The technology and application of sedimentation particle size measurement

1. General description

Sedimentation particle size measurement technology depends on the sedimentation rate of the particles in the liquid to measure the particle size distribution. The paper will introduce mainly the principle and usage of gravity sedimentation and centrifugal sedimentation light penetrating particle size analyzers. The pipette and sedimentation balance are used seldom now, so the paper will not introduce them.

As to sedimentation particle size analysis, first of all, the sample and liquid are mixed to make suspending liquid with certain consistency. The particles in the liquid begins to sedimentate due to gravity or centrifugal force, the sedimentation rate depends on the particle dimension, the sedimentation rate of the large particles is fast, and small particles is slow. The particle dimension and particle size distribution are measured according to different sedimentation rate.

But in fact, it is very difficult to measure the sedimentation rate of the particles. So the sedimentation rate is judged indirectly by measuring the variance ratio of suspending liquid consistency of some depth under liquid level, and the particle size distribution is also gotten.

Before large particles falls into the measurement area from liquid level, the consistency of this position is invariable; after large particles falls into the measurement area, the consistency of this position begins to drop, with the measurement process going on, the consistency will drop further, the measurement process is not over until all expected measured particles sedimentate under the measurement area. See diagram:

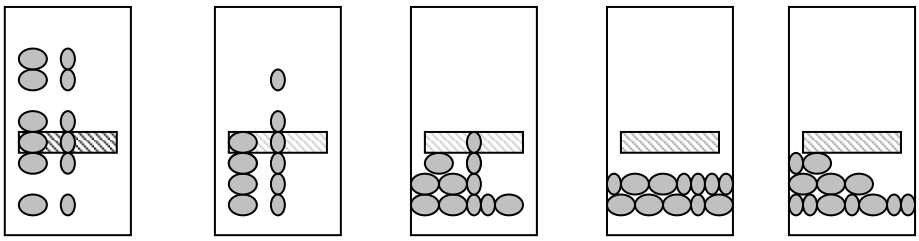

Diagram 1, the sedimentation state figured diagram of particles in the liquid

Then, what relation is between the sedimentation rate of the particle and the particle diameter? Stokes law tells us, under certain situation, the sedimentation rate of the particle is direct proportion to square particle diameter, inverse proportion to the viscosity of liquid. For large particles, we choose bigger viscosity liquid as medium to control the sedimentation rate of the particles in the gravity field center. For small particles, the sedimentation rate under the gravity is slow, adding the effects of Brownian motion, temperature, and other situation, so the measurement error will increase. In order to get over these disadvantageous factors, adopt centrifugal means to raise the sedimentation rate of fine particles. So the available sedimentation

particle size analyzers combine the gravity sedimentation with centrifugal sedimentation two measurement means, which may measure the course samples by the gravity sedimentation, also the fine samples by the centrifugal sedimentation.

New sedimentation particle size analyzers integrate traditional theory with modern technology. The computer technology, microelectronic technology and even Internet technology have been used, and the intellectualization and automation etc have made great progress. Common types are BT-1500、SA-CP3、SKC-2000 etc. The features are:

1. Convenient operation and maintenance, low price.
2. Long continuous running time, even up to above 12 hours.
3. Low running cost, few samples, low medium quantity, less wear parts.
4. Wide measure range, usually up to 0.1~200μ.
5. Short measure time, commonly 10min/time.
6. Low circumstance requirement, only room temperature.

Because the shapes of most of actual particles are nonsperical, it is impossible to show their dimension in a value. As other type particle size analyzers, the sedimentation particle size analyzer measures equivalent particle diameter of the particles, called Stokes diameter. Stokes diameter is the diameter of some consubstantial sphere that has the same sedimentation rate as the particle measured under certain situation. When the particle measured is spherical, Stokes diameter and the real diameter of the particle is consistent.

2. The principle

1) Stokes law

We know, that the sedimentation particle size analyzer measures the particle size distribution by way of the sedimentation rate in liquid. When particle is subsiding in liquid, there are three kinds of forces on the particle, downward gravity W, upward buoyancy V, upward resistance FD. According to Newton's sports law, its kinematic equation is:

$$Mg - M'g - F_D = M\frac{du}{dt} \text{ ----------(1)}$$

In the equation, M is the weight of the particle, and M` is the weight of the liquid that has the same volumes as the particle, and U is the speed of the particle, and G is the gravity acceleration, and T is time, and FD is the viscosity resistance.

When the gravity, buoyancy and viscosity resistance reach the balance, the sedimentation rate of particle is constant, and the particle is in the uniformly sedimentation state. At this moment,

$$\frac{du}{dt} = 0, \textbf{From } (1) \textbf{ Then } F_D = (M - M')g = \frac{\pi}{6}(\rho_s - \rho_f)gD^3 \text{ -----(2)}$$

In the equation, D is the particle diameter，ρs is the density of the sample, ρf is the density of the medium.

