

A STUDY OF DIFFERENTIAL THERMAL ANALYSIS
UTILIZING STATISTICAL EXPERIMENTAL DESIGN

BY

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in

Ceramic Engineering

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ABSTRACT

A study was made of the problem of differential thermal analysis utilizing statistical experimental design. A factorial type of design was chosen to yield maximum efficiency of the data obtained. "Lakeland", Florida Kaolin of commercial whiteware grade was selected because of its relative purity. Due to the large number of samples to be analyzed, it was found necessary to choose a commercial brand rather than a very pure kaolinite which was found impossible to obtain in sufficient quantity.

A statistical 2 x 3 x 3 factorial design was used and the calculations incident thereto made in detail. Data, which to the eye appeared erratic, was found to be acceptable within the probability limits of error of the experiment. The effects of two different furnaces, three rates of heating, and three densities of the samples were analyzed to determine the area under the endothermic reaction peaks as measured by Cr-Al differential thermocouples. It was found that densities and rates both yielded highly significant values and that the interactions of the two showed significant results when examined by a distribution of "F"¹. These effects were analyzed for the regressions therein and were found to be significant in the linear trends only. From this indication it would seem evident that the optimum density and rate of heating had not been reached in the limits of this experiment. Replications and furnaces did not yield significant results when tested by "F" distribution. From this it was deduced that the experiment could be conducted with sufficient precision under the present procedure and conditions and reproductibility obtained irrespective of the furnace used. Examination of the data will

(2)

indicate that the level of the two furnaces is different, but the trends within are the same in both cases.

A certain amount of erratic data were obtained due to a lag in heating rates and this difficulty will be discussed.

I INTRODUCTION

Two of the newer concepts and ideas in the research field were combined in this study. Differential thermal analysis, while not new in the strictest sense, has only recently had any real significance attached to it. This has been brought about by the efforts of several of the Government agencies² and others in attempts to introduce this method as a reliable method of analysis of the clay minerals. These minerals within each group in the following table each have an identical chemical analysis and empirical formula, yet they differ to rather large extents in crystalline and physical properties.

The Clay Minerals ³			
Kaolinitic $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$	Montmorillonitic $(\text{Mg} \cdot \text{Ca})\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 5\text{SiO}_2 \cdot n\text{H}_2\text{O}$	Micaceous $\text{K}_2\text{O} \cdot 3\text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2$	Hydrous Alumina $\text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$
Kaolinite	Montmorillonite-nontronite	Muscovite	Gibbsite
Dickite	Beidellite	Sericite	Diaspore
Nacrite	Talc	Potash Clay	
Halloysite	Pyrophyllite		
Allophane			

The problem of identification of almost any of the minerals is complicated by the fact that their particle sizes are so minute that they resist identification by the petrographic microscope and X-ray diffraction. In the microscopic techniques little success has been had in getting the optical properties of the minerals. The same has been true with X-ray diffraction patterns; the particles are so minute that they tend to give bands instead of clearly defined lines.⁴ Analysis by the electron microscope has proven

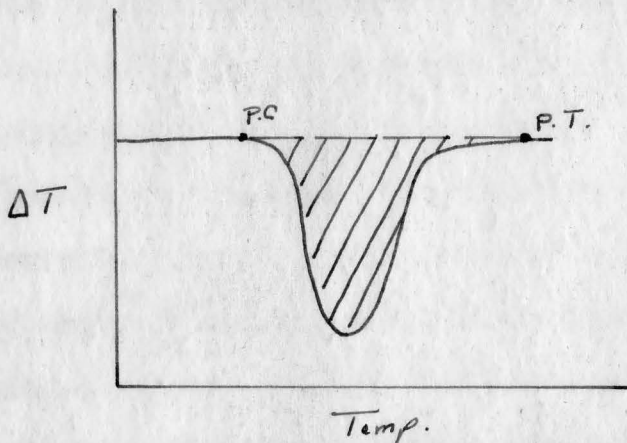
to be a fertile field but the comparatively high cost of this instrument has prevented its widespread use.

Taking these factors into consideration, the need of some supplemental method of analysis was seen evident. Differential thermal analysis has offered considerable promise in filling this gap. By its very nature it cannot be used to supplant any of the recognized means of analysis, but it does offer a powerful tool within its limitations to determine the mineral phases present as well as the relative amounts of each. Norton⁵ has done some of the leading work in this field. It has been known for some time that the chemically combined water in the clay minerals is driven off on heating at some temperature just below dull red heat. Chemical analyses have shown that these temperatures vary depending on the mineral being analyzed. It is this peculiar phenomenon that makes differential thermal analysis useful.

Differential thermal analysis may be explained basically in this manner. Materials to which heat is applied at a uniform rate will increase in body temperature at some uniform rate depending on the specific heat⁶ and the absorption characteristics of the substance. If at some temperature an inversion is undergone, this body temperature will increase over that of the atmosphere if the reaction is exothermic in nature; or if endothermic, the temperature will be below that of the medium. Such is the case upon dehydration due to absorption of the heat of decomposition of the mineral. If the difference between the temperature of the body and the temperature of a reference specimen, which undergoes no reaction at that temperature, is measured with a differential

(5)

thermocouple, as described by Wood and Cork,⁷ and the resulting plot made of ΔT vs. T , a curve of the type shown below will be obtained.



By studying the locations of the endothermic peaks, created on heating due to the absorption of the heat of decomposition by the sample, it is possible to identify each of the mineral phases present in a sample. It has been found by most all workers in this field that the area under the endothermic peaks when plotted as Δ Temperature vs. Temperature is a function of the molecular activity of the mineral, this activity being characteristic of only one mineral in the group.⁸

The second concept which has come into prominence in the last five to ten years concerns statistical and experimental design as applied to research problems in all fields of study. Originally statistical design was used almost entirely on agricultural experiments. During the past decade, however, due to a large amount of pioneering work by the Bell Telephone Laboratories, Radio Corporation of America, and some of the outstanding statisticians in the world, statistical design has been applied and used successfully in most all phases of research work.⁹ More and more companies and research organizations are utilizing these designs as a means of

obtaining highly efficient experiments. It has been found that results obtained from statistically designed experiments are generally more reliable than those obtained from experiments not utilizing this method. One of the greatest features of the statistical analysis of the data is that the final interpretation rests in the comparison of a series of numbers to certain probability functions. This relieves the experimenter or the person reviewing the work of a large amount of opinionating in the interpretation of the results. This is not meant to carry the connotation that statistics is the universal solution and can replace fundamental reasoning entirely. It does mean, however, that statistics when properly used is a powerful tool in the hands of the researcher, especially in untried territories.

II REVIEW OF LITERATURE

The theory of differential thermal analysis is not completely new in the realm of ceramic research. As early as 1887, Le Chatlier (Fr.)¹⁰ suggested that an analysis of this type might prove beneficial in the study of the minerals of the hydrous alumino or hydrous alumino-silica groups. A few years later H. Wallach (Fr.)¹¹ showed that the method worked with some precision on these minerals. After this work the method was forgotten to a large extent until the past ten years. In a period since 1939 the study by this method has been revived and considerable work done. To a large extent the work was uncorrelated between the different experimenters until 1945 when the Bureau of Mines published a consolidated paper on the works of Speil, Berkelhamer, Pask, and Davies¹². These papers cover the investigations of a large number of the commercial clays found in the world. In addition, the work of Speil proposes a furnace design and temperature control arrangement for a very nominal cost. This paper by Speil was unobtainable until after the furnaces used in this thesis had been constructed; consequently, they are not as efficient as the ones proposed. Norton¹³ has done most of the preliminary work in the exploration of the field with a view of using the process as a method of quantitative analysis. He has found that with care an analysis can be determined to limits of $\pm 2\%$.

All clays undergo endothermic reactions between 100°C. and 675°C., the location of the reaction and the heat of reaction varying from mineral to mineral.¹⁴ Exothermic reactions at approximately 980°C. yield 10-40 cal/gm for most minerals. A more complete study has further proven to observers

that clays are complex minerals and mineral aggregates, and exhibit very complex and interesting thermal characteristics upon heating¹⁵.

As to a specific explanation of the actual state of the molecules at the temperatures of the reactions, there is much disagreement. Mellor and Holdcroft¹⁶ claim that at approximately 500°C. kaolinite decomposes to alumina, silica, and water. The alumina and silica recombine at 1200°C. to form sillimanite. The first reaction mentioned has been rather widely argued, but little concrete proof of this to substantiate or refute the hypothesis has been offered. Most researchers agree, however, that the alumina and silica recombine in molecular ratios at approximately 980°C. as evidenced by the rapid exothermic reaction occurring at that temperature. Petrographic analysis of the resulting mineral has been proven to be mullite¹⁷.

Inasmuch as the author did not make any determinations or investigation along these lines, he can do nothing but relay the theories proposed and express no opinion of his own. Actually it was not necessary in the course of this investigation to use any of these assumptions as to whether the entire molecule was decomposed or just the chemically combined water driven off. The inversion at 980°C. was assumed to be mullitization of the alumina and silica, but for an elementary study such as was made here, it was not important whether the mullitization occurred by the combination of alumina and silica separately or the inversion of alumino-bisilicate.

The recommended construction of the nickel specimen holder varied from one researcher to the next. Norton¹⁸ used a block with two holes of equal size drilled therein. One of the holes was for the test specimen while the other was for an inert alumina (Al_2O_3) reference sample. The differential

thermocouple was inserted in both, and the temperature of the alumina reference sample used as the temperature of the furnace for plotting purposes. Grim¹⁹ found that by taking a very pure nickel and measuring the temperature of the block at a point laterally opposite the sample that equally satisfactory results were obtained. This was the method followed in this study.

The mass of the sample has no appreciable effect on the height of the peak value of the reactions as long as the mass of the inert sample is increased a like amount²⁰. This was found to be applicable on a few preliminary analyses using different size samples. For this reason the weight of each sample was kept as nearly constant as possible. Examination of the data relative to densities will show ± 0.02 gm. of variation in the weight of the samples and this amount only in rather rare cases. By using a constant sample weight the elimination of the reference specimen of the same mass as the sample was accomplished.

The means of forming the samples for this study was different from that of Norton and Grim. In their work the samples were formed by hand-packing the mineral being studied into the specimen cavity around the thermocouple. By weighing the specimen holder before and after the sample had been introduced and knowing the volume of the test hole or cavity, the density could be calculated. It was felt by the author that this method was subject to variation of density within the sample, so another scheme was used. In this scheme the samples were formed in a separate press and a hole slightly smaller than the thermocouple bead drilled therein. Samples, when formed in this manner, were much more dense than could be obtained by hand-packing

and also more uniform, both in size and density throughout. (See Experimental Procedure).

Berkelhamer²¹ and Norton²² used an automatic recording device of their own design for all determinations. This equipment is rather bulky and cumbersome and, inasmuch as it operated at intermittent periods of time, left a rather large amount of interpolation to be done in completing and plotting the curves. A much more satisfactory arrangement would be to use a continuous electronic recorder as was done by Kerr²³ in his work along these lines. Dr. Kerr uses a multiple setup of apparatus in such a way as to analyze six samples simultaneously. This arrangement is to be commended to anyone embarking upon an investigation of this type on a large scale.

Most of the research in this field has proven that the rate of heating and the density of the sample affect the height, width, and consequently the area of the reaction peaks. Before any comparisons can be made in a study of this nature the rate of heating, mass of the sample, density of the sample, and similarity of procedure must be established. It is felt that here is the greatest need at present in studies of this nature. Once this is effected, it is also felt that the method will prove satisfactory for commercial usage in supplementing the knowledge already existing relative to the nature of the minerals being sold or used by the ceramic industry.

III EXPERIMENTAL PROCEDURE

A. Equipment

Two furnaces, similar but not exactly identical in design, were constructed as shown in Figure 1, Page 16a. These furnaces were made slightly different in order to test the hypothesis; that there was no difference between furnaces as used in this problem. The differential sample block was machined from 99.9% pure hot-rolled nickel from a specimen obtained from the International Nickel Company, New York. (See Fig. 1). The differential thermocouples were made from 22 gage Chromel-Alumel wire and were spot-welded at the tips by means of a soft carbon electrode connected in series with an 80 ohm variable rheostat to a 110 volt ac source. The size of the bead formed was controlled so that the thermocouple would just slide into the nickel block and into the sample. According to Berkelhamer²⁴, the size and location of the thermocouple bead affects the base line characteristics of the sample. A model 2241 Wheelco "Capacitrol" and relay, when used in series, with model 60-A "Variac" (General Radio Corporation) were used to control the temperature rate of heating in the furnaces.



General View of
Equipment

B. Preparation of the Samples

The samples of "Lakeland" Florida Kaolin were made by weighing out 1.80 grams of the material and pressing this into a small pellet or cylinder by means of the press shown in Fig. 2, and a laboratory size Riehle Testing Unit. After the samples were formed at loads of 700 lbs., 850 lbs., and 1000 lbs. pressure, they were reweighed and the volumes determined in a mercury volumeter according to a method proposed by Dear²⁵. The samples then had a 1/16" hole drilled to the center from a point midway between the bases. This hole was just under-size for the thermocouple bead, thus insuring intimate contact between the material and the thermocouple.

A model 2401 "Spotlight" Galvanometer, manufactured by Rubicon Company, Philadelphia, and having internal resistance of 24 ohms and a sensitivity of 20 μ v per millimeter was used to measure the developed emf of the differential thermocouple. A Leeds and Northrup Portable, Double-Range, No. 8657 Potentiometer was used to determine the temperature of the nickel block.

The sample was placed in the drilled cavity of the nickel test block and the differential thermocouple inserted, one end in the test specimen, and the other end to a corresponding point of the nickel block. The temperature thermocouple was inserted in a small hole just below the differential thermocouple in the nickel block. A nickel cover of the same thickness as the walls surrounding the test specimen was placed over the block and the entire assembly inserted half-way through the furnace. The thermocouple leads were connected to the potentiometer and the differential thermocouple leads connected through a 25 ohm precision-wound series resistor to the Spotlight Galvanometer. Each of these instruments had been previously zero-set and standardized.

C. Development of the Rate of Heating

The desired time-rate cam was aligned on the "Capacitrol", the furnace current adjusted to 24 amperes with the "Variac", and the current turned on. By measuring the temperature of the nickel block with the potentiometer and checking the temperature-time schedule, the rate of heating of the furnace was determined. An effort was made to smooth out this rate by adjustment of the voltage to the lowest value which would permit the rate of heating desired. Once the heating was commenced, the rate of rise was controlled by the "Capacitrol." By pre-setting the L & N Potentiometer at 20°C. intervals, commencing at 100°C., the time at which the nickel test block reached the set value and the galvanometer deflection at that time could be determined. Errors in readings were found to be within ± 7 seconds for any individual. An attempt was made to record the exact temperature of the peaks of the reactions, but due to the rapid movement of the galvanometer, especially with the exothermic reaction peaks upon mullitization of the Al_2O_3 and SiO_2 , this was not as precise a determination as the other readings. For this reason the area of the exothermic reactions were not studied. An electronic continuous recorder would be necessary to accurately measure these reactions.

Samples made to densities of 3.39 gms/cc, 3.67 gms/cc, and 3.96 gms/cc were analyzed in each furnace and at the rates of 9°C/min., 12°C/min., and 15°C/min. A complete replication of the problem was made in order to secure a valid error for the tests of significance in the statistical analysis of variance. It should be noted here that the values of densities and rates of heating increased in equal increments. These were established in this manner to enable analysis to be made utilizing a table of orthogonal polynomials

to determine the significance of any trends or regressions that may have been present in the course of the investigation.

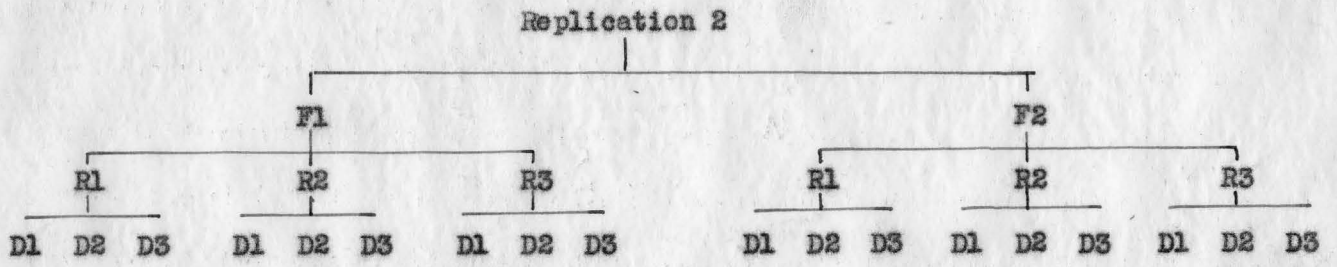
After the endothermic reactions had gone to completion, the leads of the differential thermocouples were interchanged in order that the entire scale of the "Spotlight" Galvanometer might be made available for the reading of the exothermic reactions which developed at approximately 980°C. In addition, a 50 ohm precision-wound resistor was substituted for the 25 ohm resistor used for endothermic reactions. In the cases when the endothermic reactions exceeded 100 mm scale deflection, the 50 ohm resistor was inserted in place of the 25 ohm resistor to move the readings back within the limits of the scale (100 mm). Conversion to the 25 ohm resistor basis was then made by using the calibration curves shown in Section V, Results.

D. Plotting of the Curves

Curves were plotted with the data obtained using galvanometer scale readings vs. temperature (°C.). From the calibration curves, it may be seen that the galvanometer deflections vary in a linear manner with the temperature differentials. The areas under the endothermic peaks from point of curvature to point of tangency were measured by means of a planimeter. A series of three readings were made with the planimeter and the average areas used in the statistical computations.

