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Determination of the percentage of quartz (SiO2) in samples of mine dust by the method of differential thermal analysis

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similar arrangements previously described is that the differential thermocouple junctions are introduced from below and are rigidly located within the crucibles. (See Figs: II, 3 and 4, and Fig: II, 12). Every effort was made to make this set-up as symmetrical as possible after some preliminary results had been obtained with a simpler system. The ceramic tube on which the baseplate is mounted was ground with a diamond wheel as were the twin-duct and four-duct thermocouple insulating tubes that carry the thermocouple wires. Alundum refractory cement was used to cement all the components into position. The thermocouple beads are axially situated in the crucibles about 5 mm. from their bases. The system of location of the holder in the furnace is such that the crucibles are mounted in exactly the same position each time. The test sample is always packed into the crucible furthest from the mouth of the furnace and the inert material into the other crucible.

The cylindrically shaped nickel block (See Fig: II, 6 and Fig: II, 13) is 30 mm. high and 50 mm. in diameter. It weighs 515 gm., and is supported by a nickel tube with an internal flange which in turn rests on a ceramic tube 12 inches long with an external diameter of 2 inches and internal diameter 1½ inches. The thermocouple junctions are also rigidly located in the sample wells as described in section 3 (b)(ii), the beads being very nearly centrally situated in the cavities. The whole set-up is located at a fixed height in the vertical furnace by means of a plate

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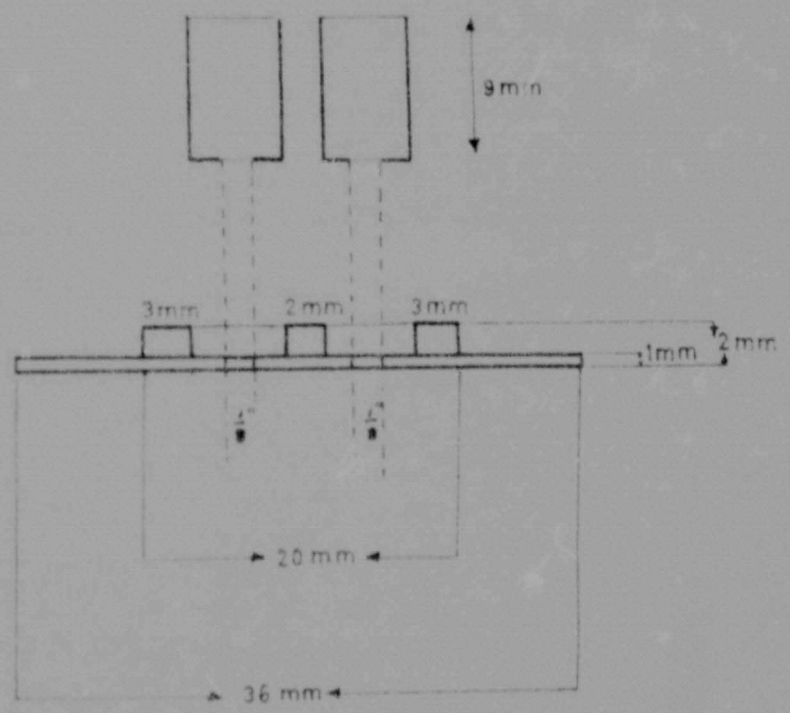
Fig. II, 12 HORIZONTAL FURNACE SAMPLE HOLDER - PLATINUM CRUCIBLES AND BASE PLATE



