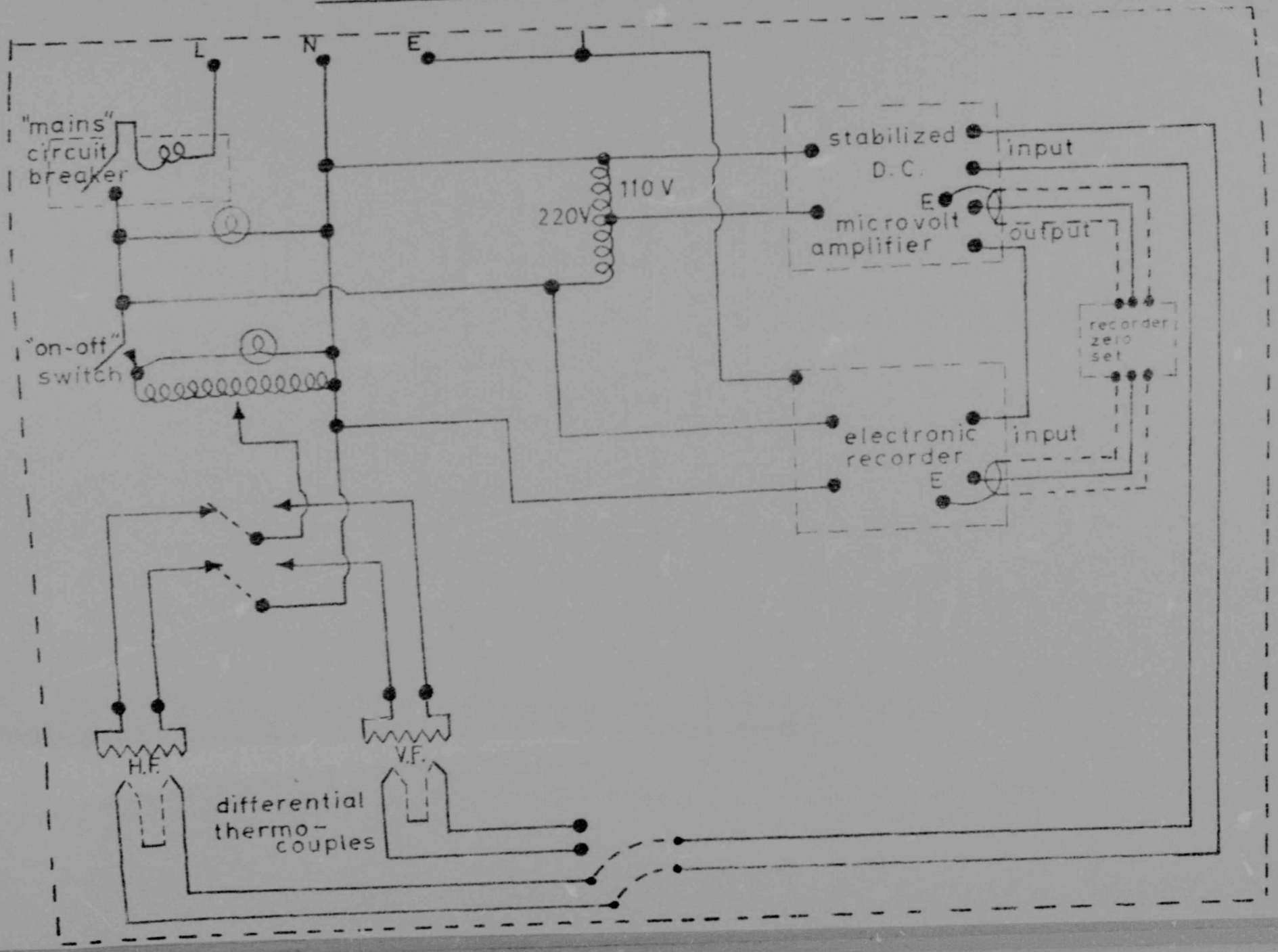
FIG. 11, 18

WIRING DIAGRAM OF D.T.A. APPARATUS



(f) Conclusion :

Details of the D.T.A. apparatus that has been built in this Laboratory have been given, together with the results of a few preliminary tests that were conducted, mainly on the horizontal furnace. The design of the rack and assembly details of the various components of the equipment have not been given as it is not felt that these are necessary for an understanding of the work to be described. Only a schematic wiring diagram of the apparatus will therefore be given at this stage (See Fig: II, 18),. The work presented has shown that the D. T. A. equipment would at least be a useful research tool, and that routine assessment of the quartz content of mine dust samples would require very careful investigation if accurate results were to be obtained by either D. T. A. or X-ray methods.

"DETERMINATION OF THE PERCENTAGE OF QUARTZ (S_iO_2) IN SAMPLES OF MINE DUST BY THE METHOD OF DIFFERENTIAL THERMAL ANALYSIS"

CHAPTER III

DETAILED EXPERIMENTAL WORK WITH THE D.T.A. APPARATUS

1. Introduction

The work described in Section 5e of the previous Chapter has given some indication of the magnitude of the problems involved in the quantitative determination of quartz by the D.T.A. method. More problems unfolded themselves as the work progressed and this led to many reassessments of the thermograms already obtained, and also to the planning of many tests to investigate the various difficulties that arose. No strict chronological order has been adhered to in the presentation, since where it was felt that it would be advantageous to present a certain group of results out of order this has been done.

Details of the use of the apparatus and of the assessment of the differential thermal curves are given before any further results because it is felt that this facilitates an understanding of them. It should be mentioned that the assessments were only evolved to their present state after many of the tests and a certain amount of the statistical analysis had been conducted.

2. Use of the D.T.A. Apparatus:

(a) Preparation of Dust Samples:

The preparation of dust samples is important since it may have a profound effect on the D.T.A. thermograms produced. We are concerned here with both the inert and the test samples, and will discuss them separately.

