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ABSTRACT

In this thesis an investigation into the dependence of the thermal conductivity of a powder on ~~its~~ **particle size** is described.

Measurements of the conductivity of six aluminium powders with interstitial air pressures from atmospheric to  $10^{-5}$  cm.Hg. are reported. The conductivity of a powder is constant at higher pressures until the mean free path of the molecules of the gas becomes comparable to the size of the pore spaces of the powder. The conductivity then decreases with decreasing pressure to a small asymptotic value at low pressures. These results accord with those of previous workers. Measurements of conductivity with varying temperature are also reported and it is concluded that the heat transfer is due to conduction and that convection and radiative heat transfer are not important.

The differences in conductivity of the six powders are explained by considering the mean particle size and the porosity of the powders. A finer powder tends to have a low conductivity because a temperature discontinuity can exist at each solid-gas interface. A powder which packs to a high porosity also tends to have a low conductivity because the thermal resistance of a powder is due to the presence of the voids. It is shown that the porosity of a packed powder

depends on its particle size distribution and it is concluded, therefore, that the conductivity of a powder depends on both the mean particle size and the size distribution. This hypothesis is confirmed by further measurements on mixtures of two of the aluminium powders.

THE EFFECTIVE THERMAL CONDUCTIVITY  
OF PACKED BEDS OF  
METAL POWDER.

by

BRIAN SCARLETT.

A Thesis

Submitted to the Faculty of Science  
of the University of Durham

for the degree of  
Master of Science.

May, 1964.



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In this thesis an investigation into the dependence of the thermal conductivity of a powder on ~~its particle size~~ is described.

Measurements of the conductivity of six aluminium powders with interstitial air pressures from atmospheric to  $10^{-5}$  cm.Hg. are reported. The conductivity of a powder is constant at higher pressures until the mean free path of the molecules of the gas becomes comparable to the size of the pore spaces of the powder. The conductivity then decreases with decreasing pressure to a small asymptotic value at low pressures. These results accord with those of previous workers. Measurements of conductivity with varying temperature are also reported and it is concluded that the heat transfer is due to conduction and that convection and radiative heat transfer are not important.

The differences in conductivity of the six powders are explained by considering the mean particle size and the porosity of the powders. A finer powder tends to have a low conductivity because a temperature discontinuity can exist at each solid-gas interface. A powder which packs to a high porosity also tends to have a low conductivity because the thermal resistance of a powder is due to the presence of the voids. It is shown that the porosity of a packed powder



depends on its particle size distribution and it is concluded, therefore, that the conductivity of a powder depends on both the mean particle size and the size distribution. This hypothesis is confirmed by further measurements on mixtures of two of the aluminium powders.

READING OF THE THESIS.

In order to make reading this thesis easier, it is proposed first to explain its arrangement.

The figures, which are used to illustrate the text, are mounted facing the relevant page. If more than one figure is referred to on a single page, the figures are all bound together facing that page.

The graphs, however, are bound together in numerical order, between the Appendices and the Tables. They are mounted so that they will fold out and enable a relevant portion of the text and a graph to be studied simultaneously.

The references are arranged alphabetically by the Author's name. If there are several authors, the first name is used for this purpose. The references are numbered and the number is inserted into the text the first time the reference is mentioned. If the same reference is mentioned subsequently, it can easily be traced by the alphabetical arrangement.

Some of the tables list values which are displayed quite adequately on the graphs. It is felt, however, that some readers may wish to use the original readings for further work or manipulation and they are thus given for completeness.

CHAPTER 1.INTRODUCTION.1.1 Purpose of the Investigation

The need for an understanding of the influence of the particle size distribution of a powder on its effective thermal conductivity was first suggested because of variations in the sintering behaviour of different batches of metal powder which might have been attributable to variations in thermal conductivity.

Thermal conductivity has been used as a parameter to express heat transfer even though it is not rigorously applicable to homogeneous media, or those in which the heat transfer is not entirely by conduction.

Interest in the effective thermal conductivity of powders was first shown at the beginning of this century and many experiments have been performed since. The topic has increased in industrial importance and has found direct application in cryogenic engineering, soil technology, chemical engineering and other fields.

One source of difficulty when making measurements of this sort is the large number of variable parameters such as porosity, size and shape of the pores, size and shape of the grains, temperature and temperature difference, heat flow, nature and pressure of the interstitial gas, geometry of cont-

ainer, nature of solid and many others, which may influence the heat transfer. The value of much of the previous work would seem to be limited because too many parameters have been varied at the same time and many have not been measured, making it impossible to distinguish the effect of each.

Several formulae have been derived, using both theoretical and empirical methods which express the thermal conductivity of a powder in terms of some of the variables. Agreement between these formulae and experimental results is not satisfactory. With the present state of knowledge it would seem that any formula which could accurately predict the conductivity would be of such complexity and involve the measurement of so many parameters that it would be far more inconvenient to use than it would be to measure the conductivity itself. Since the conductivity of a powder is in no sense a fundamental property it would seem more important, first, to understand the basic mechanism of the heat transfer. Thus, for example, if the influence of particle size distribution on thermal conductivity was completely understood, maximum or minimum conductivity could be obtained by choosing a suitable powder. The actual conductivity could be obtained by direct measurement.

It would seem essential, therefore, to vary only one parameter at a time in an experimental investigation of the thermal conductivity of a powder. In order to elucidate the

effect of the particle size distribution of a powder on its conductivity, then, as far as possible, all other parameters should not be varied.

## 1.2 A Survey of Previous Investigations.

Theoretical expressions relating the conductivity of a heterogeneous medium to the conductivities of its constituents were derived by Maxwell (41) in 1881, Lord Rayleigh (48) in 1892 and Lees (36) in 1900. Maxwell's equation assumed a medium containing dispersed spheres of another material, Lord Rayleigh assumed a system of spheres arranged in rectangular order in a containing medium and Lees considered alternate squares of two materials. Maxwell's formula was found to be consistent with measurements on a suspension of mercury in fat by Meitner (42) in 1910 and was generalised further by Burger (10) in 1919 and Eucken (19) in 1932. However, none of these formulae are applicable to a packed powder bed because of the approximations which are made in their derivation and no successful theoretical approach to this problem has yet been made.

Experiments were first carried out by Sir James Dewar (16) in 1898. In the course of his classic work on vacuum flasks, he found that he could further reduce the heat conduction through a flask by filling the evacuated space with finely divided powder. At atmospheric pressure, however, the conductivity of the powder was greater than that of air. He

concluded that the powder shortened the mean free path of the molecules and thus reduced the heat conduction at lower pressures. Early patents utilising this principle were taken out by Fate (20) in 1909 and Stanley (56) in 1913.

The basic mechanism of heat transfer through a powder and an interstitial gas was established by Smoluchowski (55) in 1910. He measured the thermal diffusivities of several powders by measuring the rate of decrease in temperature of a thermometer which acted as the inner cylinder of a concentric system, the outer cylinder being surrounded by ice. He made measurements in air at various pressures between 0.2 and 760 mm.Hg. and found that a graph of thermal conductivity plotted against the logarithm of the interstitial gas pressure was an S-shaped curve, the powder tending to constant conductivity at the higher and lower pressures. All the powders had comparable conductivities at the lower pressures. He concluded, therefore, that the conductivity of the powder depends mainly on the interstitial gas, the residual conductivity in vacuum being due to conduction through the points of contact of the grains and to radiation passing between the grains. In an attempt to utilise this knowledge to derive an expression for the conductivity of a powder, Smoluchowski assumed each contact between grains to be displaced by a small distance  $\lambda$ . Thus he avoided the implication that the heat transfer at the point of contact would be infinite. This apparent paradox has

been discussed, more recently by Jaffe (26). Having made this assumption, Smoluchowski calculated the heat conducted through the gas at the point of contact of two spheres. He was thus able to calculate the conductivity of arrays of equal spheres, which he did for cubical and tetrahedral packing, assuming the displacement to be a function of pressure. This formula related the conductivity of the powder to the conductivity of the gas and its pressure. His experimental results gave some measure of agreement but did not confirm the theory.

Smoluchowski further calculated the conductivity of a powder of spherical grains in *vacuo*, assuming that the heat transfer was due to conduction through the solid and that the area of a point of contact was given by the Hertz (25) relationship. His calculated values were about 1,000 times greater than the experimental values. He thus concluded that there is not a good solid-solid contact between the grains of powder. This is easily understood in the light of the more modern work on friction between solids (7). Smoluchowski had thus shown that the thermal resistance of a powder is due to heat being conducted from grain to grain through the gas and, even if the solid phase is a poor conductor, the conductivity of the interstitial gas is the dominating factor. As the pressure is reduced, the conductivity decreases when the mean free path of the molecules becomes comparable to the distance between the grains, as might be expected by considering the kinetic theory of gases. The

effective conductivity thus depends mainly on the nature and pressure of the interstitial gas. These general principles of heat conduction have been confirmed by all subsequent investigations. Smoluchowski also concluded from his measurements that the conductivity of a powder depends on its particle size.

The next investigations were made by Aberdeen and Laby (1) who, in 1926, published measurements of the conductivity of silox powder in air,  $\text{CO}_2$  and  $\text{H}_2$  with interstitial pressures of 1 to 760 mm. Hg. They also used a concentric cylinder arrangement but measured a steady state temperature drop and the heat input from an electrical heater. By making the cylinders much longer than those used by Smoluchowski they assumed that a region of linear heat flow existed over the central portion of the cylinders.

They found the conductivities to be greater than those determined by Smoluchowski and pointed out that his values were open to doubt because of the short length of the cylinders which he used. However, Smoluchowski's qualitative conclusions would not be affected. When their results were plotted as a graph of conductivity against the logarithm of the interstitial pressure they gave approximately a straight line relationship, not the S-shaped curve found by Smoluchowski. They therefore proposed the experimental



relationship

$$k = \frac{1}{2} k_0 \log_{10} \frac{p}{n}$$

$k$  = conductivity of the powder.

$k_0$  = conductivity of the gas.

$p$  = interstitial gas pressure.

$n$  = constant for each gas.

Aberdeen and Laby do not specify the particle size of their silox powder but they do say that the density of the powder was only  $\frac{1}{56}$  times that of the constituent grains. It seems probable, therefore, that the void spaces were fairly large and that they did not attain sufficient vacuum to produce an S-shaped curve. They did point out the possible use of evacuated powders as thermal insulation for liquid oxygen storage vessels.

The work of Aberdeen and Laby was continued by Kannuluck and Martin (28) who, in 1933, published the results of a more extensive set of measurements of the conductivities of several grades of carborundum powder and also magnesium oxide, glass and diphenylamine powders over with a gas pressure range of 0.5 to 76 cm.Hg. Their experimental technique was again a concentric cylinder arrangement, a straight wire forming the inner cylinder and the heat flow being calculated from the electrical energy dissipated in the wire. When plotting conductivity against the logarithm of the gas pressure they obtained the S-shaped curve as found by

Smoluchowski. They found that, for a single powder, it was as though the scale of the pressure was successively magnified in passing from carbon dioxide to air to hydrogen.

They considered the possibility that conduction was due to an adsorbed layer of gas causing conduction between the grains but showed that this explanation is unlikely because the powders did not show any variation in their conductivity at atmospheric pressure before and after evacuation. They also measured the conductivity of a powder filled with helium which does not show any adsorption and yet the conductivity of this powder was high. Kannuluick and Martin also pointed out that Aberdeen and Laby had in fact obtained a slightly "S-shaped" curve if full weight was given to each of their points. In an attempt to obtain a mathematical expression for the conductivity of a powder Kannuluick and Martin considered it to be made up of a series of successive laminae of solid and gas and thus derived the formula

$$k = \frac{k_g}{1 - w(1 - \frac{k_g}{k_s})}$$

where:  $w = \frac{p}{p_0}$

$k$  = conductivity of the powder  
 $k_g$  = conductivity of the gas  
 $k_s$  = conductivity of the solid  
 $p$  = effective density of the powder  
 $p_0$  = density of the solid

Agreement between the measured conductivities and values calculated using this formula were not wholly satisfactory and this they attributed to some of the interstitial spaces being

smaller than the mean free path of the gas. Their results, therefore, agreed in principal with Smoluchowski's, and it is interesting that the change of gas gave the results which might have been expected if the conductivity of the powder depends mainly upon that of the interstitial gas.

In 1931 Amberry (4) had attempted to obtain an expression for the conductivity of a powder by applying the principal of similitude. Assuming a medium in which all the bodies are similar and are situated in the same position with respect to the surrounding ones, he balanced the dimensions of conductivity with those of the size of the powder bed and its constituent grains. Saunders (52) extended this theory in 1932 and concluded that the effective conductivity is independent of the size of the grains, and if the conductivity of the gas is low, the conductivity of the powder is proportional to that of the constituent grains. This conclusion clearly does not accord with experimental values and it seems that there are so many parameters which can be varied to describe an actual powder that very little progress can yet be made by this approach. Obviously, unless all the variables are included, the conclusions are not likely to be worthwhile.

It was by this time established that the basic mechanism of heat transfer is conduction and the effective conductivity of the powder depends mainly on that of the gas.

Several later authors have stated that conduction is mainly by the gaseous phase which is a misinterpretation of the results, the gaseous phase constitutes the thermal resistance. A mathematical description of the process was obviously very difficult, if not impossible, and simple models did not describe the results completely. All the experimental measurements were open to objections but they yielded comparative results which were almost as useful as absolute ones.

Meanwhile, the subject was increasing in practical importance and was approached by several people with specific problems in mind.

Griffiths' (24) work, published in 1929, was a thermal insulation test for the Food Investigation Board. He used a parallel plate and guard ring arrangement and worked at atmospheric pressure with relatively coarse materials. Under these conditions, convection contributed considerably to the heat transfer when the apparatus was in a vertical plane. Terres (58) measured the conductivity of crushed coal, in 1928, in order to try to improve its 'coking' time. Burke Schumman and Parry (11) were concerned with the same problem and in, 1931, they published values for the thermal diffusivity of crushed coal. They found that the diffusivity of coal,  $\frac{1}{2}$ "- $\frac{1}{4}$ " mesh, was greater than that of  $\frac{1}{16}$  mesh coal. Their experimental method was a dynamic one and from the shape of the

heating curve they concluded that the laws of conduction for solid materials hold also for granular materials.

In 1934 Schumann and Voss (54) made an empirical attempt to derive a formula which would predict the conductivity of a granular material from a knowledge of its constituents. They assumed that the conductivity could be expressed in the form  $K = f(K_A, K_B, a)$

where: -a = fractional volume of the continuous phase A

$K_A$  = conductivity of phase A

$K_B$  = the conductivity of the solid phase B

$K$  = the effective conductivity of the medium.

By considering all the limiting cases where  $K$  was known, they empirically arrived at the formula

$$K = \frac{K_a K_b}{K_a + p(K_a - K_b)} \left\{ 1 + \frac{p(1+p)(K_a - K_b)}{K_a + p(K_a - K_b)} \log_e \frac{K_a(1+p)}{K_b p} \right\}$$

where  $a = p(p+1) \left( \log \frac{1+p}{p} \right) - p$ .

They measured the diffusivity of steel shot, lead shot, quartz and coal, in air and in hydrogen at various pressures. Their experimental technique was one of plunging a concentric cylinder arrangement into a bath of boiling water and plotting the rise in temperature of the inner cylinder against time. The results corroborated the conclusions of earlier investigators, the conductivities in hydrogen being much greater than those in air but both approaching similar values at lower pressures. They also pointed out that convection

cannot be an important mechanism of heat transfer through fine powders. Unfortunately, the experimental results did not fit their empirical formula very well.

In 1935, Russell (51) attempted to derive a formula to explain the behaviour of porous and granular materials. His model consisted of cubical pores of the same size on a regular lattice or alternatively, cubical particles separated by layers of air and he assumed plane isothermals. Thus for a granular material his formula takes the form:-

$$\frac{K_b}{K_a} = \frac{(1-p)^{\frac{2}{3}} + \frac{k_a}{k_s} \left[ 1 - (1-p)^{\frac{2}{3}} \right]}{(1-p)^{\frac{2}{3}} - (1-p) + \frac{k_a}{k_s} \left[ 1 - (1-p)^{\frac{2}{3}} \right] + (1-p)}$$

where  $k_b$  = conductivity of the material  
 $k_s$  = conductivity of the solid  
 $k_a$  = conductivity of the air  
 $p$  = fractional porosity

Russell also calculated the contribution to the heat transfer made by radiation assuming the same model as before

$$\text{hence :- } K_r = 4 \epsilon A_{12} T_m^2$$

where  $K_r$  = apparent conductivity across a pore due to radiation  
 $T_m^2$  = absolute temperature  
 $x$  = width of air space  $\epsilon$   
 $A_{12}$  = a constant depending upon the emissivity and geometry of the surfaces.  
 $\epsilon$  = radiation constant

He concluded therefore that with small pores and

lower temperatures the conductivity of the pores is that of the air alone. With higher temperatures radiation plays an increasing part and with pore spaces bigger than about  $\frac{1}{4}$ " convection also

occurs. He does not compare his formula with experimental data but concedes that it only approximates to the truth, it does not, for example, predict that for lowest conductivity, the continuous phase should be the material of lower conductivity.

It was now apparent, therefore, that convection is entirely eliminated with small pore size. Radiation was thought to constitute some part of the total heat transfer but its importance was not known.

In 1934 Kistler and Caldwell (31) published measurements of the conductivity of various grades of crushed silica aerogel at various gas pressures down to  $10^{-4}$  mm. Hg. They also measured the conductivity of the powder when it was filled with dichlorodifluoromethane which has a conductivity lower than that of air. As was expected the conductivity of the powder was lower than when filled with air. Their apparatus was of the guarded plate type and was adapted so that mechanical pressure could be exerted on the upper plate by means of a calibrated spring. Readings of the conductivity when evacuated to a low gas pressure were then made with different mechanical loads on the spring. It was thought that by this means the particle to particle contact would be increased and hence also the heat conduction. This was indeed the case, the conductivity increasing from  $1.27 \times 10^{-5}$  to

$1.50 \times 10^{-5}$  cal/sec/°C/cm when a load of 15.3 lb/sq.in. was applied. Since the silica aerogel seemed to behave elastically under repeated compression and release, after an initial decrease in volume, this experiment would seem to show that much of the residual conductivity of a powder under vacuum is due to particle-particle contact.

In a paper published in 1935, Kistler (30) went further and pointed out that, since the conductivity of a porous material depends very closely on the relative sizes of the pores and the mean free path of the interstitial gas, this could be a powerful tool in measuring the size of the pores, which, in silica aerogel, were too small to be measured by microscopic means.

Kistler first deduced a formula relating the conductivity of silica aerogel, its mean pore size and the mean free path of the gas as follows:-

From the kinetic theory of gases the conductivity of a gas is proportional to the mean free path of the molecules of the gas

$$k = k^1 \ell$$

where  $\ell$  = mean free path of the molecules.

If  $L$  is the mean pore size of the silica aerogel, a molecule may collide either with another molecule or with a solid surface and the mean free path of the molecules is  $l_a$  where

$$l_a = \frac{L \ell}{L + \ell}$$



If the conductivity of the aerogel is proportional to this mean free path, and this would seem to be open to discussion, then

$$k = k' \frac{L \ell}{L + \ell}$$

and hence

$$k = 0.058 \frac{M}{T} C_v \ell_0 \frac{L}{L + \frac{\ell_0}{P}}$$

where  $M$  = the molecular weight of the gas.  
 $\ell_0$  = the mean free path of the gas at a pressure of 1 mm. Hg.  
 $p$  = pressure of the gas.  
 $T$  = temperature of the gas.  
 $C_v$  = specific heat of the gas at constant volume.

Kistler modified this formula to allow for an accommodation coefficient between the molecules and the gel surface, but concluded that the error was probably small in this case and neglected it in actual calculation. By estimating the residual conductivity of the powder at very low pressures and using the measurements at two other pressures, Kistler was able to calculate  $L$ , the mean pore size from his equation. He performed this calculation using values of conductivity of the same powder measured with three interstitial gases, air,  $CO_2$  and  $CCl_2 F_2$  and obtained consistent values for the pore size. If the calculation is repeated using some of the values of conductivity measured by Kistler but not used in his calculations, however, the agreement is not so good. However, this is the only real attempt to use conductivity measurements

as a means of measuring pore size and would seem to be a promising idea.

In 1936 Bartens (5) measured the thermal conductivity of steel, glass and lead balls in oil and in air at atmospheric pressure and at 0.2 mm.Hg. The conductivities at atmospheric pressure were greater than the evacuated case but were much less than those for oil immersion. These conductivities increased when the volume of metal was increased.

These results agree with what might be expected. Since the conductivity of a liquid is much greater than that of a gas system in which the continuous medium is a liquid will have a higher conductivity. It is also of interest to note that as early as 1909 Patten (45) had published measurements of the thermal conductivities of soils with various moisture contents and had found that the conductivity increases sharply with increasing moisture content. This is obviously an intermediate case and emphasises the importance of the continuous phase in transferring the heat between the solid grains. Patten also concluded from his experiments that the conductivity is not greatly affected by grain size. In 1938 Kling (32) published a set of measurements of the conductivity of powders at pressures between one and fifty atmospheres. He did not find any appreciable effect with increased pressure over this range which again accords well with what might be predicted from the kinetic theory of gases since the conduc-

tivity of the gas would not be expected to increase over this range.

