DOWNLOAD PDF MODERN METHODS OF PARTICLE SIZE ANALYSIS

Chapter 1 : How To Select A Particle Size Analyzer - HORIBA

User Review - Flag as inappropriate The book "Modern Methods of Particle Size Analysisâ€Ž" by Howard G. Barth -and especially Chapter 7 â€žField-Flow Fractionation of Particles" written by Karen Caldwell, has been an extremely helpful reference for us.

Volume 7 In Part 1 of this guide Technical Brief Volume 6 we stated that the aim is to provide a pathway through the decision-making process of choosing a particle sizing analyzer by means of asking and answering three general questions: How do I classify the various techniques? How do I set specifications quantitative or qualitative? Which technique s have the best chance of solving my problems? We started by classifying the different particle sizing techniques in four ways: Information Content A fifth way to classify a particle sizer is by information content. This final major classification revolves around the amount of information required to solve a particular problem. There are two key questions to ask that determine which techniques are useful. What do you want? How will you use it? For average and width, an ensemble averaging instrument is sufficient. However, the more information needed, the more resolution is required. But regarding the "zero-to-infinity" trap set by over-hyped marketing claims made for many instruments. Answering the first question -What do you want? For example, in most process control environments, varying a single parameter is reasonable; varying multiple parameters is difficult. Additional size distribution information, often hard to come by reliably, might be the skewness of a single, broad distribution, or the size and relative amounts of several peaks in a multi-modal distribution, or the existence of a few particles at one extreme of a distribution. Where the distribution has several, closely-spaced, features a true high resolution technique is an imperative Specifications: Specifications are of two types: Quantitative Specifications of this type comprise size range, throughput and definitions: Throughput The novice often mistakenly assumes that the measurement duration is sufficient to characterize the typical time per sample. Sometimes the measurement duration is only a fraction of the actual time per cycle. Throughput is the total sum over all of the following: Throughput is probably most important to a QC laboratory where, often, large numbers of samples must be run in one day. Speed of analysis is sometimes a major consideration even for one measurement in process control applications. Sample preparation may be as short as a few minutes or require overnight. Warm-up, calibration or instrument adjustment all add to the overall time. Generally, with most modern instruments the actual measurement or analysis time can be short. Yet, for broad distributions, sieving and sedimentation techniques including field-flow fractionation are relative slow compared to most forms of light scattering. Single particle counting SPC is fast for narrow distributions but can be slow for broad distributions. Data reduction and printout are fast given modern computers. The time to interpret the data depends on the analyst and what criteria have been set. Cleanup time is often seriously underestimated. Finally, it is wise to consider whether a fast measurement or analysis time is worth it if the sum of all the other times is considerable. If the total throughput time is not much different a higher resolution but slower technique is a better choice. Definitions Accuracyis a measure of how close an experimental result is to the "true" value. For irregularly shaped particles or techniques that cannot be calibrated, or for any other set of conditions where a "true" value is either unknown, or not well defined, then accuracy has no meaning. For spheres and other simple shapes accuracy can be established between several techniques. Precision is a measure of the variation in repeated measurements under the same conditions instrument, sample, and operator. Accuracy associated with systematic error and precision associated with random error are related: However, if a measurement is highly accurate, then repeated measurements must have grouped around the true value. Still, accurate mean values may consist of either high or low precision. In such cases, precision limits accuracy. Precision limits resolution and reproducibility and is a useful criterion by which to assess instruments even if the accuracy cannot be determined. Resolution is a measure of the minimum detectable differences between distinct features in a size distribution. For broad, unimodal distributions, resolution is still an important concept. If the measured breadth of a distribution is meaningful, then the instrument that produces it should be able to separate narrow size peaks closer than or equal to that breadth. Otherwise, the measured breadth is really an

