DUST SAMPLING IN MINES

A REVIEW OF SOME METHODS OF SAMPLING, EXAMINING AND ANALYSING MINE DUSTS. COMPARISON OF RESULTS OBTAINED AND CRITERIA FOR THE ESTIMATION OF AIRBORNE DUST LEVELS WITH A VIEW TO DETERMINING MAXIMUM ADMISSIBLE CONCENTRATIONS.

INTERNATIONAL LABOUR OFFICE - GENEVA
The interest of the International Labour Organisation in the question of pneumoconiosis and its prevention both in industry generally and in the particular problems of the mining industry has been evinced by five international conferences and meetings of experts, as well as by the regular publication by the Office of medical and technical information relating to the prevention of dust diseases.

Regional organisations such as the European Coal and Steel Community have also directed efforts towards the prevention of accidents and industrial disease and in particular have encouraged scientific research. In this respect, the question of pneumoconiosis has been given a high priority.

In the course of these activities it has become apparent that little or no progress is being made in the standardisation of dust sampling methods and instruments and that this state of affairs renders the comparison of results very difficult.

As it is one of the traditional tasks of the International Labour Office to work towards international agreement on standards of attainment relating to conditions of work, a useful step in this field would be to prepare the ground for agreement on methods of airborne dust sampling and measurement and the interpretation of results or at least to enable valid comparisons to be made with a view to obtaining common standards that would be both practicable and acceptable to a majority of industrial and mining authorities.

With this end in view, the present review of methods of sampling, examining and analysing airborne dusts is presented. Those instruments in general use in the mining industry are briefly described, together with the procedures for dust analysis, and these are related to the criteria that have been adopted by different authorities. A proposal is made for a "reference" sampling procedure to which the results of existing procedures could be related, thereby enabling a greatly extended range of valid comparisons to be made of the hazards as they in fact exist throughout different mining fields.

This proposal is put forward as one approach towards greater comparability and a more standard interpretation of dust hazards. The theme may stimulate thought and lead to discussions aimed at a wider agreement in this field.

The present study was prepared by Dr. G. Degueldre, Senior Engineer at the Mining Health Institute, Hasselt, Belgium.
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INTRODUCTION

The present paper does not claim to deal comprehensively with the vast problem of industrial dusts; it is confined to the measurement of the various dusts that are produced and become airborne in the commonest mining operations.

The essential purpose of those organisations responsible for dust prevention is to protect as effectively as possible all miners employed in underground workings. Hence the sampling, examination and analysis of dusts are not to be considered as ends in themselves. In fact, all the known techniques, however important they may be in themselves, are only a means of improving working conditions by making it possible to restrict sources of dust, organise preventive measures and verify their efficacy. Unfortunately, the very many methods used in the different mining countries and the existence of sampling instruments of different types make it difficult, if not impossible, to make an accurate assessment of the results of measures against dusts and pneumoconioses which may be based on unusual, or even unknown, methods of dust measurement. Moreover, the efficiencies of the methods or the degrees of dust suppression achieved are not always directly comparable: incomplete knowledge of the experimental conditions and methods, even of routine methods, is often an obstacle to the generalisation, and sometimes even any application, of effective preventive methods. It is thus desirable to make a general survey of methods of dust sampling, examination and analysis before considering their comparability.

The technician must be able to sample, examine and analyse particles smaller than five microns. Agreement on this upper limit seems to be unanimous; in fact the so-called respirable dusts, that is those capable of reaching the alveoli of the lungs without being stopped in the upper respiratory tract, consist mainly of particles below five microns. The same considerations do not apply to the lower size limit. At first sight it would seem logical to provide against the smallest possible particles, but as can be seen from figs. 1 and 2 presenting some pulmonary retention graphs considered as classical, the maximum retention in the alveoli is between 1 and 2 microns, and there is a fall to a minimum around 0.2 - 0.3 micron (1)(2). Since it is not a matter of pure research into dusts, the lower limit of 0.2 micron, which is the limit of the resolving power of a very good optical microscope, is usually admitted to be more than satisfactory. In current practice, for routine measurements, the lower limit is fixed at 0.5 micron for reasons of convenience and sometimes even at one micron.
Consequently we shall review the principal measuring methods used in mines that enable particles from 5 to 0.5, or better from 5 to 0.2 micron to be studied and dealt with.

**Fig. 1.** Curves showing alveolar and upper respiratory tract retention of dust.

**I. SAMPLING AND EXAMINATION OF AIRBORNE DUST**

When dust is sampled for purposes of industrial hygiene it is usually desired to ascertain the concentration, particle-size distribution, mineralogical composition, and perhaps also the shape of the particles. It will be realised from the outset that it will be difficult to obtain all these data with one instrument and one method. A choice has therefore to be made according to the end in view and the method of expressing the results.

Measuring methods and instruments might be classified according to the parameter used to express the concentration: number of particles per unit volume, weight of particles, surface area of particles.

The choice of this parameter is of capital importance for it determines the significance attaching to particles in a given size.
Determination of the concentration by counting is liable to exaggerate the importance of the very numerous small particles, while determination by weighing exaggerates the importance of the large particles which are much heavier. Measuring the surface area is a middle course between these two extremes. It should however be noticed that the relation between counted and weighed concentrations is constant if the particle-size distribution is constant.

Fig. 2.- Alveolar retention of particles of unit density.

Whatever the merits of the different methods - and further reference will be made to this important matter - we prefer to classify measuring instruments according to their operating principle. Since, moreover it is difficult to separate sampling clearly from examination, the best known sampling instruments will be discussed with an indication of their greatest advantages and disadvantages, and the method of examination that is most appropriate or is simply imposed by them.

We shall first of all try to reply as briefly as possible to some questions concerning sampling techniques. When, where and how should samples be taken? What is a representative sample? Should sampling be continuous? Can it be discontinuous?

To measure dustiness the general view is that samples should be taken during the normal activity of a working-place by putting the air intake of the instrument (whichever it may be) at a place where the air current seems to be most uniform at about head height, usually in the centre of a return airway about 20 metres from a face or a pillar being extracted.

Unless it is desired to ascertain the quantity of dust inhaled by a particular individual, in which case he has to be followed whenever he moves, there is no advantage in coming too close to the sources of the dust, for that would only overload the samples with coarse particles. Since dust concentrations fluctuate considerably,
Continuous sampling would in theory appear to be the better. However, it is most useful to follow the fluctuations, which are sometimes abrupt, and to record them, and for this reason, although measurement is more onerous, some authorities prefer numerous separate samplings at regular intervals, which give an acceptable average if enough samples are taken. Lastly, sampling should be more or less iso-kinetic if its purpose is to determine a concentration by weight, because the distribution of air streams near the collector head (filter or probe intake) determines the proportion of the coarsest particles collected. This consideration is not so important when samples are only collected for counting particles. It may be observed that in the light of certain medical theories, some authors stated as early as 1953 (3) that the ideal index of the pneumoconiosis risk in coal mines should be in the form \( K D^2 (\text{silica}) + D^3 (\text{coal}) \), where \( D_2 \) and \( D_3 \) are not the square and the cube of the average diameter of the particles collected but the average of the \( d^2 \) and \( d^3 \) of the particles below five or three microns.

From different starting points French research workers reached similar conclusions (4): "The factor that really determines the seriousness of the danger is not actually the number of particles, but very probably the surface or the weight, or perhaps even the surface and the weight in relation to the dimensions, since the mechanism can vary with the dimension".

Thus at the time it was recognised that the number of particles of a certain kind could not alone determine the character of a dust concentration. Nevertheless, in several countries investigations into dust have continued to express the results of measurements as numbers of particles.

The abandonment, at least in theory, of counting as the sole method of measurement has become more marked from year to year. In 1959 one could read in the literature "whatever the theory accepted to explain the origin of silicosis, it will necessarily be the extent of the surface area or the mass of the particles deposited in the lung alveoli and not their number that will determine the harmful character of the environment" (5). This way of looking at the problem implied an accurate knowledge of the rates of alveolar retention and lung cleansing in relation to the size distribution, and perhaps also the nature of the particles, matters on which there is not unanimous agreement (6)(7)(8)(9). At the same time there was a proliferation of selective gravimetric instruments that collected only a certain proportion of dust in a given particle-size range. The fact that some of these instruments were not reproducible and the results of theoretically identical instruments were not comparable largely accounts for the survival of counting methods. The advocates of these methods considered that it was easier and surer to count all the particles capable of penetrating to the lungs than to make a distinction and to try to separate out those likely to be retained in the lung tissue.
A. Sampling Instruments

The operating principles of most sampling instruments are based on filtration, sedimentation, centrifuging, scrubbing or washing and precipitation - precipitation by impact, thermal precipitation, electrostatic precipitation, etc. Some instruments on the other hand permit the direct measurement of some characteristics (optical) of dust clouds but do not collect particles.

1. Filtration Devices

As a rule filtering instruments consist of an aspirator (fan, pump, water suction pump, compressed-air ejector), a filtering substance for collecting the dust, and an air flow meter (counter or aggregator) that measures the quantity of air aspired. The aspirator and the flow meter may be replaced by a hand pump of known capacity, the number of strokes being counted.

Filters today are made either of paper or of cellulose membranes with extremely fine pores (as small as 0.4 and even 0.2 microns).

It should be noted that papers are a mass of fibres with irregular interstices that are very often larger that the particles to be retained. The only way of measuring the porosity of these papers is by way of comparison to determine the partial vacuum resulting from the passage of a given quantity of air through a similar surface. The retention of particles on a filter is due not only to the sifting effect but also to the inertial effects produced by the sudden changes of direction of the air-stream (labyrinth), certain electrical effects that are very debatable, and, according to some authorities, Brownian movement.

(a) Solid filters, especially those of the cartridge type - the Soxhlet filter - are used for the continuous sampling of relatively large quantities of dust, usually with a view to subsequent analysis.

Differential weighing before and after sampling will give an exact measure of the amount of dust in the air. Part of the dust collected can be suspended in a liquid and particle-size distributions can be determined by fractional decantations and deep-cell counting under a microscope. This presupposes that the fine particles in the paper pores can be extracted and that the initial dispersion can be reconstituted in the suspension.

The following instruments are in fairly common use in coal mines:

- The Göthe filter which maintains iso-kinetic conditions for sampling with the aid of a double system of differential pressure take-offs that measure the air velocity at the entrance to the filter and in the immediate vicinity of the instrument; air intake = 12 m³/h (fig. 3).
The Staser instrument (fig. 4) aspiring one cubic metre of air an hour, enables the internal and external air velocity to be approximately balanced by selecting the diameter of the intake tube in accordance with local ventilation conditions; one type of filter holder used is shown in fig. 5.

The instrument of the Netherlands Dust Institute, an improved version of the Staser instrument, aspiring 4 m³/h.

The Hexhlet filter (fig. 6) uses a multi-duct elutriator to collect dust below a certain size limit (10); during sampling it effects a theoretical separation at about seven microns in the case of...
spherical particles of density one when the air flow is 100 l/min (original version of the instrument). The efficiency of separation varies with the air velocity, and thus largely depends on stabilisation of the aspirated air flow and the resistance of the filtering material (11).

These types of instrument, used in the mines of Austria, Belgium (13), Czechoslovakia, Germany (12), Great Britain (10), the Netherlands (14)(15) etc., are designed for connection to the underground compressed-air system.

(b) Soluble filters are used when a smaller sample is needed especially for examining dust in suspension. The best known is the tetrachloronaphthalene filter of the Le Bouchet and Mines du Nord du Pas-de-Calais types (fig. 7).

The filter is made from tetrachloronaphthalene and ether anhydride with the addition of alcohol; it is soluble in benzol. Its retentive capacity is equal to that of a good filter paper (16).

The preparation of filters and the examination of samples requires many but simple operations (decantations, centrifugings). The particles collected can be counted and weighed after evaporation of the liquid. In some conditions counting can be done without dissolving the filter (17).
Fig. 6.—Schematic view of Hexhlet sampler.

1. Elutriator. 2. Filter. 3. Injector.

(c) Filter papers other than the Soxhlet thimbles are suitable for different sampling methods.

- Small filters with a useful diameter of about 10 mm and made of Whatman, Tullis Russell and other types of paper are mounted on clips for very small-scale discontinuous sampling. The dust is neither counted nor weighed; measurements are made either of the opacity of the loaded paper, or, more simply, the percentage of light retained or transmitted by the loaded paper, or the pressure drop created by a certain air flow.

The best known of these instruments is the PRU hand pump (fig. 8). This simple instrument gives a general picture of dust conditions and can be used for many routine examinations; the inaccuracy of the measurements is unfortunately not compensated by the large number of samples (18)(19). Its inventor himself has shown that when using it for sampling and examination it was very difficult, if not impossible, to obtain accurate results (20).

- An instrument of the Le Bouchet laboratories directly and continuously records dust conditions by measuring the pressure drop; it does give an absolute reading, requires calibration for each application, and is hardly suitable for the rough conditions underground in mines. However, it has proved to be a useful research instrument in some industries (21).

- The MRE instrument takes continuous samples for weighing; it is self-contained and aspirates 2.5 litres of air a minute through a
Whatman glass-fibre filter with a useful diameter of 47.5 mm (22). It is a selective sampler; an elutriator (fig. 9) retains spherical particles above 6 microns (density 1.37).

These instruments, and more especially the PRU hand pump, have been widely used in Belgium and Great Britain.

Fig. 7.—Le Bouchet soluble filter apparatus.
1. Air intake. 2. Compressed air. 3. Air outlet.

(d) Millipore cellulose membranes are now used either for taking small samples for the purpose of counting the particles on the filter itself, or for taking large continuous samples for the purpose of ascertaining a concentration by weight, with or without separation of the large particles, or, again, for dust analysis.

The capacity of some of these membranes to retain submicroscopic dust is remarkable: at least 99.2 per cent for particles of 0.04 - 0.05 micron and 100 per cent for those of 0.1 micron (23).

Although the membrane technique is based on filtration it does not entail the inconvenience of suspension for counting the particles, since it permits of direct microscopic examination without elimination of the support and it gives a representative sample (24). The microscopic examination can be made in reflected light or by transparency after impregnating the membrane with a suitable liquid: cyclohexanone, benzyl alcohol, butyl alcohol or the like, either mixed or unmixed, so as to obtain the required refractive index.
Fig. 8.—Pneumoconiosis Research Unit handpump.