Cone (12) submitted a thesis to London University in 1938 entitled 'The flow of heat through a granular material'. He measured the thermal diffusivity at atmospheric pressure of a variety of materials including steel spheres, steel plates, crushed brick and glass by plunging a cylinder containing the material into a constant temperature bath. A larger temperature drop gave an increased diffusivity of the crushed brick. Polishing the spheres appeared to decrease the conductivity. The results were not very accurate and were only taken at atmospheric pressure. The main variable in the readings was the material, which was very coarse. However, the results may be taken as an indication that temperature and the state of the surface are variables which may influence the conductivity. Cone did not offer any explanation of his results but it is interesting to note that polishing the spheres decreased the effective conductivity and to see how this could be explained as a reduction of the accommodation coefficient as discussed later.

In 1939 Damkohler (13) calculated the contribution to the heat transfer made by radiation assuming that radiation, passed from solid to solid through the interstices and that no absorption occurred in the gas. No experimental support for the formula was offered.

His formula was  $K_a = 4 A e \sigma S dp \frac{T^3}{10^8}$

where:-

$K_a$  = equivalent conductivity caused by radiation  
 $A$  = fractional free area in granular material  
 $e$  = emissivity  
 $\sigma$  = Stefan's constant  
 $S$  = factor relating the length of the radiant path between particles to the particle diameter  
 $dp$  = diameter of the particles  
 $T$  = absolute temperature.

In the same year Austin (3) published a general review of the conductivity of porous materials. He pointed out the large number of variable parameters and discussed their effect using some of the results which have been described previously. Austin also discussed the conduction of heat through a powder in terms of the scattering of heat quanta, or phonons as postulated by Delye (14).

Cone's work was continued by Waddams (61) who, in 1944, published measurements of the conductivity of steel spheres and of coarse calcite powder, again at atmospheric pressure. The increase in conductivity with increasing particle size he attributed to convection. Again the cleanliness of surface of the particles affects the conductivity but Waddams did not make a direct comparison of the conductivity of the same spheres with different surface conditions.

In 1948 Wilhelm et. al (63) collected all the available experimental measurements and compared them with the formula derived by Schümann and Voss. They computed a

correction factor to be added to the formula which would give the best average of the experimental results. Thus, of course, they ignored the parameters which were not included by Schuman and Voss.

In 1950 Prins et al (47) measured the heat transport through glass spheres and through magnesium oxide with gas pressures from  $10^{-4}$  - 800 mm. Hg, using hydrogen, carbon dioxide and air as interstitial gases. They obtained the S shaped curves and also the variation in conductivity with the interstitial gas found by earlier workers. They also claimed that a calculation of the contact radius of glass spheres using the Hertz relationship, predicts residual conductivities which are of the same order of magnitude as experimental values. In the same year, Gemant (23) calculated the thermal conductivity of soil assuming a layer of moisture between each particle. He compared his formula with a set of measurements which had been made by Krischer (33) in 1934. These papers do indicate that a three phase system will be very different from one consisting of two phases and that the two cases cannot be easily correlated.

Gemants' formula was criticised by Webb (62) because of the neglect of the air phase and by De Vries (18) because of the assumption of parallel heat flow. Most of the theoretical predictions of effective conductivity have been derived assuming parallel flow and thus they can only be, at the best,

order of magnitude estimates. There seems little point in trying to make experimental results fit these formulae or to deduce from them the effect of a parameter.

Three sets of measurements were published in 1951 by Rowley et al (50), Katan (29) and Allcutt (2). Rowley et al measured the conductivity of several powdered and fibrous materials. They found the usual reduction in conductivity with pressure and also a reduction in conductivity of the fibrous materials when they were compressed. Rowley stressed the importance of investigating the effect of one parameter on the heat transfer instead of overall changes due to several parameters. Much of the work reported in this survey would seem to be of limited value for this reason. Katan measured the conductivity of several powders over a range of pressures and obtained the familiar S-shaped curve. By noting the pressure when the conductivity first started to increase he equated the mean pore size of the powders to the mean free path of the gas at this pressure. Allcutt measured the conductivity of fibrous materials at different bulk densities obtained by compression. A graph of conductivity against bulk density was a hook shaped curve, the material having a higher conductivity at the lower and higher densities and passing through a minimum at an intermediate density. The high conductivity at low densities was attributed by Allcutt to increased heat transfer by radiation and convection. Rowley had found only a decrease

in conductivity but possibly if he had compressed his material further he may have obtained a similar effect . In 1952, Deissler and Eian (15) again assumed a cubical array of spheres and parallel lines of heat flow in order to relate the effective conductivity of a powder to those of its constituents.

Marathe and Tendolkar (39) published measurements of the conductivities of marble haematite and copper powders (1953). The experiments were only performed at atmospheric pressure and the object of the experiment was to determine how the porosity of the bed and the particle size of the powder affected the conductivity. They relied on sieving as a particle size analysis and varied the porosity by putting an appropriate weight of powder into the thermal conductivity apparatus. It would seem almost certain that in some cases the powder would settle to a lower porosity during the experiment and a more reliable procedure is to measure the porosity of the most dense packing. Marathe and Tendolkar concluded that conductivity varied linearly with the porosity of the bed and was not affected by particle size. These conclusions do not agree with those described later and it must be questioned whether Marathe and Tendolkar controlled and measured the size and porosity of their powders adequately. Marathe and Tendolkar also developed a formula for calculating the conductivities of powders by considering the solid conduction and a "fictitious gas condition" to operate in parallel.

In 1954 Johnson et. al (27) measured the conductivity of a number of powders and fibres and obtained the S-shaped curves on graphs of conductivity plotted against the logarithm of the interstitial gas pressure at temperatures as low as 20°K.

In the following year Leidenfrost (37) published a study of the use of evacuated powders as a support for thin-walled flasks.

In 1958 Woodside (65) also calculated the conductivity of a cubical array of spheres in a gas assuming parallel lines of heat flow.

Laubnitz (35) published measurements of the conductivity of various powders at atmospheric pressure in 1959. He measured the conductivities over a mean temperature range of 100°C to 1,000°C. Although Laubnitz does not state whether the temperature difference was the same in each case, his results do demonstrate a considerable increase in conductivity with increasing temperature which he attributed to an increase in the component of heat transfer due to radiation. He compared his results with some of the formulae derived by earlier workers. Using the basic model of Russell, symmetrically placed cubical particles, he derived a formula expressing the contribution of radiation. He was, however, unable to obtain agreement between this theory and his experimental results. It may be pertinent to mention that the temperature



gradient in his apparatus was measured by two thermocouple junctions located in the powder which were separated by a radial distance of only 5 mm.

Three sets of measurements were published in 1959 by Bretznajder and Ziolkowski (8), Schafer and Gr<sup>u</sup>ndler (53), and Fulk et al (21, 22, 49). Bretznajder and Ziolkowski studied the effect of the packing of cylinders on the effective conductivity at atmospheric pressure. They found a higher conductivity for a uniformly packed bed with the axes of the cylinders perpendicular to the heat flow than for a disorderly packed bed and an even higher conductivity for a uniformly packed bed with the axes of the cylinders parallel to the heat flow. In the latter case the bed had a higher porosity and the increase in conductivity was attributed to the increased area of contact. Bretznajder and Ziolkowski also found an increase in conductivity with temperature gradient and attributed this to radiative heat transfer.

Fulk measured the effective conductivity of several combinations of powder and gas over a pressure range of  $760-10^{-6}$  mm.Hg. The measurements were made at low temperatures and he obtained S-shaped curves on graphs showing the variation of conductivity with pressure. Fulk was particularly interested in the residual conductivities at low gas pressures, a large proportion of which he attributed to radiation. There was a paradox in some of his results in that there was an apparently

greater heat flow through some of the powders with a smaller temperature drop.

"  
Schafer and Grundler measured the heat transfer through a quartz powder with several different gases. They measured the residual conductivity at low pressures and subtracted this from the effective conductivity at higher pressures. They found a linear relationship between the effective conductivity of the powder and the known conductivity of the gas at lower temperatures, the constant of proportionality depending on the particle size. They therefore extrapolated the conductivity of the gas to higher temperatures and obtained values which agreed reasonably well with direct measurements.  
" Schafer and Grundler were the first people to realise that it may be more profitable to try to deduce the fundamental properties of a gas from measurements of the conductivity of a powder rather than to try to predict the conductivity of the powder from the properties of its constituents.

In 1960 Strong, Bundy and Boverkerk (57) measured the conductivity of evacuated fibrous materials and concluded that random orientation of the fibres in a plane perpendicular to the heat flow gave the least conductivity. They also made an estimate of the heat transfer due to radiation and concluded that it must constitute a large proportion of the total in evacuated materials.

In 1962 Pratt (46) calculated the conductivity of a

fibrous material, using Kistler's approach of modifying the mean free path of the gas to allow for collisions with the fibres and showed that it compared fairly well with experimental results obtained by Verschoor and Greebler (60).

In the same year, Mischke and Smith (43) measured the thermal conductivity of alumina pellets and in 1963 Masanume and Smith (40) measured the conductivity of beds of spherical particles. For four grades of spherical glass beads the conductivity increased with increasing particle size. Masanume and Smith calculated a formula to explain these conductivities by considering the different mechanisms of heat transfer to operate in parallel.

### 1.3 Discussion of the Literature Survey.

It is evident from the work of previous investigators that the effective thermal conductivity of a powder is dependent mainly upon the nature and pressure of the interstitial gas and that a graph showing the conductivity of a powder plotted against the interstitial gas pressure produces an S-shaped curve, the conductivity tending to asymptotic values at the higher and lower pressures.

This basic mechanism of heat transfer was elucidated by Smoluchowski at the beginning of the century and yet it is still not possible to say how the thermal conductivity will vary from powder to powder. There are several parameters whose

influence on the conductivity is not understood but it would seem that the powder itself can be characterised if the nature of the solid and the particle size distribution of the powder are known. The conductivity of the solid grains does influence the conductivity of the powder but it would seem to be a secondary effect and there is no investigation needed to understand it. The influence of the particle size distribution of a powder on its effective conductivity is, however, not clear. Several authors, such as Smoluchowski and Schafer and Gründler, have stated that the particle size of a powder does influence its conductivity, many authors have simply ignored the effect of particle size and a few, notably Marathe and Tendolkar, have stated that the thermal conductivity of a powder at atmospheric pressure, does not depend on its particle size. Several authors, such as Kannuluick and Martin, have indicated that coarser powders tend to have a higher conductivity and Waddams has attributed this effect to an increase in convective heat transfer as the pore size is increased. It would seem that, if the dependence of the conductivity of a powder on its particle size was understood, then the mechanism which causes the conductivity of different powders, measured under identical conditions, to be different would be understood and an investigation of the influence of other parameters on the conductivities of all powders could then proceed from there. Many of the investigations described previously are open to the

objection that the various parameters have not been controlled closely enough and the results are, therefore, almost impossible to interpret. In this case, it was decided to vary the particle size distribution of a powder and to keep the other parameters constant as far as possible.

An aluminium powder was chosen since metallic powders were of prime interest and air was obviously the most convenient interstitial gas. The work of Leubnitz, Fulk et al and other workers shows that the conductivity of a powder varies with the temperature at which it is measured and several authors have stressed the importance of radiative heat transfer through a powder. It seemed important, therefore, that the measurements should be made at fixed temperatures and also that the importance of radiative heat transfer should be investigated if interpretation of the results were to avoid ambiguity. It was also clear that for a comprehensive investigation, the conductivity of a powder must be measured over a range of pressures wide enough to fully define the S-shaped curve.

Most of the different techniques used for measuring the thermal conductivities of powders by previous investigators are open to objections. Because of the time involved in making steady state measurements, the deduction of thermal conductivities from measurements of thermal diffusivity has been a popular approach to the problem. It is, however, by no means

clear that the conception of thermal diffusivity can be so easily applied to an inhomogeneous mass and, in any case, it is not possible to make measurements with fixed boundary temperatures by this method.

It was concluded, therefore, that an investigation of the variations in thermal conductivity of powders of the same material with differing particle size distributions and other parameters held constant, as far as possible, was needed in order to be able to understand the variation in the conductivity of one powder to another.

#### 1.4 Packing of Particles.

The study of the packing of granular materials has been widely treated both theoretically and experimentally, and an account of much of the earlier work is given in "Micromeritics" (66). The effect of several variables has been summarised by MacCrae and Gray (38).

MacCrae has pointed out that only two situations in the packing of a powder can be explicitly stated, that at the point of incipient fluidisation and that of the most dense packing when tapping does not further reduce the porosity. It is the latter packing which is relevant to this investigation and which can be considered characteristic of the powder. Further compression of a powder by the application of pressure would seem to be inadmissible since grinding and deformation may take place and the particle size distribution thus be

changed. For the purposes of this investigation, therefore, a packed powder is one whose container has been tapped until the maximum density is achieved.

Before the overall properties of a powder bed are measured it is necessary to ensure that the bed is uniform, otherwise the results would be very difficult to interpret. MacGrae and Gray found a variation of porosity in the radial direction but not in the vertical direction. More recently, Benenati and Brosilow (6) have shown that an increased porosity exists at the wall of a container and also in the vicinity of an object inserted into the bed. This wall effect is, therefore, a valid component of the thermal resistance whose importance depends on the relative sizes of the particles and their container. Benenati and Brosilow say that the effect is important for the first five particle diameters, although whether this is generally applicable is not known. As will be seen in section 2.7, this wall effect may cause an appreciable temperature drop but it is not large enough to detract from the significance of the measurements.

Experiment 7. A variation in the properties of the bed with the height of the bed would make it impossible to produce a radial heat flow pattern, and hence, to find the conductivity of the powder. In order to ascertain whether vertical inhomogeneity exists in a packed bed of the aluminium powders used in the experiments described in Chapters 2 and 3 a sample of

the original, unsieved aluminium powder was packed into a tube which was made in three sections. By inserting a thin blade between the sections, the bed was split and the particle size distribution of two samples of powder from each section was measured using the Bound Brook Photosedimentometer described in Appendix 2. The results are shown in Graph 9 which is a plot of relative surface against Stokes' diameter for samples from each section. No appreciable variation of particle size distribution with height exists and it has been assumed, therefore, that particle segregation did not occur when these powders were packed into the thermal conductivity apparatus, providing the powder is well mixed initially. This conclusion is supported by the results of McCrae who also in a packed bed found no vertical segregation.

In Experiment 6 mixtures of  $>150$  mesh aluminium and  $<300$  mesh aluminium were used and it was decided to check that this conclusion applies to these powders since the possibility of particle segregation is probably greater. A mixture containing approximately 30% by weight of the finest grade was packed into the sectional tube and the tube split into three parts as described previously. This mixture contains a reasonable amount of both components but is not the mixture of minimum porosity. The two grades of powder were separated, for each section, by sieving on a 200 mesh sieve, the  $<300$  mesh grade passing easily through the sieve and the



>150 being retained. The relative percentages of each grade in the three sections are shown in table 10. It is clear that each section of the powder is still a representative sample of the whole.

Experiment 10. While interpreting the results of the thermal conductivity measurements described later in this thesis the porosity of the packed powder was found to be a critical parameter. Thus, to understand completely the effect of particle size distribution on the thermal conductivity of a powder some knowledge of its effect on the porosity of the powder seemed desirable.

An experiment was carried out using glass powders of spherical particles since their particle size distribution is easily defined and measured. The powders were available as sieve fractions and their particle size distribution was found by microscope counting as discussed in Appendix 2. Five successive sieve fractions were then blended in different proportions to give powders with the same size limits but different size distributions. The porosities of these powders were measured in a graduated cylinder as discussed in Appendix 2. The measurements were repeated for powders of even wider distribution and for the individual sieve fractions. The results show that, for powders with the same size limits, the porosity of the bed is lower for a powder whose size distribution, plotted on a cumulative percentage weight basis, is linear than

for those powders which have an excess of larger or smaller particles. Further, if the ratio of the size of the largest particle to that of the smallest is increased, the porosity of the powder will be less than that of a powder with a similar type of size distribution but with narrower size limits.

More experiments are required before it is possible to say whether these conclusions are generally applicable to irregular powders. It is plain, however, that although the geometry of the container and the method of packing a powder are contributing factors, the particle size distribution of a powder determines primarily its packed porosity and hence, as is shown in Chapter 3, its effective thermal conductivity.

Three of the powders were blended a second time using coloured heads, a different colour being used for each of the five sieve fractions. When the powders were packed no obvious segregation of the different colours could be observed, which supports the conclusions of Experiment 7 that vertical inhomogeneity does not exist in a packed bed.

CHAPTER 2.EXPERIMENTAL WORK2.1 Design of the Apparatus

In all thermal conductivity measurements it is difficult to accurately define the heat flow pattern. Insulation is not possible with very poor conductors, such as powders, and linear heat flow is difficult to obtain.

Thermal conductivities can be measured either by steady state or dynamic methods. The flow pattern is more difficult to define when using the dynamic methods and they suffer from the further disadvantage that a close control of parameters such as temperature is not possible. The need for a close control of conditions has already been stressed and thus a steady state method was adopted even though the results were consequently obtained very slowly.

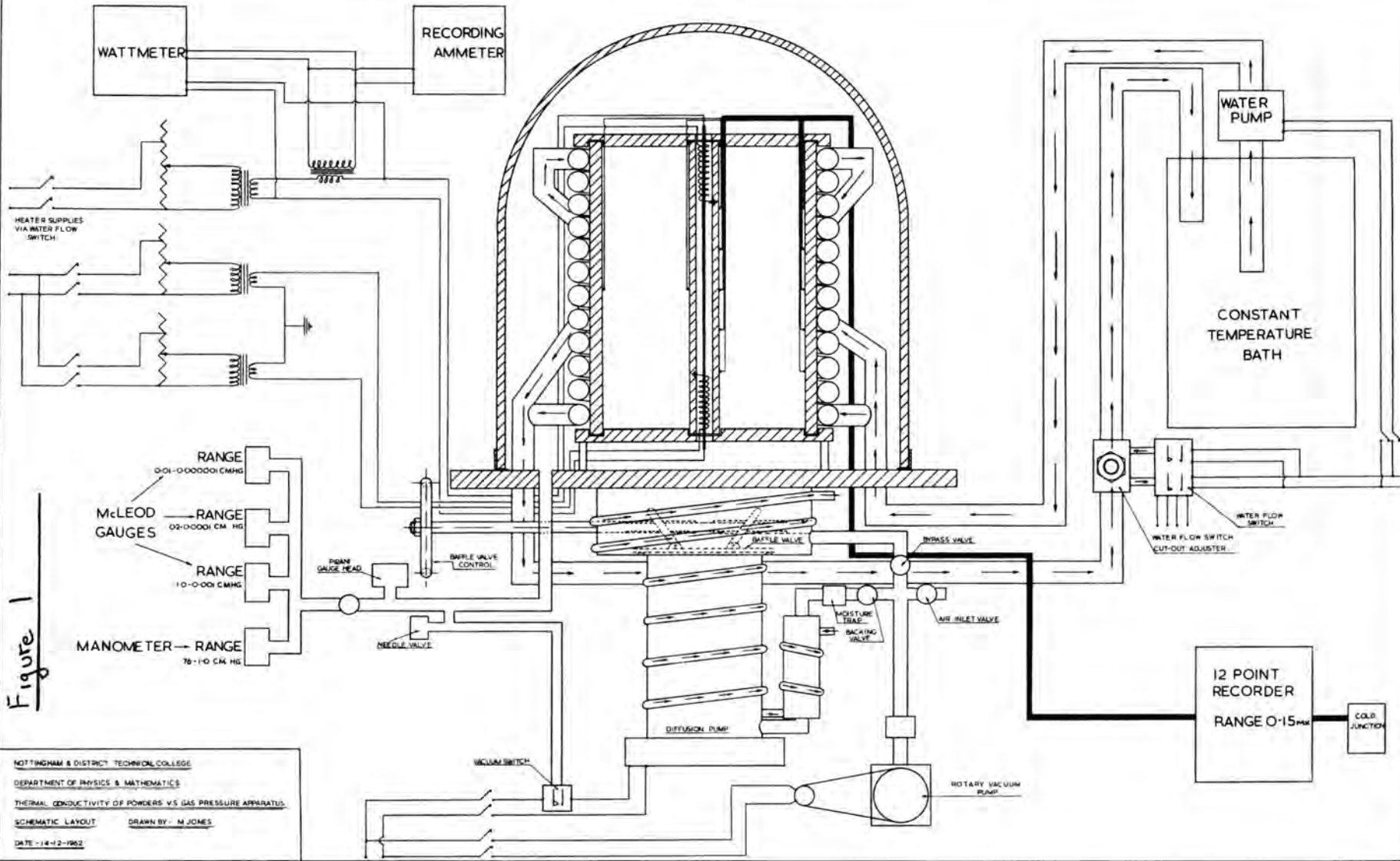
There are basically three geometrical arrangements which are convenient for measuring thermal conductivities, concentric spheres, concentric cylinders, and parallel plates. The concentric sphere arrangement was rejected because it necessitates thermocouple and heater wires lying in the direction of heat flow which would distort the flow pattern. Since the powder must be contained, the other methods involve a conducting surface in the direction of heat flow and

therefore compensation for end losses must be made. The concentric cylinder arrangement was chosen as the more convenient of the two because the compensating regions can be separated from the central region more easily and also because it is possible to ensure that the heat produced flows through the powder.

