25 characters.



WARNING:

Do not forget your user name or your password. CILAS recommends creating a table with all the usernames and all the passwords.

Into the menu "Tools\Identification", you can manage these options using the "Delete" button. It can be used to delete a user name. Be careful when removing users. Once the username has been deleted, you can not restore it. You can also change of username using the "Select" button.

We have described all the items needed to configuration "The Particle Expert" software. Please configure the software carefully. Problems can occur if the user makes bad choices for the settings described in this section.

5.7- OPTIONAL ACCESSORIES:

There are a number of accessories available for your CILAS particle size analyzer. These options are designed to better meet all your measurement needs. If you have a special need, our accessories can solve your problem and improve your measurements. The accessories are:

- autosampler: improves your productivity when performing routine analysis (controls, production, etc),
- small volume unit: a device for reducing the amount of sample and solvent necessary to perform a measurement,
- video camera: allows the user to see the shape and the sample dispersion,
- alcohol regenerator: reduces the amount of solvent necessary for analysis, which results in less waste removal.

"The Particle Expert" software controls all of the CILAS accessories. If you want to know more about these different devices, please refer to the specific "User Manual" available on your USER CD-ROM.

Some optional software packages are provided with the CILAS particle size analyzer and its main software "The Particle Expert". They are designed to complete the information obtained from the particle size analyzer:

- "ExpertShape" software allows you to analyze the size and the shape of your samples. To use this software, you must have a video camera. The video camera comes standard on the 1180 and is an optional accessory on all other models,
- "ExpertGrab" software takes images of your sample if you have a video camera installed on your particle size analyzer. A video camera comes is standard on the 1180 and an optional accessory on all other models,
- "ExpertReport" software allows the user to customize reports from the CILAS particle size analyzer,
- "ExpertConcentration" software allows you to have better reproducibility of the sample concentration for measurements in wet mode on CILAS particle size analyzers.

All of these software packages can be launched from "The Particle Expert" software. If you want more information about how to use these different software packages, please refer to their specific "User Manual" and/or the "Tutorial" available on your USER CD-ROM in the manuals section.

5.8- WASTE FROM A MEASUREMENT:

When dealing with waste from a measurement, the user needs to throw the waste away while following the rules of their company and the environmental laws of their country. CILAS particle size analyzers do not themselves produce any waste. If you have to discard the entire instrument, do it in a special location that is dedicated to electronic waste (PCs, etc).



WARNING:

Samples and solvents used in the particle size analyzer can be dangerous chemical products. The user must take the necessary precautions after reading the safety data sheet of the products they manipulate.

✓ In wet mode:

Waste can come from the sample, the dispersing agent and the solvent. You will need to connect the outlet of the particle size analyzer to a path where the solvent will be treated in order to eliminate all the pollutants contained in the carrier liquid. If you are using organic solvents, there are specific devices available to reduce the pollution and its cost. For example, if you use alcohol, CILAS has designed an alcohol regenerator which limits the amount of solvent used.

For all the instruments used to prepare the sample (dish, etc), please refer to your company rules and the laws of your country. The operator must be knowledgeable about these issues before making a measurement.

✓ In dry mode:

For particle size analyzer in dry mode, waste comes only from the sample. No solvent or dispersing agent is used. The dry mode limits the emission of waste. In order to eliminate the emission of waste, you will need it to go in to the aspirator connected to the particle size analyzer. Refer to the user manual of the aspirator to find out how to remove the waste from the aspirator. When throwing the waste away, be sure to respect your company's environmental policy and your country's environmental laws.



WARNING:

CILAS recommends that all the user of its particle size analyzers set up a circuit compliant with the standard ISO 14000 (or similar) in order to treat all the waste from measurements. Users have to follow their company's policy and their country's laws for handling environmental waste. CILAS can not be held responsible for damages caused by waste from particle analysis.

CILAS particle size analyzers are designed to limit the waste created when making a measurement. Special accessories are also available as options (Small Volume Unit, Alcohol regenerator, etc) in order to limit the waste and improve productivity. If you want more information about this optional equipment, please refer to section 1.3- in this manual.