E. General Considerations

Before any of the samples were analyzed the circuits through the differential and temperature thermocouples were checked by a Weston Type 30 Ohmmeter. It was found to be most difficult to locate a broken lead or thermocouple



> to Chronotrol
 > to Variac

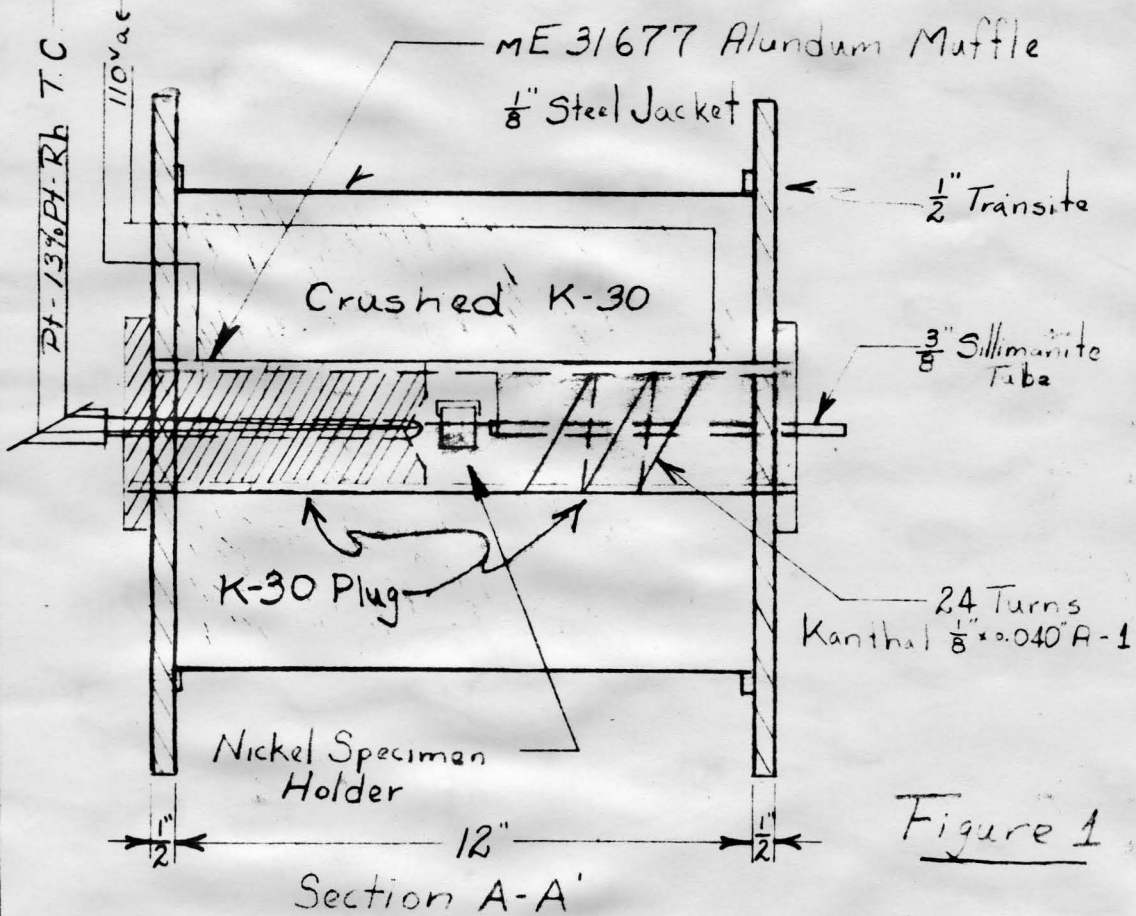
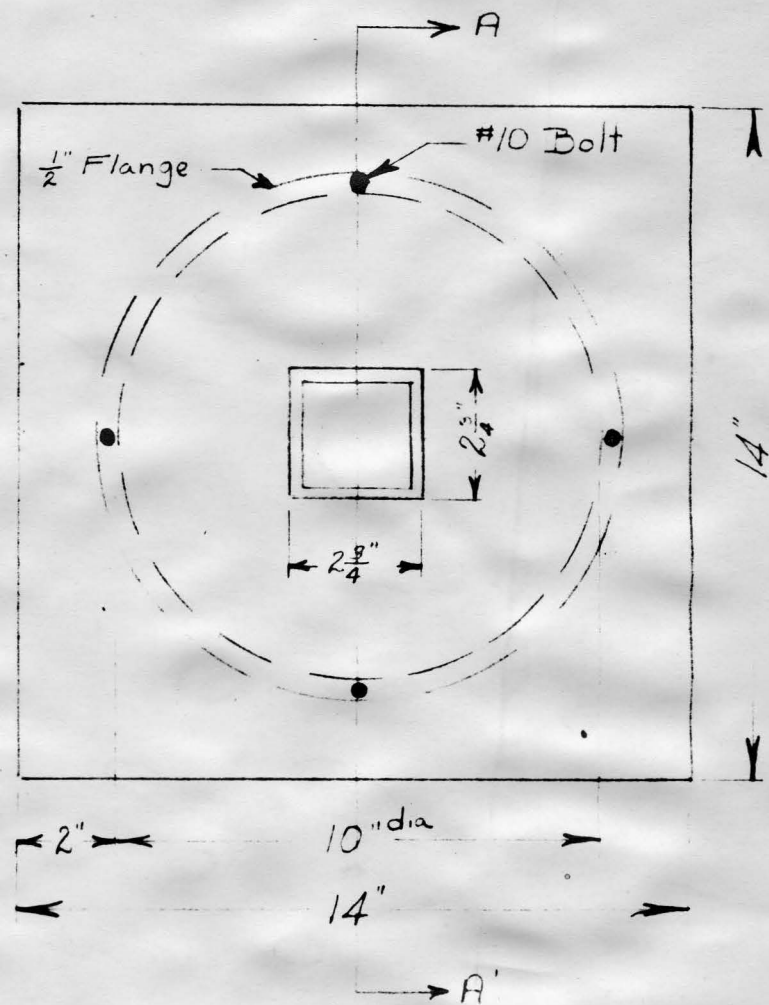
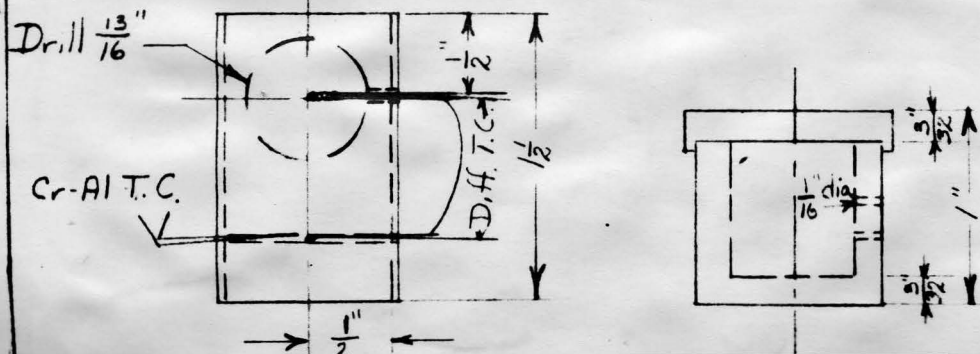


Figure 1



Front View

Nickel Specimen Holder Detail



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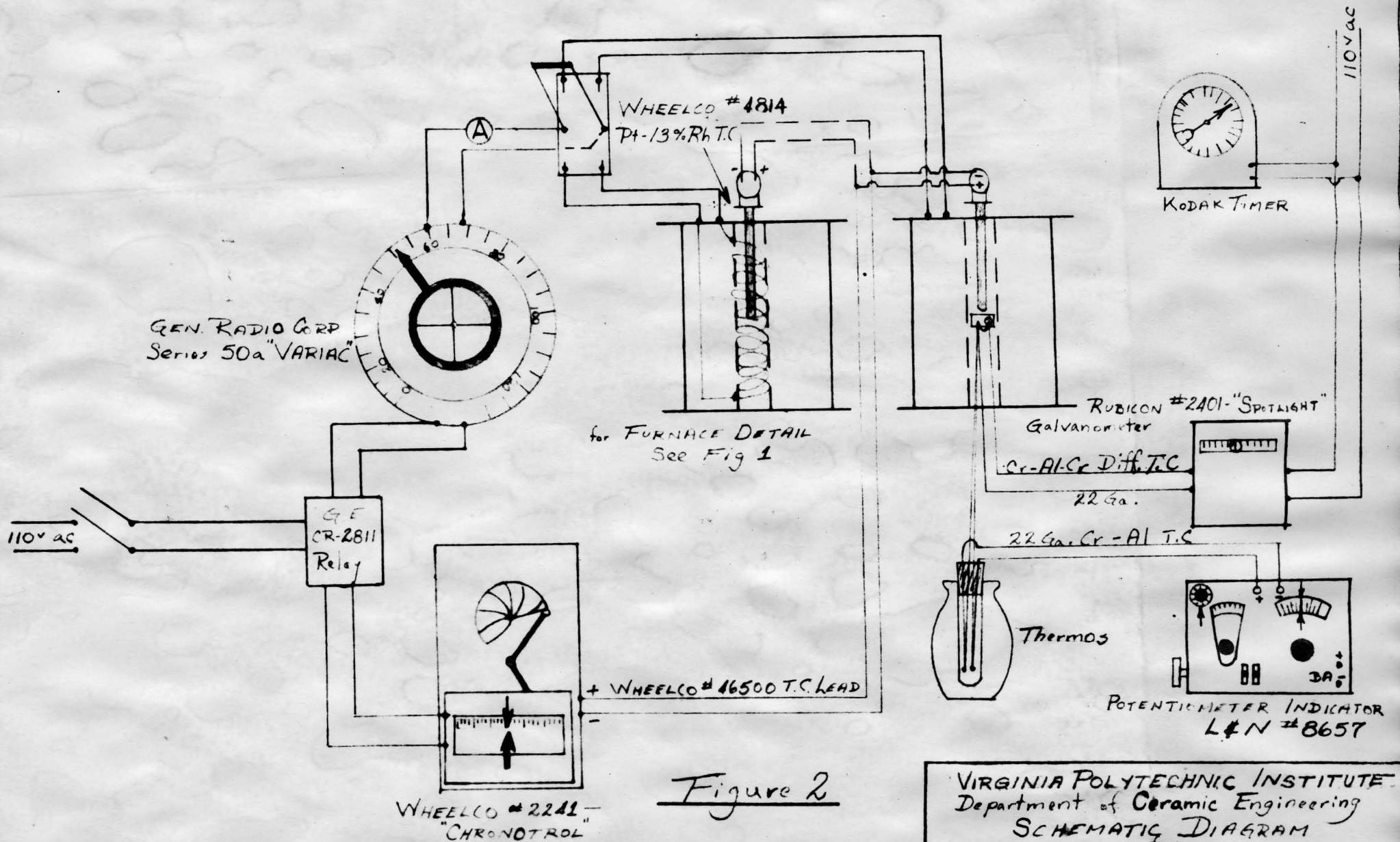


Figure 2

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IV OBSERVATIONS AND DATA

The following tables of data were obtained during the course of this investigation and are reproduced here with all comment being reserved for "Discussion of Results."

Table #1

Replication 1

Rate 1

Furnace 1

Density 1

<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm.)</u>	<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm.)</u>
000	100		- 7	49-15	60		- 6
1-20	20		- 7	50-45	80		-10
3-15	40		- 6	52-15	600	9.5	-14
5-00	60		- 6	54-00	20		-16
6-30	80		- 5	55-30	40		-16
8-00	200	12	- 4	57-30	60		-13
9-45	20		- 4	59-30	80		- 8
11-30	40		- 3	64-00	700	9.3	- 5
13-30	60		- 2	72-00	20		- 5
15-45	80		- 1	76-00	40		- 4
19-00	300	10.5	0	78-15	60		- 4
22-30	20		- 1	80-00	80		- 4
27-00	40		- 1	81-30	800	8.6	- 4
30-30	60		- 1	83-00	20		- 3
33-15	80		- 2	84-30	40		- 3
35-45	400	8.5	- 2	86-00	60		- 3
38-00	20		- 2	87-00	80		- 3
39-45	40		- 2	88-30	900	9	- 3
41-30	60		- 3		20		
43-15	80		- 3		40		
45-00	500	8.9	- 3	94-30	60		- 2
46-30	20		- 4	95-30	65		62
48-00	40		- 5	97-30	1000	9.2	0

(18)

Table #2

Replication 1

Rate 1

Furnace 1

Density 2

<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm.)</u>	<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm.)</u>
000	100	- 9	- 9	52-00	60		-24
2-15	20		-11	54-00	80		-34
4-30	40		-11	56-15	600	8.9	-51
6-45	60		-10	58-30	20		-59
9-00	80		- 9	60-45	40		-54
11-00	200	9.1	- 8	62-45	60		-38
13-00	20		- 8	64-30	80		-21
15-15	40		- 8	66-00	700	9.1	-14
17-15	60		- 8	67-30	20		-13
19-15	80		- 7	69-00	40		-11
21-30	300	9.3	- 7	70-00	60		-11
23-45	20		- 6	71-30	80		- 9
26-00	40		- 6	73-15	800	9.6	- 8
28-45	60		- 6	75-45	20		- 5
31-30	80		- 6	79-15	40		- 5
34-30	400	8.7	- 7	81-30	60		- 6
37-30	20		- 9	83-30	80		- 6
39-45	40		-10	85-00	900	9.4	- 6
42-00	60		-11		20		
44-00	80		-12		40		
46-00	500	8.7	-14	89-15	60		0
48-00	20		-15	90-00	70		130
50-00	40		-17	91-00	80		12
				94-00	1000	9.5	0

(19)

Table #3

Replication 1				Rate 1			
Furnace 1				Density 3			
<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm.)</u>	<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm.)</u>
000	100		-21	51-30	600	9.7	-80
2-30	20		-24	54-30	20		-94
5-00	40		-25	57-15	40		-76
7-15	60		-24	59-30	60		-43
9-45	80		-24	61-30	80		-30
11-45	200	8.5	-23	63-15	700	9.5	-28
14-00	20		-22	65-00	20		-24
16-10	40		-21	67-30	40		-20
18-30	60		-19	71-00	60		-18
21-00	80		-17	73-45	80		-20
24-00	300	8.4	-17	75-45	800	9.2	-21
27-30	20		-21	77-30	20		-22
30-00	40		-24	79-15	40		-19
31-45	60		-27	80-30	60		-18
33-30	80		-31	82-45	80		-14
35-00	400	8.6	-34	88-00	900	9.1	-11
36-30	20		-35		20		
38-00	40		-35		40		
39-15	60		-36	95-00	60		-70
40-30	80		-37	95-45	70		160
42-00	500	9.8	-38	96-30	80		62
43-30	20		-39	98-30	1000	9.1	0
45-00	40		-41				
47-00	60		-46				
49-15	80		-60				

(20)

Table #4

Replication 1

Rate 2

Furnace 1

Density 1

<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm.)</u>	<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm.)</u>
000	100		- 7	41-30	600	12	-18
2-00	20		- 7	44-00	20		-32
3-45	40		- 7	46-15	40		-44
5-15	60		- 7	48-30	60		-26
6-45	80		- 6	50-00	80		-15
8-15	200	12.2	- 5	51-30	700	11.7	-14
10-00	20		- 5	52-45	20		-13
11-30	40		- 4	54-00	40		-13
13-00	60		- 3	55-15	60		-12
14-45	80		- 2	56-15	80		-10
17-00	300	11.8	- 1	57-30	800	12.1	-8
19-30	20		0	58-45	20		- 3
21-45	40		- 1	60-00	40		- 3
24-00	60		- 1	62-00	60		- 4
25-45	80		- 2	64-45	80		- 4
27-30	400	10.9	- 2	67-15	900	12	- 4
29-00	20		- 2		20		
30-30	40		- 3		40		
31-45	60		- 3	71-45	60		- 3
33-00	80		- 3	72-30	70		0
34-15	500	11.7	- 4		75		64
35-30	20		- 4	73-15	80		4
36-45	40		- 4	74-40	1000	12.0	0
38-15	60		- 4				
39-30	80		- 6				

(21)

Table #5

Replication 1 Rate 2
Furnace 1 Density 2

<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm.)</u>	<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm.)</u>
000	100		- 9	43-15	600	11.6	-57
1-45	20		-12	45-15	20		-65
4-00	40		-12		30		-66
5-30	60		-12	47-30	40		-61
7-15	80		-11	49-15	60		-45
9-00	200	11.1	-11	51-00	80		-24
10-30	20		-10	52-30	700	11.4	-17
12-00	40		-10	54-00	20		-15
13-45	60		-10	55-15	40		-14
15-15	80		-10	56-30	60		-14
17-00	300	11.8	-10	57-45	80		-13
18-30	20		-10	59-00	800	11.8	-13
20-45	40		-10	60-15	20		-12
22-00	60		-10	61-15	40		-11
23-15	80		-11	62-30	60		-10
24-45	400	12.1	-12	63-45	80		- 9
26-30	20		-12	65-30	900	12.2	- 5
28-00	40		-13		20		
29-30	60		-13		40		
31-15	80		-14	72-45	60		- 3
33-00	500	12.1	-15	73-30	70		0
34-45	20		-15	74-15	80		90
36-30	40		-18	75-30	1000	11.9	0
38-45	60		-24				
41-00	80		-39				

Table #6

Replication 1 Rate 3

Furnace 1 Density 3

<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm).</u>	<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm.)</u>
000	100		-21	32-00	600	15.6	-65
1-00	20		-24	34-00	20		-76
2-30	40		-29	36-30	30		-77
4-00	60		-26	38-30	40		-70
5-15	80		-24	40-00	60		-57
6-45	200	14.8	-23	41-15	80		-40
8-15	20		-23	42-45	700	14.6	-26
9-15	40		-23	44-15	20		-21
10-30	60		-22	45-30	40		-19
11-45	80		-21	46-45	60		-19
13-15	300	15.1	-20	48-00	80		-18
14-30	20		-18	49-15	800	14.6	-17
16-00	40		-17	50-30	20		-16
17-45	60		-19	51-30	40		-16
19-00	80		-20	52-30	60		-16
20-30	400	14.6	-22	53-45	80		-16
21-45	20		-23		900	14.9	-15
22-45	40		-25		20		
23-45	60		-28		40		
25-00	80		-28	57-30	60		- 5
26-15	500	15.2	-29	58-00	70		- 3
27-15	20		-31	58-45	80		94
28-15	40		-34		90		0
29-30	60		-37	60-00	1000	15.0	0
30-30	80		-48				

Table #7

Replication 1 Rate 3

Furnace 1 Density 1

<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm.)</u>	<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm.)</u>
000	100						
1-20	20		- 4	36-00	600	13.9	-64
2-30	40		- 1	37-15	20		-85
3-45	60		- 2	38-30	40		-95
4-45	80		- 2		45		-100
5-45	200	18.3	- 3	40-00	60		-85
6-45	20		- 3	41-00	80		-73
7-45	40		- 3	42-00	700	14.5	-47
8-45	60		- 3	43-15	20		-26
10-00	80		- 7	44-30	40		-20
11-30	300	19.0	-10	45-30	60		-17
12-45	20		-12	46-45	80		-15
14-15	40		-13	47-00	800	14.9	-14
16-15	60		-14	48-15	20		-13
18-45	80		-14	50-30	40		-13
21-15	400	14.4	-14	52-00	60		-12
23-30	20		-15	53-15	80		-12
25-15	40		-17	54-30	900	14.7	-11
27-00	60		-17.5		20		
28-30	80		-19		40		
30-00	500	13.3	-21	58-00	60		0
31-00	20		-23	59-00	80		150
32-15	40		-27	60-00	1000	15.0	0
33-30	60		-34				
34-45	80		-45				

(24)

Table #8

Replication 1 Rate 3

Furnace 1 Density 2

<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm.)</u>	<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm.)</u>
000	100		- 4	33-45	600	14.9	-51
1-30	20		- 9	35-00	20		-62
3-00	40		-11	36-15	40		-66
4-30	60		-11		50		-66
6-00	80		-10	37-30	60		-64
7-15	200	14.0	- 9	38-30	80		-56
8-30	20		- 9	39-30	700	15.2	-45
9-45	40		- 9	40-30	20		-29
10-45	60		- 9	41-30	40		-19
11-45	80		-10	42-30	60		-15
12-45	300	15.7	-10	43-30	80		-12
14-00	20		-10	44-30	800	15.7	-10
15-00	40		-11	45-45	20		- 9
16-00	60		-12	47-15	40		- 7
17-00	80		-13	49-00	60		- 6
18-15	400	16.5	-13	51-00	80		- 6
19-30	20		-13	53-00	900	15.0	- 6
21-00	40		-12		20		
22-45	60		-12		40		
24-30	80		-12	57-00	60		0
26-15	500	15.3	-12		70		110
28-00	20		-14	58-00	80		21
29-30	40		-17		95		0
31-00	60		-21		1000	15.1	0
32-30	80		-34				

Table #9

Replication 1

Rate 2

Furnace 1

Density 3

<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm.)</u>	<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm.)</u>
000	40		-28	40-45	600	11.3	-91
1-40	60		-28	42-00	20		-84
3-30	80		-27	43-15	40		-97
5-15	200	12.0	-27	44-45	55		-100
7-00	20		-26	45-45	60		-96
8-30	40		-26	47-15	700	11.8	-28
10-10	60		-26	49-00	20		-22
11-45	80		-25	51-00	40		-18
13-30	300	11.9	-24	53-15	60		-17
15-20	20		-22	55-00	80		-17
17-15	40		-20	56-30	800	11.7	-18
19-45	60		-19	58-00	20		-18
22-00	80		-19	59-30	40		-18
24-45	400	10.6	-20	60-30	60		-17
26-45	20		-22	61-45	80		-15
28-45	40		-24	63-15	900	12.0	-12
30-30	60		-25		20		
32-15	80		-28		40		
34-00	500	10.6	-31	70-00	60	(50 ohms)	- 4
35-30	20		-34		70		0
36-45	40		-39		75		77
38-10	60		-48	72-00	80		15
39-30	80		-64	73-00	1000	11.8	0

