

THE PERFORMANCE AND  
IMPROVEMENT OF  
DUST SAMPLING INSTRUMENTS

BY

D. G. BEADLE

---



THE PERFORMANCE AND IMPROVEMENT

OF

DUST SAMPLING INSTRUMENTS.

---

Thesis submitted for the degree of  
Master of Science at Rhodes University

by

D.G. BEADLE, B.Sc.

May, 1954

THE PERFORMANCE AND IMPROVEMENT OF  
DUST SAMPLING INSTRUMENTS.

<u>Contents</u>	<u>Page</u>
Acknowledgements.	1
Summary.	3
1. Introduction.	6
2. The optimum sampling period.	36
3. Investigations of the performance of the konimeter.	45
4. An investigation of the thermal precipitator.	68
5. A modified form of thermal precipitator.	89
6. The examination of dust samples under the microscope.	100
7. A photo-electric method of assessing dust samples.	124
References.	144

ACKNOWLEDGEMENTS.

The investigations described here were undertaken for the Transvaal Chamber of Mines. The author wishes to express his appreciation to the Chamber of Mines for permission to use the results for this thesis.

The author worked originally under the direction of Mr. H.S. Patterson and latterly under Mr. J.P. Rees. Their guidance and advice has always been of great assistance.

Sections 2, 3, 6 and 7 are substantially the author's own work. The only assistance received was that of various laboratory assistants making routine observations under the author's direction. Mr. John Talbot carried out the research on the electron microscope described in section 6.6, working under the author's supervision; he took the electron micrograph in Figure XXIV.

Section 4 describes work carried out in collaboration with Mr. J.E. Kerrich, Consultant Statistician to the Chamber of Mines Research Laboratories. The author planned and supervised the work of obtaining the basic ob-

servations required; Mr. Kerrich analysed the results and suggested further experiments necessary to elucidate obscure points. The full statistical analysis made by Mr. Kerrich has not been quoted here.

The development of the modified thermal precipitator described in Section 5 was undertaken in collaboration with a colleague Mr. P.H. Kitto; the work was shared equally between the collaborators.

SUMMARY

The design of accurate dust sampling instruments for use in mines should logically depend on the process by which dust causes silicosis, the extent to which dust particles of different sizes are retained in the lungs, and on various physical properties of the dust. These subjects are reviewed in the Introduction, which concludes with a history of dust sampling on Witwatersrand gold mines and a description of the two instruments in common use - the konimeter and the thermal precipitator.

The period of time over which dust samples should be taken is discussed in Section 2. Experiments are quoted which lead to the conclusion that 10 minutes is a suitable sampling time. The very short sampling time of the konimeter - approximately  $\frac{1}{4}$  second - is criticised.

Investigations of the performance of the konimeter are described in Section 3. The konimeter is shown to have a low sampling efficiency for high concentrations of fine dust, but to overestimate low concentrations

of coarse dust. The need for a more accurate instrument is indicated.

Although the thermal precipitator has been proved to collect practically all the dust particles in the air sampled, down to the limits of visibility of the electron microscope, errors arise in estimating the dust concentration with it. The sources and magnitude of these errors are discussed in Section 4, and a single thermal precipitator measurement is shown to have a standard error of approximately 13%.

The standard form of thermal precipitator is not suitable for routine dust sampling in mines. A modified thermal precipitator, which overcomes most of the disadvantages of the standard form and which has other advantages, is described in Section 5.

Section 6 describes experiments made to improve the microscope technique at present used for counting dust samples. These experiments have resulted in finer dust particles being made visible. A high contrast photomicrographic method which reveals still smaller particles is described, and recent preliminary

studies of Witwatersrand gold mine dusts with an electron microscope are mentioned.

A photo-electric method of assessing dust samples is described in Section 7. This method is shown to possess advantages of speed in measurement, to eliminate the human factors and statistical errors inherent in the microscope method, and to measure a property of the dust, related to the surface area, which is probably a better measure of the danger to health than the number concentration.

## 1. INTRODUCTION

Dust is a serious hazard in mining because it causes silicosis. Dust sampling plays an important part in dust control and many dust-sampling instruments have been developed.

The South African gold mining industry has for many years taken a leading part in the world-wide efforts to eliminate silicosis. It was the first large industry in the world to introduce systematic dust sampling on a significant scale, and has always been active in testing existing instruments, and developing new types, for this work. This thesis describes some of these researches in which the author has taken part.

Before describing this work, information must be given on aspects of the silicosis problem on which the theory of dust sampling necessarily depends.

### 1.1 Theories of silicosis.

Silicosis is a disease of the lungs caused by the inhalation of silica dust. Although recognition of the disease dates back

many centuries<sup>1</sup>, the action by which the dust produces the characteristic fibrotic changes in the lung is not, even now, fully understood.

For a long time it was thought that the dust particles damaged the lungs by their mechanical action of scratching or irritating the tissues. This theory was disproved by several investigations in the period 1922 to 1932, notably by Gye and Purdy<sup>2</sup>, Gye and Kettle<sup>3</sup>, Gardner<sup>4</sup> and Kettle<sup>5</sup>. Their experiments showed, inter alia, that -

- (a) Dusts of extremely hard material such as diamond and carborundum, when introduced into the lungs of animals, failed to produce the typical fibrosis caused by silica.
- (b) Silica particles coated with an insoluble layer of iron oxide, too thin to alter their shape, failed to produce the reactions caused by uncoated particles.
- (c) For a given weight of material, small particles caused more damage than large particles.

These experiments indicated that silica might be harmful to the lungs, not

because it was hard and sharp, but because it dissolved in the lung fluids to produce a substance which was toxic to the tissues. This substance is probably silicic acid.

This "solubility theory" of silicosis was critically reviewed by King<sup>6</sup>, who has since extended his work and produced further evidence in support of the theory. King and his colleagues have shown that, in general, the more soluble a silicious dust is in fluids similar to those in the lung, the more fibrogenetic it is, but there are anomalies. Thus certain silicious substances, although comparatively soluble, are found to have a low toxicity. Also it has long been known that extremely fine silica particles, about 20 Angstrom Units diameter, which are highly soluble, do not cause a fibrosis<sup>6</sup>, although they can produce immediate death from shock when injected into animals<sup>7</sup>. King makes a clear distinction between this acutely toxic "pharmacological" effect of very fine silica particles, and the slowly appearing fibrogenetic effect of large, more slowly soluble silica particles. There is another form of rapidly soluble silica which does not produce

fibrosis. Particles subjected to prolonged grinding develop a highly soluble amorphous layer - the so-called Beilby layer<sup>8</sup>. Experiments with silica particles have shown that the presence of this layer, despite its high solubility, does not increase the fibrogenetic properties of the dust<sup>9</sup>.

King has recently summarized his views on these apparent anomalies in the solubility theory of silicosis. He says<sup>7</sup> -

"The formation of silicotic nodules is a slow chronic phenomenon. It appears that to produce this, silica particles must be small enough to liberate slowly and over a long time soluble silica into solution from their surfaces in sufficient amount to produce a chronic local irritative effect; they must not be so small that they will quickly and completely dissolve and either be carried away or produce only an acute local effect which will lack the continuous stimulus of constantly released colloidal silicic acid. It may be this prolonged irritative or stimulating effect of constantly released colloidal silica which is responsible

for the formation of the fibrous scar tissue of a silicotic nodule."

Similarly Nagelschmidt<sup>10</sup> expressed the view that "if the solubility is too high ..... the material is dissolved and eliminated too fast to produce fibrosis." He considered that below a size in the region of 0.1 micron diameter, the fibrogenetic properties of dust would decrease.

Holt and Osborne<sup>11, 12</sup> have given a feasible explanation of some of these anomalies. Accepting that solution of the silica is the first essential step in the production of silicosis, they produce evidence supporting their theory that high polymerisation of the silicic acid molecules is an essential second step. Polymerisation of silicic acid is most rapid at a pH of 5.5-6.0, decreasing sharply on either side of this value. When quartz dissolves the initial pH is low (about 2) and to reach the pH of the lung fluids (7.4) the solution has to pass through this zone of rapid polymerisation when large polymers are formed and, for this reason, according to their theory, quartz has its well known high toxicity. Cement on the

other hand does not cause fibrosis even when breathed in high concentrations, although it also produces silicic acid. Since it is alkaline it does not pass through the zone of rapid polymerisation, and hence the large polymers required for the production of fibrous tissue are not formed. Similarly it might be that in the case of the small, very rapidly soluble, silica particles and the amorphous Beilby layer, the rate of solution is so rapid that there is insufficient time for the production of large polymers.

King evidently regards his term "colloidal silica" in the quotation above as synonymous with "polymerised silicic acid."

Many other theories of silicosis have been suggested. For example Evans<sup>13</sup> suggested that the piezo-electric properties of quartz made it harmful, and Heffernan<sup>14</sup> attributed the fibrotic action to unsatisfied valencies on the surface of freshly fractured quartz. Jones<sup>15</sup> claimed that the mineral sericite, often present in quartzitic rocks, was responsible for the tissue damage. These, and many other theories, have not withstood critical examina-

tion, and the solubility theory remains that most commonly accepted by the majority of workers in the field of silicosis.

### 1.2 The rate of solution of silica particles.

Large particles of most substances dissolve at a rate proportional to the surface area exposed to the solvent. When however, the particle size decreases to diameters of the order of one micron, many substances show an increased rate of solution per unit surface area. This effect has been studied in the case of silica by Kitto and Patterson<sup>16</sup>. They showed that silica particles below one micron diameter (which are relatively numerous in mine dusts) had a rate of solution per unit surface area considerably greater than that of larger particles. The rate of solution is further increased if the particles have sharp spikes, many edges, or numerous cracks on their surface. Particles produced by mining processes are likely to show these features.

Kitto and Patterson also discussed the increased solubility of the amorphous Beilby layer on fine particles produced by any grinding

process. Such particles show a high initial rate of solution, but when this amorphous layer has dissolved, the rate of solution decreases sharply.

### 1.3 The dust particles retained in the lung.

Only the dust particles which are deposited in the terminal sacs of the lung, the alveoli, and which remain there, can produce silicosis. Studies have been made in two principal ways of the sizes of particles thus retained.

The first method is to extract the mineral particles from the lungs of deceased silicotics, and to measure the sizes of these particles under the microscope. McCrae<sup>17</sup> used this technique and was the first to draw attention to the large number of particles of diameter one micron and less in the lungs of deceased Witwatersrand miners. His work was extended by the present author<sup>18</sup> using superior microscope equipment and technique to show that as still smaller particles were made visible, further large numbers of particles were found in such lungs. Recent studies with the

electron microscope<sup>19</sup> have shown that silico-tic lungs contain enormous numbers of particles below the limit of visibility of the optical microscope.

The second method is to measure the concentration of particles of different sizes in inspired and expired air, and from this determine the percentage retention of particles of different sizes. van Wijk and Patterson<sup>20</sup>, working with dusts in Witwatersrand gold mines, measured the overall retention in the whole respiratory system in this way. Hatch and Hemeon<sup>21</sup> used their results to calculate the actual retention in the alveoli. Other measurements have since been made of the overall retention in the whole respiratory system<sup>22, 23</sup>. The literature has been reviewed by Davies<sup>24</sup>.

More recently the percentage of particles of different sizes deposited in the human lung has been measured directly. Wilson and La Mer<sup>25</sup> used particles labelled with a radioactive tracer. The subject breathed a known dose of particles of a uniform size, and with Geiger counters placed against the chest wall, the amount retained in the lungs could be

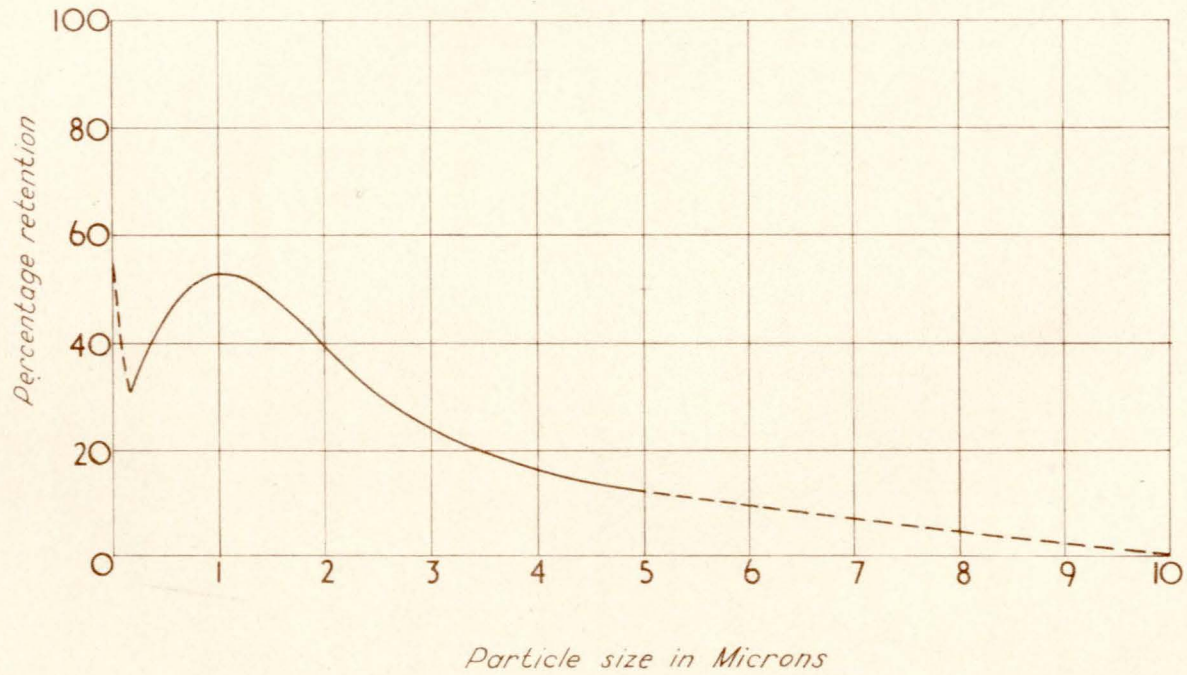
determined. Brown et al<sup>26</sup> measured the concentration of dust in the inspired air and divided the expired air into successive portions; they could identify the part of the respiratory system from which each portion of the expired air came by determining the CO<sub>2</sub> content. From these measurements they were able to make an estimate of the alveolar retention of each particle size.

The problem was investigated theoretically by Findeisen<sup>27</sup>. He took into account the velocity of air in different parts of the respiratory system, the momentum of particles of different sizes, the time the air remained in the lungs, the rates of settling of particles under gravity, and the effects of diffusion and Brownian motion.

There is reasonable agreement between the results of these various experiments, providing corrections are made for variations in the breathing patterns of the subjects, and for the density of the different dusts used. Most of the experiments have been confined to particles in the size range 0.2 to 5 microns dia-

FIGURE I

LUNG RETENTION CURVE



meter; it is desirable to extrapolate on each size of this range. There is general agreement that silica particles larger than about 10 microns diameter do not reach the alveoli<sup>28</sup> since the upper parts of the respiratory system have efficient protective devices against large particles. There have been no published experiments showing the percentage retention of particles below about 0.2 microns diameter, but the retention is likely to be greater than for 0.2 micron particles due to Brownian motion becoming appreciable. In a private communication Hatch has stated that he has some experimental evidence of this.

Based on the results quoted above, the full line in Figure I indicates what may be accepted as the best measure at present available of the alveolar retention of silica particles in the range 0.2 to 5 microns diameter. The broken lines are extrapolations based on the statements above.

The two basically different methods of determining alveolar retention of particles of different sizes can be shown to give results

which are mutually consistent. Table I gives a typical size distribution of dusts in Witwatersrand gold mines - it is based on several hundred samples. Using the percentage retentions shown in Figure I, the number of particles (per 100 breathed) retained in the lung is calculated. This is then reduced to a percentage size distribution, and is compared with the results obtained by the author by direct measurement<sup>18</sup>.

The agreement is reasonable, but rather more large particles were found in the lung than would be expected. This may be due to some of the smaller particles coagulating, either after deposition in the lung or during the process of extracting the dust from the lung. It is not easy to distinguish, under the microscope, aggregates of fine particles from single discrete particles in the size range studied. Complete or partial solution of the particles may also affect the size distribution of the dust found in the lungs, but there is insufficient quantitative data available to assess this effect.

Dust particles deposited in the lung are also affected by phagocytosis. This is a

TABLE I.

Particle diameter (microns)	Percentage size distribution in mine air	Percentage retention in lung	Number of particles retained in lung	Percentage size distribution of particles retained in lung.	
				Calculated	Observed
0.15	40	40	16.0	42	48
0.25	20	30	6.0	16	18
0.4	10	40	4.0	11	12
0.8	7	50	3.5	9	5
1.2	5	53	2.7	7	3
1.6	4	46	1.8	5	3
2.0	3	39	1.2	3	2
2.5	3	31	0.93	2.5	2
3.0	2	23	0.46	1	2
4.0	2	18	0.36	1	2
5.0 and over	4	14	0.56	1.5	3

protective mechanism of the lung in which wandering scavenger cells, called phagocytes, ingest foreign particles, such as dust, entering the lung and transport them into the special draining mechanism, known as the lymphatics. The phagocytes, however, appear to become overwhelmed when the dust load is heavy and this mechanism fails. The selective removal of particles of different sizes by the phagocytes is a subject apparently not yet investigated by experimental pathologists, and its significance therefore cannot be assessed.

#### 1.4 The properties of dust which should be measured.

There are three physical parameters which can conveniently be used to express the 'amount' of dust per unit volume of air, namely the number, the surface area and the mass of particles. The size distribution of the dusts found in mines varies considerably and hence there cannot be a fixed relationship between measurements made in these different ways. The parameter measured should obviously be that which is most closely related to the object of

the sampling; in mines this is the determination of the possible danger to health of the dust in the place sampled.

Some evidence on this subject is available as a result of pathological experiments on animals, in which dust of known size and concentration is injected into, or inhaled by, the animals. Although the extrapolation of these results to the human being may be open to some uncertainty, it remains the only direct way in which the pathogenicity of dusts can be determined. It must also be noted here that many of the experimenters on this subject do not report their results in a form in which their statistical significance can be tested. This is an unsatisfactory aspect of much of the pathological work on silicosis. The experiments of King and his colleagues, quoted below, are an exception to this criticism.

The mass concentration is not a sound measure of the silicosis risk associated with a dust. In typical mine dusts the mass concentration is controlled principally by the number of large particles, say 10 microns diameter

and larger, but practically none of these can enter the alveoli. Even if a sampling device is used which collects only the particles able to reach the alveoli there is evidence that the degree of fibrosis produced is not proportional to the mass of dust. Thus Tebbens et al<sup>29</sup> treated different animals with equal masses of dust of different particle sizes, and found that considerably more tissue damage was caused by the finer particles than by the coarser particles. King et al<sup>30</sup> have recently produced similar evidence. Mass concentration should therefore not be used to measure the danger to health of silica dust.

Tebbens and his colleagues reported that the damage caused by a given mass of dust was inversely proportional to the particle size, but did not discuss this point further.

Suppose one group of animals is treated with X particles of size A and another group with Y particles of size B, and that the mass of particles in each case is equal, i.e. that

$X A^3 = Y B^3$  (density and shape factors being similar for each dust). According to

Tebbens the tissue damage in the first group will be proportional to  $\frac{1}{A}$  and in the second group proportional to  $\frac{1}{B}$ . That is, X particles of size A cause  $\frac{B}{A}$  times the damage caused by Y particles of size B.

Presumably this implies that X particles of size A will cause the same damage as  $\frac{B}{A}Y$  particles of size B. This latter dose of particles will have a surface area proportional to  $\frac{B}{A}Y B^2$ , which equals  $XA^2$ , since  $XA^3 = YB^3$ . Therefore equal tissue damage results from equal surface area of particles, irrespective of particle size.

Recently King et al<sup>30</sup> have studied the fibrogenetic properties of dusts of various sizes. Their results show that the toxicity of a silicious dust is much more closely related to the surface area of the particles than it is to the mass or number of particles. They also showed in experiments using equal surface areas of dusts of different sizes that the maximum fibrosis was produced by particles in the size range 1 to 2 microns diameter; particles below 0.5 microns diameter appeared to be signifi-

cantly less toxic. The increased toxicity of particles in the range 1 to 2 microns conforms with their increased solubility per unit surface area; the decreased toxicity below 0.5 microns can be explained by Holt and Osborne's theory, and confirmation of decrease in toxicity with further decrease in particle size is provided by the non-fibrogenetic particles of 20 Angstrom unit diameter.

These results, and the solubility theory of silicosis, clearly indicate that surface area is likely to be soundest measurement to make to determine the danger to health of a dust. A measuring technique which over-estimates the surface area of the particles in the region of 1 to 2 microns, and under-estimates the surface area of the particles below 0.5 microns diameter would presumably provide a better measurement of the danger to health than a direct measurement of the surface area. In the present state of knowledge it is not however possible to put this desired over-estimation and under-estimation on a quantitative basis.

There is an additional reason for underestimating the surface area of the finer particles. Small, rapidly soluble particles, must eventually dissolve completely in the lung, and cannot then exert any fibrogenetic effect. It has often been argued that they should not be included in dust measurements for this reason, and in Great Britain all particles below 0.5 microns diameter are ignored in normal dust measurements.

For many years most countries, including South Africa, have used the number concentration as a measure of dustiness. There has, however, never been any published evidence that a given number of particles of one size will produce the same amount of tissue damage as an equal number of particles of another size. In King's experiments discussed above  $135 \times 10^9$  particles below 0.5 microns diameter caused appreciably less fibrosis than  $10 \times 10^9$  particles in the range 1 to 2 microns diameter. The choice of number concentration as the property to be measured was probably based, in the

past , more on expedient than on theory; that is, on what could be measured conveniently, rather than on what should be measured.

Some evidence of the soundness of using surface area instead of number count can be obtained by comparing the incidence of silicosis in different occupations in Witwatersrand gold mines with average dust measurements associated with those occupations.

Due to miners tending to follow a variety of underground occupations during their careers it is possible to give only an approximate value for the silicosis risk of a given occupation. In Table II the rates of four main occupations are given; a positive sign indicates an occupation in which the silicosis rate is higher than average, and vice versa. The dust concentrations were measured with the thermal precipitator, and the surface area obtained by calculation from the number count and size distribution.

The correlation between surface area and silicosis rate is better than the correlation between number concentration and silicosis

rate.

TABLE II

Occupation	Relative Silicosis rate	Average dust concentrations.	
		Particles per c.c.	Square microns per c.c.
A	+ 0.9	1960	780
B	+ 0.6	840	410
C	+ 0.1	510	320
D	- 1.7	690	210

The suggestion of adopting surface area as the best parameter for the measurement of dust samples was put forward by the author<sup>31</sup> and several overseas workers<sup>32, 33, 34</sup> at the International Conference on Dust at Geneva in December 1952.

Measurement of the surface area of the particles in a dust sample to assess the danger to health should include only those particles which can reach the alveoli. Inclusion of larger particles will invalidate the measurement due to the large surface area of these particles. A sampling device which collects particles in proportion to their lung retention is a desirable improvement on instruments

which collect all particles irrespective of size.

Since, at present, dust measurements are still generally made on the basis of number concentration all results in this thesis will be expressed in such units, namely the number of particles per cubic centimetre (p.p.c.c.)

The chemical composition of the dust breathed is a matter of considerable importance in silicosis studies, but as it does not materially affect the design of instruments used to measure the amount of dust, it falls outside the scope of the present work.

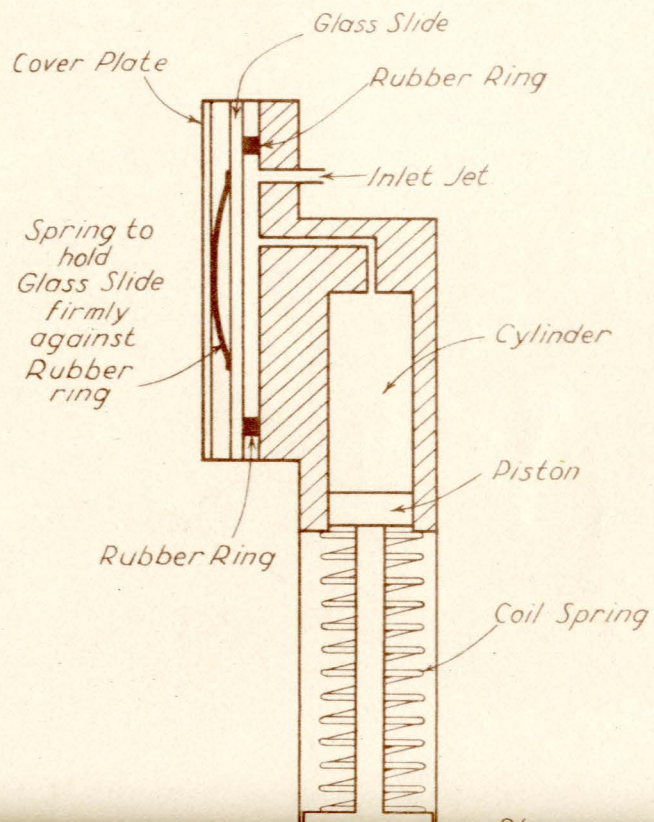
#### 1.5 The history of dust sampling on Witwatersrand gold mines.

Dust sampling started in 1902. The first dust samples were taken by drawing the dust-laden air through cotton-wool, which was subsequently ignited and the remaining dust then weighed<sup>35</sup>.

In 1907 the "sugar-tube" method of dust sampling was first suggested and later came into general use<sup>35</sup>. The apparatus con-

FIGURE II

THE KONIMETER



sisted of a hand-operated pump which sucked a known volume of air through a glass tube containing crystals of sugar. On return to the laboratory the sugar was dissolved in water, the solution filtered, the filter paper incinerated and the dust weighed. The results were vitiated by the enormous contribution of the large particles to the total measurement. It was early realised that such measurements were probably of limited value, and this led to the development of the konimeter, a device for measuring the number concentration of dust particles<sup>36</sup>. For many years both the sugar-tube and konimeter were used together in Witwatersrand gold mines, until the sugar-tube method was finally abandoned in 1938.

Figure II illustrates diagrammatically the construction of the konimeter. The spring-driven piston sucks a known volume of air (usually 5 ccs) through a fine jet (0.5 mm. diameter) at a high velocity, which exceeds, at its peak, 100 metres/second. The sampling period is about 0.2 to 0.3 seconds. A glass collecting slide is placed about 0.5 mm. from

the outlet of the jet; this slide is moved after each sample is taken so that a number of samples can be collected without the necessity of removing the slide from the instrument. The dust in the air impinges onto the slide, and to increase the number of particles adhering to the slide, the latter is coated with a thin adhesive film, usually vaseline.

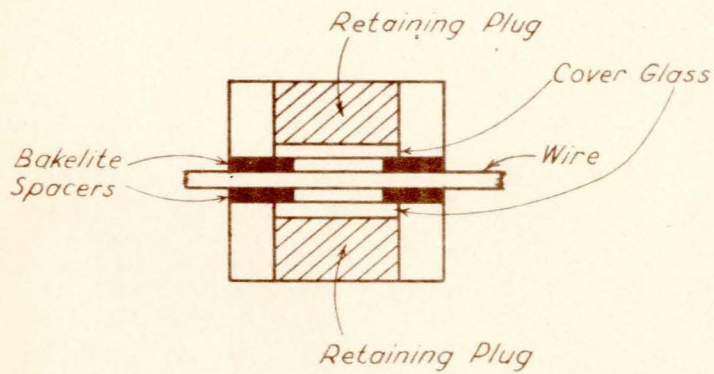
After the samples have been taken the glass slide is removed from the konimeter and heated to about  $550^{\circ}\text{C}$  to remove any carbon particles and organic matter. It is then treated in hot dilute HCl to remove any soluble salts derived from the water used for dust suppression.