instrumental broadening effect. Resolution is a function of the signal-to-noise ratio of the instrument. Reporting more than this is like magnifying the noise; more numbers are obtained but they are meaningless. However, if the fundamental resolution of an instrument is undetermined then how can one know if the broad distribution is really hiding practical and, possibly, significant information? Reproducibilityis a measure of the variation between different machines, operators, sample preparations, etc. It becomes most important when comparing the results produced on two different machines of the same type. It is surprising how often the resolution, expressed as a range of values, exceeds the basic precision for any one of the machines. In such cases, it is useful to have round-robin tests conducted on the same sample, and under the same set of prescribed conditions, to isolate any machine-to-machine variations. A classic example is the big differences obtained on FD instruments with high angle light scattering detectors from the same manufacturer because of evolving software variations on how best to handle the necessary light scattering Mie corrections. These include the following: Is training, service, and applications assistance available during the installation, warranty period and for as long as the instrument is still serviceable? An instrument might be available at a lower price from a supplier in another country but check that it comes with the expected type and level of support. Ask for references to verify any claims that are made. Ask also about any continuing program of development to ward against obsolescence. This is a very subjective concept. Will the instrument be used by experts or by inexperienced users? If this concept is important then, initially, be sure to watch measurements being made â€" the entire process from sample preparation to clean-up. This is defined as the ability to measure a wide variety of samples under a wide variety of conditions. Does the instrument handle samples in air, liquids, or both? Does the instrument work with polar as well as nonpolar liquids? Does the instrument work with dilute samples or concentrates or both? Try to estimate a realistic range of sample types and the corresponding size ranges intended to be measured. Experience has shown that it is usually better to choose dedicated instruments that do a good job for their intended purpose rather than a poor job on a wide variety of samples. The basic instrument cost is only one factor to consider. The total price is best judged in terms of the life-cycle cost. This includes purchase price, operating cost, maintenance, and repair costs. Every instrument needs some type of maintenance. It may be as simple as cleaning air filters once a month; it may be as difficult as replacing mechanical parts or aligning an optical system. And every instrument will, sooner or later, require repairs. If labor is intensive, the life-cycle cost can be quite high. If special solvents or expensive environmental costs are involved, the life-cycle cost may be high enough to consider alternate choices. Of all these qualitative considerations, support is, perhaps, the most important. When choosing between vendors of similar equipment, the one with better support may tip the scale in its favor. Do not assume that the largest vendor, or the one with the fanciest brochure, will provide the best support. Today, many companies use representatives to sell and service instruments. Just as you would choose any professional service, asking for references and getting second opinions should be an integral part of the purchase process. Identify techniques whose mid range covers your expected size range. Get some preliminary measurements made but pay attention to sampling and sample preparation. The biggest mistake at this point is to choose the apparent zero-to-infinity devices. Given the list, narrow it further by deciding if you need imaging irregular particle shapes that correlate with end-product performance or not, single particle counting absolute concentration or not, and what degree of information you require. Now carefully consider the quantitative and qualitative specifications, giving the most weight to those aspects that pertain to your situation. While automated, high throughput instrumentation is convenient, if it sacrifices the resolution you need to make good decisions, consider carefully. Accuracy, precision, resolution and reproducibility are functions of the size range. Errors are always greatest at the extremes. A common mistake is to check an instrument in its midrange and then proceed to use it at one or other of the extremes. Be skeptical of claims if these refer only to the average size. The average of any distribution is least subject to variation. Higher moments such as the measure of width, or skewness, are much more sensitive to uncertainties; so pay particular attention to the variance in these statistics. Finally, before purchasing ask the vendor for a list of users who have had the instrument for at least one year. Contact them and ask for their experience with maintenance and repairs. Provder ed , Particle Size Distribution: Particle and Particle Systems Characterization, 12 C. Journal Pharmaceutics, Particle Sciences is

a leading integrated provider of formulation and analytic services and both standard and nanotechnology approaches to drug development and delivery.

Chapter 2 : Particle Size Distribution

Specialists in the field discuss the latest developments in particle size analysis, presenting an overview of state-of-the-art methodologies and data interpretation. Topics include commercial instrumentation, photon correlation spectroscopy, Fraunhofer Diffraction, field-flow fractionation, and detection systems for particle chromatography.

Operation[edit] The sample mixture to be separated and analyzed is introduced, in a discrete small volume typically microliters , into the stream of mobile phase percolating through the column. The components of the sample move through the column at different velocities, which are a function of specific physical interactions with the adsorbent also called stationary phase. The velocity of each component depends on its chemical nature, on the nature of the stationary phase column and on the composition of the mobile phase. The time at which a specific analyte elutes emerges from the column is called its retention time. The retention time measured under particular conditions is an identifying characteristic of a given analyte. Many different types of columns are available, filled with adsorbents varying in particle size, and in the nature of their surface "surface chemistry". The use of smaller particle size packing materials requires the use of higher operational pressure "backpressure" and typically improves chromatographic resolution i. Sorbent particles may be hydrophobic or polar in nature. Common mobile phases used include any miscible combination of water with various organic solvents the most common are acetonitrile and methanol. The aqueous component of the mobile phase may contain acids such as formic, phosphoric or trifluoroacetic acid or salts to assist in the separation of the sample components. The composition of the mobile phase may be kept constant "isocratic elution mode" or varied "gradient elution mode" during the chromatographic analysis. Isocratic elution is typically effective in the separation of sample components that are very different in their affinity for the stationary phase. In gradient elution the composition of the mobile phase is varied typically from low to high eluting strength. Periods of constant mobile phase composition may be part of any gradient profile. A rotary fraction collector collecting HPLC output. The system is being used to isolate a fraction containing Complex I from E. About 50 litres of bacteria were needed to isolate this amount. Depending on their affinity for the stationary and mobile phases analytes partition between the two during the separation process taking place in the column. This partitioning process is similar to that which occurs during a liquidâ€”liquid extraction but is continuous, not step-wise. The choice of mobile phase components, additives such as salts or acids and gradient conditions depends on the nature of the column and sample components. Often a series of trial runs is performed with the sample in order to find the HPLC method which gives adequate separation. History and development[edit] Prior to HPLC scientists used standard liquid chromatographic techniques. Liquid chromatographic systems were largely inefficient due to the flow rate of solvents being dependent on gravity. Separations took many hours, and sometimes days to complete. Gas chromatography GC at the time was more powerful than liquid chromatography LC , however, it was believed that gas phase separation and analysis of very polar high molecular weight biopolymers was impossible. Early developmental research began to improve LC particles, and the invention of Zipax, a superficially porous particle, was promising for HPLC technology. Researchers began using pumps and injectors to make a rudimentary design of an HPLC system. The practical disadvantages stem from the excessive pressure drop needed to force mobile fluid through the column and the difficulty of preparing a uniform packing of extremely fine materials. The Nobel Prize in chemistry was earned by Archer John Porter Martin and Richard Laurence Millington Synge for their development of the technique, which was used for their separation of amino acids. Analyte molecules partition between a liquid stationary phase and the eluent. HILIC most often uses a bonded polar stationary phase and a mobile phase made primarily of acetonitrile with water as the strong component. Partition HPLC has been used historically on unbonded silica or alumina supports. Each works effectively for separating analytes by relative polar differences. HILIC bonded phases have the advantage of separating acidic , basic and neutral solutes in a single chromatographic run. The stronger the interactions between the polar analyte and the polar stationary phase relative to the mobile phase the longer the elution time. The interaction strength depends on the functional groups part of the analyte molecular structure, with more polarized groups e. Coulombic