1. Pump. 2. Filter.

The instruments commonly used to measure concentrations by counting particles are the following:

- The Dräger pump (fig. 10), a bellows pump aspiring 100 cm$^3$ of air; membranes with a diameter of 20 mm are fitted on metal clips (25).

Fig. 9.—Schematic view of M.R.E. Sampler.

The Morin-Cerchar self-contained instrument does not need any outside power source (weight 4.1 kg including the battery) and is specially designed to be safe in coal mines (fig. 11). The volume aspirated ranges from 0 to 500 cm³/min. according to the adjustment of the electric pump. The instrument has a magazine for 20 spare membrane holders and so permits successive samplings to give an acceptable average over a sufficiently long period.

The Zurlo instrument (26) is a mercury pump of the clepsydra type with a capacity of 100 cm³ connected to a filter holder for membranes of 20 mm diameter. The airflow aspirated is 50 cm³/min. with membranes having pores of 0.1 - 0.2 micron diameter. In principle the quantity of air sampled is unlimited; a new cycle can be begun by merely overturning the instrument (fig. 12).

Gravimetric sampling on cellulose membranes is becoming increasingly frequent with the appearance of modern selective instruments.

An instrument for large scale sampling of dust of all particle sizes has been used in France for many years. The Jouan-Cerchar instrument (fig. 13) aspirates 10 - 12 m³/h through a membrane with a useful diameter of 134 mm mounted on a porous plate to avoid any deformation or tearing. A special device enables the airflow to be regulated as a function of the increasing resistance of the filter due to progressive clogging. The instrument is worked by compressed air.

Among the gravimetric instruments equipped with an elutriator or pre-separator that are beginning to be used in coal mines are the following:

The BAT fine-dust filter (fig. 14) whose pre-separator is a cyclone (27); the respirable dust fraction is collected on a membrane of 110 mm diameter. The acceptance curve of the cyclone coincides almost entirely with the pulmonary retention curve proposed by Cartwright and Nagelshmidt (28)(29). The airflow is adjustable. Study of the influence of the aspiration rate between 12 and 15 m³/h on the sharpness of separation and of the collection efficiency in relation to concentration shows that the error is
Fig. 11 a.- Layout of self contained CERCHAR apparatus.

1. Magazine. 2. 12 V. D.C. electric motor. 3. Reduction gear.
slight, about 5 per cent (30). This filter is used in German coal mines to determine the quartz content of dust capable of penetrating to the lung alveoli.

- The SFI-Dräger instrument (fig. 15) is equipped with an elutriator having sedimentation plates. Dust below five microns is collected on a membrane with a useful diameter of 40 mm (31) and the coarser dust is retained in the elutriator through which the air velocity is kept constant (0.238 m/s for a flow of 3 m³/h).

In these two last instruments the dusty air is aspirated by a compressed-air ejector connected to the mine network.

- The SIMGARD instrument (Safety in Mines Gravimetric Apparatus for Respirable Dust) was designed to collect 3 l/min of dusty air over a period of 9 – 12 hours (fig. 16). It is self-contained. The dusty air is aspirated by an ejector worked by compressed carbon dioxide contained in two cylinders. The dust is collected on a membrane with a nominal diameter of 47 mm after passing through an elutriator with sedimentation plates. The particles are in the accepted size range for lung retention. The penetration of unit-density spherical particles through the elutriator with an airflow of 3 l/min. is plotted against particle size in fig. 17 (32).
To close this section on instruments for gravimetric sampling on membranes it may be mentioned that the French mines are about to introduce a CERCHAR instrument that is entirely self-contained and aspires 2 m³/h over a period of 5 hours.

2. Instruments Collecting Dust by Sedimentation

Sedimentation instruments are of a rudimentary character, consisting essentially of a glass slide, covered or not with vaseline, which is exposed to the air for a certain time. The slide is laid horizontally in still air and is slightly inclined in a ventilation current.

The principle of sedimentation chambers is the imprisonment of part of the air to be examined in a confined space. Under still-air conditions, the dust settles by gravity on collection slides at the bottom of the chamber. The collection of dust is almost instantaneous but the settlement time is very long. Consequently instruments of this type (fig. 18) are little used in mines and moreover temperature variations are apt to set up convection currents that hamper the settlement of fine particles.

However the Wright sedimentation instrument (fig. 19) is an improvement; it automatically collects samples every 30 minutes,
and the dust so collected is deposited on a single plate at regular intervals for several hours or even several shifts. The average of the separate samples is obtained after counting the particles under a microscope. In certain conditions, and for particles above one micron, agreement is very satisfactory when the results are compared with those obtained with a standard instrument such as the thermal precipitator (33).

Fig. 13.- Bulk sampler Jouan - Cerchar.


3. **Sampling Instruments based on Centrifuging with or without Filtering or Sedimentation**

Centrifugal instruments collect by inertia particles suspended in an air stream moving with a controlled velocity; the principle may be compared with that of the centrifugal pump, depending on the flow of a fluid near a rotating disc and the flow of a rotating fluid near a fixed plate (34)(35).

The particles are classified according to their impact velocity, that is to say according to their size-range, form and nature; only
Fig. 14. - B.A.T₁ apparatus for sampling fine dust (section and external view).

those particles are retained whose impact velocity falls within a certain range depending on the airflow, the speed of rotation and the dimensions of the system.

(a) The Conicycle Selective Sampler can aspire 10 l/min. of dusty air during an eight-hour shift. It is self-contained, being operated by a battery. The sampling head (fig. 20) makes 8,000 revolutions a minute. There is good agreement between the results of a number of conicycles operating simultaneously at the same spot;

Fig. 15.- S.F.I.- Drager apparatus.

Fig. 16 a.- Schematic view of Simgard apparatus.

Fig. 16 b.- Simgard in use.

Fig. 17.- Standard acceptance curve for size selector.

1. Penetration of unit density spheres, per cent. 2. Particle diameter, microns.
the standard deviation is said to be less than 5 per cent (36). The cut-off curve for respirable dust at the moment of sampling is in good agreement with that of the Hexhlet instrument for spherical particles of unit density in the size range 1 - 7 microns.

(b) The T.C.1 Turbo-collector for continuous sampling is a prototype that has recently been on trial in the Nord and Pas-de-Calais coal mines. It measures the aggregate dust concentration over one or more shifts. It can operate for 40 hours on the power supplied by a battery of cap lamps (fig. 21). It gives a measure of the dust inhaled, and is an efficient collector of half-micron particles. It is very compact and light (2 kg). The rotation of the sampling head is effected by an electric micromotor at a speed of 6,000 revolutions a minute. It aspires 75 l/h. The working principle of the instrument is shown schematically in fig. 22.

(c) The Zurlo Pneumoclassifier (37) also collects dust by inertia but without recourse to centrifuging. The dust-laden air aspired by a compressed-air ejector or a vacuum pump undergoes abrupt changes of direction while flowing in a tapering channel. This process eliminates the particles whose inertia is too great to enable them to keep in the air stream; they are collected in small lateral receptacles as shown in fig. 23 (1st version). Suitable adjustment of the channel orifices gives an increasingly sharp separation of particles; the finest are collected on a Soxhlet filter placed after the last stage of separation. The instrument is essentially a classifier since it can fractionate a powder into five or six particle-size ranges, but it can be used without modification for sampling in mines, and is used as such in Italy.
Fig. 20 a.- Conicycle.

Fig. 20 b.- Schematic view of Conicycle sampling head.
With these instruments, which in fact should be classified as impingement samplers, the dusty air is aspired by some device, projected against an obstacle, and given an abrupt change of direction in a liquid.

(a) The Midget Impinger (fig. 24), the best known of this type, is derived from the Greenburg-Smith Impinger. The dust-laden air is aspired by a small portable pump at the rate of 3 l/min., or by an ejector, and then passes through a wash bottle that contains a liquid with a low surface tension (isopropyl alcohol). The sample can be as large as desired; it can be weighed after evaporation of the alcohol, or counted in a suspension that can be diluted or concentrated as needed. Unfortunately, the retentive capacity of the instrument is insignificant for particles below 1 micron, and the violent impingement on the bottom of the bottle at a velocity of 60 m/s alters the particle-size distribution of the dust (38)(39).
(b) The Midget Scrubber (fig. 25) was designed to increase the retentive capacity of scrubbers by achieving a more complete and intimate mixture of the collecting liquid and the dusty air. The chief disadvantages are disintegration of aggregates and shattering of coal particles resulting in an increased concentration with the duration of sampling (40). The instrument requires a compressed-air supply and normally aspirates 6 litres of air a minute.

(c) The pre-impinger as first used was adapted to the Midget Scrubber to sample non-respirable particles (41). It acts as an elutriator and improves the performance of the scrubber; by retaining the coarse particles it prevents the shattering of coal particles above 5 microns.

The pre-impinger is a glass sphere 30 mm in diameter with a circular opening 4 - 6 mm in diameter at the top; it is connected sideways to a midget scrubber or other instrument. The sphere is half filled with collecting liquid, usually isopropyl alcohol. The coarse particles are released from the aspired air at the moment of impact on the surface of the liquid. Naturally the efficiency of pre-selection depends on a number of factors: air flow, diameter of the opening, surface tension of the liquid, nature of the dust, etc. If the loss due to evaporation of the
liquid is compensated the instrument can take continuous samples over long periods, but so far it has only been used for research purposes in German mines (42).

5. Jet Instruments based on Impaction

In these instruments airborne dust particles are collected when dust contained in a small volume of aspired air is blown on to a glass slide, which may or may not be covered with an adhesive, by a hand pump or a mechanical pump. The commonest instruments of this type are the konimeters: the British, Kotze, Zeiss, Witwatersrand, Bausch and Lomb, Bergbau, Riken and others. They have been and still are used in a number of countries: Australia, Austria, Belgium, Canada, Germany, Italy, Japan, Poland, South Africa, Sweden, United Kingdom, etc.
(a) Konimeters, the principle of which is shown in fig. 26, aspire a very small volume of air, 2 - 5 or 10 cm$^3$ in a fraction of a second. The collecting slide is usually mounted on a rotating support so that several successive samples, sometimes 30 of 36, can be taken, which is some compensation for the excessive rapidity of sampling. But the collecting efficiency is low, and varies with the concentration, the particle-size distribution and the nature of the dust to be sampled.

With friable coal dust the collection efficiency is apparently improved by the shattering of large particles and the disintegration of aggregates (the impingement velocity is as high as 100 m/s in some instruments), which completely distort the particle-size distribution as determined by microscope.

The efficiency of collection also depends on the quality and the thickness of the film of adhesive, and the various constants of the instrument, which include the diameter of the intake orifice (annular or circular), and similar constructional details.

With the Witwatersrand konimeter (fig. 27) it has been found that the correlation of the average result of counts with the result given by the thermal precipitator might range from 0.46 to 1.24 according to the concentration, and from 0.31 to 1.45 when the size of the particles varies from one to 25, this size being defined as the percentage of particles above 2 microns as counted by the thermal precipitator (43). Investigations on the British konimeter, the use of which was for a time compulsory in Belgium coal mines, led to similar findings (44). Konimeters have proved to be unusable for continuous sampling owing to mechanical defects that could not be foreseen and could not be immediately detected. Moreover, for fine dust having 50 per cent by number of particles below 1 micron, collection efficiency falls from 66 to 15 per cent when the true concentration rises from 1000 to 10,000 p/cm$^3$ between 5 and 0.5 micron.

However, the Bergbau konimeter (fig. 28) which is widely used for routine measurements in German mines, does not have the same disadvantages. It has a settling chamber that eliminates particles above 5 microns in 60 seconds, and an adjustable aspirator. This konimeter is not used in Germany for counting particles but for determining the proportion of stone dust in a cloud of coal dust. The ratio between the two concentrations is determined after incineration of the coal particles (12). At the present time this determination is not made by counting but by a differential densitometric method.

(b) The Owens Jet Dust Counter (fig. 29) also collects dust by impingement on a slide but combines additionally the effect of the expansion of dust-laden air in an atmosphere close to saturation point. This effect is achieved by a piece of absorbent paper
Fig. 27. - Witwatersrand konimeter (sectional side elevation).

1. Rubber ring.
2. Glass slide.
4. Slide-retaining spring.
5. Cover plate.
7. Winding-handle spindle.
8. Barrel.
9. Head.
12. Trigger.
13. Piston.
15. Piston rod.
17. Push rod.

Fig. 28. - Bergbau-Konimeter.

1. Annular suction channel.
2. Jet housing.
3. Tapered jet.
4. Sedimentation chamber.
5. Sample number pointer.
6. Carrying handle.
7. Cover plate.
8. Glass slide for 36 samples.
10. Adjusting ring.
11. Channel to cylinder.
12. Steel piston.
15. Release button.
16. Cover.
soaked in water placed in the aspiration tube. The collection efficiency is only 40 per cent of that of the settling chamber, and from 38 to 41 per cent of that of the thermal precipitator, depending on sampling conditions (45).

The Cascade Impactor (fig. 30) consists of three or four collectors with different orifices arranged in series and at right angles one to another. This arrangement is designed to separate the particles into three or four size fractions (45) by a progressive reduction in the diameter of the orifices with a corresponding increase in the velocity of the air jet. This instrument has all the disadvantages of the konimeter, and the additional one of requiring three or four times as many counts per sample.

Fig. 29.—Owen's jet dust counter.
1. Intake. 2. Pump. 3. Collecting slide.

Fig. 30.—Cascade impactor.
6. **Electrostatic Precipitators**

Under the influence of a powerful magnetic field airborne particles, ionised and electrically charged, tend to move towards the anode or the cathode according to the magnitude and sign of the charge. In industrial dust-extraction practice electrostatic precipitators are of the Cottrell type. Similar instruments, but on a smaller scale, have been adapted for sampling in laboratories, more especially for the investigation of particles with the electron microscope (47). Since it is not safe to use them in an atmosphere containing methane, up to the present portable types for sampling underground have only been used in metal mines.