Table #10

Replication 1 Rate 1

Furnace 2 Density 1

<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm.)</u>	<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm.)</u>
000	100		-14	55-15	600	9.0	-48
2-30	20		-16	57-00	20		-55
4-30	40		-17	58-45	40		-56
6-45	60		-16	60-30	60		-47
9-00	80		-16	62-45	80		-21
11-15	200	8.9	-14	66-00	700	9.1	-5
13-45	20		-11	69-30	20		-4
16-30	40		-9	72-30	40		-4
19-45	60		-9	74-30	60		-6
22-30	80		-9	76-30	80		-7
25-00	300	8.0	-10	78-00	800	9.0	-8
27-30	20		-11	79-45	20		-7
29-45	40		-12	81-30	40		-6
31-45	60		-14	83-30	60		-6
33-30	80		-16	85-00	80		-6
34-45	400	8.7	-17	87-00	900	9.2	-4
36-15	20		-16		20		
37-45	40		-15		40		
39-30	60		-13	96-30	60		4
41-45	80		-10	98-00	70		83
44-30	500	9.0	-8	98-30	80		10
47-15	20		-10	100-00	1000	9.0	0
49-30	40		-14				
51-45	60		-20				
53-30	80		-31				

(27)

Table #11

Replication 1 Rate 1

Furnace 2 Density 2

<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm.)</u>	<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm.)</u>
000	100		-13	58-15	600	8.6	-59
2-00	20		-17	60-20	20		-76
4-00	40		-20	62-15	40		-76
6-00	60		-19	64-00	60		-55
7-15	80		-18	65-15	80		-31
9-00	200	11.1	-16	66-30	700	9.1	-22
11-00	20		-14	68-00	20		-18
13-15	40		-11	69-30	40		-15
15-30	60		- 9	71-15	60		-11
18-15	80		- 8	73-30	80		- 5
21-15	300	9.4	- 9	77-45	800	9.0	- 2
24-15	20		- 9	80-00	20		- 3
26-45	40		-10	82-10	40		- 4
29-15	60		-11	84-30	60		- 4
31-30	80		-14	86-45	80		- 4
33-30	400	9.0	-15	88-45	900	9.00	- 5
35-15	20		-17		20		
37-00	40		-18		40		
38-45	60		-18	94-15	60		- 3
40-15	80		-18	96-15	70		130
42-30	500	9.4	-18	98-15	80		52
44-30	20		-18	100-15	1000	9.0	0
47-15	40		-17				
52-00	60		-21				
55-45	80		-35				

(28)

Table #12

Replication 1 Rate 1
Furnace 2 Density 3

<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm.)</u>	<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C./Min.</u>	<u>Diff. Galv.</u> <u>(mm.)</u>
000	100		-17	56-00	600	8.9	-44
2-10	20		-20	58-30	20		-51
4-15	40		-21		30		-51
6-15	60		-20	61-00	40		-44
8-15	80		-19	63-00	60		-30
10-00	200	10.0	-18	64-45	80		-16
11-30	20		-17	66-15	700	9.0	-12
13-15	40		-16	68-00	20		-10
15-00	60		-13	69-45	40		- 8
17-00	80		-11	71-45	60		- 6
19-45	300	10.1	- 8	73-45	80		- 4
22-30	20		- 7	76-15	800	9.1	- 2
25-15	40		- 7	79-15	20		- 1
28-15	60		- 8	82-45	40		- 2
30-45	80		- 9	85-30	60		- 2
33-15	400	9.0	-10	87-45	80		- 3
35-00	20		-11	89-30	900	9.0	- 3
37-15	40		-11		20		
39-00	60		-11		40		
41-30	80		-11	95-30	60		0
43-45	500	9.1	-12	96-30	70		10
46-00	20		-12		75		120
48-15	40		-15	97-30	80		8
51-00	60		-21	99-30	1000	8.9	0
53-45	80		-31				

Table #13

Replication 1 Rate 2

Furnace 2 Density 1

<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm.)</u>	<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm.)</u>
000	100		-11	41-45	600	12.0	-25
1-30	20		-13	43-30	20		-23
3-20	40		-14	45-30	40		-27
4-45	60		-13	47-15	60		-22
6-30	80		-11	48-45	80		-15
8-00	200	12.5	-10	50-15	700	11.9	- 6
9-30	20		- 6	51-45	20		- 4
11-15	40		- 4	53-00	40		- 3
13-15	60		- 3	54-00	60		- 3
15-15	80		- 3	55-15	80		- 2
17-15	300	11.6	- 2	56-30	800	12.6	- 2
19-30	20		- 2	57-45	20		- 2
21-30	40		- 2	59-15	40		- 1
23-15	60		- 3	60-30	60		0
24-45	80		- 4	62-30	80		- 2
26-15	400	11.4	- 5	65-15	900	12.2	- 2
27-45	20		- 6		20		
29-00	40		- 6		40		
30-15	60		- 6	73-00	60		- 3
31-45	80		- 6	74-00	70		76
33-00	500	12.1	- 7	75-00	80		12
34-30	20		- 7	76-00	1000	11.9	0
36-15	40		- 8				
38-00	60		-10				
40-00	80		-16				

(30)

Table #14

Replication 1

Rate 2

Furnace 2

Density 2

<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm.)</u>	<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C./Min.</u>	<u>Diff. Galv.</u> <u>(mm.)</u>
000	100		-21	41-15	600	12.1	-46
1-00	20		-24	43-30	20		-55
3-45	40		-27	45-15	40		-56
5-30	60		-26	47-15	60		-49
7-15	80		-26	48-30	80		-38
8-45	200	11.4	-25	50-00	700	12.0	-22
10-30	20		-25	51-30	20		-16
11-45	40		-24	52-45	40		-14
13-00	60		-24	54-15	60		-10
14-20	80		-22	55-30	80		-9
15-45	300	12.6	-20	57-15	800	12.2	-8
17-00	20		-18	58-45	20		-6
19-15	40		-15	60-45	40		-5
21-15	60		-13	62-30	60		-5
23-15	80		-13	64-30	80		-5
25-15	400	11.9	-13	66-15	900	12.0	-5
27-15	20		-14		20		
29-00	40		-14		40		
30-45	60		-16	70-45	60		-5
32-30	80		-17	71-30	70		-2
34-00	500	11.8	-19	72-15	80		130
35-30	20		-20		90		32
36-45	40		-22	73-30	1000	12.2	0
38-00	60		-25				
39-30	80		-32				

(31)

Table #15

Replication 1 Rate 2

Furnace 2 Density 3

<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm.)</u>	<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm.)</u>
000	100		-17	41-15	600	12.1	-80
1-40	20		-20	42-15	20		-100
3-40	40		-21	43-30	40		-120
5-30	60		-20	44-45	60		-120
7-15	80		-19	46-00	80		-86
8-50	200	11.8	-19	47-30	700	12.6	-36
10-30	20		-19	50-00	20		-16
12-00	40		-19	53-30	40		-14
13-30	60		-19	56-15	60	11	-13
15-00	80		-19	58-30	80		-15
16-40	300	12.0	-19	60-00	800	11.7	-16
18-15	20		-19	61-20	20		-18
19-45	40		-18	62-30	40		-18
21-45	60		-16	63-40	60		-19
24-30	80		-15	64-45	80		-19
26-00	400		-15	65-45	900	12.1	-18
28-00	20		-16		20		
30-00	40		-18		40		
31-50	60		-20	69-30	60		- 5
33-30	80		-22	70-45	70		115
35-00	500	11.4	-26	72-30	80		47
36-20	20		-29	74-15	1000	12.1	0
37-40	40		-33				
38-50	60		-42				
40-00	80		-56				

(32)

Table #16

Replication 1 Rate 3

Furnace 2 Density 1

Time Min.-Sec.	Temp. °C.	dH °C/Min.	Diff. Galv. (mm.)	Time Min.-Sec.	Temp. °C.	dH °C/Min.	Diff. Galv. (mm.)
000	100		- 3	35-30	600	14.1	-42
1-15	20		- 6	36-30	20		-55
1-30	40		-11	37-45	40		-61
3-45	60		-12	39-00	60		-61
5-00	80		-11	40-00	80		-58
6-15	200	16.2	-11	41-00	700	14.6	-52
7-15	20		-11	41-45	20		-39
8-15	40		-11	42-45	40		-31
9-15	60		-11	43-45	60		-23
10-15	80		-11	44-30	80		-20
11-30	300	17.3	-11	45-30	800	15.3	-19
12-45	20		-10	46-30	20		-18
14-00	40		-10	47-30	40		-17
15-30	60		- 9	48-30	60		-16
17-30	80		- 9	49-30	80		-16
19-15	400	15.5	- 8	50-30	900	15.8	-15
21-30	20		- 8		20		
23-30	40		-10		40		
25-15	60		-12	54-45	60		- 6
27-00	80		-12		70		58
28-45	500	14.0	-14	56-15	80		
30-15	20		-15	58-45	1000	15.3	0
31-30	40		-17				
33-00	60		-21				
34-15	80		-28				

(33)

Table #17

Replication 1 Rate 3

Furnace 2 Density 2

Time Min.-Sec	Temp. °C	dH °C/Min	Diff. Galv (mm)	Time Min.-Sec	Temp. °C.	dH °C/Min	Diff. Galv (mm)
0-00	100		-17	53-00	600	15.1	-46
1-30	20		-20	54-15	20		-58
2-45	40		-24	56-00	40		-62
4-00	60		-24	57-45	60		-56
5-15	80		-24	59-00	80		-48
6-30	200	15.3	-23	40-15	700	14.9	-36
7-45	20		-22	41-30	20		-23
9-00	40		-21	42-45	40		-19
10-00	60		-20	43-45	60		-19
11-30	80		-18	44-30	80		-18
12-45	300	15.6	-16	45-30	800	15.3	-18
14-15	20		-13	46-15	20		-17
16-00	40		-11	47-00	40		-16
17-45	60		-11	48-00	60		-15
19-15	80		-13	49-15	80		-14
21-00	400	14.3	-14	51-30	900	15.5	-12
22-30	20		-15		20		- 8
23-45	40		-16		40		- 4
25-00	60		-18		60		0
26-15	80		-19		70		94
27-30	500	14.5	-21	56-00	80		0
28-30	20		-22	58-00	1000	15.6	0
29-30	40		-24				
30-30	60		-28				
31-45	80		-34				

(34)

Table #18

Replication 1 Rate 3
Furnace 2 Density 3

Time Min-Sec.	Temp. °C.	dH °C/Min.	Diff. Galv. (mm)	Time Min.-Sec.	Temp. °C.	dH °C/Min.	Diff. Galv. (mm)
0-00	100		-17	33-45	600	14.8	-49
1-30	20		-22	35-00	20		-65
3-00	40		-26	36-30	40		-70
4-30	60		-26		50		-71
5-30	80		-25	37-30	60		-66
6-45	200	14.8	-24	38-45	80		-58
8-00	20		-23	40-00	700	15.0	-45
9-00	40		-23	41-00	20		-26
10-00	60		-22	42-00	40		-16
11-00	80		-22	43-15	60		-13
12-15	300	16.3	-21	44-15	80		-11
13-15	20		-20	45-30	800	15.3	-10
14-15	40		-18	47-00	20		- 9
15-30	60		-17	48-30	40		- 9
16-30	80		-16	50-00	60		- 7
17-45	400	16.0	-15	51-15	80		- 6
19-15	20		-11	52-45	900	15.1	- 6
21-00	40		-10		20		
22-30	60		-10		40		
24-30	80		-10	57-45	60		0
26-15	500	15.2	-11	58-30	70		125
27-45	20		-12	59-00	80		0
29-30	40		-16	60-30	1000	14.9	0
31-00	60		-21				
32-30	80		-29				

Table #19

Replication 2

Rate 1

Furnace 1

Density 1

<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm)</u>	<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm)</u>
0-00	100		-15	55-30	600	9.0	-45
2-10	20		-16	57-45	20		-52
4-20	40		-17	59-45	30		-54
6-30	60		-17	61-45	40		-50
8-30	80		-16	64-00	60		-41
10-40	200	9.4	-15	66-15	80		-23
12-45	20		-14	68-45	700	9.0	- 7
14-00	40		-11	71-00	20		- 6
16-15	60		-10	73-15	40		- 5
18-15	80		- 9	75-30	60		- 5
20-30	300	9.8	- 7	77-30	80		- 6
22-45	20		- 6	79-45	800	9.0	- 5
25-15	40		- 6	82-00	20		- 4
27-45	60		- 5	84-15	40		- 4
30-00	80		- 4	86-45	60		- 5
32-15	400	9.3	- 2	89-00	80		- 5
34-45	20		- 2		900	9.0	- 4
37-15	40		- 4		20		
39-45	60		- 5		40		
42-15	80		- 7	96-15	60	50 ohms	0
44-30	500	9.2	- 9	97-15	70		80
46-45	20		-12	98-30	80		5
49-00	40		-14	100-45	1000	8.9	0
51-15	60		-21				
53-30	80		-28				

(36)

Table #20

Replication 2 Rate 1

Furnace 1 Density 2

<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm)</u>	<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm)</u>
0-00	100		-13	55-45	600	9.0	-55
2-15	20		-12	57-45	20		-68
4-30	40		-12		30		-70
6-45	60		-11	59-45	40		-68
9-00	80		-11	61-30	60		-42
11-15	200	8.9	-10	63-30	80		-25
13-30	20		-10	65-45	700	9.1	-18
15-45	40		-10	68-00	20		-17
18-00	60		-10	70-15	40		-16
20-00	80		-11	72-30	60		-16
22-00	300	9.1	-11	74-45	80		-16
24-15	20		-11	77-00	800	9.1	-15
26-30	40		-11	79-15	20		-13
28-45	60		-11	81-30	40		-11
31-00	80		-11	84-00	60		-7
33-15	400	9.0	-11	86-30	80		-7
35-30	20		-12	88-45	900	9.0	-7
37-45	40		-13		20		
40-15	60		-14		40		
42-45	80		-15	95-15	60		0
45-00	500	8.9	-16	96-15	70		125
47-15	20		-18	97-15	80		15
49-30	40		-20	99-15	1000	9.1	0
51-45	60		-27				
53-45	80		-37				

(37)

Table #21

Replication 2 Rate 1

Furnace 1 Density 3

<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm)</u>	<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm)</u>
0-00	100		-14	56-30	600	8.8	-64
2-30	20		-16	58-45	20		-76
5-00	40		-17		30		-76
7-15	60		-17	61-00	40		-58
9-30	80		-17	63-00	60		-25
11-45	200	8.5	-16	65-00	80		-12
14-00	20		-15	67-00	700	9.0	-10
16-15	40		-14	69-15	20		- 7
18-30	60		-13	71-30	40		- 3
20-45	80		-12	73-45	60		- 3
23-00	300	8.7	-12	76-00	80		- 3
25-00	20		-13	78-15	800	9.0	- 3
27-00	40		-14	30-45	20		- 4
29-00	60		-14	83-15	40		- 4
31-15	80		-15	85-30	60		- 3
33-30	400	8.9	-15	87-45	80		- 4
35-45	20		-15	90-00	900	8.9	- 5
38-00	40		-15		20		
40-15	60		-16		40		
42-30	80		-18	96-45	60		0
45-00	500	8.9	-20	98-00	70		156
47-30	20		-22	99-00	80		50
49-45	40		-24	101-00	1000	8.9	0
52-00	60		-29				
54-15	80		-44				

(38)

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Table #22

Replication 2 Rate 2

Furnace 1 Density 1

<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm)</u>	<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm)</u>
0-00	100		-11	41-30	600	12.0	-38
2-00	20		-14	43-00	20		-46
4-00	40		-15		30		-47
5-45	60		-15	44-45	40		-45
7-30	80		-14	46-30	60		-38
9-00	200	11.1	-14	48-00	80		-24
10-15	20		-13	49-45	700	12.0	-6
12-00	40		-12	51-30	20		-2
13-30	60		-11	53-30	40		-1
15-00	80		-11	55-45	60		0
16-15	300	12.3	-11	57-45	80		0
17-45	20		-9	59-45	800	11.8	0
19-15	40		-7	61-30	20		0
21-15	60		-5	63-00	40		0
23-00	80		-5	64-30	60		0
25-00	400	12.0	-5	65-45	80		0
27-15	20		-5	67-00	900	11.9	0
29-00	40		-6		20		
30-50	60		-6		40		
32-30	80		-7	71-15	60		0
34-15	500	11.8	-8	72-00	70	50 ohms	75
35-45	20		-10	72-45	80		35
37-15	40		-12	74-30	1000	12.1	0
38-45	60		-16				
40-15	80		-26				

(40)

Table #24

Replication 2 \ Rate 2

Furnace 1 Density 3

<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm)</u>	<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm)</u>
0-00	100		-15	43-30	600	11.5	-72
1-50	20		-19	45-00	20		-96
3-45	40		-22	46-15	40		-106
5-30	60		-22	47-30	60		-100
7-00	80		-21	48-45	80		-78
8-45	200	11.4	-20	50-15	700	11.9	-38
10-15	20		-20	51-30	20		-24
11-45	40		-20	53-00	40		-18
13-00	60		-20	54-30	60		-15
14-30	80		-19	56-00	80		-12
15-45	300	12.7	-19	57-45	800	12.1	-9
17-15	20		-18	60-00	20		-7
18-45	40		-16	62-00	40		-6
20-15	60		-16	64-00	60		-6
21-45	80		-16	66-15	80		-6
23-30	400	12.7	-14	68-00	900	11.8	-8
25-30	20		-12		20		
27-30	40		-12		40		
30-30	60		-11	72-00	60	50 ohms	-3
32-30	80		-12	72-30	70		0
35-00	500	11.4	-15	73-15	80		112
37-00	20		-18	74-00	1000		10
38-45	40		-23				
40-15	60		-30				
42-00	80		-47				

(41)

Table #25

Replication 2 Rate 3
Furnace 1 Density 1

<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm)</u>	<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm)</u>
0-00	100		-11	33-00	600	15.1	-52
2-00	20		-14	34-45	20		-68
4-00	40		-16	36-30	40		-75
5-30	60		-16		45		-75
7-00	80		-17	38-30	60		-70
8-30	200	11.8	-17	39-45	80		-51
10-15	20		-17	41-15	700	14.6	-27
11-00	40		-17	42-30	20		-20
12-15	60		-17	43-45	40		-18
13-30	80		-17	45-00	60		-15
14-45	300	13.6	-17	46-00	80		-15
16-00	20		-17	47-00	800	14.9	-14
17-00	40		-16	48-00	20		-13
18-15	60		-18	49-15	40		-11
19-15	80		-19	50-30	60		-10
20-30	400	14.6	-20	51-45	80		- 9
21-30	30		-20	53-00	900	15.1	- 7
22-45	40		-21		20		
23-45	60		-21		40		
25-00	80		-21	57-15	60		- 4
26-00	500	15.4	-22	58-15	70	50 ohms	122
27-15	20		-22	59-30	80		52
28-15	40		-24		90		12
29-45	60		-28	60-45	1000	14.8	0
31-15	80		-36				

(42)

Table #26

Replication 2

Rate 3

Furnace 1

Density 2

<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm)</u>	<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm)</u>
0-00	100		-19	37-00	600	13.5	-66
1-30	20		-23	38-30	20		-81
3-00	40		-26	40-00	40		-85
4-30	60		-26	41-20	60		-76
6-45	80		-25	42-45	80		-57
7-00	200	14.3	-24	44-00	700	13.6	-36
8-20	20		-22	45-15	20		-22
9-30	40		-22	46-30	40		-19
10-45	60		-22	47-45	60		-16
11-45	80		-21	49-00	80		-15
13-00	300	15.3	-20	50-15	800	13.9	-13
14-15	20		-19	51-45	20		-12
15-30	40		-18	53-00	40		-10
16-45	60		-18	54-30	60		-10
18-00	80		-19	56-00	80		-10
19-15	400	15.6	-20	57-20	900	14.0	-10
20-30	20		-20		20		
22-45	40		-19		40		
23-00	60		-18	61-00	60	50 ohms	- 5
24-30	80		-16	61-45	70		0
26-45	500	15.0	-15	62-15	80		0
29-00	20		-16		90		100
31-15	40		-19	63-00	1000	14.3	0
33-30	60		-28				
35-15	80		-43				