The size of the apparatus was limited by the dimensions of the vacuum system available and by considerations of the quantities of powder necessary to fill the apparatus. The easiest way of obtaining a uniform low pressure in the powder bed was to mount the whole apparatus in a vacuum dome. The cylinders had to be short enough to fit inside the dome and also have a diameter smaller than their length. The powder annulus had to be reasonably thick, however, and this, combined with the necessity for keeping the powder samples small, called for an inner cylinder of small diameter. Since the heaters had, therefore, to be fitted into a small space a low voltage A.C. electrical system was used.

To deduce the thermal conductivity of the specimen it was necessary to measure the heat flow and the temperature drop. Three methods were considered for measuring the heat flow; calorimetry, electrical measurements and measurement of the temperature drop across a known conductor placed in series with the specimen. Calorimetry was considered to be

Figure 1



NOTTINGHAM & DISTRICT TECHNICAL COLLEGE  
 DEPARTMENT OF PHYSICS & MATHEMATICS  
 THERMAL CONDUCTIVITY OF POWDERS VS GAS PRESSURE APPARATUS  
 SCHEMATIC LAYOUT DRAWN BY - M JONES  
 DATE - 14-12-1962

inconvenient and a large thickness of conductor would have been necessary to give a reasonable temperature drop in series with the powder. It was, therefore, decided to rely on electrical measurements alone.

From these considerations an apparatus was designed which would measure the effective thermal conductivity of powders, over a range of pressures and temperatures, with sufficient accuracy to detect any appreciable change in conductivity.

A schematic diagram of the apparatus is shown in Fig.1 and a general view in Fig.2.A and 2B

## 2.2 Construction of the Apparatus

The cylinders were made of graphite and were mounted vertically and concentrically the inner cylinder containing the electrical heaters, the outer one being surrounded by a heat sink. The powder under test was packed between the two cylinders and was contained by two graphite slabs. Holes were drilled through the top slab to allow the thermocouple wires to pass through and to assist in evacuating the air from the powder. A scale drawing of the apparatus is shown in Fig.3 and a general view in Fig.4.

The main heater consisted of a straight constantan wire insulated by a glass tube. At each end of this tube was wound a compensating heater and the whole was enclosed in

another glass tube. This composite heater was then inserted into the inner cylinder. The end of each of the compensating heaters passed through a hole in the insulation and made contact with the graphite cylinder, thus making only one electrical connection with each of the compensating heaters necessary. Each of the heaters was fed from the A.C. mains through a Variac transformer and a step-down transformer. By adjusting the appropriate Variac the output of each heater could be controlled. The voltage drop across the main heater was measured by an Avometer, the current passing through it being measured by another Avometer connected across the secondary winding of a current transformer. The mains voltage fluctuated slightly but it was thought that a voltage stabiliser would distort the wave form and introduce inaccuracies into the power measurements. The fluctuations were, therefore, monitored by a recording ammeter connected across the larger transformer. A circuit diagram of the electrical arrangement is shown in Fig.5.

The heat was dissipated by a spiral cooling coil soldered to a copper cylinder which fitted round the outer graphite cylinder. Good thermal contact was ensured by interposing an aluminium shim. In order to ensure symmetry and an even sink temperature the spiral was wound with two tubes lying side by side, their opposite ends being joined.

The cooling water was circulated from a constant temperature bath and an Edwards' "Flowtrol" switch was included to break the heater circuits if the water pump should fail.

The apparatus stood on a copper base inside the vacuum dome. Into the base was cut a hole equivalent in size to the outer cylinder so that the base did not contribute to the end losses. The dome could be evacuated by means of a rotary pump and an oil diffusion pump, the rotary pump being connected to the vacuum dome by a flexible tube to minimise vibration. A phosphorus pentoxide moisture trap was included in series with the diffusion pump which was protected by a pressure switch. The gas pressures were controlled by means of a leak valve and were measured on a series of three McLeod gauges and a mercury manometer, used in conjunction with a Fortin barometer. A Pirani gauge was also included for control purposes.

Twelve thermocouple junctions were spaced over the central region of the cylinders, six on the inner and six on the outer. Fine Chromel-Alumel wires, insulated with glass braiding were used. Two thermocouple junctions were placed diametrically opposite each other at the centre of each cylinder, thus indicating any assymetry. The remaining thermocouple junctions were then placed at points 1" and 2" on either side of the central ones. The outer thermocouples



Figure 2 A



Figure 2 B

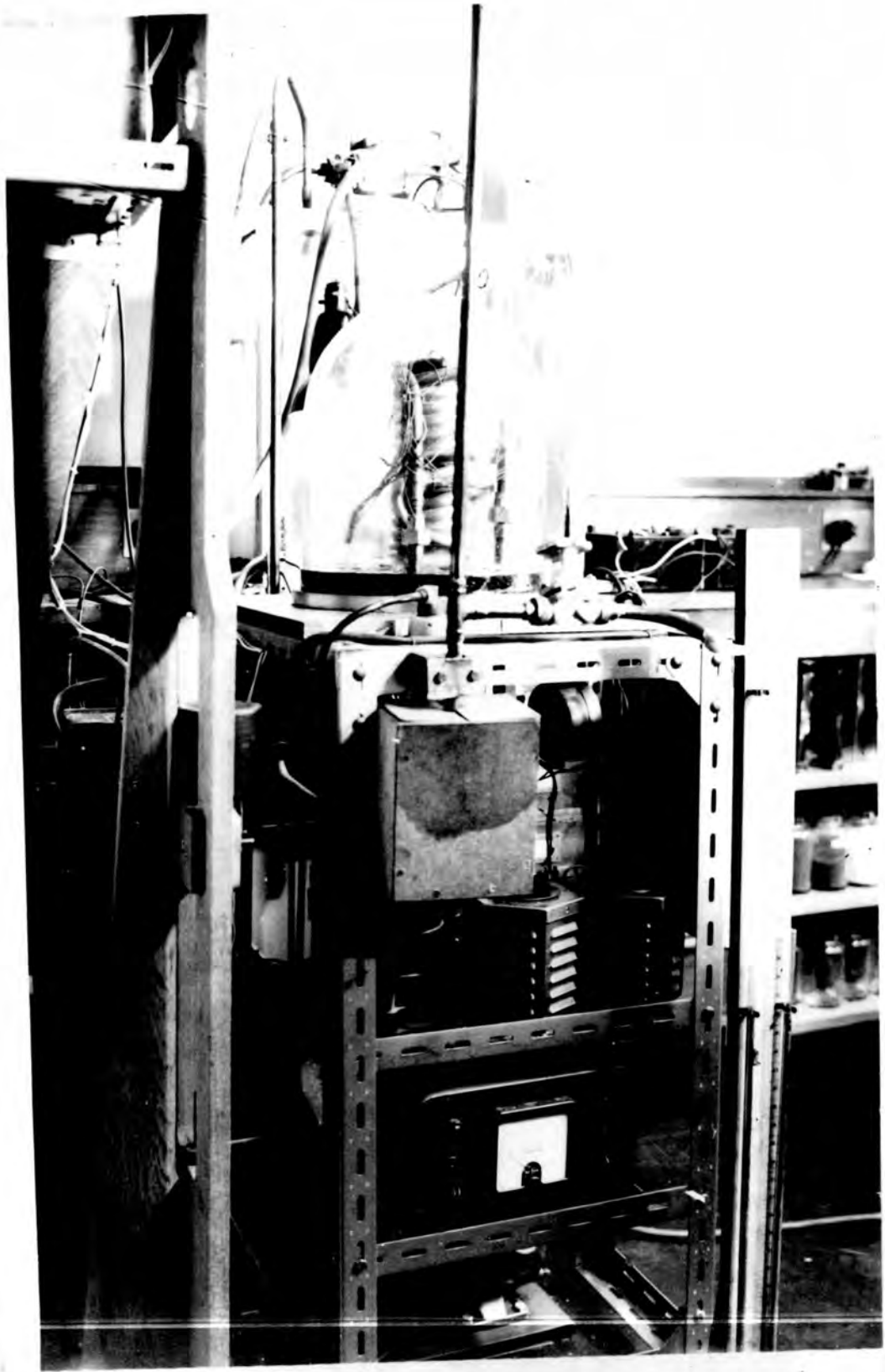
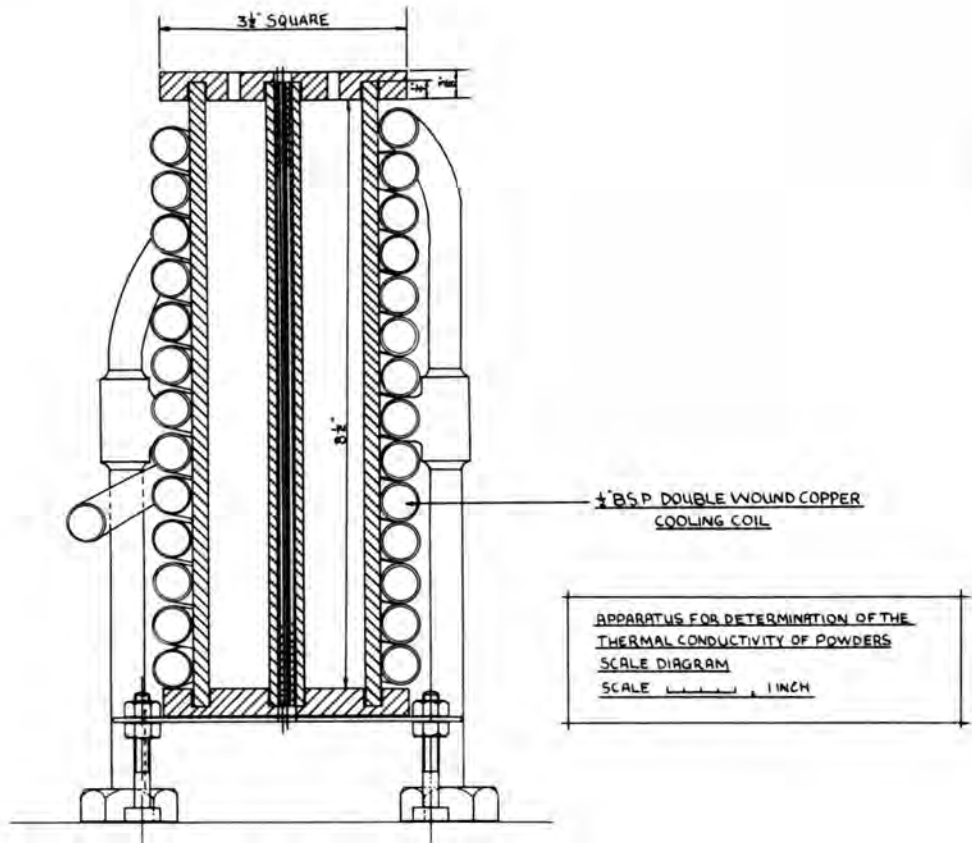


Figure 3



SECTIONAL FRONT ELEVATION ON 'AA'

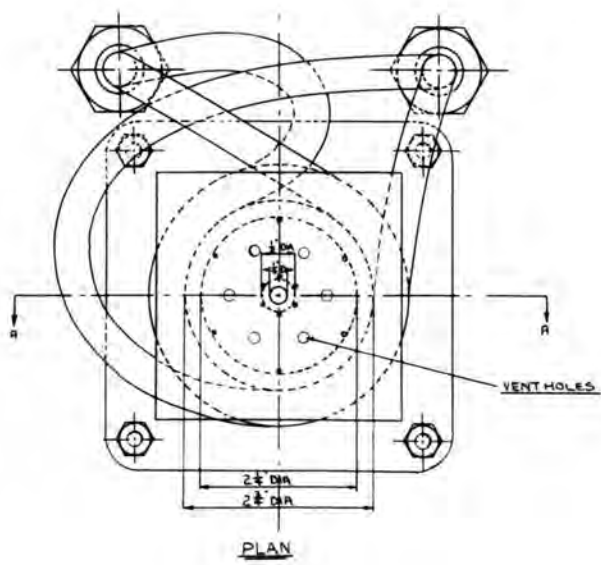
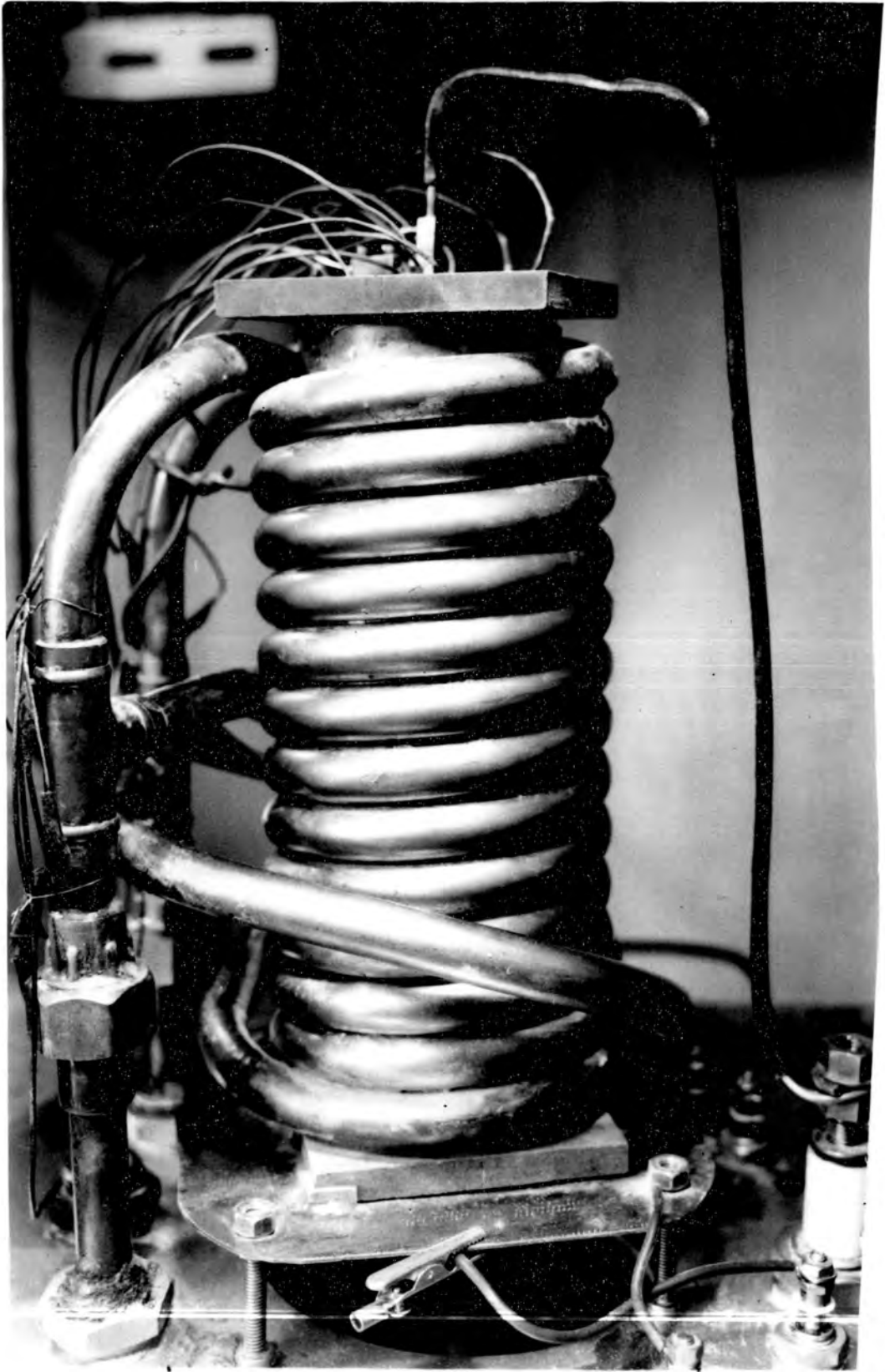


Figure 4



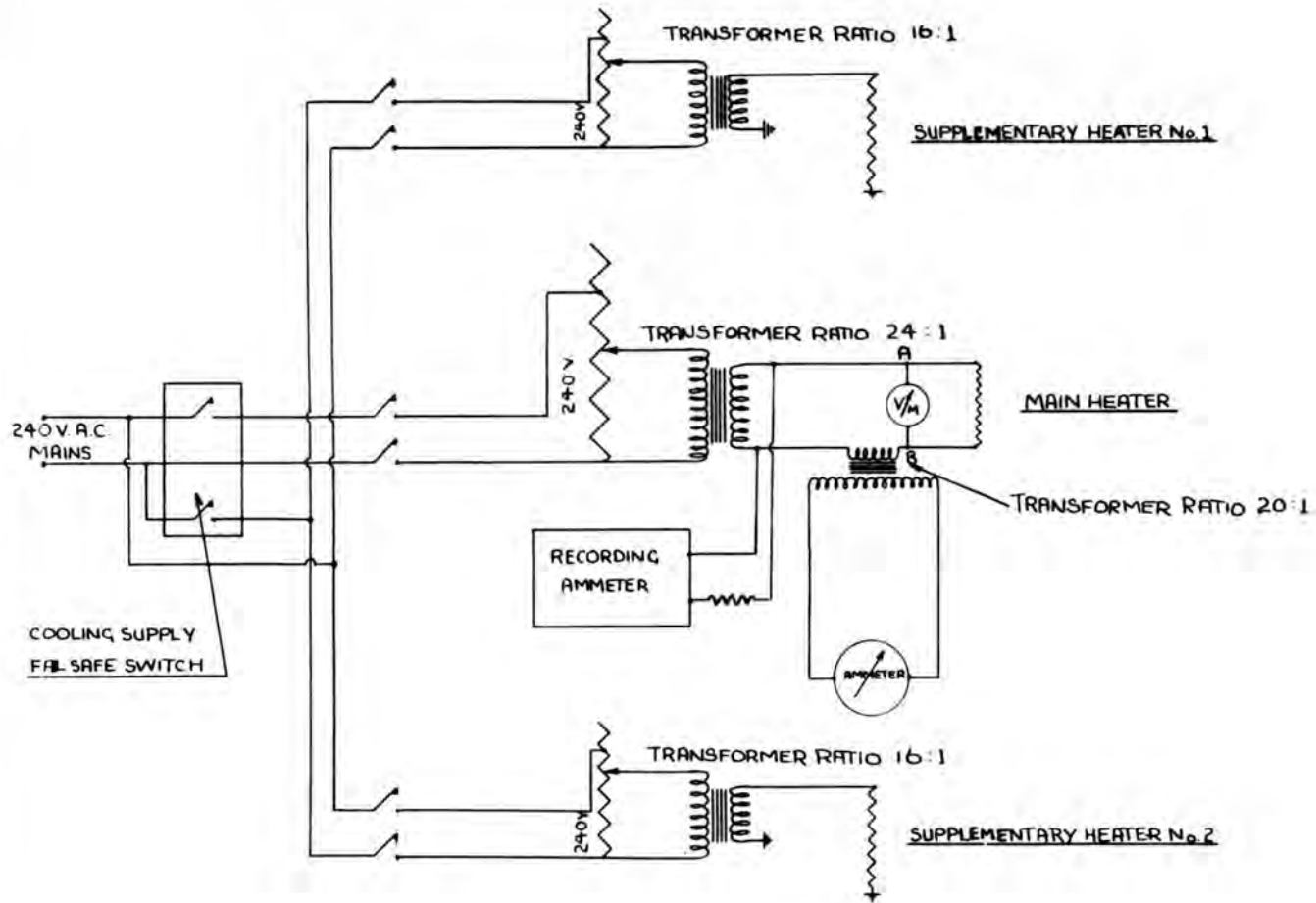
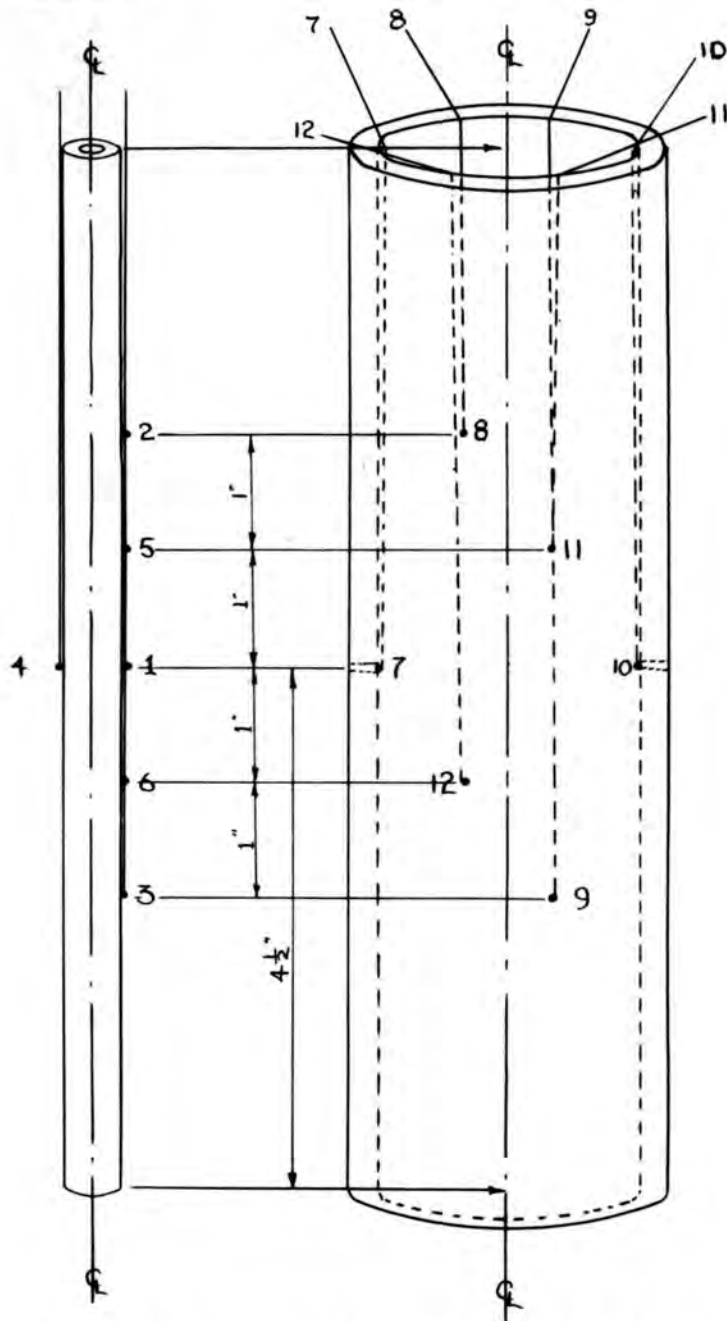


Figure 5

CIRCUIT DIAGRAM SHOWING HEATER SUPPLY AND CONTROL CIRCUITS, AND ALSO SHOWING POWER MEASURING EQUIPMENT FOR THERMAL CONDUCTIVITY APPARATUS.

