10

Measurement Techniques

A range of experimental or measurement techniques is available for determining the functioning of gas cyclones and swirl tubes. The choice of technique depends on the situation: the techniques giving the best results in the laboratory on relatively small-scale equipment under controlled conditions, may be quite different from those giving the best results in industrial equipment.

In the laboratory, the goal of most measurement campaigns is to further ones understanding of the basic phenomena that govern the performance of cyclone and related centrifugal separation apparatus. Such things as:

- detailed velocity and pressure profiles,
- specific erosion patterns and rates,
- dust concentration profiles,
- particle paths/trajectories,
- particle attrition rates,
- the effect of design modifications,
- the effect of upflow or hopper crossflow for flow maldistribution,
- dust discharge configuration or hardware,
- agglomeration effects,
- the effect of changes in operating conditions (flow rate, operating pressure, solids loading, and particle size, shape and density, etc.)

are often the target of the investigation, aside from more traditional measurements of grade and overall efficiency and overall pressure loss. Laboratory cyclone equipment is designed 'from the ground up' to facilitate accurate measurement of such effects.

In an industrial cyclone system, the goal of measurements has related but somewhat different objectives. Here, one is more interested in ascertaining the overall performance of the cyclone under plant operating conditions. This may be done to so that plant personnel can:

- check out the general state of the equipment,
- compare performance with design targets,

- verify vendor guarantees or warranties,
- check on compliance with air quality standards,
- check on the effectiveness of certain process or design features or changes,
- help determine the remaining service life of the equipment, that is, when the equipment needs to 'come down' for repair.

Depending on the specifics of the plant, other measurement objectives could be added to the above list. However, in virtually all cases, one can be certain that the commercial unit was not built to facilitate accurate measurement or testing.

Plant measurements pose some very special challenges. They must often be performed outdoors under very hot, cold, or inclement weather conditions, on windblown platforms or on stacks that may be many stories tall, with inlet and outlet ducting geometries that are far removed from ideal, and with systems that have few, if any, working sample ports. In some cases, an accurate knowledge of even some basic operating conditions, such as volumetric flow rate, operating pressure and temperature, or gas composition, may not be known or it must be estimated in a rather crude fashion. Alternatively, if such information is available, it may not be known at the point in the system where the cyclones are installed. In some cases, one may not even have up-to-date drawings of the cyclones and any inspection records that may assist in the interpretation of the measurements may be lost or woefully lacking in information.

Commercial cyclone installations are usually subject to a performance evaluation after they are brought on-line and process conditions have stabilized. Such "Start-of-run" measurements may be performed to verify vendor performance predictions or, a closely related objective, to determine if the cyclone(s) are performing their intended process duty. Aside from these important reasons, early in-the-run performance measurements also provide a benchmark with which to compare preformance later in the run. cyclone performance will usually deteriorate with run time in commercial systems since they are normally required to run for months or even years between turnarounds. Such deterioration may be due to a number of factors: erosion (increased wall roughness or holes in walls), corrosion, weld cracking, deposit formation, distortion, seal problems, etc. (see related discussion in Chap. 12). Periodic performance measurements allow plant personnel to detect and monitor the rate of performance degradation and thereby estimate its useful remaining run life.

Cyclones in commercial service are often installed in parallel arrays or in series with other cyclones. Under such conditions, it is rarely possible to determine the performance of the individual cyclones. Thus, even after the data is collected, it is often a challenge to interpret it correctly.

In some parallel cyclone arrays, wherein it is suspected or known that the cyclone system is not performing up to design expectations, it may be possible to inject a tracer, such as helium gas, in a common header upstream of the cyclones and measure the time required for the tracer to exit out the

overflow line from each cyclone[1]. Such measurements can provide clues as to the cause of the sub-par performance. The technique may tell us, for example, if gas is short-circuiting one or more cyclones, or whether one or more cyclones are running with plugged, or partially plugged, underflow pipes, or if one or more cyclones are filled or partially filled with solids. It may also tell us how uniformly the gas is being distributed among the individual cyclone units. This latter problem may be the result of an inlet gas or solids distribution problem. The same applies to a gas/liquid cyclone installation.

Even after one obtains performance data, correctly interpreting it can be quite challenging when the results are not what we are expecting. In some situations, we might even *expect* a certain outcome (such as poor separation performance) but determining the root cause from the performance data is anything but straight forward. When two or more cyclone sets are arranged in series, for example, it may be very difficult to determine which stage is malfunctioning. It is in situations like this where an in-depth understanding of cyclone behavior and experience comes most into play. Because cyclone systems vary so widely from one industry to the next, and from one service to another, it is not possible to prepare a detailed troubleshooting guide that would cover all of the various cyclone failure modes that are possible for every industry or application. On the basis of the authors' experience in analyzing and troubleshooting various cyclone installations, however, we do want to point out that *the majority of cyclone problems relate, in some way or the other, to the inability of the solids to properly discharge out the cyclone(s) underflow chute or pipe*. This certainly includes such things as gas leakage up the solids discharge opening, plugging of the underflow piping or hopper, or an unstable solids discharge. We'll have more to say about cyclone underflow sealing in the next chapter but, an example of a plugged or unstable discharge would be poor aeration of one or more cyclone underflow pipes (i.e., "diplegs") in applications wherein they are required to be "sealed" in a fluidized bed. Thus, as we can glean from the above discussion, measurement and analysis go hand-in-hand.

It is often only possible to obtain relative measurements of performance. One may be 'stuck' with a very poorly located sample point or tap, for example, yet this particular sample tap may reveal *changes* in performance that are still very meaningful to plant personnel.

Measurements are very costly to undertake, whether in the laboratory or in the plant. Plant measurement errors are almost always greater than those one can obtain under a controlled, laboratory environment, although meaningful laboratory measurements present their own, unique set of challenges.

