The Technique of Particle Measurement in Industrial Hygiene

G. L. FAIRS

From the Research Department, Imperial Chemical Industries Ltd., General Chemicals Division

A knowledge of the concentration and size distribution of a suspension of air-borne particles has two main applications in the field of industrial hygiene. It can be used in the general investigation of pneumoconiosis and allied diseases but it has a further and wider use in providing the data essential for the successful design of gas cleaning equipment. This latter application is assuming increased importance in view of the impending legislation embodied in the Clean Air Bill at present before Parliament.

One of the main uses of particle measurement in industrial hygiene is therefore to provide data for the design of gas cleaning plant and of equipment for the efficient collection of the particles so that they are not merely transferred from the plant atmosphere to the open air outside. Except for electrostatic precipitators all dust and mist collectors depend for their operation upon dynamic forces and these forces increase rapidly as the particle size decreases thus increasing the operating costs. It is therefore most important that an accurate assessment of the size distribution of any collection of air-borne particles should be available so that the right type of collectors can be chosen and designed to operate under the best possible conditions.

The subject of particle measurement is a very wide one and any survey of it which is to be kept within reasonable limits must of necessity be sketchy and generalized. It is hoped, however, that such a survey, while containing little which is novel for the specialists, will prove of value to those not well acquainted with the subject.

Sampling Air-borne Suspensions.—Before dealing with the question of particle measurement for this purpose, it is necessary to consider the previous operation of providing a representative sample of the suspended particles. There are a number of difficulties in this which in general increase as the size of the particles and the inertial forces acting upon them increase.

Sampling Air-borne Particles in Ducts.—If the particles are solid and are suspended in air which is passing along a duct (as for instance in a ventilation system) all that is required basically is to extract a sample from the air stream by means of a nozzle, isokinetically, that is, at a volume rate at which the linear velocity of the air in the nozzle is the same as that of the main air stream in the duct. Departures from isokinetic sampling result in under- or over-sampling the larger particles according as the nozzle velocity is above or below that in the duct. Precautions have also to be taken to ensure that the selected sampling point is not affected by disturbances such as those caused by bends or valves and if no suitable point is available it may be necessary to install a mixing baffle of the type developed by Stairmand (1951a). Such a baffle will ensure that a representative sample is obtained 3-6 pipe diameters downstream from

the baffle. The extracted dust can then be collected in one of a variety of sampling devices, including miniature cyclones which can give cuts down to about 1μ for particles of density 2g/cm^3 (Stairmand, 1951a).

In the case of mists similar considerations apply except that a sampling cell has to be used to collect the particles. A sample of mist is drawn into the cell which is then isolated and the droplets allowed to settle into a film of immiscible liquid on a prepared cover slip which can subsequently be examined under the microscope. Care has to be taken when dealing with volatile liquids, or aqueous mists which are not at atmospheric temperature, so that the size distribution of the suspension of droplets is not altered by evaporation or condensation. This difficulty has sometimes to be overcome by the use of a modified cell (Stairmand, 1951a) which can be inserted into the fume main itself, sampling and sedimentation being carried out at the temperature of the main, the cell being subsequently flushed out with a stream of dry air at the same temperature.

Sampling Air-borne Suspensions in Stagnant Atmospheres.—When dealing with suspensions in the ordinary atmosphere of a chemical plant the problem is rather different from that of sampling suspensions in ducts since it is impossible to sample isokinetically when the velocity of the ambient air is negligible.

Under conditions, however, where the airflow is streamline and the inertial forces on the particles are negligible, the velocity of settling particles is the vector sum of their sedimentation velocity and the air velocity at that point. Walton (1954) has demonstrated that a change in air movement in a cloud of such particles set up by the introduction of a sampling flow will not give rise to a change in concentration at any point in the cloud. It is therefore possible to design low velocity sampling nozzles for collecting bulk samples of dust from plant atmospheres. Such elutriation nozzles can be particularly useful where bulk samples of dust are required in the pneumoconiotically active range since they can be designed to cut at any desired particle size. One such nozzle consists of a rectangular duct $4\text{ cm.} \times 6\text{ cm.}$ cross section and 20 cm. long having internal shelves set 1 cm. apart. If a stream of air is drawn through at a rate of 250 l. per hour all particles of density 2g/cm^3 and greater than 5μ diameter will settle in the nozzle and the $< 5\mu$ fraction can then be trapped in a small cyclone or other suitable device and weighed. The concentration in the atmosphere can then be calculated in terms of the actual volume of air sampled and size analyses can also be carried out upon the collected fractions.