In hydromechanics, in order to be easy to study and express, we use a kind of characteristic measureless constant Renault number. Its definition as follow:

$$R_e = \frac{\rho_f uD}{\eta} \quad ----------(3)$$

Renault number Re indicates the ratio between the inertial force and the viscosity resistance when the fluid is flowing. The ratio could be neglected when Re is very little, and at this moment, the resistance of the particle is completely from the viscosity resistance of liquid. It can be expressed as following:

$$F_D = 3\pi D\eta u \quad ----------(4)$$

In the equation, u is the sedimentation rate of the particle, and η is the viscosity coefficient of the medium. This is Stokes resistance formula. In order to be easy to study, we introduce the concept of resistance coefficient. Its definition as follow:

$$C_D = \frac{F_D}{\frac{\rho_f u^2}{2} \times \frac{\pi D^2}{4}} = \frac{F_D}{AB} \quad ----------(5)$$

In the equation, A is the kinetic energy of unit volume fluid; B is the projection area in the direction of particle movement. We get from formula (4) and (5):

$$C_D = \frac{24}{R_e} \quad ----------(6)$$

When Re is very little, the resistance coefficient is very large. When Re<0.2, the fluid is in the laminar flow area, when 0.2<Re<2000, in the middle area, and when Re>2000, in onflow area.

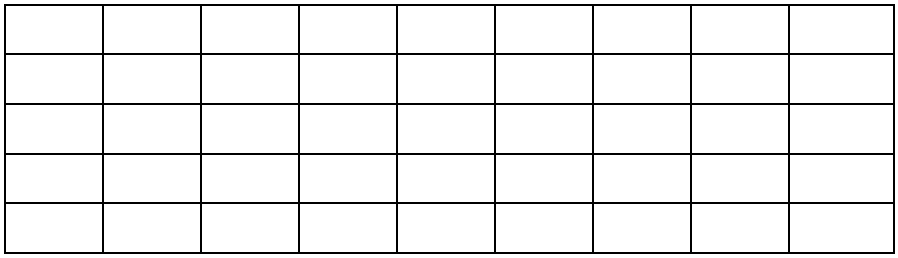

Diagram 2，the relation curve between Renault number and resistance coefficient

The scope of application of Stokes law is in the laminar flow area.

According to equation (4), we know that the resistance of the sedimentating particle is increasing along with the sedimentation speed increasing. The gravity and resistance reach the balance when the sedimentation speed increases to some extent. At this time, if we put the formula （4） into formula, we can get:

$$u = \frac{(\rho_s - \rho_f)gD^2}{18\eta} \text{----------} (7)$$

This is Stokes law.

Stokes law explains the relation between sedimentation rate and particle diameter under the laminar flow condition. It is the theory base of sedimentation method to measure particle size.

2)、The critical diameter of gravity sedimentation

The above discussion shows when Re>0.2，Stokes formula doesn't hold true，so when Re=0.2, the calculated diameter Ds is the critical diameter of gravity sedimentation. Merge the formula（3） and（7）:

$$D_s^3 = \frac{3.6\eta^2}{(\rho_s - \rho_f)\rho_f g} \text{----------} (8)$$

When the largest measured particle diameter is less than the critical diameter, the measured value is valid, or the error is very big.

The table 1 gives the critical diameter of different status (20℃, unit: mm)

Medium / Sample	**Water**	**Alcohol**	**Water+20%glycerin**	**Water+40%glycerin**
Graphite	67.4	77.9	127.0	195.1
Quartz	60.6	72.2	113.8	173.8
Talcum	60.0	70.4	112.6	171.9

Kaolin	60.0	70.4	112.6	171.9
Heavy calcium	60.0	70.4	112.6	171.9
Aluminium powder	60.0	70.4	112.6	171.9
Silicon carbide	55.0	65.2	103.0	156.9
Zirconium silicate	46.6	57.5	86.9	131.8
Zinc powder	39.0	47.2	72.8	110.0
Molybdenum	34.0	41.4	63.6	96.0
Tungsten powder	27.0	33.0	50.5	76.2

Table 1 the critical diameter of gravity sedimentation

When the largest particles of measured sample are more than the critical diameter, take measures to change the test situation. We know from the formula (8) and table (1), increasing medium viscosity may raise the critical diameter, so we take usually glycerin and water solution as the sedimentation medium of course samples.

It should be pointed that the critical diameter in table 1 is theoretical value, in practice, the instrument factor must be considered.