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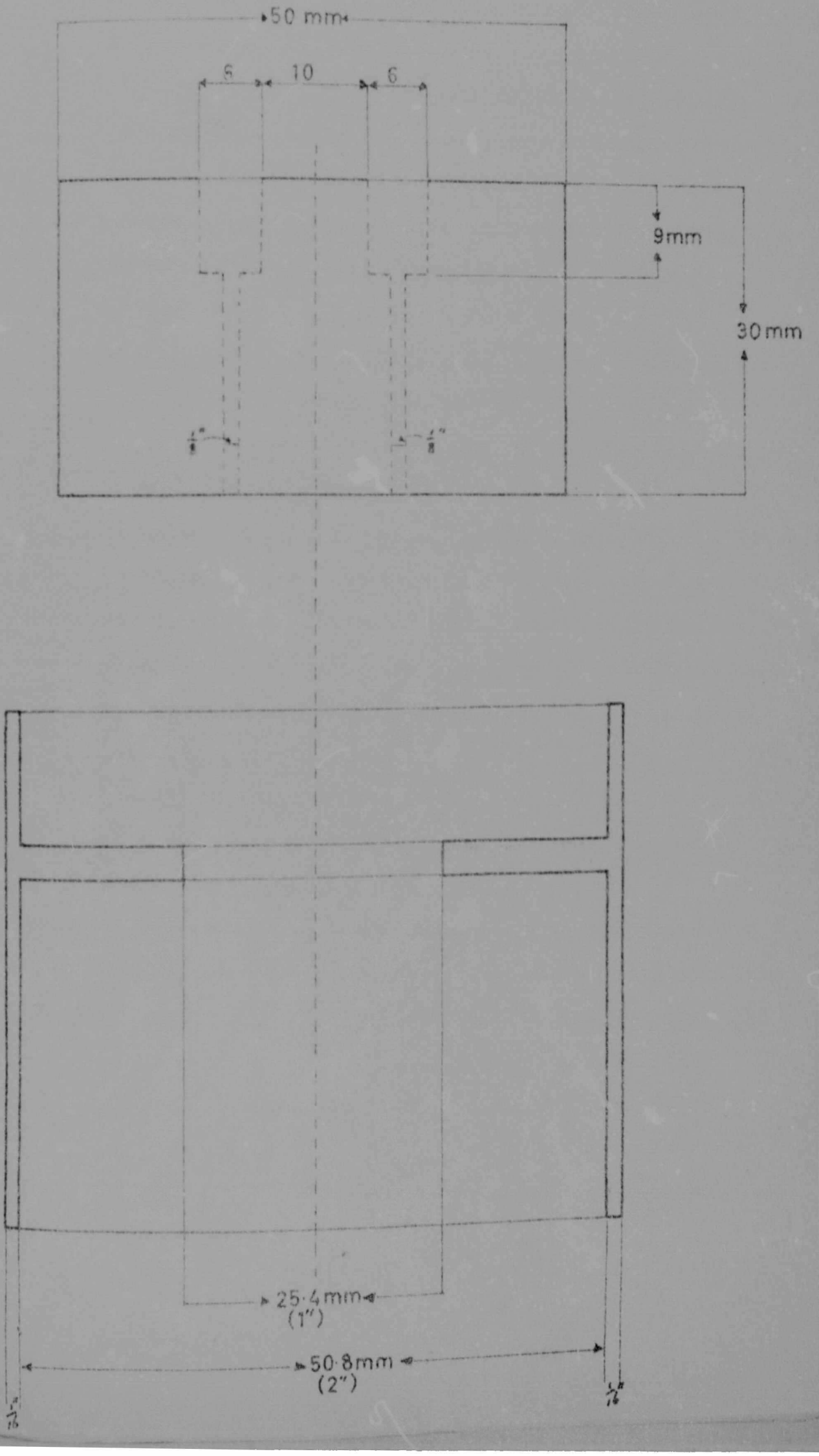
VERTICAL FURNACE SAMPLE HOLDER,

NICKEL BLOCK AND SUPPORT



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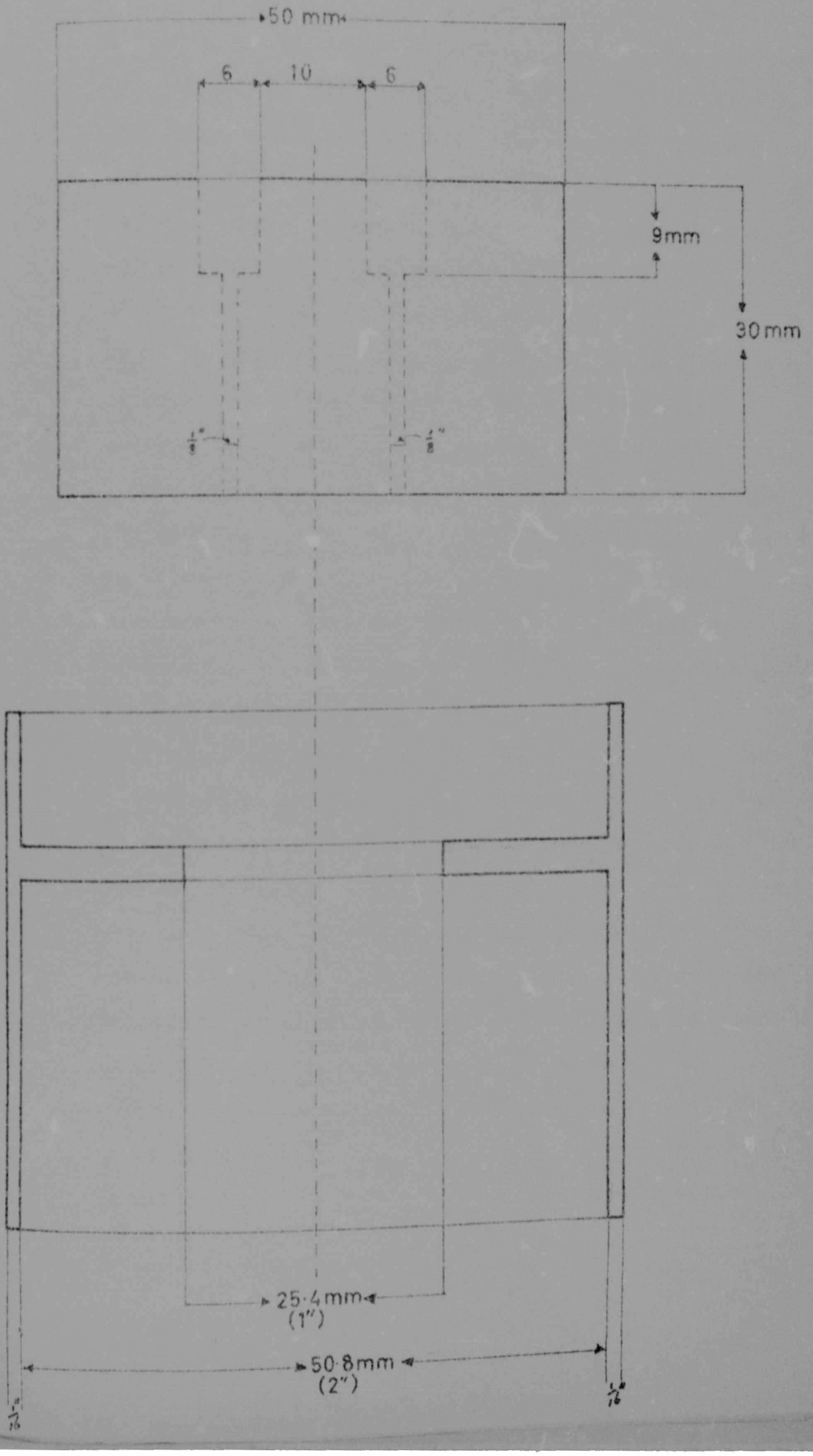
VERTICAL FURNACE SAMPLE HOLDER,