With this in mind, we wish to present some of the most commonly employed cyclone measurement techniques. While all of the following techniques

[1] In some installations it may be possible to also measure the time it takes for the tracer to exit out the underflow line from each cyclone, but this is not very common

can be applied in a laboratory research environment, a number of them can be usefully applied in a commercial plant facility also. In particular, this would include the procedures for pressure drop, on-line and off-line sampling, and methods for particle size analysis.

10.1 Gas Flow Pattern

Since the flow in a cyclone or swirl tube is complex, the direction of the axial and radial velocity components are not known in advance. This has to be borne in mind when considering how to measure the flow field.

A 'hot-wire anemometer' measures the gas velocity at a given point from the cooling of an electrically heated wire stretched out in a fork. The faster the gas flows over the wire, the stronger the cooling. A traditional hot wire anemometer will measure the absolute gas velocity (the speed), but not the direction. 3-D anemometers have been developed, which will also measure direction by comparing the cooling of wires stretched out in different directions. Although much has been done to develop the theory, hot wire anemometers still require calibration.

The traditional 'pitot tube' (see Figure 10.1.1a) has the advantage that, if properly configured, it needs no calibration. When pointed against the direction of the gas velocity, it will measure the dynamic pressure directly as $(p_1 - p_2)$ (see the figure). From this the velocity can be calculated using the formula[2]:

$$v = \sqrt{\frac{2\,(p_1 - p_2)}{\rho}}. \tag{10.1.1}$$

The error in using this formula without a calibration will be normally only a couple of percent. If the pitot tube is of the elliptical type, the error will drop to a fraction of a percentage point.

It is possible to use a conventional pitot tube to ascertain the direction of a velocity within a cyclone. It is far easier, however, to use a '5-bore pitot tube' (Figure 10.1.1b) rather than try to glean the direction by tilting a traditional pitot tube. After calibration, a 5-bore pitot tube will give information about the magnitude and direction of the gas velocity from the pressure differences between orifices located at various points on the spherical measuring head, as indicated in the figure.

A major problem with both the hot wire anemometer and the pitot tube is that the measurements are intrusive. A probe is inserted, and this may well disturb the flow pattern. Obviously, the smaller the probe is relative to the vessel, the less the disturbance.

In 'laser-Doppler anemometry' (LDA), the velocity of the gas is measured as the speed of small seed particles that follow the gas flow faithfully. In the

[2] This formula is valid up to about 60 m/s, at higher velocities the compressibility of the gas has to be taken into account

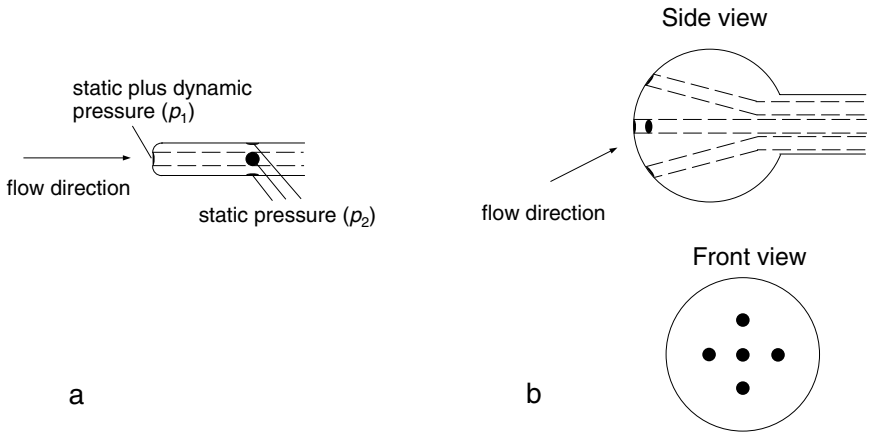

Fig. 10.1.1. Diagrams showing the principles of **a** a traditional Prandtl-type pitot tube, and **b** a 5-bore pitot tube

'fringe anemometer' two laser light beams are caused to cross, which creates an interference pattern (the measuring volume) through which the seed particles fly (a visualization of this is shown in Figure 10.1.2[3]). The light scattered by the particles as they pass through the measuring volume is detected as a 'Doppler burst', and the frequency of this is a measure of the speed with which the particles traverse the measuring volume. In classical LDA each of the components of the gas velocity are measured consecutively. In newer systems, different velocity components can be measured in the same volume simultaneously using different colored laser light, which can be detected separately.

The main advantage of LDA is that it is nonintrusive. Problems can be caused by the seed particles not faithfully following the gas in a strongly swirling region of gas flow, and by the optical breaking of the laser beams in the cylindrical wall of the cyclone or swirl tube.

The three methods discussed above are all suitable for determining the mean gas velocity. If we wish to determine also the fluctuating velocity component due to turbulence, a meter with a short response time is needed. Both hot-wire anemometry and LDA can be used for this. The response time of hot-wire anemometers is being reduced by equipment miniaturization, but LDA is the most widely used technique for turbulence characterization.

[3] Although the picture is a good way of visualizing the interference pattern, it does not quite reflect the situation correctly. If, for instance, the intensity peaks in the incoming radiation are the black lines, then the intensity peaks in the interference pattern will be where the black lines cross, which should then be the location of the darker bands in the interference pattern, and not, as they are in the picture, the location of the lighter bands.