3) Centrifugal sedimentation

We adopt the centrifugal sedimentation to quicken the sedimentation rate of fine particles. This will shorten the measurement time and raise the measurement precision. Under the centrifugal state, two forces, centrifugal force and resistance act on the particles. The formula in laminar flow area:

$$\frac{\pi}{6}(\rho_s-\rho_f)D^3\frac{d^2x}{dt^2}=\frac{\pi}{6}(\rho_s-\rho_f)D^3\omega^2x-3\pi D\eta\frac{dx}{dt}\text{-------}(9)$$

x：the distance from axes to particle，

dx/dt：the sedimentation rate of the particle，

ω：the revolution of centrifuger（r/s）；

when the centrifugal force and resistance are banlance，the movement of the particle is:

uniform velocity, the formula（9）becomes：

$$\frac{dx}{dt}=u_c=\frac{\rho_s-\rho_f}{18\eta}D^2\omega^2x\text{----------}(10)$$

This is the expression of Stokes law in the centrifugal state. It shows that the sedimentation rate of the particle has relation with particle diameter, the revolution of centrifuger and the distance from axes to particle.

The ratio between Stokes gravity sedimentation formula and the formula（10）:

$$\frac{u_c}{u} = \frac{\omega^2 x}{g} \text{----------(11)}$$

The critical diameter of centrifugal sedimentation:

$$D_{s离心}^{\ 3} = \frac{3.6\eta^2}{(\rho_s - \rho_f)\rho_f \omega^2 X} \text{----------(12)}$$

X is the distance from axes to measure position

The gravity sedimentation is use to measure the particle size of course particles, the lower limit is about 3μm; The centrifugal sedimentation is used to measure fine samples; the lower limit can be gotten from the following formula:

$$D_{\min} = \sqrt[3]{\frac{1200RT\ln(\frac{r}{s})}{\pi L \Delta\rho\omega^2 (r-s)^2}} \text{----------(13)}$$

“s” is the distance from the axes to liquid level，r is the distance from the measurement position to axes，L is Avogadro constant，R is gas constant，T is absolute temperature.

When T=300K，s=0.04m，r=0.07m，Δρ=1000，ω=838rad/s，Dmin=0.0112μm。

4) A few effect factors of sedimentation rate

(1) Brownian motion

Brownian motion is a kind of irregular motion of liquid molecular. When the particles suspending in the liquid are enough little, the irregular motion of liquid molecular will knock on the particles to occur obvious displacement, and the displacement influences the directional motion of the particles in the medium.

Table 2 the displacement of the particles which density is 2 in one second acted by Brownian motion, gravity and centrifugal force (water medium)

Particle diameter (μm)	The movement distance in water（μm）		
	Brownian motion （μm）	Gravity （μm）	Centrifugal force （μm）
0.10	2.36	0.005	2.50

0.25	1.49	0.0346	8.12
0.50	1.052	0.1384	32.45
1.0	0.745	0.554	130.95
2.5	0.334	13.84	846.15
10	0.236	55.4	13000

For the particles below 1μm, the Brownian motion displacement is more than the gravity displacement，but less than the centrifugal displacement, so the centrifugal sedimentation can get over the effect of Brownian motion to fine particles.

(2) The time reaching the uniform velocity motion

When stop stirring, the motion state of the particles will transit to uniform velocity sedimentation state. What time is from stop to uniform velocity motion? The modern theory deduction and experiments show that when Re is very little，the time is very short, the displacement is very little, so the effect can be neglected. See table 3：

Particle diameter（um）	The time reaching the uniform velocity motion in water（ms）
5	0．017
10	0．068
50	1．70

Table 3，The time reaching the uniform velocity motion in water of different particles

(3) The effect of consistency

Stokes law is effective when the consistency is every low. When consistency increases, the particles will interact to change the sedimentation rate. The reasons are so. First, the consistency increasing will make the particles agglomerate easily and hasten the sedimentation rate. Second, the velocity field produced by particles will raise the sedimentation rate of other particles. Third, there is compensative motion of fluid upward reducing the sedimentation rate. Research indicates that when consistency is 0.3%， the error of the particle size is 4%. When the distance of two particles is more 10 times than particle diameter, the interaction can be neglected. The consistency of suspending liquid is commonly 0.02%~0.2%. Every instrument is specified certain allowable consistency range and control method.

But if the consistency is extremely little, the representative of the samples is bad, and will brings bigger error to measured outcome.

(4) The effect of nonspherical particles

Stokes law applies to the spherical particles. But in fact, the particle shape is very complicated.

Their sedimentation regulation and sedimentation rate are different from the spherical particles. The sedimentation rates of irregular particles are different and have a scope. The experiment indicates that the ratio between biggest velocity and smallest velocity is 2：1. The sedimentation track of irregular particles is not vertical.

In order to get over above-mentioned problems, some instruments introduce shape factor and sphericity factor etc to correct the sedimentation rate; other instruments adopt multi-point sampling to ensure the repetition of the instruments. But these measures cannot change the complexity of the particle size measuring of nonspherical particles radically; some problems are been still studied.