(43)

Table #27

Replication 2

Rate 3

Furnace 1

Density 3

<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm)</u>	<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm)</u>
0-00	100		-26	35-15	600	14.2	-80
1-15	20		-29	36-30	20		-101
2-30	40		-34	37-45	40		-111
4-00	60		-33		50		-111
5-15	80		-32	38-00	60		-108
6-30	200	15.4	-31	40-15	80		-88
7-45	20		-30	41-30	700	14.5	-65
8-45	40		-29	42-30	20		-45
10-00	60		-29	43-30	40		-36
11-00	80		-29	44-45	60		-31
12-15	300	16.3	-27	45-45	80		-30
13-30	20		-27	46-45	800	15.0	-27
14-45	40		-26	48-00	20		-25
15-45	60		-26	49-30	40		-21
17-00	80		-27	51-30	60		-19
18-15	400	16.5	-27	53-15	80		-20
19-30	20		-26	54-45	900	14.6	-22
21-00	40		-25		20		
22-30	60		-24		40		
24-30	80		-22	58-30	60	50 ohms	-11
26-30	500	15.0	-23	59-00	70		-10
28-30	20		-25	59-30	80		118
30-30	40		-31		90		35
32-30	60		-43	60-30	1000	14.9	0
34-00	80		-58				

(44)

Table #28

Replication 2

Rate 1

Furnace 2

Density 1

<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm)</u>	<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm)</u>
0-00	100		-20	55-30	600	9.9	-82
2-15	20		-24	58-00	20	50 ohms	-70
4-30	40		-25		30		-72
6-45	60		-24	60-30	40		-69
9-00	80		-23	63-00	60		-52
11-00	200	9.1	-23	64-45	80		-34
13-00	20		-23	66-15	700	9.1	-31
15-00	40		-22	67-45	20		-30
17-00	60		-22	69-30	40		-26
19-00	80		-19	71-15	60		-21
21-30	300	9.3	-18	73-30	80		-15
24-00	20		-16	78-00	800	9.0	-13
27-00	40		-15	80-30	20		-17
30-00	60		-16	82-30	40		-20
32-30	80		-18	83-45	60		-21
35-15	400	8.5	-20	85-15	80		-21
37-30	20		-22	86-30	900	9.2	-20
39-30	40		-24		20	50 ohms	
41-30	60		-24		40		
43-15	80		-28	93-00	60		- 3
45-00	500	8.9	-29	95-30	65		0
47-00	20		-31	95-30	10		81
49-00	40		-35	97-30	80		0
51-00	60		-41	99-30	1000	9.0	0
53-00	80		-56				

(45)

Table #29

Replication 2

Rate 1

Furnace 2

Density 2

<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm)</u>	<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm)</u>
0-00	100		-15	55-45	600	8.9	-62
2-30	20		-18	58-00	20		-78
4-45	40		-21	60-15	40		-79
7-00	60		-21	62-15	60		-57
9-15	80		-20	64-15	80		-34
11-30	200	8.7	-17	66-15	700	9.1	-24
13-45	20		-16	68-15	20		-20
15-45	40		-15	70-30	40		-19
17-45	60		-12	72-45	60		-15
19-45	80		-11	75-00	80		-14
21-45	300	9.2	-12	77-30	800	9.0	-9
24-00	20		-14	80-00	20		-9
26-15	40		-16	82-15	20		-9
28-30	60		-17	84-30	60		-8
31-00	80		-19	86-45	80		-7
33-30	400	9.0	-19	89-00	900	9.0	-6
36-00	20		-19		20		
38-15	40		-19		40		
40-30	60		-19	95-45	60		0
42-45	80		-19		70		130
44-45	500	9.0	-20	98-00	80		55
46-45	20		-21	100-15	1000	8.8	0
49-00	40		-22				
51-15	60		-24				
53-30	80		-38				

Table #30

Replication 2 Rate 1

Furnace 2 Density 3

<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm)</u>	<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm)</u>
0-00	100		-15	55-00	600	9.1	-42
2-15	20		-16	57-30	20		-94
4-30	40		-18		30		-95
6-45	60		-18	60-00	40		-41
8-45	80		-18	62-30	60		-28
10-45	200	9.3	-18	64-45	80		-13
13-00	20		-16	67-00	700	9.0	-11
15-15	40		-15	69-15	20		-10
17-30	60		-14	71-30	40		- 8
20-00	80		-13	73-45	60		- 8
22-30	300	8.9	-10	76-00	80		- 8
24-45	20		-10	78-15	800	8.9	- 8
27-00	40		-10	80-30	20		- 7
29-15	60		-10	82-45	40		- 6
31-30	80		-10	84-45	60		- 6
33-30	400	9.0	- 9	86-45	80		- 5
35-45	20		- 9	88-45	900	9.0	- 5
38-00	40		- 9		20		
40-15	60		- 9		40		
42-15	80		- 9	95-30	60		0
44-15	500	9.0	-10	96-30	70		125
46-15	20		-11	97-45	80		19
48-15	40		-13	100-00	1000	9.0	0
50-30	60		-19				
52-45	80		-28				

(47)

Table #31

Replication 2 Rate 2
Furnace 2 Density 1

Time Min.-Sec.	Temp. °C.	dH °C/Min.	Diff. Galv. (mm)	Time Min.-Sec.	Temp. °C.	dH °C/Min.	Diff. Galv. (mm)
0-00	100		-22	44-15	600	11.6	-66
1-45	20		-27	45-00	20		-90
3-30	40		-29	46-45	40		-95
5-15	60		-29	48-30	60		-75
7-00	80		-28	50-00	80		-36
8-30	200	11.8	-27	51-30	700	11.7	-22
10-00	20		-26	53-00	20		-18
11-30	40		-26	54-30	40		-17
13-00	60		-25	56-00	60		-17
14-30	80		-23	57-30	80		-15
16-00	300	12.5	-22	58-45	800	11.9	-13
17-30	20		-21	60-30	20		-11
19-00	40		-19	62-00	40		-10
21-00	60		-17	63-30	60		- 9
22-45	80		-17	65-30	80		- 8
25-00	400	12.0	-17	67-30	900	11.8	- 7
27-00	20		-17		20		
28-45	40		-17		40		
30-45	60		-18	73-30	60	50 ohms	- 5
32-30	80		-19	74-15	70		0
34-30	500	11.6	-21	74-45	80		82
36-15	20		-22		90		0
38-00	40		-25	76-00	1000	11.9	0
39-45	60		-31				
41-30	80		-44				

Table #32

Replication 2

Rate 2

Furnace 2

Density 2

<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm)</u>	<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm)</u>
0-00	100		-27	42-00	600	11.9	-86
2-00	20		-32	44-00	20		-111
3-45	40		-36	46-00	40		-118
5-30	60		-35	48-00	60		-104
7-15	80		-34	49-45	80		- 52
9-00	200	11.1	-34	51-30	700	11.7	-33
10-30	20		-33	53-15	20		-29
12-00	40		-33	54-45	40		-28
13-30	60		-33	56-15	60		-27
15-00	80		-32	57-45	80		-27
16-30	300	12.1	-32	59-15	800	11.8	-27
18-00	20		-31	60-30	20		-26
19-30	40		-30	61-30	40		-25
21-00	60		-30	62-45	60		-22
22-15	80		-31	64-15	80		-18
24-00	400	12.5	-30	66-00	900	12.1	-16
25-30	20		-30		20		
27-15	40		-27		40		
29-00	60		-28	72-00	60		0
30-45	80		-28		75		106
33-00	500	12.1	-29	73-00	80		55
35-45	20		-32		95		0
37-00	40		-36	74-15	1000	12.1	0
38-30	60		-44				
40-15	80		-60				

(49)

Table #33

Replication 2

Rate 2

Furnace 2

Density 3

Time Min.-Sec.	Temp. °C.	dH °C/Min.	Diff. Galv. (mm)	Time Min.-Sec.	Temp. °C.	dH °C/Min.	Diff. Galv. (mm)
0-00	100		-27	41-15	600	12.1	-32
1-20	20		-31	44-00	20		-94
2-45	40		-36		30		-97
4-00	60		-39	46-40	40		-95
5-20	80		-38	48-15	60		-88
6-45	200		-35	49-30	80		-62
8-45	20		-33	50-45	700	11.8	-32
10-45	40		-32	52-00	20		-10
13-30	60		-30	53-15	40		- 8
16-00	80		-31	54-00	60		- 6
18-00	300		-32	55-15	80		- 5
19-45	20		-33	56-30	800	12.3	- 4
21-45	40		-33	59-00	20		- 4
22-40	60		-35	61-30	40		- 3
24-00	80		-37	63-45	60		- 3
25-15	400	11.9	-39	65-00	80		- 3
26-40	20		-39	66-15	900	12.1	- 3
28-00	40		-39		20		
30-00	60		-39		40		
31-45	80		-38	70-00	60	50 ohms	- 3
33-45	500	11.8	-39	71-45	70		0
35-30	20		-42		75		80
36-45	40		-46	73-30	80		17
38-00	60		-51	75-15	1000	12.0	0
39-15	80		-63				

(50)

Table #34

Replication 2

Rate 3

Furnace 2

Density 1

<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm)</u>	<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm)</u>
0-00	100		-25	31-00	600	16.0	-74
1-15	20		-43	32-15	20		-83
2-30	40		-52	33-45	40		-96
	60				50		-97
	80			35-00	60		-94
	200	18.5		36-30	80		-75
	20			38-15	700	15.7	-30
7-30	40		-50	40-15	20		-28
8-30	60		-47	42-00	40		-25
9-30	80		-42	43-45	60		-25
10-45	300	18.4	-40	45-00	80		-25
12-00	20		-35	46-15	800	15.1	-26
13-30	40		-31	47-30	20		-26
15-30	60		-30	48-45	40		-26
17-00	80		-29	50-00	60		-27
18-30	400	16.0	-31	51-00	80		-27
20-00	20		-32	52-15	900	15.4	-26
21-15	40		-34		20		
22-30	60		-36		40		
23-45	80		-38	56-45	60	50 ohms	-10
25-00	500	16.0	-41	57-45	70		0
26-15	20		-43	58-45	80		123
27-30	40		-47	59-45	95		0
28-45	60		-54	59-45	1000	15.0	0
30-00	80		-68				

(51)

Table #35

Replication 2

Rate 3

Furnace 2

Density 2

<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm)</u>	<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm)</u>
0-00	100		-30	35-45	600	14.0	-68
1-30	20		-38	37-00	20		-90
3-30	40		-44	38-30	40		-101
4-30	60		-43	39-15	50		-102
5-45	80		-40	39-45	60		-99
7-00	200	14.3	-37	40-45	80		-89
8-15	20		-35	42-00	700	14.3	-67
9-30	40		-34	43-00	20		-36
10-45	60		-31	44-15	40		-26
12-00	80		-29	45-15	60		-22
13-15	300	15.1	-27	46-15	80		-20
14-30	20		-25	47-15	800	14.8	-19
16-00	40		-23	48-15	20		-18
17-30	60		-22	49-15	40		-17
19-50	80		-22	50-30	60		-15
21-00	400	14.3	-20	51-30	80		-14
22-30	20		-19	52-30	900	15.2	-13
24-00	40		-19		20		
26-00	60		-19		40	50 ohms	
27-30	80		-19	56-30	60		- 5
29-30	500	13.6	-21	52-15	70		0
30-45	20		-24		75		93
32-00	40		-27	58-00	80		13
33-15	60		-34	60-00	1000		0
34-40	80		-46				

(52)

Table #36

Replication 2

Rate 3

Furnace 2

Density 3

<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm)</u>	<u>Time</u> <u>Min.-Sec.</u>	<u>Temp.</u> <u>°C.</u>	<u>dH</u> <u>°C/Min.</u>	<u>Diff. Galv.</u> <u>(mm)</u>
0-00	100		-34	53-00	600	15.1	-67
1-20	20		-41	34-30	20		-91
2-45	40		-48	35-50	40		-109
4-00	60		-49		50		-104
5-20	80		-46	37-15	60		-100
6-40	200	15.0	-43	38-45	80		-90
7-00	20		-40	40-00	700	15.0	-66
8-30	40		-39	41-15	20		-34
9-45	60		-36	42-45	40		-28
11-00	80		-34	44-00	60		-21
12-20	300	15.4	-32	45-15	80		-19
13-40	20		-28	46-45	800	14.9	-18
15-00	40		-23	48-00	20		-18
16-30	60		-22	49-15	40		-17
17-45	80		-22	50-45	60		-15
18-15	400	16.3	-22	52-00	80		-15
19-45	20		-20	53-15	900	15.0	-14
21-15	40		-20		20		
22-45	60		-19		40		
23-30	80		-19	56-15	60	50 ohms	- 3
25-00	500	16.0	-20	57-15	70		0
26-30	20		-25		75		97
28-15	40		-26	58-15	80		12
29-45	60		-35	59-45	1000	14.9	0
31-30	80		-47				

(53)

Table #37

Data on Density of Samples

	<u>Code</u>	<u>Wgt. Air</u>	<u>Factor</u>	<u>Wgt. Hg.</u>	<u>Factor</u>	<u>Vol. (ccs)</u>	<u>Density</u>
Rep. I	F1, D1, R1	1.80	.03042	68.0	0.50252	0.53	3.40
	F1, D1, R2	1.78	.03008	67.5	0.49883	0.53	3.36
	F1, D1, R3	1.80	.03042	68.0	0.50252	0.53	3.40
	F2, D1, R1	1.81	.03059	68.0	0.50252	0.53	3.42
	F2, D1, R2	1.80	.03042	67.0	0.49513	0.53	3.40
	F2, D1, R3	1.78	.03008	67.0	0.49513	0.53	3.36
Rep. II	F1, D1, R1	1.80	.03042	67.5	0.49883	0.53	3.40
	F1, D1, R2	1.81	.03059	68.0	0.50252	0.53	3.42
	F1, D1, R3	1.81	.03059	68.0	0.50252	0.53	3.42
	F2, D1, R1	1.80	.03042	67.0	0.49513	0.53	3.40
	F2, D1, R2	1.80	.03042	66.0	.48774	0.53	3.40
	F2, D1, R3	1.80	.03042	67.0	.49513	0.53	3.40

(54)

D1 Average Density

3.39 gms/cc

Table #37 (Continued)

Data on Density of Samples

	<u>Code</u>	<u>Wgt. Air</u>	<u>Factor</u>	<u>Wgt. Hg.</u>	<u>Factor</u>	<u>Vol. (ccs)</u>	<u>Density</u>
Rep. I	F1, D2, R1	1.80	.03042	61.0	0.45079	0.48	3.75
	F1, D2, R2	1.80	.03042	62.0	0.45818	0.49	3.68
	F1, D2, R3	1.79	.03025	62.0	0.45818	0.49	3.66
	F2, D2, R1	1.78	.03008	62.0	0.45818	0.49	3.64
	F2, D2, R2	1.80	.03042	62.0	0.45818	0.49	3.68
	F2, D2, R3	1.80	.03042	62.0	0.45818	0.49	3.68
Rep. II	F1, D2, R1	1.79	.03025	62.0	0.45818	0.49	3.66
	F1, D2, R2	1.81	.03059	63.0	0.46557	0.50	3.62
	F1, D2, R3	1.79	.03025	62.0	0.45818	0.49	3.66
	F2, D2, R1	1.80	.03042	62.5	0.46188	0.49	3.68
	F2, D2, R2	1.80	.03042	62.0	0.45818	0.49	3.68
	F2, D2, R3	1.79	.03025	62.0	0.45818	0.49	3.66

(55)

D2 Average Density

3.67 gms/cc

Table #37 (Continued)

Data on Density of Samples

	<u>Code</u>	<u>Wgt. Air</u>	<u>Air Factor</u>	<u>Wgt. Hg.</u>	<u>Hg Factor</u>	<u>Vol. (ccs)</u>	<u>Density</u>
Rep. I	F1, D3, R2	1.78	0.03008	57.0	0.42123	0.45	3.96
	F1, D3, R2	1.79	0.03025	58.0	0.42862	0.46	3.89
	F1, D3, R3	1.79	0.03025	58.0	0.42862	0.46	3.89
	F2, D3, R1	1.79	0.03025	57.0	0.42123	0.45	3.98
	F2, D3, R2	1.80	0.03042	57.0	0.42123	0.45	4.00
	F3, D3, R3	1.79	0.03025	58.0	0.42862	0.46	3.89
Rep. II	F1, D3, R1	1.80	0.03042	57.0	0.42123	0.45	4.00
	F1, D3, R2	1.79	0.03025	57.0	0.42123	0.45	3.98
	F1, D3, R3	1.79	0.03025	57.0	0.42123	0.45	3.98
	F2, D3, R1	1.81	0.03059	57.5	0.42493	0.45	4.02
	F2, D3, R2	1.79	0.03025	57.0	0.42123	0.45	3.98
	F2, D3, R3	1.80	0.03025	57.5	0.42493	0.45	4.00

(56)

D3 Average Density 3.96 gmas/cc

(57)

Table #38

Calibration of the Chromel-Alumel Differential Thermocouple When
with Spotlight #2401 Galvanometer

1. With 25 ohms External Resistance

<u>Ref. Temp. °C.</u>	<u>Bath Temp. °C</u>	<u>T(°C.)</u>	<u>Galv. Defl.</u>
0	4	4	9
0	13	13	45
0	19	19	69
0	20	20	74
0	25	25	87
0	30	30	100

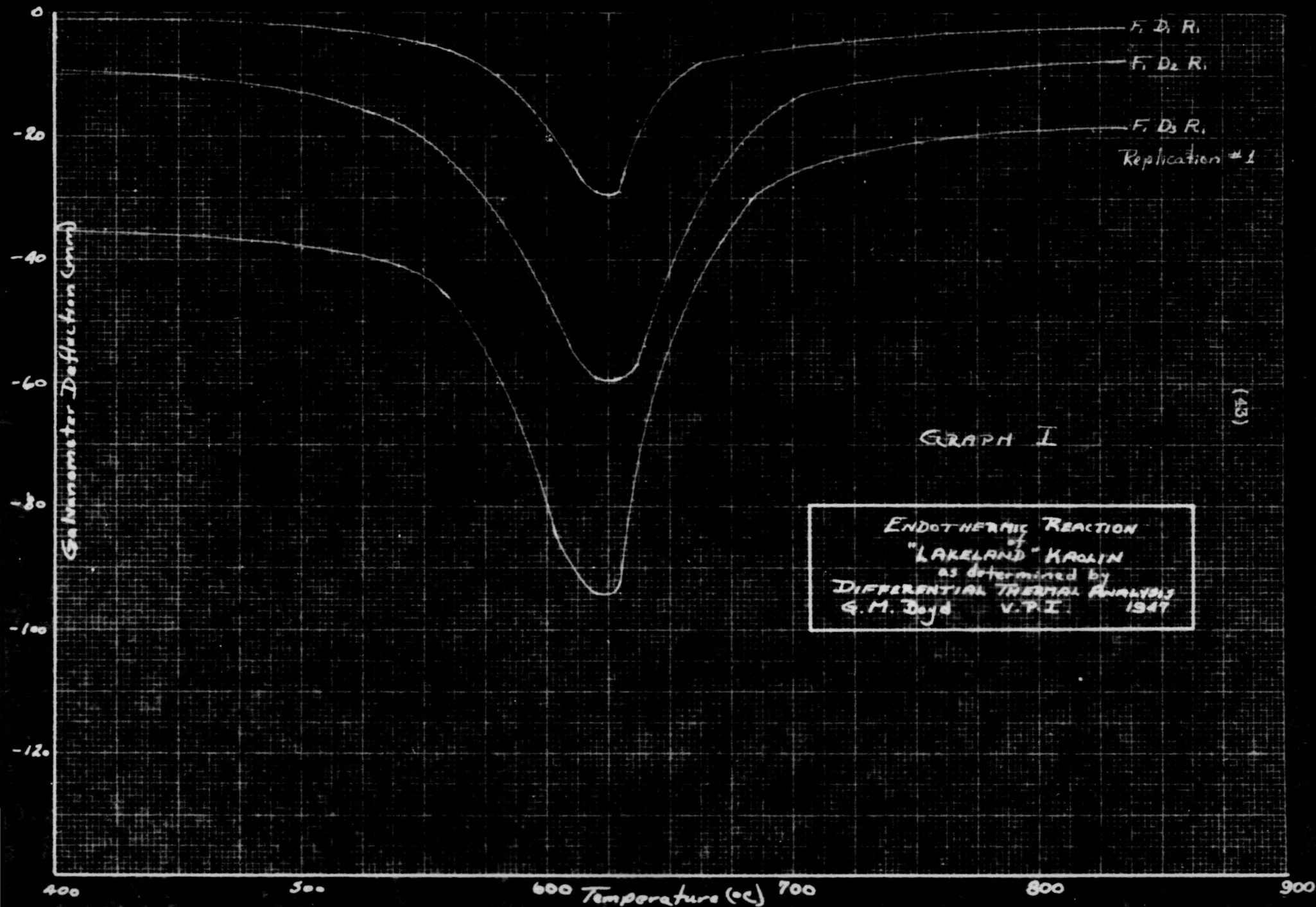
2. With 50 ohms External Resistance

<u>Ref. Temp. °C.</u>	<u>Bath Temp. °C.</u>	<u>T(°C.)</u>	<u>Galv. Defl.</u>
0	15	15	39
0	23	23	57
0	30	30	68
0	35	35	79
0	44	44	92