(a) The MSA electrostatic sampler (fig. 31) consists of a tube (the collecting electrode) in which a rod (the ionising electrode) is placed along the axis. The rod is connected to a D.C. generator delivering 15 - 20 kV. An electric fan delivers about 85 l/min. of dusty air. The electrically charged particles are diverted from the air stream and are deposited on the inner face of the collector. The retention efficiency is practically 100 per cent for particles down to 0.1 micron. Sampling is generally done for gravimetric determinations, but the instrument can be arranged to collect directly for counting. The sampling head shown weighs less than 2 kg; the whole instrument, which is completely self-contained, weighs a little over 13 kg.

(b) The electrostatic sampler of the Transvaal and Orange Free State Chamber of Mines has been specially designed to sample a
sufficient quantity of dust at places where the concentration is very low (48), and where consequently more than 85 l/min. must be sampled.

The instrument briefly described in fig. 32 is a collector with parallel plates preceded by a pre-ionisation chamber. The requisite voltage (6,000 - 12,000 V) is obtained from a 6 V battery after conversion, transformation and rectification. The pre-ionisation chamber is equipped with five brass rods 1/2 in. in diameter between which are placed tungsten wires 0.006 in. in diameter, 0.7 in. from the rods. The wires are positive. A voltage of 12,000 V is applied. The collector is constituted by a series of aluminium plates 3 in. long and 5.5 in. high, spaced 5/16 in. apart, and at a voltage alternating between 0 and 6,000 V. The collector and the high-voltage generator are not very bulky, measuring respectively 8 x 8 x 8.5 in. and about 6 x 7 x 15 in. By suitably positioning the wires in relation to the rods in the pre-ionisation chamber, an "electronic wind" can be created corresponding to 40 cfm for a current of 400 micro-amperes (1.13 m³/min.).

The efficiency of particle collection in terms of numbers has been determined by two thermal precipitators, one upstream and one downstream; it varies with the quantity of air traversing the collector. From practically 100 per cent for a flow of 565 l/min. it falls to 70 per cent for 4.52 m³/min., and the efficiency in terms of weight is 90 - 95 per cent for 2.83 m³/min.

The Gast electrostatic dust balance is a recording instrument the principle of which is outlined in fig. 33. The deposited dust is weighed on an electronic balance, an arrangement that enables the variations in dust concentration to be followed over a very long period (49). In its original form this delicate instrument was only used in surface installations but an improved version that has recently been under consideration is designed as a flame-proof recording instrument for use in German mines (air-flow rate = 1.5 l/s over 6 hours).

7. **Thermal Precipitators**

The collection of dust by thermal precipitators is based on the phenomenon of the "shadow zone" around a hot wire reported as early as 1870 by Tyndall and studied by Aitken, Ledge and Clark from 1884 onwards. Later studies by Green and Watson (50)(51)(52) finally led to the construction of the first thermal precipitator used by Patterson in 1935 in the South African gold mines.

If a hot wire suspended in a dust cloud is examined under a microscope it will be seen that around the wire looked at end on there is a zone about 1/10 mm wide that is free of particles (fig. 34).

In the light of various investigations of which the most important have been mentioned above, it has been concluded that this phenomenon is due to an intensification of the molecular bombardment in the direction of the thermal gradient.
The particles in the thermal field are subjected to a force directly proportional to the thermal gradient and inversely proportional to the actual temperature

\[ f = k \frac{1}{T} \frac{dT}{dx} \]

the factor \( k \) differing according as the particles are larger or smaller than the mean free path. The width of the shadow zone remains almost constant when the temperature difference is between 70°C and 250°C; it depends solely on the diameter of the hot wire (53). The maximum rate of precipitation is practically independent of the diameter of the particles at least down to 0.1 micron, and there is agreement that a thermal precipitator has a collection efficiency of 100 per cent for particles below 5 microns. It is the most reliable sampling instrument, furnishing a dust sample over the whole range below 5 microns, that is truly representative as regards concentration and particle-size distribution as well as the state of aggregation of the dust.

(a) Standard Thermal Precipitator

This instrument (fig. 35) consists of a platinum or nickel-chrome wire 250 microns in diameter heated by the Joule effect which is stretched between two brass blocks separated by a bakelite plate 500 microns thick in which a channel is provided for the airflow. Two brass plugs carrying glass discs 18 mm in diameter are placed on either side of the wire. After sampling, the dust is laid down along the image of the wire on the cover slips, provided that the airflow does not exceed 6 - 7 cm³/min.

![Fig. 35. - Section through thermal precipitator head.](image-url)

1. Air inlet. 2. Glass slide. 3. Platinum wire. 4. Twin cable. 5. Outlet to suction.
In the Mines Casella MKIII type (fig. 36) the battery has two lead elements for 7.8 or 10 A h and the temperature of the wire is about 100 - 105°C for a current of 1.3 A.

Fig. 36.- Casella thermal precipitator, mines type.

The sampling periods are short without being strictly discontinuous. Since the air has necessarily to pass slowly over the coverslips there is imposed a maximum flow rate. Consequently a time limit must be related to the concentration, and the duration of sampling has to be fixed so as to obtain an adequate deposit and to facilitate counting by microscope (55)(56).

All research establishments and institutions use the thermal precipitator for most of their laboratory measurements, and the instrument is very suitable for special investigations for the purpose of examinations with the electron microscope. In addition, it is used for routine or semi-routine measurements in coal and metal mines, more particularly in Australia, Belgium, Canada, Italy, Poland, South Africa, the United Kingdom and the United States.

A disadvantage of using the standard thermal precipitator is the need to count both coverslips for one sample, and also
the frequent changing of cover slips whilst underground. A remedy has been found:

- by inventing new slide holders that enable several samples to be taken on the same slide on each side of the wire (samples in cross or square formation); or

- by modifying the collecting head so that all the samples fall on one slide; or

- by replacing the hot wire by a band so as to broaden the deposit of particles and so take samples for a longer time; with this arrangement there is a risk of the thermal gradient becoming insufficient (57).

Because of the small amount of air aspired some authorities have held that the reproducibility of measurements made with the thermal precipitator and the collection efficiency are affected by excessive ventilation velocities in the working place. Within the usual velocity range (under 3 m/s) this effect is negligible for particles below 5 microns (57)(58)(59).

(b) Modified South African Thermal Precipitator

This instrument (fig. 37) was designed for sampling directly on a single standard microscope slide (3x1 in.) and to replace microscope counting by densitometric examination of particles. The slide in its support is moved by a rack and pinion fixed to a milled knob and equipped with a position indicator. The nickel-chrome hot

![Fig. 37. - Sampling head of modified thermal precipitator.](image)

wire 0.25 mm in diameter is supported over its whole length by a small grooved refractory block. The intake channel for the dusty air is so arranged that the aspirated air has to pass between the slide and the wire. The deposited dust forms a single strip on the slide; there is a 5 mm gap between successive strips.

The water aspirator has been replaced by one driven by a spring-operated motor. The improvement over the original standard precipitator is considerable as regards both weight and dimensions. With this instrument it was hoped to take selective samples containing all particles with a diameter corresponding to the maximum pulmonary retention curve and a percentage of other particles corresponding to their existing frequency. However, its efficiency in the desired zone is below that of the standard thermal precipitator (60). Fig. 38 shows the collection efficiency of the instrument compared with the desired performance for an airflow of 10 cm$^3$/min.

![Graph](image)

**Fig. 38.-** Collection efficiency of modified thermal precipitator (S. Africa).


A new instrument, still more compact and practical, is now on trial (61).

(c) Long-Running Thermal Precipitator

This instrument built in the laboratories of the National Coal Board (62) is self-contained like those described above, and has the incontestable advantage of sampling on a single slide by spreading the deposited dust over a much larger surface (12 times larger). Less air is aspirated (2 cm$^3$/min.) so that sampling can last several
hours without any risk of an overload, especially as an elutriator eliminates the particles normally retained in the upper respiratory tract (fig. 39). The motor driving the pump is supplied by an alkaline battery of the mining type (flameproof). This battery also supplies the current (0.8 A) for heating the wire in the sampling head. The complete instrument only weighs 4.6 kg. The 112 A model of the Casella Company (fig. 40) is used systematically in the British coal mines for routine dust control.

Fig. 39.- Section of sampling head of long-running thermal precipitator.

8. Instruments Measuring Certain Optical Properties of Dust Clouds

When a beam of light passes through a dust cloud or aerosol, light is absorbed and scattered. The extent of scattering in particular depends on the concentration of the aerosol; it will thus be understood that in certain conditions it will be possible to assess a dust concentration by measuring the intensity of the light scattered by the particles. Searching theoretical studies and
investigations carried on for many years in Germany, the United Kingdom, the United States and other countries have been described in important publications since 1962 (63)(64)(65)(66). This work has resulted in the construction of laboratory instruments whose field of application extends considerably beyond the bounds of the present paper.

However, one routine instrument that is easy to transport enables dust concentrations in mines to be measured underground by direct observation of dust clouds. This instrument, the Leitz tyndalloscope (fig. 41), includes a dust chamber through which the air passes, a source of light and a photometer. The intensity of the light scattered by the particles is compared with that of light from a reference source that can be adjusted by rotating a Nicol prism. Readings are instantaneous: it is only necessary to measure the angle of rotation of the analyser at which the illumination of the two fields observed through the eyepiece is equal. The scattered
light varies with the nature, concentration and size of the particles, its intensity being proportional to the intensity of the incident beam, the concentration (for a dust of a given type), and a factor $r^q$, in which $r$ is the radius and $q$ an exponent varying with the fineness of the dust. It is thus necessary to know the nature and the average size of the particles examined.

Fig. 41. - Leitz tyndalloscope.


- The nature is ascertained by a mineralogical examination made either by incinerating the coal dust to determine the stone content (12) or by photo-electric measurement of samples taken at the same time with the Bergbau konimeter (67).

- The particle-size distribution is estimated approximately by taking readings before and after settlement of the coarsest particles, that is before and after closing the observation chamber: 10 or 20 seconds are allowed to elapse so as to eliminate coal particles above 5 or 10 microns.

When the particles are very large in comparison with the wavelength of the light used, as with mine dusts even below 5 or 10 microns, the exponent $q$ is about 2 (68); thus the intensity of scattered light measured is proportional to the surface of the particles. With the end of graphs the results of tyndalloscope measurements may be expressed as a weight of a given type of dust in the range smaller than a given size. The results, however, are distorted in a humid atmosphere near the saturation point, and in an atmosphere containing oil aerosols or diesel fumes, since for very small particles below the wave length of light, the exponent $q$ is 6 (69). These defects have been largely remedied by improvements made in the
The tyndalloscope is in general use in German mines where it is proposed to introduce a new recording instrument based on the same principle (72). The tyndalloscope is also used in Japan, the Netherlands, Sweden and other countries.

B. Examination of Dust Samples

In this section the examination of dust is only considered in so far as concerns quantitative analysis, that is weighing or counting, particle-size distribution by weight or by number and densitometry. Moreover it is only treated to a limited extent, the discussion being confined to the methods recommended for so-called routine measurements that in fact are or can be applied in mines.

1. Weighing and Particle-Size Distribution by Weight

There is no difficulty in weighing dust samples even if it is impossible to separate the dust from its support; in such a case the support is weighed before and after sampling, subject to certain precautions such as drying the filters, and weighing at a constant temperature.

If it is desired to ascertain a particle-size distribution by weight the first essential is to collect a sufficient quantity of dust. Because of the dimensions of the particles sieving is impossible; the customary dry separators do not ensure a sufficiently sharp separation between the different sizes.

What is done is to suspend the dust in a suitable liquid in which the particles settle at rates varying with their size and specific weight and the viscosity and density of the medium (Stokes' and Cunningham's laws).

The classical methods are direct measurement of settlement by the Oden balance, measurement of variations in specific weight in a suspension by the Mohr balance (73) and measurement of the concentration in relation to time at a given level in the liquid with an Andreasen pipette. The last is the commonest method: at regular intervals the same quantity of liquid is aspirated at a given level, and the residue is weighed after evaporation.

It is true that in industrial hygiene detailed particle-size distributions are rarely required for routine measurements. In most cases one is satisfied with ascertaining the weight of particles below a certain size. Nevertheless, except as regards particles of the same nature and shape, this is a mistake, for the presence together of components whose shape and density are not identical seriously complicates the problem. The fractions collected successively contain particles that probably have the same settlement rate as a result of the disintegration of aggregates in the liquid.
This is one of the reasons for which attempts have been made to separate the size fractions during sampling by equipping filters with the elutriators or pre-selectors described in the preceding section. In a horizontal pre-selector, separation depends on the resultant of the air velocity and the velocity of the free fall of the particles.

2. Counting of Particles - Particle-size Distribution by Counting

Counting by microscope is the general rule, whether the dust has been sampled by midget impinger, konimeter, thermal precipitator or cellulose membrane. For counting, a microscope is often supplemented by a micro-projector; the particles are counted in different fields and compared with reference samples of known dimensions placed at the edge of the projection screen or incorporated in the eyepiece (graticule).

(a) Counting of Particles in a Suspension

This is the usual method of examining dust samples with impingement instruments, soluble filters, and sometimes solid filters. In the last case it is essential to remove the fine particles from the pores of the filter paper. This is done by turning the filter over and placing it on an Erlenmeyer flask where it is subjected to progressive negative pressure so that it is penetrated by a certain quantity of water or isopropyl alcohol.

After homogenising the liquid by agitation, and repeatedly filling a small pipette, the operator deposits a few drops in a counting cell 1 mm or 0.1 mm deep over which a cover glass is placed for protection.

The filled cell is placed on the microscope stage, and 10, 20, 30 or more fields are examined, depending on the density of the suspension. However, it is advisable to fix a time for prior decantation and to limit the duration of the examination so that the measurements will be reproducible (38)(40).

Counting particles in a suspension has serious drawbacks; in fact it is hard to discover whether the suspension accurately reflects the particle-size distribution of the dust sampled. It is also possible that the smallest particles visible under the microscope used have not had time to settle on the bottom of the cell.

(b) Counting of Dust Deposited on Glass Slides

There are very many variants of counting methods. In some cases the slides are examined as they are, with or without a cover glass. Sometimes they are previously treated with hydrochloric acid before or after, or before and after, heating in a furnace or on a stainless steel plate at temperatures ranging from about 200°C to about 600°C.