Such elutriation nozzles can only be used for sampling mists for determination of weight concentration: they are not generally applicable for collecting mist samples for size analysis. Where such data are required a more suitable instrument is the cascade impactor (May, 1945). It consists of a number of nozzles in series set close to microscope slides upon which are spread a thin sticky film in which the droplets to be collected are immiscible. The cross sections of the nozzles and their separation from the slides decreases through the series thus causing any stream of mist-laden air which may be drawn

through the impactor to emerge with increasing velocity from each successive nozzle. This increasing velocity and the corresponding decrease of the turning circles of the emergent jets as the air stream meets the various microscope slides results in smaller and smaller particles being tangentially thrown out from the streamlines to make contact with the sticky films. The main difficulty with this type of apparatus is so to adjust the volume of air sampled that sufficient of the largest particles are trapped on the first slide to ensure a statistically accurate analysis without producing too dense a deposit of fine particles on the last slide. There is, moreover, the inherent disadvantage that individual particle counts have to be made on each slide which have then to be calculated as a composite size analysis.

Where a potential silicosis or pneumoconiosis hazard is under investigation the data generally required are the number of potentially active particles in suspension rather than a weight concentration. In such circumstances it is generally sufficient to extract a sample of the dust from the ambient atmosphere by means of a thermal precipitator (Green and Watson, 1935). This instrument depends for its action upon the phenomenon of the dust-free space around hot bodies. It consists essentially of a resistance wire heated by means of a 2-volt accumulator controlled at a constant temperature by means of a rheostat. The wire is bounded on either side by two parallel cover slips set within the dust-free space and kept cool by brass backing plugs. When a slow stream of dusty gas passes the heated wire there is a movement of the particles in the "cold" direction. The thermal precipitator has been proved in practice to be a very efficient sampling device.

The main disadvantage of the instrument in its original form is that the dust particles are precipitated in a narrow band in which the concentration of particles is heaviest nearest the centre line tailing off towards the edges of the trace. This means when the trace is being counted and sized under the microscope that instead of being able to select representative areas for sizing, complete strips of the trace have to be examined at right angles to its axis and it is usually very difficult to decide at what point the trace ends. To overcome this disadvantage Wainwright and Walton (private communication) have designed a modified precipitator in which one of the cover slips may be oscillated by means of a cam mechanism backwards and forwards across the heater wire in a direction at right angles to its axis. This produces a relatively wide trace of even density of deposit from which typical areas may be selected for counting and sizing.

While the original thermal precipitator was designed for use with the light microscope the increasing interest in particles less than 1μ has rendered it necessary to extend the investigation into the range of the electron microscope. The thermal precipitator, whether in its original or modified oscillating form, can be easily modified to accommodate electron microscope grids in small depressions in the brass backing plugs.

Methods of Size Analysis.—The choice of a suitable method of size analysis is a question which until recently

has not received adequate consideration, far too many people selecting a method by personal preference very often without taking any steps to see whether the technique selected is really accurate.

There are three main methods of size analysis available for air-borne particles: microscopical examination, sedimentation, and elutriation; I.C.I. has over a number of years investigated many techniques under these three heads and has developed ones which can at any rate be relied upon to give results of known reproducibility. In certain cases, as for instance when examining mist deposits collected in sampling cells or thermal precipitator traces, the nature of the deposit will limit the investigator to size analysis by microscopical examination. On the other hand, if, for example, a bulk sample of dust is available for the purpose of designing a wet scrubber for a plant ventilation system the most suitable method of size analysis would be by water sedimentation since the actual data required are the falling speeds of particles rather than their actual diameters. Similar considerations apply to air elutriation methods which would be particularly applicable where a determination was being made of the dust deposition round a factory chimney.

While considering the general question of size analysis it is important to realize that the parameter of particle size is different from the various methods of analysis. In the case of sedimentation or elutriation analysis where falling speeds are measured, the diameters are expressed in terms of the "equivalent sphere", which is a hypothetical sphere of the same density as that of the particle measured and which has the same falling speed under identical conditions. In the case of microscopical examination the diameter measured is that of the "equivalent projected area", *i.e.*, of a circle of the same area as the projected area of the particle. This is not necessarily the same as the diameter of the equivalent sphere and it must not be expected that size analyses by sedimentation and microscopical examination carried out on identical samples will give the same result.