Figure 6



SCALE DIAGRAM SHOWING POSITION AND SPACING OF THERMOCOUPLES

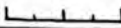
SCALE:  = 1 INCH

Figure 7



were spaced round the circumference of the cylinder and their junctions were attached by gluing them into a small hole. Five of the inner thermocouple wires lay side by side and the junctions were attached by binding them onto a copper shim with wire. The arrangement of the thermocouple junctions is shown in Fig.6. Since the diameter of the inner cylinder was only  $\frac{1}{2}$ " it was thought that the presence of the thermocouple wires might appreciably distort the heat flow pattern and hence introduce error into the measurements of thermal conductivity. A plot of the equipotentials on an electrical analogue of the apparatus showed that they were not significantly altered by the presence of a conductor corresponding to the thermocouple wires.

The thermocouple wires passed through holes in the top graphite block and passed out of the vacuum chamber through an Araldite seal to a Honeywell Multi-point Elektronik Recorder which was calibrated periodically using a Cambridge potentiometer and a standard cell. The cold junctions were immersed in melting ice and were electrically insulated from each other by enclosing each in a glass tube.

### 2.3 Operation of the Apparatus.

The apparatus was packed with powder by disconnecting the electrical heaters and sliding the top graphite block over the thermocouple wires. A semi-circular template

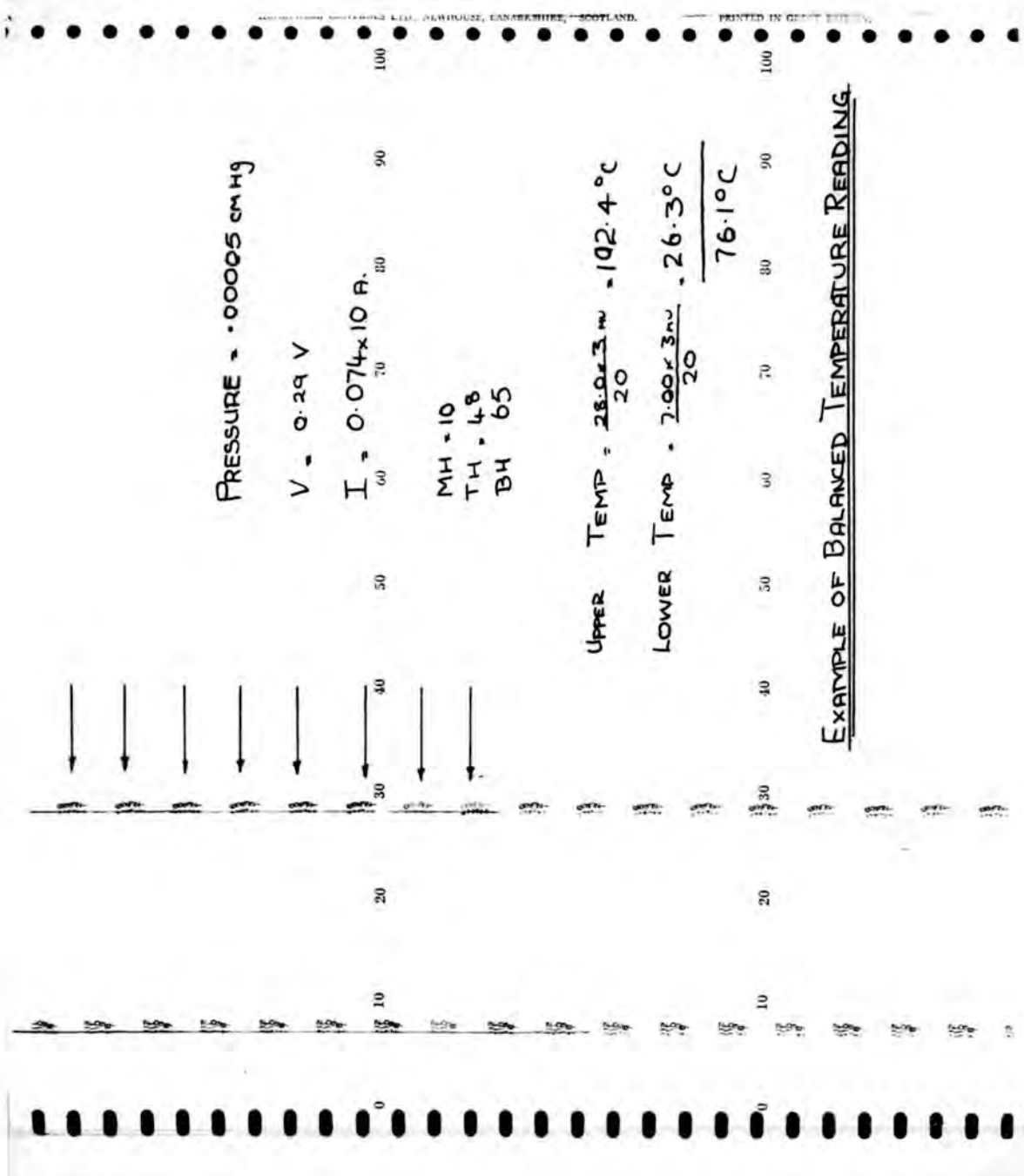


located the inner cylinder and the powder was packed into the annulus, the apparatus being continually tapped. A photograph of this operation is shown in Fig. 7. When the space was tightly packed with powder the top block was replaced, the electrical connections were made and tested and the vacuum dome put into place. A beaker of silica gel was placed inside the dome to indicate excessive moisture if the coiling coil should leak.

The apparatus was next evacuated to  $10^{-4}$  mm. Hg, the pressure being reduced slowly to avoid disturbing the powder, and the apparatus was allowed to de-gas for at least 24 hours. The heaters were used to assist this de-gassing process.

The main heater was next adjusted until the inner cylinder was at approximately the required temperature and the end heaters were then adjusted until the six inner thermocouples and the six outer thermocouples read the same temperatures. The apparatus was considered to be in balance if each set of readings was within a band, one degree centigrade wide, and if the mean temperature of the inner cylinder was within five degrees of the nominal temperatures required. This was a practical expedient because the time taken to balance the apparatus exactly on the nominal temperature would be prohibitive. Using the criterion

Figure 8



ALDRIDGE & CO., ALDRIDGE, LANSHIRE, ENGLAND. PRINTED IN GREAT BRITAIN.

stated above it was possible to take, on average, about one reading each day while the apparatus was running. When this condition was reached the recorder continued to print the results for at least half an hour to show that the apparatus was in equilibrium. A typical chart is shown in Fig.8.

The voltage and current in the main heater circuit and also the interstitial air pressure were then measured. The pressure was then adjusted to another suitable value and the procedure repeated. When sufficient measurements had been made over the whole pressure range the conductivities were calculated as illustrated in Appendix 1.

The apparatus was emptied by placing a tray underneath the graphite cylinders and removing the bottom graphite block.

#### 2.4 Preparation of the Samples of Powder.

Since metal powders are of particular interest in this investigation, aluminium was chosen as being readily available. Six samples were separated from one hundredweight of powder by sieving. The fractions were not sharp cuts, the coarser grades contained some fine powder, but they were six powders of the same material with differing size distributions. The sieve sizes have, however, been used to designate them. The densities, porosities and particle size distributions of the powders were determined as described in Appendix 2.

### 2.5 Experiment 1. Conductivities of the Aluminium Powders

The conductivities of the six grades of aluminium powder were measured over a pressure range from atmospheric to  $10^{-5}$  mm.Hg. About 15 readings were taken at different pressures for each grade, chosen such that the curves were closely defined. The results are given in Table 1. Graph 1 shows a plot of conductivity against pressure for the six powders and Graph 2 shows the same results plotted as conductivity against the logarithm of the pressure. The nominal temperature of the inner cylinder was  $100^{\circ}\text{C}$  for all these experiments except Experiment 4.

### 2.6 Experiment 2. Reproducibility of the Results.

In order to assess the reproducibility of the results the three coarsest grades of powder were packed into the apparatus again and the readings repeated. The readings on the 170-200 mesh grade were taken a third time, without re-packing the apparatus, in order to test the reproducibility using the same packing. The results are given in Table 2 and are also plotted in Graph 3.

The scatter on the results in the atmospheric and intermediate pressure regions is seen to be much less than the differences between the different grades of aluminium and the apparatus is therefore sensitive enough for the purpose intended. From the results of the experiments on the 170-200 mesh grade, the two curves plotted without repacking the powder are not significantly closer than the other pairs of curves and

the scatter can not be attributed only to repacking; probably it is due to a combination of the inherent experimental error and variations of the nominal temperature. This is discussed further in Section 2.8. The asymptotic values of the conductivity at low pressures are not as accurate as the other readings. The points fall on a straight line which suggests that the error is not random but is due to some change in conditions between runs. This is discussed further in Section 2.9. However, it is felt that no significance can be attached to the relative values of these low pressure asymptotic values.

### 2.7 Experiment 3. Boundary Effects.

Several authors have discussed the possible existence of a wall effect or a temperature discontinuity between the boundary and the powder. In order to test whether such an effect does exist the 150-170 grade aluminium was packed into the apparatus with a  $1\frac{1}{2}$ " diameter copper tube inserted coaxially into the powder bed. Three of the thermocouples from the outer cylinder were soldered onto the tube, giving a set of three thermocouples at 2" intervals on both the intermediate and outer tubes and leaving the inner set of six thermocouples intact.

The thermal path now traversed four boundaries instead of two and thus any boundary effect should decrease the conductivity. Otherwise, the presence of a layer of copper and

the smoothing of any asymmetries would tend to increase the effective conductivity. A reduced conductivity would, therefore, indicate the existence of a boundary effect. The results of this experiment are given in Table 3 and are plotted in Graph 4.

It is seen that presence of the tube does slightly reduce the effective conductivity, which implies that the powder and cylinders are not making good thermal contact. A better explanation of the decrease in conductivity is the existence of a region of high porosity near to the wall of a packed powder as discovered by Benenati and Brosilow. Thus, the overall porosity of a powder varies with the geometry of its container (as discussed in Appendix 2) and this is a variable which must be kept constant when comparing the conductivities of different powders. The three thermocouples on the copper tube also gave a value of the temperature at an intermediate radius in the bed and enabled this temperature to be compared with that predicted by Fourier's equation. The comparison is given in Table 3.

The measured and calculated temperatures are coincident providing further evidence that the heat transfer is mainly by conduction.

## 2.8 Experiment 4. Radiation and Temperature Effects.

Several authors have commented on the increase in

conductivity with increasing temperature and also on the contribution made by radiation to the total heat transfer. Allcutt has attributed an apparent variation in conductivity with porosity to radiative transfer and it was thus necessary to clarify this point since it offers a possible explanation of the results shown in Graphs 1 and 2.

The 150-170 grade aluminium was packed into the apparatus and the conductivity measured over the full pressure range for nominal temperatures of the inner cylinder of 100°C, 150°C and 200°C, the  $\pm 5^\circ\text{C}$  scatter round these means being accepted. The outer cylinder was in general between 25°C and 30°C depending on the total heat flow, this temperature not being so critical. It was not considered necessary to make quite as many measurements of conductivity as in previous experiments, to be able to plot the graphs of conductivity against pressure, since their form was now established. The results of this experiment are given in Table 4 and are plotted in Graph 5.

The implications of the results of this experiment, when considering the contribution made by radiative heat transfer, are discussed in the next chapter. However, it is also seen from these results that the variation of conductivity with temperature can be fairly large and that a  $\pm 5^\circ\text{C}$  variation on a nominal inner cylinder temperature can produce appreciable

variations in the measurements of conductivity. It is suggested that much of the scatter of the points about the curves on the graphs may be due to the use of this practical expedient. In future work it may be better to build an apparatus whose boundary temperatures are rigidly fixed, for instance by using steam and ice in order to eliminate this error.

### 2.9 Experiment 5. Residual Conductivity.

The results obtained so far had raised doubts, about the reproducibility of the values of the residual conductivity at low pressures. It was thought that this might be attributable to varying moisture content and that the addition of a cold trap to the apparatus might improve the reproducibility of these readings. This was fitted by surrounding the pipe connecting the pressure gauges to the apparatus with an insulated calorimeter. The  $M50$  grade aluminium was packed into the apparatus which was evacuated to  $10^{-4}$  mm. Hg. and balanced in the usual manner. The calorimeter was then filled with a mixture of methcol and solid carbon dioxide and the effect on the balanced temperature noted. Over a period of three hours the reading was not effectively changed.



CHAPTER 3.INTERPRETATION OF THE RESULTS3.1 Variation of the conductivities with pressure.

The graphs showing the variation of conductivity with gas pressure for the six fractions of aluminium are Graphs 1 and 2. Each of these curves has the general shape found by earlier observers, the conductivity tending to an asymptotic value at higher pressures and a much smaller asymptotic value at low pressures.

The actual values of thermal conductivity at atmospheric pressure are of the same order of magnitude as those found by previous investigators and it is the shape of the curves and the values of conductivity relative to each other which are of interest.

The accepted explanation of the S-shaped curve is that the thermal resistance of the powder is provided mainly by the gas, the heat passing from particle to particle through the interstitial spaces. The small residual conduction at low pressures is due to conduction through the actual areas of contact of the grains and to transfer of heat in the form of radiation. The remaining, and major component, of the conductivity is independent of the pressure of the interstitial gas until the mean free path of the molecules in the gas becomes comparable to the pore size when the conductivity decreases with

decreasing pressure. Although the mechanism is possibly more complicated than this, the results of these experiments give no reason to doubt it as a first order explanation.

### 3.2 Atmospheric Pressure Region.

For all the aluminium powders except the coarsest grade, the  $\gt 150$  fraction, the tendency is for the conductivity at atmospheric pressure to increase with increasing particle size. However, the  $\gt 150$  mesh sample does not conform with this tendency.

The reproducibility of the curves (Graph 3) eliminates the possibility of this result being spurious. In addition it is interesting that the results of Kannuluick and Martin show a similar tendency although they have not thought it significant. From the graphs of conductivity against pressure which they plotted for four grades of carborundum powder with three different interstitial gases they concluded that, in general, the finer the powder the lower the conductivity at atmospheric pressure. However, with each interstitial gas, the conductivity of the coarsest grade is less than that of two other grades of powder at atmospheric pressure and only becomes the greatest conductivity at lower pressures. This is exactly the form of the results in Graphs 1 and 2 and provides strong evidence that this is not a spurious effect.

If the porosities of the powders (Table 8) are studied it is seen that they decrease with increasing particle size of

the sample except for the 200-240 mesh fraction and the  $>150$  fraction, which has a much higher porosity than any of the other grades. Thus it seems that the  $>150$  grade may have a lower conductivity than the others because its porosity is higher. However, the decreasing porosities of the other fractions cannot alone account for the increasing conductivities of the other grades or the  $>150$  mesh grade would have the lowest conductivity of all and the 200-240 mesh fraction would not have a conductivity substantially bigger than the two finest grades. Since there are no other variables but the porosity and the particle size distribution it would seem that the conductivity in this pressure region must also depend on the particle size. In addition, since the porosity depends on the particle size distribution (as discussed in Section 1.4) the conductivity is affected by both the mean particle size and the size distribution.

No mechanism has yet been proposed to account for the dependence of the thermal conductivity of a powder on its mean particle size. It has, however, been recognised that a temperature discontinuity can exist between a solid surface and a gas, the "accommodation coefficient" being the ratio of the energy which a gas molecule acquires when striking a surface to that energy which it would acquire in attaining a speed corresponding to the temperature of the surface. An excellent account of this phenomena has been given by Devienne (17) and some

interesting measurements have been made by Thomas and Olander (59) (see footnote). It is now suggested that this mechanism can explain the tendency of coarser powders to have a higher conductivity at atmospheric pressure. If there is a temperature discontinuity at the surface of each grain in a powder, then the greater number of surfaces which the heat flow must traverse, the greater the thermal resistance of the powder and hence the less the effective conductivity of the powder. Thus it is suggested that a fine powder will have a lower conductivity at atmospheric pressure than a coarse powder of the same material, because a larger number of gas-solid surfaces and hence temperature discontinuities will exist in the heat path, assuming, of course, that the accommodation coefficient for that particular system is less than unity.

#### Footnote

Schumann and Voss imagined the particles to be surrounded by a stagnant layer of gas, as first postulated by Langmuir (34), in order to explain the mechanism of decreasing conductivity when the mean free path of the gas molecules becomes equal to the pore size. It is interesting to speculate whether the boundary layer theory and the accommodation coefficient theory are not two descriptions of the same phenomena.

The effect of porosity on the conductivity of a powder is more easily understood. The greater the volume of the voids, and hence gas of lower thermal conductivity, which fills the heat path the greater the thermal resistance and hence the lower the effective conductivity. Thus a powder whose particle size distribution is such that it packs to a low porosity might be expected to tend to have a relatively higher conductivity.

It should be pointed out that these two mechanisms which have been suggested to explain the dependence of the thermal conductivity of a powder at atmospheric pressure on its mean particle size and its particle size distribution could also account for Allcutt's results on the conductivities of compressed fibres. The porosity of the material will decrease as the material is compressed, tending to increase the conductivity. However, the number of fibre surfaces per unit length of heat path increases with compression, tending to decrease the conductivity. Thus, the conductivity might be expected to have a minimum value at some intermediate porosity. However, Allcutt's own explanation of his results is quite reasonable but could not apply to the aluminium powders as discussed in Section 3.5.

### 3.3 Intermediate Pressure Region.

The  $\gt 150$  mesh curve crosses those of the two coarser grades at intermediate pressures and this fraction then has the

highest conductivity. Previous workers have agreed that the shape of the intermediate portion of the curve will depend largely on the pore size of the packed powder. Since the  $>150$  mesh fraction is much coarser than the others (See Appendix 2 and Graph 8) it is reasonable to assume that the pore size is greater. Thus, a lower pressure must be reached before the conductivity of this fraction decreases because a larger percentage of the pores are still greater than the mean free path of the molecules of the gas. Similarly, it is suggested that the 200-240 mesh grade has a fairly high conductivity at a gas pressure of about 1 cm.Hg. because it has a narrower particle size distribution (Graph 8) than the three coarser grades. Hence the pores will tend to be of larger size than in the 150-170 and 170-200 mesh grades in which the small particles pack between the larger ones. The results are, therefore, concordant with the accepted mechanism of the decrease in conductivity and there seems no doubt that it is the main mechanism.

Thus, over this intermediate region, the value of the conductivity depends on that at atmospheric pressure but the rate at which it decreases from this value depends on the interstitial pore structure. The expression 'pore size' is possibly a misnomer since the pores are not distinct but are a continuous random shape. Also, the section of the pores surrounding the point of contact of two grains may contribute more to the effective conductivity than the rest of the pore,

so that the pore shape may be as important as its size and possibly powders of the same porosity but different pore shape could have a different thermal conductivity. In the case of the aluminium powders the grains are completely irregular and the pore shape cannot be considered as a parameter. The calculation of pore size of a packed powder from a knowledge of particle size is a very difficult problem and has not been solved to date for irregular grains. An interesting paper describing the pore sizes produced by the packing of spheres was published by Wise (64) in 1952 and the technique of Broadbent and Callcott (9) in expressing the particle size distribution of a powder as a column matrix would seem to be a very promising method of describing the bulk behaviour of powders mathematically.

The two methods available for pore size measurement, mercury impregnation and microscopic measurement are not suitable for a loose packed, fine powder. Kistler used his thermal conductivity measurements to deduce the mean pore size of his sample and, although his method of doing this was not very convincing, the idea would seem to be worthwhile. Possibly, as a first order calculation, the pore size distribution could be calculated by differentiating the curve on the graph of conductivity against pressure and postulating that the gradient at any pressure, expressed as a fraction of the steepest gradient, is the fractional area of the pores which is opposite a gap less than the mean free path of the gas molecules at that pressure.

### 3.4 Low Pressure Region.

The asymptotic values of the conductivity at low pressures are not as reproducible or accurate as the other readings. The error must be due to some change of conditions between sets of results because of their consistency for any one run. The magnitude of the residual conductivities is small and thus the other conclusions will not be affected. The obvious explanation is the presence of vapours in the vacuum chamber or as an adsorbed layer on the powder. However, when the cold trap was fitted to the mercury gauges it collected a little mercury but did not affect the thermal conductivity readings. Also, if the thermal conductivities at atmospheric pressure were measured both before and after evacuation of the apparatus, the values did not vary appreciably. Both these facts tend to indicate that the variation in the conductivity readings is not due to the presence of adsorbed layers of moisture. Possibly, oil vapours from the diffusion pump escaped into the system or alternatively the surface of the powders may have become contaminated during the experiments. The fact that the Pirani gauge measurements did not agree with those of the McLeod gauges could possibly indicate the presence of some vapour.