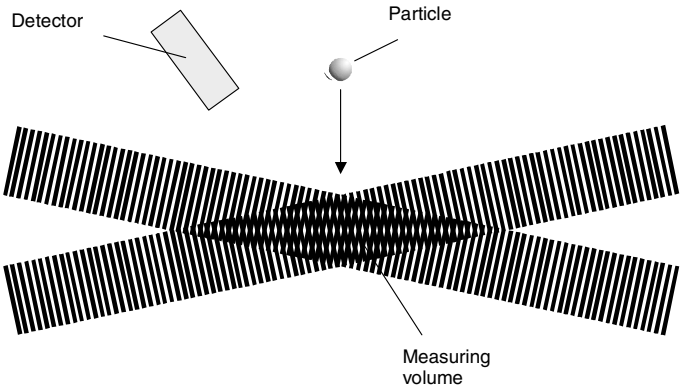

Fig. 10.1.2. Visualization of a seed particle flying through an interference pattern created by two crossing laser beams

10.2 Pressure Drop

When deciding how best to measure the pressure drop over centrifugal separation equipment, we first have to decide what we mean by the term 'pressure drop'.

As discussed in Chap. 4, the swirl in cyclones and swirl tubes means that the static and dynamic contributions to the total pressure vary strongly throughout the equipment. It is therefore not sufficient to measure the static pressure at a given position, subtract the static pressure at the inlet, and call this the 'pressure drop'. We take 'pressure drop' to mean *the drop in total pressure*, dynamic plus static. The drop in total pressure is equal to the dissipative loss of mechanical energy per unit volume in the flowing gas[4].

Measuring the pressure at the inlet of a cyclone is not a problem: there is no swirl there, and the static pressure is uniform over the cross section, so we can measure the pressure with a standard pressure tapping at the wall.

At the gas outlet, on the other hand, residual swirl is the problem: a significant dynamic pressure is stored in the swirling motion, and the static pressure is not uniform over the cross section. Some researchers studying the pressure drop in cyclones have solved this by letting the cyclones discharge directly to the atmosphere, taking the outlet pressure as atmospheric. This is a good solution if solids are not used in the system. Others have placed rectifying or 'straightening' vanes in the outlet pipe from the cyclone or swirl tube, and measured the static pressure after the rectifier (the axial velocity in inlet and outlet is about the same for normal cyclones and swirl tube designs).

[4] Note that in most other process equipment not involving swirling flow, we are not confronted with this problem in defining pressure drop, since the drop in *static* pressure is proportional to the dissipative mechanical energy loss, as long as the inlet and outlet fluid velocities are approximately the same.

Others, fearing that rectifiers would influence the flow in the separator body, measured the static pressure by a conventional pressure tapping at the wall in the outlet of a cyclone. Because the static pressure happens to vary in an approximately linear manner with radius in the cyclone outlet, this wall static pressure is approximately equal to what the cross-sectional average static pressure would have been after an ideal rectifier. This is discussed in Chap. 4. If the two latter methods are used one should keep in mind that, without rectification of the outgoing stream, more dissipative loss will occur in the downstream tubing as the swirl energy is dissipated. If one does use wall pressure taps to measure static pressure, the taps should be gas purged so that they do not plug while in service. A purge velocity of 0.2 to 0.5 m/s will usually suffice in keeping particles from entering the pressure taps. To avoid flow disturbances from the pressure taps, they should be small, of the order of 1 mm in diameter for laboratory-scale units, but may be as large as 4 to 5 mm on large commercial-scale installations. The inside edge of the taps should be smooth and flush with the inner surface of the pipe or duct into which they are inserted.

The differences in measured pressure drops and in model predictions of pressure drop in the research literature, particularly the difference between the Barth model predictions and the experimental results in Fig. 5.3.2, can probably be traced back to different cultures in measuring the outlet pressure.

It is difficult to give general recommendations for the method of measuring the outlet pressure when solids are being used in the system. The most important thing is to interpret the measurements correctly.

10.3 Particle Flow

Tracing an individual particle as it moves through the separator body is not yet possible. Several research groups are developing methods for tracing of radioactive particles in 3-D based on the detection and cross-triangulation of γ-rays emitted back-to-back. However, the tracer particles still have to be relatively large and the method is at present only suitable for particle velocities up to a few meters per second. Both of these limitations are being pushed back rapidly, though, and experimental particle tracking in cyclones and swirl tubes should become possible in the near future.

Phase Doppler anemometry (PDA) is a technique akin to LDA. In PDA the laser light scattered in the measuring volume by a particle is detected at two angles, making it possible to gain information not only about the velocity of a particle but also its size. Mothes and Löffler (1985) report a study wherein they used this technique to gain information about the distribution of particle sizes within the body of a gas cyclone.

10.4 Overall Separation Efficiency

When determining the efficiency of cyclones and swirl tubes, samples can be taken at three positions: the inlet, the gas outlet and the dust outlet (see Figure 10.4.1).

Inspecting Eq. (3.2.2) shows that the overall separation efficiency, η, can be calculated from the mass flows of solids at any two of the three sampling points. If also the solids mass flow at the third is determined, one can check on the 'material balance' by way of Eq. (3.2.1).

If there is no net flow of gas out the cyclone underflow, the captured solids fraction is a pure solids stream, and it may be possible to determine the mass flow of this fraction by collecting the underflow solids for a known time, and weighing. This method can often be applied in both an industrial and a laboratory installation.

In other cases, the underflow rate is not available but a sample of the underflow can be taken. Providing the sample is representative of the underflow, the underflow particle size distribution (PSD) can be ascertained. This PSD may also be independently computed from measured PSDs of the feed and the overflow streams and compared with the measured underflow PSD. The two underflow PSDs thus determined should agree. If not, there is something wrong with one or more of the measurements. This technique has been used in practice as a means of checking the accuracy of the feed and overflow measurements. See also the discussion around Eqs. (3.2.3) and (3.2.4).