NICKEL BLOCK AND SUPPORT



on which the eramic tube rests when the holder is in position. The nickel block is almost at the centre of the furnace tube, the top of which is covered with a flat sheet of asbestos-cement. Completely sealing off the top of this tube was tried but it reduces the natural cooling rate of the furnace to too low a rate.

In the interests of durability it was decided to use noble-metal couples in this work. Platinum versus platinum - 13% rhodium couples have most often been used, and these have an output of approximately 11.6 microvolts per degree centigrade difference in temperature at 600°C. Pallaplat, which is composed of 90% platinum - 10% rhodium versus 60% palladium - 40% Gold, has a sensitivity of about 51 μV per degree in the range of temperatures in which we are interested in the present work. It was considered that the factor of 5 by which the sensitivity would be increased by using this material for the differential thermocouple instead of the more usual material would be very important with such a small thermal reaction as the $\alpha \rightleftharpoons \beta$ quartz inversion. Platinum versus platinum - 13% rhodium was chosen for the temperature measuring couple.

Initially 0.1 mm. diameter wire was tried for the differential thermocouple. This had a low heat capacity, but in the interests of rigidity this was later changed for 0.3 mm. diameter wire. The results obtained enable a limited investigation of the effect of wire diameter on the D.T.A. response to be made, and this will be given in a later section.

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(c) Heating Programme Controller:

What is required in this work is that the heating and/or cooling rate of the furnace should be reproducible. Most workers have been interested in a wide variety of clay minerals, et cetera, and had to have a constant rate of rise or fall of temperatures from 100 to 1000°C. In the differential thermal analysis of quartz we are only interested in the rate of temperature change between 500 and 650 degrees centigrade. As long as this can be kept highly reproducible, the necessity for introducing a complicated programme controller need not arise.

Measurements of the thermal response of the dust samples have been made on both the $\alpha \rightarrow \beta$ and the $\beta \rightarrow \alpha$ reactions in all this work. For the latter, both furnaces are allowed to cool at their natural rates after being heated to a more or less fixed temperature. Variations of up to 20°C. in this temperature and of 10°C. in the ambient room temperature seem to have a completely negligible effect on the thermograms provided this temperature is sufficiently high to allow stability of cooling to be reached by the time the thermal record is started. For the horizontal furnace the power is switched off exactly fifty minutes after switching it on, and the natural cooling rate through the $\beta \rightarrow \alpha$ quartz inversion is about 8°C per minute. For the vertical furnace the comparative figures are 60 minutes and just under 4°C per minute.

It was decided to control the heating cycle by keeping the power input into the respective furnaces constant. A variac and a wattmeter were therefore

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introduced into the circuit, the former being adjusted to give a predetermined value on the latter for each cycle. Variations in the heating rate can be achieved by altering the power input, and the results have so far been satisfactory.

The furnace temperature is measured by means of a platinum versus platinum - 13% rhodium thermocouple, this giving a direct reading on an indicating meter (Fig: II, 2) to which the thermocouple is matched. The cold junction for this is provided by the mercury-filled U-tubes in the widenecked thermos flask (Fig: II, 7), which is filled with water at room temperature. Spurious thermal effects in the differential thermocouple are eliminated in this way, and the method of making electrical contacts is very convenient. The mercury does not attack platinum-rhodium alloys, and stainless steel is used for the contacts in the other arms of the U-tubes.

As previously mentioned, the exact temperature at which the $\alpha \rightleftharpoons \beta$ quartz inversion takes place is of no concern in the present work, and the furnace temperature is used only to give an indication as to when thermograms should be begun or to determine the rate of the temperature change when desired. Many such determinations have been made with both furnaces, and the reproducibility of the heating and cooling cycles has been found to be high when initial conditions have been the same. A striking example of this is provided by Fig: II, 14, in which two cycles conducted under identical conditions on the vertical furnace were superimposed by feeding the temperature-measuring thermocouple output directly into a 10 mV.

