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VALIDATIONS OF PARTICLE SIZES ANALYSIS

BY MEANS OF

S I E V I N G S

The knowledge of the particle size distribution of powders is in many cases a very important question for the characterization of the manufactured or used product, especially for quality and production control.

In the case of quality control as to ISO 9000 and also especially under the aspect of the manufacturing of high quality and competitive products, the particle size analysis of solids in dry state or suspensions/emulsions becomes a control element more and more important from day to day.

Today, as in the past and for a further long time ahead, for most of the particle sizing applications the classical and proven system of the dry sieving for relatively large size powders and the wet sieving for the fine size particles has been and will remain the most used system for particle size analysis. There are of course more elegant and in some cases more accurate/exact and fast systems than the sieving, but in most cases they have the disadvantage of needing very small quantities of samples and in such cases the taking of a representative small sample becomes the major and most critical part of the complete analysis. A further important aspect when looking at these systems is that a rather important financial investment must be made when purchasing such instruments.

FRITSCH Company based in Idar-Oberstein, Germany, a small city in the South West of the

country about 120 km away from Frankfurt/Main, has specialized since more than 75 years on the production of laboratory instruments for

sample preparation (crushing/micro-milling)

particle sizing

sample dividing and ultrasonic cleaning.

The company has a very comprehensive line of crushing/milling instruments for the sample preparation of either very hard, brittle, soft, elastic or fibrous materials.

Our line of instruments for the particle size analysis consists of sieve shakers, a scanning photo-sedimentograph and laser-diffraction particle sizers.

Finally a serie of sample dividers and ultrasonic cleaners (especially for the cleaning of fine mesh sizes of analytical sieves) are rounding up our programme.

Our instruments are well known all over the world under the trade names

pulverisette

analysette

laborette.

Sample taking and sample dividing.

This is the first and most important step for the particle size analysis. If the sample is not truly representastive for the total quality to be controled, all following steps will be subject to important errors. In most cases, national, international or internal standards or rules prescribe how to do the sample taking.

The next step, which means the sample dividing to obtain a small representative quantity for the analysis, is either done by manual quartering which has been the traditional method for many years or by the use of automatic sample dividers which guarantee a more exact dividing compared with the manual method.

We have developed a modular and very flexible rotary sample divider which allows to divide the samples either in 8, 10 or 30 individual fractions beeing highly representative for the total introduced quantity. The combination of the dividing by gravity and centrifugal force results in obtaining an optimal flow behaviour especially also for fine powders or samples which have a tendancy to agglomerate and which are difficult to divide accurately. This guarantees a very high accuracy and representativity of the small individual divided samples.

This sample divider is also perfect for the dividing of suspensions.

The instrument is fully appropriated for the dividing of food, chemical and pharmaceutical samples without introducing any undesired contamination to the samples which could result from an eventual abrasion of the parts of the sample divider beeing in contact with the samples.

The test sieves.

Test sieves which are used for production control according to ISO 9000 are **official measuring//control tools.** Therefore they are subject to very high production standards and must absolutely correspond to the given standards.

The difference must be made between:

a) test sieves with a W**orks Certificate (2.1, EN 10204),**
without indications of the results of the control,

b) test sieves with a **Certified Works Certificate (3.1B, EN 10204),**
with the results of the control as sper ISO 3310,

c) test sieves beeing **Calibrated with Works Certificate (3.1B, EN 10204),**
of which 20 times more meshes have been controled compared with b).

For the validation of particle sizes analysis it is essential that either test sieves b) = **Certified with Works Certificate** or test sieves c) = **Calibrated with Works Certificate** are being used.

Certified test sieves with Works Certificate and Calibrated test sieves with Works Certificate can be re-certified in our works.

A further obligatory point is that the sieving process must be absolutely reproducible. Therefore this excludes the manual sieving.

Choice of the sieve shaker.

In the past, the sieve shakers which were available on the international markets did not work under reproducible conditions. Depending especially upon the weight placed on the instruments (weight of the sieves and the sample) and eventual electrical supply fluctuations, the results for an identical sieving time were not the same.

Since a few years leading manufacturers of sieving machines have developed electromagnetic principle working sieve shakers which incorporate an **automatic reading and regulation** of the selected working amplitude. Whenever the incorporated amplitude reading device registers a deviation of the selected amplitude, the electronic system of the instrument regulates and resets the amplitude to the selected one. These sieve shakers work under absolutely reproducible conditions and are therefore recognized for the validation of particle size analysis according the Quality Control ISO 9000.

The new generation of the Fritsch „analysette 3 PRO“ sieve shaker guarantees an absolute reproducibility of the working conditions of the instrument (DIN 66165).

This means that independently of the weight placed on the instrument (number of sieves and sample), the selected amplitude is automatically permanently controled and in the case of a differencee between the selected and measured amplitude (variance comparison) the amplitude is automatically re-set on the selected value.

Of course it goes without saying that such a modern and high tech instrument is fitted with a precise digital timer, with soft touch and ergonomic control panel and that the indications of the amplitude, frequency and other additional fonctions are digital.

The „analysette 3 PRO“ is therefore itself a measurement/control tool and corresponds to the requirements of the standard ISO 9000. Prior to the issuance of a Works Certificate, the instrument is thoroughly controled by a particle size analysis using a calibrated sieve set.

The analysed sample (**Standard**) is a particle size control sample provided by the European Community Bureau of Reference under the title BCR 68. Its particle size distribution and the tolerated deviations are determined and published in the information BCR 6825 dtd. 1980. The basis is the **Certification Report on Reference Materials of Defined Size, BCR 66, 67, 68, 69 and 70.**

The comparison and conformity with the tolerated limits of the BCR standard leads to the validation of the sieve shaker.

The sieve shaker „analysette 3 PRO“ can be re-validated by our authorized technical service staff with a Works Certificate 3.1B EN 10204.

„autosieve“ programme for auto-validation of the sieving processs by means of the RS 232 interface.

This programm allows the automatic evaluation of the results of the sieving and the storage of the results according to ISO 9000. The programm works with a database for management of sieve sets with up to 30 individual sets and provides the results presentation either in tabular date, frequency or cumulative distribution graphs. Up to 100 comparison curves can be displayed simultaneously.
This programme is the basis for an auto-validation „on line“ for the working parameters of the sieve shaker and the results of the particle size analysis and therefore conforms to the highest requirements of quality control.

The use of the „autosieve“ programme requires a balance with a load of about 4 kg and a lecture of 0,01 g as well as a customary standard WINDOWS PC with a minimum of 8 Mbyte RAM.

Finally a few words as to the handling of the test sieves.

Test sieves and especially the ones smaller than about 100 micron are relatively sensitive measuring

tools. They must be treated with utmost care. By no way they should be strongly brushed to clean them as the woven mesh gauze would be damaged and the particle sizing results would be wrong.

For the gentle, efficient and fast cleaning of the test sieves we strongly recommend the ultrasonic cleaner „laborette 17“.