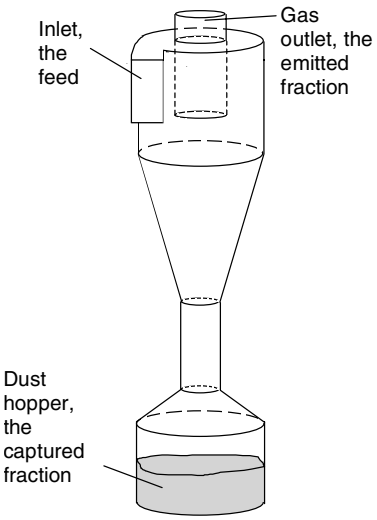

Fig. 10.4.1. Sketch indicating the sampling points for determining cyclone separation efficiency

The mass flow of the feed solids is sometimes known in an industrial installation from measurements taken elsewhere in the plant. In an experimental laboratory rig, a test is often of a limited duration, and the mass of feed solids can be determined accurately by weighing the entire charge. If these options are not workable, the inlet solids flow has to be determined by on-line sampling from the piping upstream of the cyclone or swirl tube, or inferred from measurements or information regarding the solids flow rate in the overflow and underflow streams.

The mass flow of the overflow fraction normally has to be determined in an industrial installation by on-line sampling downstream of the cyclone, while in a laboratory test rig total capture of the overhead fraction by filtering may be feasible.

Once the mass flowrates of solids at two of the three points shown in Fig. 10.4.1 have been determined with sufficient accuracy, the efficiency of the cyclone or swirl tube can be determined from Eq. (3.2.2).

The error in a measurement is often roughly proportional to the absolute value of the measured variable. This is used in Appendix 10A to show that the error in the value of η calculated from the three mass flows is by far the lowest if the overflow fraction is one of the two mass flows measured.

10.4.1 On-line Sampling of Solids

Sampling for determining solids concentrations in gas streams and for size analysis needs to be done carefully. Below we highlight some of the intricacies of sampling on-line in the piping both upstream and downstream of a cyclone or swirl tube.

Sampling on-line involves drawing off a small stream of the solids-laden gas by means of the sampling probe. The small stream of dust-laden gas is led through a filter in which the solids are captured, and from which they can be removed for subsequent analysis.

The first rule of on-line sampling is that it has to be done 'isokinetically', that is, the gas velocity at the mouth of the sampling probe has to be the same as that of the flowing gas at that point. Figure 10.4.2 illustrates this principle.

We note two extreme cases where isokinetic sampling may be less critical for measuring particle size distribution:

- at high gas velocities, and when the smallest particles or droplets are rather large and the particles have a high density relative to the fluid, all particles approaching the nozzle will travel in a more or less straight line "ignoring" any curvature of the fluid streamlines caused by deviations from isokineticity. Note, however, that although isokinecitity is not crucial for measuring the particle size distribution in this extreme case, it is still a prerequisite for measuring the true particle concentration.

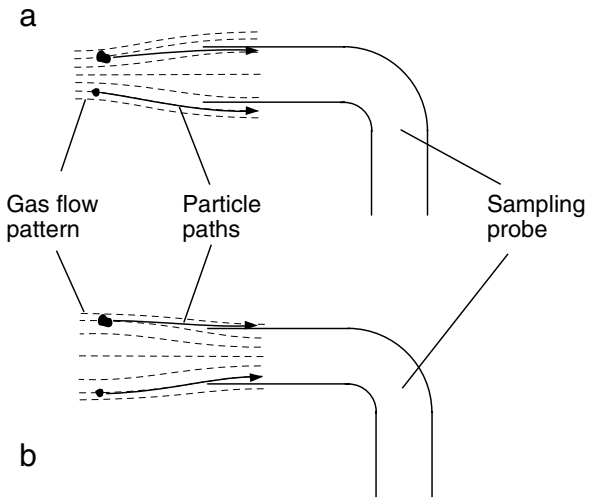

Fig. 10.4.2. Sketches showing the consequence of not sampling isokinetically. In **a** the sampling velocity is too low, and the gas flows around the probe in the pattern indicated. Small particles manage to flow around the probe, while larger ones at the same position relative to the probe mouth enter the probe by their inertia. The sampling will overestimate the solids concentration in the gas, and the size distribution of the captured material will be biased toward large particles. In **b**, the sampling velocity is too high, and the converse takes place, the solids concentration is underestimated, and fraction of fine particles is overrepresented in the captured material

- at low gas velocities, and when the largest particles are relatively small and have a low density, all the particles may follow the gas streamlines faithfully, the measured size distribution will therefore not depend on isokineticity.

The former of these two cases may well arise in cyclone research. If the maximum and/or minimum particle size and the particle density are not known *a priori*, however, it is impossible to tell whether one of these conditions apply, and in general it is advisable to sample isokinetically whereever possible. Hangal and Willeke (1990) discuss the issue of sampling from a gas stream, and present a model for the capture efficiency of particles as a function of the particle's Stokes number, the ratio of gas to sampler inlet velocities and the angle of the sampler relative to the gas flow.

The second important point is that the sampling should be representative of the entire cross-sectional area of the pipe. This means that in most cases sampling has to be done at a number of cross-sectional positions. Ensuring representative sampling can be difficult if sampling is done too close to an upstream flow disturbance, such as a bend. The gas velocity profile will then

be skewed, making isokinetic sampling more difficult. But, more importantly, the solids concentration is likely to be nonuniform over the cross section of the pipe. The former of these two problems can be overcome by determining the cross-sectional gas velocity profile and making sure that sampling is isokinetic, but there is really no acceptable way of overcoming the latter problem.

In order to minimize errors arising from such effects, a number of guidelines have been laid down in industry norms or specifications. It is useful to consult such a norm or specification when faced with the task of determining solids concentration and size distribution by on-line sampling. We mention some of the most important points below, enough to carry out a reasonably accurate sampling, and refer to, for example, the norm ISO 9096 for more information.