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Fig. II.14 SUPERIMPOSITION OF TWO TEMPERATURE CYCLES ON THE VERTICAL FURNACE

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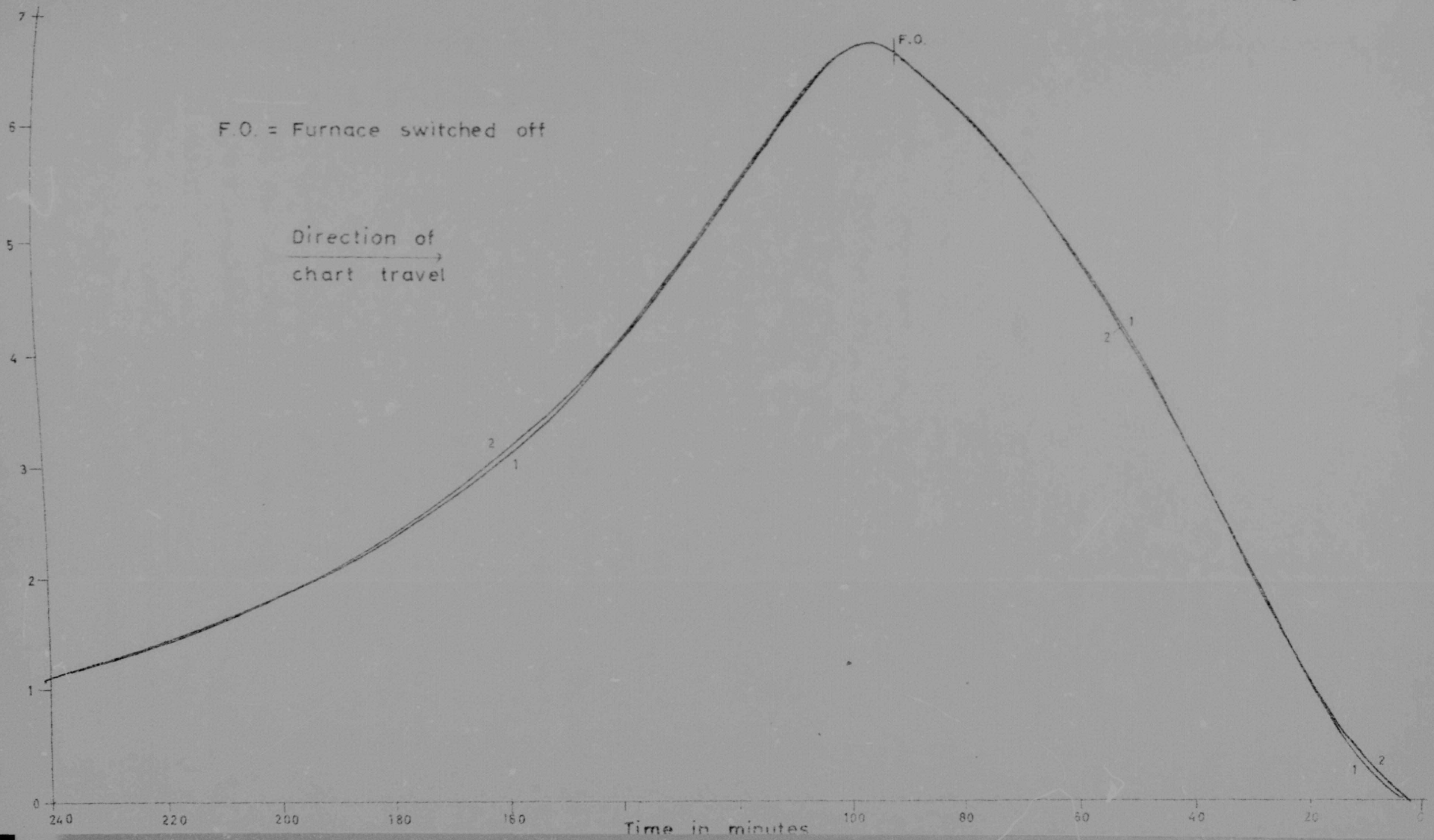

potentiometric recorder. The thermocouple used has an e.m.f. of 10.47 mV at 1000°C, and the chart speed was three inches per hour for this test, so the rate of temperature change at any point can be determined from the record. Figs: II, 15 and 16 show how the heating and cooling rates on both furnaces varied for two cycles carried out on the same day, that is, with different initial conditions, the furnace laggings still being warm when the second cycle was started. It will be seen that, although the inversion point is reached at slightly different times in each case, the actual rate of heating or cooling through the inversion point was not significantly altered.

The last test described above is of considerable importance since for many of the D.T.A. cycles to be described in the following chapter, two and even three determinations were made with one furnace on the same day. This was necessary in order to complete the programme of work, but it was feared that it might have had a detrimental effect on the reproducibility of the results.

(d) Differential Temperature Recorder:

The requirements of the differential temperature recorder have already been given in Section 1 of this chapter. Using Pallaplat for the differential thermocouple, a temperature difference of 0.15°C between the test and the inert samples would give rise to a potential of about 7.5 μV . In view of its vastly superior convenience for routine use, it was decided to use an electronic pen recorder of the potentiometric type to record the D.T.A. response if a

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suitable preamplifier could be found for such an instrument.

Many preliminary D.T.A. tests were made using a sensitive galvanometer and recording the results manually. These indicated that for a 300 mgm. sample of -325 mesh ground quartz crystal, a temperature difference of about 2.5°C was developed between the crucibles in the horizontal furnace. From this it was clear that an amplifier giving a stable gain of 200 would be sufficient to detect as little as 1% quartz in a sample.