Each sample is examined in turn under a low power microscope, total magnification about  $\times 150$ , with dark field illumination. Using an eyepiece graticule, the number of particles visible in one-tenth of the whole sample is counted. A simple calculation gives the number concentration of the dust in the air sampled.

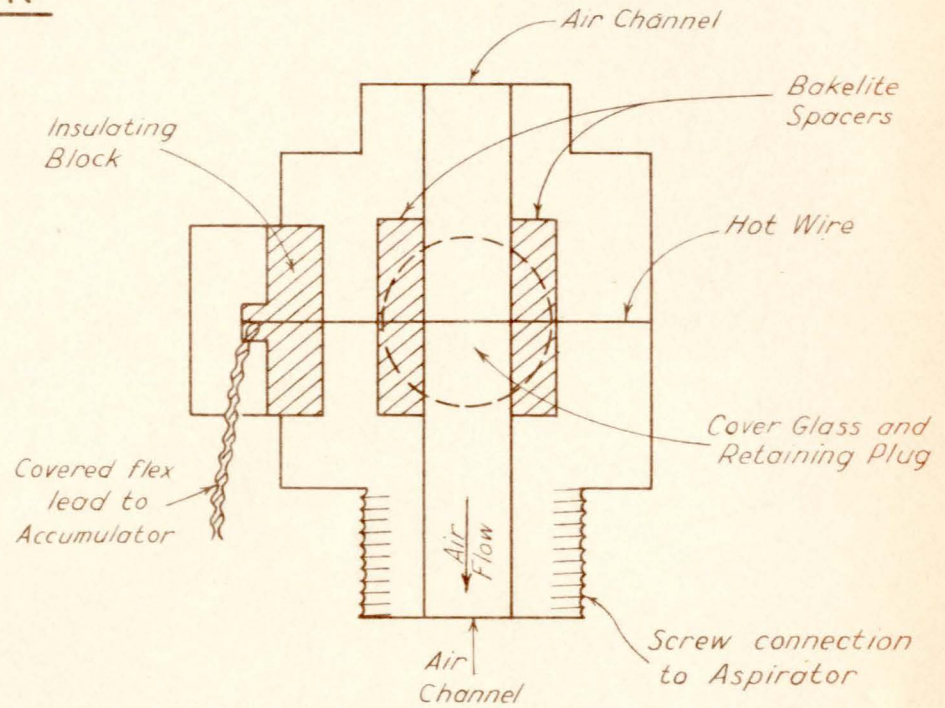
In 1934 the British Government after

FIGURE III

# THE THERMAL PRECIPITATOR



PLAN VIEW



SECTIONAL VIEW

examining all existing types of dust sampling instruments, including the konimeter, and finding that none of them were suitable and accurate enough for precise industrial use, developed the thermal precipitator<sup>37, 38</sup>. This is still the most accurate instrument available for industrial dust sampling. The thermal precipitator was introduced into Witwatersrand gold mines in 1935 by H.S. Patterson, who has described his experiences with it<sup>39</sup>.

The thermal precipitator works on the principle, first noted by Aitken<sup>40</sup>, that surrounding any hot body there is a zone into which dust cannot enter. The size of this dust-free zone depends on the temperature difference between the hot body and its surroundings. In the thermal precipitator the hot body is an electrically heated thin wire, suitably supported at each end. Microscope cover glasses are placed on each side of the wire, parallel to each other, so that the wire is midway between them and along a diameter. The cover glasses are held a fixed distance away from the wire but intersect the dust-free space around it.

Figure III shows the principal details of construction.

A water aspirator is attached to the sampling head and the dust-laden air is drawn between the cover glasses past the wire; the dust cannot penetrate the dust-free zone and is therefore deposited on the cover glasses as a strip slightly above the wire. The sample is about 1 cm. long and 1mm. wide. The instrument samples continuously for any desired period. The cover glasses are changed after each sample, and before examination under the microscope they are ignited to remove carbon particles and organic matter; they can also be treated to remove acid-soluble salts.

The dust deposit is examined under a high power microscope using a 2mm. apochromatic objective with light field illumination. The total magnification is x1500. The number of particles in a known fraction of the whole sample is counted, and the particles are also matched for size against a scale in a special eyepiece graticule<sup>41, 42</sup>. Knowing the volume of air sampled and the fraction of the sample

examined, the dust concentration in the air is calculated.

The thermal precipitator has been an invaluable instrument for research work ever since it was introduced in South Africa, but has not proved suitable for the routine dust sampling carried out by the dust inspectors attached to each gold mine. An ideal instrument for this work should -

- (a) Take a sample of the dust which will give an accurate measure of its danger to health.
- (b) Be easily portable (preferably pocket-size), and robust.
- (c) Have a self-contained power supply and not require external supplies such as compressed air or electric mains.
- (d) Be simple to use, dependable, and require a minimum of care and maintenance.
- (e) Take the sample over a period long enough to give a representative sample, but not so long that the operator cannot sample the requisite number of working places in a day.

(f) Collect the dust in a form in which it can conveniently be treated to remove various contaminants, and in which it can easily be assessed for its danger to health.

(g) Enable the measurement of the dust to be made as soon as possible after the sample is collected.

No instrument at present available fulfils all these requirements satisfactorily. For example the konimeter does not satisfy requirements (a) or (e), (see sections 2 and 3 of this thesis) while the thermal precipitator, in its standard form, does not satisfy (b) or (d).

Many other instruments have been tested under local conditions but have been found unsuitable. These are described briefly, and reasons given for their rejection, in a paper by the author and a colleague<sup>43</sup> - a copy is attached as Appendix I, see pages 285 - 287.

Section 5 of this thesis describes a modified form of thermal precipitator which overcomes the objections to the standard form.

The present position in regard to dust

sampling on Witwatersrand gold mines is that the konimeter, despite its known limitations, remains the instrument used for routine dust sampling, the standard form of thermal precipitator is used only for research work, but the modified form may later replace the konimeter for routine dust sampling. The amount of dust is at present expressed in terms of number concentration, obtained by counting the number of dust particles under the microscope, but there are sound reasons for adopting surface area as a basis for assessing the danger to health. The photo-electric apparatus described in Section 7 measures basically the surface area of the particles in the dust samples collected by the modified thermal precipitator (which collects particles approximately in proportion to their retention in the human lung), but with the desired over-estimation of particles in the region of 0.5 to 2 microns diameter, and under-estimation of the particles below 0.5 microns.

These new methods of collecting and assessing dust samples are more convenient to use than those at present in use, and provide

more accurate measurements of the danger to health of dust than has previously been practicable.

---

## 2. THE OPTIMUM SAMPLING PERIOD

### 2.1 The Problem.

There are two main ways in which the results obtained from dust-sampling can be considered -

- (a) The result gives the average dust concentration existing during the sampling period.
- (b) The result gives an indication of the average dust concentration likely to exist in that place over a longer period than that covered by the actual sample. The period to which the result is extrapolated may be a few hours, or a complete working shift, or even a number of shifts.

In research work the first is the only justifiable use of the data, and the sampling period will depend on the nature of the experiment. However in routine dust sampling work some extrapolation as suggested in the second interpretation is unavoidable. A dust inspector during a visit underground must sample a number of working places, and in each place must make a number of other observations in

addition to taking the dust samples. The time available for dust sampling is thus limited. The measurement of the dust during this visit has to be taken as representative of the conditions at other times. Obviously the sampling period should be as long as possible to reduce the errors of this extrapolation.

This section deals with the problem of selecting the optimum sampling period for the konimeter and the thermal precipitator.

## 2.2 Observations with the konimeter.

The konimeter takes a sample in about  $\frac{1}{2}$  second. Dust conditions in a mine normally vary rapidly with time and a single konimeter sample may not represent at all accurately the average dust concentration. The effective sampling period can be extended by taking a number of samples to obtain an average and, in general, the larger the number of samples the more reliable the average, but the labour involved in counting the samples under the microscope becomes considerable. In practice the number of samples taken in one place is usually limited to three or four, taken at intervals

ranging from a few seconds up to a minute.

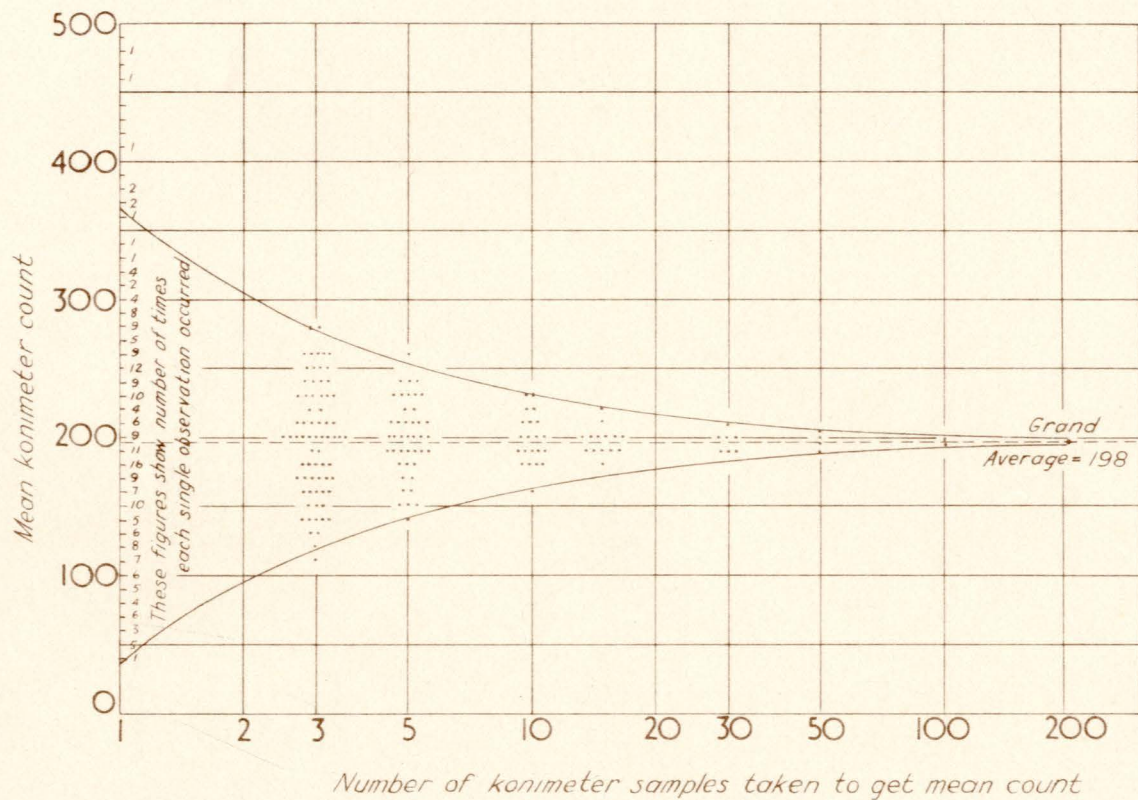
In the following discussion the means of different numbers of konimeter counts are compared with the "grand average" over a period - this "grand average" refers only to the average as shown by the konimeter; it is not an accurate measure of the actual dust in the air, as will be shown in section 3.

Five experiments were made in each of which a large number of konimeter samples (from 187 to 320) were taken at 10 second intervals. Normal work continued throughout the period of sampling and a dust inspector would have been justified in taking samples in that place at any time during the experiment to determine the dust conditions. Full experimental details, and the individual results have been published<sup>44</sup> - a copy is attached as Appendix II, see pages 266 - 268.

In one of the experiments, the grand average of 210 samples was 198 p.p.c.c. with a standard error of  $\pm 6$ . The individual observations were analysed to show the results which would have been obtained if various lesser

FIGURE IV

MEANS OF VARIOUS NUMBERS  
OF KONIMETER SAMPLES



numbers of samples had been taken to determine the average concentration during the period of the experiment; thus 70 results were available of the mean of 3 samples; 21 results of the mean of 10 samples and so on. The results of this analysis are shown graphically in Fig.IV. The standard deviation has been calculated for each set of results, and the lines drawn on the graph enclose values lying within twice this deviation from the mean. In accordance with standard statistical practice, this can be interpreted as meaning that if a large number of observations had been available for any given number of samples, 95% of the observations would fall between these two lines.

Fig.IV indicates that the mean of only 3 samples might, on occasion, give results up to 45% different from the grand average. Assuming that errors of  $\pm 20\%$  (in 95% of the observations) are acceptable in routine dust sampling, these results indicate that not less than 8 samples should be taken to give an average within this limit of error.

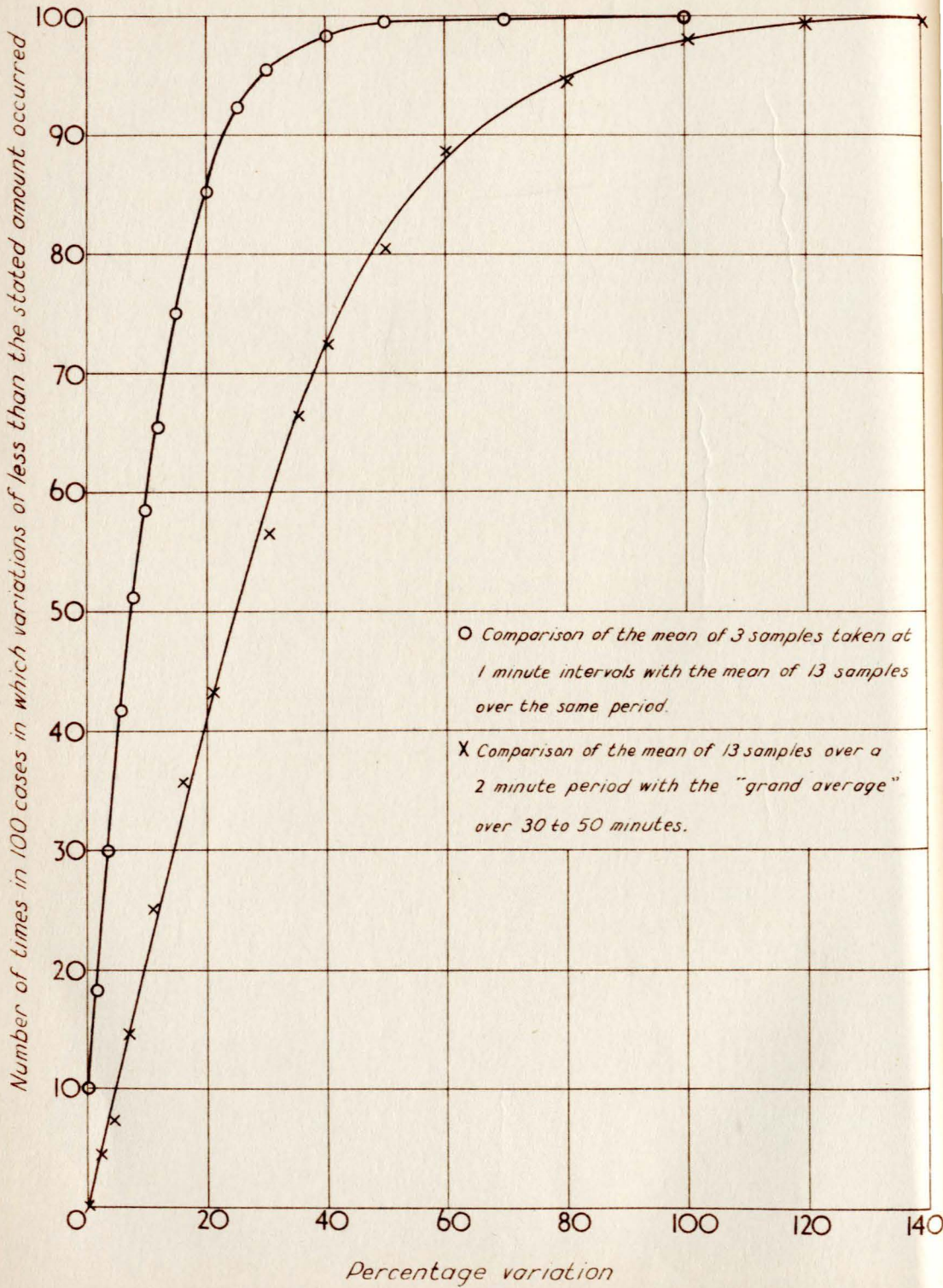
This experiment was made in a working

place where dust conditions were, by mining standards, comparatively steady, In another experiment of this type where the dust conditions were more variable, a similar analysis indicated that not less than 15 samples should be taken to ensure that the error of their mean did not exceed  $\pm 20\%$ . The mean of only three samples gave results up to 50% too low and 100% too high.

The data has also been analysed to determine how accurately the mean of three samples spaced one minute apart represents the average over the two minute period covered by the samples. The mean of the thirteen samples taken during each 2 minute period has been taken as a measure of the average dust concentration during the period, and this has been compared with the mean of the 1st, 7th and 13th samples in the group, i.e. with the mean of three samples taken at one minute intervals. The percentage variation between the two measures has been calculated for each two minute period in the five basic experiments. The number of times this percentage variation falls

FIGURE V

VARIATION OF MEAN KONIMETER  
COUNT FROM THE AVERAGE



within given limits is plotted as one curve in Fig. V. This indicates that in most cases three samples taken at minute intervals will give a reasonably accurate estimate of the average concentration over that period of 2 minutes.

However even an accurate measurement of the average dust concentration over 2 minutes (i.e. the mean of 13 samples) does not give a good estimate of the average conditions likely to exist in the working place over a longer period, say half-an-hour. The second curve in Fig. V shows the number of times the average dust concentration over two minutes varies by different amounts from the grand average over half-an-hour. Large errors frequently occur if this extrapolation is made, and the errors are likely to be larger if the results are taken to represent conditions over, say, a whole shift.

These observations with the konimeter lead to the conclusions that -

- (a) Three konimeter samples at 1 minute intervals give a reasonably accurate estimate of the average dust concentra-

tion during the 2 minute period covered by the samples.

- (b) A measurement of the mean dust concentration over a 2 minute period cannot be extrapolated to predict with reasonable accuracy, the average over half-an-hour, or longer.
- (c) The mean of three samples, spread over 30 minutes, does not agree satisfactorily with the grand average over that period.
- (d) The number of samples necessary to give a mean in reasonable agreement with the grand average over half-an-hour depends on the relative steadiness of the dust conditions. With "steady" conditions 8 samples were sufficient; with "variable" conditions 15 samples were required.

To take this number of samples as routine at each working place, would result in an excessive amount of microscope work. The need for so many samples to attain reasonable accuracy is due largely to the very short sampling time of the konimeter.

### 2.3 Observations with the thermal precipitator.

The thermal precipitator has the great advantage compared with the konimeter, of sampling continuously over any desired period.

Six experiments were made in each of which 24 successive 5 minute samples were taken with the thermal precipitator. During the period of two hours covered by each experiment the same work continued in the working place. Full experimental details and a typical set of results have been published<sup>43</sup> (Paper attached as Appendix I. - see pages 290 - 292).

The various measurements over 5 minute periods were combined to show the averages which would have been obtained had samples been taken over other longer periods, namely 10, 15, 20, 30, 40 and 60 minutes.

The percentage standard deviations of the observations for each period of time were calculated. The average deviations for the six experiments are given in Table III.

TABLE III.

<u>Sampling Period in Minutes.</u>	<u>Average Percentage Standard Deviation.</u>
5	24.4
10	18.5
15	17.3
20	15.8
30	14.8
40	13.8
60	12.0

A sampling time of 5 minutes gives results which have a variation larger than is desirable. The variation with a sampling time of 10 minutes is probably acceptable for routine dust sampling work, and the slight improvement with longer sampling times does not appear to warrant the extra time involved.

---

### 3. INVESTIGATIONS OF THE PERFORMANCE OF THE KONIMETER

The proportion of the total dust in the air collected by the konimeter has been a subject of controversy ever since it was first introduced.

Green<sup>37, 38</sup> is the only worker who appears to have compared the konimeter with accurate laboratory dust sampling devices. He found that the results obtained with the konimeter varied with the concentration and size of particles in the dust clouds sampled. This probably explains the widely differing results obtained by other workers.<sup>45, 46, 47, 48, 49</sup>

Section 3.1 below deals with an experimental investigation of the performance of the konimeter when used to sample typical dust clouds in Witwatersrand gold mines and Section 3.2 describes the application of certain theoretical studies to explain some of the results found.

#### 3.1 An experimental investigation.

The thermal precipitator was accepted as a satisfactory standard against which to

measure the performance of the konimeter.

The same konimeter (number H24) and thermal precipitator (Number 441) were used throughout the tests. They were in good working order at all times; each was checked against other instruments of the same type by taking simultaneous adjacent sets of samples. The results of these preliminary tests are given in Tables IV and V respectively.

TABLE IV.

Test Number K1	Konimeter H24	Konimeter 12	Konimeter H22
Number of samples taken	31	31	29
Average count, p.p.c.c.	72	62	75
Test Number K2			
Number of samples taken	29	29	28
Average count, p.p.c.c.	620	570	710

These results indicate that Konimeter H24 is an average instrument.

TABLE V.

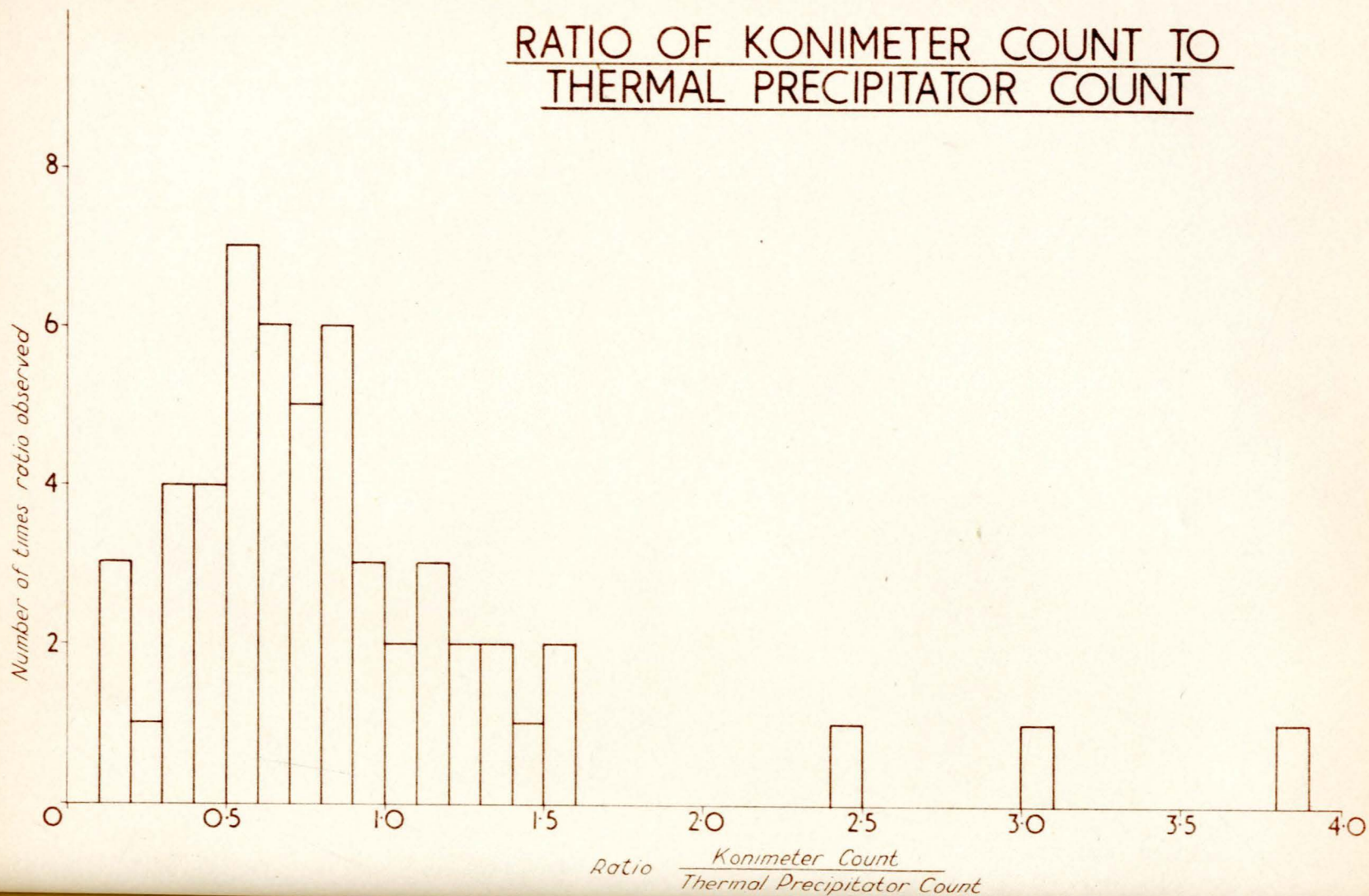
	Thermal Precipitator No. 441	Thermal Precipitator No. 439	Thermal Precipitator No. 431
Test Number TP1			
After ignition, before acid treatment.	394	416	431
After acid treatment.	246	298	300
Test Number TP2			
After ignition, before acid treatment.	342	368	334
After acid treatment.	193	254	192

The counts before acid treatment indicate that there is no appreciable difference between the three thermal precipitators. There is evidence however that the effect of acid treatment is not consistent, and this point is dealt with later.

54 experiments were made in which the average konimeter count was compared with the thermal precipitator count. These were carried out in 17 different working places in 8 mines. In each experiment a thermal precipitator sam-

FIGURE VI

RATIO OF KONIMETER COUNT TO  
THERMAL PRECIPITATOR COUNT



ple was taken over a period of approximately half-an-hour. During this period about 16 konimeter samples were taken within a few inches of the thermal precipitator head at two-minute intervals. The studies described in section 2.2 have indicated that this number of samples over this period should give a good indication of the average konimeter count. All samples were taken according to standard practice for each instrument. All samples were acid-treated before they were counted.

The ratio of the mean konimeter count to the thermal precipitator count was calculated for each experiment. The detailed results, together with a discussion of the confidence limits of the ratio, have been published<sup>44</sup> - see Appendix II, pages 272 - 275. The results are summarized in graphical form in Figure VI where the histogram shows the number of times the ratio fell between certain limits.

Possible reasons for this wide range of ratios have been investigated by analysing the results in more detail.

(a) Effect of dust concentration. The experi-

ments were divided into groups according to the dust concentration shown by the thermal precipitator. The mean ratio for each group was calculated and is shown in Table VI.

TABLE VI.

Range in dust concentration shown by thermal precipitator p.p.c.c.	Average concentration in range p.p.c.c.	Mean ratio :- $\frac{\text{Konimeter}}{\text{Thermal Precipitator}}$
0 - 150	109	1.24
151 - 300	201	0.98
301 - 600	408	0.82
601 - 1000	790	0.96
Over 1000	1860	0.46

(b) Effect of size distribution. The 'coarseness' of the dust was defined arbitrarily as the percentage of particles of size 2 microns diameter and greater in the thermal precipitator sample. The experiments were divided into groups according to the 'coarseness' of the dusts. The mean ratio for each group was calculated and is shown in Table VII.

TABLE VII.

Range in percentage of particles 2 microns and larger.	Average percentage in range.	Mean Ratio :- $\frac{\text{Konimeter}}{\text{Thermal Precipitator}}$
Less than 5	Approx. 1	0.31
5 - 14	12.0	0.84
15 - 19	17.0	0.94
20 - 30	24.7	1.45

The results in Tables VI and VII show that the dust measurement made by the konimeter depends on the concentration and size frequency of the dust. Possible reasons for this will now be discussed.

(c) Effect of microscope technique. Konimeter and thermal precipitator samples are counted by different microscope techniques. Konimeter samples are counted under low power dark field illumination, which shows particles down to about 0.20 microns diameter, providing too many particles are not present. The high power light field technique used for counting thermal precipitator samples shows particles down to about 0.13 microns diameter. The ratio of the konimeter count to thermal preci-

pitator count must obviously depend therefore on the percentage of particles in the range 0.13 to 0.20 microns. This will vary from one dust cloud to another.