(i) Inert Sample

For quantitive differential thermal analysis, choice of the inert dust is very important. Because the peaks depend on the thermal conductivity of the

/dust .....

dust in the sample holders: it is desirable to use as the inert sample material which has thermal properties as similar to the test sample as possible. In practice it has been found that calcined alumina (Al_2O_3) is a suitable material to use when the test sample contains quartz.

The particle size distribution of the inert dust also affects the magnitude of the D.T.A. response. For quantitive work it is, therefore, necessary to use a standard inert sample. In the work described in this thesis, it was decided to use aluminium oxide which had been passed through a 270 mesh sieve and calcined at about 950°C. Calcined alumina is slightly hygroscopic and must ce kept either in a sealed bottle or in a dessicator.

(ii) Test Sample:

The preparation of the test sample will depend on the nature of the dust, and also on the quantity available. All samples should be passed through a 325 mesh sieve in order to eliminate coarse particles. If necessary the dust should first be ground down in an agate mortar. The sample is then calcined at 950°C for 20 minutes in order to eliminate any irreversible reactions that might interfere with the quartz inversion.

If there is insufficient dust to fill the sample holder it is mixed in known proportions with -270 mesh calcined alumina. To ensure efficient mixing the test sample and the diluent are lightly ground in an agate mortar.

(b) Packing of the Sample Holders:

The test and inert dusts are packed into their respective sample holders in the same way. It is not always necessary to repack the inert dust when changing the test sample in the horizontal furnace, but in the vertical furnace it is necessary since the nickel block cannot be

/separated .....

separated from the thermocouples without disturbing the calcined alumina.

About 1 gram of the test sample is placed in a watch glass and weighed. A plunger device has been designed to ensure that the dust is packed into the sample holders at a constant pressure. Dust is packed into the barrel of this device with a spatula. The device is held over the sample holder to be filled and the plunger depressed, forcing the dust into the sample holder. When the piston is flush with the bottom of the barrel the pressure on the surface of the dust is about 3.75 pounds. Any excess dust should be returned to the watch glass and weighed again. The difference between the initial and final weights of the watch glass will give the weight of dust that has been packed into the sample holder.

The above packing device became available only after cycles H 145/10 and V 115/10 had been completed. Up to this point the dust was tamped down firmly with a glass rod. A disc of alundum cement 50 mm. in diameter and about 2 mm. thick should be placed on top of the nickel block after the sample wells have been packed. This is found to stabilize the $\alpha \rightleftharpoons \beta$ quartz inversion peaks obtained from this furnace.

(c) Insertion of Sample Holders into Furnaces:

(i) Horizontal Furnace:

Whenever the horizontal furnace sample holder is not in position in the furnace, it should be resting on the wooden stand that has been made for the purpose (See Section 3a (xi) of Chapter II p.42). When the crucibles have been charged, the holder is inserted into the furnace from the front. The asbestos cement end-mounting fits snugly into the furnace tube. The knurled nuts are screwed onto two threaded bolts which slide into holes in the end-mounting. These nuts serve both to keep the sample holder firmly in position

/and .....

and to orientate the platinum baseplate horizontally. The cylindrical metal cap is screwed in position so that the thermocouple leads emerge through a slit in its side. The leads are placed in the appropriate arms of the U-tubes in the top of the thermos flask, the thin wires in the left-hand pair of tubes and the other two in the right-hand pair. The door is closed on the flask and the horizontal furnace is ready to operate.

(ii) Vertical Furnace:

This sample holder will stand upright on any flat surface, its thermocouple leads issuing from a slot that has been cut at one end of the supporting tube. The door concealing the thermos flask is opened, and the thermos moved to one side. The plate under the furnace tube is pushed back, the sample holder inserted into the furnace and the plate slid underneath it to hold it in position. The position of the supporting tube is adjusted so that the nickel cylinder which holds the block does not touch the walls of the furnace tube. After dipping the thermocouple leads into the mercury in their respective U-tubes, the vertical furnace is ready for use.

(d) Operation of the D.T.A. Apparatus:

As there is no programme controller in the apparatus, it has been necessary to determine by trail and error the power input that gives a convenient rate of temperature change in each furnace in the vicinity of the $\alpha \rightleftharpoons \beta$ quartz inversion. In order not to waste the recorder chart paper it has also been necessary to determine when the recorder should be switched on and off.

Separate instructions for the operation of the two furnaces are given below:-

(i) Horizontal Furnace:

1. Place the sample holder in position as described in

/Section .....

Section 2. c (i).

2. Switch on the mains, and then the power switches to both the amplifier and the recorder.
3. Depress the recorder "Test" switch for five seconds in order to standardise the recorder.
4. Set the selector switch on the amplifier to "Linear" and the "Range" switch to 200.
5. Check that the recorder gears are adjusted to give a chart speed of 1 inch per minute.
6. Switch the furnace selector to "Horizontal".
7. Switch on the power and set it to 500 watts on the wattmeter by means of the "Furnace Power Control" variac.
8. Note the time, say t_o.
9. Periodically check the power input to the furnace and readjust to 500 W. if necessary.
10. If the approximate percentage of quartz in the test sample is known, the amplifier range switch may be adjusted to either range 100 or 50 as necessary. If it is not known, the amplifier should be left on range 200 (i.e. 200 microvolts fullscale deflection on meter or recorder).
11. At time t_o + 20 minutes, take the reading on the amplifier meter, the result being positive or negative according to whether the needle is to the right or left of the centre zero. If the reading is positive, the inert dust is at a slightly higher temperature than the test dust or vice versa.
12. Use the "Set Recorder Zero" knob to adjust the position of the recorder pen, such that with the amplifier selector switch in the "Recorder" position a deflection of about 30 divisions is obtained.
13. Note this recorder zero position with the amplifier selector switch on "Linear". This is usually about 10, 20 or 30 divisions.

/14 .....