On this basis a Leeds and Northrup Stabilized D - C Microvolt Amplifier (Type 9835-B) and a 10 mV. Sunvic High Speed Recorder (Type RSP2) were purchased. These instruments have been described in Section 3(a) and it only remains here to give the accuracy claimed for them by their manufacturers. The limit of error of the amplifier response is given as ± 0.4% of the reading with a maximum zero offset of ± 0.5 microvolt within which the drift will not exceed 0.2 μV during any one minute interval after warm-up. The response time is given as two seconds for a source resistance of 2,000 ohms. maximum. The source resistance in the present application is only about 5 ohms. The instrument is intended either as an indicator or as a preamplifier yielding ± 5 millivolts maximum across 500 ohms, corresponding to any selected input range. This gives it a maximum amplification of 200, and it is designed for use with a potentiometer null-balance recorder only.

The recorder is matched with the above amplifier, a 10 millivolt input giving a full-scale deflection

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of 9 7/8 inches. The chart is marked with 0 - 100 equal divisions and has transverse rulings every inch; being a single-pen instrument it gives a continuous trace, and an added advantage is that the chart speed can be adjusted to 1, 2 or 3 inches per minute or per hour respectively. The overall accuracy is stated to be ± 0.5% of the scale span, the sensitivity being limited only by the slidewire steps, i.e. 0.1% of the scale span. The speed of response is given as approximately 2 seconds for the full scale traverse. The slide-wire standardisation against a standard Weston cell is fully automatic but can also be initiated by depressing the "Test" switch for 5 seconds. The automatic standardisation has been disconnected in the present instrument as it might interfere with the quartz peak.

Both the above instruments appear to have fully justified the manufacturers' claims. The latter has been in frequent use for ten months, and both have been used almost every day for six months without any trouble whatsoever. The overall accuracy of the recording equipment should be within ± 1 μV . As it is desired to be able to set the recorder zero at any scale value for any input, a centre-tap potentiometer device by means of which a positive or negative bias can be placed on the recorder was designed. This is shown in Fig: II, 17, and is placed in the positive lead from the amplifier to the recorder. The control knob for the recorder zero can be seen below the temperature indicator on the control panel (Fig: II, 2). It is found to be

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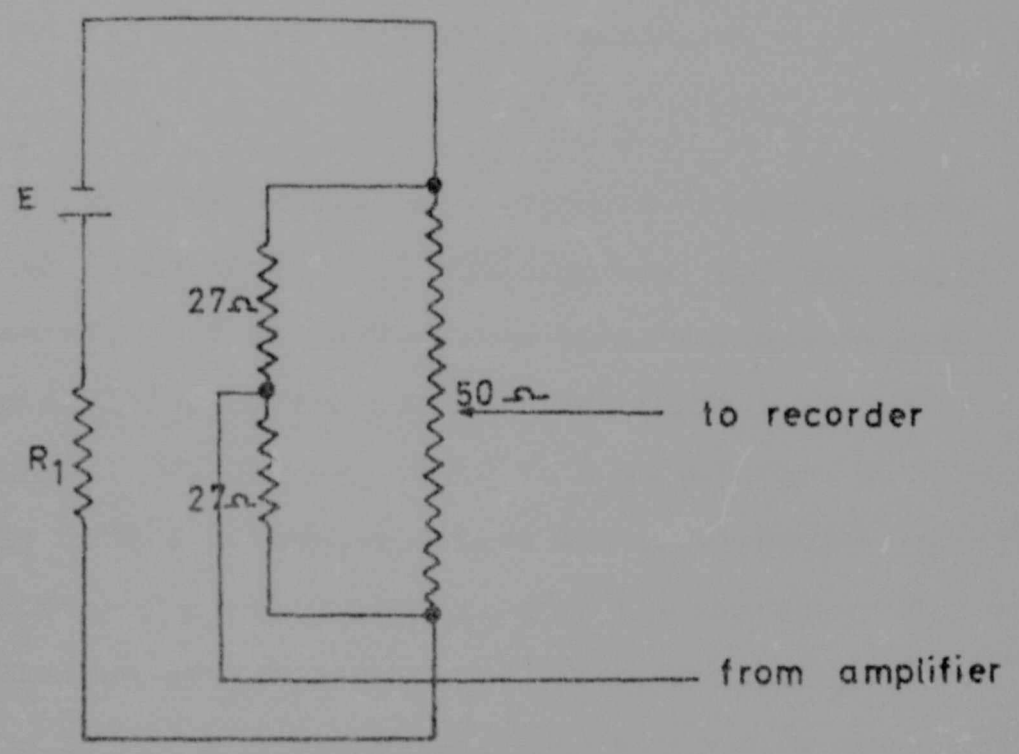
Fig. II, 17

CENTRE - TOP POTENTIOMETER DEVICE FOR RECORDER ZERO SETTING



E = 1·5V

R_1 = 1800Ω

quite satisfactory in operation, and it serves to increase the sensitivity of the equipment as a greater amplification can be employed by setting the recorder zero on either the left or the right of the chart according as the deflection of the peak is going to be the right or the left. The input leads from the amplifier to the recorder have been shielded against A - C pickup, as has the recorder zero control circuit.