V RESULTS

A. General

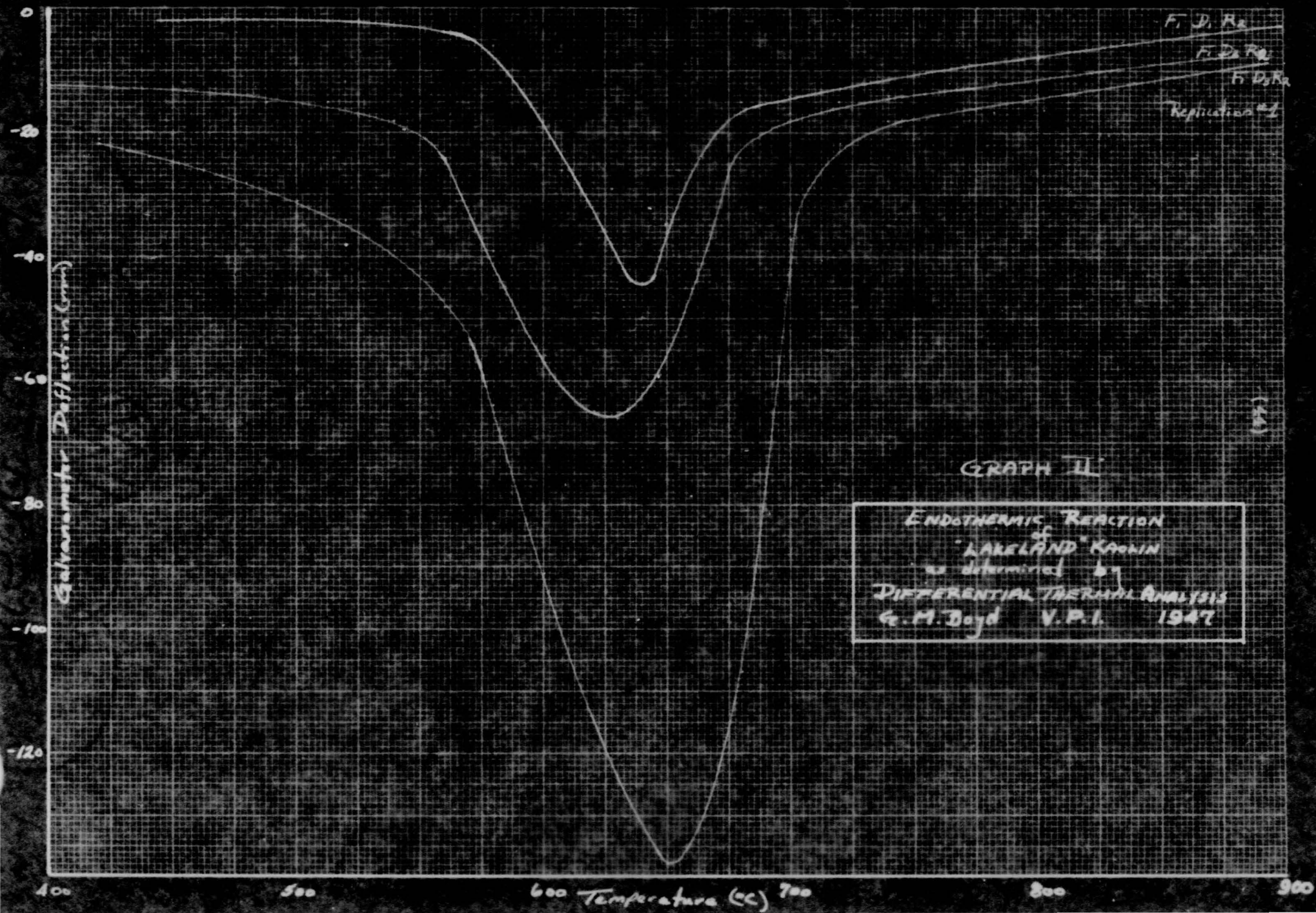
The curves on the following pages were plotted from the data tables in the preceding section. The curves are a result of the best fitting line through the plotted points. These curves, lettered according to the symbolism adopted for this study, were used for the determination of the areas as reported in Table 39 of Results.

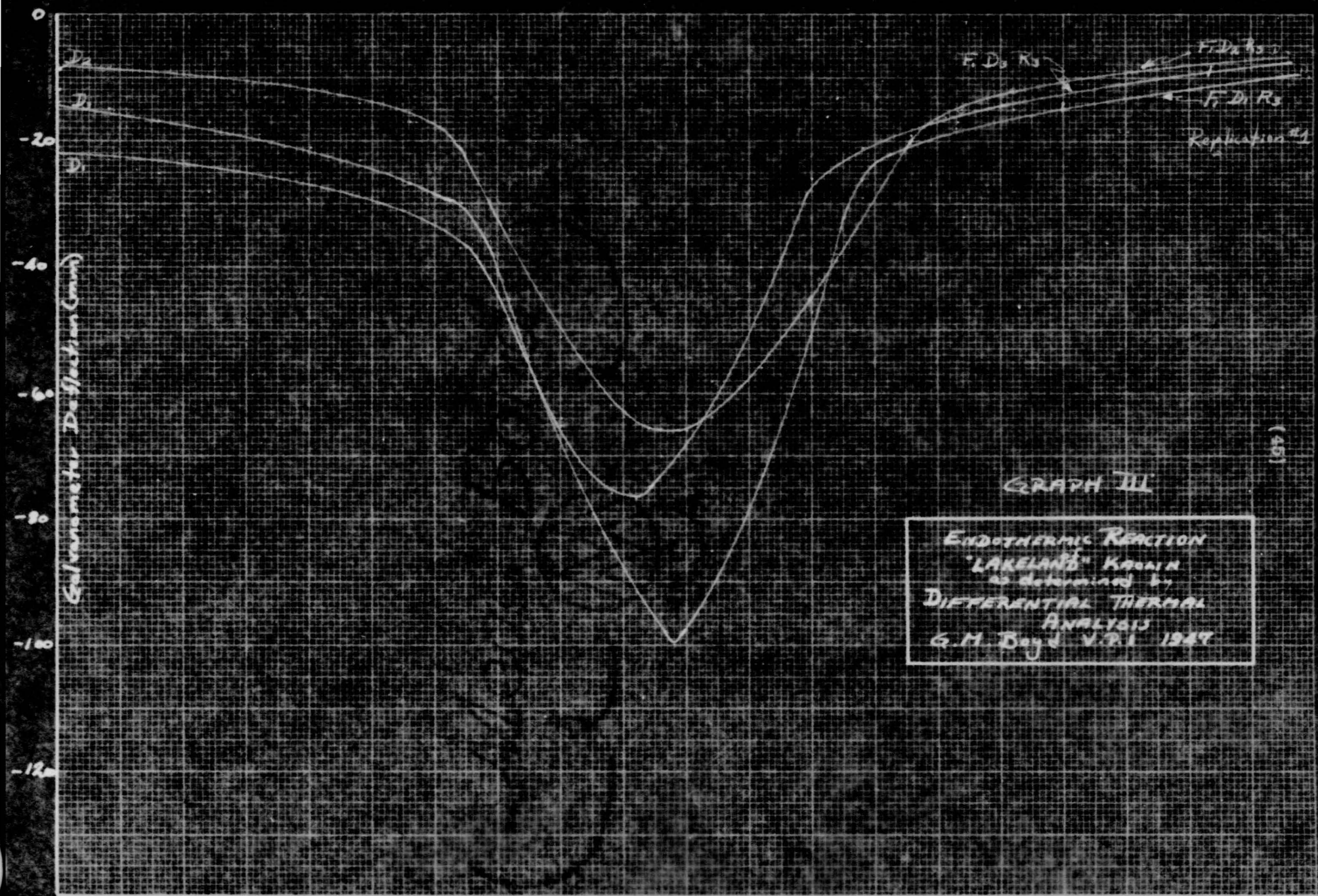


GRAPH I

ENDOTHERMIC REACTION
of
"LAKELAND" KOLIN
as determined by
DIFFERENTIAL THERMAL ANALYSIS
G. M. Boyd V.P.I. 1947

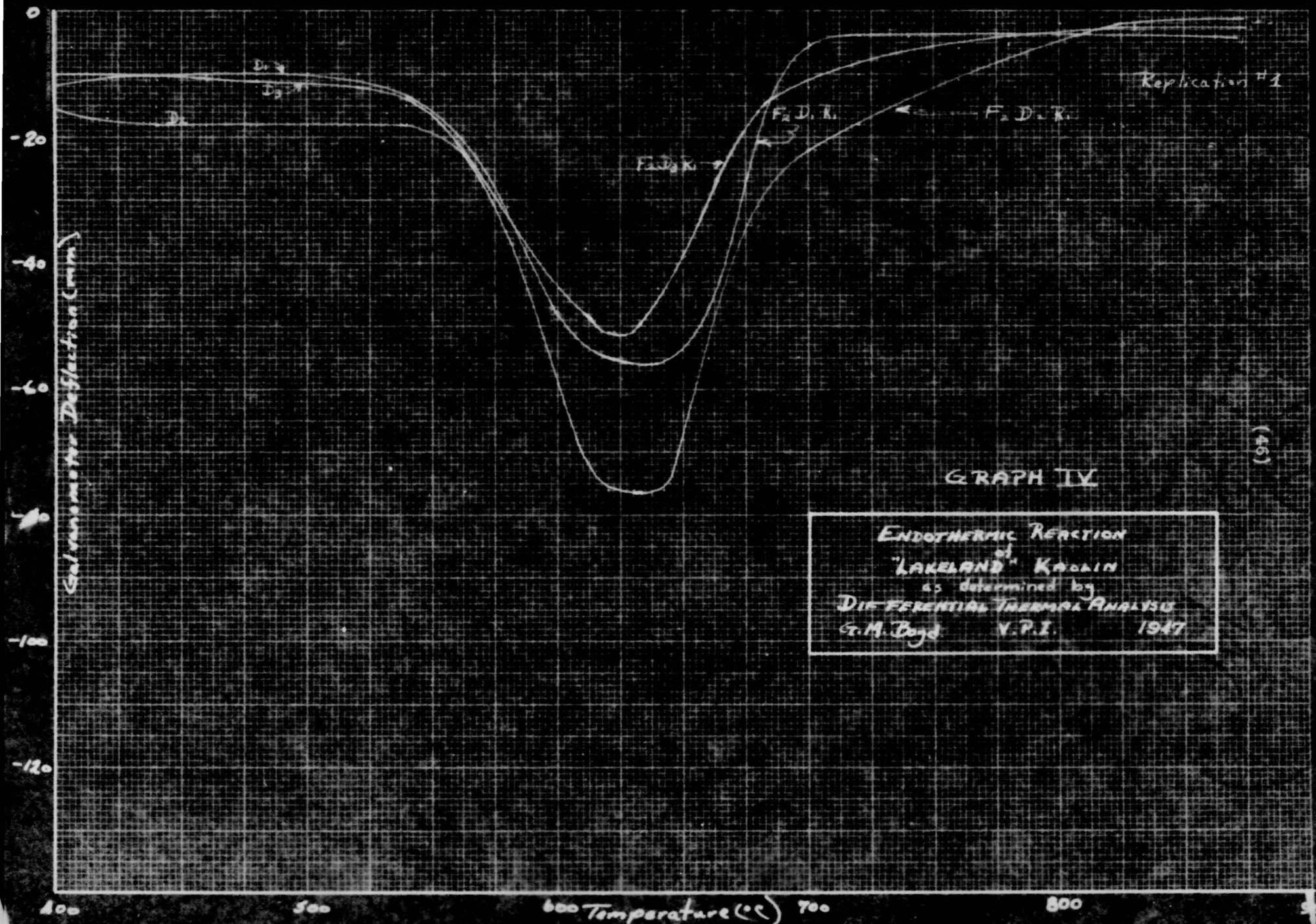
(43)

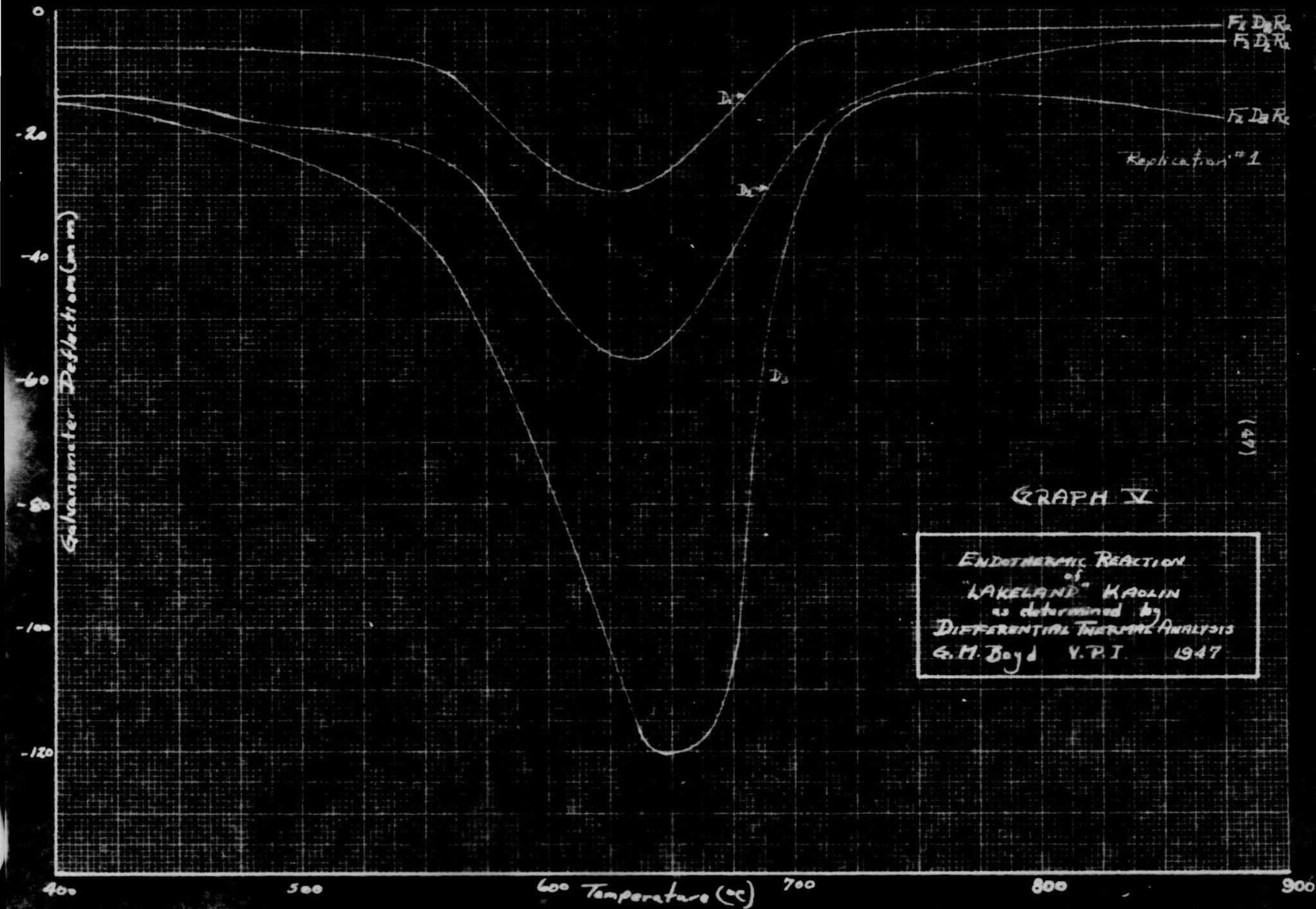


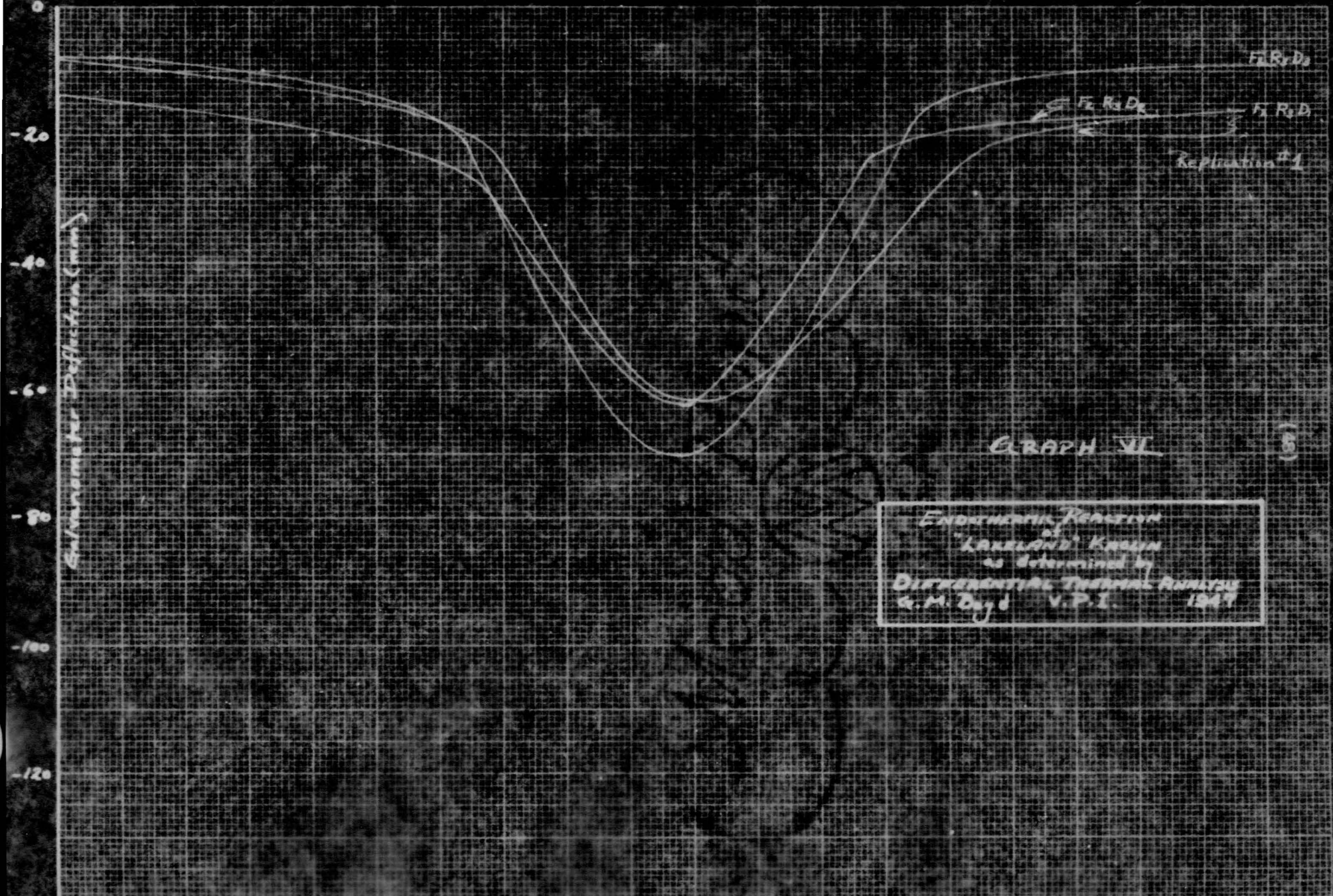


GRAPH III
 ENDOTHERMIC REACTION
 "LAKELAND" KRAVIN
 as determined by
 DIFFERENTIAL THERMAL
 ANALYSIS
 G. M. Boyd V.P.I. 1947