electrostatic interactions can also increase retention. Use of more polar solvents in the mobile phase will decrease the retention time of the analytes, whereas more hydrophobic solvents tend to increase retention times. Normalâ€”phase chromatography[edit] Normalâ€”phase chromatography was one of the first kinds of HPLC that chemists developed. Also known as normal-phase HPLC NP-HPLC this method separates analytes based on their affinity for a polar stationary surface such as silica, hence it is based on analyte ability to engage in polar interactions such as hydrogen-bonding or dipole-dipole type of interactions with the sorbent surface. The analyte associates with and is retained by the polar stationary phase. Adsorption strengths increase with increased analyte polarity. The interaction strength depends not only on the functional groups present in the structure of the analyte molecule, but also on steric factors. The effect of steric hindrance on interaction strength allows this method to resolve separate structural isomers. The use of more polar solvents in the mobile phase will decrease the retention time of analytes, whereas more hydrophobic solvents tend to induce slower elution increased retention times. Very polar solvents such as traces of water in the mobile phase tend to adsorb to the solid surface of the stationary phase forming a stationary bound water layer which is considered to play an active role in retention. This behavior is somewhat peculiar to normal phase chromatography because it is governed almost exclusively by an adsorptive mechanism i. Adsorption chromatography is still widely used for structural isomer separations in both column and thin-layer chromatography formats on activated dried silica or alumina supports. Partition- and NP-HPLC fell out of favor in the s with the development of reversed-phase HPLC because of poor reproducibility of retention times due to the presence of a water or protic organic solvent layer on the surface of the silica or alumina chromatographic media. This layer changes with any changes in the composition of the mobile phase e. Recently, partition chromatography has become popular again with the development of Hilic bonded phases which demonstrate improved reproducibility, and due to a better understanding of the range of usefulness of the technique. Displacement chromatography[edit] The basic principle of displacement chromatography is: A molecule with a high affinity for the chromatography matrix the displacer will compete effectively for binding sites, and thus displace all molecules with lesser affinities. In elution mode, substances typically emerge from a column in narrow, Gaussian peaks. Wide separation of peaks, preferably to baseline, is desired in order to achieve maximum purification. The speed at which any component of a mixture travels down the column in elution mode depends on many factors. But for two substances to travel at different speeds, and thereby be resolved, there must be substantial differences in some interaction between the biomolecules and the chromatography matrix. Operating parameters are adjusted to maximize the effect of this difference. In many cases, baseline separation of the peaks can be achieved only with gradient elution and low column loadings. Thus, two drawbacks to elution mode chromatography, especially at the preparative scale, are operational complexity, due to gradient solvent pumping, and low throughput, due to low column loadings. Because the process takes advantage of the nonlinearity of the isotherms, a larger column feed can be separated on a given column with the purified components recovered at significantly higher concentration. Reversed-phase chromatography RPC [edit] Further information: With such stationary phases, retention time is longer for molecules which are less polar, while polar molecules elute more readily early in the analysis. An investigator can increase retention times by adding more water to the mobile phase; thereby making the affinity of the hydrophobic analyte for the hydrophobic stationary phase stronger relative to the now more hydrophilic mobile phase. Similarly, an investigator can decrease retention time by adding more organic solvent to the eluent. RP-HPLC operates on the principle of hydrophobic interactions, which originates from the high symmetry in the dipolar water structure and plays the most important role in all processes in life science. The binding of the analyte to the stationary phase is proportional to the contact surface area around the non-polar segment of the analyte molecule upon association with the ligand on the stationary phase. This solvophobic effect is dominated by the force of water for "cavity-reduction" around the analyte and the Cchain versus the complex of both. The energy released in this process is proportional to the surface tension of the eluent water: The retention can be decreased by adding a less polar solvent methanol, acetonitrile into the mobile phase to reduce the surface tension of water. Gradient elution uses this effect by automatically reducing the polarity and the surface tension of the aqueous mobile phase during the course of the analysis. Structural properties of the