(5) The effect of centrifugal sedimentation to particle motion state

Diagram 3 is the figured diagram on particle centrifugal sedimentation state

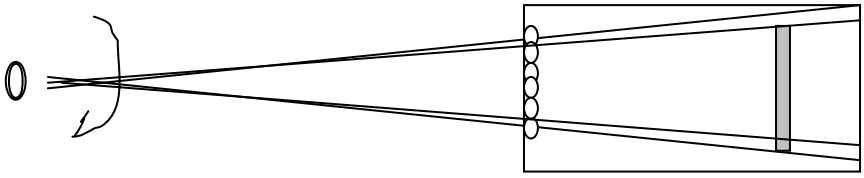

Diagram 3，particle centrifugal sedimentation state

We know from formula (10), the sedimentation rate of the particle under the centrifugal field is direct proportion to the distance x from the particle to axes O. The particle motion direction is divergence motion along centrifugal radium direction in centrifugal sedimentation. See diagram 3, there are four particles among six particles may pass through the measurement area in centrifugal sedimentation, two on the side move along the radium direction until fall to the bottom of vessel along the wall, beyond the measurement area.

The sedimentation rate and direction changement of centrifugal sedimentation particle leads to the nonnormal consistency drop of measurement area, which brings the reduce of fine particle in the measurement outcome.

The settled means are that one is to use long arm centrifugal device, when OS>>SR, the effect of the distance x can be neglected. Other is proper correct method when long arm centrifugal device is not suitable.

(6) Extinction coefficient

We know that the sedimentation particle size analyzer measures the transmitting light efficiency of suspending liquid to reflect the sedimentation rate of the particle, and the particle size distribution is obtained further. When particle diameter is far more than the light wavelength, the light attenuation after passing suspending liquid results from mainly the shadow of particle projection; when particle diameter approaches the light wavelength, the light will occur scattering, interference, and diffraction etc phenomena, and also occur refraction, and reflection etc

phenomena if transparent particles. The light signal received is anomalous. To the brought error, we introduce extinction coefficient to compensate. Table 4 is the extinction coefficient of different particle diameters.

Particle diameter	Extinction coefficient		Particle diameter	Extinction coefficient
22.5	1		3	0.62
20	0.97		2	0.76
15	0.84		1	1.4
10	0.64		0.8	1.8
7	0.56		0.6	2.8
5	0.56		0.4	5.6
4	0.58		0.2	9.8

Table 4 the extinction coefficient of different particle diameters.

4) Translucidus principle——Beer law

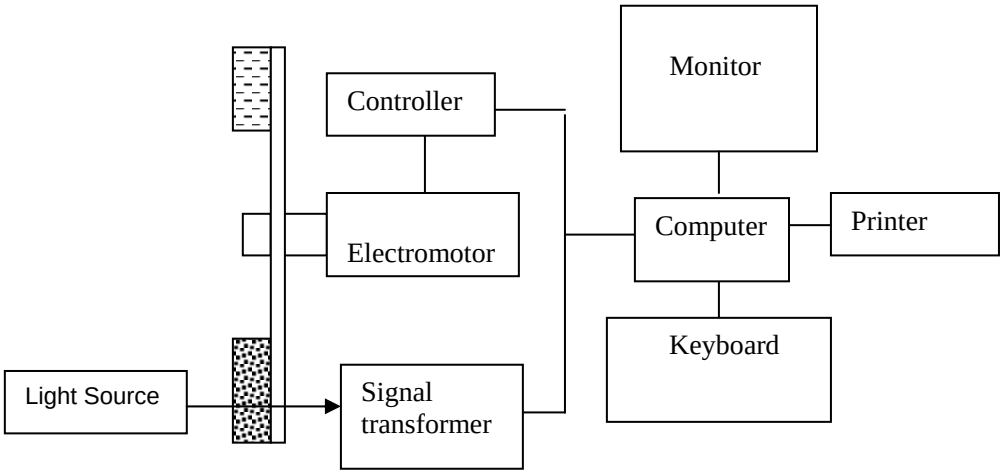

Table 4，the structure schematic diagram of sedimentation particle size analyzer

Table 4 is the structure schematic diagram of sedimentation particle size analyzer. The work process is so. The prepared suspending liquid is transferred to the sample tank, and the sample tank is placed on the analyzer. We use parallel light beam to irradiate the suspending liquid. The permeated light signal is received, converted and input the computer, at the same time, the change curve is displayed. With the sedimentation going on, the consistency of suspending liquid drops generally, and the permeated light increases generally. When all prospected particles fall under the measurement area, the measurement is over. After computer finishes processing the light signal, we will the particle size distribution.