400 500 600 Temperature (°C) 700 800 900







GRAPH VI

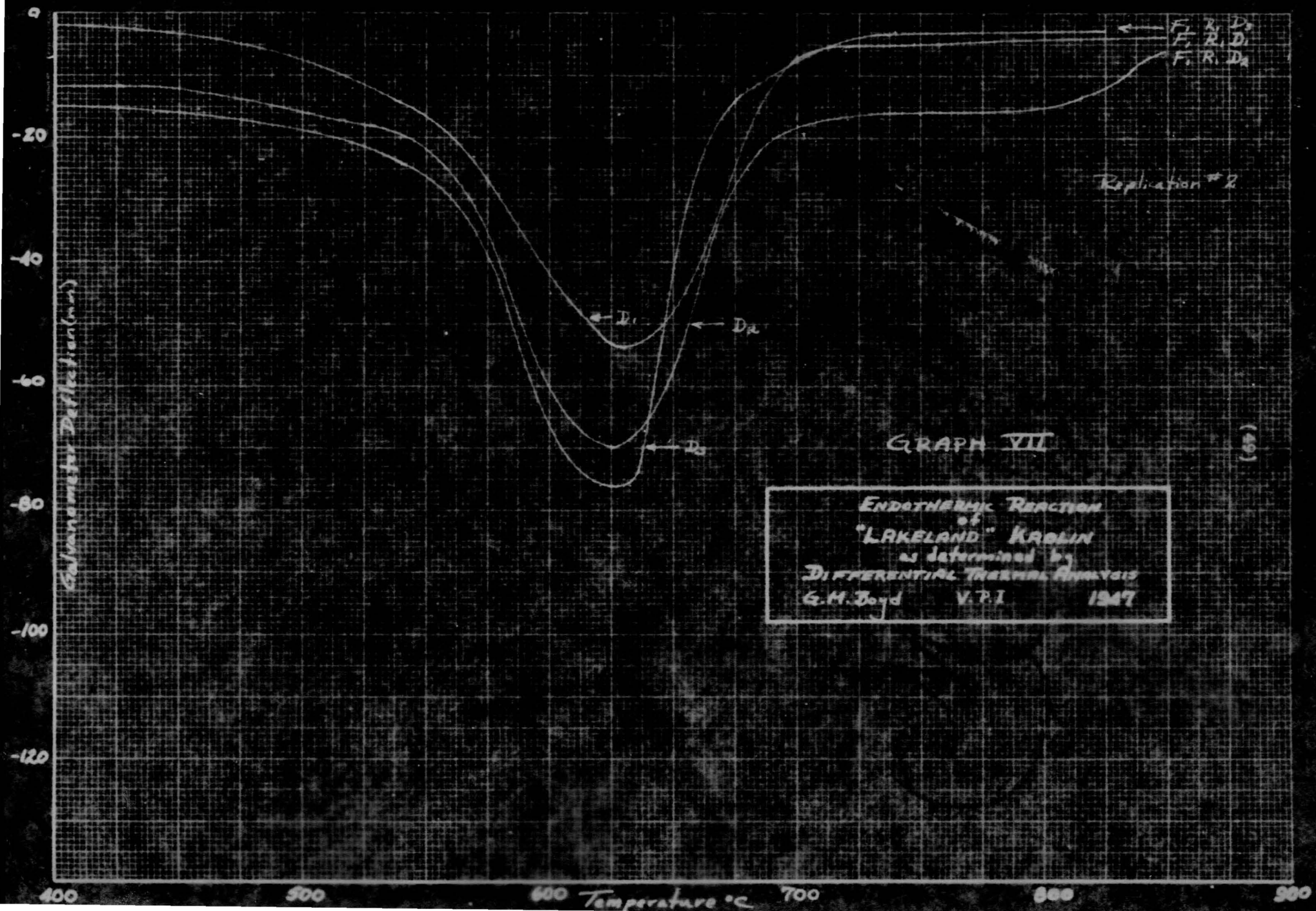
Enrollment Paper
 LAWRENCE KNOWN
 as determined by
 DIPLOMA IN TECHNICAL ANALYSIS
 R.M. D.J. V.P.I. 1947

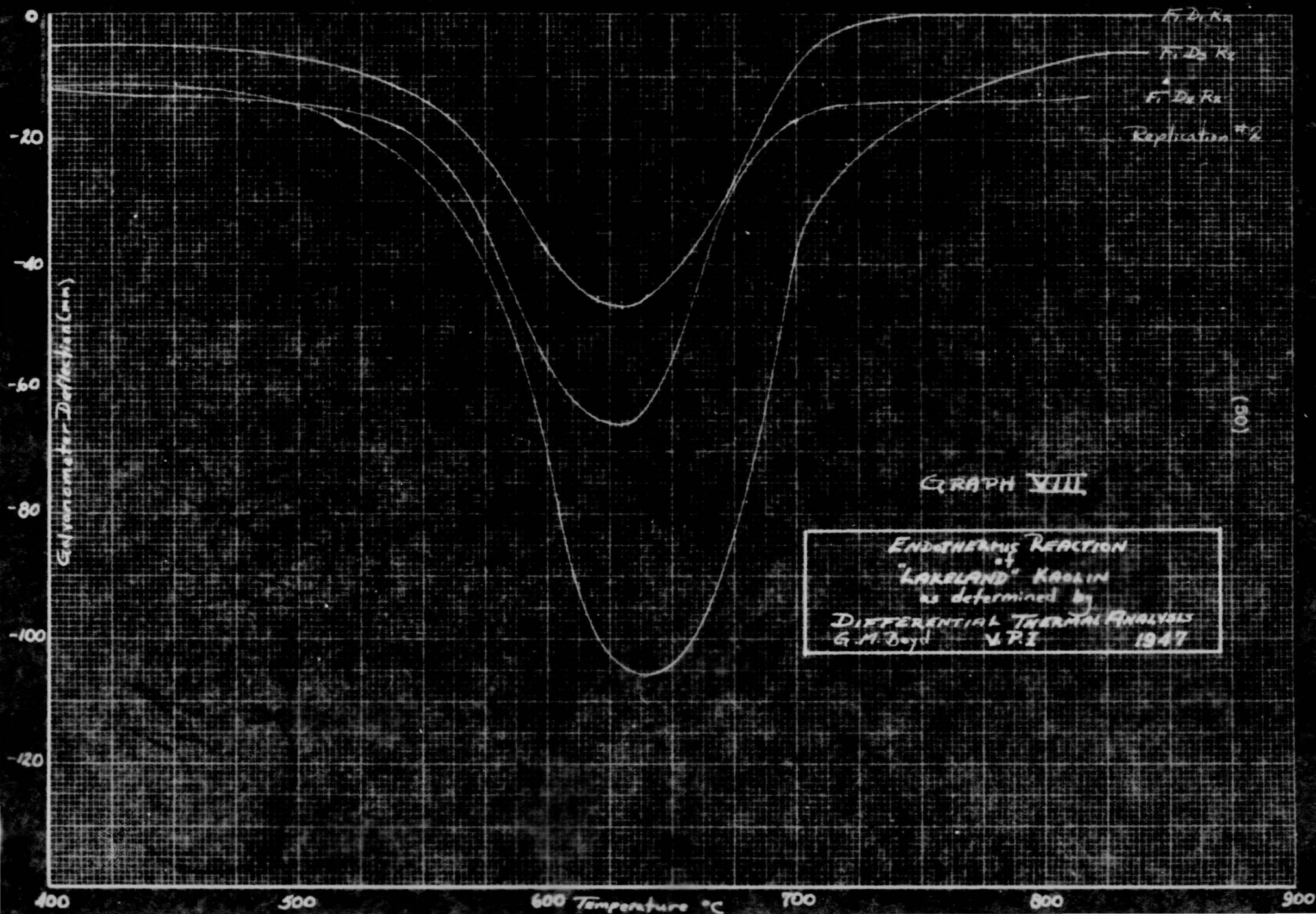
500

Temperature (°C)