14. At a time $t_o + 23 = t_1$, minutes, switch amplifier to "Recorder" and switch on the recorder motors. Note the time and the furnace temperature. If it is desired to know the heating rate, the furnace temperature should be read every minute.

15. The $\alpha \rightarrow \beta$ quartz inversion should occur at about time $t_1 + 7$ minutes, a deflection to the right being obtained. The recorder motors are switched off at a time $t_1 + 12$ or $t_1 + 13 = t_2$ minutes. The amplifier selector switch is changed to "Linear" and the furnace temperature recorded.

16. Set the recorder zero to about 70, 80 or 90 divisions on the recorder chart.

17. Switch off the power supply to the horizontal furnace at a time $t_o + 50 = t_3$ minutes, and record both the time and the furnace temperature. This is usually between 770 and 780°C.

18. Repeat 11, 12 and 13 at a time $t_3 + 10$ minutes and switch on the recorder motors at a time $t_3 + 12 = t_4$ minutes. Note the furnace temperature.

19. The $\beta \rightarrow \alpha$ quartz reaction should occur at about time $t_4 + 6$ minutes, the deflection this time being to the left. Switch off the recorder motors at a time $t_4 + 12$ minutes and record the furnace temperature.

20. The chart recording thus obtained may now be cut off. The metal strip which is used to keep the chart taut over its rollers should be removed, and reattached to the new end of the paper in the recorder by means of adhesive tape. A slot has been cut in the base of the recorder door in order to allow the door to be closed while the chart is unrolling.

(ii) Vertical Furnace:

1. Place the sample holder in position as described in Section 2. c (ii).

2 to 6. As above, substituting "Vertical" for "Horizontal"

/where .....

where necessary.

7. Switch on the power, and set it to 750 watts.

8 to 13. As above, substituting $t_o + 40$ for $t_o + 20$ in 11.

14. At a time $t_o + 45 = t_1$ minutes, switch to "Recorder" and switch on the recorder motors. Note the time and the furnace temperature.

15. The $\beta \rightarrow \alpha$ quartz inversion should occur at about time $t_1 + 6$ minutes. The recorder motors are switched off at a time $t_1 + 12 = t_2$ minutes, the amplifier selector switched to "Linear", and the furnace temperature recorded.

16. As above.

17. Switch off the power supply to the vertical furnace at a time $t_o + 60 = t_3$ minutes. Record the time and the furnace temperature.

18. As above, substituting $t_3 + 20$ for $t_3 + 10$ minutes and $t_3 + 23$ for $t_3 + 12$ minutes.

19. The $\alpha \rightarrow \beta$ quartz reactions should occur at about $t_4 + 5$ minutes. The recorder motors are switched off at time $t_4 + 12$ minutes and the temperature recorded.

20. As above.

3. Assessment of the Differential Thermal Curves

An examination of the literature on D.T.A. revealed that only a few of the workers in this field give details of their methods of measuring the D.T.A. response. Webb (52) has made a comprehensive survey of these methods and the point that stands out is that they are all somewhat arbitrary, except in the very rare event of the base line before and after a peak being perfectly horizontal and on the same level. In the case of quartz dust this never occurs on account of the change of specific heat and thermal conductivity of the sample as the quartz changes from the α- to the β- crystalline structure.

In the present work various methods were tried for measuring both the peak height and the peak area of the response. These will now be described.

/(a) .....

(a) Method A

This method of measurement is illustrated in Fig III,1 for both the $\alpha \rightarrow \beta$ and $\beta \rightarrow \alpha$ inversions. A line perpendicular to the direction of chart travel is drawn through the apex of the D.T.A. peak. The speed of the chart travel was 1 inch per minute in all these tests. A distance of four inches on either side of this centre line is measured, and lines parallel to the centre line are drawn. From the points of intersection of these lines with the curve two lines parallel to the direction of chart travel are drawn.

The height of the D.T.A. response is given in microvolts by

a = peak height

b = base displacement height

a + b = total height

The area of the D.T.A. response is given in microvolt minutes by

1 + 2 = peak area

3 + 4 = base displacement area

1 + 2 + 3 + 4 = total area

When it was found that either of the two base lines would have intersected the curve twice on one side of the centre line if drawn by the above method, then it was taken simply as the tangent to the curve.

(b) Method B.

This half-peak method of assessing the response is illustrated in Fig III, 2. Again the arbitrary distance of four inches is measured on either side of the centre line, but this time the base lies from the α- and β- sides of the peak are only drawn to the centre line. Peak height responses are exactly the same as before, and the area of the D.T.A. response is given by