The mouth of the sampling probe should be at least 4 mm in diameter and be sharp edged and tapered to avoid turbulence around the inlet to the probe. If the process stream is at elevated temperature, and contains moisture or 'condensables', the part of the sampling system outside the process piping may need to be heated to avoid condensation. If there is moisture or condensables in the system, one has to be careful to account for this when working out the flows, for instance, if the gas is dried before it is metered in the sampling train.

The gas velocity can best be measured using a (Prandtl type) pitot tube. Either this can be done in a separate measurement, or the sampling system can incorporate a separate probe for velocity measurement.

The sampling position should be at least 5 diameters downstream and 2 diameters upstream of a flow disturbance, such as a bend or a valve. Some vendors or users of sampling or flow measurement equipment demand much larger undisturbed sections. If the undisturbed section cannot be made long enough, where feasible, a half-area mixing baffle (a transverse plate having half the cross-sectional area of the pipe) can help in distributing the gas and solids flow uniformly over the pipe cross section. It is better to sample in a vertical than in a horizontal pipe section. The ISO norm recommends sampling at a series of specified cross-sectional positions in the pipe to obtain a more representative sample. Each of these positions represents an equal fraction of the pipe cross section.

A sampling time of at least 3 minutes is recommended, although the sampling time obviously has to be adjusted in accordance with the solids concentration.

Before closing this section on sampling we wish to point out that, once a plant is built, it is normally too late to think about accurate sampling if this was not considered in the original design phase. In such instances, one may not be able to get accurate measurements of a particular feed or overflow stream. However, even a less-than-ideal sample point may still prove very useful for monitoring *trends* in the cyclone system's performance, such as a sudden increase in the coarse fraction of solids reporting overhead or a steady increase in pressure drop. A trend of increasing pressure drop over time could be indicative of material depositing or growing on the inside of the gas outlet

pipe. A steady decrease in cyclone pressure drop over time could be a sign of increased wall roughness brought about by wall deposits or wall erosion and/or corrosion. Recall that an increase in effective wall roughness will result in an attenuation of the core spin velocity and this, in turn, will reduce the cyclone's overall pressure loss.

10.5 Grade-Efficiency

There are two ways of determining the grade-efficiency $\eta(x)$ of cyclones:

1. In the case of a laboratory cyclone unit, inject monosized particles in the cyclone, and measure the overall efficiency to obtain one point on the grade-efficiency curve.
2. Inject a feed of a wide size distribution into a laboratory cyclone or, in the case of an commercial installation, utilizing the solids already reporting to and from the cyclone, collect samples and perform size analyses on any two of: the feed, the overflow and the captured (underflow) fractions. Then use Eq. (3.2.6) to calculate $\eta(x)$. To obtain reasonably accurate results, the overflow fraction should be one of the fractions analysed.

The first method eliminates a host of potential sources of error. The measurement is very direct, and if the cyclone is run properly with a reasonably low solids loading, not much can go wrong. The drawback of using this method is that it is obviously a very time-consuming and cumbersome procedure, and it will not normally be practical in an industrial context, not to mention the problems of finding fine, monodisperse particles at a reasonable price. We will therefore concentrate on the second method in this section.

The main issues of concern in this second method are:

- obtaining and preparing samples for size analysis in the case of a laboratory cyclone
- choosing the method for size analysis

These two issues will be considered together in what follows, since the sample preparation depends on the method used for size analysis.

10.5.1 On-Line *vs.* Off-Line Size Analysis

Two main options exist for the measurement of particle size distributions: on-line and off-line.

Off-line analyses involve collecting samples of the solids fractions, dispersing them and analyzing them off-line, often using a liquid-borne technique. This method is more accurate than 'real time' on-line methods and it is often the most user friendly. Still, it has one important drawback. When collecting a particle sample from the process stream and re-dispersing it in liquid, one

loses the information of its state of dispersion in the cyclone or swirl tube. For instance, a small particle may have been traveling through the system as part of an agglomerate which, due to its large size, was captured in the separator. But when a sample of the captured fraction is dispersed in a liquid, it will normally disperse into its constituent or elementary particles so that our particle will appear in the size analysis as a small particle. Thus, the calculated grade-efficiency would show an anomalously high efficiency for such small particles. This is probably the reason for the typical 'tail' seen at the fine end of many experimental grade-efficiency curves, as mentioned in Chap. 5.

On-line analysis, which measures the particle size distribution in 'real time' while the particles are still dispersed in the carrier gas, avoids this problem. These methods characterize the particles as they appear in the gas stream, whether they are present as agglomerates or as dispersed particles. This can be an important advantage, especially in industrial situations, where the state of dispersion of the particles in the system is not always known. However, on-line analysis of particle sizes is normally far less accurate than off-line analysis and some serious errors can be introduced in the process of conveying the solids-bearing bleed stream through the size analyzer.

In summary: if the state of dispersion of the particles in the process is known or can be discovered (perhaps by comparing an on- and an off-line size analysis), off-line sizing gives the best results. Otherwise, on-line methods should be used, if feasible. In some situations, a relatively minor degree of particle agglomeration is known to occur but may be ignored if it does not significantly affect the purpose of the measurements.

10.5.2 Sample Capture and Preparation

We have already discussed the problems of isokinetic sampling. The issues are the same whether one wishes to measure only the solids concentration to determine the overall efficiency, or to subject the collected sample to size analysis either off-line or on-line by leading the bleed stream through some on-line size analyzer. In the latter case, the analyzer may be calibrated only for certain fixed gas flow rates passing through it, making it necessary to vary the opening of the sampling tube (or the number of parallel sampling tubes), to achieve isokinetic sampling.

When preparing a sample for off-line size analysis, one needs to ensure that the particles are dispersed in the suspension. This mostly means adding some surface tension reducing agent and dispersing agent to the suspension to make sure that the particles are fully wetted and dispersed (examples of such agents are Teepol and Calgon). The surface tension reducing agent ensures wetting of the particle surfaces, so that no gas is trapped between them. The dispersing agent avoids particle agglomeration in the suspension, often by imparting an electrical charge to the surface of the particles, so that they

repel each other. Vendors of sizing equipment normally list dispersing agents suitable for different types of particles.