Then, what relation is between the light signal and particle diameter? According to Bill law, the relation of light intensity of suspending liquid Ii, incidence light intensity Io and particle diameter D

is:

$$\log(I_i) = \log(I_0) - K\int_0^{\infty} n(D)D^2 dD \quad (14)$$

In the equation, K：a constant concerned with instrument constant, shape, and extinction system; n(D)：the particle quantity of D~D+dD diameter in light road; Io：incidence light intensity; Ii：light intensity through suspending liquid；

Bill law gives the relation between light intensity and particle quantity. In the calculating course, the system calculates the every time of different particles reaching the measurement area according to Stokes law, and writes down every light intensity through suspending liquid at corresponding moment. The particle size distribution of the sample can be worked out by formula (12), and the algorithm is as follows:

For example, there is a sample consisted of particle diameter D1、D2、D3、D4, and D1>D2>D3>D4. Their quantities are n1、n2、n3、n4，and the corresponding light intensity are I1、I2、I3、I4. We calculate the above equation:

$$\log I_1 = \log I_0 - K(n_1 D^2{}_1 + n_2 D^2{}_2 + n_3 D^2{}_3 + n_4 D^2{}_4)$$

$$\log I_2 = \log I_0 - K(n_2 D^2{}_2 + n_3 D^2{}_3 + n_4 D^2{}_4)$$

$$\log I_3 = \log I_0 - K(n_3 D^2{}_3 + n_4 D^2{}_4)$$

$$\log I_4 = \log I_0 - K(n_4 D^2{}_4)$$

Two equations subtracts each other among above four equations, then multiply Di, we get:

$$D_1(\log I_2 - \log I_1) = k n_1 D^3{}_1$$

$$D_2(\log I_3 - \log I_2) = kn_2D^3{}_2$$

$$D_3(\log I_4 - \log I_3) = kn_3D^3{}_3$$

$$D_4(\log I_0 - \log I_4) = kn_4D^3{}_4$$

From the above equation, we know that particle quantity multiply by particle weight on the right of the equation. It is also the total weight of particles with such diameter. So we could work out their respectively percentages according to the left of the equation.

3. Sample preparation

Sample preparation is sample and test situation preparation process before measuring particle size. Sample preparation includes sampling, the preparation of sedimentation and suspending liquid, dispersing, dispersant, and the check of dispersing effect.

1) Sampling

Because we use a little sample to represent the great deal of particles characteristic, the sample must be appropriate. But in fact, we always neglect the importance of sampling. This kind of situation must be changed.

Taking a sample from large quantities of materiel can be divided into following four steps:

large batch of materiel（or produce process）→ course sample(kg) → experiment sample(g）→

measure sample（suspension liquid） ←analyzing sample (mg)←

(1). The common rule of sampling and eduction phenomenon:

The coarse and fine particles will occur eduction phenomenon during the production, convey, packing, store and transmission. For example, the fine material will be concentrated at the central section, and coarse materiel will be concentrate at the surrounding. And on the conveyer, the mostly coarse material is on the both sides and surface, and the mostly fine material is at the central section and bottom. And in the bags, the mostly coarse material is at the surrounding than central section. If we know the eduction trend, we will conquer the insouciant attitude in the work.

The total principle of sampling is:

The first, we should take samples from moving material as possible as we can, in production process.

The second, we take the sample from many sections (different section, different deep), and the sampling section will be not less than four. We will get the experiment sample after we mix these samples.

The third, the sampling method must be fixed. We must make a strict standard according to the practical circumstances, and avoid sampling optionally.

Sampling tools have many kinds. Keyway sampler is for dry powder, and wide mouth vessel (suck as breaker and graduate) etc for pulp material.

(2). The sample division method

We should divide the experiment sample into proper quantity. The method is as follow: i) Use ladle. The sample must be fully mixed (put the sample into container and vibrate tempestuously or put the sample on the glass board and mix fully). And then we take the sample from many sections. ii) Cone four division. We put the whole sample on the glass board and fully mix, and stack the sample into a cone. Now we can use a thin plate cut the cone into crisscross from the top, and we put the diagonal parts in one and mix fully and repeat the course mentioned above until we take proper quantity sample. It is very important that the cone must be regular and the cross line of two cut surfaces must be superposition with the axis of the cone. iii) Instrument division. There are the fork flow type divide apparatus，the dish divide apparatus and so on.

(3). Divide the laboratory sample into analysis sample

The above experiment sample will be divided into analysis sample. Because the sample is more and more less, the representative divided sample is very important. We take out commonly 0.5~2g sample to make up suspending liquid. The division method is multi-point (at least four points) ladle sampling after mixing completely. We must note the sample in the ladle is used fully, and cannot loss.