The low magnification used for counting konimeter samples often makes it impossible to distinguish as individual particles those which lie close together. They are therefore counted only as one particle, whereas under higher magnification they would be counted as two or more. Also the light scattered by larger particles under dark field illumination is often sufficiently bright to mask nearby small particles. Both these factors tend to cause some particles to be missed in counting, and the effect becomes greater as the density of deposit in the sample increases. The statistical problem of the overlapping of particles collected in a small area has been investigated<sup>50</sup>.

The 2 millimeter oil immersion objective used for counting thermal precipitator samples cannot be used to count konimeter samples as the glass slide is too thick, but some estimate of the magnitude of the effects

discussed above has been obtained by counting a number of konimeter samples with a medium power (4 millimeter) objective and comparing this with the low power dark field count. The 4 millimeter objective, with light field illumination, shows particles down to approximately 0.25 microns diameter. The ratio of the counts for different densities of deposits is shown in Table VIII.

These results indicated how the "counting efficiency" of the low power technique falls off as the dust samples become more dense. This is undoubtedly part of the explanation of the decreased overall efficiency of the konimeter as higher dust concentrations are measured.

(d) Effect of acid treatment of the samples.

The acid treatment of samples is carried out to remove soluble particles which are derived from the water used for dust suppression. After acid-treatment only particles derived directly from the rock are left on the slide. Unfortunately the acid treatment process also tends to dislodge from the

TABLE VIII

Particles per c.c. shown by 4 mm. objective	0 to 100	101 to 200	201 to 300	301 to 400	401 to 500	501 to 700	701 to 900	901 to 1500	Over 1500
Ratio									
<u>Low power dark field count</u> 4 mm. light field count	2.2	2.0	1.7	1.3	1.2	1.2	0.9	0.9	0.5
Number of samples counted	14	9	15	14	9	8	9	7	5

slide a number of the rock particles, and the percentage of particles thus lost varies from one sample to another. For example there is evidence of this in the results quoted in Table V. Some of the variations in the ratio of konimeter count to thermal precipitator count are likely to arise from this factor but the effect has not been fully investigated as it is believed to be comparatively small.

(c) Effect of the collecting efficiency of the konimeter.

It might be thought that the variations between the konimeter count and the thermal precipitator count are largely due to the factors discussed in (c) and (d) above. Comparisons were therefore made under conditions where these factors did not operate. Thirteen experiments were made in each of which approximately 10 konimeter samples were taken at regular intervals during a thermal precipitator sample. The samples from both instruments were ignited only, and were counted using a 4 mm. objective with light field illuminations. The sizes of all particles were measured using an eyepiece graticule. The results have been

published in full (see page 279 of Appendix II) They are summarised below in Table IX, in which they are divided into groups according to the concentration and 'coarseness' of the dust as shown by the thermal precipitator sample. The table shows the ratio of konimeter count to thermal precipitator count; when two observations fell into one group the mean ratio is given.

These results indicate clearly how the actual proportion of particles collected by the konimeter depends on the concentration and size distribution of the dust sampled.

A study of the actual numbers of particles of various sizes collected by the two instruments gives interesting results. The calculations have been made for each of the experiments but only the two extreme cases are given in Table X.

In the latter experiment there were many particles of size 10 microns and greater in the dust.

The sampling efficiency of the thermal precipitator is not 100 percent for quartz particles greater than about 5 microns diameter,

TABLE IX.

Percentage of particles 2 microns & over	Count in p.p.c.c. shown by thermal precipitator.			
	Under 500	500-1000	1000-1500	Over 1500
Under 4			0.33	0.18
4 to 8			0.70	0.64
8 to 12	1.59	0.45		
12 to 16	1.77	0.90		
Over 16		1.80		

TABLE X.

Size of Particles, microns.	Actual number of particles per c.c. of this size shown by		Ratio <u>Konimeter</u> Thermal Precipitator
	Konimeter	Thermal Precipitator	
<u>Fine dust</u>			
< 0.4	28	1,040	.03
0.4	29	528	.05
0.8	18	187	.10
1.6	5	19	.26
2.4 and 3.2	3	12	.25
4.0 and greater	1	4	.25
Total all sizes	84	1,790	.05
<u>Coarse dust</u>			
< 0.4	332	112	3.0
0.4	350	127	2.8
0.8	314	104	3.0
1.6	175	67	2.6
2.4	114	42	2.7
3.2	66	20	3.3
4.0	39	12	3.3
5.0 and greater	68	16	4.2
Total all sizes	1,460	500	2.92

but it has been proved to be 100 percent efficient for particles smaller than this, down to the limits of visibility of the electron microscope<sup>38, 51</sup>. Yet in the second experiment quoted there were many more particles of all sizes present in the konimeter samples than in the thermal precipitator sample. This effect only occurred in the experiments in which there were considerable numbers of coarse particles in the dust cloud being sampled. When there are very few coarse particles present, as in the first experiment, the efficiency of the konimeter for the finest particles is extremely low.

An explanation of the presence of particles in the konimeter sample which do not exist in the air is that the konimeter is breaking up coarse particles, or aggregates of particles. This may be due either to a shearing action as the particles pass through the jet, or to a shattering effect as they strike the collecting plate at high velocity.

The author, working in the laboratory with specially prepared dusts, showed that

the konimeter and another instrument of the impinger type, the Owens dust counter, shattered dust particles of quartz<sup>52</sup>. Other workers have also shown that instruments of the impinger type shatter the dust particles<sup>53, 54, 55</sup>, but Green<sup>38</sup> stated that there was no evidence that particles were shattered on impact with the plate at the jet velocity he used. This velocity was 58 metres/second, and is low compared with that normally employed in the konimeter, where it is of the order of 100 metres/second. This difference may well explain his results.

It may be argued that it is correct to disrupt aggregates of particles and count them as individual particles. This is based on the assumption that similar effects may occur when aggregates are breathed into the human lung. However the velocity of impingement in the konimeter is of the order of 100 metres/second, which is many times greater than the velocity of air in the respiratory system which appears to have a maximum value of under 2 metres/second<sup>56</sup>. Aggregates are not therefore

likely to be broken up by impingement in the respiratory system to anything like the extent they are in the konimeter. They may be broken up to some extent by chemical action once they have settled on the surfaces of the respiratory system, but the larger aggregates will be collected in the nasal passages, the trachea and other upper parts of the respiratory system; here there are effective mechanisms for removing them and they cannot play a part in the production of silicosis. In physical properties such as rate of settling, Brownian motion, and removal by impingement and filtration in the defence systems of the nasal passages etc, aggregates will act as single particles of equivalent size. It appears therefore that it is more likely to be correct to count an aggregate as a single particle than as the number of individual particles forming the aggregate.

### 3.2 A theoretical investigation.

Davies and Aylward<sup>57</sup>, and Ranz and Wong<sup>58</sup> have calculated the trajectories of solid particles in the air flowing out of a jet and striking

a nearby flat plate. These results can be used to calculate the efficiency of collection of particles in a konimeter, if it is assumed that all particles striking the plate adhere to it and are counted under the microscope.

The mathematical theory in the first paper is complex and the final result is not given in a form which permits easy calculation of the collecting efficiency. The second paper however gives the result

$$\psi = \left(1.0 + \frac{0.16}{D}\right) \frac{SVD^2}{18nd} \times 10^{-8}$$

where  $\psi$  = a function related to the percentage of particles which strike the plate.

D = diameter of the dust particles in microns.

S = density of dust particles in grams/c.c.

V = velocity of air flow through the jet in cms/sec.

n = viscosity of the air in poises.

d = diameter of the jet in cms.

The relationship between  $\psi$  and the percentage of particles striking the collecting plate is given in the original paper. The re-

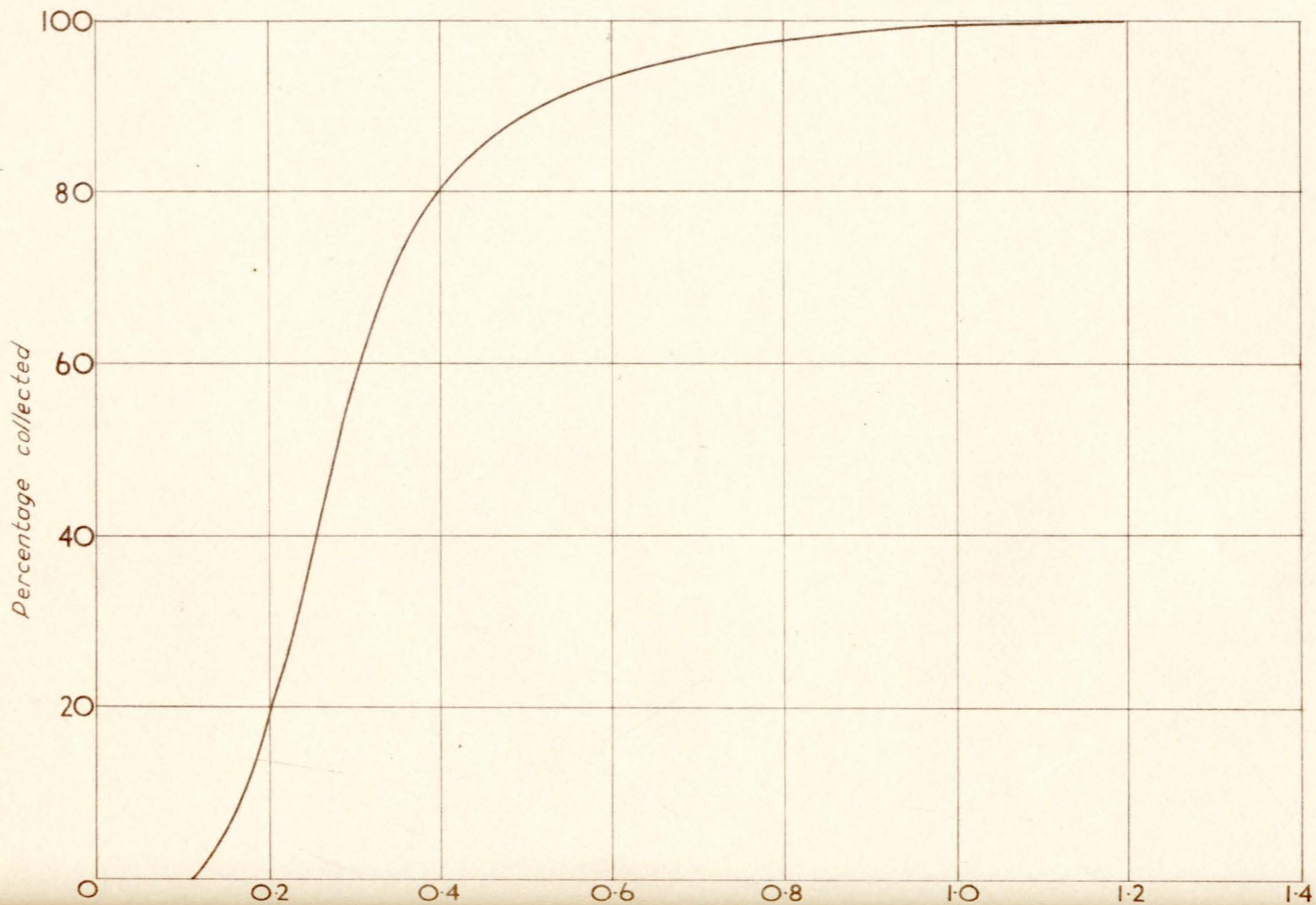
relationship was derived theoretically and confirmed experimentally by the authors. The distance of the collecting plate from the outlet of the jet is taken to be of the same order as the diameter of the jet.

V is not constant in the konimeter. Its variation with time has been studied by Rabson<sup>47</sup>. At the beginning of the piston stroke V is low, but increases rapidly and after approximately 0.05 seconds reaches a maximum value, of the order of 120 metres/second in a typical konimeter. Thereafter V dies away gradually over a period which, due to the compressibility of air, is longer than the actual time of the piston stroke. As a result, different portions of the total volume are sampled at different velocities. These proportions can be calculated from Rabson's results.

The percentage of particles of various sizes striking the collecting plate in a konimeter has been calculated for different velocities using the following values of the constants in the formula above -

FIGURE VII

PERCENTAGE OF PARTICLES OF DIFFERENT  
SIZES COLLECTED BY KONIMETER



$$S = 2.5 \text{ gms/c.c.}$$

$$n = 1.8 \times 10^{-4} \text{ poises}$$

$$d = 0.055 \text{ cms.}$$

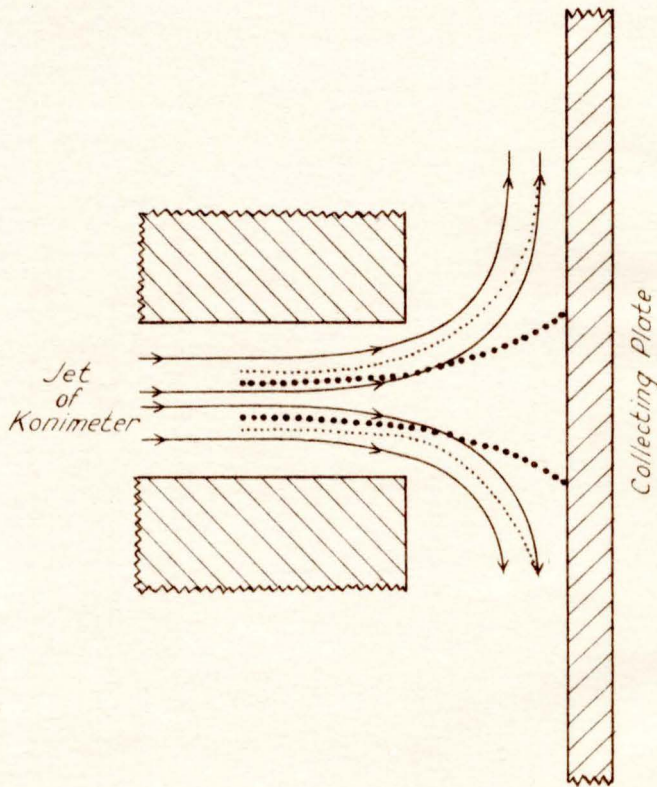
The results are given in Table XI which also shows the percentage of the total volume of air sampled at different velocities.

From this the total percentage of particles striking the plate during the whole sampling process can easily be calculated for any desired particle size. This has been done and the results are plotted in Figure VII.

The physical reason for the decrease in the percentage of particles striking the plate with decreasing size is that the smaller particles due to their very small momentum tend, after leaving the jet, to follow the line of air flow and are swept away without striking the plate. Larger particles, with their greater momentum, tend to continue to follow a straight line after leaving the jet and cross the air flow lines to reach the plate. This is illustrated in Figure VIII. The actual path of a particle of any stated size will depend on its position in the airstream when passing

FIGURE VIII

FLOW OF AIR AND DUST PARTICLES  
THROUGH KONIMETER JET



- AIR STREAMLINES
- ..... TRAJECTORY OF FINE PARTICLES
- ..... TRAJECTORY OF COARSE PARTICLES

TABLE XI.

Velocity metres/ second.	Percentage of particles of stated size striking the collecting plate.							Percentage of total volume of air sampled at this velocity
	0.15	0.20	0.25	0.30 microns	0.40	0.60	1.00	
120	13	33	63	85	100	100	100	19
100	8	28	59	80	100	100	100	16
100	8	24	54	72	98	100	100	14
90	4	19	40	63	94	100	100	11
80	2	13	33	54	89	100	100	9
70	2	8	24	40	82	100	100	8
60	0	4	13	33	72	100	100	7
50	0	2	8	24	59	99	100	6
40	0	0	4	13	40	91	100	5
30	0	0	0	4	19	72	100	3
20	0	0	0	0	4	40	99	1
10	0	0	0	0	0	4	59	1

through the jet.

The formula shows that the collecting efficiency for any particle size can be improved by increasing the velocity of impingement, but this will lead to a greater danger of breaking up the larger particles.

The actual percentage of particles collected on the konimeter slide will not necessarily agree with the above calculated values as other factors will also affect the process, for example -

- (a) Not all the particles which strike the plate will adhere to it. Hamilton et al<sup>59</sup> show that with adhesive films of the thickness used on the Witwatersrand a considerable percentage of particles rebound from the collecting slide.
- (b) Some large particles may be broken up, thus reducing the number of large particles and increasing the number of fine particles apparently collected.
- (c) The air is sucked through the sampling jet by reducing the pressure inside the konimeter; this air undergoes a sudden

expansion as it passes through the jet.

If the air contains sufficient moisture this expansion will result in water condensing on the particles, and their effective size is thereby increased, resulting in more efficient deposition. The extent to which the particles will grow in size will however depend on the amount of water available for condensation, that is, on the humidity of the air, and this introduces another variable in the performance of the konimeter. Also the growth will depend on the number of particles in the air; if the number is low the amount of water available per particle will be greater than when there are large numbers of particles present, and the particles will therefore grow to a larger size. Thus the percentage of particles collected will be greater when the dust concentration is low than when it is high.

These theoretical studies offer some explanation of the results observed in the experimental investigation of the performance of

the konimeter. Both studies indicate that when sampling a high concentration of fine dust the konimeter will have a low efficiency and the recorded results will be significantly lower than the corresponding thermal precipitator count. When sampling a low concentration of coarse particles the konimeter will appear to have a high efficiency and will sometimes give a higher count than that given by the thermal precipitator. Coarse dust is relatively harmless because it cannot reach the lungs. Results obtained with the konimeter may therefore be very misleading, and a more exact method of determining dust concentrations is desirable.

Kerrich<sup>60</sup> has criticized the konimeter from another point of view. He has shown, using data obtained in part by the author, that if two konimeters are used simultaneously, side by side, to sample dust in a mine, the results differ significantly from each other, and the ratio between the two measurements wanders in an unpredictable way.

---

#### 4. AN INVESTIGATION OF THE THERMAL PRECIPITATOR.

During the development of the thermal precipitator it was compared with the three fundamental laboratory methods of measuring dust concentrations accurately, namely the slit-ultra microscope<sup>61</sup>, Green's condensation apparatus<sup>62</sup> and a precise form of sedimentation cell<sup>63</sup>. These tests showed that the thermal precipitator deposited all the dust visible under the optical microscope below a certain size<sup>38</sup>. This size is about 5 microns in the case of silica particles<sup>64</sup>, and the slight loss of particles above this size is not important in silicosis studies. The complete deposition of finer particles is the outstanding advantage the thermal precipitator possesses over most other dust sampling instruments, and subsequent studies have shown that this sampling efficiency extends down to particles at the limit of visibility of the electron microscope<sup>51</sup>.

This however does not necessarily mean that a single thermal precipitator sample will give an accurate measurement of the dust

concentration. As in all types of measurement there will be random errors associated with the observations, and there may be mutual bias between instruments or between the observers counting the samples under the microscope. A particularly likely source of error arises from the practice of counting the particles in only a small fraction of the deposit, and assuming that this is a representative portion of the whole sample.

There appears to have been no published information dealing with this aspect of thermal precipitator sampling.

The experiments described here were therefore carried out to measure the size and frequency of the errors that occur in practice, and to try to isolate the factors which contribute to the total error.

Because dust concentrations vary over a wide range it is convenient to express the variations as percentage standard deviations. It was found that, for each given set of experimental conditions, the percentage standard deviations were roughly constant, and indepen-

COMPARISON OF INDIVIDUAL DUST COUNTS

WITH AVERAGE COUNTS

OBSERVER A

Individual Observation P.P.C.C.

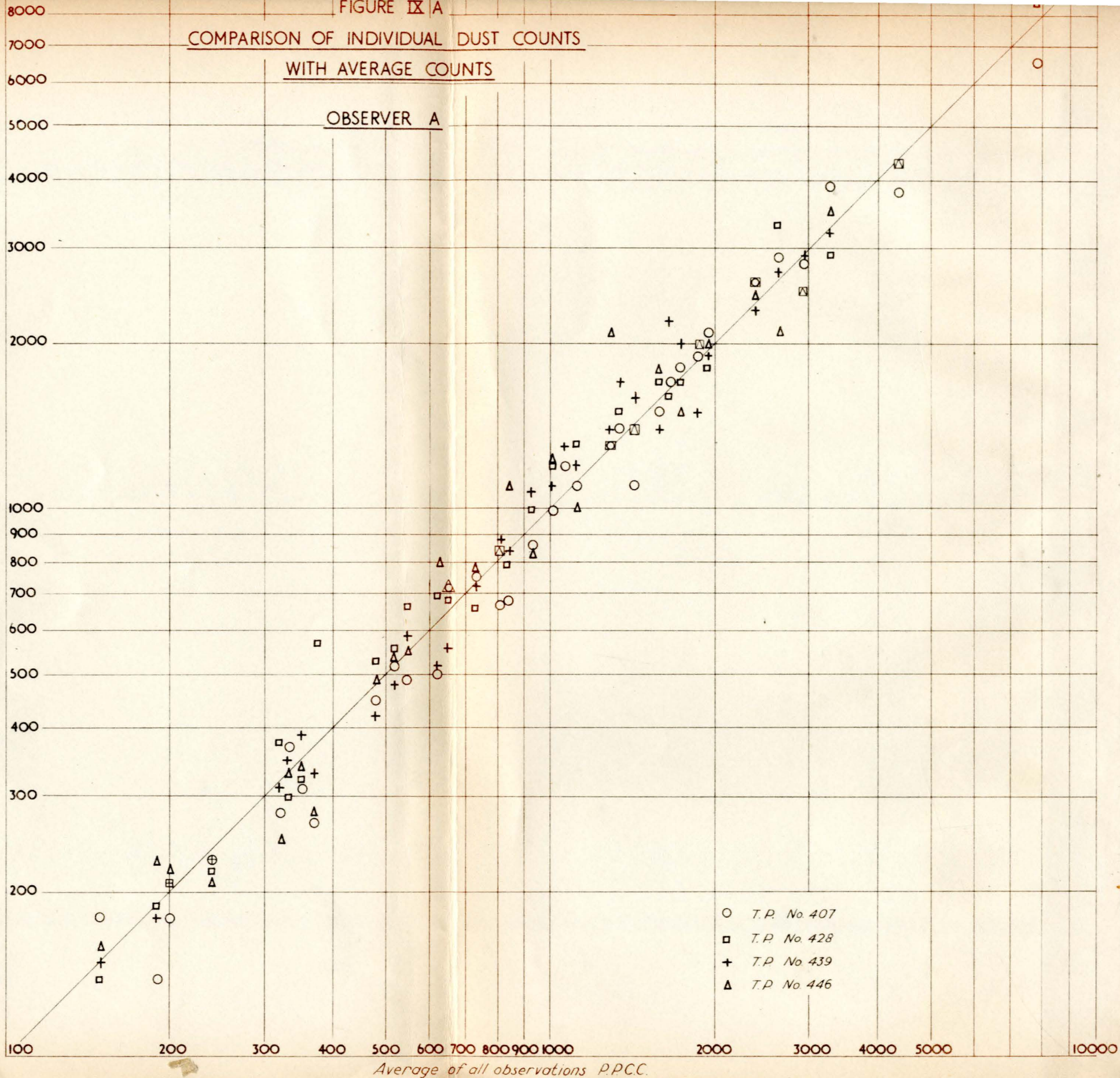


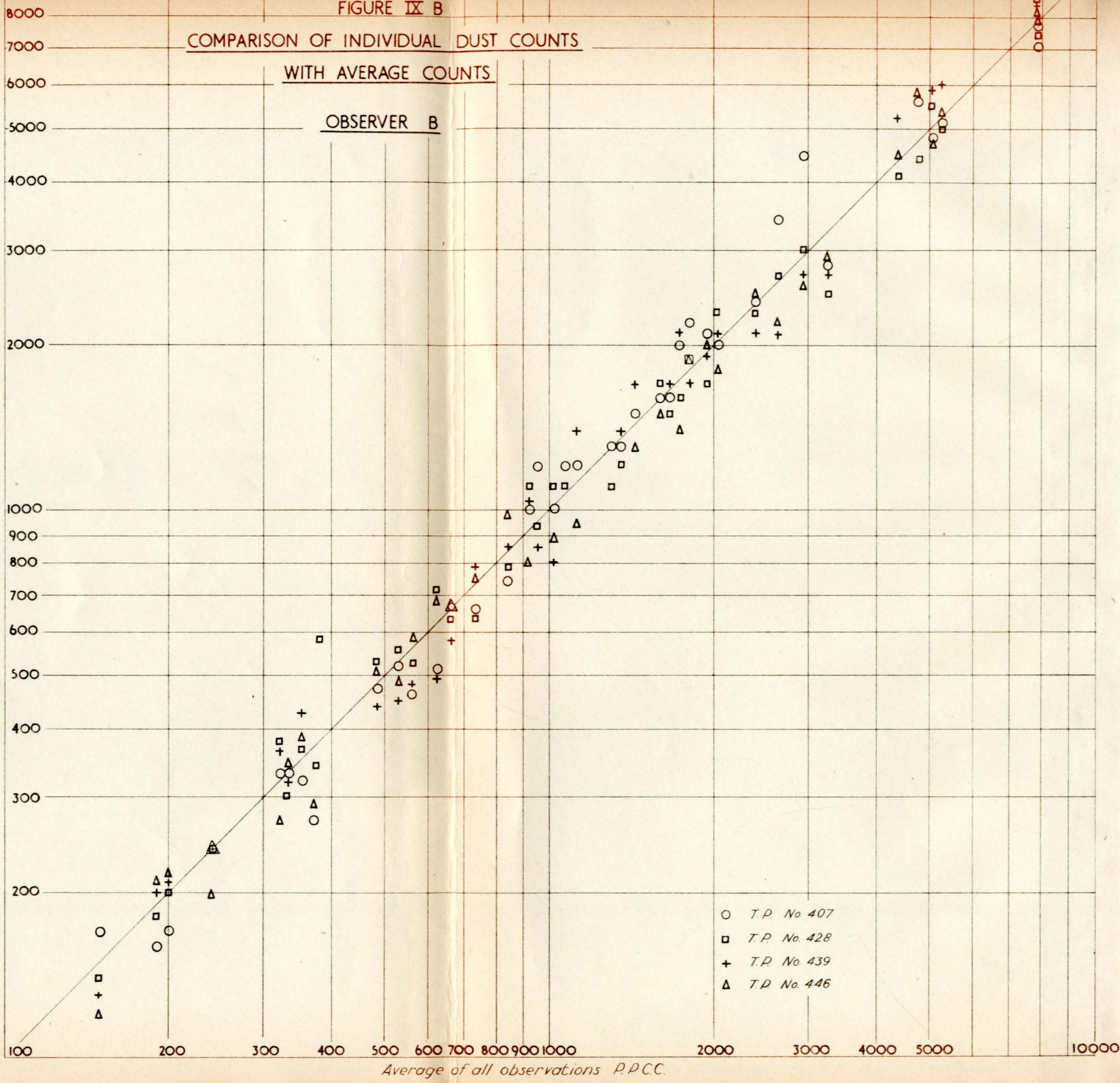
FIGURE IX B

COMPARISON OF INDIVIDUAL DUST COUNTS

WITH AVERAGE COUNTS

OBSERVER B

Individual Observation P.P.C.C.