5. GENERAL :

The performance of the equipment described above has been found to be most satisfactory, and many hundreds of beautiful D.T.A. curves have been obtained to date. The sensitivity of the differential thermal recorder is such that 1 μV, corresponding to a temperature difference of only 0.02°C between the test and inert sample, gives a deflection of 2 small divisions on the recorder, that is, a deflection of 1/5 inch. The recorder reading can be read to 1/5 of a division, corresponding to a temperature of 0.002°C.

It has been found that a test sample consisting of 100% S_iO_2 gives a potential of 125 μV in the horizontal furnace and 75 μV in the vertical furnace under roughly the same conditions. In other words, on the most sensitive amplifier range (50) deflections of 25 and 15 inches would be obtained on the recorder for the $\alpha \rightarrow \beta$ quartz transition from the two furnaces. It is clear that under these circumstances it should be possible to detect as little as 1 per cent of quartz in the test sample quite easily. Whether or not this amount of quartz would yield

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reproducible results is another question.

Before closing this chapter, a few details of interest will be given of some of the preliminary D.T.A. tests that were conducted with the apparatus described above. The results are to be regarded as comparative only.

(a) Effect of Differential Thermocouple Wire Diameter:

The preliminary mounting for the platinum crucibles in the horizontal furnace was equipped with 0.1 mm. diameter thermocouple wire. In the final set-up this was replaced with 0.3 mm. diameter wire of the same material, so a limited comparison of the D.T.A. responses can be attempted. It must be emphasised that the height and area measurements given here, although consistent among themselves, are not obtained according to the conventions eventually adopted.

TABLE II, 1

Effect of Thermocouple Wire Diameter on D.T.A. Response.

e No.	Wire Diam in mm.	Inversion	Peak height in μV Peak height	Peak height in μV Average	Peak area in μV min. Area	Peak area in μV min. Average
/1	0.1	$\alpha \rightarrow \beta$	176	176	106.5	106.5
/1	0.3	$\alpha \rightarrow \beta$	147	146	79.0	85.8
/1			144		89.0	
/1			146		89.4	
/1	0.1	$\beta \rightarrow \alpha$	150	158	161.5	165.3
/1			161		164.0	
/1			159		165.5	
/1			160		170.0	
/1	0.3	$\beta \rightarrow \alpha$	113	113	81.0	83.8
/1			111		80.5	
/1			111		85.5	
/1			116		87.4	

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These were among the very first results obtained from the apparatus, and even at this early stage certain interesting facts emerged. Firstly, it was apparent that the peak height measurements tended to be more reproducible than peak area measurements. Secondly, while both heights and areas decreased for the thicker wire for both the $\alpha \rightarrow \beta$ and the $\beta \rightarrow \alpha$ inversion, there was no apparent uniformity in the magnitude of the decrease. This indicated that separate calibration curves for each measure of the D. T. A. response would probably be necessary. It will be seen that, depending on the method used for measuring the D. T. A. response, increasing the wire diameter from 0.1 to 0.3 mm. decreases the magnitude of the peak by anything from 18 to 50 per cent.

(b) Effect of Heating Rate on D. T. A. Response :

A few preliminary tests were conducted by varying the power input into the horizontal furnace heating element. Simple theory suggests that the peak area under a thermal peak should be independent of the heating rate, whereas the height of the response increases with the rate of temperature change.

TABLE II, 2

Effect of Heating rate on $\alpha \rightarrow \beta$ Quartz D. T. A. Response :

Cycle No.	Power Input in Watts.	Peak height in μV		Peak Area in μV- min.	
		Height.	Average.	Area.	Average
H018/1	-	83.0	-	58.5	-
H019/1	375	83.0	82.3	60.0	59.2
H020/1	-	81.0	-	58.6	-
H021/1	400	89.6	89.6	59.2	59.2
H021a/1	425	93.0	93.5	58.2	58.7
H022/1	-	94.0	-	59.2	-
H027/1	450	106.8	106.8	72.4	72.4
H029/1	-	121.4	-	72.2	-
H030/1	500	123.2	122.5	74.6	75.0
H033/1	-	122.8	-	78.2	-
H014/1	600	144.0	144.0	89.0	89.0

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These results must be treated with some caution as exact rates of temperature change are not available, but they do show that not only the peak height but also the peak area of the response increases as the heating rate increases. Further tests on the heating rate will be reported in the following chapter.

(c) Effect of Particle Size of Inert Material on D. T. A. Response :

α - alumina (Al_2O_3) is by far the most commonly used reference material for D. T. A. tests. The reason for this is that after preliminary calcination it has no heat effects and its thermodynamic properties are similar to those of most ceramic materials. It is nevertheless possible that the particle size of the inert sample could have an influence on the D. T. A. response. Some -270 mesh calcined alumina was ground down to pass through a 325 mesh sieve. Comparative results using these two grain sized inert samples are given in Table II, 3.

TABLE II, 3.