analyte molecule play an important role in its retention characteristics. In general, an analyte with a larger hydrophobic surface area Câ€”H, Câ€”C, and generally non-polar atomic bonds, such as S-S and others is retained longer because it is non-interacting with the water structure. Such interactions are subject to steric effects in that very large molecules may have only restricted access to the pores of the stationary phase, where the interactions with surface ligands alkyl chains take place. Such surface hindrance typically results in less retention. Retention time increases with hydrophobic non-polar surface area. Branched chain compounds elute more rapidly than their corresponding linear isomers because the overall surface area is decreased. Aside from mobile phase surface tension organizational strength in eluent structure , other mobile phase modifiers can affect analyte retention. For example, the addition of inorganic salts causes a moderate linear increase in the surface tension of aqueous solutions ca. This technique is used for mild separation and recovery of proteins and protection of their biological activity in protein analysis hydrophobic interaction chromatography, HIC. Another important factor is the mobile phase pH since it can change the hydrophobic character of the analyte. For this reason most methods use a buffering agent , such as sodium phosphate , to control the pH. Buffers serve multiple purposes: Ammonium formate is commonly added in mass spectrometry to improve detection of certain analytes by the formation of analyte-ammonium adducts. A volatile organic acid such as acetic acid , or most commonly formic acid , is often added to the mobile phase if mass spectrometry is used to analyze the column effluent. Trifluoroacetic acid is used infrequently in mass spectrometry applications due to its persistence in the detector and solvent delivery system, but can be effective in improving retention of analytes such as carboxylic acids in applications utilizing other detectors, as it is a fairly strong organic acid. The effects of acids and buffers vary by application but generally improve chromatographic resolution. Reversed phase columns are quite difficult to damage compared with normal silica columns; however, many reversed phase columns consist of alkyl derivatized silica particles and should never be used with aqueous bases as these will destroy the underlying silica particle. They can be used with aqueous acid, but the column should not be exposed to the acid for too long, as it can corrode the metal parts of the HPLC equipment. RP-HPLC columns should be flushed with clean solvent after use to remove residual acids or buffers, and stored in an appropriate composition of solvent. The metal content of HPLC columns must be kept low if the best possible ability to separate substances is to be retained. It is generally a low resolution chromatography and thus it is often reserved for the final, "polishing" step of the purification. It is also useful for determining the tertiary structure and quaternary structure of purified proteins. SEC is used primarily for the analysis of large molecules such as proteins or polymers. SEC works by trapping these smaller molecules in the pores of a particle. The larger molecules simply pass by the pores as they are too large to enter the pores. Larger molecules therefore flow through the column quicker than smaller molecules, that is, the smaller the molecule, the longer the retention time. This technique is widely used for the molecular weight determination of polysaccharides. SEC is the official technique suggested by European pharmacopeia for the molecular weight comparison of different commercially available low-molecular weight heparins.

Chapter 3 : Measurement of particle size distributions

Description Specialists in the field discuss the latest developments in particle size analysis, presenting an overview of state-of-the-art methodologies and data interpretation.

Types[edit] The way PSD is usually defined by the method by which it is determined. The most easily understood method of determination is sieve analysis , where powder is separated on sieves of different sizes. Thus, the PSD is defined in terms of discrete size ranges: The PSD is usually determined over a list of size ranges that covers nearly all the sizes present in the sample. Some methods of determination allow much narrower size ranges to be defined than can be obtained by use of sieves, and are applicable to particle sizes outside the range available in sieves. However, the idea of the notional "sieve", that "retains" particles above a certain size, and "passes" particles below that size, is universally used in presenting PSD data of all kinds. The PSD may be expressed as a "range" analysis, in which the amount in each size range is listed in order. It may also be presented in "cumulative" form, in which the total of all sizes "retained" or "passed" by a single notional "sieve" is given for a range of sizes. Range analysis is suitable when a particular ideal mid-range particle size is being sought, while cumulative analysis is used where the amount of "under-size" or "over-size" must be controlled. The way in which "size" is expressed is open to a wide range of interpretations. A simple treatment assumes the particles are spheres that will just pass through a square hole in a "sieve". In practice, particles are irregular â€” often extremely so, for example in the case of fibrous materials â€” and the way in which such particles are characterized during analysis is very dependent on the method of measurement used. Sampling[edit] Before a PSD can be determined, it is vital that a representative sample is obtained. In the case where the material to be analysed is flowing, the sample must be withdrawn from the stream in such a way that the sample has the same proportions of particle sizes as the stream. The best way to do this is to take many samples of the whole stream over a period, instead of taking a portion of the stream for the whole time. The material to be analysed must be carefully blended, and the sample withdrawn using techniques that avoid size segregation, for example using a rotary divider [3] p. Particular attention must be paid to avoidance of loss of fines during manipulation of the sample. Sieve analysis[edit] This continues to be used for many measurements because of its simplicity, cheapness, and ease of interpretation. Methods may be simple shaking of the sample in sieves until the amount retained becomes more or less constant. Alternatively, the sample may be washed through with a non-reacting liquid usually water or blown through with an air current. Two common uses in the powder industry are wet-sieving of milled limestone and dry-sieving of milled coal. Another disadvantage is that the amount of energy used to sieve the sample is arbitrarily determined. Over-energetic sieving causes attrition of the particles and thus changes the PSD, while insufficient energy fails to break down loose agglomerates. Although manual sieving procedures can be ineffective, automated sieving technologies using image fragmentation analysis software are available. These technologies can sieve material by capturing and analyzing a photo of material. Air elutriation analysis[edit] Material may be separated by means of an elutriator, which consists of a vertical tube up which fluid is passed at a controlled velocity. When the particles are introduced, often through a side tube, the smaller particles are carried over in the fluid stream while the large particles settle against the upward current. If we start with low flow rates small less dense particle attain terminal velocities, and flow with the stream, the particle from the stream is collected in overflow and hence will be separated from the feed. Flow rates can be increased to separate higher size ranges. Further size fractions may be collected if the overflow from the first tube is passed vertically upwards through a second tube of greater cross-section, and any number of such tubes can be arranged in series. Each cut-point can be recovered for future size-respective chemical analyses. This technique has been used for decades in the air pollution control industry data used for design of control devices. This technique determines particle size as a function of settling velocity in an air stream as opposed to water, or some other liquid. It is a fairly time-consuming analytical technique. The actual test method [4] has been withdrawn by ASME due to obsolescence. Instrument calibration materials are therefore no longer available. Optical granulometry Materials can now be analysed through photoanalysis procedures.