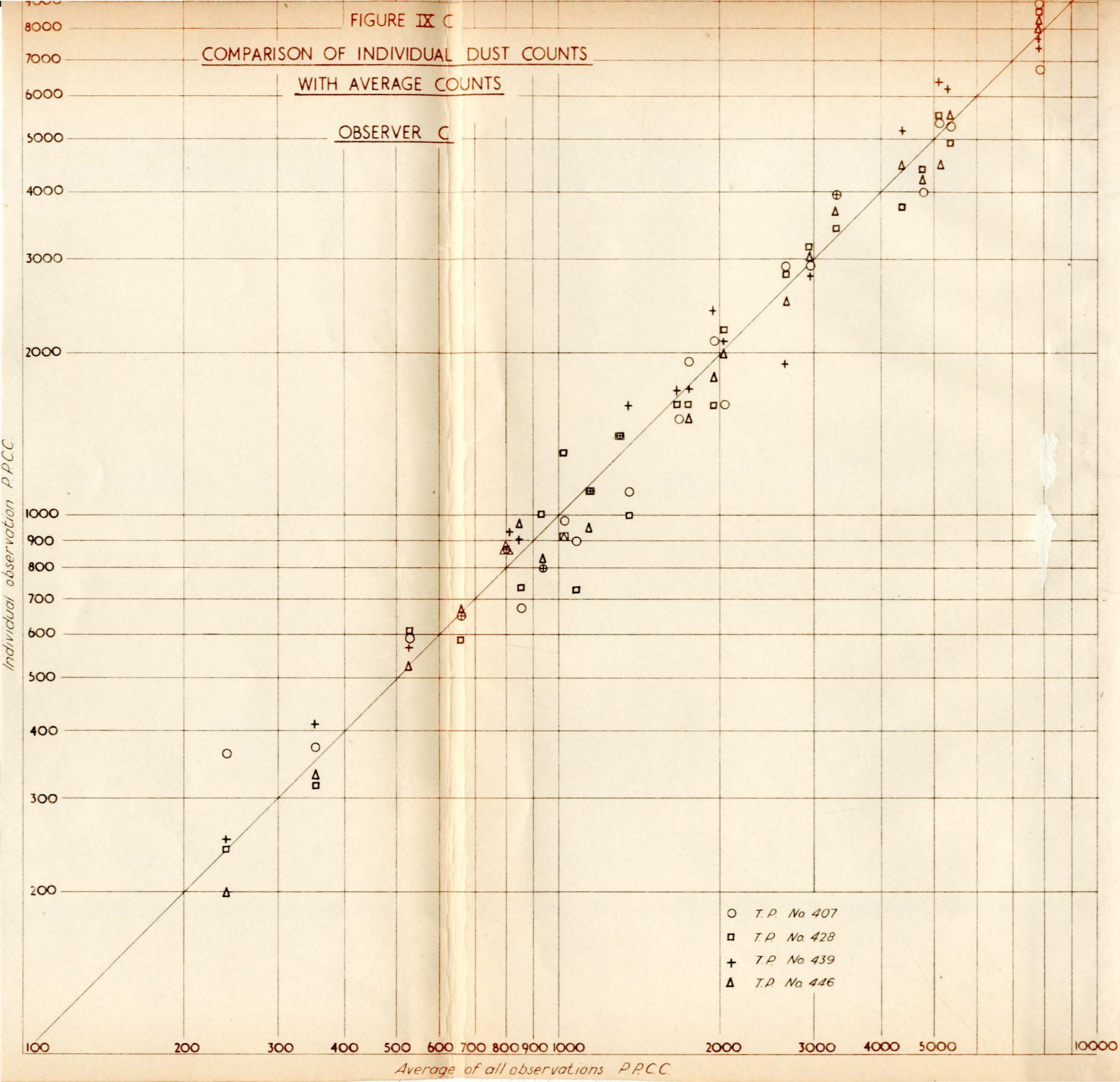
- T.P. No. 407
- T.P. No. 428
- + T.P. No. 439
- △ T.P. No. 446

FIGURE IX C

COMPARISON OF INDIVIDUAL DUST COUNTS

WITH AVERAGE COUNTS

OBSERVER C



dent of the dust concentration. This means that the logarithms of the dust counts will have approximately a constant standard deviation. In the statistical analysis connected with these investigations the logarithms of the dust counts were therefore used since well established statistical procedures could be applied directly to such data, but the results have been converted back to actual counts for presentation here.

#### 4.1 The overall error.

Four thermal precipitators were placed close together and simultaneous samples taken on each. The dust samples were ignited before counting but were not acid treated since this introduces another source of variation. Each sample was counted by two or three observers. 43 such experiments were made. The range of dust concentrations sampled (150 p.p.c.c. to 9,000 p.p.c.c.) covers that normally found in Witwatersrand gold mines.

The results are plotted in Figures IXa, IXb and IXc and show the individual counts obtained by each observer from each instrument.

These are compared with the average result obtained in each experiment.

Analysis of these results showed that a single thermal precipitator sample counted by a single observer had an overall percentage standard deviation of 13%.

#### 4.2 Mutual bias between instruments and observers.

The same data was used to measure the mutual bias between the four thermal precipitators.

The result given by each thermal precipitator in each experiment was divided by the average result given by all thermal precipitators in that experiment. The average ratio for each thermal precipitator over the whole series of experiments is given in Table XII.

TABLE XII.

Thermal Precipitator No.	Average Ratio
407	.985
428	1.006
439	.996
446	1.013

Further analysis showed that none of

these values were significantly different, at the 95% confidence level, from unity. There is therefore no evidence of mutual bias between any of the instruments.

The data was next examined to see if there was any mutual bias between the observers who had counted the samples. The count obtained by each observer was compared with that of each other observer, for each sample. The average ratios of the counts are given in Table XIII.

Observer A was a very experienced observer, B had fair experience and C limited experience of counting dust samples.

TABLE XIII.

Observers	Average Ratio of Counts	Number of Comparisons
$N_A/N_B$	1.023	134
$N_A/N_C$	1.045	79
$N_B/N_C$	1.015	104

$N_A/N_C$  was found to be significantly different (at the 95% confidence level) from unity, and the indications are that observer A

tends to count a few percent more particles than the others, and Observer C to count a few percent particles less than the others. This might be expected from their degrees of experience.

#### 4.3 The sources of the errors in a thermal precipitator sample.

A thermal precipitator measurement is obtained by drawing a measured volume of air through the sampling head. The pair of cover glasses on which the dust is deposited is then examined under the microscope. The number of particles in a small strip across the width of the sample is counted on each cover glass of the pair and added together - this is usually referred to as one traverse of the sample. This strip is a known fraction of the whole sample; usually about  $\frac{1}{1000}$ th. If the number of particles per traverse is small it is customary to count a number of traverses of each cover glass to improve the accuracy of the count.

The concentration of dust in the air, N, in particles per c.c. is given by

$$N = \frac{n F}{t v}$$

where  $n$  = total number of particles counted on the two cover glasses comprising the sample.

$F$  = the ratio between the area of the thermal precipitator sample and the area of a traverse.

$t$  = number of traverses made of the cover glasses.

$v$  = volume of air sampled.

The following sources of error have been investigated -

- (a) The errors which arise in counting the number of particles in a given traverse - the "counting error".
- (b) The errors which arise from assuming that the number of particles per traverse in the portion examined is equal to the average number per traverse in the whole sample - the "traverse error".
- (c) The variations between the results of different observers counting the same slide - the "observers error".

- (d) Errors in V, arising from mistakes in measuring the volume of water aspirated - the "volume error".
- (e) The variations between the results obtained from a number of thermal precipitators sampling simultaneously side by side - the "instrument error".

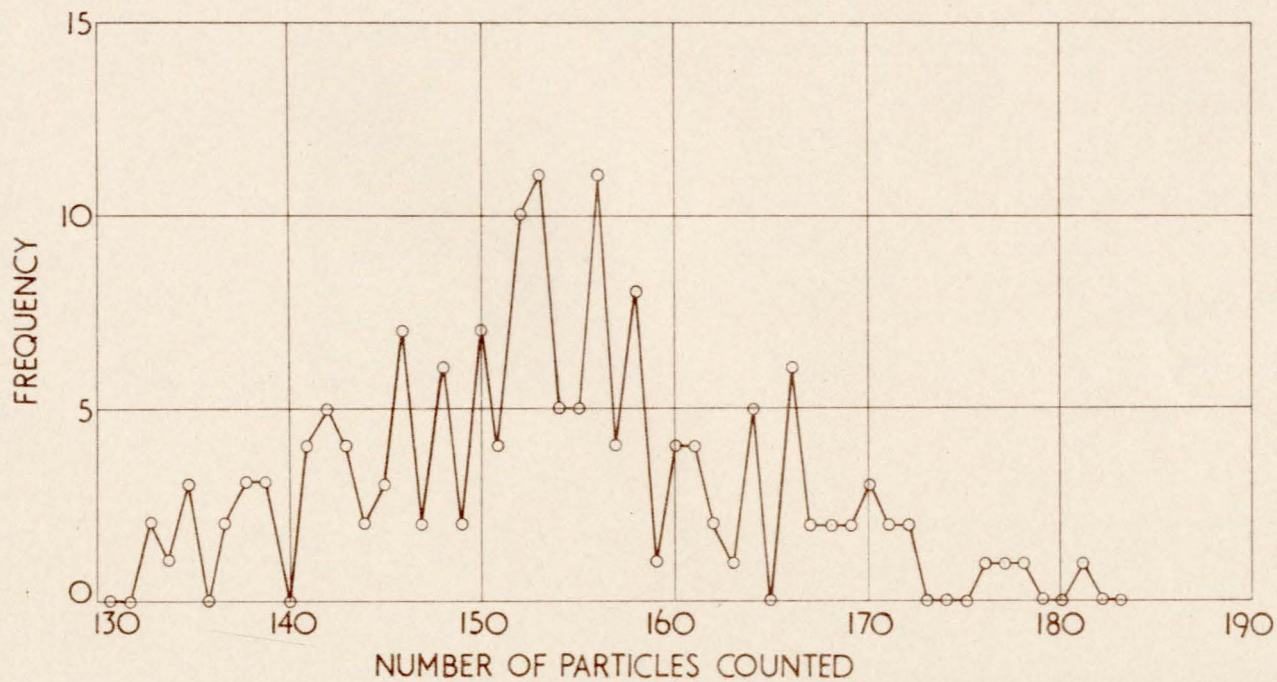
These were believed to be the principal sources of error, and it was expected that if these individual errors could be assessed separately they would account for most of the overall error. If the individual sources of variation have standard deviations  $\sigma_1$ ,  $\sigma_2$ ,  $\sigma_3$ , etc. and the overall standard deviation is  $\sigma_{\text{Total}}$  then  $\sigma_{\text{Total}}^2 = \sigma_1^2 + \sigma_2^2 + \sigma_3^2 + \text{etc.}$

(a) The counting error ( $\sigma_1$ ).

Many dust particles in samples taken in Witwatersrand gold mines are at the limit of visibility of the optical microscope and are very difficult to see. The actual count obtained may therefore be affected by the degree of fatigue in the observer, lack of concentration, and similar factors. It is also well

FIGURE X

FREQUENCY WITH WHICH DIFFERENT COUNTS WERE  
OBTAINED FROM SAME TRAVERSE



known that when counting a number of objects without some method of marking off those already counted, some variation from the correct value is likely to occur. For these reasons the count in a given traverse may not represent accurately the true number present, and repeated counts of the same area can be expected to show some variation about a mean.

The magnitude of this variation has been investigated by counting the same traverse 154 times. The author used a mechanical recorder so that he would not be influenced by knowing the count reached during each traverse. Three variables which it was thought might affect the results were introduced in a random way, namely, speed of counting, the time interval between successive counts, and the number of counts made at one counting session.

Figure X shows the number of times various counts were obtained. The mean of all the counts is 153.6 particles, and they have a percentage standard deviation of 6.6.

The results were divided into groups according to the speed of counting. The mean

count in each group was calculated.

Similarly the mean count after rest intervals of different lengths was calculated. These results are given in Table XIV. In 18 experiments this information was not recorded.

The observations also indicated that if more than 18 traverses were counted at one counting session the results became erratic. Thus in counts made after the 18th in any session the percentage standard deviation rises to 8.2. No similar fatigue effects could be discovered when less than 18 counts were made in a counting session.

These results indicate that -

- (i) The counting speed should not be greater than about 70 particles per minute - at higher rates some particles are missed.
- (ii) An interval of not less than 2 minutes should be allowed between successive counts.
- (iii) Not more than 18 counts should be made in one counting session.

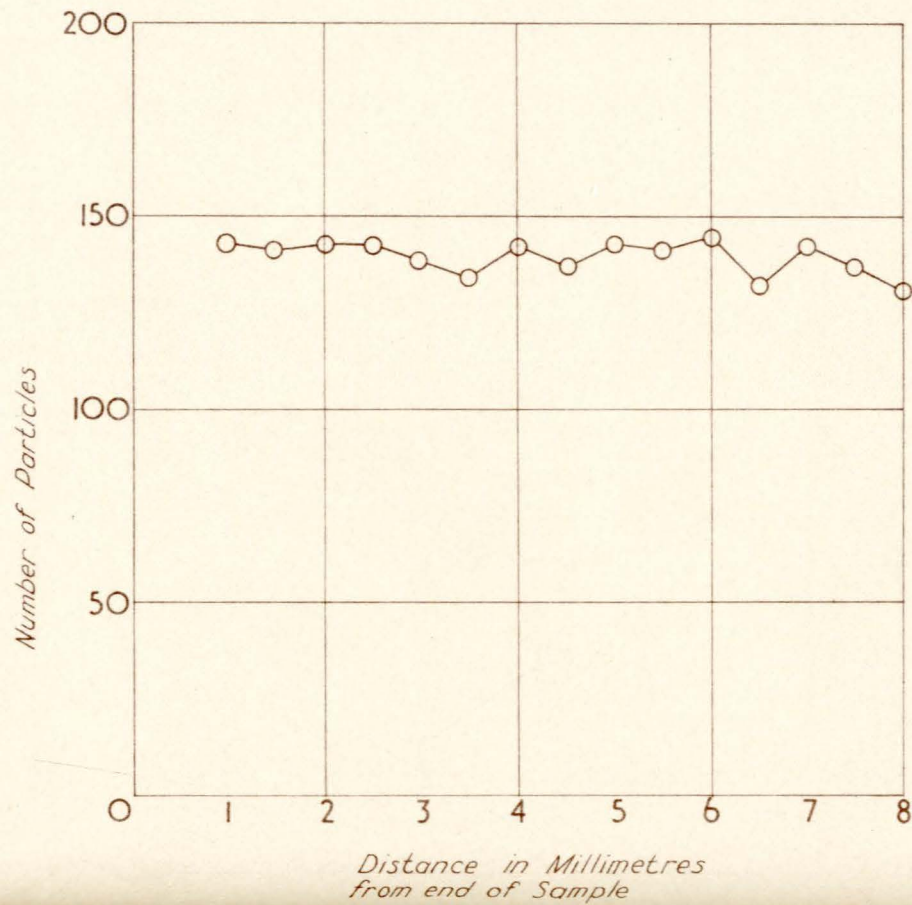
All experiments which had been made under conditions shown to affect the count appreciably (counting time under 2 minutes,

TABLE XIV.

Time taken for count Minutes	Average count	Number of Experiments in this Group	Interval before count Minutes	Average count	Number of Experiments in this Group
$1\frac{1}{4}$	141.0	2	$\frac{1}{4}$	145.6	5
$1\frac{1}{2}$	145.8	5	$\frac{1}{2}$	152.9	67
$1\frac{3}{4}$	151.7	27	$\frac{3}{4}$	155.5	19
2	153.0	44	1	154.8	6
$2\frac{1}{4}$	157.1	23	$1\frac{1}{4} - 2$	152.4	11
$2\frac{1}{2}$	164.4	17	$2\frac{1}{4} - 3$	161.5	8
$2\frac{3}{4}$	154.8	13	$3\frac{1}{4} - 4$	158.9	7
3 & $3\frac{1}{4}$	158.0	5	4 & over	160.5	13

FIGURE XI

VARIATION IN NUMBER OF PARTICLES ALONG  
LENGTH OF A THERMAL PRECIPITATOR SAMPLE



interval less than  $\frac{1}{2}$  minute, or more than 18th count in a session) were then eliminated. This left 74 counts, which have a mean of 156.3 and a percentage standard deviation of 5.6.

This latter figure is taken to be a measure of the "counting error" (C1).

(b) The traverse error (C2).

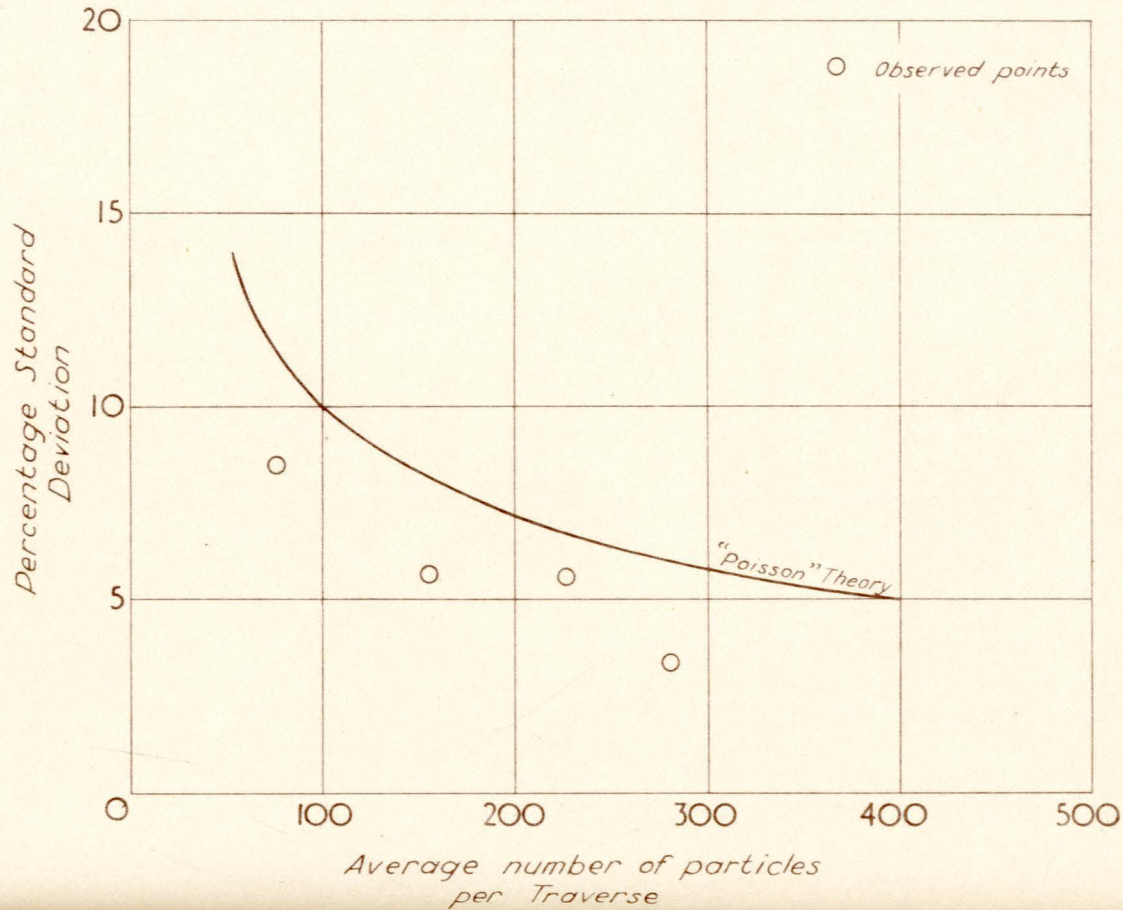
15 traverses spaced equidistantly along the length of a sample were each counted three times. The average count for each traverse is shown in Figure XI.

From this data the "counting error" in this experiment was estimated and then eliminated from the results thus providing information on the magnitude of the variation between different traverses. The "traverse error" (C2) was found to have a percentage standard deviation of 3.3 in this experiment.

Several experiments have been carried out in which a single count was made of each of a number of traverses in a sample. The standard deviation in such experiments will combine the "counting error" (C1) and the "traverse error" (C2). If the combined error is  $C_{1+2}$

FIGURE XII

RELATIONSHIP BETWEEN PERCENTAGE STANDARD  
DEVIATION OF COUNT AND NUMBER OF  
PARTICLES COUNTED



$$\text{then } \sigma_{1+2}^2 = \sigma_1^2 + \sigma_2^2 .$$

It is a measure of error likely to arise when a single count of a thermal precipitator sample is taken to be representative of the true average number of particles per traverse.

Various observed values of  $\sigma_{1+2}$  are plotted in Fig. XII against the average number of particles per traverse. Previously Goch<sup>65</sup> had advanced the theory that the distribution of this error would follow the Poisson Law; this, if correct, would imply that the standard deviation of the count was equal to the square root of the number of particles counted, i.e. that the standard deviation  $= \sqrt{n}$  and the percentage standard deviation  $= \frac{\sqrt{n}}{n} \times 100 = \frac{100}{\sqrt{n}}$ .

Acceptance of this theory has led to the local practice of usually counting at least 400 particles in each sample, in order to limit the percentage standard deviation to 5%.

The error, based on this theory, associated with counting various numbers of particles is also plotted on Fig. XII, and it will be noted that the observed values of the

combined error are in all cases less than the values predicted by Goch's theory. The accuracy of counting is therefore somewhat better than previously believed to be possible. The combined counting error and traverse error totals about 6% if 200-250 particles are counted.

(c) The observer's error (63).

In section 4.1 experiments were quoted in which two or three observers each counted the same sample in 45 different experiments. It was shown in Table XIII that the mutual bias between these observers was small, but this does not mean that they will agree so closely in their counts of each individual slide. The count obtained by each observer of each slide has been compared with the count obtained by the other observers of that slide. The observers did not count the same traverse of each slide. The percentage deviation between each pair of observers counts of each slide has been calculated, and the average values are given in Table XV.

FIGURE XIII

RELATIONSHIP BETWEEN PERCENTAGE STANDARD DEVIATION  
AMONG COUNTS BY DIFFERENT OBSERVERS AND  
AVERAGE NUMBER OF PARTICLES COUNTED

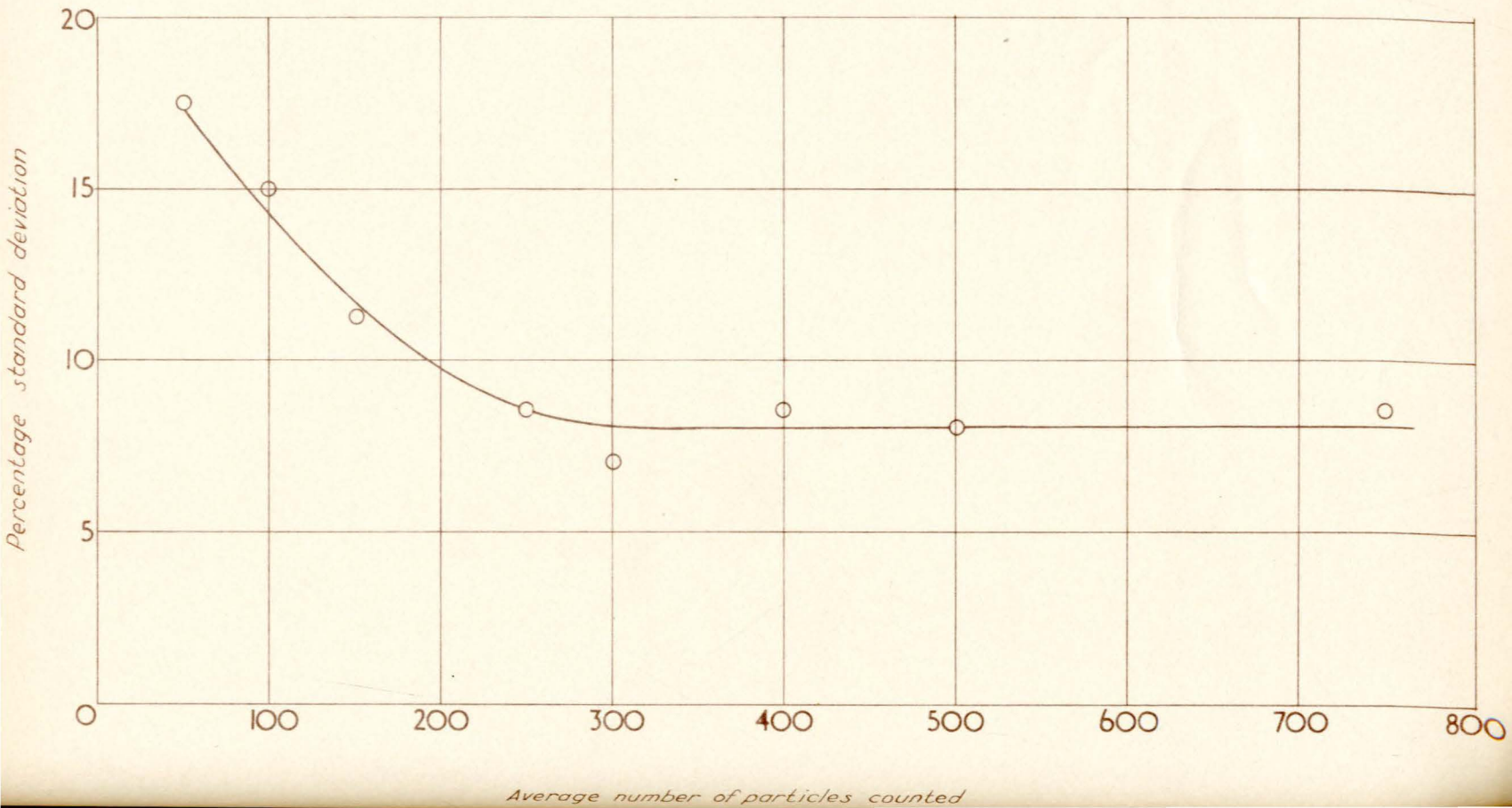


TABLE XV.

Observers	Average Percentage Standard Deviation	Number of Comparisons
A & B	8.5	134
A & C	13.3	79
B & C	11.8	104

In another similar experiment two experienced observers A and D had an average percentage deviation of 7.6 in a series of 63 comparison counts. Observer C was relatively inexperienced, and if his results are excluded it appears that 8% may be a typical value of the average difference between two observers' counts of the same slide.

These variations were further analysed to see if they depended on other factors. Fig. XIII shows how the percentage standard deviations depend on the numbers of particles counted per sample. Each result is the mean of more than 30 comparisons. The percentage variation between observers increases rapidly as the number of particles counted decreases below 250, but above this number, the variation remains constant. No increased accuracy

results from counting more than this number of particles per sample; this again points to a saving in labour in the present practice of counting at least 400 particles per sample.

Another analysis was made to see if the variation between observers depended on the density of deposit in the samples, i.e. on the average number of particles per traverse. Samples in which the total number of particles counted was less than 150 were excluded, due to the increased errors arising in such counts. The results are given in Table XVI.

TABLE XVI.

Number of Particles per Traverse	Average Percentage Standard Deviation
Less than 50	7.0
50 - 100	6.7
100 - 150	8.7
150 - 200	4.9
200 - 250	11.3
250 - 300	7.2
300 - 350	9.2
More than 350	8.8

There is no indication that the variation between observers depends on the density of deposit.

The variation of about 8% between the counts by experienced observers of the same slide is partly accounted for by the combined "counting and traverse" error associated with each observers count of the slide, previously assessed as about 6%. The remaining error i.e.  $\sqrt{8^2 - 6^2} = 5.3\%$  is due to other random variations between the observers' counts, and is called the observer's error,  $\sigma_3$ . It is probably higher when one or both of the observers is inexperienced.

(d) The volume error ( $\sigma_4$ ).

This is the only measurement actually made underground when taking a thermal precipitator sample. The water leaving the aspirator is collected in a suitable container and measured in a 100 c.c. measuring cylinder. Volumes range from about 10 c.c.s. to 1000 c.c.s. depending on the type of experiment. Because conditions in a mine are not conducive to accurate work, this measurement may be a source of considerable error.

Various difficulties arose in attempting to design experiments to measure the magni-

tude of this error in tests underground. Instead an experiment was carried out in the laboratory. 27 beakers, containing from 75 c.c.s. to 570 c.c.s. of water were each measured 6 times by six different observers; i.e. 36 measurements were made of each volume of water. The observers were made to take their measurements as rapidly as possible - each observer made 162 measurements in under 2 hours. It was assumed that this would lead to errors of the same order as would arise in the case of an observer making a single observation underground when he is not hurried but is subject to the difficulties of bad lighting, cramped surroundings and various distractions sometimes encountered in a mine.

The results showed two types of error -

- (i) Small random errors of the order of a few ccs -
- (ii) Occasional gross errors, e.g. 10 ccs (in 3% of the observations) and 100 ccs (in 0.6% of the observations).