The particles also have to be dispersed mechanically in the liquid. This can be done by stirring the suspension and/or by treating it in an ultrasonic bath to break up particle agglomerates. If the particles have a relatively high settling velocity, one needs to ensure that the large particles do not settle out. If the particles are soluble in the liquid one may be able to circumvent solubility concerns by ensuring that the liquid is first saturated in the material of which the particles consist.

10.5.3 Methods for Size Analysis

It goes beyond the scope of this book to give a full account of the methods available for particle size analysis. For a further study of this, we refer to the book of Allen (1990). The principles behind the methods differ widely, and they yield different measures of the particle size.

The size that determines the behavior of the particle in cyclones and swirl tubes is the dynamically equivalent size. Using methods that measure this size avoids errors arising from such things as a varying particle density or particle shape and, for this reason, are considerably preferable to others. Unfortunately, these methods are also rather labour intensive. Below we mention the most used sizing techniques, and discuss briefly their usefulness for grade-efficiency analysis. We start with on-line methods.

The 'cascade impactor' measures the particle size by leading the particle-bearing gas through a series of stages, each consisting of a jet and an impaction plate. When the particle-bearing gas, accelerated in the jet, impacts the plate, the particles that are below the 'cut size' for that stage will flow around the plate with the gas, while the coarser ones will impact the plate and stick to it. Each subsequent stage is configured such that it will have a lower cut size. If the cut size for each stage is known, or determined by testing, the particle size distribution can be found by weighing the material collected on each impaction plate. This method thus measures a dynamically equivalent particle size on-line.

A 'cyclone train' consists of a series of small cyclones (a few cm in diameter) with a progressively lower cut size. Like the cascade impactor, the cyclone train permits on-line measurement of the dynamically equivalent particle size distribution. The advantage of the cyclone train is that the cyclones can collect more particles than a cascade impactor.

A 'laser diffraction particle analyzer' can measure, in principle, the size distribution of particles suspended in a gas. However, if one attempts to determine the size distribution of agglomerates, laser diffraction may not give a very accurate result due to the nonsphericity of agglomerates. See also the discussion of this method below. Laser diffraction does not give a dynamically equivalent particle size.

Turning now to off-line methods

The 'disc centrifuge' classifies particles off-line in a liquid suspension in a centrifugal field. A suspension of particles is injected onto the surface (at radius R_o) of a 'spin fluid', housed in a disc-shaped cavity and spun at a precisely known rate (angular velocity Ω). The centrifugal force field propels the particles radially outward. The concentration of particles is measured as a function of time, t, at a fixed radial position. This can be done either by sampling or, as in the 'photosedimentometer', optically (see Figure 10.5.1). A light source/photocell couple located close to the edge of the disc-shaped cavity (at R_{dc}) measures the level of light obscuration. The velocity of the particles is a function of the diameter, density and shape. A force balance on a particle (treated as a sphere of diameter x) gives the following expression:

$$x^2 = \frac{18\mu \ln\left(\frac{R_{dc}}{R_o}\right)}{(\rho_p - \rho)\ \Omega^2\,(t - t_o)}, \tag{10.5.1}$$

where μ is the viscosity of the spin fluid and t_o the time at which the suspension is injected.

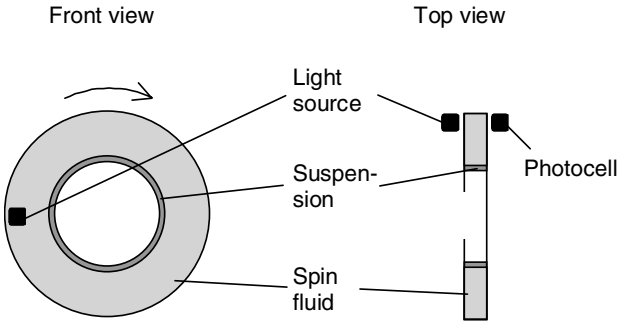

Fig. 10.5.1. Sketch showing the working principle of the disc centrifuge

The output from disc centrifuges has the form of turbidity versus time. The turbidity of a uniform dispersion is given by Lambert-Beer law (Devon et al., 1991):

$$\frac{I}{I_o} = \exp\left(-\tau\ l\right), \tag{10.5.2}$$

where I_o is the incident intensity, I the transmitted intensity, τ the turbidity and l the path length of the light. The theory behind the disc centrifuge has been set out by Allen (1987), although his treatment needs to be corrected on a couple of points of detail. Centrifugal sedimentometers measure dynamically equivalent particle sizes down to a fraction of a micron. They are therefore eminently suitable for cyclones and swirl tubes. Nevertheless, they are very labour intensive, and the technique can be difficult. If one is not careful,

instabilities can occur wherein a whole pocket of the suspension will settle out rather than the individual particles separately ('streaming', a phenomenon similar in nature to the effect of solids loading in cyclones as discussed in the Chap. 9), thereby spoiling the results. Streaming can be revealed by a stroboscopic light, but can also be recognized in the shape of the output curve by a trained operator.

Gravitational, as opposed to centrifugal, sedimentometers are also used to measure particle size distributions. In these, an initially homogeneous suspension is allowed to settle under the influence of gravity in a column. Particle sizes are determined either by sampling or by optical detection.

In the 'Andreasen settling bottle', samples are taken at fixed time intervals at a given level in the suspension. At any given time, particles with a 'critical' size have just settled from the surface of the suspension past the detection level. Particles finer than this are still present at the detection level at their original concentration, while particles coarser than the critical size are not present. The critical size corresponding to each sampling time can be found using Stokes law, and in this way the cumulative size distribution can be calculated directly from the particle concentrations in the samples.