Unlike sieve analyses which can be time-consuming and inaccurate, taking a photo of a sample of the materials to be measured and using software to analyze the photo can result in rapid, accurate measurements. Another advantage is that the material can be analyzed without being handled. This is beneficial in the agricultural industry, as handling of food products can lead to contamination. Photoanalysis equipment and software is currently being used in mining, forestry and agricultural industries worldwide. Optical counting methods[edit] PSDs can be measured microscopically by sizing against a graticule and counting, but for a statistically valid analysis, millions of particles must be measured. This is impossibly arduous when done manually, but automated analysis of electron micrographs is now commercially available. It is used to determine the particle size within the range of 0. Electroresistance counting methods[edit] An example of this is the Coulter counter , which measures the momentary changes in the conductivity of a liquid passing through an orifice that take place when individual non-conducting particles pass through. The particle count is obtained by counting pulses. This pulse is proportional to the volume of the sensed particle. The results are only related to the projected cross-sectional area that a particle displaces as it passes through an orifice. This is a physical diameter, not really related to mathematical descriptions of particles e. Sedimentation techniques[edit] These are based upon study of the terminal velocity acquired by particles suspended in a viscous liquid. Typical apparatus disperses the sample in liquid, then measures the density of the column at timed intervals. Other techniques determine the optical density of successive layers using visible light or x-rays. Sample must be dispersed in a liquid medium Density is highly dependent upon fluid temperature remaining constant. X-Rays will not count carbon organic particles. Many of these instruments can require a bulk sample e. Laser diffraction methods[edit] These depend upon analysis of the "halo" of diffracted light produced when a laser beam passes through a dispersion of particles in air or in a liquid. The angle of diffraction increases as particle size decreases, so that this method is particularly good for measuring sizes between 0. Advances in sophisticated data processing and automation have allowed this to become the dominant method used in industrial PSD determination. This technique is relatively fast and can be performed on very small samples. A particular advantage is that the technique can generate a continuous measurement for analyzing process streams. Laser diffraction measures particle size distributions by measuring the angular variation in intensity of light scattered as a laser beam passes through a dispersed particulate sample. Large particles scatter light at small angles relative to the laser beam and small particles scatter light at large angles, as illustrated below. The angular scattering intensity data is then analyzed to calculate the size of the particles responsible for creating the scattering pattern, using the Mie theory of light scattering. The particle size is reported as a volume equivalent sphere diameter. Each randomly scanned particle obscures the laser beam to its dedicated photo diode, which measures the time of obscuration. Acoustic spectroscopy or ultrasound attenuation spectroscopy[edit] Instead of light , this method employs ultrasound for collecting information on the particles that are dispersed in fluid. Dispersed particles absorb and scatter ultrasound similarly to light. This has been known since Lord Rayleigh developed the first theory of ultrasound scattering and published a book "The Theory of Sound" in The resulting ultrasound attenuation frequency spectra are the raw data for calculating particle size distribution. It can be measured for any fluid system with no dilution or other sample preparation. This is a big advantage of this method. However, as concentration increases and the particle sizes approach the nanoscale, conventional modelling gives way to the necessity to include shear-wave re-conversion effects in order for the models to accurately reflect the real attenuation spectra. Cascade impactors use the principle of inertial separation to size segregate particle samples from a particle laden gas stream. The mass of each size fraction is determined gravimetrically. The California Air Resources Board Method [8] is currently the most widely accepted test method for particle size distribution emissions measurements. Probability distributions[edit] The log-normal distribution is often used to approximate the particle size distribution of aerosols , aquatic particles and pulverized material. The Weibull distribution or Rosinâ€”Rammler distribution is a useful distribution for representing particle size distributions generated by grinding, milling and crushing operations. The log-hyperbolic distribution was proposed by Bagnold and Barndorff-Nielsen [9] to model the particle-size distribution of naturally occurring sediments. This model suffers from having non-unique solutions for a range of probability coefficients. The skew log-Laplace model was proposed by Fieller, Gilbertson and Olbricht [10]

as a simpler alternative to the log-hyperbolic distribution. It is still widely used in mineral processing to describe particle size distributions in comminution processes.

Chapter 4 : Particle Size Analysis| 6 Methods Used For Particle size distribution

Barth, Howard G. , Modern methods of particle size analysis / edited by Howard G. Barth Wiley New York Wikipedia Citation Please see Wikipedia's template documentation for further citation fields that may be required.