The total magnifications used range from 100 to 3,000 diameters. There does not seem to be any marked preference for light field, dark field, phase contrast or other microscope technique. However,
the result of a microscope count has no value unless the method of examination is clearly stated.

Some recommended counting methods are very briefly outlined below by way of example:

- Counting konimeter slides without special preparation. The glass slide on which the particles are deposited is held by a screw-threaded ring in a rotating support that can occupy 30 different positions. By merely rotating the support each of the 30 dust spots can be brought under the microscope incorporated in the instrument (light field). A graticule in the eyepiece defines two opposing sectors whose angle at the apex is 18° (fig. 42).

![Fig. 42. - Graticule used in konimeter work.](image)

The distance between one of the diameters and the line parallel to it represents 5 microns (magnification x 200). A count is made of the number \( n \) of particles below 5 microns deposited in the two sectors. Since in the present case the volume of air aspired is 5 cm\(^3\), the number \( N \) of particles per cm\(^3\) of air is

\[
N = \frac{10 \times n}{5} = 2n
\]

since theoretically the two sectors represent one tenth of the circular surface of the spot.

- Counting of thermal precipitator slides. Before examination the cover slips are mounted on a support such as a microscope slide. The microscope has a number of objectives, usually three, with low magnification for adjustment and centring and high magnification for counting. The objective used for counting is almost always of the apochromatic type with 2 mm immersion and numerical aperture 1.3; its own magnification is about x100. The eyepiece has a magnification of x6, 10 or 12. The image is often projected to obtain a final magnification of x1,000, 2,000 or 3,000. When a sample is taken with the standard thermal precipitator, counts are taken of the particles in 2, 3 or 4 traverses 30 or 40 mm wide, perpendicular to the line of deposit. An endeavour is made to keep the number of particles per traverse or per sample as low as possible so as to reduce counting errors; for example, 75 - 100 particles from 1 to 5 microns are counted per traverse or 400 per sample.

This brief account of some features of examination methods will suffice to show that there is no comparability of results of counts of dusts sampled by two different instruments (konimeter and thermal precipitator). Even if it is admitted that the same collection
efficiency might apply to different instruments, it must be realised that the results of counts of the same sample may be spread over a whole range of values of which the extremes differ by a factor of 20 or even 30 according to the type of microscope used. In fact it is not enough to employ microprojectors with the same total magnification to arrive at identical results, neglecting counting errors. The results depend essentially on the numerical aperture of the objective, not to mention the wave length of the light which determines the resolving power of the system. In examinations with a microprojector in a light field at magnifications x 1000, 500 and 200 of dust sampled with a thermal precipitator in rock workings in coal mines, and using the same light, the same eyepiece and very high quality objectives of numerical aperture 1.3 - 0.85 - 0.50 it has been found that whereas the magnifications are in the ratio 1 to 5 the concentrations are sometimes in the ratio 1 to 20 when particles below 5 microns but without any lower limit are counted (73). Moreover it is not possible to seek a correction factor for conversion from one result to another for such a factor would depend on the particle-size distribution of the dust and the density of the sample deposit. In the investigation referred to (73) it was found that the practical resolving power of the optical instruments used might be about 0.2 - 0.7 to 2.2 - 2.3 microns and the median diameter of the particles counted 0.7 - 1.2 - 2.4 microns according to the final magnification adopted and the numerical aperture of the objectives used (fig. 43).

Fig. 43.- Cumulative curves from the same sampling, using different magnifications and N.A.
(x 1000, N.A.1.3; x 500, N.A.0.85; x 200, N.A.0.50)

1. Cumulative per cent. 2. Diameter in microns.

On the other hand if a lower limit is fixed for counting, for example 0.5 micron, the ratio between the number of particles of 0.5 - 5 microns at a magnification of x 1000 and the number of particles below 5 microns at a magnification of x 500 is more nearly constant. The difference between results obtained in a light field and in a dark field also decreases when high magnifications and large numerical apertures are used.

However, for low magnifications and numerical apertures of 0.25 - 0.30 - 0.50 comparison is impossible, since the result of counting depends almost entirely on particle-size distribution, which is one of the unknown quantities to be found. It is idle to try to count particles of 0.5 - 5 microns with objectives of numerical aperture 0.25 - 0.30 - 0.50.
To these difficulties related to optical instruments must be added another due to differing views of dimensions. Some authors consider a mass of small particles that are in contact (aggregates) to be a large particle; others try to separate them as much as possible when counting. This difference of attitude necessarily leads to considerable discrepancies, which increase when there is a risk of similar errors arising from the superimposition of particles. There is thus an advantage in using objectives with high resolving power (immersion in oil, numerical aperture 1.3, etc.), and also a need to limit the duration of sampling.

(c) Counting of Particles Deposited on Cellulose Membranes

The considerations set out above also apply to the counting of particles deposited on membranes, whether by direct observation in reflected light, by transparency in a light field, by phase contrast, or in a dark field after impregnating the membrane with cedar oil, amyl acetate, cyclohexanone, etc. Moreover, the choice of the impregnating liquid is of the greatest importance. The refractive index of the solvent has a direct influence on counting; particles become invisible in a homogeneous medium of the same index, and this must be avoided at all costs. On this point it may be noted that the solvent with the refractive index closest to that of silica is benzyl alcohol \((n = 1.544)\) which is still sometimes used. Methyl glycol has an index of 1.399, while that for a mixture of equal parts of amyl acetate and cyclohexanone is 1.423.

(d) Particle-Size Distribution of Dusts—Expression of Results

The particle-size distribution of a dust cloud can be determined by counting the number of particles with a diameter in a certain size range, the diameter being that of a circle having a surface equal to the surface projected by the particle under examination. It is customary to work with the following particle-size fractions: 0.2 to 0.5 micron; 0.5 to 1 micron; 1 to 2.5 or 3 microns; 2.5 or 3 to 5 microns; over 5 microns. If the average number of particles in each size range in the microscopic field is known, and taking into account the surface area of the field and of the deposit or preparation, as well as the volume of air aspirated by the sampling instrument, it is possible to determine directly the number concentration of particles in the different size ranges.

Investigations have been made into the reproducibility and the accuracy of certain recommended methods of operation. The overall error in the measurement of a concentration can be found by comparing the results of samples taken simultaneously at the same place with a number of instruments of the same type by different operators, and possibly having the samples examined by a number of persons. The imprecision of this procedure is the resultant of a number of errors: error in counting in a field; error due to the non-homogeneity of the deposit (choice of sector for counting); errors of instruments; errors in measuring the volume of air aspirated, and so on. It is considered that a total error of \(\pm 15\) per cent is not excessive \((73)(74)\).
The graphical representation of particle-size distribution, in the form of cumulative distribution curves, makes it possible to calculate useful parameters: fineness of the dust, average diameter of particles in a given size range, and others. The ordinates of these curves show the percentage of the number of particles smaller or larger than values shown on the abscissae and vice versa. It is also possible, on bases corresponding to each size fraction, to construct rectangles whose areas are proportional to the number or the percentage of particles in the fraction concerned. In this way a stepped frequency diagram is obtained that approximates to the frequency curve derived from the cumulative curve (fig. 44).

For certain investigations it is very convenient to use semi-logarithmic or logarithmic diagrams. Although this is a matter outside the usual field of routine measurements, it may be mentioned that the particle-size distribution hardly ever gives a Gauss frequency curve, but generally an asymmetric curve (Galton's curve; log-normal distribution). Different distribution laws have been proposed (75)(76)(77)(78)(79)(80)(81), but in very many cases the S-shaped cumulative particle-size distribution curves become straight lines when graphs are plotted with the abscissae on the logarithmic scale and the ordinates on the Galton scale (log-probability diagrams). In this case, with a log-normal distribution, construction of the cumulative curve giving the percentage of spherical particles smaller than a given size makes it possible to obtain graphically
cumulative curves for the surface or the volume of these particles (82) (4) (83). It is thus theoretically possible with reasonable accuracy to convert the number to the surface area or weight of particles of identical shape. Unfortunately the "shape factor" is not constant, even, in some cases, for the particles of a dust cloud considered to be homogeneous because of the nature of its components. Particles of the same nature, with the same rate of free fall may have diameters as measured by the microscope differing by a factor of 3 (84). It is not sure that the difference is wholly due to the shape of the particles, but it shows that the risk of error is great if the volume or the weight is calculated from the number of particles, especially in a heterogeneous medium. Investigations into these matters have more than a theoretical interest; knowledge of the relationship between number and mass of particles is becoming more important with the trend towards selective gravimetric sampling (85) (86). Developments have even reached the point where laboratory instruments have been designed to measure particle-size distribution both by counting and by weighing (87).

To conclude this section on the counting of particles, it may be mentioned that attempts have been made for more than 15 years to find an automatic method for counting and analysing the particle-size distribution of mine dusts (88)(89). Automatic instruments have come into general use in some laboratories for counting particles or objects having practically the same dimensions as dust particles, blood globules for example, or for surface studies of monodisperse aerosols, but it does not appear that up to the present a really satisfactory routine method has been found for determining the particle-size distribution of mine dusts in the size range 0.2 to 5 microns.

3. Densitometric or Photo-electric Examination

Photo-electric methods based on the measurement of scattered light, measurement by extinction, etc. and densitometric methods based on the measurement of opacity were devised to avoid the difficulties of counting by microscope.

(a) The simplest instrument used for routine measurements is the PRU densitometer, which is used together with the PRU hand pump (see page 8). This instrument has a lamp bulb (6 V - 6 W) a barrier-layer photo-electric cell, connected to a galvanometer, an aperture normally closed by an opaque shutter that prevents the light from the lamp from acting on the cell except in examination periods and a device for adjusting the distance between the lamp and the cell by means of which the galvanometer needle can be moved to its extreme position for a clean paper. A simplified expression of the results is \( r = L/L_o \) where \( L \) is the galvanometer reading for the luminous flux passing through the soiled paper and \( L_o \) is the galvanometer reading for a clean paper (former Belgian regulations). This ratio multiplied by 100 is called the percentage of transmitted light; it decreases as the dust concentration increases and only measures the concentration for a constant number \( n \) of aspirations. Conventional limiting values of \( r \) enable dusty atmospheres to be classified.
To express measurements British researchers (90) have used the formula
\[ p = 100 \times \frac{D1.5}{n} \] (1)
where \( n \) is the number of aspirations and \( D \) is \( \log_{10} \frac{L_0}{L} \), the opacity of the soiled paper. The magnitude \( 100 \times D^{1.5} \) may be read directly on the upper scale of the densitometer if the maximum deviation for a clean paper is found. The factor \( p \) must be multiplied by a constant \( c \) obtained by calibration with the thermal precipitator. The number of particles is given by the formula
\[ N = c \cdot p. \] (2)
However, \( c \) varies from one workplace to another according to the seam, air flow, method of working, dust suppression equipment, etc.

Other researchers (91) have suggested replacing formulas (1) and (2) by
\[ N_n = a \cdot D^{1.5} + k \] (3)
where \( a \) and \( k \) are constants obtained by calibration with the thermal precipitator. Lastly, the Safety in Mines Research Establishment has proposed to calculate the number of particles \( N \) from the formulas
\[ N_n = k' s \]
and
\[ s = 100 \left( 0.3010 - \log_{10} \log_{10} \frac{L_0}{L} \right) \]
where the symbols have the same significance as before and \( k' \) again is given by calibration with the thermal precipitator. The spread of these factors \( k' \) is less than that of the other factors \( c, a \) and \( k \) (92).

If it is admitted that dusts collected in coal mines are opaque, and that the scattering of light is trifling compared with its absorption, and, further, if the photo-electric cell receiving the luminous flux and the measuring galvanometer have linear response curves (practically correct assumptions) it can be shown that the difference between unity and the ratio of the transparency of a soiled paper to the transparency of the same paper when clean varies with the total surface of the sample when the density of the deposits is low, that is, when the chances of covering the shadows of the different particles are negligible (\( L/L_0 \) greater than 0.7 – 0.8).

If account is taken of the possible covering of particles it is found that the function
\[ s = k' \log_{10} \log_{10} \frac{K}{L_0} - \log_{10} \log_{10} \frac{L}{L_0} \]
calculated from the readings \( L \) and \( L_0 \) should vary with the total surface of the particles collected, or, if the particle-size distribution is constant with their number (18).

Lastly, the assessment of the dust concentration is very inaccurate when the results of the measurements are expressed as the percentage of light transmitted, \( r = 100 \frac{L}{L_0} \), which is only a measure of the surface of the sample when \( L/L_0 \) approximates to 1, that is when the concentration is low.
Densitometric examination is therefore justified when the parameter sought is the surface of the particles collected, and is appropriate for dusts of the same kind that have a constant particle-size distribution.

In mines generally, and in coal mines more particularly, dusts are heterogeneous: the same seam will liberate dust clouds that differ in composition and particle-size distribution according to the method of working, the operation in progress, the means of transport, the preventive measures, and so on. It is for this reason that measurements with filter clips are difficult to make and interpret. It is impossible to compare one face with another, and even one shift with another at the same face (19). Figure 45 shows, with a probability of 90 per cent, the number of particles in the range 0.5 - 5 microns corresponding to a percentage of transmitted light determined by 10 hand-pump clips used at the same face during three shifts: morning (M), afternoon (m) and evening (s).

(b) When considering the design of new sampling instruments, it seems logical, as we have said, to take into account only particles capable of reaching the lung alveoli, and if possible aim to collect them in proportion to the extent to which they are retained in the alveoli. These criteria are satisfied by the modified South African thermal precipitator (60). In the light of the results of experiments on both animals and human beings, and of certain properties of siliceous particles (93) (94) (95) (96), in the case of dusts with a very high silica content it is considered that
concentrations should be expressed as a function of the surface of the respirable particles and not of a number or a weight (97). It also seems desirable to attach more importance to the surface of siliceous particles between 1 and 2 microns and underestimate that of the dust below 0.5 micron.

This being so, since the dust in the South African gold mines is highly siliceous, their particle surface is the most important dimension to determine; efforts have been made to work out a routine method for measuring the surface of particles deposited on the slides of the modified thermal precipitator instead of counting the particles (98).