α = α- half peak area

β = β- half peak area

$\alpha + \beta$ = total of half peak areas

Fig. III, 1 ASSESSMENT OF D.T.A. RESPONSE - Method A

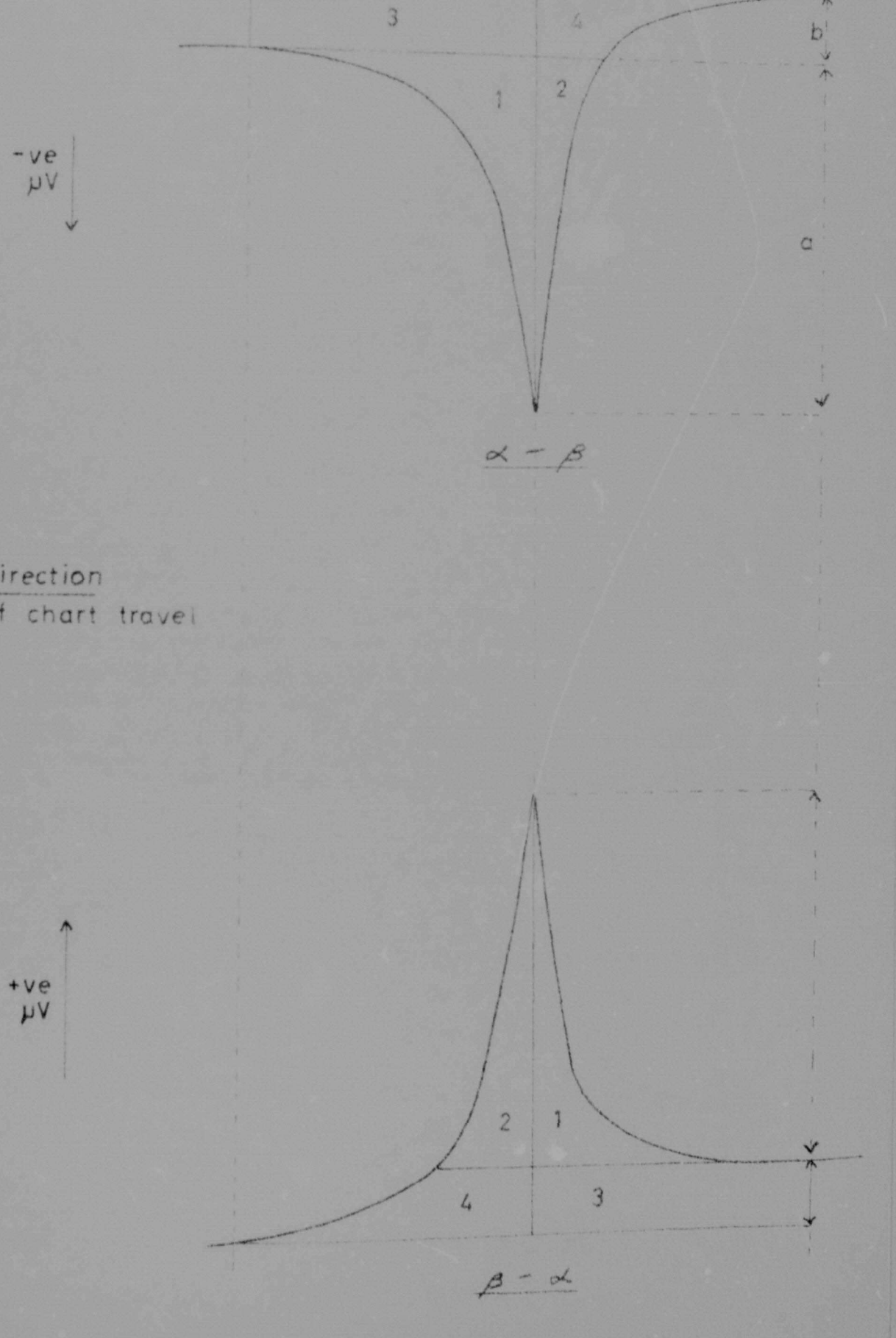
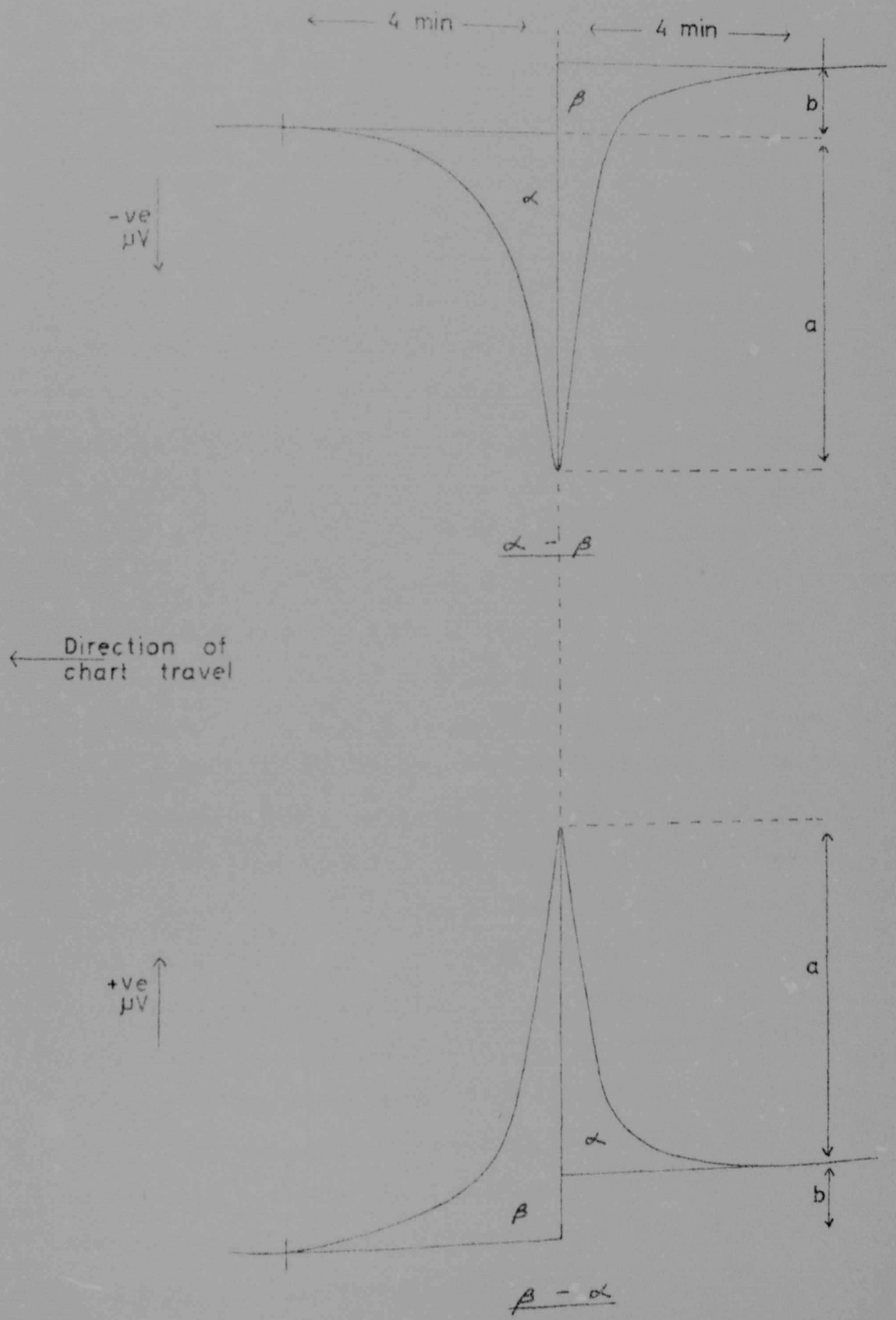