Compare Methods of Particle Size Analysis The use of sieves to separate particulate material into fractions of various sizes has occurred throughout the period of recorded history. Sieving is one of the simplest methods of determining particle size distributions, and is probably used in industrial laboratories more than any other method. It is generally overlooked when carrying out fundamental research, which is unfortunate, since it can be a very accurate technique. Sieving has been classed along with optical and electron microscopy as a method which classifies particles according to geometric similarity, regardless of density or optical properties. Sieves with openings smaller than about 50 microns are seldom used for determining the size of dry powders. However, membranes having submicroscopic pores have been used to estimate the size of particles in colloidal solutions, and membrane filters with pore diameters of about 1 micron have been suggested for separating the particles in aerosols into two size fractions. Types of Sieves Most sieves which are used for Particle Size Distribution determinations have square openings. However, circular and rectangular openings, and even slits, are sometimes used. Both the shape of the particle and the shape of the opening affect the size of particle which can pass through a given opening. However, the probability of such a particle passing through such a hole when it is adjacent to the hole is quite small because of the small probability that the particle will be properly oriented. Fortunately, the sizes of screen openings have been standardized. Two standard series of screen openings are used in the United States. The screens are made from woven wire cloth. The Tyler scale is based on the size of openings in wire cloth having openings per linear inch mesh. The diameter of the wire used for the mesh screens is 0. Thus the areas of the openings of each sieve are double those of the next finer sieve. Also, the ratio between the width of openings of alternate sieves in the series is 2. The Tyler scale is shown in Table I. Tyler screens are manufactured by the W. The National Bureau of Standards in proposed a series using the same ratio as the Tyler Standard Scale Sieve Series, but based on an opening of 1 mm. The differences between the two series are so small that, for most purposes, sets of screens designed according to the two series can be used interchangeably. Sieve Series is shown in Table II. The ratio is the same as for the U. The slight variations are due to the differences between U. Between and the British used a standard series adapted by the Institution of Mining and Metallurgy. There was no fixed ratio between the openings. Instead, the opening and wire diameter were the same. This proportion was used in the belief that this would minimize shifting of the wires. However, such shifting is not a major problem when modern methods of manufacturing wire screens are used, and the series has been largely abandoned in favor of the British Standard Sieve Series. The sieves are designated by number and also by the number of meshes per square centimeter. Sieves of either the Tyler Series or the U. Sieve Series can be obtained commercially which have been certified by the National Bureau of Standards as coming within the specified tolerances. The sieves are tested by actual measurement of the wire diameters and openings. The American Society for Testing Materials has set up standards for sieve cloth and frames. According to these standards, the cloth is woven not twilled, except the cloth of the , , , and micron sieves from brass, bronze, or other suitable wire, and shall not be coated or plated. Frames for all sieves of the fine series openings microns or less are circular and are 8 in. The latter are used primarily in the testing of paint pigments. The height of the sieve from the top of the frame to the cloth is either 2 in. Sets of standard testing sieves are shown in Figure 1. Methods of Making Particle Size Determinations Determinations of particle size distributions using sieves usually involve placing a sample on the wire mesh of the top sieve of a nest of sieves, shaking for a predetermined period of time, and weighing the portion of the sample retained on each sieve. The sieves in the nest are of progressively finer mesh, so the weights of the portions retained on the screen constitute a set of classified particle size distribution data. The weight of the sample screened is quite important. The weight of sample must not be too great, since oversize particles or even small particles may be jammed into the openings, clogging and distorting them. Also, the sieving time required to obtain reproducible results increases with increasing sample

size. On the other hand, a sufficiently large sample must be used; otherwise accuracy is lost in both sampling and weighing. One hundred grams is often a satisfactory size when 8-in. A method which has been suggested for determining the optimum sample size starts by splitting out, with a sample splitter, samples of varying weights, for instance 25, 50, , , and g. Each sample is sieved for the same length of time, for example 5 min. If the three smallest samples 25, 50, and g. The number of sieves used for a given determination depends largely on the reason for making the determination. If, for example, the only information required is the percentage of material less than a given size, a single screen is sufficient. If a particle size distribution must be obtained, five sieves is often a satisfactory number. The sieving operation starts by mounting the sieves in a nest, the coarsest at the top. The sample is placed on the top screen, and the screens are shaken for several minutes. It is generally impossible to screen a sample completely; the longer one shakes the screens, the more material comes through, although to a continuously diminishing extent. Therefore, if the results of sieve tests are to be compared, the method of shaking and the time of shaking should be standardized. The screens can be shaken by hand, either all at the same time or consecutively. However, mechanical shakers produce superior results be- cause of their uniformity of action. The Ro-Tap shaker, manufactured by the W. It im- parts a circular motion to the nest of screens while tapping them from above. Portable shakers and hand-driven shakers, such as the one shown in Figure 3, are also available. High-frequency vibrators, such as the Hummer electric and the Leahy No-Blind screen, are also available. Wet sieving techniques are of value for substances which tend to form aggregates during shaking, or which are already suspended in a liquid. Water or some other liquid is mixed with the material. The suspension is run through one or more sieves and additional liquid is poured over the residue. Sieves for wet testing are especially de- signed with deep frames which prevent loss of sample when washing the fines through the sieve. Wet sieving is commonly used to isolate oversize particles. It usually greatly increases the total amount of material passing through the sieve. However, Sehweyer prepared closely fractionated samples of a number of materials which passed a number sieve when dry but not when wet. Apparently the liquid breaks up aggregates and increases the agitation, but when added to an easily dispersed, closely fractionated sample it may increase the effective particle size. The sieving time is somewhat arbitrary. It should be sufficiently long that the changes produced by continued agitation can be considered to be negligible. If accurate comparisons of various materials are to be made, the sieving time, sample size, screens, and method of agitation should all be standardized. Shergold studied the effect of sieving time and sieving load on the efficiency of sieving sand. The efficiency decreased slowly as the sample weight was increased from to g. Shergold conchided that if the screens are not overloaded, that is, if the samples are between about 40 and g. Weber and Moran state that enough time must be allowed for the fractions retained on the several sieves to approach constancy, but that a protracted time allows additional opportunity for the closely sized particles to seek out openings slightly larger than average in each sieve. They suggest that the period after which the greatest sieve fraction begins to lose a constant amount for each succeeding minute is the optimum period on which to standardize. Details of the sieve analyses of a number of materials are given in the ASTM standards. An interesting procedure is that given for the sieve analysis for glass spheres used for highway traffic lines. Fifty grams of the dried glass spheres are placed on the screen with the smallest opening in the series designated for the test. The covered sieve is held with one hand in a slightly inclined position while tapping with the palm of the other hand at the rate of taps per minute. The sieve is turned one-sixth of a revolution every 25 strokes. Sieving is continued until not more than 0. The residue is weighed and placed on the sieve with the next larger opening. Sieving is continued in a similar manner using each sieve successively in the order of increasing size of opening. The material is weighed and the percentage of the sample passing each of the sieves is calculated. Calibration of Screens Sieves are usually calibrated by measuring with a microscope a number of openings and wires. The results are then compared with the specifications, such as those of the U. The National Bureau of Standards recommends measuring the diameters of 5 to 10 wires, making four measurements on each wire. The number of wires per centimeter are then determined and the average opening calculated. Weber and Moran measured representative groups of five adjacent individual openings along two diameters of the sieve parallel to the warp and woof, respectively, taking the same number of measurements in each direction. Two hundred measurements gave about the same accuracy for sieves No.