The design of the instrument is shown in fig. 46. The lamp A (8.5 V - 35 W) is supplied by a 12 volt battery with a rheostat and an ammeter in the circuit. This lamp emits two beams collimated at B when passing through two lenses C (focal length 2 in.) as they travel towards two mirrors D. The two beams, refocussed by the lenses E (focal length 4 in.), cross in the slide F (slide of the thermal precipitator) and are received by the photo-electric cells G. The slide F mounted on the support H can be moved sideways by the screw-threaded rod J and the handle K. Since the cover is fixed, the slides are changed in the compartment L which has a lightproof door M. The inside of the box which is painted dull black is divided by two partitions N to prevent the dispersion of light.

![Diagram](image-url)
A telltale P is provided for seeing the position of the slide. The two photo-electric cells are connected in opposition to the potentiometer Q (25 ohms) and the galvanometer at zero R (150 ohms), which is so sensitive that the displacement of the needle is 4 mm per microampere. The purpose of the potentiometer is to reset the galvanometer at zero when a beam of light is passed through a virgin area of the sampling slide (to remedy any asymmetry in the distribution of the light beams). To make a measurement the slide with the samples is moved to the intersection of the light beams one of which traverses the glass and the dust and the other only the glass, and the deviation of the galvanometer needle is a measure of the quantity of light scattered or absorbed by the dust.

In all, measuring errors do not exceed 1 per cent.

Comparison of the results of counts with photo-electric measurements of dust from slides of the modified thermal precipitator enable the photo-electric cell to be calibrated. By calculating \( \xi(n, d) \) and \( \xi(n, d^2) \) when \( n \) is the number of particles of diameter \( d \), it is possible to find the relationship between the average particle diameter and the ratio photo-electric reading: projected surface of particles.

It has been found that for particles with an average diameter over 1.8 microns the photo-electric measurement is proportional to the surface of the particles, but that as the size decreases the photo-electric measurement per unit of surface increases, reaching a maximum when \( d = 0.5 - 0.6 \) micron, and then decreases rapidly when the average diameter is below 0.4 micron. This method of measuring is said to reflect more faithfully the harmful character of the siliceous dust in question.

(c) A third routine method is widely used in the Federal Republic of Germany; it is the photo-electric assessment of samples taken with the Bergbau konimeter, but solely for the purpose of determining the stone content of coal dusts (99). It is a simple and rapid method but it calls for certain special precautions. Since the intensity of the scattered light depends largely on the particle-size distribution, it is essential to collect only particles below a certain size, in this case 5 microns. This is why the Bergbau konimeter is fitted with a sedimentation bell and a suction channel which for heavy concentrations may be supplemented by an elutriator eliminating particles above 5 microns. Before the samples are examined, the film of vaseline on the slide has to be removed; this is done by rinsing with carbon tetrachloride, care being taken not to carry away the deposited particles. If the vaseline is not removed the results of the measurements will be higher.

The actual measurement is made with a microscope using transmitted light and equipped with a dark field condenser; a selenium photo-electric cell incorporated in the eyepiece is connected to a galvanometer.

When the conditions mentioned above are satisfied, that is elimination of the vaseline or other adhesive and pre-selection of
the coarse particles, the intensity of the scattered light is proportional to the number of particles between 1 and 5 microns in a given dust (coal or stone).

The size of the stone fraction of coal dust is found by photo-electric measurement before and after incineration of the samples at 550°C. Figure 47 shows the average ratio between stone-dust fractions as determined by counting particles from 1 to 5 microns with a microscope and as determined by photometry. This ratio is considered to be satisfactory for routine measurements. One of the advantages of the method is that it does not require any special apparatus, since the microscope used can be prepared almost immediately for ordinary counting.

(d) Measurement by photo-electric densitometry has also been used for slides of the long running thermal precipitator to determine the proportion of inhalable particles (100). The special construction of the densitometer together with certain artifices of manipulation enable the measure of the opacity to be converted into a number of particles, and the weight of dust to be found by means of suitable conversion factors.

Attempts have been made to use methods of this kind to determine particle-size distributions of dusts (101) (102), but so far these methods have been confined to highly specialised research laboratories.

C. Comparison of Sampling and Examination Techniques.

There is hardly any point in comparing sampling instruments alone, because it implies the choice of an instrument and a reference
method of examination, and assumes the prior verification of the reproducibility of each of the instruments in question over the whole range of use.

In fact it is not enough to operate simultaneously at the same place with two different instruments and then to examine the samples by suitable methods, even if the same units are used to express the results. If when the same upper limit is taken for counting, the results differ statistically speaking, it may be that one of the two instruments favours small particles at the expense of large ones, or allows some of the finest particles to escape, or that one of the two methods of examination does not allow the finest particles to be counted when they have been collected. These contingencies are also possible when a lower limit has been fixed for counting.

Further, if by operating simultaneously at the same place with two different instruments whose samples are examined by different methods, results are obtained that do not differ significantly when the same upper and lower limits are taken for counting (for example 5 to 1 micron), there is no proof that the two techniques are valid for another particle-size distribution, especially if the distribution has a wider spread, for example 5 - 0.5 micron.

If samples are taken simultaneously at the same place with a number of identical instruments used in the same way, and are then examined by different methods, it should be concluded that one method of examination is better than the others if the results differ significantly. This procedure however is not worth following unless it is quite certain that the so-called respirable particles can be counted, that is all particles between 5 and 0.5 micron at least, whatever their nature.

But if samples are taken simultaneously at the same place with identical instruments used in different ways, and are examined by the same method (that described in the preceding paragraph) it must be concluded that one sampling method is better than another if the results differ significantly.

It is thus advisable to use the sampling method associated with the method of examination that, for the range selected (5 - 0.5 micron), gives the largest number of particles irrespective of their nature, provided that the operating methods do not distort the particle-size distribution, in particular by shattering particles.

Consequently, sampling instruments cannot be compared by means of a single method of examination, or methods of examination by means of a single sampling instrument. Since certain counting methods are bound up with certain sampling methods, in practice the whole set of operations - sampling plus examination - has to be compared in a given field of use.

Notwithstanding these restrictions, comparisons prove to be extremely complicated because it is difficult to create homogeneous and reproducible dust clouds and to collect dust at the same place over the same period of time. Finally, to obtain figures comparable from country to country it is also necessary to have all the necessary apparatus for following the methods peculiar to each in every detail.
Many comparative studies of the results of simultaneous samplings have however been made during the last 15 years.

(1) One investigation comprising a large number of measurements in the underground workings of coal mines made with the thermal precipitator, the midget impinger, and the Soxhlet thimble, was carried out in Belgium in 1952 (39). A satisfactory correlation was found between the results given by the midget impinger and those given by the Soxhlet thimble. The correlation between the results given by the midget impinger and those given by the thermal precipitator (greater than 1 micron) was about 2 for coal dust owing to the shattering of particles and the disintegration of aggregates. In the other cases it varied widely.

(2) Comparison of the average measurements of concentrations made with the thermal precipitator, the soluble filter, the midget impinger, the midget scrubber and the electrostatic precipitator was undertaken almost at the same time by the CERCHAR laboratories (103). This systematic comparison, made with quartzite drilling dust, and supplemented by a searching statistical analysis, showed that the total number of particles as measured with a phase contrast microscope with a magnification of x 900, increases with the true concentration increasingly rapidly for the different instruments in the following order: soluble filter, thermal precipitator, electrostatic precipitator, midget impinger, midget scrubber. If only particles above one micron are counted, the thermal precipitator and the electrostatic precipitator form one class, and the soluble filter, the midget impinger and the midget scrubber form another, quite distinct from the first; the correlation between the two classes averages 1.7, and there is a 95 per cent probability that it will be between 1.4 and 2.2 because of the existence of aggregates that are disintegrated during the preparation of samples. Further, the authors have considered that the explanation of the fact that the total count with the electrostatic precipitator is higher than with the thermal precipitator although the number of particles above one micron is much the same must reside in the partial disintegration of aggregates by the mutual repulsion of charged particles.

(3) A later investigation also carried out by CERCHAR showed that the only reliable instruments at the time were those with solid filters and the electrostatic and thermal precipitators (24). In particular, the authors compared results by using, on the one hand, the thermal precipitator, the soluble filter (from which the particles are systematically disintegrated before counting) and the membrane filter (retaining the support), and on the other, the soluble filter and the membrane filter, but with this last employing the method of examination recommended for the soluble filter, namely, dissolving the support, and disintegrating the deposit by peptisation after separation by centrifuging.

They found that the thermal precipitator seems to give results a little lower than the membrane, although the difference is not statistically significant, and that the membrane and the soluble filter when treated in the same way give identical results, while the soluble filter always gives results definitely higher than the thermal precipitator and the unseparated membrane.
Since then other comparative investigations have been carried out on instruments based on the same principle as well as on instruments of fundamentally different design.

Investigation of the validity and reproducibility of measurements made with three types of konimeter have shown that each type has a particular performance, and that this performance of a given type varies with the length of time it has been in use (104).

Short-running (standard type) and long-running thermal precipitators have also been investigated in laboratories and underground in mines (105). The correlation between the results given by the two instruments is quite satisfactory provided that the sampling time is kept short to avoid overlapping of particles. The sampling period with the long-running thermal precipitator should be considerably reduced - 2 or 3 hours - if the dissociation from aggregates of particles between 5 and 0.5 micron are counted (106).

The most recent comparisons of sampling and examination methods made with other instruments have often been limited, as were those just referred to, to very special cases; some were made with gravimetric instruments equipped with elutriators having different separation curves and operated in ways differing from those recommended by the inventors.

During the past few years such comparisons have been carried out more especially with the Hexhlet instrument and the Conicycle (34), the T.C. Turbo-collector and the Morin-Cerchar instrument (35), the Conicycle and the long-running thermal precipitator (36), the tyndalloscope and the BAT filter (27), and the tyndalloscope and the SPI-Dräger instrument (31).

As matters stand at present, no evaluation or comparison of dust contents, and consequently no definition of a maximum not to be exceeded, has any significance unless the type of equipment, the method of sampling and the nature of the dust are precisely known.

Until an adequate method of operation, and an optimum method of examining samples collected by a reference instrument, have been defined, a comparison of instruments that would be valid in all circumstances is almost an idle dream. Moreover, except in the case of homogeneous dust clouds with a constant particle-size distribution, the correction factors will always vary with the particle-size distribution or the nature, or both, of the dust examined. Lastly, if concentrations are to be compared by counting and weighing, with or without an elutriator, the proportion and the nature of the ingredients of the dust, as well as the particle-size distribution, will be of decisive importance.

Without wishing to underestimate the obvious theoretical importance of such researches, we consider that if the aim is to control more effectively the efficacy of means of combating dust, it is far better to compare dusty atmospheres as a whole, and the criteria of assessment, to determine whether the mining environments rated as good, bad or doubtful by certain researchers are given the same ratings by others, for each will have his own methods of operation. This is the line being taken by the High Authority of the European Coal and Steel Community, which has entrusted a working party with studies of this nature.
II. QUALITATIVE AND QUANTITATIVE ANALYSIS OF MINE DUSTS

For present purposes, discussion of the composition of mine dusts is confined to determination of the silica content, whether silica associated or combined with all kinds of other substances, or so-called free silica. The necessary analyses must inevitably be made on dust samples collected in the atmosphere: the proportion of silica found in a specimen of sandstone, for example, is by no means identical with the proportion found in particles raised during the drilling and crushing of the same sandstone. When coal dust is to be analysed it is first incinerated and the actual analysis is carried out on the resulting ash (stone dust). Both the temperature and the duration of incineration vary considerably; they depend on the methods adopted for subsequent treatment, but the object is always to prevent inopportune reactions between the silica and the silicates present. For this reason incineration is usually performed at a low temperature, ranging from 450°C to 650°C or thereabouts.

A. Determination of Total Silica

Silica in combination with aluminium, sodium, calcium, etc., forms silicates whose structural formulas are very complex. There are several analytical methods, the majority designed for the analysis of samples of stone dust; they are not all reproducible when it comes to fine dust collected in the atmosphere.

Two chemical methods were studied thoroughly some ten years ago; they were worked out after statistical analysis of experimental results and discussion of the different stages of dust analysis so as to fix and justify all the details of the manipulations (107).

1. Gravimetric Method

(a) If the sample contains no combined silica it is enriched with silica by means of a selective reaction suited to the nature of the substance to be analysed. The concentrate obtained is treated with hydrofluoric acid in a strong acid medium to volatilise the silicon in the form of SiF₄. This is accomplished by prior sulphuric treatment and incineration at 825°C. When the amount of the substance to be examined is between 30 and 100 mg, the typical deviation with this method is about 0.50. In addition to the analysis of various substances, such as chalks, diatomaceous powders and quartz dusts, this method can be used to verify the purity of total silica after disintegration, or free silica after the treatment described below.

(b) If the sample contains combined silica, which is the commonest case, it is made soluble by being disintegrated with alkaline carbonates. The mixture of equal parts of sodium and potassium salts lowers the melting point to 180°C. The silica is made insoluble by drying the residue for one hour at 120°C after evaporating the hydrochloric acid. The amount of silica carried
away is independent of the mass of the precipitate but dependent on the method of operation. The use of 100 cm³ of a rinse consisting of a 10 per cent solution of hydrochloric acid reduces this loss to 0.000047 ± 0.00013 grams of silica. For test samples of only 30 mg after all corrections have been made the standard deviation does not exceed 0.56.

2. Colorimetric Method

Measurement of the absorption of light by yellow silico-molybdenum compounds formed in an acid medium by ammonium molybdate is one method of determining total silica. The sample, made soluble by carbonates, is concentrated to an extent that eliminates the influence of the iron, calcium, magnesium and other ions; the pH is 1.6 and the light used has a wave length of 430 millimicrons. The concentration of the solution is sufficient to nullify the influence of inequalities in the transparency of the dishes used. In the conditions described the reproducibility of the method is acceptable for a silica content below 30 per cent, when the typical deviation is 0.9. This method of analysis is thus more particularly indicated for samples rather poor in silica or weighing less than 30 mg.