The overall percentage standard de-

viation of the errors was 3.3 and this has been accepted as some estimate of the volume error ( $\sigma_4$ ).

(e) The variation between instruments ( $\sigma_5$ ).

In the experiments mentioned in 4.1 above, simultaneous samples were taken on 4 thermal precipitators. Each sample was counted by different observers, usually three. The mean of three counts of the same slide will have  $\frac{1}{\sqrt{3}} = 0.58$  the error of a single count. To provide information on the variation between the actual amounts of dust collected by each thermal precipitator the mean of the counts by all observers of each thermal precipitator sample was compared with the mean of all counts of all instruments in each experiment. These were found to have a standard deviation of 10.6%, but of this  $0.58 \times 8 = 4.6\%$  is due to combined counting, traverse and observers error, and 3.3% to volume errors so that the actual variation between instruments is

$$= \sqrt{10.6^2 - 4.6^2 - 3.3^2} = 9.0\%$$

This variation between instruments ( $\sigma_5$ ) may be due to real differences in the dust concentra-

tion in the air sampled by each instrument. However since the instruments were placed within a few inches of each other, and the samples taken over a period of fifteen minutes to an hour with the air moving continuously over them, it is considered unlikely that this can account for all the variation.

A more likely explanation is that occasionally the sample from one or more of the thermal precipitators becomes contaminated by extraneous dust settling on the slide or in some other way, leading to a spuriously high count.

It is also possible that errors sometimes arise from faulty operation of the thermal precipitator, such as failing to ensure an airtight connection between the sampling head and the aspirator, or not using the proper current to heat the wire.

One or more of these reasons may account for this relatively high variation between the results obtained with an instrument which is known to have a high sampling efficiency.

4.4 The combined variation.

The estimates derived above of the various sources of variation are summarised in Table XVII

TABLE XVII.

Counting error	=	5.6%
Traverse error	=	3.3%
Observer's error	=	5.3%
Volume error	=	3.3%
Variation between instruments	=	9.0%

Combining these, the total variation is given by

$$\sqrt{5.6^2 + 3.3^2 + 5.3^2 + 3.3^2 + 9.0^2} = 12.7\%$$

which is in satisfactory agreement with the overall standard deviation of 13% reported in section 4.1

---

5. A MODIFIED FORM OF THERMAL  
PRECIPITATOR

Because of the limitations of the konimeter there is a need for a more accurate instrument for routine dust sampling in Witwatersrand gold mines. The standard form of thermal precipitator is sufficiently accurate but is not suitable for the following reasons :-

- (a) It is too large and heavy.
- (b) Considerable skill is required to operate it correctly.
- (c) The cover glasses are fragile, they must be changed underground after each sample, and two are used per sample.
- (d) The time required to set up the instrument and take a sample is too long.
- (e) The microscope examination of the samples requires skilled observers, takes a considerable time, and has unavoidable statistical errors connected with it.

No other form of dust sampling instrument has been found which is sufficiently accurate and convenient. The principle of

thermal precipitation has therefore been retained in a modified form of the instrument which overcomes the above objections. The new instrument was also designed to collect a sample of dust in which particles of various sizes are collected approximately in proportion to their retention in the human lung. The desirability of this sampling characteristic was mentioned in Section I of this thesis.

This modified form of thermal precipitator has been fully described in a paper by the author and a colleague<sup>43</sup> - a copy of this paper forms Appendix I. The salient features only will be mentioned below. The three principal parts are the sampling head, the mechanical aspirator, and the electrical circuit.

### 5.1 The sampling head.

A number of samples are collected on a single 3" x 1" glass microscope slide; after each sample has been taken the slide is moved inside the sampling head so that a new portion faces the hot wire. 12 samples may be taken on

a single slide without removing it from the sampling head - this is sufficient for a normal days work.

The slide moves along an accurately machined face inside the sampling head. It is held firmly against this face by springs in the cover of the head. The hot wire, which deposits the dust, is supported along its whole length in a block of refractory material. This block is mounted in the sampling head so that the glass slide is 0.20 mm away from the wire.

Air flows into the sampling head through a horizontal channel 10 mm wide, 2 mm high and 10 mm long (in the direction of air flow). These dimensions were so chosen that, with the rate of air flow used, all silica particles larger than 10 microns diameter would settle to the floor of the channel during their passage through it; particles of smaller diameter settle in varying percentages depending on their size. After leaving the entrance channel the air passes the hot wire which deposits the dust onto the glass slide.

## 5.2 The mechanical aspirator.

Water aspirators have various disadvantages - the rate of aspiration varies as the hydraulic head changes, the volume of water has to be measured for each sample and reserve supplies of water have to be carried. A mechanical aspirator was therefore developed for the modified thermal precipitator. This consists of a bellows which is extended by cords attached to a drum driven by a small motor. The bellows is made of a rubberized fabric.

Originally a clockwork motor was used, but this was later replaced by a small fractional watt electric motor. The main spindle of the motor makes 2500 revolutions per minute, and this is geared down to the spindle carrying the drum and revolving at  $\frac{1}{10}$ th revolution per minute; the torque is therefore high. This spindle extends through the casing of the aspirator and carries a pointer which moves over a scale. One revolution of the drum takes 10 minutes and extends the bellows sufficiently to draw 100 c.c.s. through the sampling head.

When a sample is to be taken, the pointer is rotated and set to aspirate any desired volume, up to 100 c.c.s. The action of rotating the pointer causes the drum on the spindle to wind up cords which compress the bellows. A simple clutch, operated by pressing down the pointer, disconnects the drum spindle from the motor during this operation. When the clutch is released the electrical supply is automatically connected to the motor; this drives the drum in the opposite direction and the bellows is extended. A switch cuts off the electrical supply to the motor when the bellows is fully extended.

The torque of the drum spindle is sufficiently high to ensure that the speed of rotation does not change as the load increases when the bellows are approaching their maximum extension. Experiments have shown that the relationship between the displacement of the moving end of the bellows and the volume of air aspirated is practically linear. This means that the volume of air aspirated per minute should be constant, and actual measurement has

shown that the volume in fact is 10.0 ccs per minute, with a standard deviation of less than 0.1 ccs.

In addition to overcoming the disadvantages of water aspirators mentioned above, this mechanical aspirator has the additional advantage that when the pre-selected volume of air has been aspirated, the aspirator stops itself. The operator need not discontinue other work to attend to it until it is convenient to do so.

### 5.3 The electrical circuit.

The current for heating the hot wire and for driving the motor is taken from an accumulator of the type used for miners' cap-lamps. Providing a large number of samples are not taken in a day, an accumulator of this type has sufficient capacity to operate the thermal precipitator as well as the observer's cap-lamp. There is therefore no additional weight for the observer to carry to provide power for the thermal precipitator. A rubber-covered lead connects the accumulator to the

modified thermal precipitator. The circuit includes a small  $\frac{1}{2}$  ohm rheostat which incorporates a switch, and a  $1\frac{1}{2}$  inch diameter ammeter. These are so wired that adjustments of the rheostat to keep the current in the heating wire to the required 2 amperes also adjusts the motor supply current to a constant value, so that it maintains a constant speed independent of voltage changes in the accumulator.

#### 5.4. Methods of using the modified thermal precipitator.

The instrument can be screwed onto a light photographic tripod, or it can be suspended by a string or wire passing through an eyelet screwed into the top of the head. Alternatively it is light and small enough to be carried in the hand while the sample is being taken and this enables it to be moved around in the working place so that a more representative record of the dust conditions is obtained. It has been used in several positions in mines where the use of the standard thermal precipitator was impracticable. It has proved

to be sufficiently robust to stand up to normal use in the mines.

The total weight of the modified thermal precipitator is  $2\frac{1}{2}$  lbs; the standard form weighs 26 lbs.

#### 5.5 Tests of the modified thermal precipitator.

All tests have been made by taking simultaneous samples, side by side, on the modified instrument and two standard instruments. The samples were then counted under identical conditions, and the number of particles of various sizes collected by the modified thermal precipitator compared with the number of particles collected by the standard instruments.

Various tests were carried out during the development of the modified thermal precipitator to determine the optimum values of various dimensions in the new instrument, in particular the rate of aspiration, the heating current for the wire, and the distance between the wire and the slide. The effects of changes in these three dimensions are inter-related.

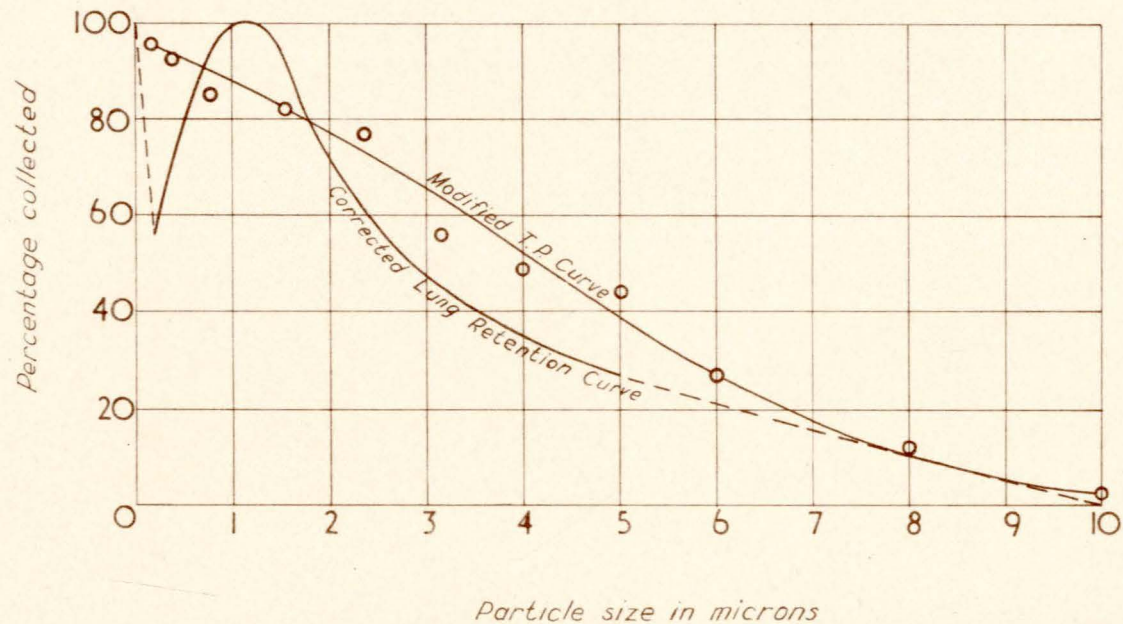
The first series of experiments with the final form were made in an airtight dust chamber in the laboratory. Dust clouds of different concentration and size distribution were created by spraying an aqueous or alcoholic suspension of dust into the air and allowing time for the liquid to evaporate. The air was kept in gentle motion by a fan while the samples were taken. Between 16 and 28 experiments were made for each size of particle.

The slope of the regression line connecting the number of particles of a given size found in the modified thermal precipitator sample with the number shown by the standard thermal precipitators, was taken as the collecting efficiency of the modified thermal precipitator for that particle size. The results were corrected for the small drop in sampling efficiency of the standard thermal precipitator for larger particles.

The results of these tests were quoted in detail, together with confidence limits and co-efficients of correlation, in the paper describing this work - see pages 300 - 301 of

FIGURE XIV

PERCENTAGE OF PARTICLES OF VARIOUS SIZES COLLECTED  
BY HUMAN LUNG (CORRECTED TO MAXIMUM OF 100%)  
AND BY MODIFIED THERMAL PRECIPITATOR



Appendix I.

The sampling efficiency of the modified thermal precipitator for particles of various sizes is given in Fig. XIV, and a smooth curve is drawn through the observed points. This figure also shows the lung retention curve of particles of various types, as previously given in Figure I, but adjusted so that the point of maximum retention is shown as 100% and other retentions increased in proportion. To a first approximation the modified thermal precipitator is sampling dust particles of various sizes in proportion to their retention in the human lung. It is the first dust sampling instrument with this important characteristic.

The second series of tests was carried out underground in various gold mines. More than 200 tests were made and the effects of various factors (depth in the mine, relative humidity, temperature, dust concentration and air velocity) on the collecting efficiency were investigated. These results are given in detail in Appendix I, pages 301 - 303

The only factor found to influence the collecting efficiency significantly was the air velocity. If the sampling orifice faces the air stream, and the latter has a velocity exceeding 250 - 300 feet per minute it was found that large particles (say size 2 microns diameter and greater) are driven through the entrance channel without settling as they normally tend to do, and are deposited on the slide resulting in a higher efficiency of collection for these sizes than shown in Fig. XIV. This effect is eliminated if the instrument is used so that the face containing the air intake is kept parallel to the air stream. Samples taken in this way showed approximately the same average collecting efficiency for particles of various sizes as were obtained in the laboratory tests described above.

Practical experience with the modified thermal precipitator over the past three years has indicated that it is suitable for routine dust sampling work, and it will give far more accurate results than the konimeter. The method of assessing dust samples taken with it is described in section 7.

---

6. THE EXAMINATION OF DUST SAMPLES UNDER THE MICROSCOPE.

The most common method of assessing dust samples is to count the number of particles present in them under a microscope. This method can also provide information on the size, shape and general appearance of the dust particles. Since it is desired to determine particles down to the smallest possible size this aspect of dust sampling has been kept constantly under review. Although the results quoted below apply specifically to dust samples taken with the thermal precipitator the methods described may be applied to dust samples collected in other ways.

6.1 High power light field illumination.

A general microscope technique for examining thermal precipitator samples was specified by those responsible for its development<sup>66</sup>. This recommended using a 2 mm. oil immersion objective with light field illumination to obtain the maximum resolution and to show the smallest possible particles. Other

details were described in general terms only, and soon after the introduction of the thermal precipitator in South Africa it was found that improvements in the microscope technique would result in finer particles being made visible.

Although text-books on microscopy commonly deal at length with the limit of resolution, there is comparatively little published work on the limits of visibility - one paper on the subject<sup>67</sup> does not extend down to the limits considered here. Although the two limits are to some extent related, factors such as contrast and glare play an appreciable part in determining the limit of visibility but are not so important when considering the resolving power. Several of the procedures recommended below improve the visibility, usually by increasing contrast or decreasing glare, at the cost of decreased resolution.

Numerous experiments were made to determine the best microscope conditions for showing the maximum number of particles. After the optimum conditions had been discovered the effect of varying each factor separately,

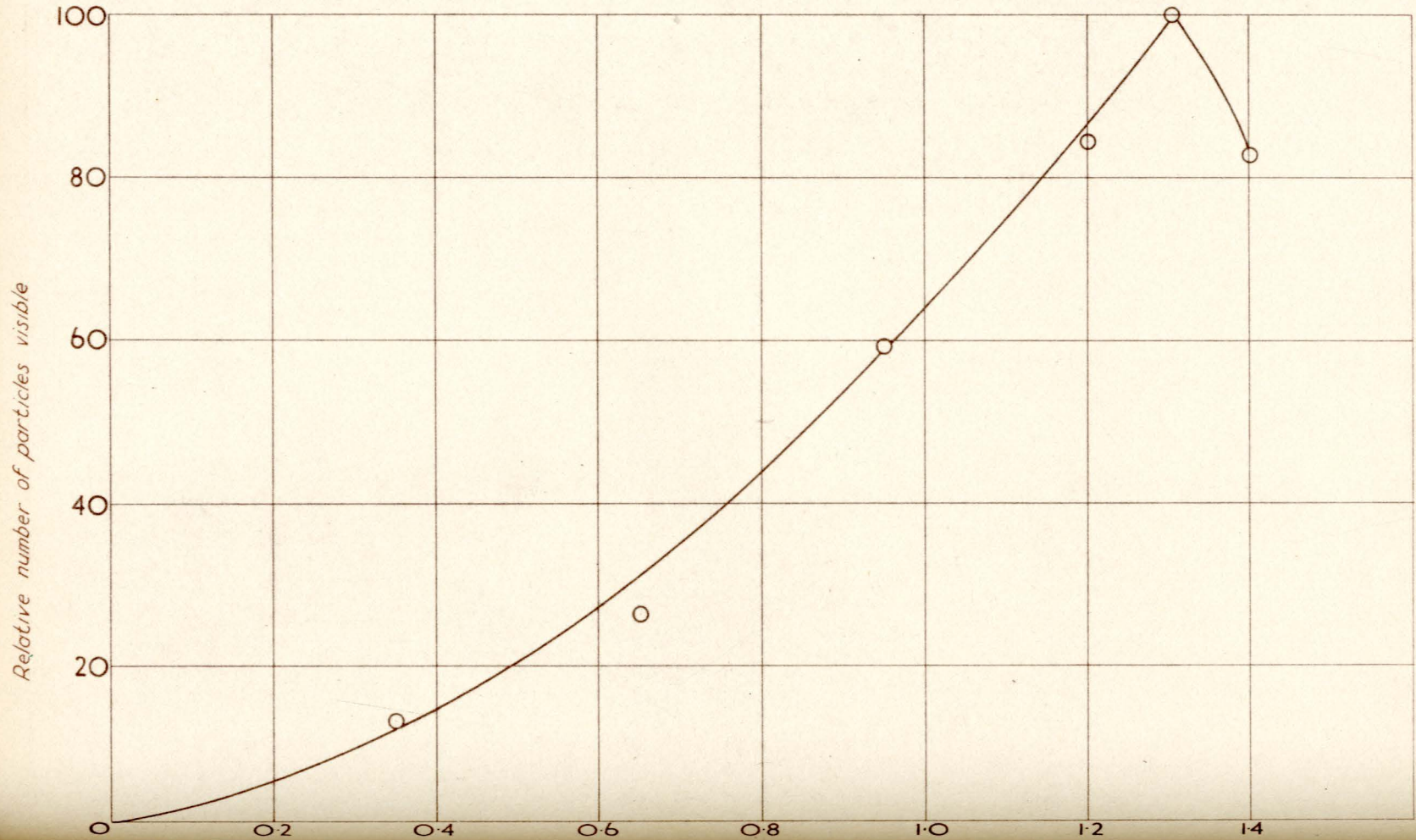
keeping all others constant, was studied by counting the number of particles visible in a fixed area of a dust sample. Since dust samples from mines contain numerous particles at and below the limit of visibility of the optical microscope, this provides a critical test of any adjustment; a very slight improvement in the limit of visibility will show an increased number of particles. The effect of the various factors will be discussed under separate headings, and wherever possible the results are given graphically.

#### Effect of mounting medium.

It is common practice to mount microscope specimens in a medium of high refractive index to increase the resolving power. However the contrast is dependent on the difference between the refractive index of the material examined and that of the mounting medium. Since quartz has a refractive index (1.55) close to that of many mounting mediums, e.g. Canada balsam, (1.52 - 1.54) the contrast is very poor and the finest particles cannot be seen. Experiments were made with many mounting

FIGURE XV

EFFECT OF NUMERICAL APERTURE OF OBJECTIVE  
ON NUMBER OF PARTICLES VISIBLE



materials but it was found that the maximum number of particles was visible if no medium was used and the dust particles examined mounted dry - i.e. against a background of air.

American work<sup>68</sup> has shown that if dust particles are coated with a film of very high refractive index (greater than 2) the difference in contrast is sufficiently great to show the particles clearly and the increased resolving power can therefore be used effectively. Such films can be produced by sputtering selenium in vacuo onto the slide carrying the dust sample. This method has not been tried since the labour involved in treating every dust sample in this way appears to preclude its application as a matter of routine.

Effect of numerical aperture of objective.

Counts were made with objectives of different numerical aperture, and are shown in Figure XV. All the objectives were apochromats; counts with achromatic objectives of the same numerical aperture gave lower counts. It was found that if a given objective was reduced to a lower numerical aperture by placing a

diaphragm behind its back lens it then gave approximately the same count as an objective of numerical aperture equal to the reduced value. This indicates that it is numerical aperture which is the controlling factor, and not the magnification of the objective.

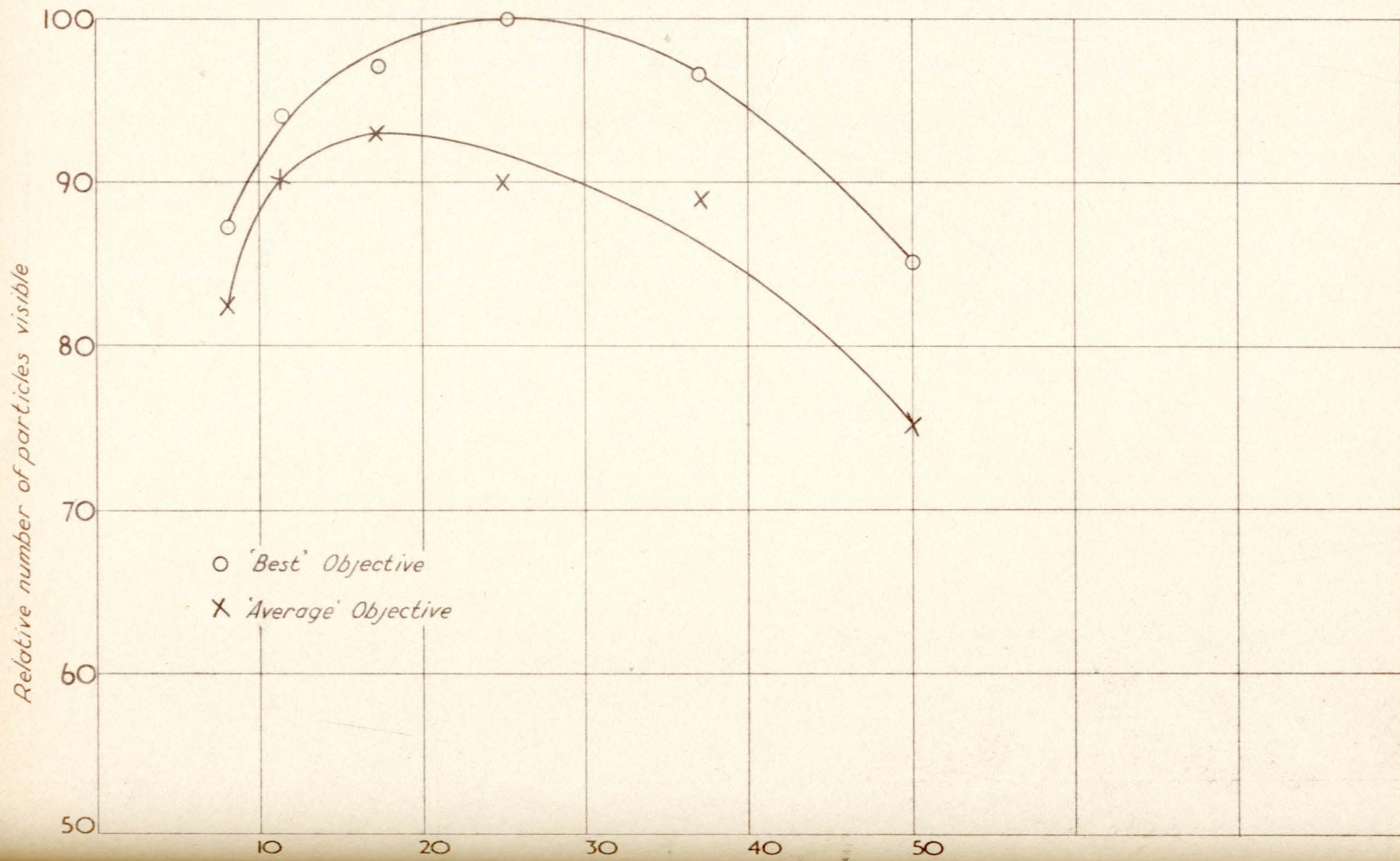
These results show that it is necessary to use a 2 mm. apochromatic objective of numerical aperture 1.3. The objective of numerical aperture 1.4 shows fewer particles and this may be due to the difficulty of providing adequate correction for spherical aberration in objectives of such high aperture. Although various manufacturers make objectives to the above optical specification, comparative tests proved that one particular make (Beck, London) showed a significantly greater number of particles than other makes.

#### Effect of condenser.

Simple uncorrected types of sub-stage condenser such as the Abbe, resulted in a loss of about 30% of the particles. Providing an achromatic and aplanatic condenser of numerical aperture 1.0 is used there appeared to be no

FIGURE XVII

EFFECT OF MAGNIFICATION OF EYEPIECE  
ON NUMBER OF PARTICLES VISIBLE



difference between different types.

Most text-books recommend that the aperture of the condenser should be reduced by an iris diaphragm to about two-thirds of the aperture of the objective to give the best resolution. However this still produces too much glare for the finest particles to be seen against a light field, and Figure XVI shows how the counts increase as the aperture of the condenser is reduced still further.

"Critical" illumination in which the image of the lamp is focussed in the plane of the object gave higher counts than "Kohler" illumination, in which the image of the lamp is focussed on the back lens of the condenser.

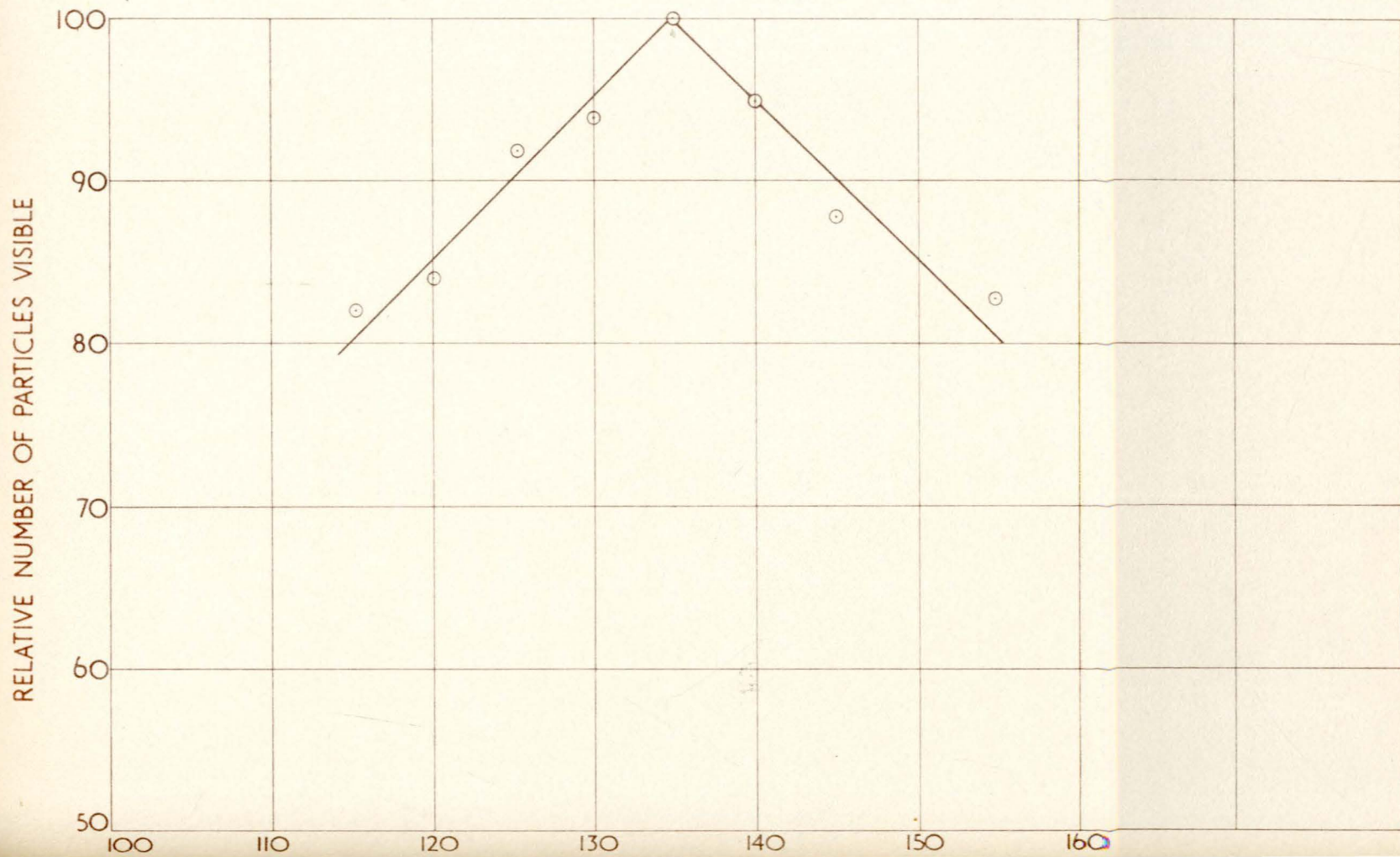
Effect of eyepiece magnification.