When the percentage standard deviation Y was less than 6, the sieves appeared to be satisfactory as judged from the results of sieving a number of standard samples. Moran and Weber suggested that when Y is greater than 6, the effective opening Xt for a shaking time of t minutes can be estimated using the empirical equation Sieve calibrations based on empirical corrections determined by sieving standard samples were not satisfactory. The correction depended not only on the sieve, but also on the slope of the cumulative curve. Corrections based on equation 1 added little to the accuracy of results obtained for regularly shaped particles having a wide particle size distribution, but greatly increased the accuracy of results obtained for closely sized but irregularly shaped particles. Equation 1 applies to plain-weave wire cloth but not twilled cloth. A glass scale is used which is carefully ruled.

Chapter 5 : Top 10 Particle Size Analyzer Priceï¸±Top 10 Used Particle Size Analyzerï¸±AimSizer

Find helpful customer reviews and review ratings for Modern Methods of Particle Size Analysis (Chemical Analysis: A Series of Monographs on Analytical Chemistry and Its Applications) at calendrierdelascience.com Read honest and unbiased product reviews from our users.

LASER particle size analysis. Direct microscopy or optical microscopy: This can be done by use of compound microscope. Here the compound microscope parts i. One can observe the particles directly for their shape and size through the microscope. Particles in the size range of 0. Here the particle dimensions are directly measured by observing into the microscope or by projecting on to a screen for ease. The field can even be photographed. A fixed quantity of powder is mixed with an oil and suspended on a slide. This slide is covered with another glass slit to prevent movement of particles. Then using the micrometer, a reflection of particle shape is created on a graph paper placed beside microscope. This shape is drawn with a pencil. After taking fixed number of particle drawings on graph paper the size is estimated by formulas. A large number of counting are needed to obtain statistically valid data. Around particles are to be measure to get the data. During measurement of particle size, dimension like a Martin diameter: Length of the line that bisects the particle irregular image. This method is used for particle size analysis in suspensions, aerosols and emulsions. It is simple and the particle shape can be directly seen. Agglomerates of particles can be seen and avoided while counting. During the measurement, the particle are not in motion and at rest which give precise measurement. Only length and breadth are measure but depth is avoided. So particle dimension is taken in 2 sides and not in all the 3 sides. The process is very slow and tedious. Large amount if sample is required. The size of particles is expressed as dseive which is the diameter of the sphere that passes through the sieve aperture. This is rough and fast method and one can obtain weight distribution of particle sizes. Sieves of different pore sizes are used to sieve the powder. The standard sieves with different mesh numbers as per U. P are available commercially. The sample of said quantity is placed on the top sieve and tapped. In doing so the particle with size larger than the particular sieve number are retained while others are passed off. Thus particles pass through sieves pores based on size and settle on different level of sieves. The smaller particles settle on the finest pore sieve i. In this manner the particle size distribution of the powder sample estimated using formulas. The method is faster, least expensive and results are reproducible. If the is wet, the pores of the sieves can get clogged with particles and sieving would be improper. Since the sieves are shaken, the particles collide with each other and there are chances of further size reduction which can lead to errors. We notice that when a particle falls into a liquid, it slowly settles down. The bigger and denser it settles fast. The smaller and lighter, settles slowly. This principle is used in particle size determination. Since in a given powder, all the particles are of same density, they settle only based on size. So large particles settle fast and at the bottom of the sediment. Similarly smaller ones settle slower and lie at the top of sediment. The size of particles here is expressed as stokes diameter dst. This represents the diameter of an equivalent sphere which has the same rate of sedimentation. There are different methods to carry out this procedure like anderson pipette method, balance method and hydrometer method. Here the particles are suspended in a liquid medium and allowed to sediment or settle down in a cylindrical tube. The rate of sedimentation varies based on the particle size. Hence different layers of sediment are formed. These different layer are taken as particles of particular size are only present in the sediment layer. The weight of the sediment layer is measured. This gives the weight of particles of particular size in the entire sample. Reliable as whole of sample is screened. But is a rough estimate of size in each layer. Here particle are suspended in a conductive solution like electrolyte solution. There are two chambers one inside other. An electrical resistance wire is suspended in the inner chamber. The solution divides two chamber by a small orifice. This is the method also used for measurement of size of bacteria and other microbes. The most reliable method as we get information of size of each and every particle. LASER particle size analyzer: This depend on the laser ray diffraction by the particle. This is suitable for online particle size determination. Reliable, less time consuming.