Effect of Particle Size of Inert Material in D.T.A. Response:

Sample Number.	Cycle Number.	Size Grading of Ref: Material.	$\alpha \rightarrow \beta$ Peak Heights. Height	$\alpha \rightarrow \beta$ Peak Heights. Av:	$\alpha \rightarrow \beta$ Peak Areas. Area	$\alpha \rightarrow \beta$ Peak Areas. Av:	$\beta \rightarrow \alpha$ Peak Heights. Height	$\beta \rightarrow \alpha$ Peak Heights. Av:	$\beta \rightarrow \alpha$ Peak Areas. Area	$\beta \rightarrow \alpha$ Peak Areas. Av:
11	H069/11	-270 Mesh	61.6	62.5	34.2	34.7	67.2	67.2	60.6	60.3
	H070/11		63.4		35.2		67.2		60.0	
12	H079/12		53.2	55.7	23.6	31.1	55.0	54.7	43.1	43.6
	H080/12		54.4		30.2		55.6		45.0	
	H081/12		57.0		34.8		54.0		43.1	
	H082/12		58.0		35.8		54.2		43.3	
11	H073/11	-325 Mesh	74.4	74.3	64.6	65.2	62.8	62.8	48.8	48.1
	H074/11		74.2		65.8		62.8		47.4	
12	H075/12		64.2	63.6	34.4	48.4	49.4	50.7	31.3	33.6
	H076/12		65.4		60.2		50.2		34.2	
	H077/12		62.0		47.9		51.2		33.4	
	H078/12		62.9		51.0		51.8		35.4	

/From .....

From these figures it is apparent that the particle size of the reference material has a somewhat irregular effect upon the D.T.A. response. As -270 mesh calcined alumina had been used with satisfactory results up to the time these tests were conducted, it was decided to revert to this for the inert material for all future tests. Many workers have recommended that the particle size grading of the inert material should be the same as that of the test sample. However, as this is likely to vary from sample to sample in the present work, it was considered more satisfactory to use standard inert material for all tests.

(d) Zero Drift of the Apparatus :

Any zero drift that is present in the D.T.A. record during a cycle of temperature change could be due to any one of the following factors :-

i) Differences in the sample holders, especially crucibles;

ii) Differences in the thermocouple junctions in the sample holders;

iii) Lack of symmetry of the sample holder mounting with respect to the furnaces;

iv) Differences in the thermal conductivity of the test and inert samples;

v) Variations in thermal properties of the test sample with temperature;

vi) Stray thermal effects outside the furnace.

Due to the reproducibility of the D.T.A. curves obtained when repeated under identical conditions, and to the constant temperature junctions in the thermos flask, the last factor is not likely to be important. The fifth factor is one over which we have no control, and assuming that the samples are consistently packed

/(something ....

(something that will be discussed in detail in the following chapter), there is little that can be done about the fourth possibility. However, tests were conducted on both furnaces by packing both sample holders with minus 270 mesh calcined alumina. The results obtained are interesting.

The recorder system was set to give maximum sensitivity, that is, to amplifier range 50. The recorder chart speed was set to 3 inches per hour, and the entire heating cycle and the cooling cycle until well below the quartz inversion peak temperature was recorded for both furnaces. For the horizontal furnace (platinum crucible sample holders) the following results were obtained. When the furnace was switched on the trace drifted steadily out to + 13.25 μv in about five minutes and then returned to zero after about fifteen minutes. As the temperature increased between about 500 and 660°C in fifteen minutes, the trace drifted from + 0.25 to -2.75 μv, that is, a drift of 0.2 μv per minute in the region of the $\alpha \rightarrow \beta$ quartz inversion. In other words the temperature difference between the two crucibles changed by only 0.06°C as the furnace temperature increased by 160°C. A further point of interest in this trace is that a minute "blip", of about 0.4 μv amplitude, was obtained at the quartz inversion temperature, indicating that even the minute traces of this dust that remained on the thermocouple junction mounting could be detected. This feature was present on both the $\alpha \rightarrow \beta$ and $\beta \rightarrow \alpha$ inversions and also on the trace for the vertical furnace. When the furnace input was cut off at about 730°C, the base line reading was -5.5 μv. This drifted to + 5.0 μv in about two minutes, but then returned steadily to zero again. In the twenty minute period that it took the furnace temperature to drop from about 620 to 470°C, the

/trace ....

trace drifted only from -1.5 to - 2.8 μv, this 1.3 μv change corresponding to a temperature change of less than 0.03°C. These drift figures are obviously completely negligible, being within the limits of accuracy of the recording equipment, so the horizontal furnace sample holders and mounting must be considered to be completely satisfactory.

The conclusions arrived at above also apply to the vertical furnace assembly, so that it is not necessary to give details of this trace. Only two points need be made: firstly, the initial drift immediately after switching the power on and off was much smaller than for the horizontal furnace, probably due to the large thermal capacity of the nickel block sample holder; secondly, a distinct peak was obtained in the trace at about 370°C, the curie point of nickel. Finally, random variations of about 0.5 μv occurred from the general drift line for the cooling cycle, probably due to the effect of convection currents.

(e) Some Preliminary Results :

The following few results are given not so much because they possess any intrinsic value, but because they revealed at an early stage the nature and magnitude of the problems that existed in the application of D.T.A. to the quantitative determination of quartz in dust samples.