Fig. III, 2 ASSESSMENT OF D.T.A. RESPONSE -

Method B, Half-Peaks



It should be noted that this method of assessment was suggested to the author by Prof. A.L. Hales. It later came to his attention that a very similar method had been developed by Sewell (private communication via Dr. T.L. Webb) in his theoretical work on D.T.A.

(c) Method finally adopted

While working with various prepared samples of quartz, the above methods, and especially the second one, were found to give quite good results. The reason for this was that for these samples, the base line of the curve was seldom found to have a large slope. This was not found to apply to some unknown dust samples that were tested, and it became necessary to develop an alternative method of assessing the response. Fig III 3 illustrates the method. The base lines have deliberately been drawn with large slopes in order to show how methods A and B could give erroneous results.

The diagrams are self-explanatory. Once again the centre line from the apex of the peak has been drawn. As the peak is usually very sharp, this point is easy to determine. It has been found that the base line preceding the reaction is more stable than that afterwards; for this reason the α - base line is extended to meet the centre line for the $\alpha \rightarrow \beta$ inversion, and the β- base line for the $\beta \rightarrow \alpha$ inversion. The areas measured are then the α- base line half peak for the $\alpha \rightarrow \beta$ and the β- base line half peak for the $\beta \rightarrow \alpha$ change. The peak height taken is the distance along the centre line from the apex of the peak to its intersection with the base line. This parameter was used by Grimshaw and Roberts (20) on the $\beta \rightarrow \alpha$ inversion as a measure of their D.T.A. measurements on quartz, and they report that it gave them a linear relationship between peak height and percentage quartz.

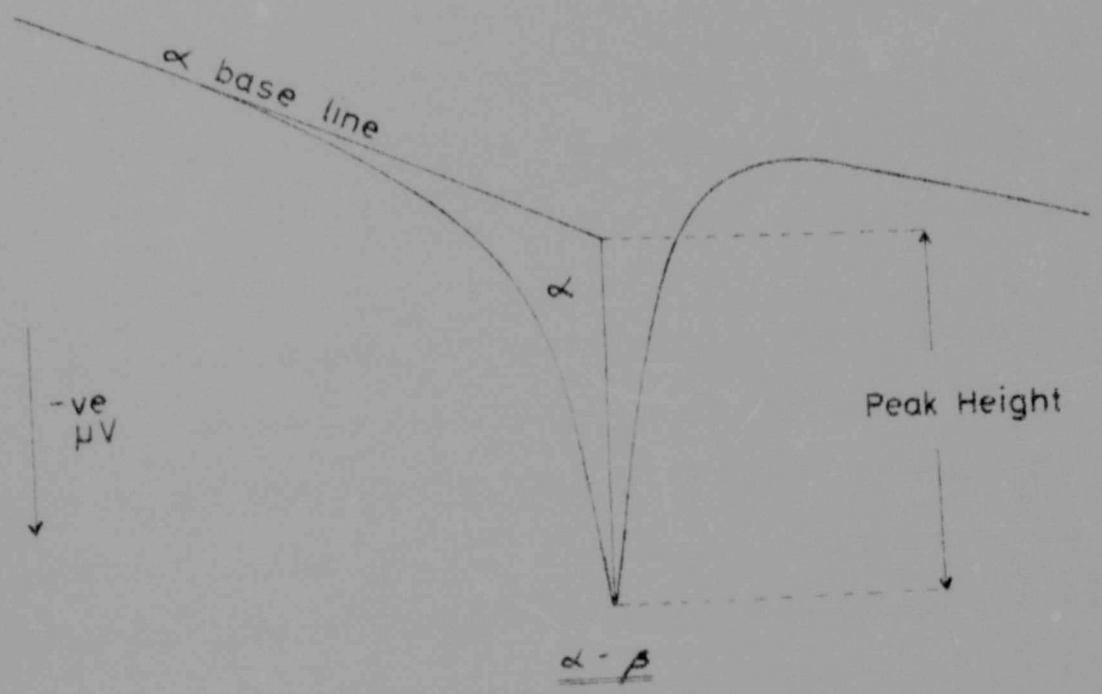
In the present work it has been found that especially