5.9- ANSWERS TO FREQUENTLY ASKED MEAUREMENT QUESTIONS:

If you have additional question or would like advice about measurements made with your particle size analyzer, please see the file available on our website (download at www.particle-size-analyzer.com) or on the user CD-ROM (USERCDROM \ Answers to frequently ask questions \ Frequently ask questions.pdf). You can also contact your local CILAS dealer.

PART 6: MAINTENANCE



Maintenance is an important part of taking measurements with any instrument. CILAS particle size analyzers do not require much maintenance. The short bench concept means your CILAS particle size analyzers is very reliable. There is no need for optical alignment if the instrument if it is used in normal operating conditions. In this section, we will describe the maintenance operations. If you have any problems with your particle size analyzer, please carefully read this section.

6.1- SAFETY REQUIREMENTS:

Before performing any maintenance operations, please carefully read the safety rules described in section 2.3- of this manual. The person in charge of the maintenance must be trained during installation by a CILAS certified technician. Other people are not authorized to perform maintenance operations. CILAS is not responsible for an accident which happen during maintenance performed by unauthorized persons.

Authorized users must be aware of laser and electrical risks involved with a maintenance procedure. To perform maintenance, users have to open the protective cover of the particle size analyzer. The laser is not activated when the cover is open because of the safety interlock.



WARNING:

Anybody who will work with or on the CILAS particle size analyzer must respect all this safety recommendations. The operator in charge of maintenance must have the necessary skills and information to understand the operation of an electromechanical device. All users must follow the safety rules and policies of their company.

Keep in mind that laser radiation is very dangerous even if all the safety tools on the CILAS particle size analyzer are working properly to ensure the operator safety.

6.2- PREVENTIVE MAINTENANCE FOR USERS:

6.2.a)- Introduction:

CILAS particle size analyzer maintenance operations cover regular servicing and cleaning of the optical parts. Users and technicians can perform both of these operations. Users should only perform preventive maintenance and should never perform any other maintenance operations. Servicing needs to be done regularly. Cleaning the optical parts needs to be done frequently in order to ensure good reliability of your results.

6.2.b)- Preventive maintenance items:

Before operating the instrument, the user should have the following items:

- 1 set of hexagonal screwdrivers,
- 1 set of flat wrenches,
- 1 flat screwdriver,
- a clean work area,
- 1 cleaning kit (wipes, absolute ethylic alcohol, lint-free cloth).

Once the user has gathered these items, preventive maintenance can begin.

6.2.c)- Preventitive maintenance:

These operations need to be carried out before each use of the particle size analyzer. If the particle size analyzer is used frequently, se preventitive maintenance should be done once a day, in the morning before the first measurement is taken.

- Make sure the particle size analyzer is disconnected from the power line,
- Fluid connector module: check the condition of hoses and replace any punctured hoses,
- Video and data input connections: check the condition of the connections,
- Remove the particle size analyzer cover,
- Check operation of the cover safety with a multimeter,
- Check pump module, hose condition; replace any punctured, polluted or worn hoses,
- Check liquid inlet electrovalve hosing and piping,
- Unclip stirrer from the top of the ultrasound tank,
- Inspect sealing on the level sensors and union flanges,
- Clean level sensors with cotton tip dipped in alcohol,
- Clean ultrasound tank with lint-free cloth dipped in alcohol,
- Clip stirrer back onto the top of the ultrasound tank,
- Check (without removing) that the optical fibers are properly connected to their respective connectors,
- Check sample cell inlet (remove accumulated matter),
- Make sure sample cell fluid connections are properly connected,
- Make sure bellows are properly positioned,
- Close particle size analyzer cover.

6.2.d)- Cleaning the Optical Parts:

The optical parts need to be cleaned whenever there is an increase in the blank signal (this can be seen using the "real time signals" button) when compared to the previous measurements. If you use a product that you know will make the sample cell dirty, please clean the sample cell regularly.

A gradual increase in the blank signal over time (up to 20% of their original value) is considered normal. Before cleaning the optical parts, rinse the particle size analyzer with detergent and recheck the value of the blank signal.

If an increase in blank signal persists:

- clean the sample cell holder,
- rinse with detergent,
- recheck blank signal value (using the "real time signals" button),
- clean the optical parts if the increase in blank value persist.