B. Determination of Free Silica

Free silica is found in nature in manifold forms both crystalline and amorphous; chalcedony, opal, tridymite, cristobalite, quartz and others, quartz being practically the only one found in coal deposits. Apart from some very rare exceptions there is no need therefore to bother about the form it takes in the mining industry, and it is almost always taken to be quartz. This would not be the case in the iron and steel and refractory industries.

1. Chemical Determination of Free Silica

The successive stages in the determination of free silica are the following: dissolution and elimination of soluble compounds, decomposition of the silicates and dissolution of the colloidal silica formed, weighing of the residue, which as a rule is the uncombined silica originally present in the sample, and verification of the residue to make sure that it is really silica.

Some 15 methods have been proposed; they are not all valid in all circumstances because silicates are difficult to attack and different methods have to be used, and also the solubility of quartz in different media varies with its particle-size distribution. All the methods have been compared, analysed and generally discussed (108), The more it is desired to spare the quartz, the narrower the scope of the method and the longer its duration. The researches undertaken have led to the elaboration of a method of operation based on the use of pyrophosphoric acid at 275°C, which determines quartz accurately in small quantities of fine dust (109)(110). The manipulations are few and simple. In fact the method is a quick one; in routine operations it gives ten results per day per operator. The test samples are from 10 to 50 mg of mineral substance
weighed in the usual conditions. The loss of quartz is small, less than 10 per cent; it can be found by introducing into the general correction formula a parameter characteristic of the dust examined (average specific diameter). This method, which is well suited to the analysis of small quantities of airborne coal dust (100 mg), has been tried with many minerals from other sedimentary rocks, or metamorphic and igneous rocks. For special cases when unusual minerals like zircon, disthene or tourmaline may be present in considerable quantities another method based on melting potassium pyrosulphate has been worked out in full detail (109).

Nowadays it can be considered that all methods of chemical analysis are capable of solving all current problems with more than sufficient accuracy, and they are also very effective in particularly difficult cases.

2. Mineralogical Determination of Quartz

Classical petrographic methods do not permit the study of particles below 5 microns and are thus of no use for the analysis of industrial dusts. The examination of thin films with the polarisation microscope, even if it were possible for such small dimensions, would be a source of considerable errors, since there is not necessarily any fixed relation between the silica content of dust and that of the rock from which the dust comes (111).

Two new methods suitable for fine powders have been proposed since 1954 (112) (113).

(a) Method of Coloration by Immersion over a Dark Background

It is known that the refractive index of a mineral varies with the wave length of the light employed. Consequently an immersion liquid is chosen with a refractive index equal to that of the mineral sought for a given wave length. When the wave lengths are altered, as they deviate more and more from the given wave length the surface of an immersed particle produces reflections and refractions that are more and more distinct. When illuminated under a white light the particle has a certain composite colour independent of its diameter. This method is applicable down to 2 microns.

(b) Method of Coloration by Immersion with Phase Contrast

By combining the relationship between the refractive index and the wave length of light with the properties of phase contrast, a relative decrease or increase in the intensity of certain wave lengths of the light source can be effected at the position of the image, and the change will be greater, the greater the difference between the refractive index of the medium and that of the particle. This method gives colorations that are less differentiated than with the preceding method, but as against this it enables particles of about 1 micron to be identified easily.

With these methods, which are directly applicable to sampling slides without vaseline, not only quartz but also kaolinites and
micas can be identified. They are commonly used for routine analyses of particles deposited on glass slides in Belgium (thermal precipitator slides) and Germany (konimeter slides).

Dust immersed in a liquid with a refractive index of 1.560 is examined under a mineralogical microscope (Ortholux Leitz) equipped with an apochromatic objective with a magnification x 40 and numerical aperture 0.7, and a binocular eyepiece 1.25 x 10, so that the total magnification is x 500. The instrument is provided with a Heine condenser by means of which, by simply turning a milled screw the illumination can be gradually changed from light field to phase contrast, half-dark field and dark field. A field of observation 50 x 130 microns bordered by reference data of known dimensions can be selected by means of a grating in the eyepiece. In each field the operator counts the total number of particles between 1 and 5 microns and those with the characteristic coloration of quartz. A simple rule of three will give the percentage number of particles of quartz between 1 and 5 microns, which is a sufficiently accurate determination if account is taken of the fact that the quartz content of a powder tends to decrease with increasing average fineness of the particles (111) (114).

Until very recently the mineralogical determination of quartz in dust deposited on cellulose membranes was not possible because of the lack of homogeneity of the mixtures of impregnation and immersion oils.

Even in routine measurements it is not logical to determine a dust concentration by counting and then calculating the quartz content in terms of weight. If the particle-size distributions of the various ingredients of a dust differ very widely it may happen that the quartz content as weighed and the content as counted are in the ratio of 1 : 20, especially when the rocks are fairly light and the minerals heavy, as in metal mines. It was thus important to determine the quartz content by counting samples collected on membranes because membranes are employed in metal mines. Researches for this purpose carried on in Italy have recently been brought to a successful conclusion (115).

It may however be observed that the difference between the quartz content by counting and the content by weighing is less in coal mines where the specific gravity of the two ingredients of the dust varies at most by a factor of two. In coal mines where prevention is reasonably effective the concordance between the chemical method of determining free silica and the mineralogical method of determining quartz is more than satisfactory, at least within the usual range of quartz contents found in coal mines (55).

3. Physical Determinations of Free Silica

(a) Differential thermal analysis consists in heating or cooling simultaneously in identical conditions and at a predetermined rate dust in which there is an unknown percentage of quartz and another sample that contains none at all, alumina for example. At 573°C quartz undergoes a reversible crystalline transformation
accompanied by the absorption or liberation of heat; because of this transformation the two samples do not heat or cool in the same manner. When the temperature difference between the two powders is plotted against time, at 573°C the curve will show a peak the height of which will depend partly on the quartz content. By comparison with values obtained from samples of known quartz content, the quartz content of any dust whatsoever can be determined.

Although very small differences of temperature can be detected by highly refined measuring techniques (116) and differential thermal analysis can give the crystalline silica content of a sample of powder in less than two hours (117), this method is hardly used any more because it requires a relatively large quantity of mineral (500 mg). Study of it has however been resumed on occasion for certain researches because it seemed likely to give a qualitative estimate of the extent of the crystallisation of quartz (116).

(b) Analysis of quartz by X-ray diffraction, a method derived from the work of Debye, Scherrer and Hull, is more practical and more accurate, both quantitatively and qualitatively, than differential thermal analysis. Its accuracy is such, and was even when it was first employed systematically in France in 1953, that quantitative analyses can be made from samples ten times smaller than those needed with the preceding method (118). It is also of incontestable value because it permits determination of the different forms of free silica.

When a rod of agglomerated microcrystals is exposed to a narrow cylindrical beam of monochromatic X-rays the diffracted rays are directed on to a photographic film. Since the position of the spectral lines is different for each mineral it is easy to identify them. The fraction of each element in the mixture analysed is then determined by measuring the relative intensity of the different lines after calibration with reference spectra.

The method has been greatly improved during the past few years (119). Improvement was necessary not to achieve greater accuracy, but to enable very small samples to be analysed, no more than 7 mg of mineral in the form of dust collected by gravimetric instruments equipped with elutriators. An automatic sample changer makes it possible to record almost continuously the diffraction diagrams of 36 successive examinations of preparations (120). With this instrument combined with a new device, also automatic, for measuring absorption (121) and supplemented by a special diffractometer (122), 36 samples a day can be analysed with the aid of four reference preparations.

This procedure is now used in the Federal Republic of Germany for the analysis of dust samples taken in coal mines by the BAT instrument.

(c) Infra-red spectroscopy is an analytical method of great refinement that requires only very little mineral, not more than a few milligrams. This method was used for the qualitative and quantitative
analysis of silica powders in 1953 by Haccuria, Lejeune and Duyshaerts¹, and for the analysis of mine dust in 1956 (123).

The atoms of molecules oscillate continuously about their position of equilibrium, and for many substances the frequency of oscillation is in the infra-red band. Infra-red spectrophotometric examination of such substances gives a spectrum whose absorption bands are determined by the chemical nature of the substance, or the type of atomic and crystallographic bonds between the excitable dipoles of the molecule. The sample is prepared by dispersing 1 to 3 mg of the dust to be examined in from 200 to 400 mg of dried and powdered potassium bromide, which has been rendered as homogeneous as possible by a ball mill agitated by a magnetic vibrator. Pastilles 0.5 mm thick are made with a powerful press and measured very accurately with a micrometer to determine the amount of substance per unit of surface. Quantitative analyses are made by reference, that is by comparing the extinctions with those for samples of known composition.

If special precautions are taken, the concordance between results obtained by this method and those obtained by chemical analysis are quite satisfactory.

The study of determination by infra-red spectroscopy has recently been resumed with the object of simplifying the manipulations and eliminating disturbing influences due to the presence of other substances in association with the quartz (124). It is not yet a routine method that can be easily generalised.

Concordance between X-ray diffraction and infra-red spectroscopy is said to be more than satisfactory (125).

III. MAXIMUM CONCENTRATIONS, THRESHOLD VALUES. CRITERIA FOR ASSESSMENT

The routine control of dusty atmospheres in mines usually involves two distinct determinations: determination of the dust concentration and determination of the quartz or free-silica content of the dust. In coal mines in some districts only the stone content (ash) is measured, and this is considered to be proportional to the quartz content of the deposit (126).

Determination of the threshold of danger is a delicate task and it can only be accomplished with the help of medical statistics compiled over very many years, and provided that one knows the quantity and the quality of the dust inhaled by the persons who throughout the period have undergone periodical and systematic medical examinations and radiographic controls. Industrial medical investigations

in this connection are in progress at the present time in Belgium, the Federal Republic of Germany, South Africa, the United Kingdom and other countries. The investigations are based on a variety of theories, the equipment employed also varies widely, and it may be expected that when they are published the results will create much confusion as a result of erroneous interpretations.

It is instructive at the present time to see how maximum concentrations are fixed by using dust sampling instruments and methods of examination that are totally different from those used to set the standards. "Standard values" defined over 20 years ago are blindly accepted when sampling with the midget impinger, the cascade impactor, the Owens jet dust counter, one or other of the konimeters, the thermal precipitator, the cellulose membrane or any other instrument, although each necessarily requires a different examination method, and in some cases methods are even altered by incinerating the samples beforehand or immersing them in acid. What is more, particles down to 0.5 micron are counted with objectives of numerical aperture 0.4 and even 0.3 although their resolving power is insufficient for particles so fine as this.

However this may be, two tendencies have become apparent in assessments of the danger of a dust cloud:

- measuring the weight of dust and determining the percentage of stone (ash) and perhaps the quartz content; and

- finding the number of particles below a certain size, or, better, between two limits, and determining the amount of quartz in these particles.

A. Proposals, Recommendations or Regulations in Force

Dust concentrations and quartz or ash contents are combined in more or less acceptable formulas to determine maximum permissible concentrations. It should however be noticed that even with gravimetric sampling, and notwithstanding the many investigations carried out recently on selective gravimetric samplers, there are not yet any standards for gravimetric concentrations of dust that can be retained by the lungs. There are too many parameters remaining to be defined. What in fact is the ideal cut-off diameter for an elutriator? How to determine the equivalent of the cut-off diameter by a standard test? How to take account of the particle-size distributions of the various ingredients which are not necessarily the same as that of the mixture? What is the influence of the particle-size distribution and the form of the ingredient with the greatest specific weight? These questions are still subjects for research. At a symposium at Cambridge (28 September - 1 October 1965), agreement could not be reached on the specifications of an ideal elutriator. For spherical particles of density 1 (Unity Density Sphere : U.D.S.), some advocated a 50 per cent cut off at 1 microns, while others adhered to the principle of a cut-off curve ranging a 50 per cent cut off at 5 microns.
For these reasons maximum permissible concentrations or threshold values are almost always based on airborne dust at workplaces.

It should also be noticed that since there is no universally applicable standard criterion, a number of methods are sometimes accepted or recommended in certain countries even if a particular method of routine examination is prescribed by regulations for one or other industry.

Below an account is given of the threshold values adopted, recommended or imposed in the mines of certain countries, together with the principles governing the methods employed, but without reference to the frequency of examinations. This account, based on the literature, is inevitably incomplete in spite of the appearance of an important general review of the subject published in 1962 (127).

Australia

Widely differing instruments are used in the different regions: Owens jet dust counter, thermal precipitator, midget impinger, Watson konimeter, etc.; microscope magnifications range from x 150 to x 1,500 and counting is done either in a light field or in a dark field for particles under 3, 5 or 6.5 microns or between 1 and 5 microns.

In New South Wales the regulations for coal mines lay down threshold values; values above those in the accompanying table are considered dangerous; they refer to the free-silica content of the material treated (133).

<table>
<thead>
<tr>
<th>Free Silica Content (Percentage)</th>
<th>Maximum Average Concentration (part/cm³ less than 5 microns)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Less than 10</td>
<td>700</td>
</tr>
<tr>
<td>10 - 20</td>
<td>600</td>
</tr>
<tr>
<td>20 - 30</td>
<td>500</td>
</tr>
<tr>
<td>30 - 40</td>
<td>400</td>
</tr>
<tr>
<td>40 - 50</td>
<td>300</td>
</tr>
<tr>
<td>More than 50</td>
<td>200</td>
</tr>
</tbody>
</table>

Dust collected with an Owens jet dust counter is counted in a light field with an immersion objective, numerical aperture 1.15 - 1.30, the total magnification under the microscope being between 700 and 1,500. When for the average free-silica content of representative samples of the strata traversed the prescribed maximum concentration is below 500 part/cm³, account is taken both of the particles in the intake air and those liberated by the work being controlled.
Austria

Assessment of dust concentrations in mines is made by using a formula:

\[ x = \frac{T \cdot G}{100} \cdot k \]  

(134)

where:

- \( T \) is the number of particles per cm\(^3\) of air collected with the Standard Sartorius H.S. konimeter and counted in a light field at a magnification of x 1,080;
- \( G \) is a factor depending on the composition and calculated from the results of analyses of dust collected with the Göthe filter (cf the danger factor of the Silicosis Research Institute of Bochum explained under Germany, Federal Republic); and
- \( k \) is a factor for the konimeter.