As an alternative to sampling, optical detection of the turbidity of the suspension as a function of height and time is sometimes practiced. This is much less labor intensive than sampling, but, in contrast to the Andreasen settling bottle, a calibration is necessary.

Contrary to the belief of many, these methods are, in principle, suitable down to sub-micron particle sizes. Brownian motion is not a problem, since the displacement due to diffusion is proportional to the *square root* of time, not to time itself (it therefore makes no sense to derive a criterion for whether the method is suitable by comparing the mean displacements due to Brownian motion and due to settling after one second). The biggest problem is convective currents, which can be induced by a nonuniform suspension temperature during the process of settling.

In the 'electrical sensing zone' technique, a current flows between two electrodes placed on each side of an orifice. The particles are suspended in an electrolyte, and are made to travel through the orifice by drawing the electrolyte into the tube through the orifice (Figure 10.5.2). When a particle flows through the orifice, a jump in the current, proportional to the volume of the particle, is registered. This is a standard method for size analysis and is the principle underlying the operation of the popular Coulter Counter, which has been a standard for particle size analysis for the past 30 years. It measures a volume equivalent particle size, and therefore does not take into account the effects of variations in density or shape upon the particle's behavior in a centrifugal separator. This instrument is capable of measuring particle sizes in the 1 to 120 micron range. The range of particle sizes that can be measured in one analysis is limited, since the particle/orifice size ratio needs to be within certain limits. If the distribution is too wide, the solids have to be fractionated

by filtering before analysis. Below a particle size of 1 or 2 microns electrical noise becomes too disturbing and the method becomes difficult to use.

Particle sizing in liquid suspension by 'laser scattering' is becoming very popular. The method is extremely user friendly and reproducible. A suspension of the particles is irradiated with a laser beam, the particles in the suspension scatter the light and their size can be inferred from the scattering pattern, which is detected by ring-shaped detectors. The authors have had mixed success with off-line laser scattering analysis for cyclone characterization. For very large cyclones working with light particles, where the cut size is large, say 20 μm, laser-scattering analyses has resulted in reasonable grade-efficiency curves. For a small cyclone, however, working on a chalk powder and having a cut size of around 1.2 μm, laser scattering gave results that appeared to be inferior to centrifugal sizing. Laser scattering does not give a dynamically equivalent size. The method is less suitable for nonspherical particles, and uncertainly is still high in the sub-micron range.

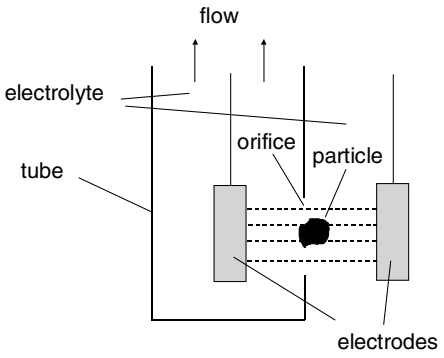

Fig. 10.5.2. Illustration of the electrical sensing zone principle for particle sizing

With any of the methods described above wherein the particles are suspended in a liquid medium, it is important that the medium itself does not significantly interact with the particles, thereby changing their size and/or density. Certain natural organic particles, such as grain flours or wood dust generated from sanding operations, will gneerally swell when suspended in water. They also become "water-logged" over time as a result of water filling up their cellular air pockets. The investigator just needs to be aware of such interactions and choose a suspension medium that is appropriate for the particle under investigation.

We have now looked at some of the different sizing techniques, the issue of off-line *vs.* on-line sizing, and the issue of which measure of particle size we obtain from the different methods. Before closing this section with some recommendations we need to consider one more issue: which type of distribu-

tion do the different methods give us—number, surface or volume—and which type do we actually want?

Let us answer the last question first. We saw in the early chapters that the distribution that gives us the most direct information about cyclone performance and that allows us to convert between cut size and overall separation efficiency is the mass distribution. If the particle density is independent of particle size, this mass distribution is the same as the volume distribution. This distribution is of overriding importance to our cyclone studies.

One question which then arises is, "What do the various particle analyzers measure?" From the electrical sensing zone technique, we get a number distribution, in spite of the fact that the method measures the volume of the individual particles. From the particle volume the instrument computes a volume equivalent particle diameter (see Chap. 2). The instrument then counts and reports the particles within a series of narrow (volume equivalent) diameter ranges.

This method can therefore be characterized as a 'counting technique', as can microscopy and those laser scattering techniques that measure one particle at a time. Although such methods are very precise, it can be difficult to obtain a good volume distribution from them. One has to count 1 million particles of 5 μm for every one particle of 500 μm to get the same volume. In other words: if the size distribution is wide it takes a lot of counting to get enough large particles included for a statistically good result in the coarse end.

Other methods, 'look at' the entire particle assembly together, such that a reading representing the the entire particle assembly has to be deconvoluted to obtain the size distribution. For light scattering methods the scattering pattern from nonspherical particles can give rise to the appearance of 'ghost peaks' in size ranges where no particles are actually present. Experience also shows that laser scattering tends to give wider size distributions than other methods.

The settling methods give us what we want: the Andreasen settling bottle yields a cumulative volume size distribution according to a dynamically equivalent diameter directly, since at any given time particles larger than the critical particle size are no longer detected. The disc centrifuge gives us a differential volume distribution: the turbidity can be shown to be proportional to the volume of the particles in a given band. Another advantage of the disc centrifuge is that it 'sees' only one particle size at a time.

The cascade impactor and cyclone train also give us the appropriate distribution: each captured fraction represents the volume (or actually mass) fraction of solids in the band between the cut diameters of two successive stages. This method also provides us with the dynamically equivalent particle size.