Next, we will explain how to clean the sample cell and the optical parts. Be careful when cleaning, the sample cell and optical parts are very fragile. Please clean the parts exactly as described in this section. If you do not follow this advice, you can deteriorate the parts. CILAS is not responsible for problems resulting from errors made by a user cleaning the system. The warranty does not apply to parts improperly cleaned by the user. The warranty is only valid for standard use in normal conditions.

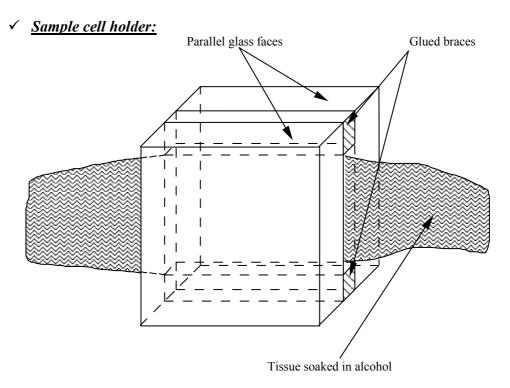


FIGURE 28: Cleaning the sample cell

Clean tank as follows:

- Remove the sample cell holder: see removal/replacement information in section 6.2.e),
- Wet a tissue with absolute ethyl alcohol,
- Wipe the tissue back and forth over the plate inner surfaces,
- Repeat several times if necessary, changing the tissue each time,
- Rinse assembly with absolute ethyl alcohol,
- Clean outer surfaces of the sample cell holder with a tissue soaked in alcohol,
- Install sample cell holder and sample cell: see removal/replacement in section 6.2.e).

✓ Lens:

Cleaning the lens is a delicate operation that must be performed only if cleaning the sample cell did not change the high level of the blank signal.

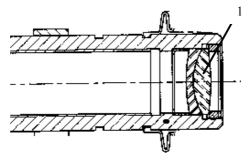


FIGURE 29: Cleaning the lens



NOTE:

Do not unscrew the permanent screws on the barrel or you will lose the optical alignment of the system.

Clean the lens barrel as follows:

- Make sure particle size analyzer is disconnected from the power line,
- Open the particle size analyzer cover,
- Clean the outer surface of the concentrator lens (item 1, figure 7) by wiping it once with tissue soaked in alcohol. Be careful not to scratch the anti-reflection coating,
- Close the particle size analyzer cover.

The above section describes all the maintenance operations authorized for users. The next section will describe what to do if you have a more difficult problem with your instrument. Keep in mind that CILAS particle size analyzers are reliable if used in standard operating conditions and if you remember to perform preventive maintenance.

<u>6.2.e)- Removal – Replacement of the sample cell:</u>

This section describes how to remove or replace the sample cell of your CILAS particle size analyzer.



WARNING:

Drain the particle size analyzer and disconnect it from the power line before proceeding with removal of any parts.



NOTE:

O-rings (item 5, 8, 10) are visible only after removing diffuser (item 3), flange (item 6), and sample cell holder (item 9).

✓ Removal:

- 1- Open the particle size analyzer cover,
- 2- Remove fluid lines (items 1 and 2),
- 3- Remove the sample cell holder by pulling it up,
- 4- Unscrew the 4 screws (item 4) securing the diffuser (item 3),
- 5- Remove the diffuser (item 3),
- 6- Remove the O-ring (item 5) from the diffuser (item 3),
- 7- Unscrew the 4 screws (item 7) securing the flange (item 6),
- 8- Remove the flange (item 6),
- 9- Remove the O-ring (item 8) from the flange (item 6),
- 10-Slide out and remove the sample cell holder (item 9),
- 11- Remove the O-ring (item 10).

✓ Replacement:

- 1- Clean the sample cell holder (item 9),
- 2- Clean seals and mechanical parts, rinse and dry,
- 3- Clean the inside of the diffuser without opening it,
- 4- Insert the O-ring (item 10) in the bottom of the base,
- 5- Insert the sample cell holder (item 9) in its support,
- 6- Insert the O-ring (item 5) in the diffuser (item 3),
- 7- Insert the O-ring (item 8) in the flange (item 6),
- 8- Fit the flange (item 6),
- 9- Fit the diffuser (item 3),
- 10-Tighten the 4 screws (items 4) of the diffuser,
- 11-Place the sample cell in the particle size analyzer,
- 12-Tighten the 4 screws (items 7) of the flange
- 13-Connect the fluid lines (items 1 and 2) to the sample cell,
- 14- Close the particle size analyzer cover,
- 15-Fill with water and check to be sure that it does not have a leak.