Size Analysis by Microscopical Examination.—With this method it is now usual to match the particles against a series of graduated circles on a graticule which may be either set in the focal plane of the microscope eyepiece or in the case of the projection microscope may be embodied in the projection screen. The particles are counted and grouped into size ranges equated to a common area of slide examined, which can be expressed as number percentages or else as weight percentages by multiplying the number of particles per unit area in each size range by the cube of the corresponding mean particle size. In this latter case, since a few large particles exert a considerable effect on the weight distribution analysis, it is essential to count sufficient of these particles to ensure an analysis of the required accuracy. This whole question has been examined statistically and it is now possible to carry out a size analysis on a weight basis at any given limit of reproducibility for the largest particles and to ensure that this level is maintained throughout the analysis (Fairs, 1951).

In the case of the light microscope under optimum conditions of adjustment and illumination and with the

highest quality of optical equipment it is possible to resolve particles as small as about 0.3μ . It is, however, important to realize that below about 1μ it is impossible to differentiate between images of larger particles and diffraction halos of much smaller particles. It is therefore becoming general practice to restrict the use of the microscope to the range $76\mu - 1\mu$. This has an important bearing on analyses of thermal precipitator traces which have in the past been quoted as low as about 0.2μ . The optical limitations of the method are, however, now beginning to be realized and where large-scale investigations of thermal precipitator traces have to be carried out below the limiting value certain investigators are now using the electron microscope instead of the light microscope.

Size Analysis by Sedimentation and Elutriation.—The general principle underlying all sedimentation analyses is to measure the rates of fall of the particles in suspension, and then to convert these rates of fall into particle diameters by means of Stokes law.

$$V = \frac{1}{18} \frac{g(\sigma - \rho)}{\eta} d^2$$

where V = falling speed

g = gravitational constant

d = particle diameter

σ = particle density

ρ = density of suspending fluid

η = viscosity of suspending fluid

All the factors in the equation are in consistent units.

This would be a simple matter if the particles were of uniform size but in the usual dispersion encountered which has a range of sizes the normal procedure is to measure the change in concentration of the suspension at definite time intervals at which it is known that all particles greater than a certain size have settled out. Stokes law only holds for conditions of free settling, and in order to achieve this condition the concentration of the suspension must not be greater than about 0.2% by volume, which raises some difficulty in measuring changes in concentration at the end of an analysis when most of the particles have settled out. This difficulty has been overcome in the I.C.I. sedimentation apparatus (Stairmand, 1947). This consists of a sedimentation tube connected by means of a side arm to a reservoir which contains clear suspending medium. A quick release run off is provided at the bottom of the sedimentation tube below the side arm which when opened allows all the sedimented particles to be flushed out by means of a stream of clear liquid from the reservoir, the weight of particles in the collected fraction being estimated by any convenient method. This procedure is very much more sensitive than any which measures the amount of solid in a sample withdrawn from the suspension itself apart from the dangers of causing disturbances in the suspension thereby. By this means sufficiently dilute

suspensions can be employed to ensure that hindered settling and mass convection effects are prevented. In all sedimentation analyses complete dispersion of the particles in the suspending medium must be achieved by the addition of an appropriate dispersing agent, satisfactory dispersion being checked by examination of a drop of dispersion under the microscope.

Generally similar techniques are used in the case of elutriation. In this case a sample of the powder is subjected to a current of air under such conditions that any aggregates are broken down, the dispersed particles being passed into an elutriation vessel. The air rate is so adjusted that the average linear velocity in an upward direction in the vessel is equal to the free falling speed of the particles at which it is desired to cut. A satisfactory apparatus of this type is the Roller elutriator (Roller, 1931) which consists of a U tube containing the powder sample which is oscillated against a spring by means of a cam mechanism, while a stream of air is directed against the heap of powder, the dispersed powder being carried into the elutriation vessel and the material less than the "cut" size passing forward and being collected in a Soxhlet thimble. When elutriation of the finest particles is complete the air rate is increased and the next fraction is removed, the full analysis taking about four to eight hours.

For analyses of friable powders which might break down under the lengthy and violent action of the Roller apparatus, I.C.I. has developed a rapid elutriator which will give a reasonably accurate analysis involving six cuts in about 30 minutes. This apparatus is very simple and consists of a vertical elutriation tube which is made in two halves joined by flanged ends (Stairmand, 1951b). A thin gauze is fitted between these flanges which serves as a support for the sample to be elutriated. The lower end of the tube is connected to a flowmeter and the upper end through a Soxhlet thimble filter to a vacuum supply. As with the Roller apparatus the smallest particles are drawn off first but in this case elutriation for each size fraction is only carried out for 30 seconds since it is found that elutriation of most non-sticky powders is reasonably complete in this time.

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In the subsequent discussion the President pointed out that in practice it was necessary to use the best sampling instrument available without waiting for the ideal; in fact, he had been using the same instrument for 20 years to cover a large part of routine dust sampling.