These readings are not sufficiently reproducible to be reliable and it would seem desirable that they be repeated and form the basis of a separate investigation so that the mechanism at very low pressures may be elucidated further.



### 3.5 Radiation and Convection Effects.

In Experiment 3 it was found that Fourier's Law was obeyed, indicating that the heat transfer is mainly by conduction. Providing the average particle size is much smaller than the total heat path, the postulation of a temperature discontinuity at the grain surfaces would not be expected to affect this result.

However, if radiation is a predominant mechanism of heat transfer, the temperature distribution would be altered. It seems, therefore, that heat transfer by radiation is not appreciable. To test this further Graph 5 shows the conductivity of the 150-170 mesh grade at three different boundary temperatures. As was expected the effective conductivity increases with increasing temperature. However, the curves come close together at lower pressures, whereas, if the increase was attributable to radiation, they would be parallel. The increase in conductivity is probably due to the increased conductivity of the gas, therefore, and much higher temperatures would have to be attained to make radiative transfer appreciable.

The consideration of radiative transfer is important because of the possibility that the differences in conductivities of the grades may be explained in similar fashion to Allcutt's results. He attributed the high conductivity of fibrous materials at low densities to increased heat transfer by radiation and

convection. A preliminary calculation, given in Appendix 3, had shown that it might be feasible for radiation to contribute an appreciable portion of the heat transfer. It is now clear, however, that Allcutt's explanation cannot apply to the aluminium powders. Convection is not possible with such fine powders and with no vertical temperature gradient. Heat transfer by radiation has been shown to be small and further, even if it accounted for the whole of the residual conductivities, which is unlikely, they are not large enough to account for the differences in conductivity of the powders at the higher pressures.

It is concluded, therefore, that radiative heat transfer is not an important mechanism in this case and cannot explain the variations in the conductivities of the aluminium powders.

CHAPTER 4.CONCLUSIONS.4.1 Statement of the Hypothesis.

The results of the experiments described in Chapter 2 can now be explained in the following manner.

A temperature discontinuity can exist at a gas-solid interface and hence a powder of small grain size tends to have a low conductivity because the heat path must traverse a greater number of surfaces. The conductivity also depends on the relative quantities of gas and solid which the heat flow must traverse. A powder of high porosity will tend to have a low conductivity. Since the porosity of a packed powder depends primarily on the relative quantities of different sized grains, its effective conductivity at atmospheric pressure depends on both the mean particle size and on the size distribution. The conductivity of a powder is independent of the interstitial gas pressure until the mean free path of the molecules of the gas becomes comparable to the pore size of the powder. Hence the shape of the curve, on a graph of conductivity plotted against pressure, depends on the pore size distribution of the packed powder.

This hypothesis explains the dependence of thermal conductivity on the physical properties of the powder itself. Several other parameters which are not properties of the powder

can influence the conductivity, one important one being temperature. The conductivity of a powder increases at higher temperatures but this is due mainly to an ~~increase in~~ conductivity of the interstitial gas. Radiative heat transfer does not contribute appreciably to the total transfer at temperatures up to 200°C.

#### 4.2 Experiment 6. Confirmation of the hypothesis.

Having now carried out a series of experiments with the six different grades of aluminium powder and having postulated a mechanism to explain the results of those experiments, it was decided to perform an experiment which would test the hypothesis. As has already been pointed out, quantitative prediction of conductivities is very difficult and hardly worthwhile at the moment. Further, it is very difficult to vary the mean particle size and the porosity of a powder independently. It was decided, therefore, to qualitatively predict the results of an experiment. If the finest and coarsest grades of powder are mixed in different proportions, the fine particles will tend to reduce the porosity of the coarse grade, by filling the interstitial spaces, and hence increase the conductivity. However, the decrease in average particle size will tend to decrease the conductivity. (In fact, if the hypothesis of Section 3.2. is correct, the conductivity might be expected to be directly proportional to the mean particle sizes at constant porosity.) With the different mixtures of these two powders, if

the mechanism described in section 3.2 is correct, a graph of the conductivity against the percentage constitution of the mixture might be expected to pass through a maximum, having lower conductivity with large average particle size and high porosity and with small particle size and low porosity than at some intermediate mixture. The conductivity of the 150 grade was measured over the full pressure range and then gradually increasing percentages of the 300 grade added. The conductivity of each mixture was measured at low pressure (i.e.  $10^{-3}$  m.m.Hg) and at atmospheric pressure. When the atmospheric readings appeared to have passed through a maximum, as expected, the readings were taken over the full pressure range again as they were yet again ~~at a high concentration~~ of < 300 grade. If the mean pore size is gradually decreasing as the percentage of fine powder is increased, the decrease in conductivity with pressure might be expected to occur at higher pressures and thus the experiment also acted as a check on the mechanism of the reduction in conductivity with gas pressure, as described in section 3.4. The results are given in Table 5 ~~and~~ SB. Graph 6 shows a plot of the conductivity at atmospheric pressure against the percentage composition of the mixture. In order to reduce the time of the experiment the balancing of the thermocouple temperatures, using the compensating heaters, was not done quite so rigorously as in previous experiments since it was only required to establish the shape of the curve. However, an

estimate of the error has been made and a smooth curve drawn through the points on the graph. The porosities of the mixtures were also measured (as described in Appendix 2) and are plotted against the percentage composition of the mixture on Graph 6 underneath the conductivity graph. The third curve on Graph 6 is simply the straight line decrease of the mean particle size. This line is fixed by reading the fifty percentile diameters for the  $>150$  and  $<300$  grades of powder from the graphs of cumulative percentage undersize by weight against Feret's diameter in Graph 9. These values are approximately a linear diameter of the particles averaged with respect to their volume, which is the relevant one since the linear diameters of the particle determine the number of surfaces in the heat path and the volume determines the proportion of the bed affected by that diameter. Values of the conductivity and the interstitial gas pressure for the  $>150$  mesh grade, mixtures containing 20% and 60% by weight of the  $<300$  mesh grade, and the  $<300$  mesh grade are given in Table 5. In order to compare the shapes of the curves they have all been scaled up to give a conductivity at atmospheric pressure of ten arbitrary units. The results are shown in Graph 7.<sup>8</sup>

From Graph 6 it is seen that the porosity does initially decrease with increasing proportion of the  $<300$  grade until the finer grade constitutes 40% by weight of the mixture when it starts to increase again. Thus the 40% mixture contains the right proportion of fines to fill the spaces between

the large particles and has the minimum porosity.

However, the maximum conductivity occurs for the 20% mixture and is clearly the predicted maximum due to the opposing influences of the decreasing particle size and porosity. Further, it is possible from the curve to compare the conductivities which powders with the same porosity but differing mean particle size would have. This experiment confirms that a decrease in the mean particle size can cause a substantial decrease in the effective conductivity.

The curves in Graph 7 show that the conductivities of the mixtures containing 60% and 100% by weight of the  $\leq 300$  mesh grade decrease more rapidly than those containing 0% and 20% of this grade. This again supports the conclusions of Section 3.4, since the coarser powders will probably have a greater average pore size. Also, these results show the feasibility of differentiating between different pore structures by this means although these results are not accurate enough to separate the powders further.

#### 4.3 Suggestions for Further Work.

The experiments described in this thesis have obviously not completed the necessary work. It would be interesting to see how measurements using different powders compared with those described, particularly if the accommodation coefficient could be measured for the relevant gases and solids. The readings at low pressures are unsatisfactory and should be repeated. If

an apparatus was constructed such that the temperature of the whole bed was in excess of  $100^{\circ}\text{C}$  it might help to remove doubts about adsorbed layers of moisture. Further, the temperature range investigated is very narrow and readings of the conductivity at very high temperatures might show radiative heat transfer to be more important in this region. In future investigations it might be advantageous if the temperature of the boundaries were fixed to reduce the scatter due to the variation of the nominal temperatures.

The packing behaviour of powders, its reproducibility and the factors which affect it obviously need further investigation. As stated previously it is necessary to be able to define a system exactly before measurements of its properties have any meaning.

Previous workers have found some powders to have conductivities less than that of air at reduced pressures but not at atmospheric pressure. It is interesting to speculate whether a very fine powder of close size distribution ground from a material with a low accommodation coefficient might not make a very efficient thermal insulator at atmospheric pressure.

Schafer and Grundler used their measurements of conductivity to deduce the conductivity of gases at high temperatures where they are not easily determined by other methods. This idea seems worthwhile. Further, it may be possible to compare the mean free paths of the molecules of



different gases by measuring the variation of conductivity with pressure of a powder containing each gas in turn or to measure the variation in mean free path of molecules with temperature by the same method. This, therefore, may be a valuable method of measuring the mean free path of the molecules of a gas.

The paradox of increased heat flow with reduced temperature difference found by Fulk would seem to invite further investigation. At the low temperatures attained in Fulk's apparatus, the interstitial gas must have been near to its liquifaction point and it might be worthwhile making measurements with an interstitial gas which is close to its liquifaction point.

The existence of a temperature discontinuity at a solid gas interface is a phenomenon which requires further investigation. One interesting explanation of the mechanism is that the molecules are adsorbed onto the solid surface and are emitted again when they undergo a collision with a phonon of suitable energy. Possibly a medium consisting of a large number of laminae instead of grains might produce a measurable total temperature drop at the surfaces and enable the dependence of the accommodation coefficient on parameters such as the nature of the solid, gas temperature and gas pressure to be investigated.

In Section 3.3, it was suggested that the pore size

distribution of a powder might be deduced from a knowledge of the variation of its conductivity with gas pressure. It is suggested that possibly a measurement of the variation of the permeability of a bed of powder to gas flow at various gas pressures may be an easier method of doing this since a transition from "Poiseuille" to "Knudsen" flow might be expected to occur when the mean free path of the molecules is comparable to the pore size.

APPENDIX NO. 1.Calculation of Conductivities.

The method of calculating thermal conductivities from the voltage, current and temperature readings is described below using the measurements on the 150-170 grade of powder from Experiment 1 to illustrate the procedure. The complete set of readings is given in Table 6 and the significance of the various readings is described below.

Column 1. Records the r.m.s. voltage between points A and B in Fig. 5.

Column 2. Sometimes the voltage scale had to be moved from 0-2.5 volts to 0-10 volts and the readings taken on the two scales were not consistent. The lower range was taken as the reference scale, therefore, and the other readings corrected to this. Twenty readings of voltage were made on both scales and the ratio of the readings found to be  $1.038 \pm .001$ . The voltages taken on the 0-10 volt range are, therefore, multiplied by 1.038.

Column 3. Records the r.m.s. current passing through the secondary of the current transformer.

Column 4. Shows the product of the voltage and current.

Column 5. Shows the total power dissipated between points A and B. The Avometers were calibrated against a sub-standard wattmeter by taking successive readings of voltage, current and

power, the voltage being taken on the 0-2.5 volts scale. The mean of twenty values of the ratio of power to the product of the voltage and current was found to be  $0.963 \pm .003$ . Assuming the wattmeter to be reliable, therefore, this factor, includes corrections for the scales of the Avometers and the power factor of the circuit. When multiplied by the ratio of the current transformer this is the factor for converting the product of voltage and current to the total power dissipated between points A and B. The reduction ratio of the transformer was 20 and this was checked by making ten measurements of the currents passing through primary and secondary of the transformer with the two Avometers and then repeating the process with the Avometers interchanged. The final conversion factor was thus  $20 \times 0.963 = 19.26$ .

Column 6. Shows the ratio of the voltage and current the average of which is proportional to the electrical resistance between points A and B.

Column 7. Shows the power, in watts, dissipated in one centimetre of the main resistor wire. The resistance of 1 cm of the main heater wire was .00731 ohms. This value was obtained, both by calculation knowing the resistivity of constantan, and by comparison of the actual wire with a standard ohm. The electrical resistance between points A and B varied slightly each time the connections were made. It was measured during the calibration of voltage, current and power by connecting a standard one ohm

resistor in series with the circuit and measuring the respective potential differences when a D.C. current of 0.00731 ampere voltage was applied. The average of three measurements was .2319 ohms  $\pm$  .00011. In this case, therefore, the total power was multiplied by  $\frac{.00731}{.2319}$  to obtain the power dissipated in 1 cm. of the wire. For the other measurements it was not necessary to measure the resistance again. The average ratio of voltage to current during this calibration was 4.325 and the factor to convert the total power to power per unit length of wire in the other experiments was thus taken as

$$\frac{.00731}{.2319} \times \frac{4.325}{\frac{V}{I}} = \frac{.13633}{\frac{V}{I}}$$

where  $\frac{V}{I}$  is the average voltage to current ratio for the particular experiment.

Column 8. Records the mean temperature of the inner cylinder, which was within 5°C of the nominal temperature aimed for. The temperatures are calculated to the nearest 0.1°C in order to make the errors of calculation smaller than those of measurement.

Column 9. Records the mean temperature of the outer cylinder.

Column 10. Shows the temperature difference between the cylinders.

Column 11. Records the air pressure measured by the McLeod gauges or the manometer.

Column 12. Records the reading on the Pirani gauge. These readings are not directly comparable with those of the McLeod gauges but serve simply as a check that the McLeod gauge has

not been read incorrectly.

Column 13. Is the ratio of the power dissipated in one centimetre of wire and the temperature difference between the cylinders. For convenience, it has been multiplied by a factor of  $10^3$ .

Column 14. Shows the calculated values of the thermal conductivity. For a concentric cylinder arrangement it is given by

$$K = \frac{\log_e \frac{r_2}{r_1}}{2} \frac{H}{(\theta_1 - \theta_2)}$$

K = thermal conductivity

$r_2$  = diameter of outer cylinder

$r_1$  = diameter of inner cylinder.

H = heat conducted per unit length of cylinder per unit time

$(\theta_1 - \theta_2)$  = temperature difference between cylinders

The radii of the cylinders were measured with vernier calipers, the averages of ten measurements of each being 1.291 cms and 5.696 cms<sup>±</sup>. Thus  $\frac{\log_e \frac{r_2}{r_1}}{2} = 0.23625$  and the thermal conductivities are calculated by multiplying  $\frac{H}{\theta_1 - \theta_2}$  in column 13 by this factor. Again the conductivities are multiplied by a factor of  $10^3$  for convenience.

Column 15. Records the settings of the various transformers. It was thus possible to check the approximate power readings if an obvious mistake was suspected in the power readings.

APPENDIX 2Properties of the Aluminium Powders.Density of the Aluminium.

The density of the aluminium was found by liquid displacement using methcol. Three determinations of the density of each of two grades, the 150-170 and the <300 mesh fractions, were made. They coincided exactly, the specific gravity of the two powders being 2.666 gms/cc and this was therefore assumed to be the specific gravity of all the powders.

Experiment 8. Particle Size Distribution of the Powders.

There are many ways of measuring the particle size distribution of a powder, but they all have limitations and the results must be interpreted carefully to avoid ambiguity. Unless the particles are spherical, there are a large number of sizes which can be measured. An analytical technique must, therefore, be chosen which measures the relevant parameters.

In this case the particle size analyses were required primarily to compare the size distributions of the six powders which had already been graded by sieving. In order to obtain fairly comprehensive results the powders were analysed by two different methods, microscope counting and photosedimentation.

Microscope Count Analysis.

The microscope count consisted simply of measuring the individual size of a number of particles, which were dispersed on a microscope slide, using an eye piece vernier. The

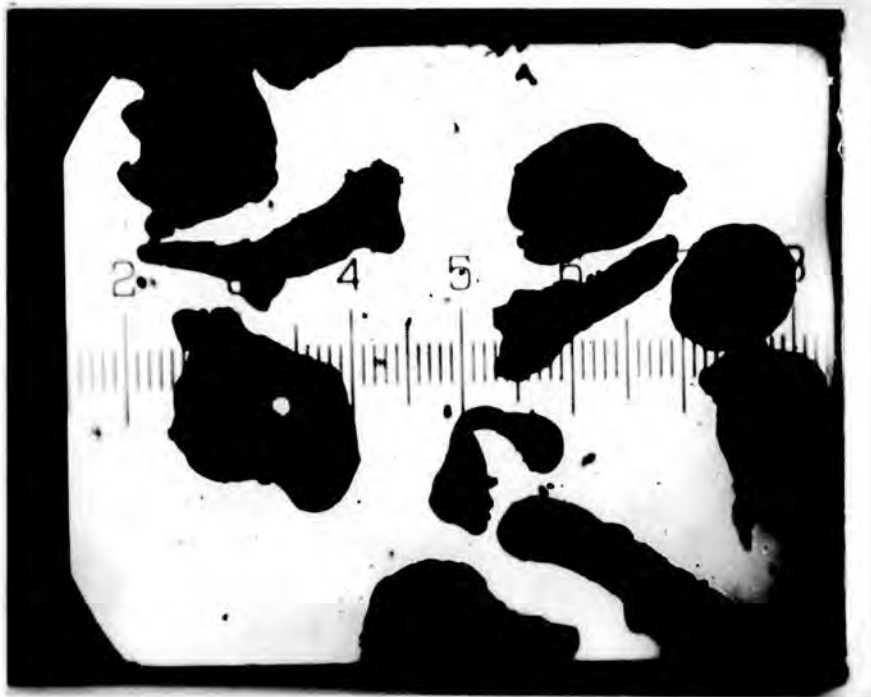
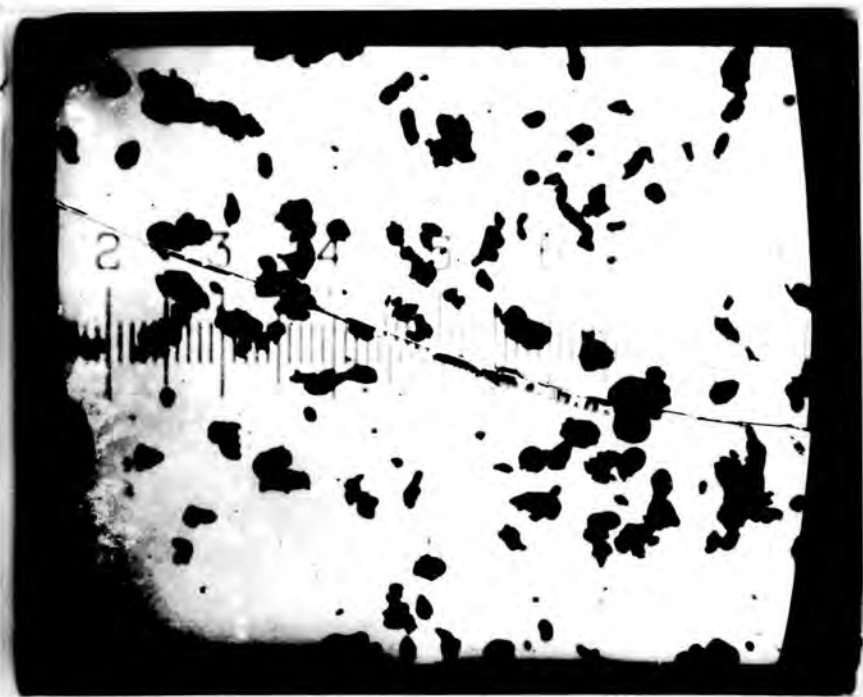


Figure 9. Photomicrographs of the  $>150$  and 170-200 mesh aluminium powders.





vernier was calibrated using a stage micrometer.

It was felt that the 'Ferret's diameter' was the most practical one to measure with such irregular particles. Ferret's diameter is the distance between the extremities of the particle in an arbitrarily fixed direction. Approximately 200 particles in each powder were measured and the results are given in Table 9A and are plotted as an  $\sum nd^3$ :d, cumulative percentage undersize curve, in Graph 8<sup>7</sup>, where n is the number of particles of size d. A photomicrograph, illustrating the irregular nature of the grains of aluminium powder<sub>4</sub>, is shown in Fig. 9.

#### Analysis by Photosedimentometer.

The aluminium powders were also analysed in a photosedimentometer which was constructed using the principle of a 'Bound Brook Photosedimentometer' (67). The three finest grades of aluminium were suspended in water and the other three grades in a mixture of water and glycerine. Two samples of each grade of powder were analysed when using this technique. In each case the results of the analyses of the two samples were similar and were combined to be converted to a cumulative percentage undersize by weight curves. Care must be taken that the concentration of powder in the suspension is low, otherwise the particles do not fall independently. The results of the analyses converted to a  $\sum nd^3$ :d curve, are given in Table 9B

and are plotted in Graph 8<sup>7</sup>.

### Results of the Analyses.

The analyses by the two different methods do not coincide, of course, the Feret's diameter measured by the microscope count method is larger than the mean projected diameter and the Stokes' diameter measured by the photosedimentometer for such an irregular powder. However, the relative size distributions of the powders seem to be represented quite well, although the sieve apertures have little relevance. This is not surprising since the sieving was carried out as a nest operation and was not intended to give precise separation but powders of different size distributions. The microscope count results give a realistic estimate of a linear diameter of the particles and the photosedimentation results confirm the relative size distributions of the powders. These analyses, therefore, give sufficient information for the purposes of this investigation.