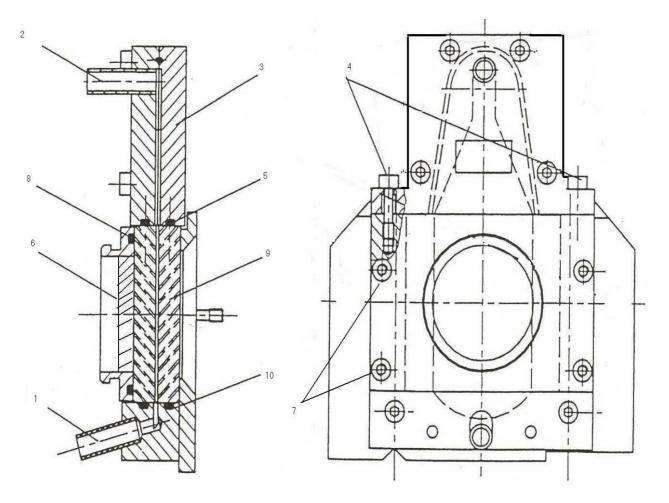


FIGURE 30: Sample cell holder

6.3- MAINTENANCE FOR DEALERS AND AUTHORISED PERSONS:

This chapter covers corrective maintenance operations. Only technicians accredited by your CILAS dealer can perform these actions. Corrective maintenance operations need to be made if a problem occurs with the hardware or the software.

CILAS particle size analyzers will identify a problem when one occurs. The system will inform you of the problem via "The Particle Expert" software. During a measurement, an error message would appear on the monitor screen. If an error message appears, there is a problem and the error message is an alarm. When an error message occurs, the user needs to do the following:

- note the error message provided by the software,
- note the model and the serial number of your particle size analyzer,
- note the version of "The Particle Expert" software you are using,
- fill in the "Bugreport.doc" document (available in the appendix of your USER CD-ROM),
- contact your local CILAS dealer. They will need all the above information in order to diagnose the problem. A technician will proceed with maintenance operations.

Users are not authorized to perform corrective maintenance. If they do, it is under their own responsibility and the warranty will become invalid. To reduce the time it takes for maintenance, communicate to your dealer as soon as the file "Bug report.doc" is completed. The bug report will help him to quickly locate and solve your problem. This document is available in your USER CD-ROM in the "software" section of the appendix.

6.4- ANSWERS TO FREQUENTLY ASKED MAINTENANCE QUESTIONS:

If you have additional question or would like advice about the maintenance of your particle size analyzer, please see the file available on our website (download at www.particle-size-analyzer.com) or in the USER CD-ROM ("Frequently ask questions.pdf"). You can also contact your local CILAS dealer.

PART 7: SPARE PARTS



User replaceable items for your CILAS particle size analyzer can be ordered from CILAS. A list of these items and their part numbers are provided in the appendix of your USER CD-ROM. Original spare parts provided by CILAS are manufactured following our specifications and are checked before shipment to prevent any damage to the instrument and to guarantee the high quality measurement results. These items must be used for maintenance if you want to maintain your warranty.



WARNING:

To ensure accurate measurements after a maintenance operation, you must carefully read this entire manual at least once. The warranty is conditional on the use of only parts certified by CILAS for maintenance.