Dust concentrations are classified as follows:
- \( x \) less than 100 = sub-critical zone
- \( x \) more than 100 and less than 150 = critical zone
- \( x \) more than 150 = supercritical zone.

Belgium

Routine control of dust concentrations in coal mines is provided for in the legislation (13). Gravimetric samples are taken on Soxhlet filters without elutriation at 15 - 20 metres from the face in the return airway and the aspiration velocity is kept approximately equal to the velocity of the ventilation air. The samples collected (coal + rock) are analysed to determine the ash content. A diagram (fig. 48) based on the recommendations of the Dust Institute of the Netherlands mines is used to classify workplaces as follows:

- Class I: slightly dusty
- Class II: moderately dusty
- Class III: dusty
- (Class IV): very dusty.

The Mining Hygiene Institute has also proposed thresholds (55) (135) that are given by a "danger index" and are calculated from the formula: \( i = 3.32 \log C \cdot t - 9.3 \) which is derived from the French dust index referred to below. In this formula:

- \( C \) is the number of particles between 5 and 0.5 micron per cm\(^3\) of air collected by a standard thermal precipitator (four samplings an hour) and counted at a magnification of x 1,000 by means of a microprojector equipped with an immersion objective with numerical aperture 1.3 and a resolving power of about 0.2 micron; aggregates are disintegrated in counting; and
- \( t \) is the quartz content of these particles as determined by the method of coloration with immersion in phase contrast over a dark background.
Fig. 48.- Classification of mine dust levels as a function of the total gravimetric concentration and of the quartz content.

1. Stone dust per cent. 2. Total concentration mg/m$^3$. 
This formula and the diagram that expresses it (fig. 49) are obviously only valid for the sampling and examination methods.

Fig. 49. — Classification of mine dust as a function of particle concentration and quartz content.

1. Thousands of particles/cm³, 5 – 0.5 micron. 2. Per cent quartz.
recommended by the Mining Hygiene Institute. An atmosphere is the more dangerous the higher its index; to limit the mass of almost pure coal dust that should not be inhaled quartz contents below 1.25 per cent are considered to be that percentage.

Four classes of environment are proposed, the threshold index being \( i = 5 \). To quote an example, for a quartz content of 5 per cent by counting, \( i = 3.5 \), and \( i = 5 \) for a concentration of 1,500 or 4,000 part/cm\(^3\) from 5 to 0.5 micron (aggregates disintegrated).

**Canada**

In metal mines routine measurements are made with the konimeter (136). The dust is counted with a microscope over a dark background at a magnification of \( x \) 150, the slides having first been heated to 575°C both before and after treatment with hydrochloric acid (HCl 50 per cent) for two minutes. Dust concentrations are assessed as follows:

- less than 300 part/cm\(^3\) : good
- 500 - 700 (or 800) part/cm\(^3\) : fair only
- over 1,000 part/cm\(^3\) : bad

In the Quebec asbestos mines, examinations are made with a microprojector over a light background at a magnification of \( x \) 1,000.

**Czechoslovakia**

In addition to screening and precipitation liquids, extraction cartridges of the Soxhlet thimble type are used for sampling, at least in the Rosice coalfield (145); the threshold values adopted are 30 mg/m\(^3\) for inert dust (fine dust); 10 mg/m\(^3\) for dust with a low quartz content; and 2 mg/m\(^3\) for dust with a high quartz content.

**France**

Since 1956 (139) regulations have required a dust index to be established for each workplace. The index is:

\[
I = 3.32 \log C.t - k, \quad (4)
\]

where:

- \( C \) is the number of particles below 5 microns per cm\(^3\) of air counted with a microscope having a resolving power of 0.5 micron and a minimum magnification of \( x \) 200 (generally \( x \) 600);
- \( t \) is the percentage content by weight of free crystalline silica as determined by X-ray diffraction of dust below 5 microns from a sample taken specially for this purpose; and
- \( k \) is a constant depending on the sampling and examination methods adopted.

This constant \( k \) is:

- 10.6 when the sample is taken on a soluble filter and examined by an accepted method (Le Bouchet or Nord and Pas-de-Calais mines); or
- 8.9 when the sample is taken on a cellulose membrane and counted in accordance with the CERCHAR rules.
The index figure determines the frequency of medical examinations. Dust concentrations with an index of 5 or less are considered to be satisfactory; those with an index between 5 and 6 to be doubtful; and those with an index above 6 to be dangerous.

Calculation of the index is facilitated by the use of diagrams similar to that shown in fig. 49.

Germany, Democratic Republic

In the mines of Saxony dust measurements appear to be made exclusively with the konimeter (132). The particles are counted under a microscope at a magnification of x300 before and after incineration at 550°C for two hours. Workplaces are classified according to the number of particles below 5 microns or the ash content. There are four classes:

- Part/cm³: 0 - 150; 150 - 500; 500 - 1000; over 1000
- Ash percentage: 0 - 10; 10 - 20; 20 - 50; over 50.

Germany, Federal Republic

Assessment of the danger of a mine atmosphere is based on measurement of the concentration by weight (fine dust only) and the application of "danger factors" for the different ingredients of the dust examined. To indicate the relative importance of these ingredients the Silicosis Research Institute at Bochum has assigned them the factors shown in the following table.

<table>
<thead>
<tr>
<th>Substance</th>
<th>Mineral Factor</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quartz</td>
<td>1.0</td>
</tr>
<tr>
<td>Feldspars and micas (more than 25 per cent quartz)</td>
<td>0.7</td>
</tr>
<tr>
<td>(sericite) less than 25 per cent quartz</td>
<td>0.5</td>
</tr>
<tr>
<td>Clayey ingredients</td>
<td>0.2</td>
</tr>
<tr>
<td>Ores, coals, carbonates</td>
<td>0.1</td>
</tr>
<tr>
<td>Calcium carbonate, gypsum</td>
<td>0.0</td>
</tr>
</tbody>
</table>

Initially designed to assess the danger of a rock by mineralogical analysis of drillings below 42 microns (129), the method has been adapted for fine airborne dust collected in large samples on filters or membranes; the concentration is measured with a tyndalloscope after proper calibration.

A dust found by analysis to consist of 20 per cent quartz, 60 per cent sericite and 20 per cent clayey minerals would be assigned an index number equal to

\[(20 \times 1) + (60 \times 0.3) + (20 \times 0.2) = 42\]

The quantity of fine dust found (mg/m³) multiplied by the index
number calculated as shown and divided by 10 gives a risk index number, which would be 63 for a concentration of 15 mg/m³ [15 x 4.2].

These risk index numbers are used to classify atmospheres in four categories (130):

0 - 25 : harmless
25 - 50 : slightly harmful
50 - 100: increasingly harmful with increasing length of exposure
over 100: harmful.

In coal mines more especially, the regulations of the mining authorities require the use of the tyndalloscope and the Bergbau konimeter (131). Konimeter samples serve to determine the percentage of stone or ash after incineration at 550°C. Densitometric examinations (see preceding section) replace counting under the microscope at a magnification of x 360 for particles between 1 and 5 microns, and photographic examination by comparison with standard films. For a given ash content, tyndalloscope measurements (formerly assimilated to gravimetric measurements expressed in mg/m³) are read by comparing the angular values with a reference table. The diagram in fig. 50 represents the classification of dusty atmospheres. There are four grades, as follows:

I : Slightly dusty
II : Moderately dusty
III : Highly dusty
IV : Very highly dusty.

Since 1958 tyndalloscope measurements have had to be supplemented by determinations of quartz contents of dusts, but the systematic application of the regulations has been delayed pending the production of a filter for the selective sampling of fine dust. Amendments have recently been approved by the mining authorities (November 1965) but they do not affect the principle of the system described above.

Italy

According to a study published in 1960 on dust concentrations in a siderite mine (140) dust sampled with the thermal precipitator or cellulose membranes is examined in a light field at a minimum magnification of x 200 and sometimes x 400 in routine operations and counted between 0.7 and 5 microns. It seems that the maximum permissible concentrations, which vary with the quartz content of the particles, are similar to the American maxima allowed before 1962. However at a magnification of x 200 with numerical aperture 0.45 of the objective, a content exceeding 500 part/cm³ of any dust is considered dangerous, and if there are more than 50 particles of quartz, very dangerous.
Fig. 50. - Classification of working places according to
Tyndalloscope reading and
stone dust level.

Japan

The commonest sampling instrument is the Riken konimeter, similar to the Owens jet counter of the Funkenmeter. The particles in the range 5 - 0.5 micron are counted under a microscope in a light field at a magnification of x 400. With this method (141) the maximum permissible concentrations are:

400 part/cm$^3$ or 8 mg/m$^3$ for more than 10 per cent silica
1,000 part/cm$^3$ or 20 mg/m$^3$ for less than 10 per cent silica.

Netherlands

In 1959 the Dust Institute of the Netherlands mines proposed an index based on gravimetric measurements and expressed in effective milligrams; it is the amount of dust below 5 microns expressed in mg/m$^3$ and calculated by multiplying the weights of the various ingredients by a factor of 5 for quartz, 3 for other minerals and 1 for coal.

A dust cloud containing $X$ mg/m$^3$ of quartz dust, $Y$ mg/m$^3$ of dust of other minerals and $Z$ mg/m$^3$ of coal dust (particles under 5 microns) has a dust index equal to:

$$5X + 3Y + Z$$

effective mg per m$^3$ air.

By means of this index four categories of dusty atmospheres have been defined (142):

I : Non-dusty atmosphere : less than 15 eff. mg/m$^3$
II : Slightly dusty atmosphere : 15 - 30 eff. mg/m$^3$
III : Dusty atmosphere : 30 - 45 eff. mg/m$^3$
IV : Very dusty atmosphere : more than 45 eff. mg/m$^3$

In the light of experience in Dutch Limburg this arrangement has been replaced by a simpler one consisting in taking gross samples without elutriation and replacing the four category ranges by the values 10 - 15 and 22 mg/m$^3$ of mixed rock and coal dust, the ash obtained by incineration being counted as rock dust. To take account of the fact that coal dust imposes an additional burden on the lungs, the original maxima have been reduced by half for ash contents of 10 per cent (that is coal contents of 90 per cent). The categories resulting from simple gravimetric measurements are shown in fig. 48, where the curve separating categories II and III indicates the maximum concentration.

South Africa

For very many years, several decades in fact, routine measurements of dust concentrations have been made with the Witwatersrand konimeter. The slides after being heated to 550°C and immersed in a solution of hydrochloric acid to remove solid or liquid organic substances and insoluble salts are counted under the microscope in a dark field at a magnification of x 150. The maximum permissible concentration fixed by the mining authorities is between 200 and 300 (usually 250) part/cm$^3$ of pure silica under 5 microns. In the
metal mines the standard thermal precipitator is also used; the slides are counted under the microscope in a light field at a magnification of $\times 1,000$ before and after the treatment just described. With this procedure the maximum permissible concentration has not been fixed officially.

In the coal mines the modified thermal precipitator is used (60); the slides are treated at 220°C and examined photo-electrically (98). The galvanometer reading ($L$) enables the atmosphere under examination to be assigned to one of three categories:

- $L$ less than 100: fairly good
- $L$ more than 100, less than 250: average
- $L$ more than 250: fairly bad.

After incineration of the sample at 220°C, the value $L = 100$ corresponds to a concentration of 500 part/cm³ between 1 and 5 microns as measured with the thermal precipitator (128).

**Sweden**

In the majority of cases routine measurements in metal mines are made with the Witwatersrand of the Sartorius H.S. konimeter. The standards in force in 1961 do not appear to have been uniform. Concentrations below 400 part/cm³ counted in a dark field appear to have been acceptable (144). In some places a maximum of 200 part/cm³ has been adopted for ores with a very high quartz content, and 500 part/cm³ for ores with 20 - 30 per cent quartz. Particles are counted in a light field at a magnification of $\times 160$.

**Switzerland**

For dust concentrations of particles below 10 microns with a quartz content averaging about 40 per cent, the silicosis risk is assessed as follows (145):

<table>
<thead>
<tr>
<th>Concentration (mg/m³ less than 10 microns)</th>
<th>Assessment</th>
</tr>
</thead>
<tbody>
<tr>
<td>More than 100</td>
<td>Serious silicosis after a few years</td>
</tr>
<tr>
<td>100 - 20</td>
<td>Risk of silicosis</td>
</tr>
<tr>
<td>20 - 10</td>
<td>Silicosis after long exposure (ten years)</td>
</tr>
<tr>
<td>10 - 5</td>
<td>Doubtful</td>
</tr>
<tr>
<td>Less than 5</td>
<td>Atmosphere probably harmless</td>
</tr>
</tbody>
</table>

**U.S.S.R.**

Maximum permissible concentrations are expressed as weights of fine dust, probably under 5 microns. 2 mg/m³ is mentioned for
particles containing over 10 per cent of quartz, and mg/m$^3$ for those with less (147). A publication dated 1960 gives the following particulars (148):

<table>
<thead>
<tr>
<th>Mineral and Organic Dust</th>
<th>Maximum Permissible Concentration mg/m$^3$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Over 70 per cent crystalline silica</td>
<td>1</td>
</tr>
<tr>
<td>10 - 70 per cent free silica</td>
<td>2</td>
</tr>
<tr>
<td>Silicate dust with less than 10 per cent free silica</td>
<td>4</td>
</tr>
<tr>
<td>Other mineral dust with less than 10 per cent free silica</td>
<td>5</td>
</tr>
<tr>
<td>Minerals and mixtures with no silica</td>
<td>6</td>
</tr>
<tr>
<td>Coals with more than 10 per cent free silica</td>
<td>2</td>
</tr>
<tr>
<td>Coals with less than 10 per cent free silica</td>
<td>4</td>
</tr>
<tr>
<td>Coals with no silica</td>
<td>10</td>
</tr>
</tbody>
</table>

United Kingdom

On 1 October 1965 the National Coal Board, after consultation with all the parties interested - Ministry of Fuel and Power, Mines Department, trade unions, medical associations, etc. - adopted new criteria and new methods for assessing dust conditions (143).