### Experiment 9. Porosity of Powders.

To determine the porosity of the powders, if the density of material is known it is necessary only to measure the volume occupied by a known weight of powder. The porosity is defined as  $\frac{\text{volume occupied by Voids}}{\text{volume occupied by powder}}$ .

The volume occupied by the powders in the thermal conductivity apparatus could not easily be measured directly and it was decided, therefore, to measure the porosities of the

powders independently. The powder was poured into a measuring cylinder, which was tapped until the powder did not subside further, when the volume and weight of the powder were determined. Hence the porosity of the powder was deduced. Each powder was measured in three different cylinders of differing diameters, and each of the measurements was made in triplicate. The results are shown in Table 7A.

As might be expected, the scatter of the results is appreciable since the surface of a packed powder is not always level or easily defined. However, the variation between the porosity of the same powder when packed into different tubes shows a greater variation and clearly the effective porosity of these powders depends upon the geometry of the container. Since the same weight of powder had not been used for each measurement the porosity of the 170-200 mesh powder was measured several times with different amounts of powder. The results are shown in Table 7B. No systematic variation existed but the standard deviation on these readings,  $\pm 0.005$ , is an estimate of the scatter to be expected on the other sets of triplicate readings. The powders tend to pack to a lower porosity in the smaller tubes although the 170-200 mesh powder results show that it is not possible to generalise from these results. Clearly, further investigations into the packing behaviour of powders are necessary.

However, for the purposes of these experiments it is

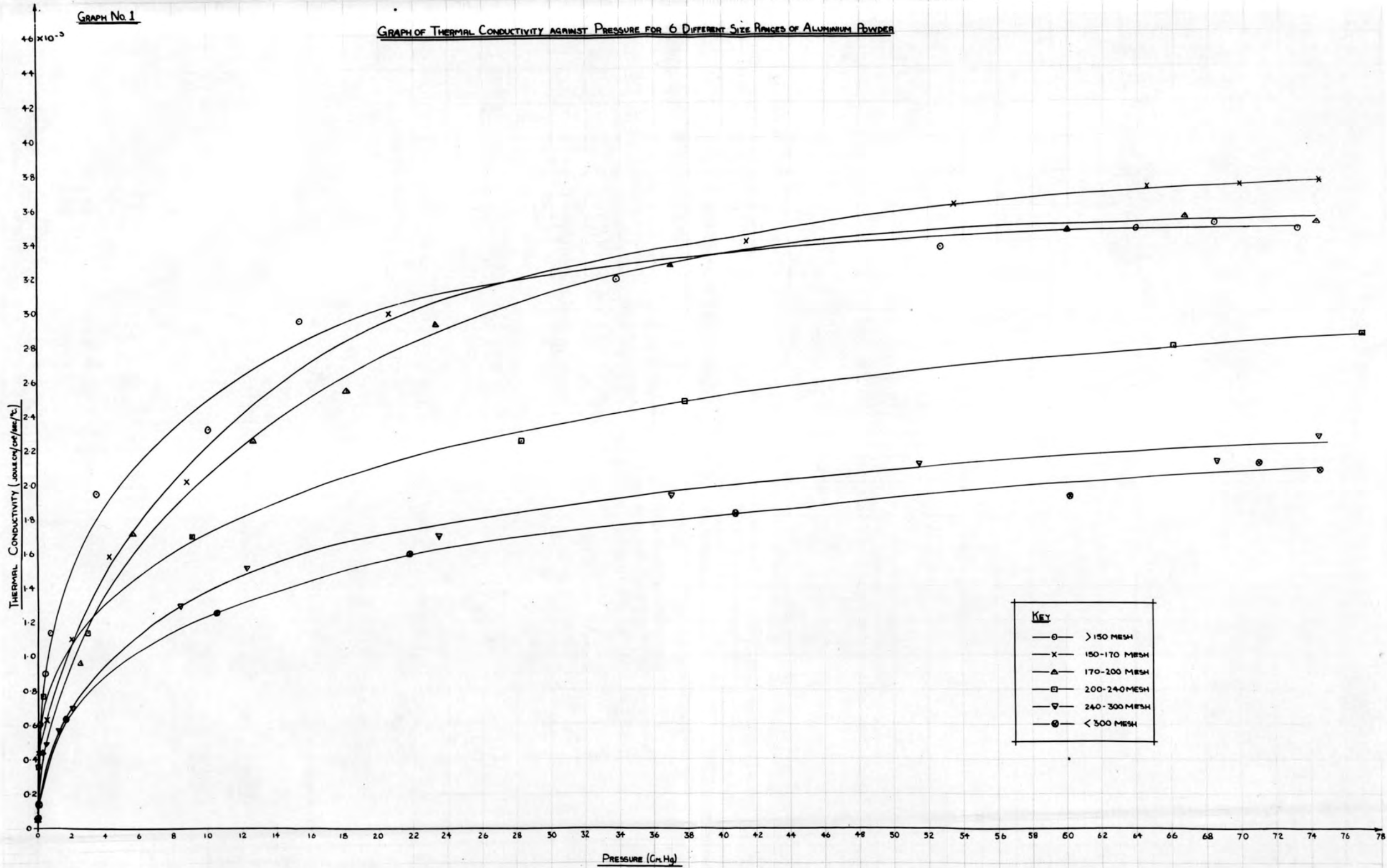
only necessary to know the porosity of the powders in the thermal conductivity apparatus.

A concentric cylinder arrangement of the same dimensions as the thermal conductivity apparatus was constructed and the porosities of the powders when packed into the apparatus were measured. Again, each experiment was performed in triplicate. The height of the powder bed was measured at four fixed points and the average taken. The powder was packed by tapping the sides of the cylinder as in the actual thermal conductivity apparatus and weighed after each volumetric measurement.

The porosities of the powders are shown in Table 8 . They all fall into the same order as when measured in the other tubes and the weights of the powder used in the actual thermal conductivity apparatus corroborate these results. For the purposes of considering how the porosity affects the thermal conductivity, therefore, the results from the concentric tube apparatus are sufficient.

GRAPH No. 1

GRAPH OF THERMAL CONDUCTIVITY AGAINST PRESSURE FOR 6 DIFFERENT SIZE RANGES OF ALUMINIUM POWDER



Case No. 2

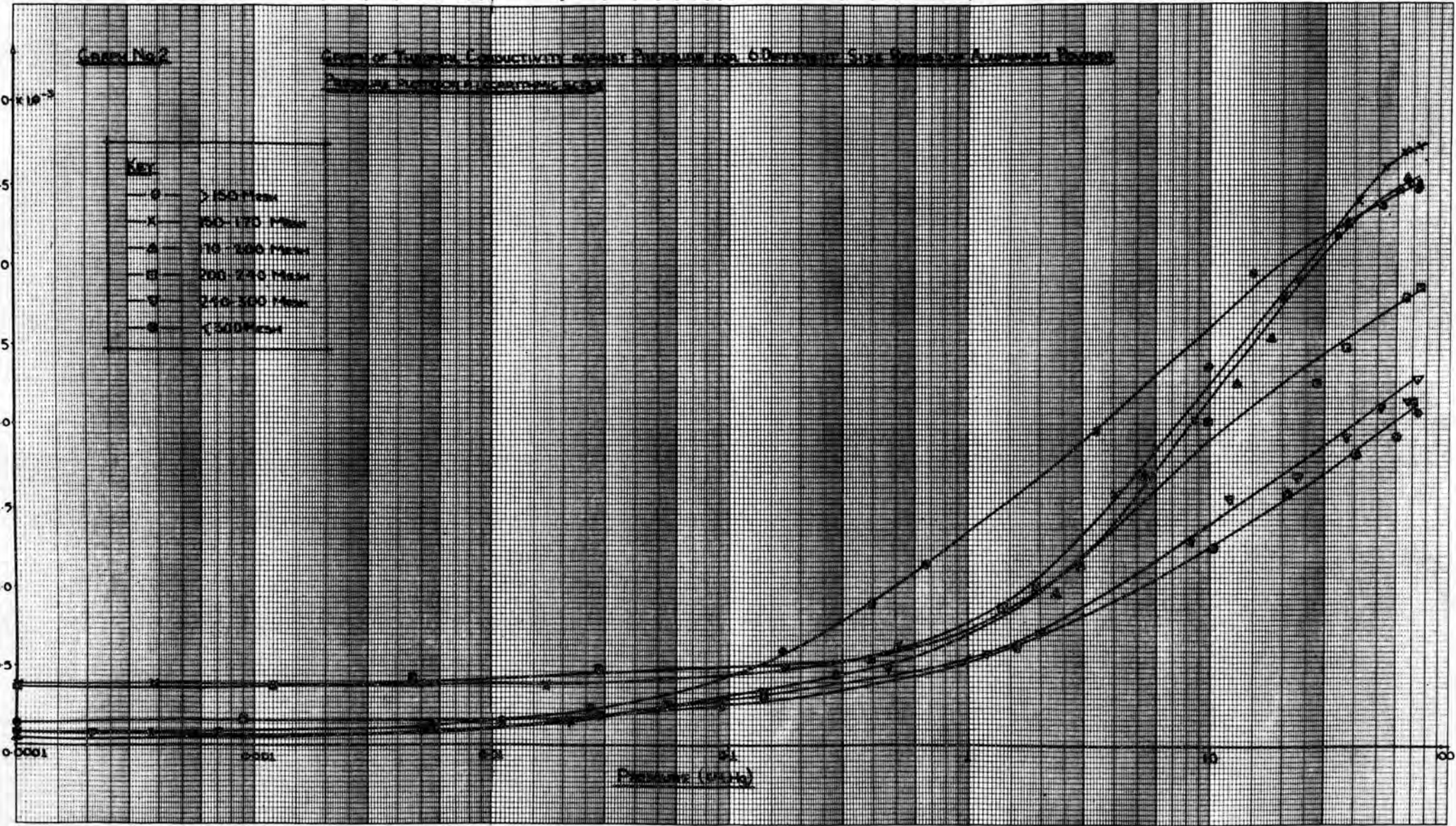
Graph of Thermal Conductivity against Pressure for 6 Different Size Grains of Aluminum Oxide

Pressure in Atmospheres

THERMAL CONDUCTIVITY (JOULE CM/SEC/°C)  $\times 10^{-3}$

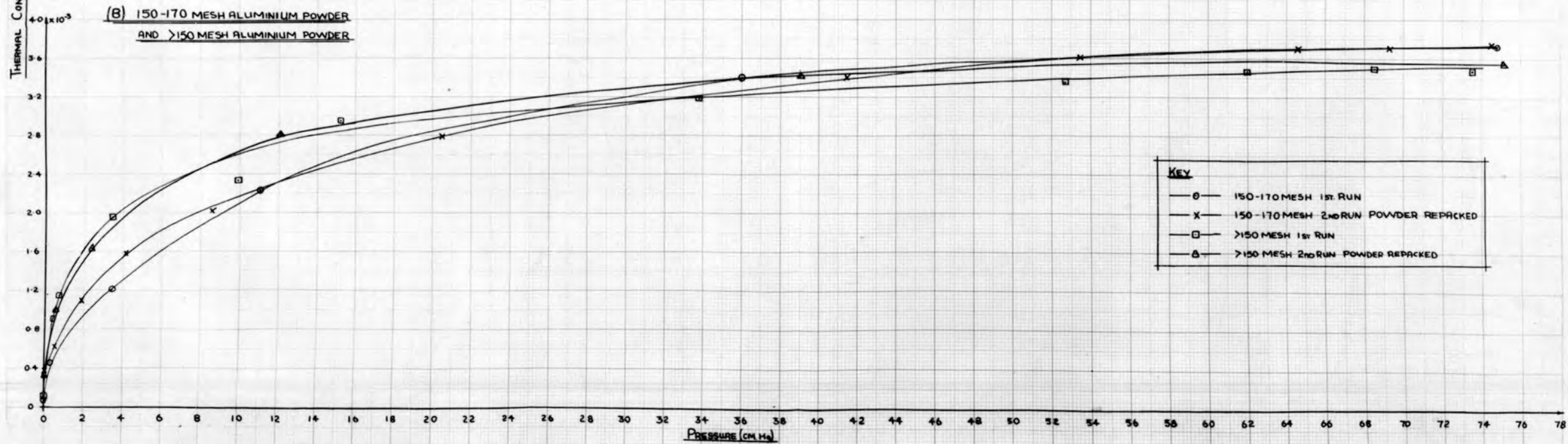
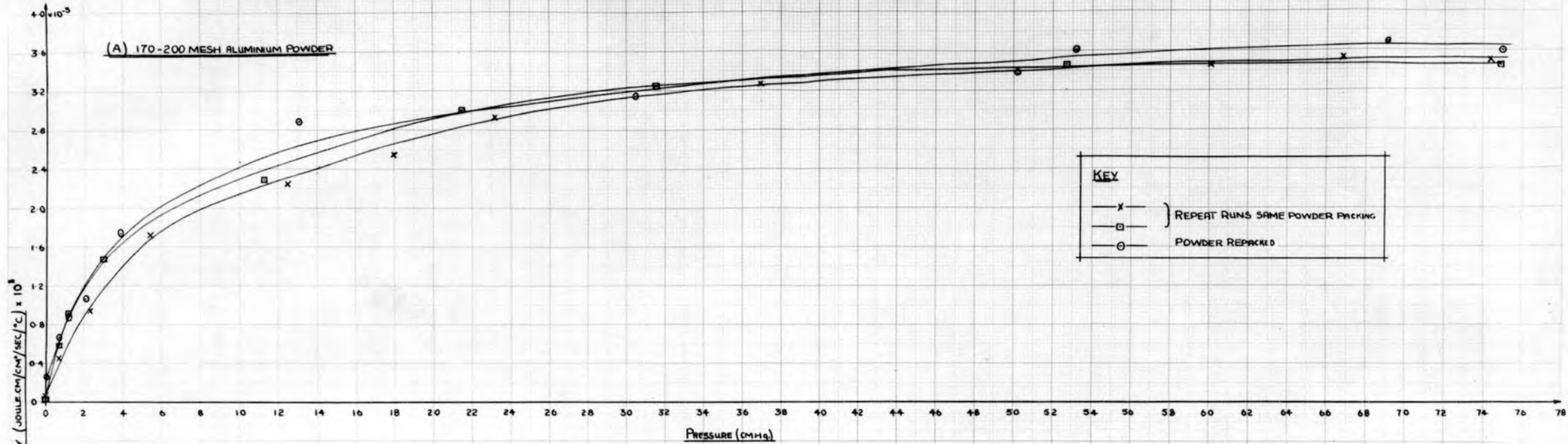
Symbol	Grain Size Range (microns)
○	> 500
×	150 - 175
△	75 - 100
□	200 - 250
◇	250 - 300
●	< 50

Pressure (atm)