7.1- LIST OF SPARE PART:

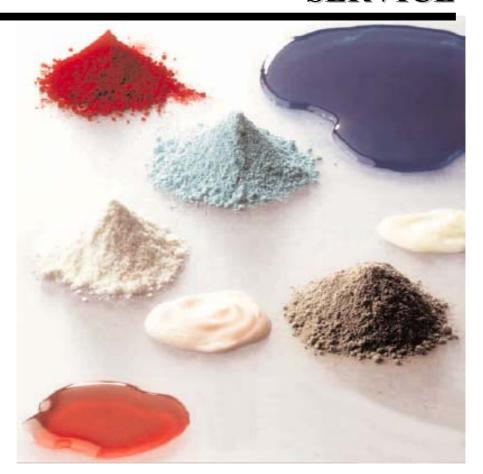
The list of all the spare parts for your CILAS particle size analyzer is given in the appendix on your USER CD-ROM. You will find a list of all the spare parts along with their code, which is needed for the spare part order form. Please make sure that the code corresponds to the spare part you want to order.

7.2- ORDER FORM:

Use the order form available in the appendix of your USER CD-ROM for all orders. This order form must be returned to your local CILAS dealer. If you need to see a list of CILAS dealers, please go to our web site (www.particle-size-analyzer.com). Please indicate on your order which model of your particle size analyzer you have, its serial number and the version of "The Particle Expert" software you are using.

You have additional question or would like advice about a command or an order form, please see the file available on our website (download at www.particle-size-analyzer.com) or in the user CD-ROM ("USERCDROM \ Answers to frequently ask questions \ Frequently ask questions.pdf"). You can also contact your local CILAS dealer.

PART 8:WITHDRAWING YOUR PARTICLE SIZE ANALYSER FROM SERVICE



Your CILAS particle size analyzer can be withdrawn from service. This would be done if you want to move the system to a new laboratory or move the system to a new building. This can happen due to the long lifetime of the CILAS particle size analyzer.

Technicians who know how to manipulate electromechanical devices must perform the withdrawal. If a skilled technician is not available, please contact your local CILAS dealer. CILAS is not responsible for damage or accidents affecting the users or the particle size analyzer.

To withdraw the particle size analyzer from service, please follow the procedure below:

- 1- Drain the particle size analyzer,
- 2- Turn off and disconnect the particle size analyzer,
- 3- Close "The Particle Expert" software and turn off the PC,
- 4- Disconnect the RS232 link between the particle size analyzer and the PC,
- 5- Turn off the water supply and/or the compressed air supply (depending on the model of particle size analyzer),
- 6- Disconnect the links between the particle size analyzer and the two supplies (air and/or water),
- 7- Remember to keep all the accessories with the instrument, including all the tubing system and other items originally provided with the particle size analyzer,
- 8- Clean the sample cell and the optical parts of the particle size analyzer,
- 9- Retrieve the shipping case and place it near the particle size analyzer,
- 10- Set the particle size analyzer in to the shipment case,
- 11-Place all the accessories and devices originally provided with the instrument in the shipping case,
- 12-Put the PC or the database (already zipped and burned on a CD-ROM) with the particle size analyzer if you want to save the results of previous measurements.

The procedure above is also applicable if you have to ship the particle size analyzer to your dealer or to CILAS headquarters in France for maintenance. If you are sending your system for maintenance, you will need to determine which parts of the system you need to send. In order to do that, you have to provide a good description of your problem (using the "Bug report" document). The technician will tell you which parts of the system you will need to ship.

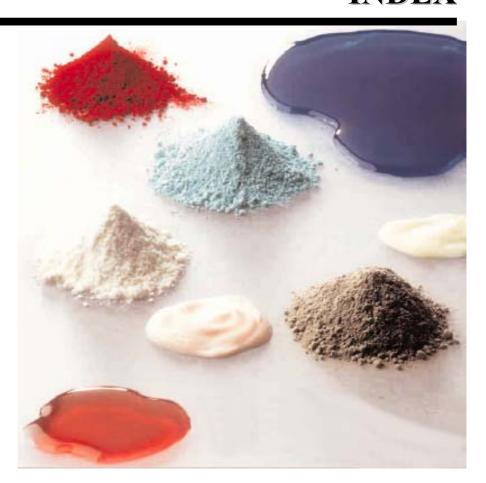


WARNING:

If you ship the particle size analyzer to the CILAS plant for the maintenance, please remember to send with the instrument the safety data sheets of all the products (samples) measured using CILAS particle size analyzer.

The withdrawal of an instrument is a very complex and difficult thing. There can be problems with your particle size analyzer if a highly skilled technician does not perform the withdrawal.

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