Counts were made with compensating eyepieces of different powers and with two objectives. One objective was that which had given the highest counts in the comparison of objectives while the other was of average performance. The results are given in Figure XVII.

The 'best' objective benefits from a higher eyepiece magnification, but in general

FIGURE XVIII

EFFECT OF TUBE LENGTH ON NUMBER  
OF PARTICLES VISIBLE



it has been found preferable to standardize on the xl7 eyepiece. With higher magnifications the images of the smallest particles become so diffused that their contrast is insufficient to enable them to be seen.

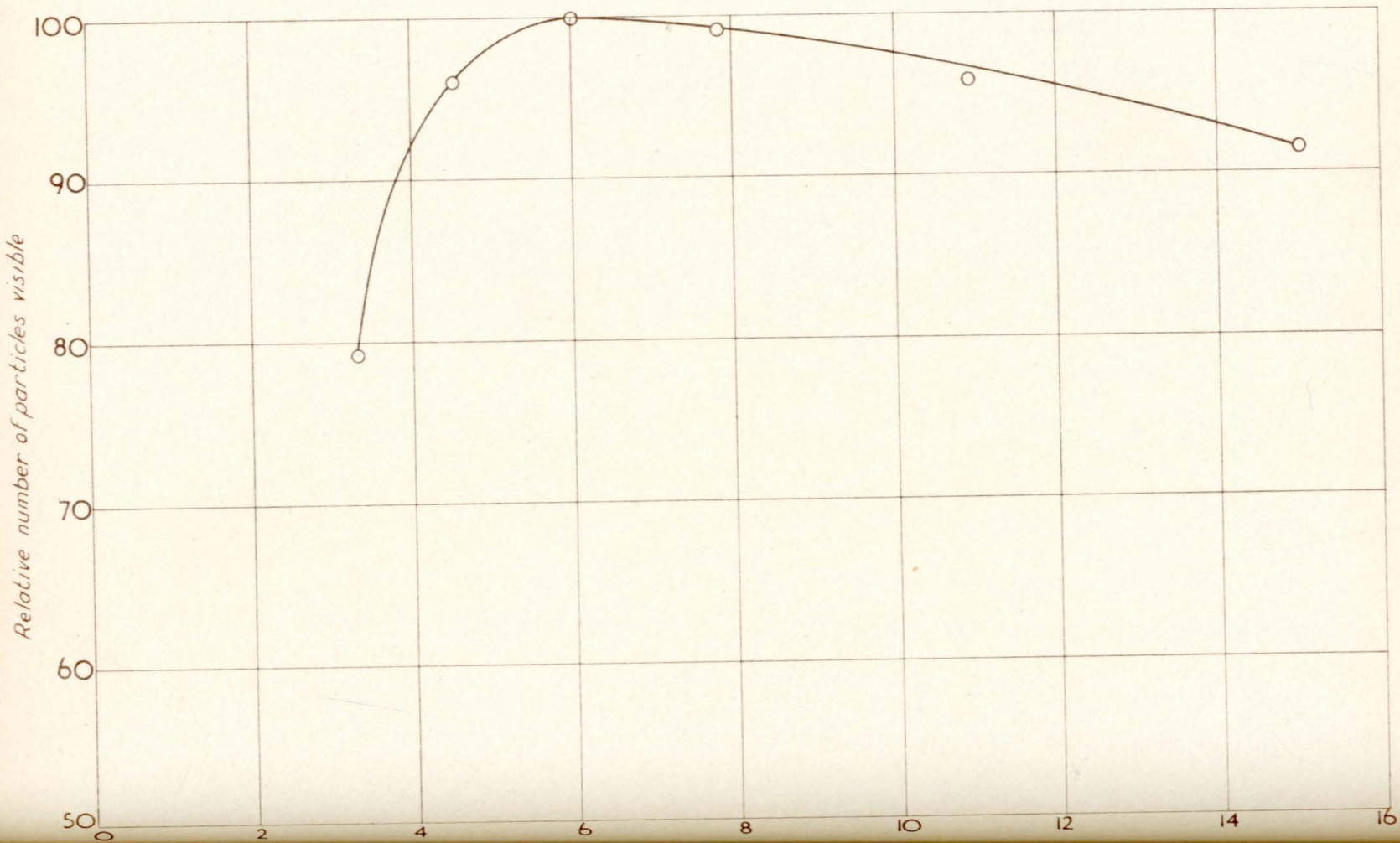
Effect of tubelength.

The objectives used were designed to work at a tubelength of 160 mms. The counts shown in Figure XVIII were obtained at other tubelengths. This result was published soon after it was discovered<sup>69</sup>. The reason may be that the objective is corrected by the manufacturer to work with a continuous medium of high refractive index between the front lens and the object (i.e. immersion oil, cover-glass and mounting medium) whereas in this work the object is in air. By altering the tubelength some compensation for upsetting the manufacturer's calculations for the correction for spherical and chromatic aberrations may result.

The optimum tubelength varies from one objective to another, but is always less than 160 mms. The best tubelength for a given

FIGURE XIX

EFFECT OF INTENSITY OF ILLUMINATION ON  
NUMBER OF PARTICLES VISIBLE



objective also depends on the thickness of the cover-glass. For one particular objective the relationship  $T.L. = 149 - 84t$  was established, where  $T.L.$  = optimum tubelength and  $t$  = thickness of cover-glass, both in millimetres.

#### Effect of intensity of illumination.

The brightness of the sub-stage lamp was varied over a wide range. The counts obtained are plotted in Figure XIX against the power used for the lamp. The results indicate that this is not a critical factor, and it is probably best for each observer to choose the level of illumination which he finds most comfortable.

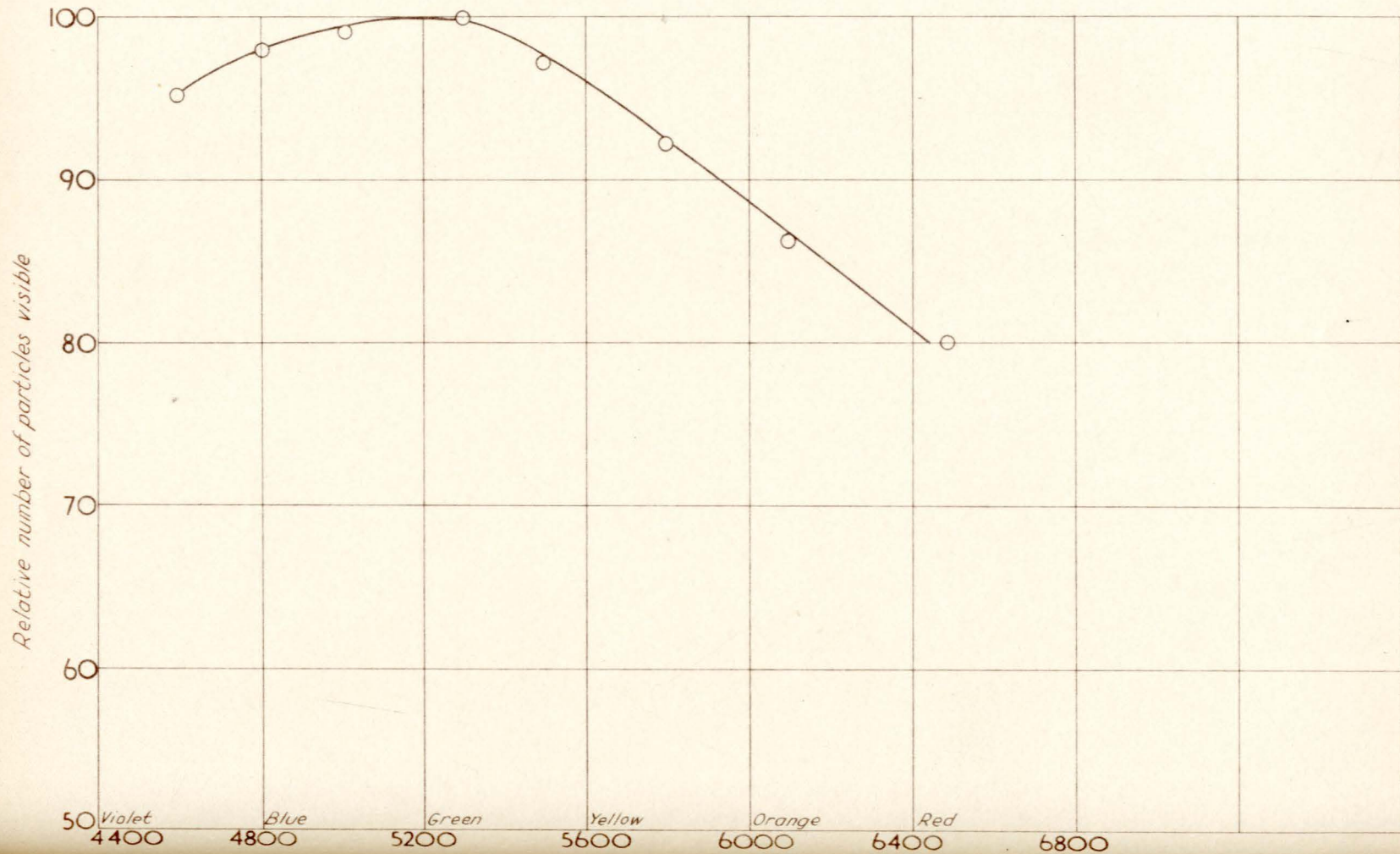
It was also found that the intensity of illumination in the room is not important; no significant difference in counts was found when this was varied from complete darkness to a normally well-lighted room.

#### Effect of wavelength of light.

Counts were made using light of different wavebands obtained from a set of spectrum filters. The lamp was adjusted to

FIGURE XX

EFFECT OF WAVELENGTH OF LIGHT ON  
NUMBER OF PARTICLES VISIBLE



give the same apparent brightness with each filter. The results are given in Figure XX. There is a wide zone, around 5,000 Angstrom units, in which wavelength appears to have little effect. Presumably the improved resolution at shorter wavelengths is balanced by the decreased sensitivity of the eye.

Comparison of counts.

A number of slides were counted using the optimum conditions found as a result of the above work, and also the conditions commonly used before this work was reported<sup>70</sup>. The improved methods showed a total of 3,540 particles while the other method showed 2,720 particles. The improved method has increased the count by 30%.

The size of the smallest particles visible.

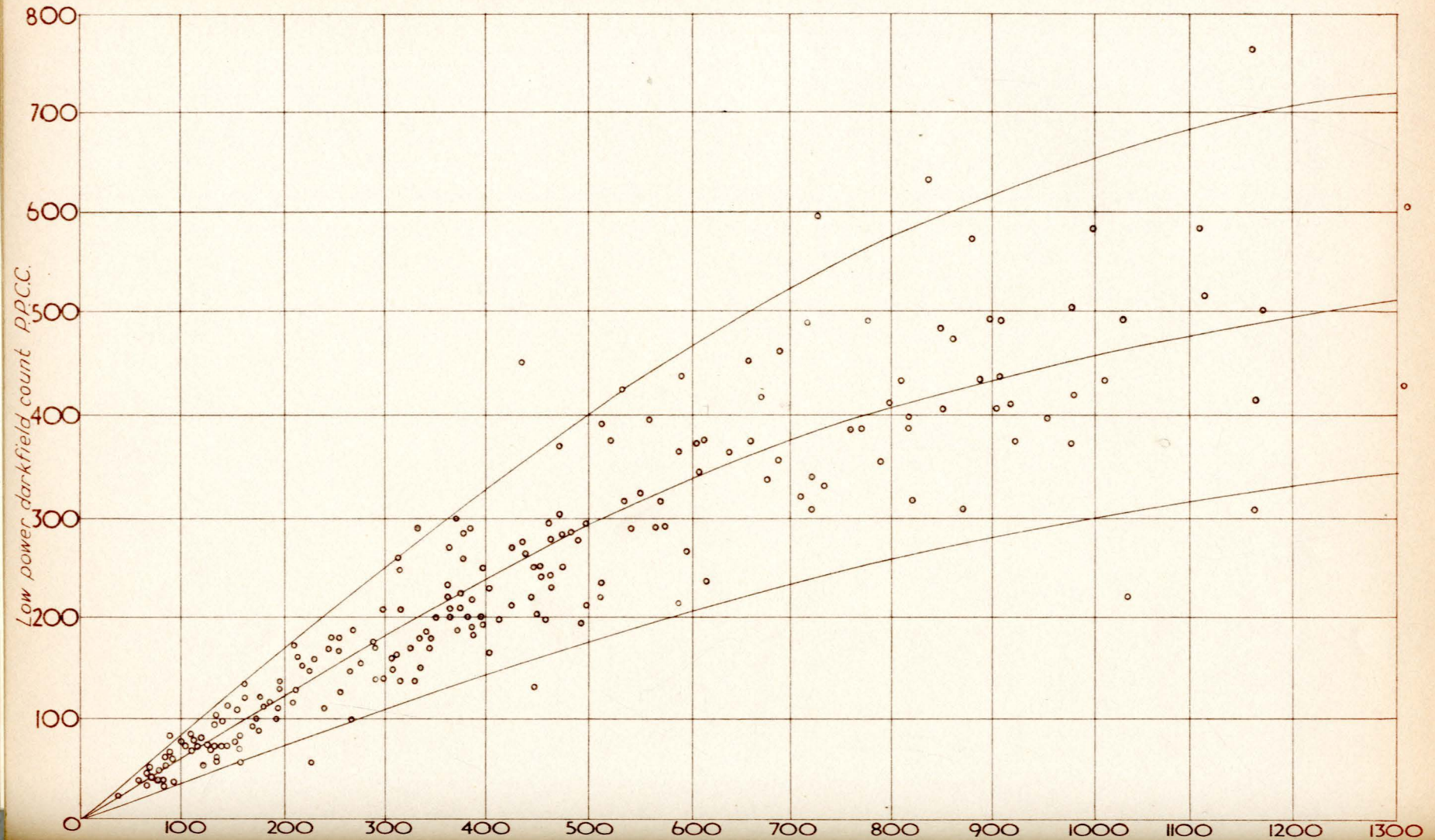
It was important to know the size of the smallest particles shown by the improved technique. At the time of the experiments (1937-1939) there was no simple method available for determining these sizes directly.

A slide containing particles covering a wide size range was examined using objectives

of various numerical apertures, and also high aperture objectives with their apertures reduced by diaphragms placed behind the back lens. The numbers of particles visible with each aperture was counted. The slide was then examined under the best conditions and the size of each particle which could be measured accurately was determined using an eyepiece micrometer. From these two sets of observations the smallest size of particle detectable at various numerical apertures was determined. These results were extrapolated to numerical aperture 1.3, and this indicated that the smallest size of particle visible was 0.13 microns diameter. The same result was obtained in another way by Kitto<sup>71</sup>; he centrifuged suspensions of particles under conditions which would remove all particles of a given size, and then observed the residue under the microscope. In this way he was able to determine the sizes of the smallest particles visible under the microscope. More recently direct comparison with electron micrographs has been possible. Results in Great Britain<sup>72</sup> have confirmed that this is the

FIGURE XXI

COMPARISON OF COUNTS WITH HIGH POWER  
LIGHT-FIELD ILLUMINATION &  
LOW POWER DARK-FIELD ILLUMINATION



order of size of the smallest particles visible under the optical microscope.

## 6.2 Low power dark field illumination.

Low power objectives with dark field illumination are used for counting konimeter samples. A 16 mm. objective and xl5 eyepiece is commonly used, giving an overall magnification of xl50. The method is simpler than the high power light field technique and has been proposed for counting thermal precipitator samples<sup>70</sup>.

This system can however only show particles down to about 0.20 microns diameter, and efforts to reveal smaller particles with it have not been successful. Protaganists of this method suggest that counts by the two methods might have a fixed relationship to each other, in which case the counts by the one method could easily be converted to counts by the other method. A number of such comparison counts were therefore made and are given in Figure XXI. The mean line is drawn through the observations, and also lines cal-

culated to contain 95% of the results.

The results show -

- (a) The correlation between the two methods of counting is poor.
- (b) For a given concentration shown by one method there is a large range in the counts obtained by the other method.
- (c) As the concentration increases, the low power dark field method shows a decreasing percentage of the particles present; that is as the dust increases in danger, the low power method tends to further under-estimate the danger.

The low power dark field method of counting is therefore not sufficiently accurate for counting thermal precipitator samples.

### 6.3 Medium power dark field illumination.

With a given objective dark field illumination will show finer particles than light field illumination. Although dark field illumination with a 16 mm objective was found to be unsatisfactory, there appeared to be a possibility of using a medium power objective

and dark field illumination to show up more particles than were visible with the 2 mm. light field system.

Experiments were made to determine the best conditions for two medium power objectives; two different light sources were used. The main difficulty occurred in finding suitable sub-stage condensers, and with the 4 mm objective the condenser had to be used in oil immersion contact with the bottom of the slide carrying the dust samples. Using the best conditions for each arrangement the counts shown in Table XVIII were obtained; these counts are relative to a count of 100 of the same slide with the 2 mm light field technique.

TABLE XVIII.

Objective	Lamp	Count
8 mm apochromat; numerical aperture 0.65	8-watt substage	108
"	500-watt arc.	100
4 mm apochromat; numerical aperture 0.95	8-watt substage	135
"	500-watt arc.	156

With the 8 mm objective the arc lamp gives too bright a background resulting in a slight loss of particles, but with the 4 mm objective the extra light is required due to the higher magnification.

Experiments were also made with a 4 mm objective of the Chapman-Aldridge type. This is fitted with an annular mirror around the objective, this reflects light down onto the particles from above and produces a type of dark field illumination. This system gave a relative count of only 52 particles.

Although medium power dark field illumination has been proved to show more particles than can be seen with the best 2 mm light field conditions the method is no longer of great interest due to the advent of the electron microscope. However the fact that the 8 mm objective shows approximately the same number of particles as the 2 mm light field method is of some use. Samples from the standard thermal precipitator are counted using the latter method, but this cannot be used for samples from the modified thermal precipitator

since these are collected on a thicker slide, and the 2 mm objective cannot focus through this. The 8 mm objective with dark field illumination can however be used with the thicker slide and this enables equivalent counts to be obtained.

#### 6.4 High contrast photomicrographic methods.

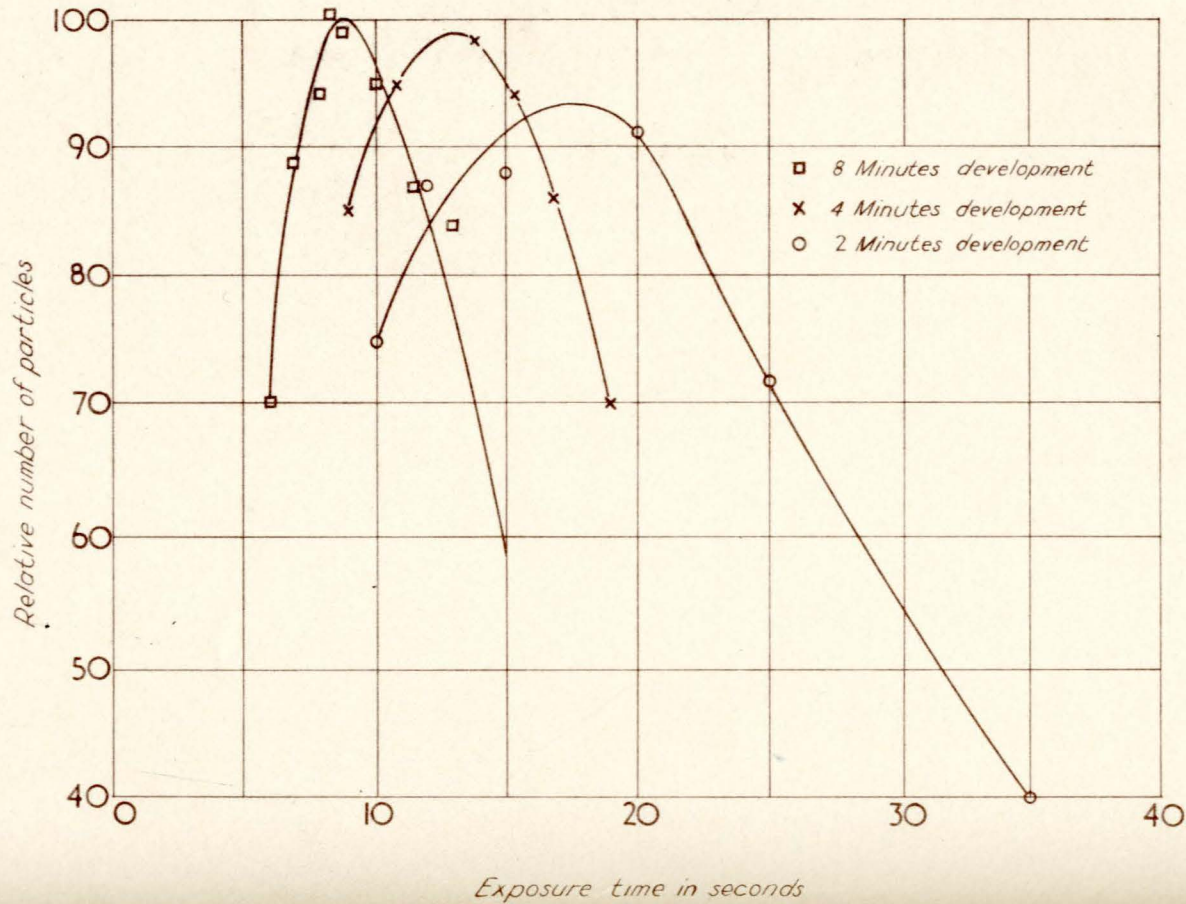
One of the principal factors controlling the visibility of small particles under the optical microscope is that with decrease of particle size the intensity of the diffraction rings decreases until it is so close to the intensity of the background that the difference cannot be detected by the human eye.

One method of revealing smaller particles is therefore to use high contrast photographic methods to detect differences of intensity smaller than can be seen by eye.

Experiments were made to show the best conditions for obtaining such photographs, and the results were published<sup>73</sup>. The investigations covered the best combination of microscope conditions, the most suitable

FIGURE XXII

EFFECT OF DEVELOPMENT AND EXPOSURE TIMES  
ON NUMBER OF PARTICLES PHOTOGRAPHED



plates and developer, the best combination of exposure and development times, and the effect of different wavelengths.

In general the best microscope conditions were those which had been found best in the research done by visual observations (section 6.1), except that the lowest possible eyepiece magnification should be used; in photomicrography the plate must be placed some distance away from the eyepiece in order to produce a reasonable size picture, this introduces additional magnification and this is presumably the reason why a lower eyepiece magnification is suitable. With the particular projection microscope used for these investigations the eyepiece used was X6 and the projection distance 42 cms.

Ilford thin film half tone plates developed in a sodium-hydroxide/hydroquinone developer showed the greatest number of particles, due to their high contrast ( $\gamma$  about 6).

The effect of various exposure and development times is shown in Figure XXII.

Photographs were taken at wavelengths

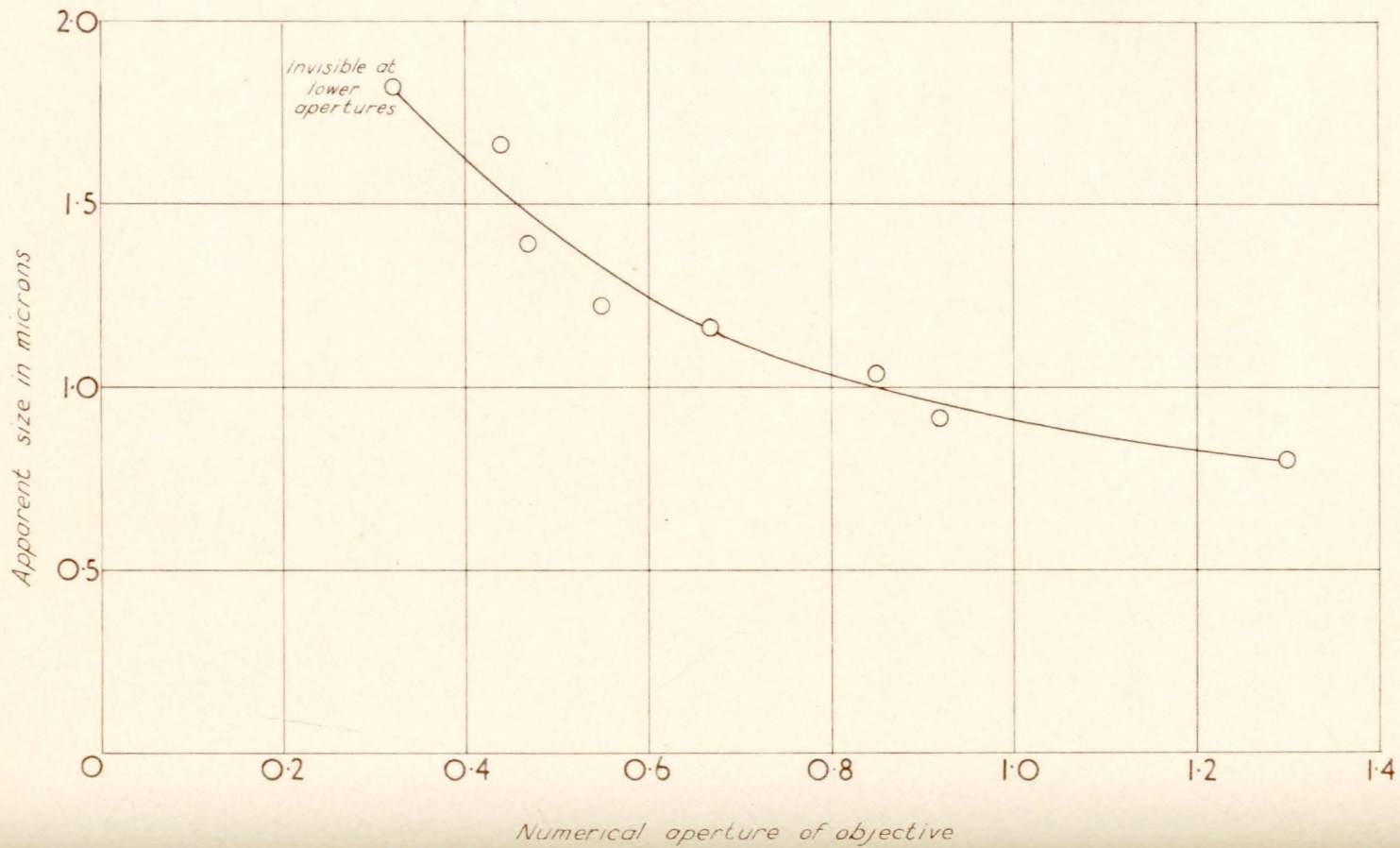
down to 4,000 Angstrom units, and it was found that the shorter the wavelength the greater the number of particles revealed. Apparatus for experiments with ultra-violet light was not available.

Using these photomicrographic methods the number of particles made visible was about twice the number which could be seen visually in the same field; the size of the smallest particles made visible was believed to be slightly less than 0.10 microns diameter. This method was used to investigate the number of particles of this size in dusts produced by various mining processes<sup>74</sup>.

During the course of these experiments two interesting subsidiary investigations were made. It was found that the focus had to be exact to show the maximum number of particles. A special slow motion drive was fitted to the normal fine focussing adjustment of the projection microscope, and photographs were taken in which the focus was changed by 0.1 microns after each exposure. The results showed that a change of this amount was

FIGURE XXIII

APPARENT SIZE OF PARTICLE AT  
DIFFERENT NUMERICAL APERTURES



sufficient to make the finest particles disappear completely, in other words that the depth of focus was less than this amount. This is less than the amount postulated by theory. A note on this observation was published<sup>75</sup>.