(4). The preparation and transfer of suspend liquid

Putting the analysis sample into beaker, and mixing the sample with medium, we can get about 60 ml volumes suspend liquid. After the sample is equally dispersed and mixed, we transfer one part of them into sample tank for measuring. We should fully mix the suspend liquid before transfer, and then use the multi-directional sampler to draw out the liquid into sample tank.

Sampling is the most important step during particle size measurement. The basic requirements are: First, the method must be appropriate; Secondly，we must attach importance to it; And the third, the method must be standard.

2) Sedimentation medium

The medium is the liquid used to disperse the sample. The sedimentation particle size analyzer samples from suspending liquid, so it is very important to select appropriate sedimentation medium.

How to select the sedimentation medium? At first, the selected medium should have the good affinity with the measured sample. In chemistry, the easily wetted matters by water and other media are called hydrophilic matters, such as calcite, SiO_2, and kaolin etc; the difficultly wetted matters by water and other media are called hydrophobic matters, such as talcum and graphite etc. The simplest method to judge the hydrophilic matters and hydrophobic matters is to see the

float phenomenon after the material is put in the medium and mixed, the matter without float phenomenon is hydrophilic matter; or hydrophobic matter. The second, the measured material will not dissolve in the medium, and will not occur expand, hydration and other physics and chemistry reaction. The third, the medium must be pure, no impurity. The fourth, the particle should have appropriate sedimentation rate.

Common sedimentation mediums are water, water + glycerin, absolute alcohol, absolute alcohol + glycerin. Here glycerin is tackifier increasing the medium viscosity, and ensuring the course particle sedimentates in laminar flow area. Generally, the largest particle less than 38um, or the sample of density less than 3 may choose distilled water or alcohol as medium directly.

The method to prepare glycerin and water solution or glycerin and alcohol solution is so. Add water (or alcohol) first, and add glycerin, then stir completely. At last, put it into ultrasonic scatter apparatus and shake about 10 minutes. Now we can use the sample.

3) Dispersing and dispersant

The sample and sedimentation medium are mixed into suspending liquid with certain consistency, and the particles are distributed in the liquid in individual state, this is called dispersing.

The dispersing is divided into three stages. First, humidification process, that is, the liquid humidifies the particle surface; second, particles separate from “Group Particle”; third, keeping dispersing state. The additive added in above three stages is called dispersant. That is to say the action of dispersant is to humidify the particle surface well, enhance the affinity between the particle surface and liquid, quicken the separate of “Group Particle”, and keep dispersing state.

Common dispersants are $(NaPO_3)_6$, $Na_4P_2O_7$ etc. The dispersant must be dissolved in the medium before using, and the consistency is usually around 0.2%. Too much or too little dispersant will have negative effect on dispersing.

If the sedimentation medium is alcohol or benzene etc organic solvent, the medium does not need dispersant.

Separating “Group Particle” is the key among above-mentioned three stages. The bond strength between separate particles of some samples, especially fine particles, is bigger, only the humidification action of dispersing medium is not enough to separate them completely and quickly, so we must impress outside force. The best is ultrasonic dispersing. It also includes stirring, grinding and boiling etc, and these methods are usually used together.

Table 5 gives the ultrasonic dispersing time of different dry powders. (unit: min)

Particle size D50 （micron）	**Talcum, kaolin, graphite**	**Calcite**	**Aluminium powder**	**Other**
>20			1~2	1~2
20~10	3~5	2~3	2~3	2~3

10~5	5~8	3~5	2~3	2~3
5~2	8~12	5~7	3~5	3~8
2~1<	12~15	7~10	5~7	8~12
>1	15~20	10~12	7~10	12~15

Table 5 the ultrasonic dispersing time of different samples

The ultrasonic dispersing time of wet method pulp is 1/2 of time in the table. It is in relation to the powder of ultrasonic disperser, the bigger the power is, the shorter the time is.

4) Make up suspending liquid and prepare before measuring

The generic samples, are directly put in the medium with dispersant, dispersed and used for measuring. The samples with wide particle size distribution, are mixed into ropiness in a little medium, taken out some in ladle, and put in the medium to prepare suspending liquid, which benefits to ensure the representative of the sample. The hydrophobic or metamorphic samples, are predispersed, and made up suspending liquid to disperse.

After ultrasonic dispersing, the humidity of the suspending liquid will increase, which benefits dispersing. But if the temperature difference between suspending liquid and circumstance is too big, it will bring disadvantageous effect on measuring. The methods getting over temperature increasing are four. First, reduce the temperature of suspending liquid to room temperature after ultrasonic dispersing. Second, measure the temperature of suspending liquid directly, and take the parameter at this temperature as initial parameter to measure. Third, change the water in ultrasonic disperser constantly, and reduce the temperature increasing during dispersing. Fourth, prepare big consistency suspending liquid to disperse completely, and take suitable such liquid to make proper consistency suspending liquid.