Table II, 4 gives D.T.A. and X-ray diffraction analysis results that were obtained on four quartz samples all prepared from the same pure quartz crystal.

/TABLE II, 4 ....

TABLE II. 4

Comparison between D. T. A. and X-ray response for various quartz samples:

Sample No.	Cycle No.	Wt: of Sample in mgm.	Description of Sample. (all -325 Mesh)	$\alpha \rightarrow \beta$ Height in μV		$\alpha \rightarrow \beta$ Area in μV-min:		$\beta \rightarrow \alpha$ Height in μV		$\beta \rightarrow \alpha$ Area in μV-min:		% SiO_2 by X-ray
				Ht.	%	Area	%	Ht.	%	Area	%	
1	H003/1	300	Ground 2-15 μ	176.5	92.2	106.5	99.1	166.5	93.0	170.0	112.2	102
2	H005/2	195	Fine, treated with aqua regia. 1-6 μ	97.5	50.9	58.0	53.9	90.5	50.6	75.0	49.5	104
3	H007/3	266	As above but treated with HF. 6-> 15 μ	176.5	92.2	101.0	93.9	160.0	89.4	121.5	80.2	84
4	H006/4	314	As No.2 but coarser fraction. 3->15 μ	191.5	100	107.5	100	179.0	100	151.5	100	106

For purposes of comparison, sample No. 4 is arbitrarily chosen as 100% quartz. Apart from one isolated area result (112.2%) where the measurement must be regarded with suspicion, the D.T.A. response, uncorrected for mass of sample, shows surprisingly consistent results as measured in the four different ways. No corrections were made for the mass of the test sample here, and this will be discussed in the following chapter. It will be noticed that the mass of sample No. 2 is only two-thirds of that for No. 1. The mean deviation of 12 X-ray determinations on each of these samples is given as 5, 14, 2.5 and 16 per cent for Nos. 1, 2, 3 and

/4 respectively ...

TABLE II. 4

Comparison between D. T. A. and X-ray response for various quartz samples:

Sample No.	Cycle No.	Wt: of Sample in mgm.	Description of Sample. (all -325 Mesh)	$\alpha \rightarrow \beta$				$\beta \rightarrow \alpha$				% SiO_2 by X-ray
				Height in μV		Area in μV-min:		Height in μV		Area in μV-min:		
				Ht.	%	Area	%	Ht.	%	Area	%	
1	H003/1	300	Ground 2-15μ	176.5	92.2	106.5	99.1	166.5	93.0	170.0	112.2	102
2	H005/2	196	Fine, treated with aqua regia. 1-6 μ	97.5	50.9	58.0	53.9	90.5	50.6	75.0	49.5	104
3	H007/3	266	As above but treated with HF. 6->15μ	176.5	92.2	101.0	93.9	160.0	89.4	121.5	80.2	84
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For purposes of comparison, sample No. 4 is arbitrarily chosen as 100% quartz. Apart from one isolated area result (112.2%) where the measurement must be regarded with suspicion, the D.T.A. response, uncorrected for mass of sample, shows surprisingly consistent results as measured in the four different ways. No corrections were made for the mass of the test sample here, and this will be discussed in the following chapter. It will be noticed that the mass of sample No. 2 is only two-thirds of that for No. 1. The mean deviation of 12 X-ray determinations on each of these samples is given as 5, 14, 2.5 and 16 per cent for Nos. 1, 2, 3 and

/4 respectively ...

4 respectively.

This matter will be taken up again in the following chapter, as will the mass of a given sample that can be packed into the sample holders. It should be noted that if these results were all corrected to a standard mass, then the percentage response for sample No. 2 would be much higher.

Table II. 5 gives some further interesting results.

TABLE II. 5

Comparison of D.T.A. and X-ray response for Quartz Samples of different Particle Size :

Sample No.	Cycle No.	Wt: of Sample.	Particle Size Range of Sample.	$\alpha \rightarrow \beta$				$\beta \rightarrow \alpha$				$\%SiO_2$ by X-ray
				Height in μv		Area in $\mu v/min$		Height in μv		Area in $\mu v/min$		
				Ht.	%	Area	%	Ht.	%	Area	%	
2	H008/2	226	1 - 6 μ	106	51.7	37	38.6	71	43.0	41	47.2	64
3	H009/3	269	6 ->15 μ	184	89.7	86	89.7	148	89.7	78	89.7	100
7	H010/7	266	2 - 5 μ	140	68.3	110	114.7	90	54.6	81	93.2	70
8	H011/8	61	0.5- 2 μ	14	6.8	-	-	16	9.7	-	-	40

Here sample No. 3 was arbitrarily chosen as 89.7% quartz, the average of the responses obtained in Table II, 4. Responses for the other samples are then worked out with respect to that for No. 3. It is apparent that for both D. T. A. and X-ray analysis the response decreases as particle size decreases. This matter will also be examined in more detail in the following chapter, and a hypotheses will be advanced for the observed discrepancies between D. T. A. and X-ray analysis results for quartz.

/(f) Conclusion ...

Author Craig D K
Name of thesis Determination of the Percentage of Quartz (S1O2) in samples of Mine Dust by the Method of Differential Thermal Analysis 1959

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