The second investigation was concerned with the apparent size of particles photographed with objectives of different numerical aperture. The sizes of the same particle as measured from the photographic plate at different apertures are shown in Figure XXIII. As the numerical aperture decreases the apparent size of the particle increases until the particle finally becomes invisible when it is so large and diffuse that there is insufficient contrast to distinguish it from the background. A theory of image formation of fine particles has subsequently been published<sup>76</sup> which explains this observation.

#### 6.5 Phase contrast microscopy.

Comparison counts have been made of the same slide with a phase contrast 2 mm

objective, and with the standard technique used for thermal precipitator samples. No increase in counts was obtained.

Phase contrast methods however appear to offer possibilities of identifying the nature of smaller particles than can be achieved with standard petrological methods or the Becke line technique, but this subject falls outside the scope of the present work.

#### 6.6 Electron microscopy.

The experiments described in sections 6.1 to 6.4 above were made before 1940 when some method of determining the number and sizes of particles below the existing limits of the optical microscope were urgently required. Electron microscopes were not then commercially available.

The advent of the electron microscope has enabled the range of investigation of dust particles to be extended by several orders of size. A small amount of work has been carried out overseas on mine dusts<sup>77,78,79</sup>.

An electron microscope has been installed at the Research Laboratories of the Transvaal Chamber of Mines. Before it could be used to investigate mine dusts a method of collecting such dust in a form suitable for examination had to be developed. The supporting film had to be sufficiently robust to withstand handling in the mine and yet be transparent to the electron beam; it also had to withstand heating to about 500°C to remove carbon and other particles from the sample and be free of crystalline structure so that electron diffraction studies could be made of the dust particles.

The standard collodion film used in electron microscopy cannot be heated to the required temperature. After experimenting with various films the following technique was evolved.

The ordinary copper supporting grid is given a thin coating of aluminium by evaporating the aluminium in a vacuum. A moderately thick collodion film is then cast on this in water in the usual way<sup>80</sup>. The grid with

the collodion film is then placed in a vacuum chamber and a thin film (approximately 100 Angstrom units thick) of silicon monoxide deposited on it by evaporating the silicon monoxide on a suitable filament nearby.

The grid with the two films on it is then taken underground and the sample collected in a specially modified thermal precipitator. The grid is kept oscillating about the hot wire in the thermal precipitator during the sampling process so that each area of the sample contains a representative sample of the dust. This is necessary since the area which can be examined in the electron microscope is very small.

On return to the laboratory the grid is heated under controlled conditions. This heating removes the unwanted carbon particles and organic matter present in the dust sample, burns off the collodion supporting film and converts the silicon monoxide into a form of silicon dioxide which shows no crystalline structure. The specimen is then shadow-cast

FIGURE XXIV.

ELECTRON MICROGRAPH OF DUST FROM

A WITWATERSRAND GOLD MINE.



1 Micron

1 Micron

Magnification x 25,000

Shadowed with gold-palladium at  $10^{-13}$ .

and examined in the electron microscope. The thin layer of aluminium on the grid appears to improve the adhesion between the bars of the grid and the silicon dioxide film.

Figure XXIV is an electron micrograph of dust from a Witwatersrand gold mine. It will be seen that there are many particles below the limit of visibility of the optical microscope. The nature of these particles and their possible danger to health remains to be investigated.

#### 6.7 Automatic counting of particles.

The counting of particles under the microscope by eye is a laborious process, and as shown in section 4 has various errors associated with it.

Automatic methods of doing this have received considerable attention in recent years, particularly in Great Britain<sup>81, 82</sup>. The basic principle of these methods is to scan the dust sample with a beam of light and arrange that each time the beam of light strikes a particle its presence is recorded electronically. By measuring the time the

beam of light is in contact with the particle, or by measuring the intensity of the scattered light, or in other ways, the sizes of the particles may also be determined.

This method was suggested by the author<sup>83</sup> prior to the publication of the overseas experiments but facilities for building the apparatus were not available. Later the author and a colleague, Mr. R.T. Jamieson commenced construction of such an apparatus but the work was abandoned as the apparatus is now available commercially.

This type of apparatus will have several advantages over the optical method of counting dust samples -

- (i) The human factor is eliminated.
- (ii) A much larger fraction of the sample, or the whole sample, can be examined, thus reducing the errors which are unavoidable if only a small fraction of the sample is counted.
- (iii) The samples can be assessed more rapidly - counting rates of 1000 par-

particles per second or higher can be used; with human counting 1 to 2 particles per second is the maximum rate.

At present these devices are still in the experimental stage, they will require skilled maintenance, and their cost is high. They are unlikely to be suitable for use by personnel on the mines, and an alternative method of assessing samples is described in the next section.

---

## 7. A PHOTO-ELECTRIC METHOD OF ASSESSING DUST SAMPLES

The method of assessing a dust sample by an observer counting under a microscope the number of particles present in it has several disadvantages :-

- (a) The process is laborious and time-consuming.
- (b) Observers require lengthy training and considerable experience before they become proficient.
- (c) The results are subject to variations due to the human factor.
- (d) Usually only a small fraction of the whole sample can be examined, and this may not be representative. This leads to unavoidable errors in the result.
- (e) There is considerable doubt if particle number is an accurate indication of the danger to health of the dust.

Some of these matters have been discussed earlier in this thesis.

### 7.1 Basic theory of the photo-electric apparatus.

There is a need for a method of

assessing dust samples which is rapid, consistent and simple to use, which eliminates the human factor, and which measures a property of the dust closely related to its danger to health. In section I it was suggested that the parameter measured should be the surface area of the particles retained in the lung with over-estimation of particles in the region 1 to 2 microns, and under-estimation of the particles below 0.5 microns. The modified thermal precipitator has been shown to sample dust approximately in proportion to the retention of the various sizes in the lung; parallel to the development of the modified thermal precipitator, a photoelectric apparatus was designed and constructed to enable the samples to be measured rapidly and conveniently. It was expected that an instrument measuring the light extinguished by the dust particles in the sample would provide a reading related closely to the surface area of the particles.

Richardson<sup>84</sup> showed that the light extinguished from a beam by a collection of

dust particles placed in the beam was proportional to their projected surface area when the particles were large compared with the wavelength of the light, but that as the particle size decreased, and approached the wavelength of the light, the light extinguished per unit surface area increased. The solubility of particles per unit surface area also increases over the same range of size. The increased solubility per unit surface area, and the increased amount of light extinguished per unit surface area, with decrease of particle size down to the wavelength of the light used, are approximately equal. The possibility of using this relationship to provide a useful method of assessing dust samples was pointed out by the author<sup>85</sup>

When the particle size is small compared with the wavelength of the light, the Rayleigh law of scattering applies, i.e. the scattering is proportional to the sixth power of the diameter, and the light extinguished per unit surface area falls rapidly to effectively zero.

Since visible light has a mean wavelength of approximately  $\frac{1}{2}$  micron this implies that apparatus measuring the extinction of visible light would give a measurement of the surface area of the particles, with an over-estimation of the surface area of particles slightly larger than the wavelength of light, i.e. particles of the order of 1 micron diameter and with a rapidly increasing under-estimation of the surface area of particles below 0.5 microns. As was shown in Section 1 of this thesis, this would reproduce qualitatively the toxicity of silica particles. At present there is insufficient pathological evidence to place this relationship on a quantitative basis.

This relationship forms the basis of the apparatus described below.

The laws of scattering and extinction of light by small particles (of the order of size equal to the wavelength of the light) are complex. They have been investigated by Rayleigh<sup>86</sup>, Mie<sup>87</sup>, and more recently by Sinclair<sup>88</sup>, and many others. Tables of

scattering function have been published<sup>89</sup>

Much of this work applies only to spherical particles, and the laws can only be applied in practice if the particles are homogeneous in composition, size and shape, if the refractive index is known, if the light is monochromatic, and if the geometry of design of the apparatus for measuring the amount of scattering or extinction is specified.

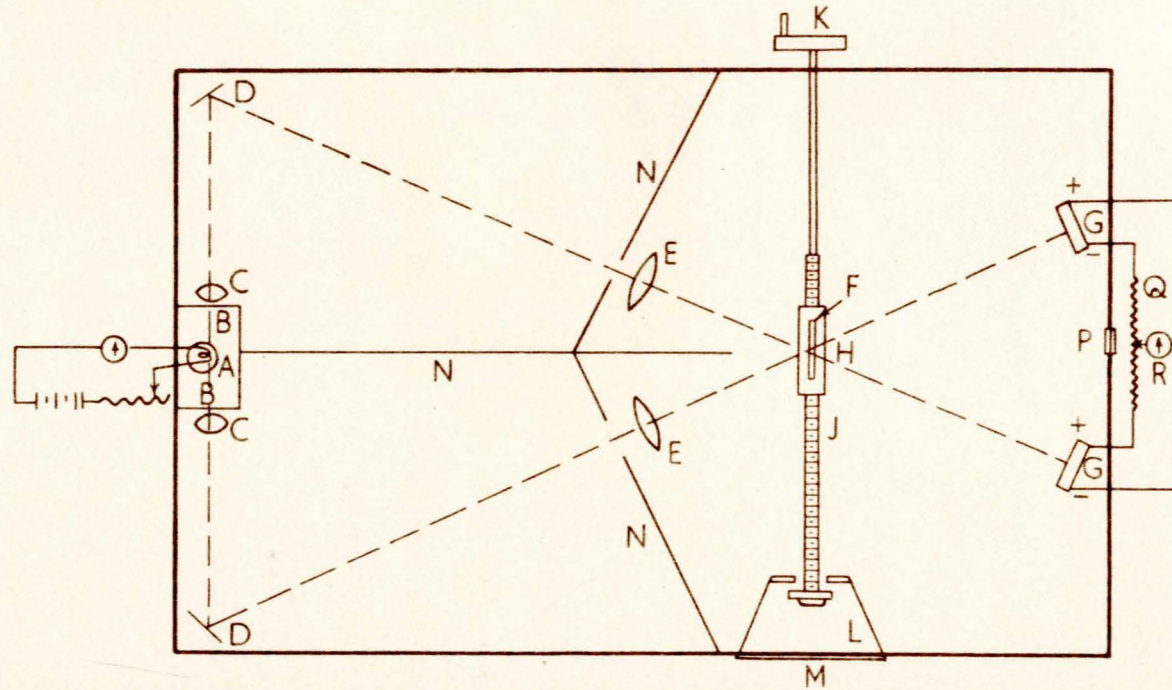
These requirements regarding the particles do not hold in the case of mine dusts, and there appeared to be no possibility of calculating the design and performance of an instrument with predetermined measuring characteristics. Instead various experimental designs were tried on an optical bench, their characteristics measured, and a final design constructed in a form suitable for use by mining personnel. The actual property of the dust measured in this apparatus was then determined in a series of calibration tests.

## 7.2 Description of the photo-electric apparatus.

The basic construction of the apparatus

FIGURE XXV

PHOTO-ELECTRIC APPARATUS FOR ASSESSING DUST SAMPLES



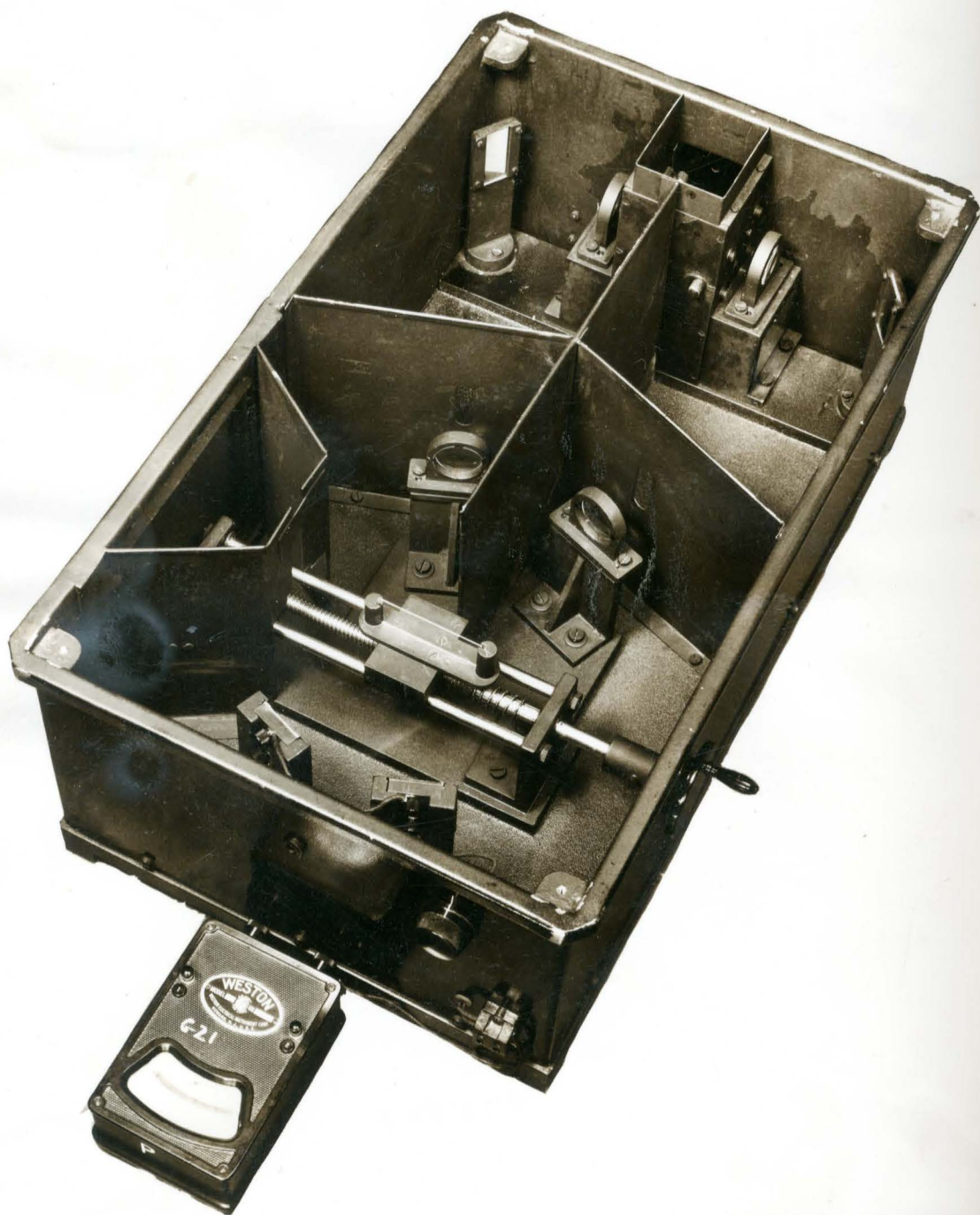
is shown diagrammatically in Figure XXV.

Figure XXVI is a photograph of the apparatus.

The apparatus is built on a heavy base plate with light-tight thin metal sides and lid. All internal metal surfaces are painted matt black to minimize stray light. The apparatus is 24" long, 15" wide and 8" high.

The lamp A has a coiled vertical filament, approximately  $3/8$ " high and 0.04" wide. It is mounted on a base which can be moved in three dimensions or tilted so that when a lamp is changed adjustment can be made to compensate for slight differences in the position of the filament. The lamp is rated at 8.5 volts, 35 watts, but is under-run slightly to increase the life. It must be fed by a stable supply; either a 12-volt car accumulator or a constant voltage transformer has been found suitable. The circuit includes a 3-ohm, 10-ampere rheostat and an ammeter. It has been found essential to solder the leads to the base terminals of the lamp to ensure a steady contact. The

FIGURE XXVI  
THE PHOTO-ELECTRIC APPARATUS FOR ASSESSING  
DUST SAMPLES.



lamp housing includes a louvred ventilator.

Two indential beams of light from the lamp pass through the apparatus. Adjustable diaphragms at B determine the dimensions of the beams which then pass through lenses C (focal length 2") and are reflected by mirrors D and refocussed by lenses E (focal length 4"). The beams pass close to each other through a glass slide F (which carries the dust samples) and fall onto barrier-layer photocells G.

The glass slide is carried on a support H which is moved laterally by the screwed rod J and handle K. The lid is sealed onto the apparatus so that the optical arrangements cannot be interfered with, and the slideholder H is therefore designed to travel out into a compartment L when the slide is to be changed. This compartment has a light-proof door M. Inside the box are baffles N to reduce stray light. A small observation hole P enables the glass slide to be observed.

The two photo-cells are connected in opposition through a 25-ohm potentiometer Q and a pointer type galvanometer R which has a

sensitivity of 4 millimetres per microampere, a resistance of approximately 150 ohms and a periodic time of  $2\frac{3}{4}$  seconds. It has a centre zero.

The apparatus has been made heavy to prevent accidental knocks upsetting the optical arrangements. It has been made fairly long since the photocells have a temperature coefficient and their output may alter if they are heated unduly by the lamp; it was found that the use of heat-absorbing filters did not reduce their drift appreciably.

The illumination at the dust deposit must be evenly distributed. This is achieved by the Kohler system of illumination, i.e. lens C throws an image of the lamp filament onto lens E, and lens E throws an image of the surface of lens C onto the slide H - since all parts of lens C received light from all parts of the lamp filament its image is evenly illuminated. The dimensions of the lamp filament and the diaphragm B are such that, taken with the other dimensions of the apparatus, the image of the diaphragm B thrown

on the dust slide is the same height as, and slightly wider than, the dust samples on the slide.

The photocells receive an out-of-focus image of the light filament. Their effective area is controlled by masks so that this image fills their surface. Any light from the beam scattered by the dust particles is therefore not recorded by the photocells.

Matched pairs of photocells were selected, but despite this and the general symmetry of the two beams, it was found necessary to incorporate the potentiometer to enable the galvanometer reading to be brought to zero when both beams of light traverse a blank portion of the glass slide. There are day-to-day variations in the galvanometer zero reading as well as a gradual drift during a day. This is adjusted by resetting the potentiometer. The sensitivity of the apparatus, i.e. the deflection of the galvanometer for a given amount of dust, is controlled by adjusting the size of the diaphragms B, and by varying the brightness of the lamp.

Two beams of light are used; this compensates to a large extent for slight variations in lamp brightness during a series of measurements, and is also necessary because the surfaces of the glass slide often scatter as much light as the dust particles. When a measurement is made, one beam of light passes through the dust sample and glass slide, while the other passes only through the glass slide nearby - variations from average in the reflectivity of the glass and in the amount of absorption of light in the glass are thus automatically compensated for and the difference in the intensity of the two light beams is therefore due to the light scattered or absorbed by the dust particles.

### 7.3 Operation of the photo-electric apparatus.

The lamp is switched on at least 15 minutes before it is desired to measure a sample. This allows the photocells to reach a fairly stable output after which drift is greatly reduced. The lamp current is adjusted to 4.0 amperes.

The glass slide is inserted into the slide holder and by turning handle K is brought into a position where both beams of light traverse portions of the slide which have no dust deposited on them. The potentiometer is then adjusted to bring the galvanometer reading to zero.

The slide is moved until the first dust sample is seen through the observation hole to be in the centre of one of the light beams; this position is judged by observation and the slide is then moved slowly from side to side until the galvanometer reading is a maximum; this reading is noted. The same dust sample is then brought into the other beam and the process repeated, with a galvanometer reading being obtained on the other side of zero. The two galvanometer readings added together give the assessment of the dust sample. The next dust sample on the slide is then measured in the same way. Each reading takes about 10 seconds to make, compared with 10 to 20 minutes normally required to count a thermal precipitator sample.

An alternative method of making the measurement is to use the potentiometer to bring the galvanometer pointer to zero when the dust sample is in one beam. This "null" method appeared to have no advantages over the method described above, and a longer time is needed to obtain the reading.

#### 7.4 Consistency of measurements.

The whole dust sample is evaluated in this apparatus. There is thus no error such as occurs in microscope counting where only a fraction of the whole sample is examined and assumed to be representative of the whole. In the studies reported in section 4.3 of this thesis, this source of error was found to have a percentage standard deviation of 3.3%.

When the same observer counts the same thermal precipitator sample a number of times under the microscope it was shown in section 4.3 that the counts have a percentage standard deviation of 6.6%. One observer made 45 measurements of the same dust sample in the photo-electric apparatus and the percentage standard deviation of the readings was

0.7%. There was no sign in the data that the observations became erratic with time, i.e. there is no indication of a fatigue effect such as was evident when repeated microscope counts were made.

When different observers count the same slide under the microscope it was found that their counts varied on the average by 8% when both observers were experienced, and by up to 13% when one was inexperienced. Five observers measured the same sample in the photo-electric apparatus, and their measurements were 23.3, 23.4, 23.4, 23.5 and 23.5. The percentage standard deviation is 0.4%. The second and fifth measurements respectively were made by observers with no previous experience of the apparatus; in microscope counting it is found that inexperienced observers tend to obtain significantly lower counts than trained observers. New observers can be trained to operate the apparatus in about 10 minutes.

The various sources of error inherent in microscope counting are therefore largely eliminated in this apparatus. Readings on it

are consistent to less than 1%.

#### 7.5 Calibration of the photo-electric apparatus.

It was important to determine how the measurements in this apparatus were related to the surface area of the particles in the dust sample. It was also desired to know if the readings were dependent to any appreciable extent on the material from which the dust was produced; it was possible that the refractive index of the particles might affect the results significantly, and mine dusts may be produced from a variety of minerals.

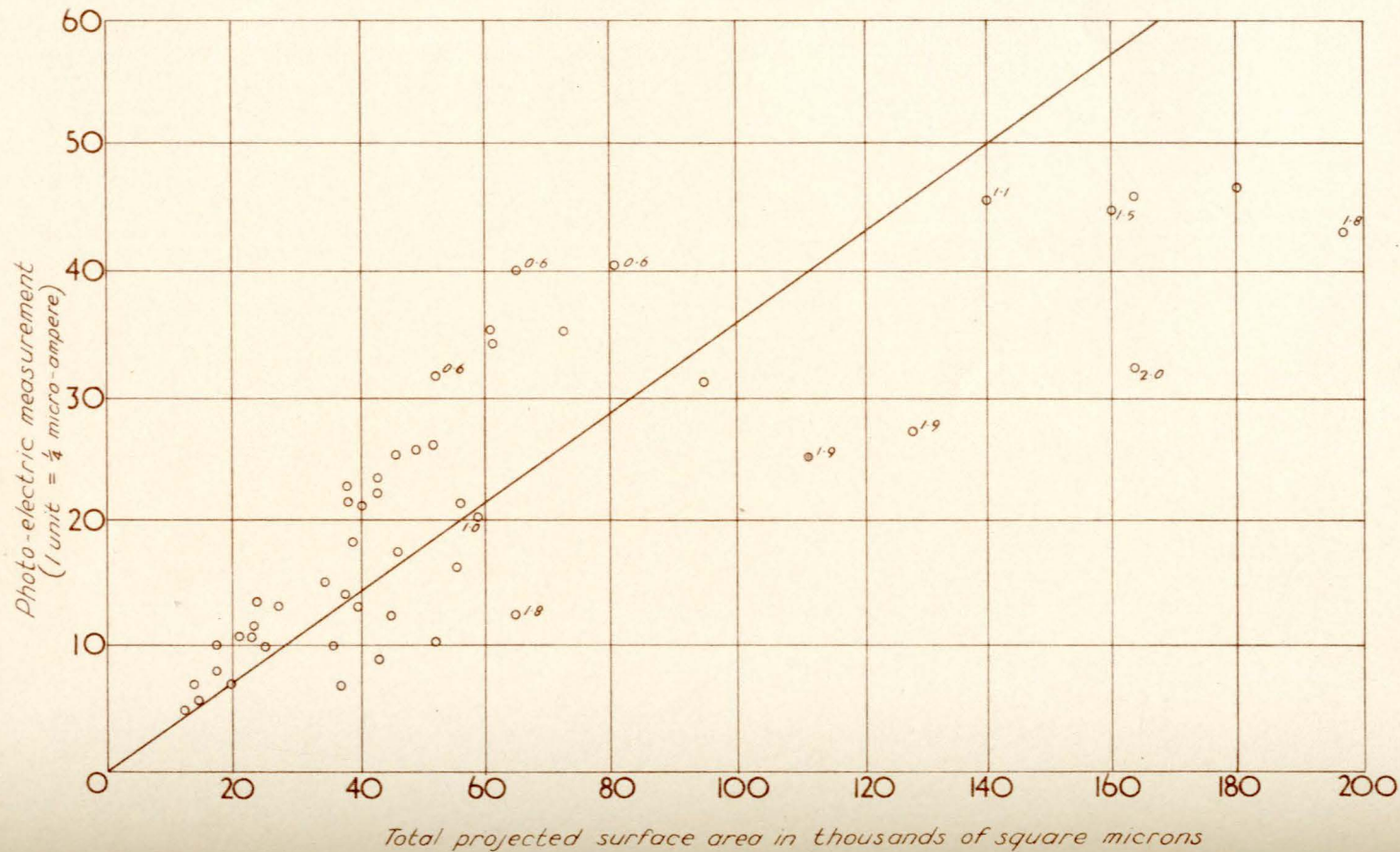
It is not practicable to produce dust clouds, of say quartz, in which all the particles are of uniform size. All that can be done is to produce clouds which are generally fine or generally coarse, but the particles in each type cover a wide range of size, and these ranges invariably overlap to some extent.

Samples of pure quartz dust of widely differing size distribution and number concentration were collected by modified thermal precipitator and evaluated in the photo-electric

FIGURE XXVII

RELATIONSHIP BETWEEN PHOTO-ELECTRIC MEASUREMENT  
& TOTAL PROJECTED SURFACE AREA OF PARTICLES

*N.B. Figures next to certain points show average size of particles in the sample*



apparatus; four or five measurements were made of each sample, and these invariably agreed to better than 1%. The dust samples were then examined under the microscope and the number and size of particles estimated as in the standard method of dust counting. About 1,000 particles were counted and measured in each sample.