Chapter 6 : Modern Methods of Particle Size Analysis | Analytical Chemistry | Chemistry | Subjects | Wiley

Method: A fixed quantity of powder is mixed with an oil and suspended on a calendrierdelascience.com slide is covered with another glass slit to prevent movement of particles. Then using the micrometer, a reflection of particle shape is created on a graph paper placed beside microscope.

Amount of sample available for analysis. Budget Each of these points is discussed in more detail below. Current analysis technique or practice The choice of particle size analyzer often depends on previous experience. For example, knowing the sample size range requires some sort of size analysis, even if it is simply looking at a few particles under a microscope or rubbing them between your fingers to feel them. Understanding the current technique will help clarify expected results or new needs. Often, there is a body of literature or practice that will guide the decision making process. For example, a manufacturing plant may already have a size analyzer and they are looking for an improvement. Or, the scientific literature may be dominated by results from one technique. It is also useful to think about the importance of correlation to past results. Many find that an updated technique allows them to escape old analytical problems such as poor resolution and do not want any correlation. Size Range To choose between different techniques, the size range is usually the prime factor. The chosen technique should identify not just the median size, but the full range of sizes in the distribution. In cases where more than one instrument covers the desired size range, consider other possible samples and possible future developments. For example, a new product with a larger particle size may be introduced in the future. This may suggest that the analyzer with the higher upper size limit is a better choice than an analyzer with a smaller lower size limit. Figure 1 below shows the size ranges of some techniques. A note of caution: The stated size range for an analyzer does not apply for all samples and all cases. Choosing an analyzer such that the particle size of the sample is in the middle of the instrument size range is almost always wise. In general the data is better and you are better able to work with variations in particle size about your expected value. Approximate size ranges of common particle sizing techniques. Chemistry or Material The material to be analyzed is important in choosing a technique as material properties are important when presenting the sample to the analyzer. Here are some questions to ask: Is the material Free Flowing? A dispersion what liquid? Are there safety and environmental considerations? Different analytical techniques allow the use of different accessories or sampling systems that may be more appropriate for the material of interest. For example, a fragile agglomerated powder or highly-soluble material would be best analyzed as a dry powder by static light scattering instruments. Materials with limited availability or that are toxic or expensive would require a small volume measurement cell available with several different analyzer systems. Desired Information Different particle size analyzers present different information about the particle sample. If this additional information is important, it is worth learning if the sample is appropriate for a technique that provides the information. For example, if particle size is large enough, then image analysis can be used to determine both particle size and particle shape. Or, if the primary interest is dissolution or catalytic activity then surface area is critical and an SA surface area analyzer may be a wiser choice than a size analyzer. Desired information can also include different points about the distribution. For example, if quantifying large particle impurities in a material with small particles is important, than laser diffraction is often better than dynamic light scattering. Or, if the only important information is the size of the largest particles, an inexpensive Hegman gauge may be the best choice. Desired Throughput A large amount of information is useful for tracking process issues. A fast analysis means that a formulation scientist can quickly identify what strategy works. All of this requires throughput, either to run many samples in one day or to allow faster decision making. Throughput is best considered in terms of the overall measurement process, from sample preparation to measurement and reporting, until the instrument is clean and ready for the next analysis. Laser diffraction has the fastest throughput of the major analysis techniques, followed by dynamic light scattering, then dynamic image analysis. Amount of Sample Available for Analysis Some materials are manufactured by the ton. Some is synthesized by the milligram. Different particle analyzers and techniques require different amounts of samples, from micrograms to grams. Thus, sample amount can be a consideration.

Often, small sample quantities require slightly more expensive analyzers. And the smallest sample cells with volumes measured in microliters require careful attention to cleaning. For submicron samples, the lowest sample volumes are achieved with dynamic light scattering and just a few micrograms of particles are required. Budget The expense of poor or slow analysis results such as poor manufacturing quality must be balanced against the cost of analysis, including equipment purchase, consumables, and laboratory labor. Additional considerations are cost and frequency of down time. And finally, the availability of training materials and classes will help ensure that personnel remain up-to-date and new personnel are rapidly trained. That is, training costs are minimized. Modern analyzers tend to incorporate features that dramatically lower operating costs while slightly increasing initial costs. For example, high reliability, precision, and continued availability of training and support mean higher productivity due to tighter control on production processes. Automated measurements can provide constant feedback, reduce operator error, and allow the operator to focus on unusual events or process improvement rather than routine measurement activities. Concluding Comments Knowing the answers to the questions above will allow the analyst to rapidly identify appropriate particle analysis techniques. This information, along with results from analysis in a supplier applications lab or on-site demonstration will allow confident identification of the best technique and instrument for a particular laboratory.

Chapter 7 : Considerations in Particle Sizing: Specifying a Particle Size Analyzer

Specialists in the field discuss the latest developments in particle size analysis, presenting an overview of state-of-the-art methodologies and data interpretation.

Chapter 8 : Particle-size distribution - Wikipedia

Modern Laser Diffraction for Particle Size Analysis, an Introduction Particle Analysis Method Workflow.

Chapter 9 : Modern Methods of Particle Size Analysis - Philip J. Elving, James D. Winefordner - Google Bo

Considerations in Particle Sizing Part 2: Specifying a Particle Size Analyzer Technical Brief Volume 7 In Part 1 of this guide (Techni-cal Brief Volume 6) we stated.