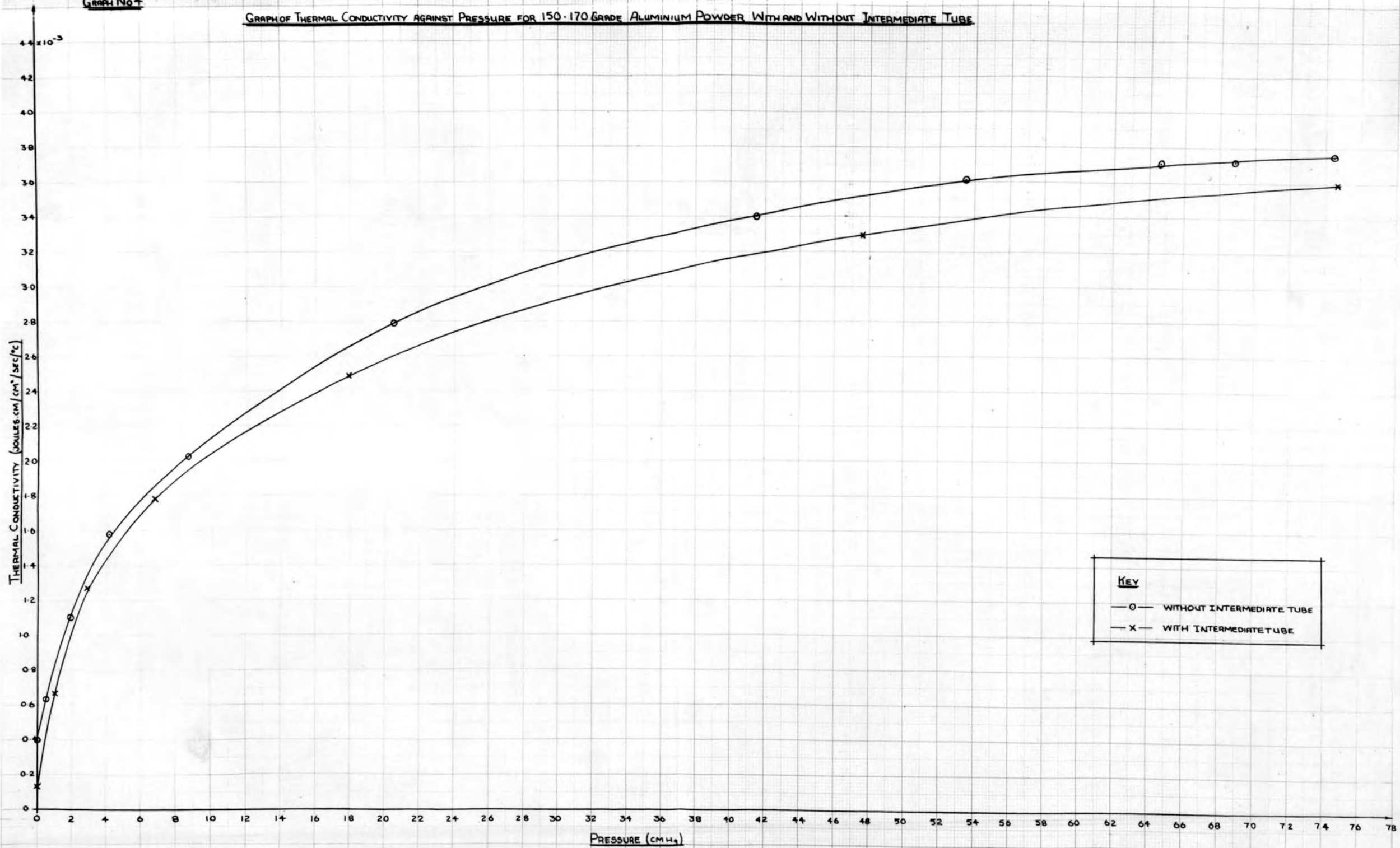
GRAPH No. 3

GRAPHS OF THERMAL CONDUCTIVITY AGAINST PRESSURE SHOWING THE REPRODUCIBILITY OF THE CONDUCTIVITY RESULTS



Graph No 4

GRAPH OF THERMAL CONDUCTIVITY AGAINST PRESSURE FOR 150-170 GRADE ALUMINIUM POWDER WITH AND WITHOUT INTERMEDIATE TUBE

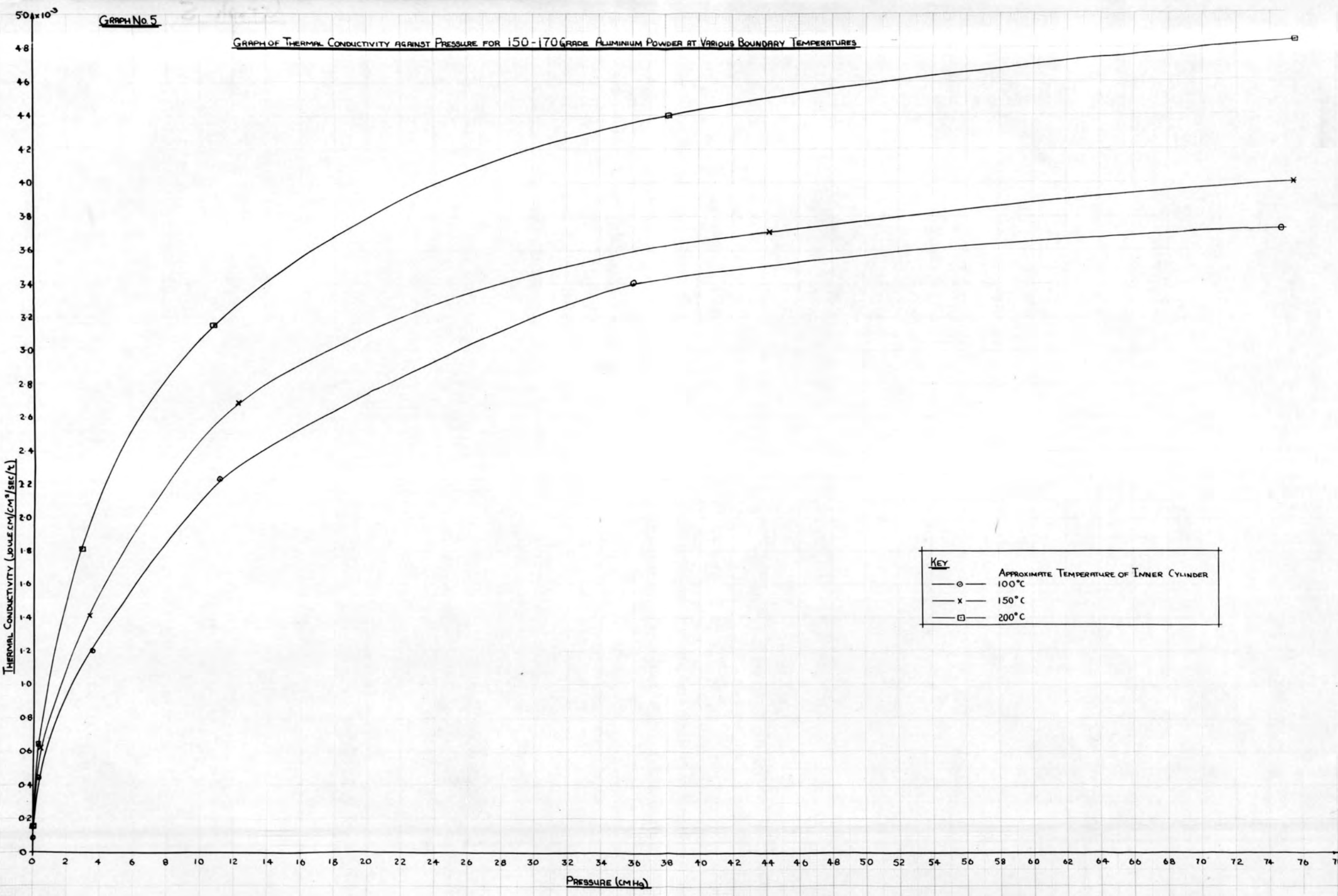


**KEY**  
—○— WITHOUT INTERMEDIATE TUBE  
—x— WITH INTERMEDIATE TUBE



GRAPH No. 5

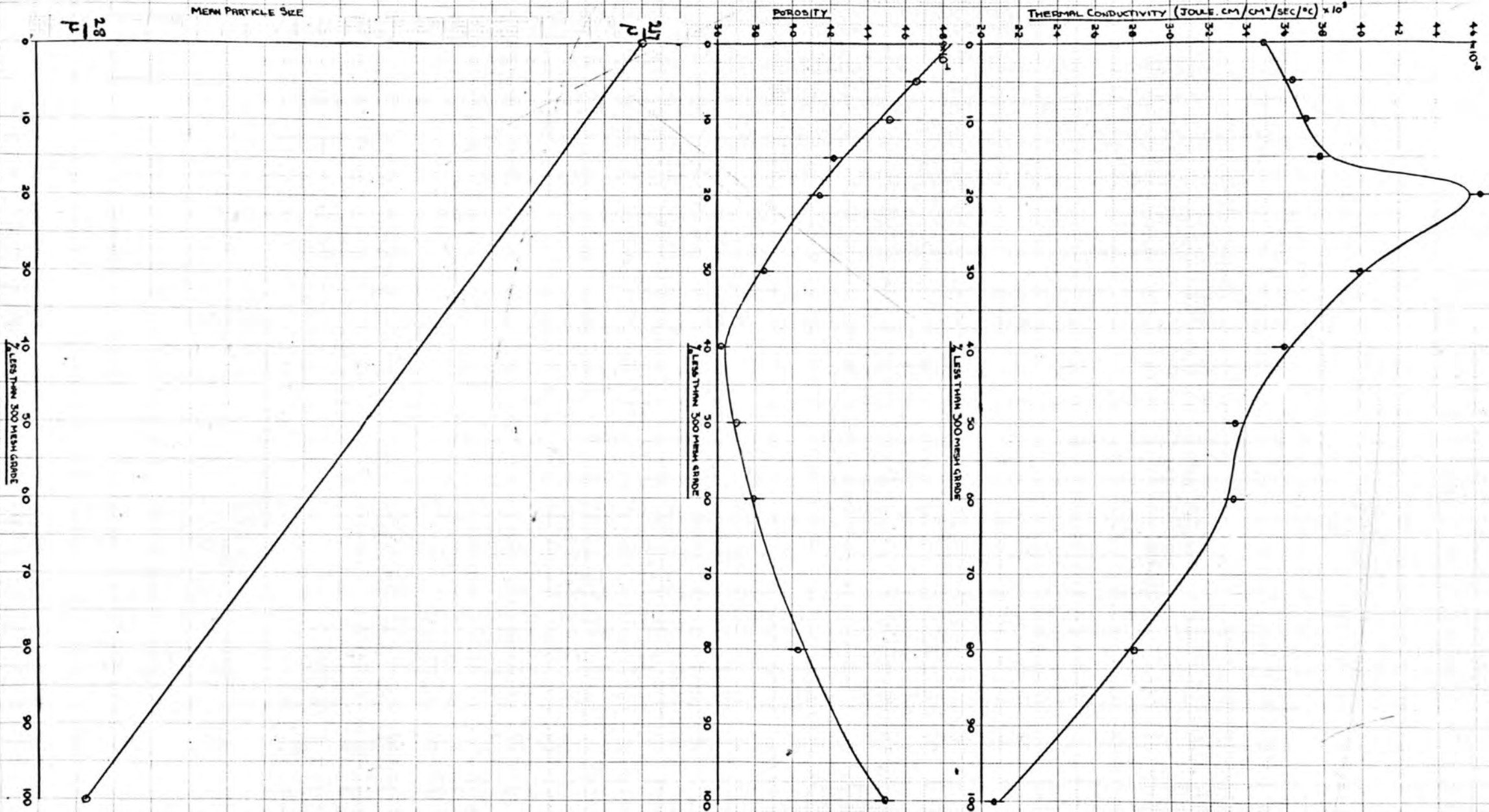
GRAPH OF THERMAL CONDUCTIVITY AGAINST PRESSURE FOR 150-170 GRADE ALUMINIUM POWDER AT VARIOUS BOUNDARY TEMPERATURES



**KEY**  
APPROXIMATE TEMPERATURE OF INNER CYLINDER  
—○— 100°C  
—x— 150°C  
—□— 200°C

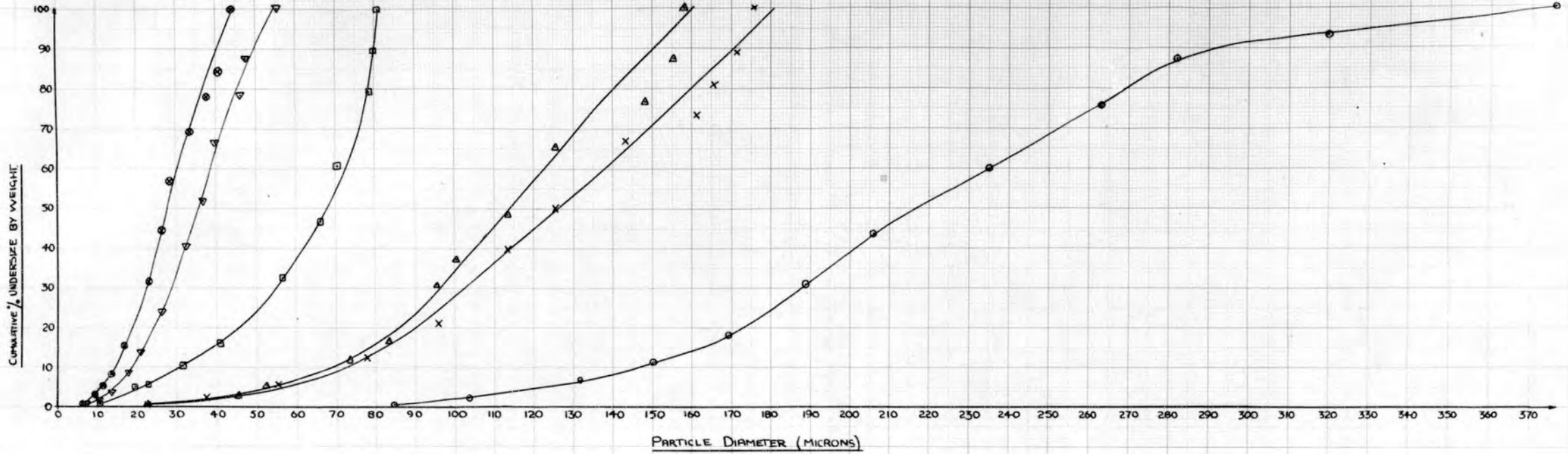
GRAPH No 6

THE CONDUCTIVITY AT ATMOSPHERIC PRESSURE OF THE MIXTURES OF >150 AND <300 MESH GRADES  
ALSO GRAPHS SHOWING POROSITY AND MEAN PARTICLE SIZE OF THESE MIXTURES

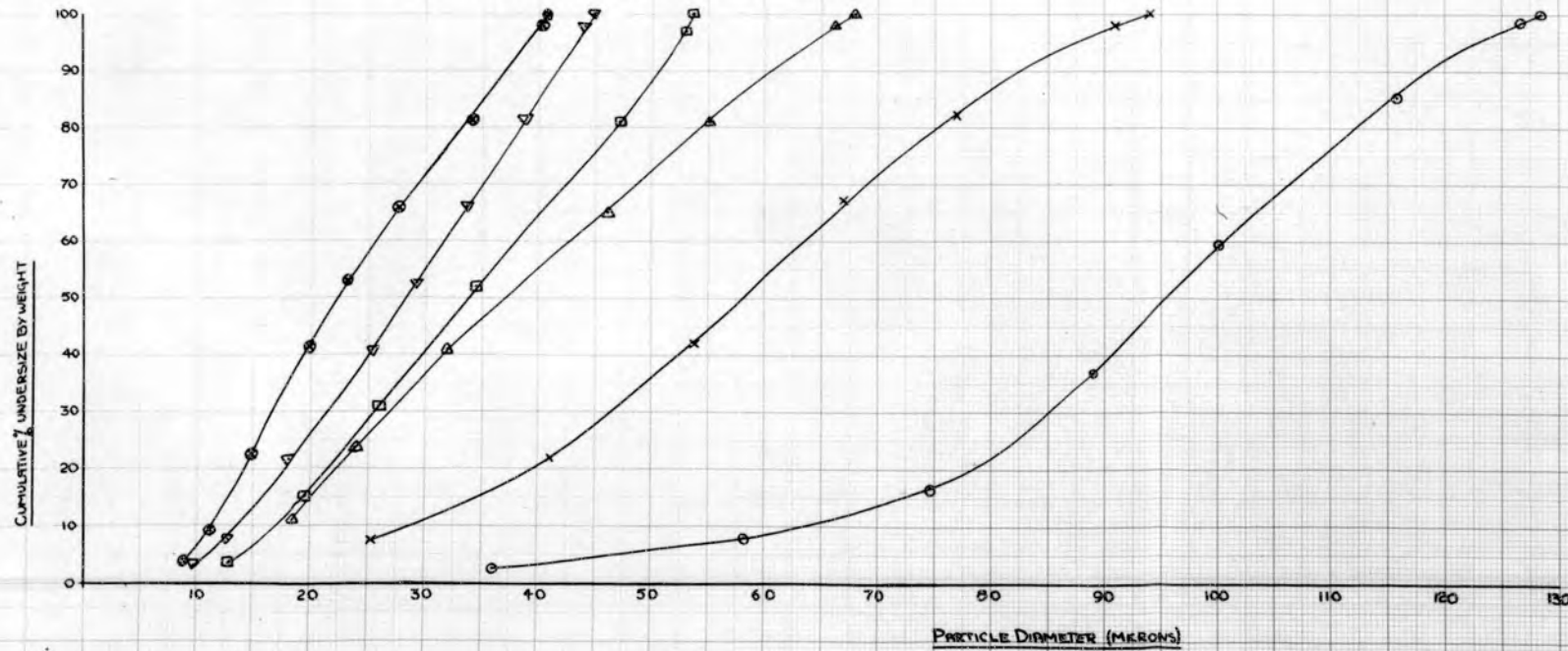


GRAPH No 8

(a) Size Distribution By Microscope Count Technique

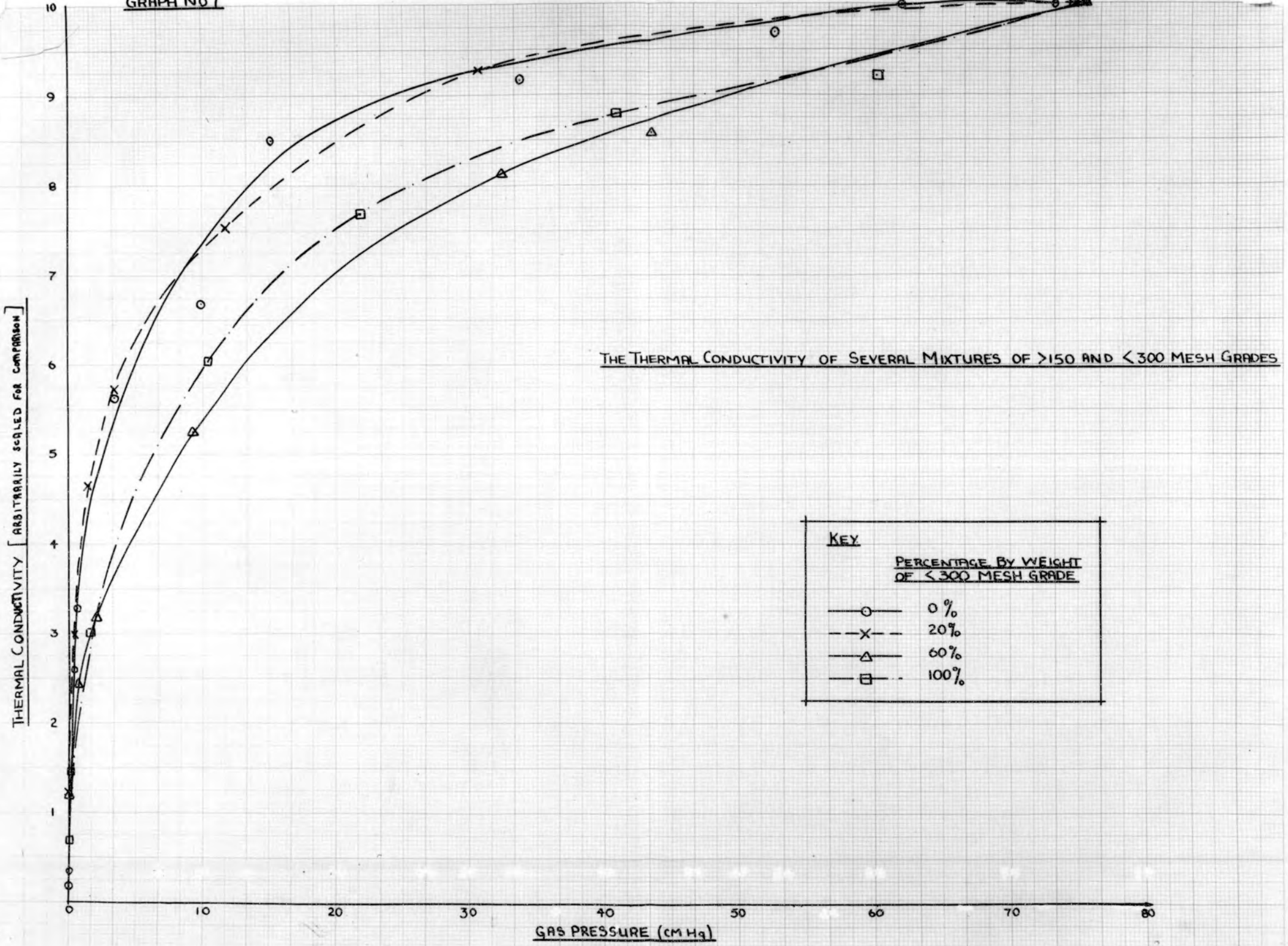


(b) Size Distribution By Photo Sedimentation



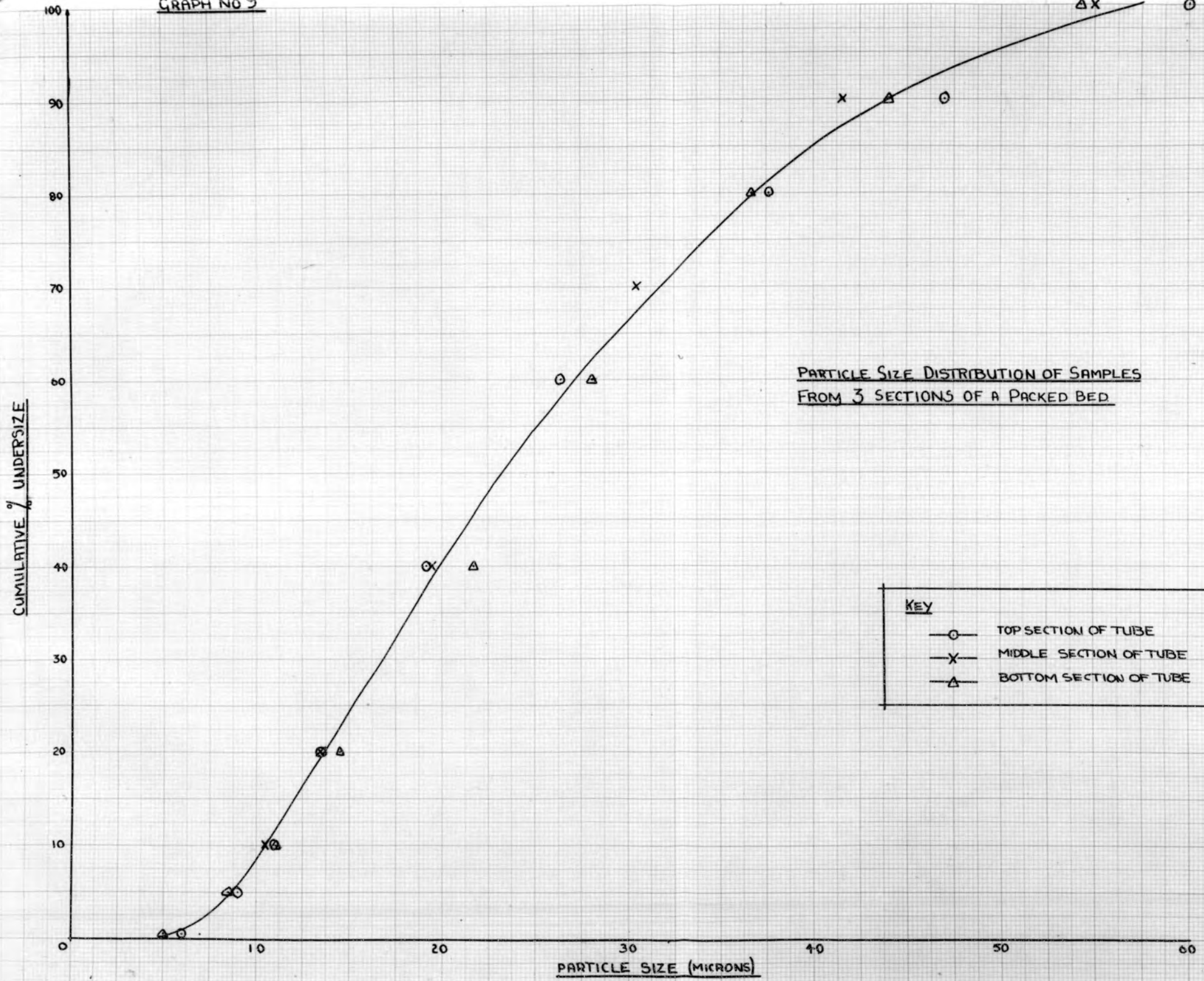
PARTICLE SIZE DISTRIBUTIONS OF SIX GRADES OF ALUMINIUM POWDER FROM >150 TO <300 MESH GRADES.

KEY:-	
○	> 150 MESH
×	150-170 MESH
△	170-200 MESH
□	200-240 MESH
▽	240-300 MESH
●	< 300 MESH



THE THERMAL CONDUCTIVITY OF SEVERAL MIXTURES OF >150 AND <300 MESH GRADES

KEY	
<u>PERCENTAGE BY WEIGHT OF &lt;300 MESH GRADE</u>	
—○—	0%
—X—	20%
—△—	60%
—□—	100%



KEY

- TOP SECTION OF TUBE
- × MIDDLE SECTION OF TUBE
- △ BOTTOM SECTION OF TUBE

APPENDIX 3.Radiative Heat Transfer.

When considering the **importance** of heat transfer by radiation through a powder, a preliminary estimate of the maximum contribution which could be made by this mechanism was made.

In the literature there are two different conceptions of radiative heat transfer through a powder. Some authors, such as Laubnitz, assume that the solid is opaque to radiation and that the heat is passed from grain to grain through the pore spaces. In this case, the effect of the powder is much like interposing a number of layers of conductor between the hot and cold boundaries and hence the radiative heat transfer must be small. Other authors, such as Fulk, consider that the powder is partly transparent to radiation which is scattered as it passes through the powder.

In either case, the total heat transfer by radiation cannot be greater than that which would be emitted by a black body radiator at the same temperature as the inner cylinder. Taking Stefan's constant to be  $5.669 \times 10^{-5}$  erg/cm<sup>2</sup>/sec/deg.<sup>4</sup>, and assuming an emissivity of unity, one centimetre length of the inner cylinder could radiate 0.445 joules/sec. Since the heat transfer through the 150-170 mesh aluminium was 1.10 joules/sec. per centimetre length of cylinder, an experimental investigation of the importance of radiative heat transfer was necessary.

TABLE 1

Results of Experiment 1Plotted on Graphs 1 and 2

Thermal Conductivity of Six Aluminium Powders with varying Interstitial Pressure.

Pressures in centimetres of Mercury.