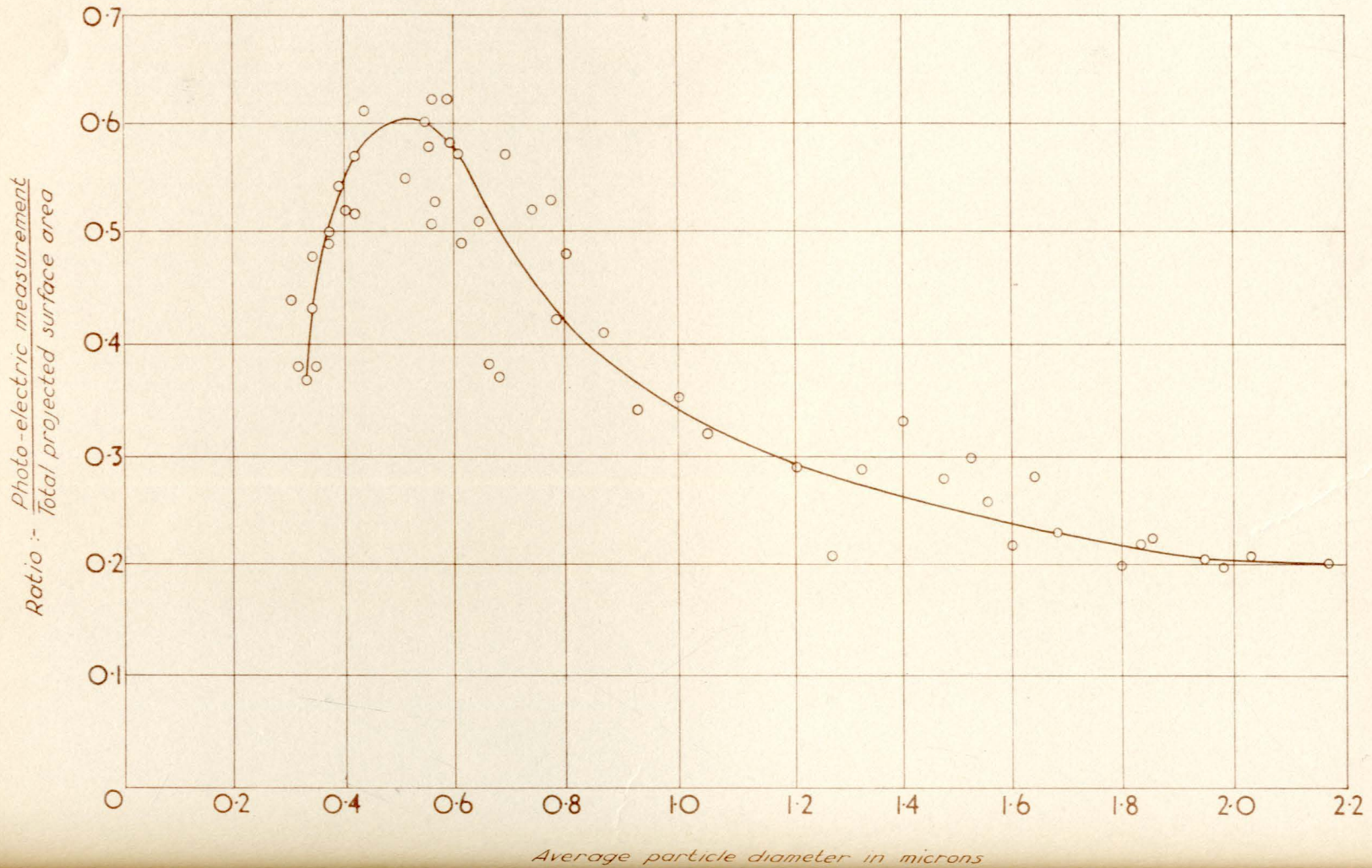
The total projected surface area of all the particles in the sample was then calculated from these observations. It should be noted here that although the microscope method leads to a determination of the projected surface area of the particles, this is related by a constant ( $\frac{1}{2}$ ) to the total surface area of the particles, even if the particles deviate from a spherical shape by a considerable amount<sup>90</sup>.

The calculated projected surface area and the photo-electric measurement for 52 such experiments are plotted in Figure XXVII.

Although there is some indication of a relationship, the scatter about the mean line is considerable. The average size of particle in each sample was then calculated using as a

FIGURE XXVIII

RELATIONSHIP BETWEEN RATIO OF PHOTO-ELECTRIC  
MEASUREMENT OVER TOTAL PROJECTED SURFACE  
AREA AND AVERAGE PARTICLE SIZE



measure of mean particle size, as is customary in such work,  $\frac{\sum n d^2}{\sum nd}$  where  $n$  is the number of particles of diameter  $d$  in the sample.

It was found that points lying above the mean line resulted from experiments in which the average particle size was small, while points lying below the mean line were for coarse dusts. The average particle sizes are shown next to a few points in Figure XXVII to illustrate this.

The ratio  $\frac{\text{Photo-electric reading}}{\text{Projected surface area}}$  was then calculated for each experiment. The value of this ratio in each experiment is plotted against the average particle size in Figure XXVIII. Deviations from the general trend are probably due mainly to errors in measuring the sizes and numbers of particles under the microscope, since the accuracy of this type of work is low.

The results in Figure XXVIII indicate that for samples of coarse dust (average diameter greater than 1.8 microns) the photo-electric reading is directly proportional to the surface area. As the size decreases the

photo-electric reading per unit surface area increases and reaches a maximum of nearly three times the normal value when the average particle diameter is in the range 0.5 to 0.6 microns diameter. Below 0.4 microns the extinction per unit surface area decreases sharply as expected from the theory of light scattering.

The size at which maximum over-estimation of surface area occurs is somewhat smaller than that desirable from a consideration of the toxicity of silica dusts of different sizes (see Section 1). However the whole curve could be moved to the right by using light of longer wavelength, i.e. light in the red or near infra-red region of the spectrum. However in the absence of more precise quantitative data on the toxicity of particles this step is not yet regarded as warranted. It would not involve any major alterations to the present form of the apparatus.

Extinction factors for each particle size were taken from Figure XXVIII and used to convert the measured surface areas in the original experiments to "extinction surface

FIGURE XXIX

RELATIONSHIP BETWEEN EXTINCTION SURFACE AREA AND  
PHOTO-ELECTRIC MEASUREMENT FOR QUARTZ DUST

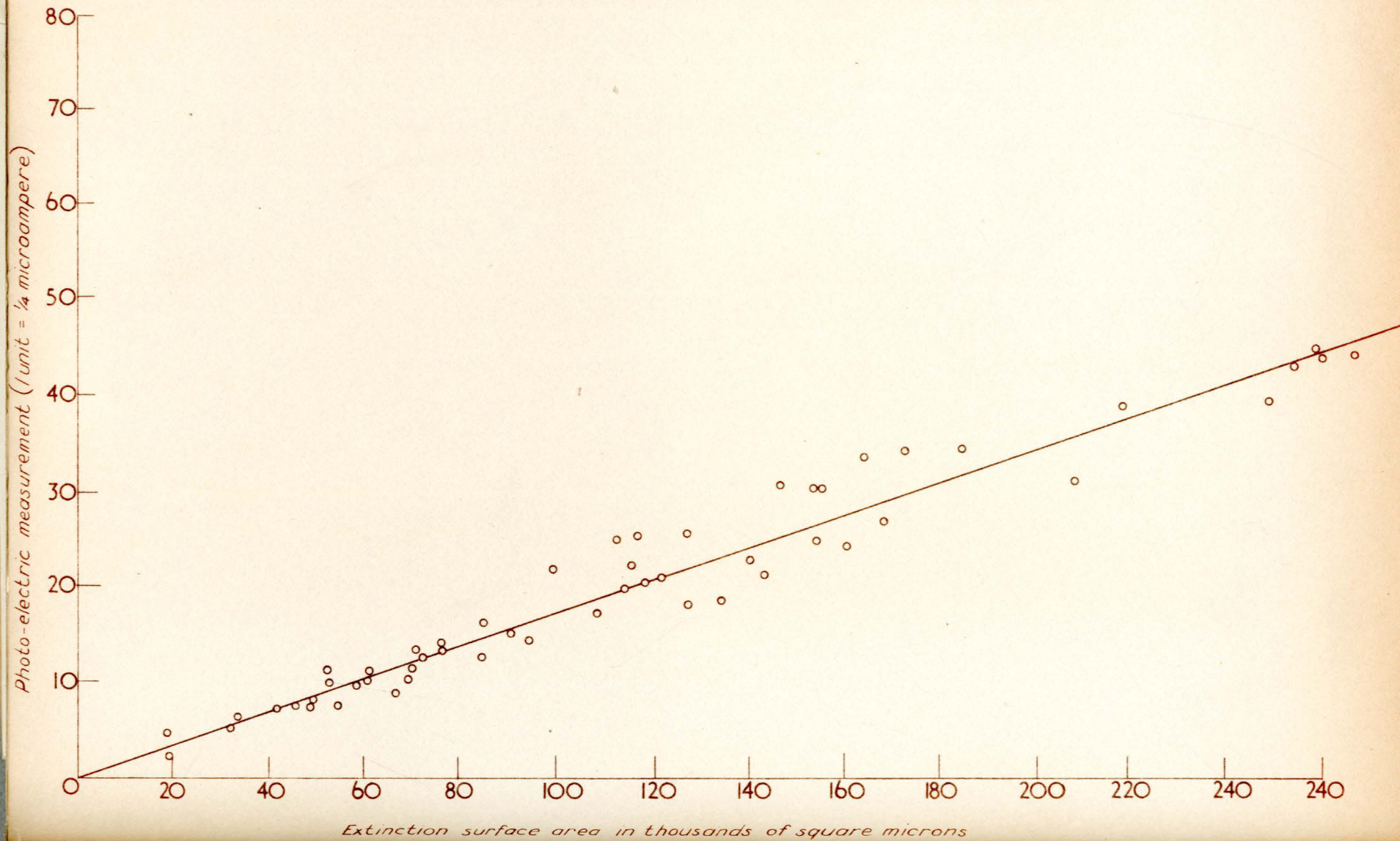
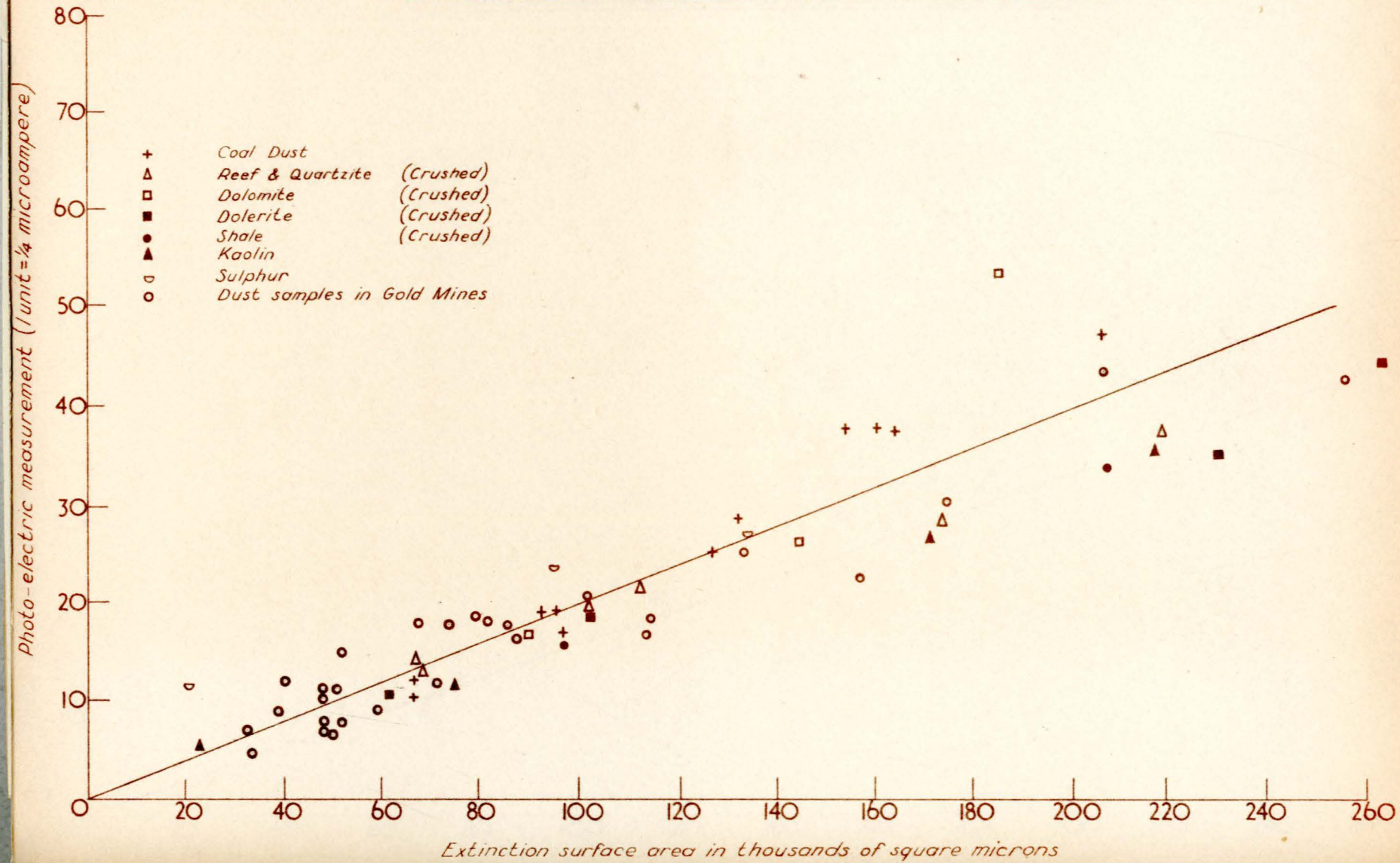


FIGURE XXX

RELATIONSHIP BETWEEN EXTINCTION SURFACE AREA &  
PHOTO-ELECTRIC MEASUREMENT FOR MISCELLANEOUS DUSTS



areas". The total extinction surface area for each sample was then calculated and is plotted against the photo-electric reading in Figure XXIX. Although there is some scatter about the mean line the correlation is satisfactory and the relationship appears to be linear within the range of concentrations tested. This range covers the conditions normally found in Witwatersrand gold mines.

Similar experiments were made using dusts produced from a variety of other substances - coal, kaolin, sulphur, and various minerals, - as well as dusts obtained in gold mines (a mixture of quartz and various silicates). The weighting factors to obtain the extinction surface area of these samples were taken from the experiments on pure quartz. Figure XXX shows the relationship between the photo-electric reading and the extinction surface area. It will be seen that the mean line through the points agrees closely with that obtained for quartz, and apart possibly from the results with sulphur there is no evidence that any of the substances tested show a

markedly different relationship to the others.

These results show that the measurement of the dust samples obtained in this photo-electric apparatus provides basically a measure of surface area of the particles, with an over-estimate of the surface of particles between 0.5 and 2 microns, believed to have an enhanced toxicity per unit surface area, and with an under-estimate of the surface of particles below about 0.5 microns diameter, which are believed to have a lower toxicity per unit surface area, and which probably play a minor role in the production of silicosis due to their comparatively rapid disappearance from the lungs by complete solution. This measurement is probably a more accurate assessment of the silicosis hazard of a dust than any previously available measurement.

#### 7.6 Advantages of the photo-electric method of assessing dust samples.

- (i) Repeated measures of the same slide are more consistent (percentage standard deviation less than 1%) than repeated measurements of the same slide under the

microscope (percentage standard deviation about 7%).

- (ii) Inter-observer variations are less (0.4%) than inter-observer variations with the microscope (8%). The human factor is practically eliminated.
  - (iii) The whole sample is assessed. With microscope methods only a fraction of the sample can be examined and unavoidable errors are thus introduced.
  - (iv) Samples can be assessed very rapidly (say 10 seconds); the usual microscope examination of a thermal precipitator sample takes 10-20 minutes.
  - (v) The property of the sample measured is believed to be a better measure of the danger to health of the dust than is measurement of the number concentration.
-

REFERENCES.

1. AGRICOLA, G. "De re metallica", A.D.1556.  
English translation by Hoover and Hoover, 1912, page 6.
2. GYE, W.E. and PURDY, W.J. "The poisonous properties of colloidal silica".  
Br.J. Exper. Path, 3, 75, 1922 and 5, 238, 1924.
3. GYE, W.E. and KETTLE, E.H. "Silicosis and miners phthisis" Br. J. Exper. Path., 3, 241, 1922.
4. GARDNER, L.U. "Studies on the relation of mineral dusts to tuberculosis".  
Am. Rev. Tuberc, 7, 344, 1923.
5. KETTLE, E.H. "Interstitial reactions caused by various dusts". J. Path. and Bact, 35, 395, 1932.
6. KING, E.J. "Solubility theory of silicosis".  
Occ. Med, 4, 26, 1947.
7. DALE, J.C. and KING, E.J. "Acute toxicity of mineral dusts" A.M.A. Arch. Ind. Hyg. and Occ. Med., 7, 484, 1953.
8. BEILBY, G. "Aggregation and flow of solids".  
McMillan, London, 1921.
9. KING, E.J., MOHANTY, G.P., HARRISON, C.V. and NAGELSCHMIDT, G., "Effect of modifications of the surface of quartz on its fibrogenetic properties in the lungs of rats". A.M.A. Arch. Ind. Hyg. and Occ. Med., 7, 455, 1953.
10. NAGELSCHMIDT, G. "Mineralogical aspects of pneumoconiosis research".  
Research, 2, 170, 1949.

11. HOLT, P.F. and OSBORNE, S.G. "Formation of silicotic tissue". Nature, 171, 892, 1953.
12. HOLT, P.F. and OSBORNE, S.G. "Studies on the nature of silicosis. The effect of silicic acid on connective tissue". Br. J. Ind. Med., 10, 152, 1953.
13. EVANS, S.M. "Tissue responses to physical forces - the pathogenesis of silicosis". J. Ind. Hyg. and Toxic., 30, 353, 1948.
14. HEFFERNAN, P. "What is silicosis?" Tubercule, 29, 169, 1948.
15. JONES, W.R. "Silicosis". J. Chem. Met. Min. Soc., S.A. 33, 99, 1933.
16. KITTO, P.H. and PATTERSON, H.S. "The rate of solution of particles of quartz and certain silicates". J. Ind. Hyg. and Toxic., 24, 59, 1942.
17. McCRAE, J. "The ash of silicotic lungs". Publications of the S.A. Institute for Medical Research, No.1 1913.
18. BEADLE, D.G. Contribution to discussion on "Certain dusts produced by mining processes and in other ways" by H.S. Patterson, Trans. Inst. Min and Met., 49, 129, 1939.
19. COLLET, E. "Researches with the electron microscope on fibrosis in pulmonary silicosis" La Presse Medicale, 60, 1419, 1952.
20. VAN WIJK, A.M. and PATTERSON, H.S. "The percentage of particles of different sizes removed from dust-laden air by breathing". J. Ind. Hyg. and Toxic., 22, 31, 1940.

21. HATCH, T. and HEMMON, W.C.L. "Influence of particle size in dust exposure" J. Ind. Hyg. and Toxic., 30, 172, 1948.
22. HATCH, T. and KINDSVATTER, V.H. "Lung retention of quartz dust smaller than one-half micron". J. Ind. Hyg. and Toxic., 29, 342, 1947.
23. LANDAHL, H.D. and HERRMAN, R.G. "On the penetration of airborne particulates in the human lung". J. Ind. Hyg. and Toxic., 30, 181, 1948.
24. DAVIES, C.N. "Dust sampling and lung disease". Br. J. Ind. Med., 9, 120, 1952.
25. WILSON, I.B. and LA MER, V.K. "The retention of aerosol particles in the human respiratory tract as a function of particle radius". J. Ind. Hyg. and Toxic., 30, 265, 1948.
26. BROWN, J.H., COOK, K.M., NEY, F.G. and HATCH, T. "Influence of particle size upon the retention of particulate matter in the human lung". Amer. J. Pub. Health, 40, 450, 1950.
27. FINDEISEN, W. "Uber das absetzen kleiner, in der luft suspendierter, teilchen in der menschlichen lunge bei der atmung". Pflugers Arch. f.d. ges. Physiol., 236, 367, 1935.
28. DAVIES, C.N. "Inhalation risk and particle size in dust and mist". Br. J. Ind. Med., 6, 245, 1949.
29. TEBBENS, B.D., SCHULZ, R.Z. and DRINKER, P. "The potency of silica particles of different size". J. Ind. Hyg. and Toxic., 27, 199, 1945.

30. KING, E.J., MOHANTY, G.P., HARRISON, C.V. and NAGELSCHMIDT, G. "The action of flint of variable size injected at constant weight and constant surface into the lungs of rats". Br. J. Ind. Med. 10, 76, 1953.
31. BEADLE, D.G. "Recent developments in dust sampling on South African gold mines". Meeting of experts on the prevention and suppression of dust in mining, tunnelling and quarrying. International Labour Office, Geneva 1952.
32. FROMAN, G. "Sampling, analysing and recording of dust in Swedish mines and quarries". Meeting of experts on the prevention and suppression of dust in mining, tunnelling and quarrying. International Labour Office, Geneva, 1952.
33. WALKENHORST, W. "Sampling, measurement and analysis of airborne dust". Meeting of experts on the prevention and suppression of dust in mining, tunnelling and quarrying. International Labour Office, Geneva, 1952.
34. WYNN, A.H.A. "The assessment of airborne dust concentrations in mines". Meeting of experts on the prevention and suppression of dust in mining, tunnelling and quarrying. International Labour Office, Geneva, 1952.
35. General report of the Miners' Phthisis Prevention Committee. Govt. Printer, Pretoria, 1916. Appendix 3.
36. Final report of the Miners' Phthisis Prevention Committee. Govt. Printer, Pretoria, 1919. Pages 10 - 12.
37. GREEN, H.L. "Recent developments in methods of sampling dusts". Trans. Inst. Min. and Met., 44, 95, 1934.
38. GREEN, H.L. and WATSON, H.H. "Physical methods for the estimation of the dust hazard in industry". Med. Res. Council Special Report Series No.199, 1935.

39. PATTERSON, H.S. "The sampling of mine dusts with the thermal precipitator". Trans. Inst. Min and Met., 49, 75, 1939.
40. AITKEN, J. Collected scientific papers of John Aitken, Cambridge, 1923, page 84.
41. PATTERSON, H.S. and CAWOOD, W. "The determination of size distribution in smokes". Trans. Far. Soc., 32, 1084, 1936.
42. WATSON, H.H. "Simplified eye-piece graticule for assessing thermal precipitator dust samples". Br. J. Ind. Med., 9, 80, 1952.
43. KITTO, P.H. and BEADLE, D.G. "A modified form of thermal precipitator". J. Chem. Met. Min. Soc. S.A., 52, 284, 1952.
44. BEADLE, D.G. "An investigation of the performance and limitations of the konimeter". J. Chem. Met. Min. Soc. S.A., 51, 265, 1951.
45. PATTERSON, H.S. "The prevention of silicosis on the mines of the Witwatersrand". Govt. Printer, Pretoria, 1937. Page 89.
46. LAMBRECHTS, J. de V., "A critical review of dust sampling methods employed on Witwatersrand gold mines". Reply to discussion. J. Chem. Met. Min. Soc. S.A., 43, 156, 1943
47. RABSON, S.R. "The performance of the circular konimeter". Paper to Mine Vent. Soc. S.A. 1946.

48. BURDEKIN, J.T. "Measurement of airborne dust clouds in mines-konimetry". Paper read at 9th International Congress on industrial medicine, London, 1948.
49. KITTO, P.H. Contribution to discussion on "An investigation of the performance and limitations of the konimeter" by D.G. Beadle. J.Chem. Met. Min. Soc. S.A. 52, 91, 1951.
50. IRWIN, J.O., ARMITAGE, P. and DAVIES, C.N. "Overlapping of dust particles on a sampling plate". Nature, 163, 809, 1949.
51. WALTON, W.H., FAUST, R.C. and HARRIS, W.J. "Electron microscopy applied to the assessment of aerosols". Porton Technical Paper, No.1., 1947.
52. BEADLE, D.G. "The shattering of dust particles by the impinger". J. Ind. Hyg. and Toxic., 21, 109, 1939.
53. ANDERSON, E.L. "The effect of certain impingement dust sampling instruments on the dust particles". J. Ind. Hyg. and Toxic., 21, 39, 1939.
54. WATSON, H.H. "A note on the shattering of dust particles in the impinger". J. Ind. Hyg. and Toxic., 21, 121, 1939.
55. DAVIES, C.N., AYLWARD, M., and LEACEY, D. "Impingement of dust from air jets". A.M.A. Arch. Ind. Hyg. and Occ. Med., 4, 354, 1951.
56. ASLETT, E.A., HART, P., and McMICHAEL, J. Proc. Roy Soc. B, 126, 502, 1939.

57. DAVIES, C.N. and AYLWARD, M. "The trajectories of heavy, solid particles in a two-dimensional jet of ideal fluid impinging normally upon a plate". Proc. Phys. Soc., 44B, 889, 1951.
58. RANZ, W.E. and WONG, J.B. "Jet impactors for determining the particle size distribution of aerosols". A.M.A. Arch. Ind. Hyg. and Occ. Med., 5, 464, 1952.
59. HAMILTON, R.J., WAINWRIGHT, T., and WALTON, W.H. "The effect of adhesive film thickness on the sampling efficiency of the konimeter". Br. J. Ind. Med., 8, 14, 1951.
60. KERRICH, J.E. "An analysis of the inter-comparison of konimeter counts". Internal report to Transvaal Chamber of Mines, 1952.
61. WHYTLAW-GRAY, R., and PATTERSON, H.S. "Smoke". Edward Arnold and Co, London, 1932. Chapter 4.
62. GREEN, H.L. "Application of the Aitken effect to the study of aerosols". Phil. Mag., 4, 1046, 1927.
63. GREEN, H.L. "Some accurate methods of determining the number and size-frequency of particles in dusts". J. Ind. Hyg., 16, 29, 1934.
64. PREWETT, W.C. and WALTON, W.H. "The efficiency of the thermal precipitator for sampling large particles of unit density". Porton Technical Paper No. 63, 1948.
65. GOCH, D.C. "Statistics of dust sampling". Internal report to Transvaal Chamber of Mines, 1943.

66. WATSON, H.H. "A system for obtaining, from mine air, dust samples for physical, chemical and petrological examination". Trans. Inst. Min. and Met., 46, 155, 1937.
67. BROWN, C.E. and FEICHT, F.L. "Size of smallest dust particles revealed by various microscopic systems". U.S.A. Bureau of Mines, Report of Investigations 3821, 1945.
68. "The pharmacology and toxicology of uranium compounds". McGraw Hill Book Co, New York, 1949, Vol. I, page 477.
69. BEADLE, D.G. and PATTERSON, H.S. "Effect of tubelength on the visibility of dust particles with an oil-immersion objective". Nature, 144, 327, 1939.
70. "Quality of Mine Air - Dust content and cooling power". Transvaal Chamber of Mines", 1947, Section I.
71. KITTO, P.H. Unpublished work for Transvaal Chamber of Mines.
72. WALTON, W.H. "The application of electron microscopy to particle size measurement." Symposium on particle size analysis. Inst. Chem. Eng. and Soc. Chem. Ind., page 69, 1947.
73. BEADLE, D.G. "The photography of fine dust particles". J. Sci. Instr., 16, 262, 1939.
74. BEADLE, D.G. Contribution to discussion on "Certain dusts produced by mining processes and in other ways" by H.S. Patterson. Trans. Inst. Min. and Met., 49, 103, 1939.
75. BEADLE, D.G. "Depth of focus of microscope objectives". Nature, 145, 1018, 1940.

76. OSTERBERG, H. and PRIDE, G.E. "Effect of particle size on the diffraction image in microscopy with narrow-coned, axial illumination". J.Opt. Soc.Am., 40, 14, 1950.
77. SHARPE, J.W. and HOUNAM, R.F. "General technique for the examination of airborne dust with the electron microscope with special reference to coal dusts". Proc. Conf. Electron Microscopy, Delft, 1950, page 186.
78. CARTWRIGHT, J. and SKIDMORE, J.W. "The measurement of size and concentration of airborne dusts with the electron microscope". Research Report No.79, Safety in Mines Research Establishment, Sheffield, 1953.
79. WATSON, J.H.L. "Applied electron microscopy". Canad. J. Res., 21A, 89, 1943.
80. DRUMMOND, D.G. "Practice of electron microscopy". J. Roy. Micr. Soc., 70, 17 et seq, 1950.
81. WALTON, W.H. "Automatic counting of microscopic particles". Nature, 169, 518, 1952.
82. DAWES, J.G. "The intercept length method for the automatic evaluation of dust samples". Research Report No. 72, Safety in Mines Research Establishment, Sheffield, 1953.
83. BEADLE, D.G. Internal report to Transvaal Chamber of Mines, 1948.
84. RICHARDSON, E.G. "Optical properties of colloidal suspensions in relationship to measurement of particle-size frequency". J. App. Phys., 11, 653, 1940.

85. BEADLE, D.G. "A possible method of estimating the relative rates of solution of fine silica particles". S.A. Science., 2, 68, 1948.
  86. RAYLEIGH, Lord. Scientific papers, Cambridge University Press, 1899, Vol. I pp 92-93; Vol. IV. p. 400.
  87. MIE, G. "Beitrage zur Optik truber Medien". Ann. Phys., 25, 377, 1908.
  88. SINCLAIR, D. "Light scattering by spherical particles". J. Opt. Soc. Am., 37, 475, 1947.
  89. "Tables of scattering functions for spherical particles". U.S.A. National Bureau of standards", Washington, 1949.
  90. KOTLER, J. "The distribution of particle sizes". J. Franklin Inst, 250, 341, 1950.
-