5) Check dispersing result

The common methods to check dispersing result are two. One is by microscope. Place a little dispersed sample on microscope to observe agglomerating phenomenon. Other is by particle size analyzer. Compare whether measured parameters of two times in different time are consistent.

The above is the brief introduction on sample preparation. The sampling method and dispersing method are more important. Because different production processes and methods, circumstance, and complicated dispersing and agglomerating principles, we must make experiment and study the measured material, sedimentation medium, dispersant and dispersing mode etc during practical particle size measurement, to reduce the error from sample preparation furthest, and make sure the particle size measurement meet quality control requirement.

4. The sedimentation particle size analyzer and its application

There are many kinds of the sedimentation particle size analyzers, such as gravity sedimentation

mode, gravity sedimentation and centrifugal sedimentation combined mode, and centrifugal sedimentation mode. And centrifugal sedimentation is divided into even suspending liquid mode and spreading layer mode etc. We will discuss the prevalent problems of the sedimentation particle size analyzer.

1) The repeatability

The repeatability is the key to examine the quality of one particle size analyzer. Here, the repeatability is deviation of measure results of the same sample measured by the same instrument many times. We should do our best to avoid the influence because of the sample shrinking, dispersing and etc.

The detailed method is:

⑴The suspension liquid used to measure is not less than 120ml, and take the test sample three times from it;

⑵The consistency of suspension liquid should to be suitable;

⑶The sedimentation medium and dispersing agent should be suitably;

⑷Disperse suspension liquid adequately

⑸Stir evenly before taking a sample to test;

The methods that guarantee and raise the instrument repeatability are:

(1) First, the suspension liquid should be in the good and steady dispersing state;
(2) Second, grasp the normative operating rules and method;
(3) Third, the stability of instrument and power supplying should be normal.

These are the basic requirements during the actual particle measurement.

The examination on repeatability deviation of sedimentation particle size analyzer adopts generally the standard sample. The standard sample used to do repeatability examination can be international particle size standard matter; also can be other sample with the corresponding condition. Under standard operating circumstances, the variation of sample measured D50 should be within 4% every time.

The calculation method of repeatability variation is:

$$\sigma = \sqrt{\frac{\sum (D_i - \overline{D})^2}{n-1}} \text{-----------------(15)}$$

$$\delta = \frac{\sigma}{\overline{X}} \times 100\% \text{-----------------(16)}$$

In the formula, Di is the value of middle position diameter each time; n is the measurement times (generally no less than 10 times); D is the average value of middle position diameter; σis

the standard variation of Di; δis repeatability fractional error.

2) The accuracy

The accuracy of any measure instrument is always the error between measured value and true value. We will find from the following analysis: the accuracy concept of the sedimentation particle size analyzer is different from other measure instruments. Because the great mass of powder particles n practical production non spherical (such as slice shape, needle shape, lozenge shape etc), such particles can not express their dimension by a particle diameter value in theory, hence, we can not get so-called true value of particle diameter in theory. The particle diameter measured by particle size analyzer is an equivalent particle diameter, not (impossible) true particle diameter. Now some instruments or standards give some accuracy indicatrixs, which indicates the difference between measured value and corresponding value (generally D50) of one or several standard substances. The calculating method is:

$$\Delta = \frac{\overline{D} - L}{L} \times 100\% \quad ----------(17)$$

In the formula, △ is accuracy fractional error.

D is the average value of middle position diameter.

L is the nominal value of middle position diameter of standard sample.

The accuracy indicatrixs is discussed on basis of special sample, conditional, relative. This shows, as to the sedimentation particle size analyze, accuracy is discussed only for standard sample. Some pursuers put forward with facticity concept. That means the difference between measured values of different instruments or methods should be within reasonable range. What is the reasonable range? Now there is no last word. The users can decide it according to relative trade standard or mature techniques demand.

3) The determination of sedimentation parameter

If we use the sedimentation particle size analyzer to measure the particle size distribution, we must predefine particle density, medium viscosity, medium density, and sedimentation height and particle diameter etc parameters.

Table 6 gives the real density of common powders

Powder material	**Density**		**Powder material**	**Density**		**Powder material**	**Density**
talcum powder	2.7		carborundum	3.2		aluminum 2.7	2.7
kaolin	2.7		silicon dioxide	2.65		molybdenum	10.2

calcium carbonate	2.7		zeolite	2.3		zinc	7.14
silicon dioxide	2.65		clay 2.6	2.6		copper	8.96
graphite 2.2	2.2		diamond	3.29		tungsten	19.3
silicon	2.85		quartz	2.7		nickel	8.9
zirconium	4.63		iron	7.87		silver	10.49

Table 6 the real density of common powders

The medium viscosity and density are variational parameter with temperature. We can get them by referring to table or by measuring with viscosimeter and gravimeter directly.