Conductivities in joules  $\text{cm}^{-1} \text{C}/\text{cm}^2/\text{sec}$  and multiplied by  $10^3$

> 150 mesh		150-170 mesh		170-200 mesh.	
Inter-stitial Gas Pressure	Thermal Conductivity	Inter-stitial Gas Pressure	Thermal Conductivity	Inter-stitial Gas Pressure	Thermal Conductivity
0.00001	0.058	0.000005	0.392	0.00001	0.047
0.00037	0.057	0.00006	0.390	0.00005	0.042
0.00070	0.076	0.00038	0.395	0.021	0.155
0.0045	0.106	0.0049	0.393	0.28	0.444
0.026	0.229	0.017	0.376	2.3	0.959
0.168	0.603	0.170	0.516	5.4	1.712
0.40	0.905	0.51	0.629	12.5	2.246
0.67	1.137	1.9	1.101	18.0	2.535
3.4	1.947	4.1	1.577	23.2	2.919
9.9	2.320	8.6	2.020	37.0	3.256
15.2	2.950	20.5	2.790	60.1	3.464
33.8	3.182	41.5	3.401	66.8	3.543
52.7	3.362	53.5	3.609	74.4	3.502
62.0	3.469	64.6	3.711		
68.5	3.496	68.9	3.715		
73.3	3.469	74.5	3.751		

continued.....

TABLE 1 (contd. from previous page)

Results of Experiment 1Plotted on Graphs 1 and 2

200-240 mesh		240-300 mesh		← 300 mesh	
Inter- stitial Gas Pressure	Thermal Conduc- tivity	Inter- stitial Gas Pressure	Thermal Conduc- tivity	Inter- stitial Gas Pressure	Thermal Conduc- tivity
0.00001	0.355	0.00003	0.054	0.00001	0.139
0.00007	0.345	0.00009	0.078	0.0001	0.142
0.0012	0.363	0.0002 1	0.073	0.0009	0.161
0.0046	0.422	0.0015	0.084	0.011	0.142
0.028	0.488	0.019	0.154	0.093	0.248
0.39	0.537	0.028	0.191	0.14	0.295
1.4	0.775	0.055	0.260	1.6	0.619
2.9	1.145	0.14	0.328	10.4	1.253
8.9	1.695	0.44	0.488	21.7	1.594
18.8	2.020	1.2	0.574	40.8	1.819
28.2	2.237	2.0	0.705	60.2	1.919
37.8	2.471	8.3	1.291	70.9	2.114
66.2	2.787	12.1	1.511	74.6	2.066
77.0	2.859	23.4	1.692		
		37.0	1.924		
		51.5	2.100		
		68.7	2.118		
		74.5	2.267		



TABLE 2

Results of Experiment 2Plotted on Graph 3

Repeated Measurements of the Thermal  
Conductivity of Aluminium Powders.

Pressures in centimetres of Mercury

(P)  
Conductivities in joules  $\text{cm}^{\circ}\text{C}/\text{cm}^2/\text{sec.}$  and multiplied by  $10^3$   
(K)

> 150 Mesh Apparatus re-packed				150-170 Mesh Apparatus re-packed			
P	K	P	K	P	K	P	K
0.00001	0.058	0.00005	0.323	0.00005	0.392	0.0010	0.082
0.00037	0.057	0.0026	0.364	0.00006	0.390	0.020	0.112
0.00070	0.076	0.51	0.999	0.00038	0.395	0.31	0.444
0.0045	0.106	2.4	1.624	0.0049	0.393	3.4	1.204
0.026	0.229	12.2	2.810	0.017	0.376	11.0	2.231
0.168	0.603	39.1	3.430	0.170	0.516	36.0	3.402
0.40	0.905	75.1	3.556	0.51	0.629	74.8	3.719
0.67	1.137			1.9	1.101		
3.4	1.947			4.1	1.577		
9.9	2.320			8.6	2.020		
15.2	2.950			20.5	2.790		
33.8	3.182			41.5	3.401		
52.7	3.362			53.5	3.619		
62.0	3.469			64.6	3.711		
68.5	3.496			68.9	3.715		
73.3	3.469			74.5	3.751		

contd.

TABLE 2 (contd. from previous page)

Results of Experiment 2Plotted on Graph 3

170-200 Mesh					
Without re-packing apparatus				Apparatus re-packed	
P	K	P	K	P	K
0.00001	0.047	0.003	0.012	0.00001	0.227
0.00005	0.042	0.028	0.047	0.00006	0.229
0.021	0.155	0.08	0.310	0.00030	0.295
0.28	0.444	0.67	0.586	0.0030	0.261
2.3	0.959	1.2	0.923	0.023	0.360
5.4	1.712	3.0	1.483	0.033	0.338
12.5	2.246	11.1	2.292	0.70	0.692
18.0	2.535	21.5	3.006	1.2	0.871
23.2	2.919	31.5	3.259	2.1	1.049
37.0	3.256	52.7	3.461	3.9	1.735
60.1	3.464	74.9	3.482	13.1	2.286
66.8	3.543			30.5	3.145
74.4	3.502			50.2	3.365
				53.2	3.611
				69.1	3.691
				75.0	3.601

TABLE 3

Results of Experiment 3.

Plotted on Graph 4

Thermal Conductivity of the 150-170 mesh Aluminium Powder with an additional coaxial tube.

Pressures in centimetres of Mercury

Conductivities in joules  $\text{cm}^{\circ}\text{C}/\text{cm}^2/\text{sec}$  and multiplied by  $10^3$ 

Interstitial Gas Pressure	Thermal Conductivity	Temperature of Inner Cylinder ( $\theta_1$ )	Temperature of Intermediate Cylinder ( $\theta_2$ )	Temperature of Outer Cylinder ( $\theta_3$ )	$\frac{\theta_1 - \theta_2}{\theta_2 - \theta_3}$
0.00001	0.127	100.6	46.5	29.1	3.109
0.00008	0.103	98.8	46.5	28.2	2.858
0.0034	0.184	101.5	45.6	29.1	3.388
0.032	0.300	102.4	43.7	26.3	3.374
0.080	0.329	99.8	41.9	24.4	3.309
1.0	0.672	100.6	41.9	25.4	3.558
2.9	1.272	100.6	43.7	27.2	3.448
6.6	1.784	99.8	42.8	25.4	3.276
15.9	2.478	101.5	45.6	28.2	3.213
47.6	3.292	101.5	46.5	29.1	3.161
74.7	3.592	102.4	48.3	31.8	3.279

Average measured value of  $\frac{\theta_1 - \theta_2}{\theta_2 - \theta_3} = 3.270 \pm .057$ Calculated value of  $\frac{\theta_1 - \theta_2}{\theta_2 - \theta_3} = \log_e \frac{d_1}{d_2} = 3.249 \pm .004$ using Fourier's equation  $\log_e \frac{d_3}{d_4}$ 

where  $d_1$  = outside diameter of inner cylinder  
 $d_2$  = inside diameter of intermediate cylinder  
 $d_3$  = outside diameter of intermediate cylinder  
 $d_4$  = inside diameter of outside cylinder

TABLE 4

Results of Experiment 4Plotted on Graph 5.

Thermal Conductivity of the 150-170 mesh Aluminium Powder with various boundary temperatures.

Pressures in centimetres of Mercury

Conductivities in joules  $\text{cm}^{\circ}\text{C}/\text{cm}^2/\text{sec}$  and multiplied by  $10^3$

Temp. of Inner Cylinder	100°C		150°C		200°C	
	Inter-stitial Gas Pressure	Thermal Conductivity	Inter-stitial Gas Pressure	Thermal Conductivity	Inter-stitial Gas Pressure	Thermal Conductivity
	0.00010	0.082	0.00004	0.151	0.00003	0.151
	0.020	0.112	0.013	0.264	0.029	0.247
	0.31	0.444	0.180	0.482	0.25	0.636
	3.4	1.204	0.45	0.619	2.8	1.809
	11.0	2.231	3.3	1.410	10.7	3.160
	36.0	3.402	12.2	2.685	38.2	4.400
	74.8	3.719	44.3	3.706	75.5	4.844
			75.5	3.990		

## TABLE 5A

Results of Experiment 6Plotted on Graph 7

Thermal Conductivity of mixtures of the  
>150 and <300 grades of Aluminium Powder.

Pressures in centimetres of Mercury.

Conductivities in joules  $\text{cm}^{\circ}\text{C}/\text{cm}^2/\text{sec}$ . and multiplied by  $10^3$

The arbitrary conductivities are the actual conductivities scaled for comparison purposes such that the conductivity of each fraction at atmospheric pressure is 1,000.

Percentage by weight of <300 mesh grade					
0%			20%		
Interstitial Gas Pressure.	Thermal Conductivity.	Arbitrary Conductivity.	Interstitial Gas Pressure.	Thermal Conductivity.	Arbitrary Conductivity.
0.00001	0.058	0.17	0.0051	0.576	1.23
0.00037	0.057	0.17	0.56	1.392	2.98
0.00070	0.076	0.23	1.4	2.165	4.65
0.0045	0.106	0.32	3.5	2.673	5.72
0.026	0.229	0.66	11.7	3.509	7.52
0.168	0.603	1.73	30.6	4.337	9.28
0.40	0.905	2.59	75.4	5.014	10.00
0.67	1.137	3.29			
3.4	1.947	5.63			
9.9	2.320	6.68			
15.2	2.950	8.50			
33.8	3.182	9.16			
52.7	3.362	9.68			
62.0	3.469	10.00			
68.5	3.496	10.10			
73.3	3.469	10.00			
				contd.....	

TABLE 5A (contd. from previous page)

Results of Experiment 6Plotted on Graph 7

Percentage by weight of 300 mesh grade					
60%			100%		
Interstitial Gas Pressure.	Thermal Conductivity.	Arbitrary Conductivity.	Interstitial Gas Pressure.	Thermal Conductivity.	Arbitrary Conductivity.
0.00001	0.404	1.20	0.00001	0.139	0.68
0.0016	0.451	1.31	0.0001	0.142	0.68
0.60	0.815	2.43	0.0009	0.161	0.77
2.1	1.065	3.18	0.011	0.142	0.68
9.2	1.754	5.25	0.093	0.248	1.21
32.3	2.709	8.13	0.14	0.295	1.45
43.5	2.856	8.58	1.6	0.619	3.00
75.6	3.333	10.00	10.4	1.253	6.04
			21.7	1.594	7.68
			40.8	1.819	8.79
			60.2	1.919	9.22
			70.9	2.114	10.05
			74.6	2.066	10.00

TABLE 5BResults of Experiment 6Plotted on Graph 7

Percentage by weight of <math>\lt; 300</math> mesh Aluminium.	Thermal Conductivity at Atmospheric Pressure.	Thermal Conductivity at 0.00001 cm. Hg.	Porosity of mixture
0	3.469	0.058	0.480
5	3.644	0.414	0.466
10	3.713	0.090	0.452
15	3.975	0.323	0.422
20	4.667	0.289	0.414
30	4.002	0.454	0.385
40	3.597	0.111	0.361
50	3.345	0.175	0.368
60	3.329	0.404	0.379
80	2.809	0.426	0.403
100	2.066	0.139	0.451

TABLE 6.

Described in Appendix 1.

Calculation of Thermal Conductivities.

Powder 150-170 mesh Aluminium

Wt. of Powder = 807.4 gms.

1	2	3	4	5	6	7	8	9	10	11	12	13	14	15		
V	Vx1.038	I	VxI	W	$\frac{V}{I}$	$\frac{W}{L}$	Ti	Te	Ti-Te	P	p	$\frac{W}{L(Ti-Te)}$	K	T.H	B.H	M.H
1.065		0.218	.2322	4.4722	4.89	.12468	104.2	29.1	75.1	0.000005	0.00073	1.6602	0.392	20	15	32
1.055		0.216	.2279	4.3894	4.88	.12238	102.4	28.2	74.2	0.00006	0.00072	1.6493	0.390	20	15	32
1.060		0.218	.2311	4.4510	4.86	.12409	100.6	26.3	74.3	0.00038	0.00133	1.6701	0.395	20	15	32
1.060		0.217	.2300	4.4298	4.88	.12350	100.6	26.3	74.3	0.0049	0.0063	1.6622	0.393	20	15	32
1.055		0.216	.2279	4.3894	4.88	.12238	102.4	25.4	77.0	0.017	0.035	1.5894	0.376	15	38	32
1.260		0.257	.3238	6.2364	4.90	.17387	103.3	23.7	79.6	0.170	0.15	2.1843	0.516	15	55	38
1.375		0.278	.3823	7.3631	4.95	.20528	101.5	24.4	77.1	0.51		2.6625	0.629	40	60	40
1.785		0.370	.6605	12.7212	4.82	.35467	101.5	25.4	76.1	1.9		4.6606	1.101	28	72	54
2.150		0.440	.9460	18.2200	4.89	.50797	102.4	26.3	76.1	4.1		6.6750	1.577	32	80	64
2.380		0.497	1.1828	22.7807	4.79	.63513	101.5	27.2	74.3	8.6		8.5482	2.020	55	70	72
2.69	2.792	0.564	1.5747	30.3287	4.95	.84556	99.8	28.2	71.6	20.5		11.8095	2.790	70	68	83
2.96	3.072	0.624	1.9169	36.9195	4.92	1.02932	100.6	29.1	71.5	41.5		14.3961	3.401	70	68	92
3.10	3.218	0.648	2.0853	40.1629	4.97	1.11974	103.3	30.0	73.3	53.5		15.2761	3.609	70	68	98
3.10	3.218	0.650	2.0917	40.2861	4.95	1.12318	101.5	30.0	71.5	64.6		15.7088	3.711	72	70	98
3.10	3.218	0.667	2.1464	41.3397	4.82	1.15255	103.3	30.0	73.3	68.9		15.7237	3.715	78	68	98
3.04	3.156	0.653	2.0609	39.6929	4.83	1.10664	100.6	30.9	69.7	74.5		15.8772	3.751	75	65	98

Average  $\frac{V}{I} = 16 \frac{78.28}{4.890}$        $\therefore$  Power in 1 cm of wire = Total power x  $\frac{.13633}{4.890} = \frac{\text{Total power}}{x 0102788}$ .

Key :-

- V = Voltage
- I = Current (amps)
- W = Power (watts)
- $\frac{W}{L}$  = power per unit centimetre of heater wire
- Ti = temperature of inner cylinder
- Te = temperature of outer cylinder

- P = interstitial gas pressure (McLeod Gauges and Manometer)
- p = Pirani Gauge reading
- K = Thermal Conductivity of powder.
- TH = Setting of variac transformer supplying top heater
- BH = Setting of variac transformer supplying bottom heater
- MH = Setting of variac transformer supplying main heater



TABLE 7A

Results of Experiment 9Porosities of the Six Grades of Aluminium Powder

Grade	Cylinder diameter = 3.57 cms.			Cylinder diameter = 2.5 cms.		
	Wt/Vol (gms/cc.)	Mean Wt/Vol (gms/cc.)	Porosity.	Wt/Vol (gms/cc.)	Mean Wt/Vol (gms/cc.)	Porosity.
> 150	1.302 1.307 1.305	1.305	0.511	1.356 1.346 1.363	1.355	0.492
150-170	1.479 1.478 1.482	1.480	0.445	1.526 1.524 1.525	1.525	0.428
170-200	1.456 1.444 1.472	1.457	0.454	1.498 1.491 1.488	1.492	0.440
200-240	1.444 1.454 1.453	1.450	0.456	1.455 1.460 1.471	1.462	0.452
240-300	1.435 1.451 1.436	1.441	0.460	1.512 1.496 1.476	1.495	0.439
< 300	1.371 1.376 1.367	1.371	0.486	1.434 1.443 1.462	1.446	0.458

contd.....

TABLE 7A (contd. from previous page)

Results of Experiment 9Porosities of the Six Grades of Aluminium Powder

Grade	Cylinder diameter = 1.92 cms.		
	Wt./Vol. (gms./cc.)	Mean Wt./Vol. (gms./cc.)	Porosity.
> 150	1.350 1.350 1.349	1.350	0.494
150-170	1.527 1.536 1.522	1.528	0.427
170-200	1.473 1.467 1.450	1.463	0.451
200-240	1.471 1.494 1.486	1.483	0.444
240-300	1.538 1.541 1.550	1.543	0.421
< 300	1.476 1.485 1.471	1.477	0.446

TABLE 7BResults of Experiment 9.Porosity of the 170-200 Grade of Aluminium Powder.

Cylinder diameter = 3.57 cms.

Volume occupied by Powder (ccs)	Weight of Powder (gms)	Weight/Volume. (gms/cc)
79	116.39	1.473
121	180.10	1.488
156	228.14	1.462
188	277.37	1.475
204	297.58	1.459
220	324.02	1.473
235	348.85	1.484
243	351.30	1.446

Mean value of weight/volume = 1.470 -  
 Standard deviation on a single value =  $\pm .013$   
 $\therefore$  Standard deviation on a single value of  
 porosity =  $\pm .005$ .

TABLE 8.Results of Experiment 9.

Porosity of the Six Grades of Aluminium Powder  
determined in the Concentric Cylinder Apparatus.

Grade	Mean <sup>Wt/Vol</sup> (gms./cc.)	Porosity	Weight of Powder in Thermal Con- ductivity Apparatus during Expt. 1
>150	1.386	0.480	659.5
150-170	1.612	0.395	807.4
170-200	1.515	0.432	755.6
200-240	1.468	0.449	708.7
240-300	1.503	0.436	718.1
<300	1.460	0.451	688.5



101  
TABLE 9A .

Results of Experiment 8

Plotted on Graph 8

Particle Size Analysis of the Six Aluminium Powders by  
Microscope Count and Photosedimentation Techniques.

Particle Size Analysis by Microscope Count Technique.

Grade > 150		150-170		170-200	
Cumulative % under-size by weight.	Particle Diameter (microns)	Cumulative % under-size by weight	Particle Diameter (microns)	Cumulative % Under-size by weight.	Particle Diameter (microns)
0.5	84.6	0.5	22.6	0.6	22.6
2.2	103.4	2.3	37.2	2.9	45.2
6.7	131.6	5.8	55.2	5.4	52.2
11.3	150.4	12.4	77.8	12.0	73.1
18.1	169.2	21.3	95.8	16.6	83.2
31.0	188.0	39.9	113.4	30.9	95.4
43.7	206.8	50.1	125.5	37.2	100.4
59.9	235.0	67.0	143.1	48.5	113.4
75.7	263.2	73.6	161.2	65.7	125.5
87.2	282.0	80.9	165.7	76.8	148.1
93.2	319.6	88.8	171.3	87.3	155.5
100.0	376.0	100.0	175.7	100.0	158.1

contd....

TABLE 9A (contd. from previous page)

Results of Experiment 8Plotted on Graph 8

Particle Size Analysis by Microscope Count Technique.

Grade 200-240		240-300		< 300	
Cumulative % under-size by weight	Particle Diameter (microns)	Cumulative % under-size by weight	Particle Diameter (microns)	Cumulative % under-size by weight	Particle Diameter (microns)
0.7	10.5	0.6	7.3	0.7	6.3
5.0	19.9	1.7	10.5	3.0	9.4
5.4	23.0	3.6	13.6	5.2	11.5
		8.7	17.8	8.2	13.7
10.5	31.5	13.6	21.0	16.4	16.8
16.0	40.9	23.9	26.3	31.4	23.1
32.5	56.7	40.6	32.5	44.7	26.3
46.7	66.2	51.9	36.8	56.7	28.4
60.6	70.4	66.7	39.9	69.3	33.6
79.3	78.8	78.3	46.1	78.0	37.8
89.5	79.8	87.4	47.2	84.3	40.9
100.0	80.9	100.0	55.5	100.0	44.0

TABLE 9BResults of Experiment 8Plotted on Graph 8

Particle Size Analysis of the Six Aluminium Powders by  
Microscope Count and Photosedimentation Techniques.

Particle Size Analysis by Photosedimentation

Grade > 150		150-170		170-200	
Cumulative % under- size by weight.	Particle Diameter (microns)	Cumula- tive % under- size by weight.	Particle Diameter (microns)	Cumula- tive % under- size by weight.	Particle Diameter (microns)
2.8	36.0	7.6	25.3	11.0	18.5
8.6	58.3	21.9	41.2	23.8	23.8
16.7	74.7	42.0	53.7	41.0	31.9
36.9	89.0	67.1	67.0	65.0	46.1
59.7	100.2	82.2	77.0	81.0	55.2
85.3	115.7	98.0	91.0	98.0	66.2
98.5	126.7	100.0	94.0	100.0	68.0
100.0	128.4				

contd.....

TABLE 9B (contd. from previous page)

Results of Experiment 8Plotted on Graph 8Particle Size Analysis by Photosedimentation

Grade 200-240		240-300		< 300	
Cumulative % under-size by weight.	Particle Diameter (microns)	Cumulative % under-size by weight.	Particle Diameter (microns)	Cumulative % under-size by weight.	Particle Diameter (microns)
3.5	12.5	3.0	9.8	4.0	8.9
15.0	19.5	7.7	12.9	9.3	11.3
31.0	25.9	21.7	18.1	22.8	15.0
52.0	34.4	40.9	25.3	41.4	19.9
81.0	47.2	52.6	29.3	52.9	23.2
97.0	53.0	66.1	33.6	65.9	27.6
100.0	53.6	81.5	38.8	81.2	34.0
		97.9	44.0	97.9	40.3
		100.0	44.8	100.0	40.7



TABLE 10

Split Tube Experiment showing the vertical homogeneity of a packed mixture of the <300 mesh and >150 mesh grades of Aluminium Powder.

Mixture consists of approximately 30% by weight of the <300 mesh grade.

Section of Tube.	Weight of Powder retained on 200 mesh sieve.	Total weight of powder in section.	Fractional percentage of coarser powder.
Top	14.777	21.489	68.77
Middle	13.819	20.263	68.20
Bottom	11.239	16.410	68.49

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