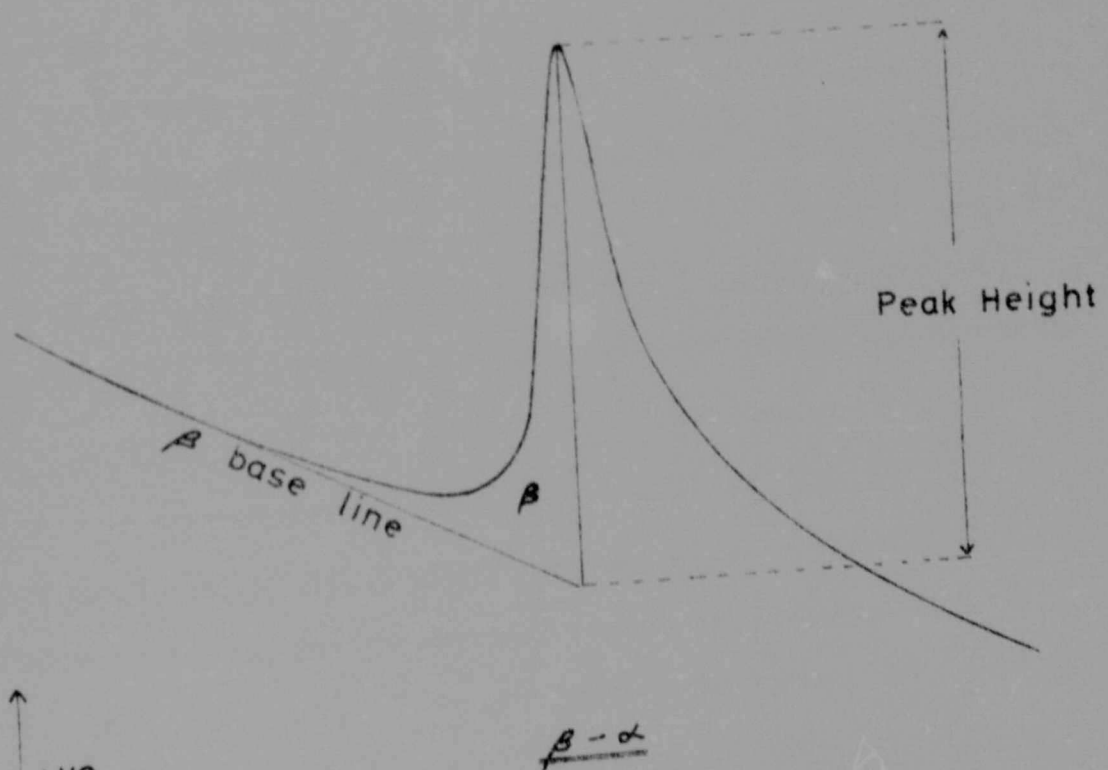
/for .....

III . 3

ASSESSMENT OF D.T.A. RESPONSE

Method finally adopted





for the horizontal furnace, these base lines, although sloping, have been very stable. There is considerable theoretical justification for assessing the response in this way, firstly because the sloping base line is caused by the change in specific heat of quartz with temperature, and secondly because the heat reaction is actually complete before the return of the curve to its new base line. However, most workers claim that it is the area, and not the height, that is a measure of the heat involved in a reaction.

Some measurements of the D.T.A. response were measured by a method which is described by Webb [52]. It is really only a slight modification of that given above, and is illustrated in Fig III, 4. Peak height measurements are exactly the same as above. Where the base line after the peak is above that before the reaction, the base line is produced to meet the curve as shown for the $\alpha \rightarrow \beta$ inversion. Where it does not return to the same level, as for the $\beta \rightarrow \alpha$ quartz inversion, the straight portion of the return curve is produced to intersect the base line. In both cases the area measurement is the sum of the areas marked α and β. This was not adopted for the present work as it was felt that the centre line was less arbitrary and could be more precisely located than the straight portion of the return curve for the $\beta \rightarrow \alpha$ reaction.

A few measurements were also made on the $\alpha \rightarrow \beta$ reactions by extending the β- base line backwards to intersect the centre line, but these were discontinued due to the instability of the base line after the peak in certain instances.

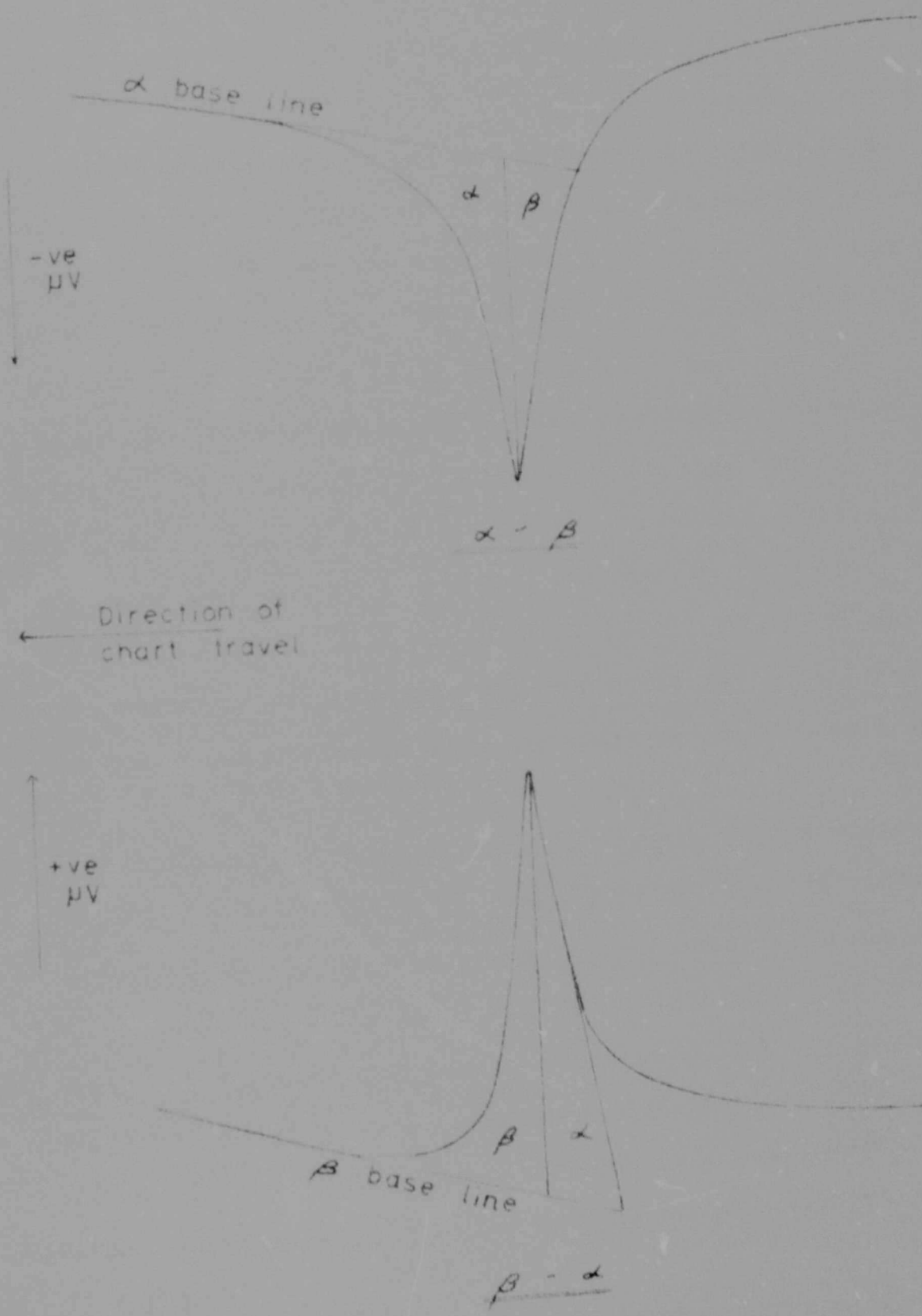
(d) <u>Comparison of results obtained for area measurements by polar planimeter and paper weighing</u>