The sedimentation height is the distance between liquid level and measurement position. The sedimentation distance of general sedimentation analyzers is 20mm~100mm. Some analyzers have several kinds of sample grooves with different heights for the need of course and fine samples. Generally, the course samples needs long sedimentation height, and the fine samples need short sedimentation height.

Besides the above parameters, we need to set consistency value and reference value of suspending liquid on the sedimentation particle size analyzer. Consistency value called is the light intensity through suspending liquid, and reference value is the light intensity through pure medium. The ratio is 1: 6.

4) Measurement mode and process

The measurement mode of the sedimentation particle size analyzer includes gravity sedimentation mode, centrifugal sedimentation mode, and gravity sedimentation and centrifugal sedimentation combined mode.

The gravity sedimentation mode is that the measurement course from beginning and ending is finished under gravity, and the centrifuger does not run. The measurement lower limit is 3μm, if less than 3μm, the influence of Brownian motion becomes obvious, and the error of measured outcome becomes bigger.

The centrifugal sedimentation mode is used to measure superfine samples, when water is medium, the measurement range is 8μm~0.1μm, the measurement lower limit of disk centrifugal particle size analyzer is even up to 0.04μm.

The basic process of combined sedimentation mode is that the gravity sedimentation mode is after the beginning of the measurement to measure course particles; when the gravity sedimentation mode reaches certain condition, the centrifuger is started up, and the fine particles are measured by the centrifugal sedimentation mode. It enlarges not only the range of the sedimentation particle size analyzer, reduces the influence of bad factors, but also shortens the measurement time.

In general, for bigger specific gravity metallic powder or little fine samples below 10μm, we select

the gravity sedimentation mode; for around 90% nonmetallic powder below 2μm, we choose pure centrifugal sedimentation mode; for other powder from 200 meshes to 2500 mesh, we use combined mode.

5) The measuration of largest particle diameter

The traditional sedimentation particle size analyzer is needed to preset a largest particle diameter value according to sample dimension before measuring, but the value is blind. The newer sedimentation particle size analyzer can measure particle diameter automatically according to critical diameter and sedimentation curve change of gravity sedimentation. The largest diameter is the diameter of the particles leading to the earliest consistency change of suspending liquid and obvious increasing of sedimentation curve in the measured suspending liquid.

Diagram 5 the position schematic diagram of largest particle diameter from several measuring samples of BT-1500 sedimentation particle size analyzer

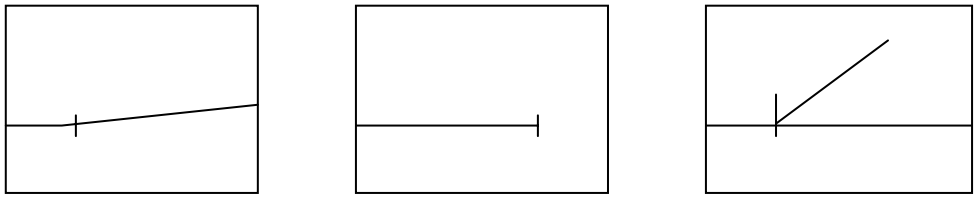

Diagram 5，the position schematic diagram of largest particle diameter from BT-1500

6) The setting of particle diameter section

The particle size distribution shows what is the particle percentage of different particle diameter among all particles. The sedimentation particle size analyzer need to set particle diameter section factitiously. The setting principles are:

⑴Meet the analysis need. The very concerned particle diameter section in production and application cannot be omitted.

⑵Particle diameter section setting of same samples should be consistent.

⑶Under the precondition of meeting need, particle diameter section of course particle end should be wide, and particle diameter section of fine particle end should be narrow.

⑷The largest value of particle grade should not be less than the measured largest particle diameter.

The optional methods on setting particle diameter section are:

⑴Fixed interval：The system sets over ten kinds of particle diameter sections, and user can select any kind.

⑵Random interval：Every particle value from big to small is set, any corresponding percentage of particle diameter can be obtained.

⑶Equidifferent interval：The dimension of all particle diameter sections is equal to a fixed difference.

⑷Geometric proportion interval：The ratio between two consecutive particle grades is a fixed value.

Some new sedimentation particle size analyzers can reset particle grade to amend the particle grade.

7) Processing the measured result

After finishing measure, the processing includes printing, saving, searching, comparing, merging and deleting etc.

8) The prospect about sedimentation particle size measure technology

Sedimentation particle size measure technology is more mature, and has wide application in production and research field. In future, its development will focus on the following aspects.

⑴Raise automation and intelligence level, easier to use.

⑵Enhance software dealing with data, provide more data, and combine with Internet to quicken information transferring and converting.

⑶Become to measure many samples at one time, increasing the efficiency.

⑷Develop centrifugal revolution into high precision, high revolution, stepless shift, and intelligent control.