In conclusion, we can say that generating grade-efficiency data places some rather stringent demands on the accuracy of the particle sizing. We saw in Chap. 3 that relatively minor errors in the size data can lead to considerable deviation in grade-efficiency data. In an industrial context, on-line methods

may be best in some situations, while in a laboratory—where the state of dispersion of the test solids is known and controlled—off-line, liquid-borne methods give the best results. For true, accurate results, one should use a sizing technique that gives the dynamically equivalent particle size. The techniques that can do this, however, are cumbersome and labour intensive. If the particles involved are relatively large, a sizing technique based on laser scattering may yield acceptable grade-efficiency data. Electrical sensing zone techniques probably give better results than laser scattering, but may be impractical if the size distribution of the particles is wide. All the same, one should be careful with both laser scattering and electrical sensing zone methods—many industrial solids contain different types of particles, such as pulp and sand particles, with very different shapes and densities. These two techniques will not distinguish between them.

10.A Estimate of Errors

For a series of equivalent and independent observations y_i (i=1...N), the mean or average value is:

$$\langle y \rangle = \frac{1}{N} \sum_{i=1}^{N} y_i. \tag{10.A.1}$$

The variance can be estimated as:

$$s^2 = \frac{1}{N-1} \sum_{i=1}^{N} \left(y_i - \langle y \rangle \right)^2 = \frac{N}{N-1} \left(\langle y^2 \rangle - \langle y \rangle^2 \right), \tag{10.A.2}$$

where the advantage of using the second formula in the equation above is that it is computationally simpler. The estimated standard deviation is simply the square root of the estimated variance:

$$s = \sqrt{s^2}. \tag{10.A.3}$$

If N tests have been made, one would not, of course, just pick out one measurement and regard this as the best estimate of the quantity being measured. Rather, one would report the mean value. If a *series* of such mean values were generated, these would also have a certain standard deviation, which would be smaller than the standard deviation of the individual results. *The standard deviation in the mean values* can be found directly from the standard deviation of the individual results:

$$s_{mean} = \frac{s}{\sqrt{N}}. \tag{10.A.4}$$

We refer to a basic book on statistics for derivation of these results (e.g. Kreyszig, 1970).

If one wishes to estimate the variance (or standard deviation) in a *result* calculated from a number of data $f(x, y, z \ldots)$, each of which have certain standard deviations themselves, we can use *Gauss' formula for error propagation*:

$$s_f^2 = \left(\frac{\partial f}{\partial x}\right)^2 s_x^2 + \left(\frac{\partial f}{\partial y}\right)^2 s_y^2 + \left(\frac{\partial f}{\partial z}\right)^2 s_z^2 \ldots \tag{10.A.5}$$

Let us apply this to the issue of cyclone or swirl tube efficiency. We stated in the main text that including the overhead fraction when calculating the efficiency would result in lower errors.

Let us call the errors in determining each of the three mass flows M_f, M_c and M_e s_f, s_c and s_e, where, again the subscripts f, c and e refer to the feed, the collected, and the emitted particles, respectively. As stated in Chap. 3, we can estimate the efficiency in three ways (Eq. 3.2.2):

$$\begin{aligned} &\text{a)}\ \eta = \frac{M_c}{M_f}, \\ &\text{b)}\ \eta = 1 - \frac{M_e}{M_f}, \\ &\text{c)}\ \eta = \frac{M_c}{M_c + M_e}. \end{aligned} \tag{10.A.6}$$

The two last expressions incorporate the mass flow of the overhead solids, the first does not. Using Gauss' formula with the first expression (10.A.6a) for η gives:

$$s_\eta^2 = \left(\frac{\partial \eta}{\partial M_f}\right)^2 s_f^2 + \left(\frac{\partial \eta}{\partial M_c}\right)^2 s_c^2 + \left(\frac{\partial \eta}{\partial M_e}\right)^2 s_e^2 = \frac{M_c^2}{M_f^4} s_f^2 + \frac{1}{M_f^2} s_c^2 + 0 s_e^2. \tag{10.A.7}$$

We apply the same procedure for the two other expressions for η.

We now assume, as suggested in the text, that the error in each of the measurements of mass flow is simply proportional to the measurement itself, so that:

$$s_f = CM_f,\ s_c = CM_c,\ s_e = CM_e,$$

where C is a constant. This gives, after some simplification, the following three expressions in (10.A.6) for the variance in the calculated value of η:

$$\begin{aligned} &\text{a)}\ s_\eta^2 = \frac{2\left(CM_c\right)^2}{M_f^2}, \\ &\text{b)}\ s_\eta^2 = \frac{2\left(CM_e\right)^2}{M_f^2}, \\ &\text{c)}\ s_\eta^2 = \frac{2\left(CM_cM_e\right)^2}{\left(M_c + M_e\right)^4}. \end{aligned} \tag{10.A.8}$$

For illustration purposes, let us now assume that the cyclone or swirl tube is operating at 95% efficiency and, accordingly, take the values of 1.0, 0.95 and 0.05 for M_f, M_c and M_e, respectively. We then obtain for the three variances the expressions:

$$\text{a) } s_\eta^2 = 1.805C^2,$$
$$\text{b) } s_\eta^2 = 0.005C^2,$$
$$\text{c) } s_\eta^2 = 0.0045C^2.$$

Thus, the estimated variance in the calculated efficiency will be about 400 times greater—and the standard deviation 20 times greater—than the one we would have obtained if we had included the overflow fraction in the calculations. A similar principle is applicable for the calculation of grade-efficiency data.

We have to qualify this discussion, however. If the feed and collected fractions can be determined a lot more precisely than the overflow fraction, we might come to different conclusion when using Gauss' formula with the new estimates of measurement errors. This could result, for instance, if we were able to accurately weigh the feed and the captured solids, while being forced to perform on-line sampling of the overflow fraction.