800

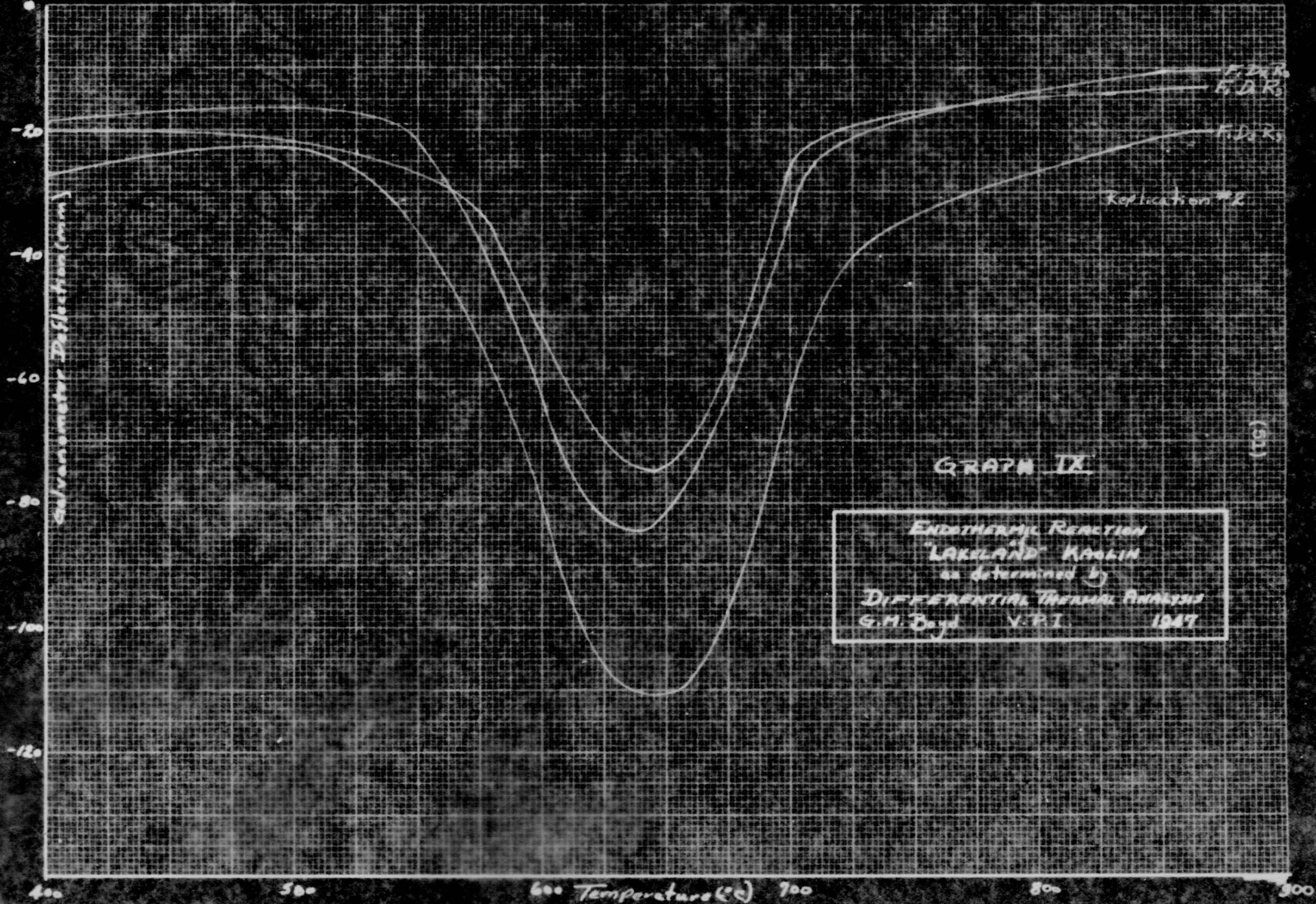
900

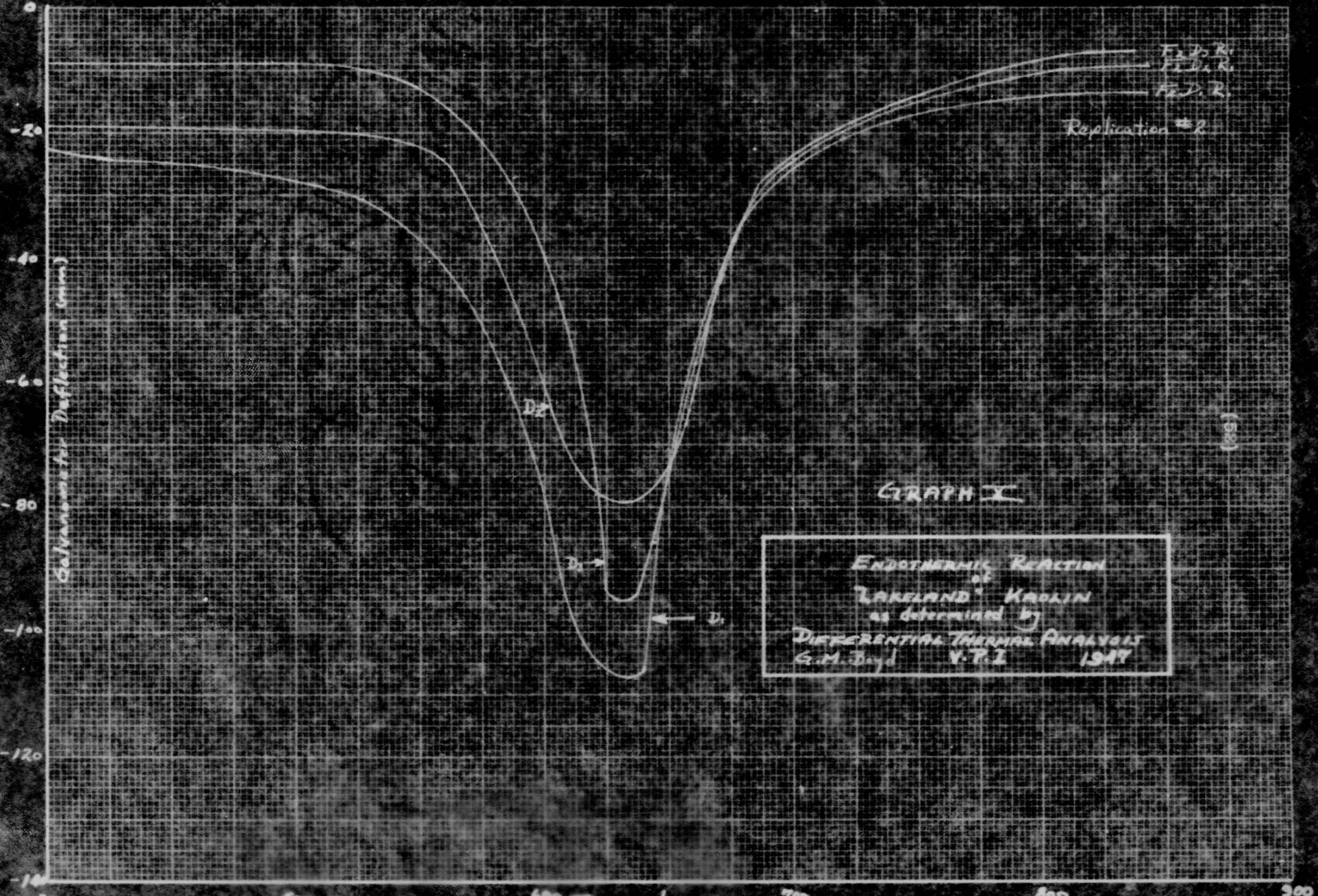


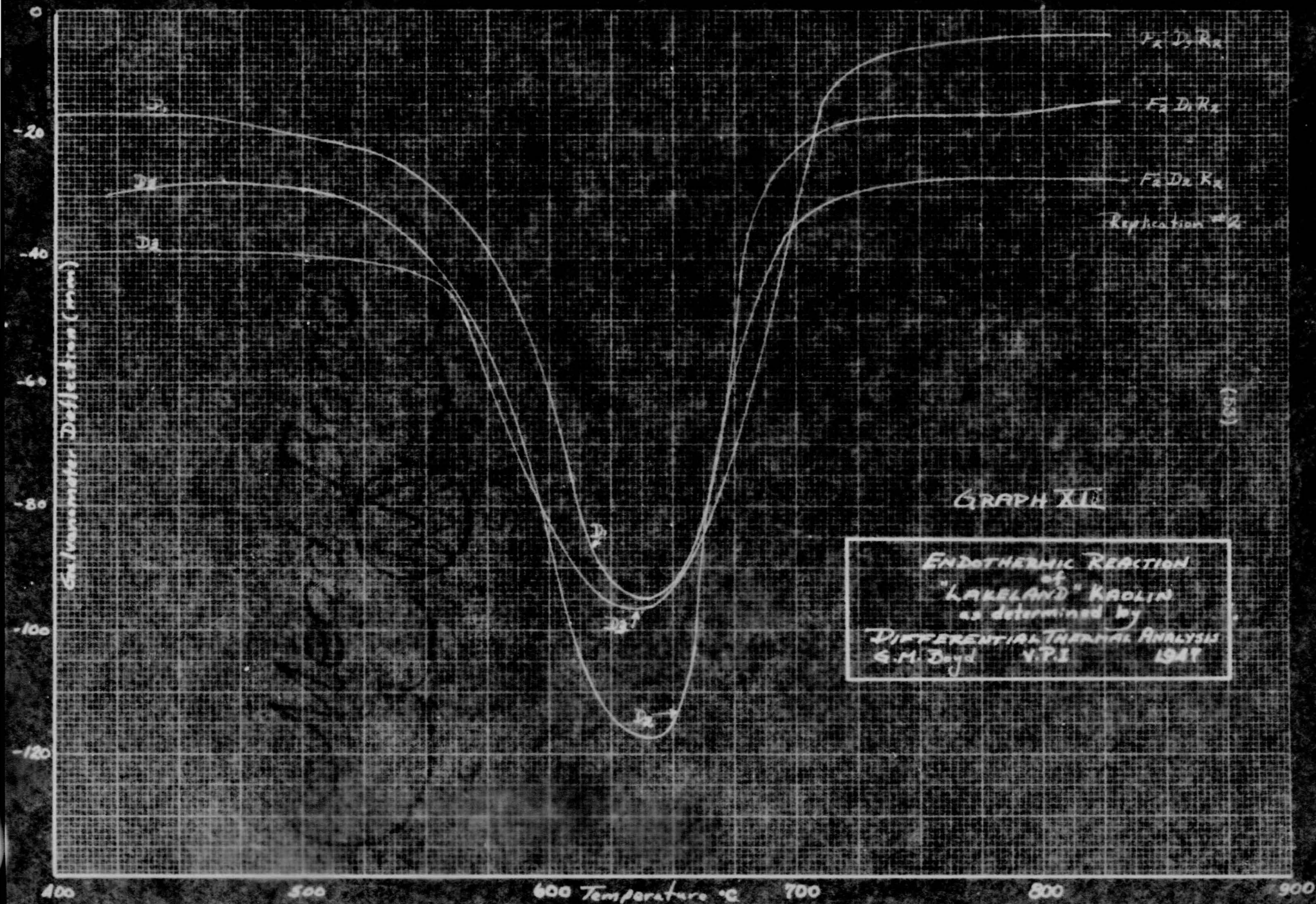


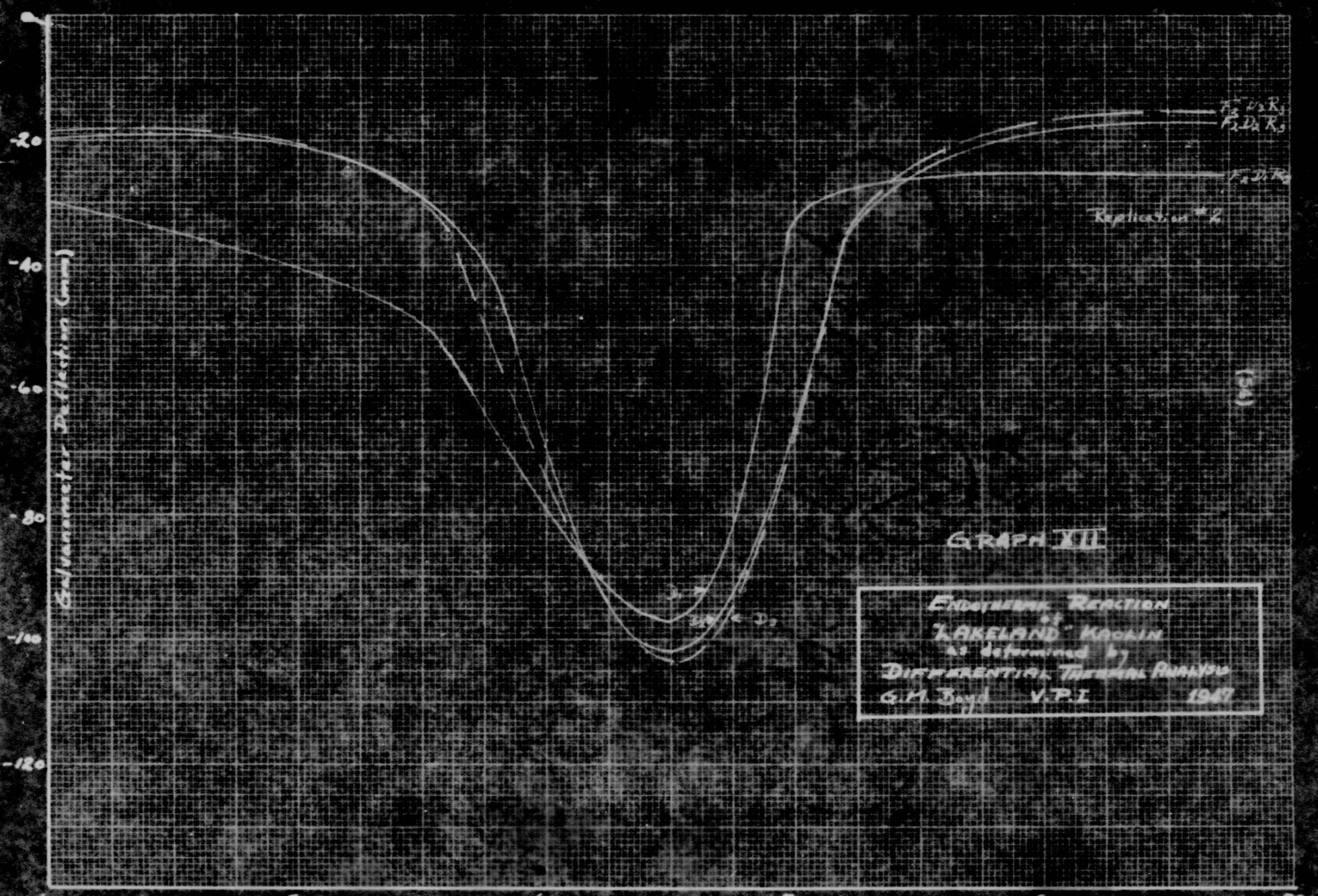
GRAPH VIII

ENDOTHERMIC REACTION
"LAKELAND" KOLIN
is determined by
DIFFERENTIAL THERMAL ANALYSIS
G. M. Boyd VPI 1947









GRAPH III

ENDOGENIC REACTION
 of
 LAKELAND KACHIN
 as determined by
 DIFFERENTIAL THERMAL ANALYSIS
 G.H. Boyd V.P.I. 1967

(55)

GRAPH VIII

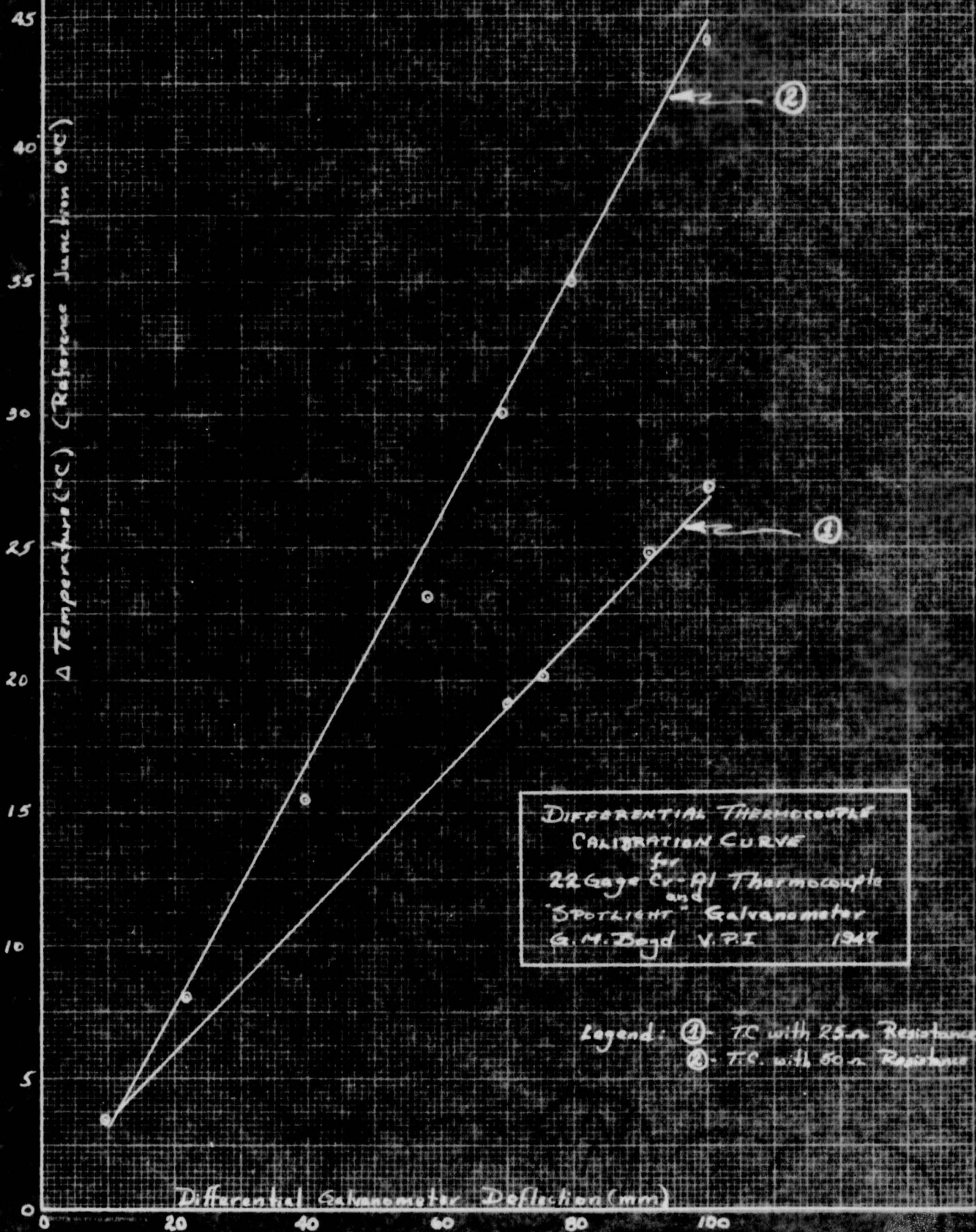


Table #39

Areas Under the Endothermic Peaks of "Lakeland"
Florida Kaolin as Determined by Differential Thermal Analysis

Replication 1*

	Furnace 1				Furnace 2			
	Rate 1	Rate 2	Rate 3	Totals	Rate 1	Rate 2	Rate 3	Totals
Density 1	12.1	20.3	59.6	92.0	31.0	15.7	44.8	91.5
Density 2	31.8	35.4	48.8	116.0	41.2	38.5	36.5	116.2
Density 3	<u>37.6</u>	<u>87.4</u>	<u>55.8</u>	<u>180.8</u>	<u>28.4</u>	<u>75.4</u>	<u>52.5</u>	<u>156.3</u>
Totals	81.5	143.1	164.2	388.8	100.6	129.6	133.8	364.0

Replication Total = 752.8

*All Areas in Sq. Cm.

Replication 2

	Furnace 1				Furnace 2			
	Rate 1	Rate 2	Rate 3	Totals	Rate 1	Rate 2	Rate 3	Totals
Density 1	31.1	31.6	33.3	96.0	45.5	44.1	43.6	133.2
Density 2	32.8	30.4	46.0	109.2	39.4	51.8	61.9	153.1
Density 3	<u>41.6</u>	<u>74.2</u>	<u>74.0</u>	<u>189.8</u>	<u>43.2</u>	<u>59.7</u>	<u>71.0</u>	<u>173.9</u>
Totals	105.5	136.2	153.3	395.0	128.1	155.6	176.5	460.2

Replication Total = 855.2

Grand Total = 1608.0

V RESULTS (Continued)

B. Statistical and Regression Analyses

The following statistical computations are presented in detail for simplicity in following through the analysis of the data obtained from the measurement of the areas under the endothermic curves of the specimens analyzed. No discussion as to the steps taken will be made, but detailed calculations will be made in each step. The theory behind all such calculations may be found in the textbook by G. W. Snedecor, "Statistical Methods", the Iowa State College Press, Ames, Iowa, 1946. All calculations in the statistical breakdown of the data were based on Table #39, page 72.

$$\text{Grand Corr. Term} = \frac{(\Sigma X)^2}{n} = \frac{1608.0^2}{36} = 71,824.0$$

$$\text{Total SS} = 12.1^2 + 20.3^2 + \dots + 71.0^2 - C = 82,254.5 - 71,824.0 = 10,430.5$$

$$\begin{aligned} \text{Furnaces SS} &= \frac{(\Sigma F_1)^2}{18} + \frac{(\Sigma F_2)^2}{18} - C = \frac{783.8^2 + 824.2^2}{18} - C = \\ &= 71,869.3 - 71,824.0 = 45.3 \end{aligned}$$

$$\begin{aligned} \text{Rates SS} &= (81.5 + 100.6 + 105.5 + 128.1)^2 + \Sigma R_2^2 + \Sigma R_3^2 - C \\ &= 415.7^2 + \frac{564.5^2}{12} + 627.8^2 - C = 73,800.0 - 71,824 = 1976.0 \end{aligned}$$

$$\text{Replications SS} = \frac{752.8^2 + 855.2^2}{18} - C = 72,115.3 - C = 291.3$$

(74)

$$\text{Densities SS} = \frac{(96.0 + 133.2 + 92.0 + 91.5)^2}{12} + D_2^2 + D_3^2 - C =$$
$$\frac{412.7^2 + 494.5^2 + 700.8^2}{12} - C = 75,497.7 - C = 3,673.7$$

Two Way Tables for Interactions:

Sub Table 1 - Determination of Interactions
Furnaces x Rates

Rates \ Furnaces	Furnaces		Totals
	1	2	
1	187.0	228.7	415.7
2	279.3	285.2	564.5
3	<u>317.5</u>	<u>310.3</u>	<u>627.8</u>
Totals	783.8	824.2	1608.0

Sub Table Analysis of Variance	d/f	ss
Furnaces	1	45.3
Rates	2	1976.0
Furnaces x Rates Interaction (by Diff.)	2	<u>106.8</u>
Total	5	2128.1

$$\text{Sub Total SS} = \frac{187.0^2 + 228.7^2 + \dots + 310.3^2}{6} - C = 73,952.1 - C = 2128.1$$

Sub Table 2 - Determination of Interaction of
Furnaces x Densities

Densities \ Furnaces	Furnaces		Totals
	1	2	
1	188.0	224.7	412.7
2	225.2	269.3	494.5
3	<u>370.6</u>	<u>330.2</u>	<u>700.8</u>
Totals	783.8	824.2	1608.0

Sub Table Analysis of Variance	d/f	ss
Furnaces	1	45.3
Densities	2	3673.7
Furnaces x Densities Interaction (Diff.)	2	365.0
Totals	5	4084.0

$$\text{Sub Total SS} = \frac{188.0^2 + 224.7^2 + \dots + 330.2^2}{6} - C =$$

$$= 75,908.0 - 71,824.0 = 4084.0$$

Sub Table 3 - Determination of the Interactions of Rates x Densities

Rates Densities	Rates			Totals
	1	2	3	
1	119.7	111.7	181.3	412.7
2	145.2	156.1	193.2	494.5
3	<u>150.8</u>	<u>296.7</u>	<u>253.3</u>	<u>700.8</u>
Totals	415.7	564.5	627.8	1608.0

Sub Table Analysis of Variance	d/f	ss
Rates	2	1976.0
Densities	2	3673.7
Rates x Densities Interaction (By Diff.)	<u>4</u>	<u>1872.1</u>
Totals	8	7521.8

$$\text{Sub Total SS} = \frac{119.7^2 + \dots + 253.3^2}{4} - C = 7521.8$$

(77)

Sub Table 4 - Determination of the Interaction of Furnace x Rates x Densities

Rates	Furnaces						Totals
	F1			F2			
	D1	D2	D3	D1	D2	D3	
R1	43.2	64.6	79.2	76.5	80.6	71.6	415.6
R2	51.9	65.8	161.6	59.8	90.3	135.1	564.6
R3	<u>92.9</u>	<u>94.8</u>	<u>129.8</u>	<u>88.4</u>	<u>98.4</u>	<u>123.5</u>	<u>627.8</u>
Totals	188.1	225.2	370.6	224.7	269.3	330.2	1608.0

Sub Table Analysis of Variance	d/f	ss
Furnaces	1	45.3
Rates	2	1976.0
Densities	2	3673.7
Rates x Densities Interaction	4	1872.1
Furnaces x Rates Interaction	2	106.8
Furnaces x Densities Interaction	2	365.0
Furnaces x Rates X Densities Interaction	4	198.1
Totals	17	8237.0

$$\text{Sub Total SS} = \frac{43.2^2 + 64.6^2 + \dots + 123.5^2}{2} - 0 = 80,061.0 - 71,824.0 = 8,237.0$$

(78)

Table #40

Preliminary Analysis of Variance

Sources	d/f	SS	MS	F	Significance
Replications	1	291.3	291.3	2.60	-
Furnaces	1	45.3	45.3	-	-
Rates	2	1976.0	988.0	8.84	xx
Densities	2	3673.7	1836.9	16.41	xx
Furnaces x Rates Interaction	2	106.8	53.4	-	-
Furnaces x Densities Interaction	2	365.0	132.5	1.17	-
Rates x Densities Interaction	4	1872.1	468.0	4.18	x
Furnaces x Rates x Densities Interaction	4	198.1	49.5	-	-
Experimental Error (Diff.)	17	1902.2	111.9	-	-
	—	—	—		
Totals	35	10430.5			

Legend: - Not Significant
x Significant, F = 0.05 level
xx Highly Significant, F = 0.01 level

Sub Table 5 - Regression Analysis of Rates and Densities

Densities	Rates			Totals
	R1	R2	R3	
D1	119.7	111.7	181.3	412.7
D2	145.2	156.1	193.2	494.5
D3	150.8	296.7	253.3	700.8
Totals	415.7	564.5	627.8	1608.0

A. Regression of the Rates

Rates	9°C/Min.	12°C/Min.	15°C/Min.	
Sum of Rates All D's	415.7	564.5	627.8	
Linear Trends	+	0	-	SC ² = 2
Quadratic Trends	+	-2	+2	SC ² = 6

$$SS \text{ due to linear regression} = \frac{(415.7 - 0 + 627.8)^2}{2 \times 12}$$

$$SS_L = 1874.4$$

$$SS \text{ due to quadratic regression} = \frac{(415.7 - 2(564.5) + 627.8)^2}{6 \times 12}$$

$$SS_Q = 101.6$$

$$SS_L = 1874.4$$

$$SS_Q = 101.6$$

$$\text{Total} = 1976.0 = \text{SS Rates as determined in Sub Table 3}$$

B. Regression of the Densities

Densities	1	2	3	
Sums of Densities (All Rates)	412.7	494.5	703.8	
Linear Trends	+	0	-	$SC^2 = 2$
Quadratic Trends	+	-2	+	$SC^2 = 6$

$$SS \text{ for Linear Regression} = \frac{(412.7 + 0 - 700.8)^2}{kc} = \frac{288.1^2}{2 \times 12} = 3458.4$$

$$SS \text{ for Quadratic Regression} = \frac{(412.7 - 2(494.5) + 700.8)^2}{kc} = \frac{(124.5)^2}{6 \times 12} = 215.3$$

$$SS \text{ Linear} = 3458.4$$

$$SS \text{ Quadratic} = 215.3$$

$$\text{Total} = 3673.7 = SS \text{ for Densities}$$

from Sub Table 3

Sub Table 6
 Determination of the Regression Equations for Heating Rates - Method of Least Squares

Rearrangement of the Data for Simplicity of Calculation

Replication I

	Furnace 1						Furnace 2					
	D1		D2		D3		D1		D2		D3	
	Rate	Area	Rate	Area	Rate	Area	Rate	Area	Rate	Area	Rate	Area
	9	12.1	9	31.8	9	37.6	9	31.0	9	41.2	9	28.4
	12	20.3	12	35.4	12	87.4	12	15.7	12	38.5	12	75.4
	<u>15</u>	<u>59.6</u>	<u>15</u>	<u>48.8</u>	<u>15</u>	<u>55.8</u>	<u>15</u>	<u>44.8</u>	<u>15</u>	<u>36.5</u>	<u>15</u>	<u>52.5</u>
Totals	36	92.0	36	116.0	36	180.0	36	91.5	36	116.2	36	156.3

(81)

Replication II

	Furnace 1						Furnace 2					
	D1		D2		D3		D1		D2		D3	
	Rate	Area	Rate	Area	Rate	Area	Rate	Area	Rate	Area	Rate	Area
	9	31.1	9	32.8	9	41.6	9	45.5	9	39.4	9	43.2
	12	31.6	12	30.4	12	74.2	12	44.1	12	51.8	12	59.7
	<u>15</u>	<u>33.3</u>	<u>15</u>	<u>46.0</u>	<u>15</u>	<u>74.0</u>	<u>15</u>	<u>43.6</u>	<u>15</u>	<u>61.9</u>	<u>15</u>	<u>71.0</u>
Totals	36	96.0	36	109.2	36	189.8	36	133.2	36	153.1	36	173.9

Determination of the Constants in the Regression Equation

$$Y = \bar{y} + b(x - \bar{x}) \text{ where}$$

Y = any calculated value of area under the curve

\bar{y} = the mean of all areas, at all rates, all densities,
and all furnaces (44.7 sq. cm.)