Initially all area measurements were made by means of a polar planimeter. Each area was assessed at least three times, and it was found that with care a maximum variation

/of .....

Fig. III.4 ASSESSMENT OF D.T.A. RESPONSE

Dr. Webb's Method



of 2% could be achieved. Planimeter work was found to be very tedious, and it was thought that a much quicker method would be to trace the D.T.A. curves on to paper, cut out the portion for which the area is desired and weigh it. Preliminary measurements indicated that the method was promising, especially when it was found possible to print the D.T.A. trace directly from the chart paper on to some high quality graph paper.

Ten square-inch pieces of paper were cut from one piece of this graph paper, and square-inch pieces were also cut from thirty different sheets. These were all weighed and the results statistically analysed. It was found that the coefficient of variation for the first readings was 4.3 per cent, and for the second 3.0 per cent. The first figure would have been very considerably reduced, (to about 1.5%), if one odd reading had been excluded. This is probably justified, as this reading may have been due to inaccurate cutting of the paper squares. Several squares of paper were kept and weighed at intervals, sometimes on dry and sometimes on wet days. The total variation in these readings was less than 1 per cent.

A number of comparative measurements of the areas under the D.T.A. curves was then made by these two methods, converting the weight of graph paper to microvolt minutes by means of the average of the 30 measurements mentioned above. A two-way analysis of variance on these figures was carried out by the Statistics Department of this Laboratory, and this showed that the difference between areas as obtained by the two methods was not significant at any level.

Area measurements obtained by weighing of paper should therefore give the result to within 3 per cent. The accuracy of the planimeter method is only about 2 per cent, and the fact that these readings take at least

/ten .....

ten times as long to obtain has led to the adoption of the weighing method wherever possible. It has been suggested that the use of polythene sheet, which is not affected by humidity and has very constant thickness, would considerably improve the accuracy of the method, but the author has not yet been able to test this.

4. Preliminary Experiments on Test Sample No. 1

(a) Description of Tests

A large clear quartz crystal was ground down in an agate mortar to pass through a 325 mesh sieve. After "aqua regia" treatment to eliminate iron contamination, the dust was calcined for 20 minutes at 950°C. It was sized under an optical microscope, and was analysed by both chemical and X-ray diffraction techniques.

The sizing showed that while 60% of the dust was smaller than 6 μ (projected surface diameter), 13% was larger than 12 μ. Chemical analysis gave the total silica content of the dust as 97.92%, but X-ray diffraction results varied between 87 and 102 per cent. This may be due to the coarseness of this sample relative to the X-ray standard, 89% of which consisted of particles with a diameter of less than 6 microns.

In this series of tests, 26 cycles using 6 different packings of the test sample were carried out on the horizontal furnace, the equivalent figures for the vertical furnace being 27 cycles and 8 packings. Measurements were made on both the $\alpha \rightarrow \beta$ and the $\beta \rightarrow \alpha$ inversions, for heights and areas by all the methods described in Section 3 of this chapter. For convenience, the data sheets have been numbered H1A, H1B, V1A and V1B respectively, the H referring to Horizontal, V to Vertical, A to $\alpha \rightarrow \beta$ and B to $\beta \rightarrow \alpha$. This method of nomenclature was used throughout, and will be adopted for the Tables in this chapter.

/The .....

The results obtained were statistically analysed by standard methods. It would serve no purpose to reproduce all the figures here, only Table III, H1A being given as an example. A summary of the statistical data is given in a separate table.

(b) Experimental Results

As mentioned above, details of only the $\alpha \rightarrow \beta$ measurements on the horizontal furnace results are given. It will be observed that twelve D.T.A. cycles were conducted on one packing of the test sample and later, after reconstruction of the sample holder mounting, a further 10 cycles on another packing. Eleven cycles on the same packing were conducted on the vertical furnace, and these were used to calculate the within-packing variances of the D.T.A. responses.

The criterion in a judgement of this nature is the coefficient of variation which is given by the

$$\frac{\text{standard deviation}}{\text{mean}} \times 100$$

and is expressed as a percentage. These results are grouped together in a convenient form in Table III, 1.

/TABLE III, 1. .....