The sampling instrument is the Casella long-running thermal precipitator, type 112 A; sampling begins with the arrival of the miners at their workplace and ends with their departure.

Counting of particles under the microscope is confined to the size range 1 - 5 microns irrespective of the nature of the dust. To take the nature of the ingredients into account indirectly, "approved" and "non-approved" airborne dust conditions vary with the type of work and the mining Division.

The average concentration for a shift may not exceed 250 part/cm$^3$ in stone drifts and roads through old workings in any Division. For other conditions the concentration must be less than:

- 700 part/cm$^3$ in any Division, except the South Western;
- 500 part/cm$^3$ in the South Western Division, except in anthracite mines;
- 400 part/cm$^3$ in the anthracite mines of the South Western Division (that is, South Wales, Forest of Dean and Somerset).

United States

The many separate investigations undertaken in the different States have led to a diversity of recommendations. For some 30 years however observance of one rule has been found to be good practice:
"When the total dust concentration (in millions of particles per cubic foot) is multiplied by the free-silica content (expressed as a percentage) the concentration should be considered too high if the product exceeds 5,000,000" (137). However, this rule is not applied to concentrations with a low content of free silica (less than 5 per cent SiO₂). Also samples must have been taken with the midget impinger and examined in a light field at a magnification of x 100.

For over 20 years the Committee on Threshold Limit Values of the American Conference of Governmental Industrial Hygienists has regularly published a list of maximum permissible concentrations that is only applicable if the samples have been taken and examined by the methods just described. Before 1962 the thresholds, for mineral dusts were as follows:

Free silica more than 50 per cent: 5 m.p.p.c. or 175 part/cm³
Free silica 5 - 50 per cent: 20 m.p.p.c. or 700 part/cm³
Free silica less than 5 per cent: 50 m.p.p.c. or 1750 part/cm³
Asbestos: 5 m.p.p.c. or 175 part/cm³
Mica: 20 m.p.p.c. or 700 part/cm³
Aluminium oxide, calcite: 50 m.p.p.c. or 1750 part/cm³

In 1962 the Committee adopted new thresholds for "mineral and non-metallic inorganic dusts" (138); fig. 51 is a graphical representation of the formula proposed:

\[
G = \frac{250}{Q + 5} \times 10^6 \text{ particles per cubic foot.}
\]

Fig. 51.- Dust limits for inorganic, mineral and non-metallic dusts as proposed by the Committee on Threshold Limit Values (1964).

1. Particles per cubic foot x 10⁶. 2. Per cent, quartz by weight.
B. Comparison of Maximum Permissible Dust Concentrations

The figures quoted in the preceding section reflect the position taken at a given moment by authorities responsible for dust prevention, but it is evident that in most countries institutions both official and private are continuing their investigations into dust problems.

On the international scale investigations into the comparison of standards or criteria for the assessment of dust concentrations in mines are nevertheless confined to some very special cases. In fact to the difficulties described in connection with the comparison of sampling instruments and methods of examination must be added another: the researcher who compares his own method with foreign methods operates in seam conditions and with ingredients and particle-size distributions that are not necessarily the same as those for which the foreign methods were designed. But as is well known, the results of dust counts depend on the size distribution of the airborne particles, and irrespective of the collection efficiency of the instruments used, the results of counts in a given particle-size range depend on the numerical aperture of the objective and not the total magnification of the microscope used.

It would thus seem more profitable to compare gravimetric methods, which at first sight are more reproducible. It should however be noticed that gravimetric standards do not always apply to particles of the same dimensions (all sizes either less than 10 microns, less than 5 microns or less than 7 or 8 microns) and that the weight of dust collected is combined in different ways with the nature of the ingredients.

(1) The criteria for assessment adopted in the mines of the Federal Republic of Germany (risk index numbers applicable to all mines) when compared with the results of gross gravimetric measures made in the Netherlands coal mines reveal some agreement between the two classifications used (127). In fact, if certain hypotheses concerning the proportion of particles less than 8 and less than 10 microns are accepted, the risk index numbers for concentrations considered "only slightly harmful" correspond to the index numbers of the Netherlands Dust Institute for "slightly dusty atmospheres".

(2) The concentrations measured by tyndalloscope in the German coal mines and the dust risk index numbers calculated from them represent Class II atmospheres of the Bonn Divisional Mining Office (moderately dusty) or the "slightly harmful" atmospheres of the Silicosis Research Institute whose standards are somewhat stricter.

(3) U.S.S.R. and United States standards before 1962 were compared some years ago (149). The Russian maximum permissible concentrations, usually expressed as weights, were converted into numbers of particles by means of conversion factors derived in particular from the results of gravimetric and numerical measurements made in some British coal mines.
The disagreement is striking; the Russian maxima are definitely lower. But there is nothing to prove that in practice Russian requirements are stricter so long as the methods of sampling, examination and analysis are not employed simultaneously at the same place so as to take account of all factors influencing the results of the measurements.

(4) The maximum allowable numerical concentrations fixed by the Committee on Threshold Limit Values in the United States have more recently been compared with the maximum gross gravimetric concentrations recommended for the Netherlands mines and in force for the Belgian mines (150).

On the basis of more than 100 simultaneous measurements made in the Belgian coal mines with the midget impinger and the Soxhlet thimble, the maximum permissible gravimetric concentration has been calculated for each ash content measured, and the maximum permissible numerical concentration was then calculated for a given quartz content. The results of this investigation are shown graphically in fig. 52. When the quartz content of the coal dust is between 10 and 15 per cent of the ash content, as is the case in the coal mines of Dutch Limburg, the two proposed curves for the maxima have the same form.

(5) There is no a priori reason why there should not be a relationship between the mode of expressing dust concentrations in terms of weight and that expressing concentrations in terms of numbers, if it is assumed that there is a fixed relationship between the ash and quartz contents, and that there is a log-normal distribution law governing the number of particles, and if certain precautions are taken in sampling, the most important being to avoid collecting all the coarse particles in the immediate vicinity of the dust source.

Comparative study of samples taken simultaneously with the thermal precipitator (method of the Mining Hygiene Institute) and the Soxhlet thimble during 144 shifts in the underground workings of Belgian coal mines showed a relationship between gravimetric and numerical methods of estimating concentrations. It was found that classifications of concentrations based on:

- the total weight of dust and the weight of its ash content; and
- the number of particles between 0.5 and 5 microns and the number of quartz particles

are equivalent in 85 per cent of cases when the ash content is between 5 and 75 per cent and in 90 per cent when the ash content is less than 30 per cent (151). This equivalence means that dust concentrations in categories I and II or III and IV in fig. 52 have on the average a risk index IHM above or below 5 (135), that is to say, on the average they are considered equally good or bad, acceptable or unacceptable, whether one adopts either classification or applies the American standards with due regard to the hypotheses referred to under point 4.
Fig. 52.—Gravimetric dust limits adopted by Belgian-Netherlands coal mines compared with American standards.

1. Total dust content after ignition (per cent).  2. Total concentration (mg/m$^3$).  3. Quartz content as percentage of total ash content.
C. Proposals for the Adoption of a Criterion of Reference

The adoption of a standard method of assessment would presuppose the existence of a method that is definitely better than the others, or unanimously recognised as such, is usable in all mines, and with an adequate factor of safety protects the health of underground personnel. Medico-occupational investigations into dust concentrations as related to the work and medical history of miners are still in progress in France, South Africa, the United Kingdom, etc. In Belgium and the Federal Republic of Germany investigations of this kind are carried on with the help of the High Authority of the European Coal and Steel Community. The results point to the possibility of fixing maximum permissible concentrations that are both technically and medically valid.

The evolution of pneumoconiosis depends first and foremost on the cumulative effect of exposure; as exposure increases the pneumoconiotic lesions become more frequent and more serious. It even appears that the mass of coal dust is more important than its stone or ash content (152). There is also a relationship between the onset or the evolution of silicosis and exposure to dust expressed as a risk index number (135).

Nevertheless, in spite of the analogy between the assessment criteria that is observed when the few considerations set out in the preceding paragraph are closely examined, the validity of the different methods of assessment is questionable from the international standpoint because the national investigations referred to have been confined to the coal deposits of Western Europe.

It would thus seem both logical and wise to abandon for the moment the very idea of a standard method of assessment. But it is important to choose a reference method, that is to say, a method that is not troublesome, is easy to apply, and would allow everybody to continue their investigations in co-ordination without any change in their methods of operation.

Such a simple method that could be applied generally at small cost, should nevertheless respect the concept of harmfulness of the atmosphere accepted by most of the researchers in the field in question. Now, gross gravimetric sampling in which all the particles of both coal and rock are collected more or less isokinetically, when supplemented by determination of the rock or ash content, gives a routine measurement, an assessment of the dust concentration that in the majority of cases corresponds fairly well with that derived from other criteria, whether these are based on tyndalloscope measurements, on gravimetric measurements of fine dust, or even on numerical measurements (risk index, dust index or American standards).

Thus, as a first step towards the standardisation of methods of sampling dust and assessing the pneumoconiosis risk, the adoption of gross gravimetric sampling appears to be indispensable.
It will probably be objected that this is taking a step back­wards. We do not think so: gross gravimetric results which are very bad where there is no prevention should stimulate those concerned to take remedial action more vigorously. Moreover it will very quickly be noticed that particles above 10 microns will become fewer and fewer as action against dust becomes more systematic.

The method of gross gravimetric sampling is unquestionably the simplest, and when necessary it readily permits the determination of the mineral ingredients and the fractionating of the samples taken. This will be a matter for consideration when the theoretical and practical cut-off curves for gravimetric instruments equipped with elutriators have been definitely accepted.

The simple gravimetric method could thus be used in the different countries at the same time as the usual method of each. This arrangement would automatically provide opportunities of establishing a relationship between the different assessment criteria when measured against one and the same reference, and of finding out whether the atmospheres judged to be good, doubtful or bad by some are judged in the same way by others in all conditions of work and dustiness.
Notwithstanding the simplicity of the principle of measurements sampling methods should be discussed and precisely specified so as to ensure the reproducibility and the comparability of the determinations; this applies for instance to the site and duration of sampling, the minimum amount of air to be aspirated, accuracy of weighings, identification of principle ingredients, and temperature and duration of incineration.

Purely for purposes of information we describe below the method of operation imposed by the Belgian regulations in October 1965 (13).

"Sec. 1. The classification of workplaces and workshops that are in operation in the underground workings of coal mines shall take into account the dust content of their atmosphere (gravimetric concentration) and the ash content of the dust (percentage by weight).

Sec. 2. The ranges of the different classes are shown in the following table:

<table>
<thead>
<tr>
<th>Ash content of dust Percentage</th>
<th>10</th>
<th>20</th>
<th>30</th>
<th>40</th>
<th>50</th>
<th>60</th>
<th>70</th>
<th>80</th>
<th>90</th>
<th>100</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dust concentration mg/m³</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Class I</td>
<td>50</td>
<td>37</td>
<td>29</td>
<td>24</td>
<td>20</td>
<td>17</td>
<td>15</td>
<td>13</td>
<td>11.5</td>
<td>10</td>
</tr>
<tr>
<td>Class II</td>
<td>85</td>
<td>58</td>
<td>43</td>
<td>35</td>
<td>29</td>
<td>25</td>
<td>22</td>
<td>19</td>
<td>17</td>
<td>15</td>
</tr>
<tr>
<td>Class III</td>
<td>110</td>
<td>88</td>
<td>68</td>
<td>55</td>
<td>45</td>
<td>37</td>
<td>33</td>
<td>28</td>
<td>25</td>
<td>22</td>
</tr>
</tbody>
</table>

For an ash content between two magnitudes indicated in the table the dust concentration is found by linear interpolation.

The class of a workplace or workshop may also be determined by marking on the diagram (in fig. 48) the dust concentration of its atmosphere in mg/m³ and the ash content of the dust as a percentage.

Sampling

Sec. 3. A sample shall be taken by passing the air whose dust concentration is to be determined through a filter of a special approved type known as the Soxhlet thimble at the rate of at least 1000 litres an hour.

The filter shall be placed in a holder, and the air shall be made to enter it without changing direction by means of a nozzle without appreciably altering its velocity. The total volume of air traversing the filter shall be determined either by maintaining the
flow constant for the entire duration of sampling and measuring both the flow and the duration, or by passing the air through a gas meter.

The sampling period shall overlap for at least two hours the working period of the workplace or the workshop so as to ensure the collection of enough dust to enable the atmosphere to be classified. It shall be interrupted during meal breaks and all stoppages of work.

In productive workings sampling shall be done during the coal-getting shift, and if appropriate during stowing and caving, along the axis of the return airway of each of the faces being worked, at a distance of 15 - 20 m from the face, and at face height subject to a minimum of 25 cm from the roof.

In workings ventilated by ducts, sampling shall be done during operations likely to produce and liberate dust, in particular coal-getting, drilling and loading out. Samples shall be taken along the axis of the road at mid-height, ten metres behind the end of the duct if the face is ventilated by a forcing duct, and at the mouth of the duct if it is an exhaust duct.

Calculation of Dust Concentration

Sec. 4. Before and after sampling, the filter shall be placed in an open holder and dried for at least two hours, until the weight remains constant in an oven that is automatically kept at a temperature of ± 85°C. The holder shall then be closed and cooled during 30 minutes under a dryer until the temperature of the laboratory is reached. After loosening the stopper for a short time, the filter shall be weighed with the holder stoppered and then the empty holder shall be weighed.

The difference between the weights of the filter before and after sampling gives the quantity of dust collected, which shall be expressed in milligrams.

The dust concentration shall be calculated from the quantity of dust and the volume of air that has passed through the filter, and shall be expressed in milligrams per cubic metre (mg/m³).

Calculation of Ash Content

Sec. 5. The dust from the filter, dried as indicated above, shall be collected in a porcelain dish. After being weighed, the dish with the dust shall be placed in an oven at 600°C.

The temperature shall be raised to 700°C in half an hour and kept at this level for half an hour. The dish shall be allowed to cool to 600°C in the oven and then to the temperature of the laboratory under a dryer.

The dish shall be weighed and the operations shall be begun again to show whether combustion was complete.

The weight of ash as finally obtained and expressed as a percentage of the weight of the sample, gives the ash content of the dust."
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