b = the regression coefficient

x = any value of heating rate

\bar{x} = the mean heating rate (12°C/min)

$$\text{Correction Terms: } C_x = \frac{(SX)^2}{n} = \frac{432^2}{36} = 5184.0$$

$$C_{xy} = \frac{(SX)(SY)}{n} = \frac{(432)(1608)}{36} = 19,296.0$$

$$\text{Regression Totals: } SX^2 = 5400 - 5184 = 216.0$$

$$SXY = (9)(12.1) + (12)(20.3) + \dots (15)(71.0) - C_{xy} =$$

$$= 19,932.3 - 19,296.0 = 636.3$$

$$\text{Regression Due to Treatments: } \frac{SX^2 = 36_1^2 + 36_2^2 + \dots 36_{12}^2}{3} - C_x =$$

$$= 5184.0 - 5184.0 = 0.0$$

$$SXY = \frac{(36)(92.0) + (36)(116.0) + \dots (36)(173.9)}{3} - C_{xy} =$$

$$= 19,296 - 19,296 = 0.0$$

Since there is no regression of treatments, no correction is necessary therefor and

$$b = \frac{SXY}{SX^2} = \frac{636.3}{216.0} = 2.95$$

and the regression equation for areas of the curves on rates is:

$$Y = 44.7 + 2.95(x - 12).$$

Sub Table 7
 Determination of the Regression Equation for Densities - Method of Least Squares

Rearrangement of the Data for Simplicity of Calculation

Replication I

	Furnace 1						Furnace 2					
	R1		R2		R3		R1		R2		R3	
	Dens.	Area	Dens.	Area	Dens.	Area	Dens.	Area	Dens.	Area	Dens.	Area
	3.39	12.1	3.39	20.3	3.39	59.6	3.39	31.0	3.39	15.7	3.39	44.8
	3.67	31.8	3.67	35.4	3.67	48.8	3.67	41.2	3.67	38.5	3.67	36.5
	3.96	37.6	3.96	87.4	3.96	55.8	3.96	28.4	3.96	75.4	3.96	52.5
Totals	<u>11.02</u>	<u>81.5</u>	<u>11.02</u>	<u>143.1</u>	<u>11.02</u>	<u>164.2</u>	<u>11.02</u>	<u>100.6</u>	<u>11.02</u>	<u>129.6</u>	<u>11.02</u>	<u>133.8</u>

(83)

Replication II

	Furnace 1						Furnace 2					
	R1		R2		R3		R1		R2		R3	
	Dens.	Area	Dens.	Area	Dens.	Area	Dens.	Area	Dens.	Area	Dens.	Area
	3.39	31.1	3.39	31.6	3.39	33.3	3.39	45.5	3.39	44.1	3.39	43.6
	3.67	32.8	3.67	30.4	3.67	46.0	3.67	39.4	3.67	51.8	3.67	61.9
	3.96	41.6	3.96	74.2	3.96	74.0	3.96	43.2	3.96	59.7	3.96	71.0
Totals	<u>11.02</u>	<u>105.5</u>	<u>11.02</u>	<u>136.2</u>	<u>11.02</u>	<u>153.3</u>	<u>11.02</u>	<u>128.1</u>	<u>11.02</u>	<u>155.6</u>	<u>11.02</u>	<u>133.8</u>

Determination of the Constants in the Regression Equation

$$Y = \bar{y} + b(x - \bar{x}) \text{ where}$$

Y = any calculated value of area under the curve

\bar{y} = the mean of all areas, at all rates, all densities and all furnaces (44.7 sq. cm.)

b = the regression coefficient

x = any value of density

\bar{x} = mean density (4.49 gms/cc)

$$\text{Correction Terms: } C_x = \frac{(SX)^2}{n} = \frac{132.24^2}{36} = 485.8$$

$$C_{xy} = \frac{(SX)(SY)}{n} = \frac{(161.64)(1608.0)}{36} = 5906.7$$

$$\begin{aligned} \text{Regression Totals: } SX^2 &= 3.39^2 + 3.67^2 + \dots + 3.96^2 - C_x = \\ &= 487.7 - 485.8 = 1.9 \end{aligned}$$

$$\begin{aligned} SXY &= (3.39)(12.1) + (3.67)(31.8) + \dots + (3.96)(71.0) - C_{xy} = \\ &= 5989.1 - 5906.7 = 82.4 \end{aligned}$$

$$\text{Regression Due to Treatments: } SX^2 = \frac{(11.02)^2 (12)}{3} - C_x = 485.8 - 485.8 = 0.0$$

$$\begin{aligned} SXY &= \frac{(11.02)(8.15) + (11.02)(143.1) + \dots + (11.02)(133.8)}{3} - C_{xy} = \\ &= 5906.7 - 5906.7 = 0.0 \end{aligned}$$

Since there is no regression of treatments, no correction is necessary therefor, and:

$$b = \frac{SXY}{SX^2} = \frac{82.4}{1.9} = 43.3$$

and the regression equation for areas of the curves on densities is:

$$Y = 44.7 + 43.3 (X - 3.67)$$

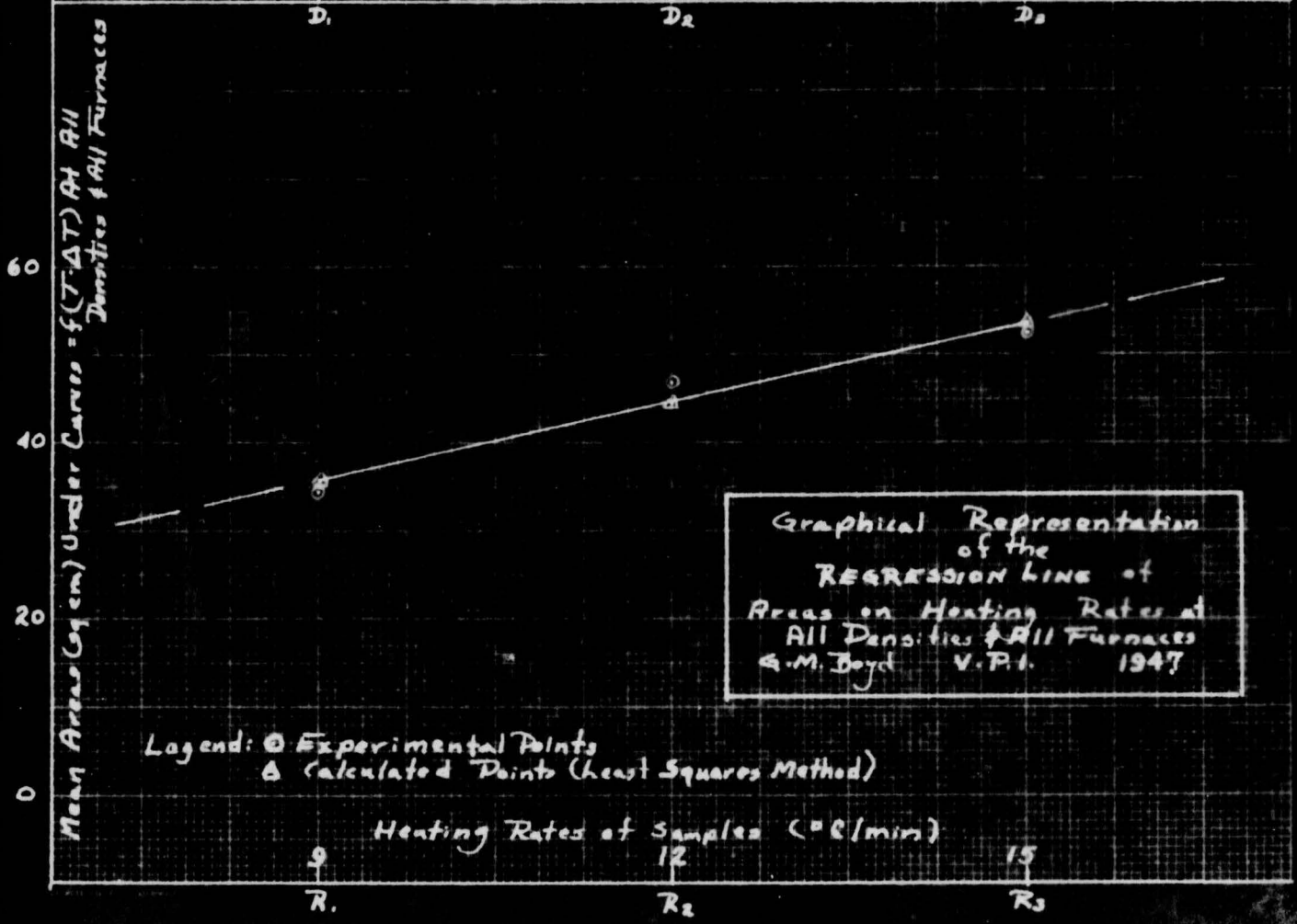
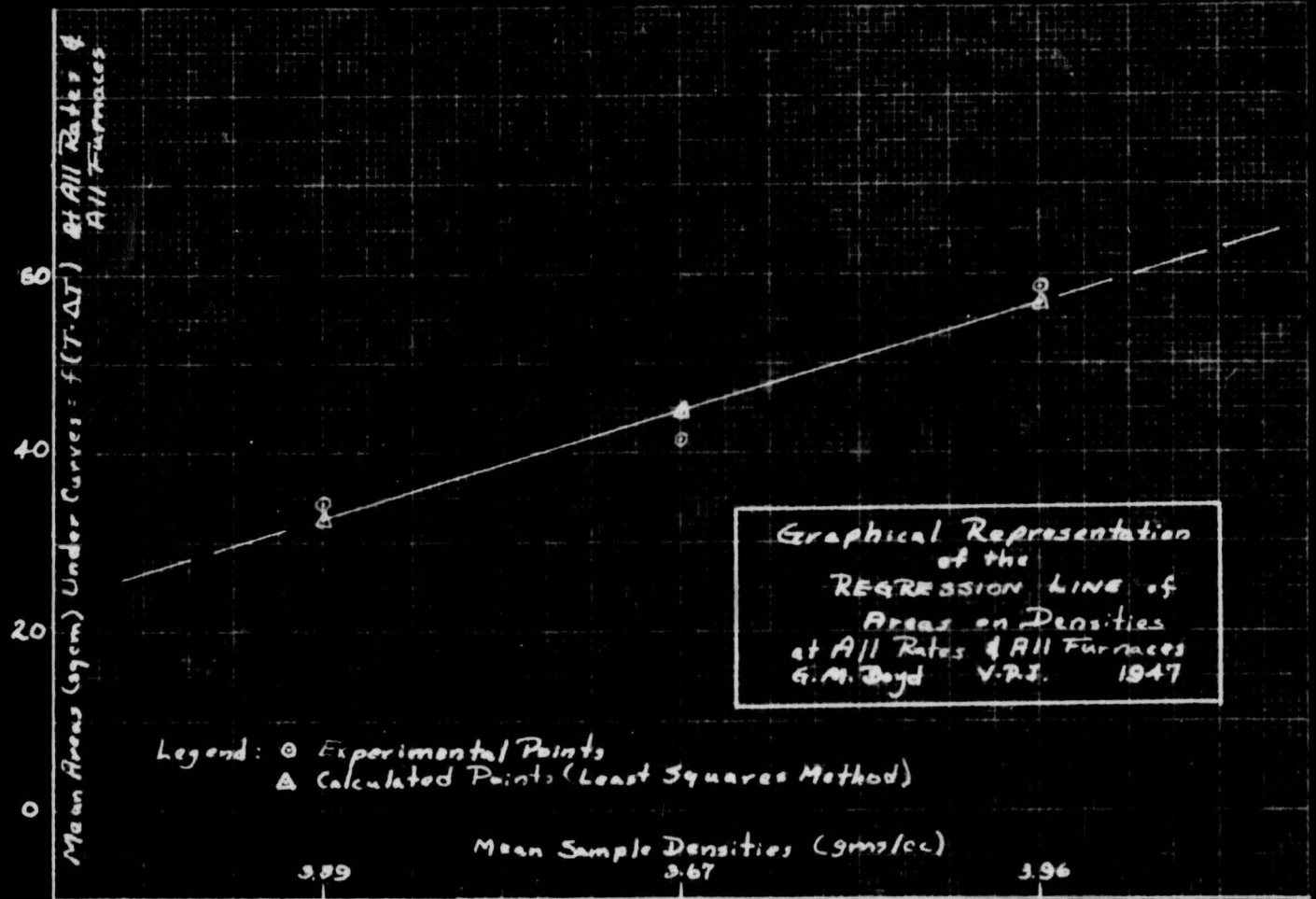


Table #41

Final Analysis of Variance

Sources	d/r	SS	MS	"F"	Significance
Replications	1	291.3	291.3	2.60	-
Furnaces	1	45.3	45.3	-	-
Rates	(2)				
(a) Linear Trend	1	1874.4	1874.4	16.80	xx
(b) Quadratic Trend	1	101.6	101.6	-	-
Densities	(2)				
(a) Linear Trend	1	3458.4	3458.4	30.90	xx
(b) Quadratic Trend	1	215.3	215.3	1.92	-
Furnace x Rates Interaction	2	106.8	53.4	-	-
Furnaces x Densities	2	365.0	182.5	1.17	-
Rates x Densities	4	1872.1	468.0	4.18	x
Furnaces x Rates x Densities	4	198.1	49.5	-	-
Experimental Error	17	1902.2	111.9		
		<hr/>	<hr/>		
Total	35	10430.5			

Legend: - Not Significant
 x Significant at 5% level
 xx Significant at 1% level

VI DISCUSSION OF RESULTS

A. Discussion of the Major Effects and Trends

This section will be devoted largely to the interpretation of the statistical analysis of variance. Once this analysis has been calculated it is the duty of the experimenter to interpret the table and to make predictions or test hypotheses. In this study the latter will be considered as the primary motive for the determinations made here. As expected, the major effects were due to differences in densities and rates of heating. This fact had been proven by all researchers in this field. However, the breakdown of these effects shows one trend that has not been previously discussed in the literature as reviewed; the linearity or non-linearity of the effects can easily be determined by the orthogonal comparisons made in the previous section. From these it can easily be seen that the optimum density and rate of heating has not been reached in the scope of this study. Another advantage of a comparison of this type is that it allows us to decide with comparatively high precision as to whether the plotted points lie on a curve or on a straight line. It must be borne in mind by all researchers that no results will be exact, but are subject to certain variations due to faulty technique and also due to the random error which is inherent whenever a measurement or determination of any type is made. This is probably one of the most difficult concepts to convey to the engineer. Too often he feels that his measurements are so precise that they are not subject to error. This is a poor assumption on his part and as a result, much valuable time and money may be wasted

in experimentation in attempting to get results which exactly duplicate each other.

The question will immediately be asked, "How do I know how much variation I will be allowed before this variation is a real difference and not just caused by random error?". The "F" test, which is a ratio of the mean square of the effect being studied to the mean square estimate of error at the levels of 1% or 5% of probability, is a measure of this characteristic. If the calculated value of the mean square of any treatment or effect exceeds the tabular probability value of "F", it may be said that the mean square of the effects or treatment has been caused by that treatment and is not a variation due alone to random error in the analysis.

Further investigation of the breakdown of any of the major effects with a view to studying the trends shows whether a quadratic or higher order curve will best fit the data. However, where there are only three points plotted, it is impossible to go beyond the quadratic curve. As was shown in the final analysis of variance the effect due to the quadratic trend was not significant. This means that in determining an empirical equation for the plotted points that a straight line is the best fitting curve available. The amount that the line would be improved by the fitting of a quadratic would be negligible. Here again the analyst does not have to make a decision as to what type of curve to draw as the analysis of variance makes this decision for him.

One other fundamental concept of statistics must be borne in mind when analyzing any sample where this sample is drawn from a large popu-

lation. Statistical analysis by its very nature works on a probability basis. It is possible to draw a sample from a normal population of known variance (σ^2) and known mean (\bar{y}) and upon testing find, that according to statistical analysis, this was not the case. This is to say that a bad sample may be drawn from a good population. Similarly, a bad sample may yield good results. However, this reversed situation will occur only in 1% or 5% of all cases depending upon the level of probability used when making the "F" test. The engineer who is not familiar with statistics will immediately say that he need not make such an assumption in his experiments and that results of his sample are used as predictions for results that would be obtained in similar determinations upon a larger amount of the population. Here again he is failing to recognize a situation which actually exists irrespective of whether or not statistics is employed in the analysis of the problem.

With this fact in mind, it may readily be seen that more reliable results may be obtained by using several samples; i.e. make replications of the experiment with different samples to minimize any chance of making a prediction from a non-representative sample.

B. Discussion of the Interactions of the Effects

An interpretation of the interactions of an experiment depends largely on the nature of the analysis being performed. Especially in the field of chemistry or any field where the additions of several substances are made independently and jointly we may often find that a significant

interaction occurs, whereas the independent treatments were non-effectual. This means that this combination of the materials gives the best (or worst) results. In this experiment where rates and densities both gave highly significant effects, the significance of the rate-density interaction indicates that it was caused by the fact that the slope of the regression line for rates and densities is different, viz., 2.95 for rates and 43.3 for densities. Had the effects of rates and densities been equal, they would have caused the interaction means square to be not significant. A visual study of the two-way table of rates and densities will show the analyst whether this interaction will develop in the calculations.

In this particular problem once the interaction effect was found to be significant, the desirability of making a regression analysis is even more evident.

C. General Considerations

In this study one source of variation in the experimental procedure was found and after discovery could not be corrected due to unavailability of material. The thickness (5/16") of the alundum muffle in the furnaces was so great that a lag occurred in the transmission of heat from the furnace windings to the nickel test specimen holder. This caused the rate of heating to follow a surging course rather than one of a very uniform rate. It is recommended that a furnace of the type described by Speil²⁸ be used in the study of differential thermal analysis. The only difference

in the described furnace and the one used in this experiment is that the former is interior wound. This change would lead to a much more rapid dissipation of the heat by the elements and more rapid absorption by the nickel test specimen holder. The top on this specimen holder will prevent direct radiation onto the surface of the sample being analyzed. The "Capacitrol" is sufficiently sensitive to control the heating rate to a very close degree. It is felt by the writer that this change in the equipment would render much more consistent results, that is, reduce the mean square of experimental error (σ^2) and consequently the standard deviation $\left(\frac{\sqrt{\sigma^2}}{n}\right)$. This, in turn, would cause the curves of the endothermic peaks to follow a much more uniform pattern and sequence.

In the study made in this thesis, had it not been for the application of statistics, it is quite possible that most or all of the data would have been discarded as useless. However, statistical analysis proved that the variation was no more than could be expected due to the inherent random error of an experiment of this type. A further conclusion from the statistical analysis would prove that a replication of the experiment is not entirely necessary. Neither is the use of two furnaces required in analyzing a sample by differential thermal analysis. Thus, a similar study for comparison of one sample against another could be considerably shortened based upon the results obtained in this experiment.

VII SUMMARY

Based on the results of this experiment the following statements may be made in summation:

1. In analyzing samples for comparison to the sample herein analyzed, a replication of the experiment is not necessary.
2. The sample could be analyzed in either or both furnaces independently. These two changes (1 and 2) could considerably shorten a subsequent study along these lines.
3. Due to equipment of different sensitivity being used by most experimenters, no real correlation can be obtained at present. In attempting correlation, the sample size, density, rate of heating, and sensitivity of the galvanometer and thermocouple used must all be specified.
4. The effects due to density and rate of heating were linear, and an optimum temperature or density was not reached in the scope of this thesis.
5. The empirical equations for density and rate of heating could be used in a future study for correlation of data with that obtained here.
6. Statistical experimental design was most useful in this study as more information was obtained from the data than would have been possible had this design not been used. The experiment was said to be efficient in that respect.
7. Statistical analysis showed that the eccentricity of the results was no more than that due to random error which is inherent in

any experiment, measurement, or determination.

8. Due to the consistency of the results, the method is to be commended as a means of qualitative analysis, and by virtue of work already done elsewhere, as a means of quantitative analysis in determining the percentages of admixtures of several of the clay minerals.

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