TABLE III, 1

COEFFICIENTS OF VARIATION OF HEIGHT AND AREA READINGS

Parameter Measured	Horizontal Furnace				Vertical Furnace	
	First Packing		Second Packing		First Packing	
	$\alpha \rightarrow \beta$	$\beta \rightarrow \alpha$	$\alpha \rightarrow \beta$	$\beta \rightarrow \alpha$	$\alpha \rightarrow \beta$	$\beta \rightarrow \alpha$
Peak Height	1.13	0.76	1.10	0.89	1.59	1.20
Base Displ. Ht	2.72	5.04	6.93	12.21	13.43	13.95
Total Ht.	1.06	0.87	1.38	1.57	1.05	1.25
α - base line height	0.40	-	1.62	-	1.61	-
β - base line height	2.24	1.20	0.96	1.11	-	1.88
Peak area	3.95	1.54	3.75	2.00	9.18	5.01
Base Displ. area	2.50	6.22	6.69	13.01	13.31	15.64
Total area	2.40	2.42	3.72	5.18	4.58	6.11
α - half peak area	4.59	3.98	4.48	2.69	15.58	8.45
β - half peak area	4.47	3.53	5.08	7.17	17.46	8.56
Total half peak area	3.28	1.77	3.24	3.45	7.47	6.24
α - base line $\frac{1}{2}$ peak area	4.05	-	11.05	-	10.09	-
β - base line $\frac{1}{2}$ peak area	6.53	2.99	3.78	3.84	-	6.33

(c) Discussion of results:

Several interesting features immediately reveal themselves. Firstly, all height measurements excepting those for the base displacement somewhat surprisingly show much better reproducibility than the corresponding measurements for the peak areas. The figures underlined are those in which we are most interested. These show that for peak heights the within-packing variance is less than 2 per cent, whereas for the areas it can be as much as 11 per cent. The coefficient of variation of 11.05% for the horizontal furnace α - base line half-peak areas can probably be discounted since it is nearly 3 times as much as those for the other area measurements on this furnace. The within-packing

/variance .....

variance for these areas is of the order of 4 per cent, or twice as large as that for the heights. Secondly, there is quite obviously a change in the readings from No. 17 onwards in Table III, H1A, the point at which readings with the new sample holder mounting began. This indicates that should the differential thermocouple wire ever have to be replaced, the apparatus will require recalibration.

A third point of interest is that the variances for the horizontal furnace readings appear to be slightly less that those obtained for vertical furnace readings. This is surprising, in view of the claims that have been made for nickel block sample holders in the literature. It must be observed that the readings from the platinum crucibles were slightly more than one-and-a-half times as much as those from the nickel block in the conditions under which the respective furnaces were operated.

Returning to Table III, H1A, the readings in row numbers 4, 11, 14, 19 and 25 were obtained on the same days as the results immediately preceding them. In other words, the operating conditions of the apparatus were not quite the same as for the other tests, since the furnace lagging would still have been warm. An inspection of the figures in the table does not reveal that this was of any significance. This conclusion is of considerable practical importance, since it means that two cycles a day can be conducted on the same furnace with confidence.

The statistical calculations carried out on these figures also showed that between-packing differences could not be accounted for by random experimental variation alone. In this analysis they used an "F" - test

$$F = \frac{\Sigma n_c (\bar{x}_c - \bar{x}_{..})^2}{(k-1)\, \sigma_1^2}$$

/where .....

where $\bar{x}_i$ = mean of the ith packing

σ_1^2 = within-packing variance

$\bar{x}_{..}$ = weighted mean = $\frac{\Sigma n_i \bar{x}_i}{\Sigma n_i}$

n_i = number of cycles

K = number of packings

The "Fs" for all but one of the parameters tested in this way proved to be significant, meaning that there is a real between-packing variance (σ^2). Each mean is therefore subject to a variance

$$\sigma^2 + \frac{\sigma_1^2}{n_i}$$

n_i being the number of cycles from which the mean is taken.

The results above were used to plan the tests to be described in the following section in so far as the number of packings (K) and the number of cycles per packing (n) executed on each test sample are concerned. The variance of the mean is given by

$$\frac{1}{K}\left(\sigma^2 + \frac{\sigma_1^2}{n}\right)$$

The value of this variance is more effectively reduced by increasing K than by increasing n. It was therefore decided to cary out 2 D.T.A. cycles on each of 4 packings of a sample rather than 3 cycles each on 3 packings, as was the original intention.

One last point in connection with these preliminary results concerns the comment at the foot of page 19, Chapter I of this thesis. Since successive runs on the same packing of a sample do not show any tendency towards a lower D.T.A. response, it is extremely unlikely that some of the β - quartz does not revert to α - quartz on cooling. Also, the first cycle on a particular packing does not seem to differ much from subsequent ones. Were it to do so, the author would be more inclined to attribute the difference to the slightly hygroscopic nature of the inert material, calcined alumina. Even

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though it is kept in a dessicator, there is still the possibility that it might absorb a small quantity of moisture. This could only be completely eliminated by re-calcining the alumina before each test.

5. Calibration Tests:

(a) Description

The purpose of these tests was to examine the linearity of the D.T.A. response for the various parameters measured and to construct calibration curves for the apparatus by means of which the percentage of quartz in an unknown test sample could be estimated. For this purpose, a pure sample of superfine Dowson and Dobson quartz was chosen as a standard. Microscopic sizing showed that this dust contained no particles of diameter greater than 7.5 μ, and that more than 70% of the particles had a diameter of 2 μ or less.

Using this dust as a 100% sample of quartz and minus 270 mesh calcined alumina as the diluent, samples containing 80, 60, 40 and 20 per cent quartz by weight were prepared. Two cycles on each of 4 packings were carried out for both furnaces on all these 5 samples (Nos. 10 to 14). The responses on the 40 D.T.A. records obtained from each furnace were assessed by all the methods described in Section 3 of this chapter for both the $\alpha \rightarrow \beta$ and the $\beta \rightarrow \alpha$ quartz inversions.

Since the effect of packing had proved to be significant, it was decided to try to correct the results to a standard mass. This was done by means of the formula

$$x = \frac{x_i \times 250}{m_i}$$

where x = corrected response

x_i = response for the ith packing

m_i = mass of dust in the ith packing.

The mass 250 mgm was arbitrarily chosen, since approximately this weight of several different samples could

/be .....