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AFML-TR-75-181

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DEVELOPMENT OF IMPROVED ENVIRONMENTAL RESISTANT ORGANIC-REINFORCED MATERIALS SYSTEMS

AEROTHERM DIVISION
ACUREX CORPORATION
SAN DIEGO, CALIFORNIA 92123

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NOVEMBER 1975

TECHNICAL REPORT AFML-TR-75-181
FINAL REPORT FOR PERIOD 1 JUNE 1974 - 31 AUGUST 1975

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This technical report has been reviewed and is approved for publication.

Gary D Hollingsworth

Gary D. Hollingsworth, 1 Lt.,USAF
Project Engineer

FOR THE COMMANDER

William J Schulz

William J. Schulz
Program Manager
Advanced Development Division
Air Force Materials Laboratory

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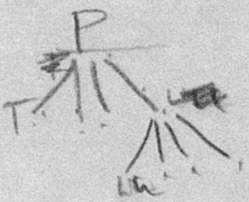

Whittaker Corp
Research and Development Div.

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Commercially available matrix resins were identified and a list of resin criteria along with target goals for properties and performance was established. A screening program consisting of dimensional change measurements, water pickup determinations, and TMA curves before and after 24-hour boil was run on a series of castings and/or moldings prepared from the various candidate resins. Several systems emerged which exhibited moisture resistance superior to that of commercially available epoxy systems. Process and cure studies were carried out on glass reinforced laminates which also exhibited excellent moisture

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resistance. Prepreg parameter and compatibility studies along with tape and laminate fabrication and testing were carried out on the most promising candidate systems. Severe difficulties were finally encountered in achieving both high quality laminates and adequate performance at elevated temperature. The difficulties were identified and alternate means of overcoming these problems were suggested.

FOREWORD

This Final Report was prepared by Aerotherm Division, Acurex Corporation, Mountain View, California and WRD, a Division of Whittaker Corporation, San Diego, California. Aerotherm assumed responsibility for the contract performance on September 12, 1975. This report was prepared for Air Force Contract No. F33615-74-C-5142, "Development of Improved Environmental Resistant Organic-Reinforced Materials Systems", Project 69CW.

The work was performed under the sponsorship of the Air Force Materials Laboratory (AFML/LC), Air Force Systems Command, Wright-Patterson Air Force Base, Ohio, under the direction of Mr. R. M. Neff and Lt. G. D. Hollingsworth. The manuscript of this report was released by the author in November 1975 for publication.

Efforts at WRD were conducted within the Advanced Composites Engineering Department, under the technical direction of Mr. M. G. Maximovich as Program Manager.

This report covers laboratory efforts from 1 June 1974 through 31 August 1975.

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SUMMARY

Commercially available matrix resins were identified and a list of resin criteria along with target goals for properties and performance were established. A screening program consisting of dimensional change measurements, water pickup determinations, and TMA curves before and after 24-hour boil was run on a series of castings and/or moldings prepared from the various candidate resins. Several systems emerged which exhibited moisture resistance superior to that of commercially available epoxy systems.

Process and cure studies were carried out on glass reinforced laminates which also exhibited excellent moisture resistance. Prepreg parameter and compatibility studies along with tape and laminate fabrication and testing was carried out on the most promising candidate systems.

Severe difficulties were finally encountered in achieving both high quality laminates and adequate performance at elevated temperature. The difficulties were identified and alternate means of overcoming these problems were suggested.

SECTION 1

INTRODUCTION

The combination of high strength and stiffness with light weight makes organic matrix advanced composites hardware extremely attractive for today's modern Air Force weapons systems. Accordingly, such materials are being employed on such programs in ever increasing quantities. Unfortunately, it has been shown that present epoxy matrix composites gradually absorb moisture, become plasticized, and lose a significant measure of their performance at elevated temperatures. Strength critical laminates often become matrix critical; elevated temperature compressive strengths are severely degraded.

As a result of this phenomenon, currently designed composite structures incorporate significant overdesigns and safety factors. This not only results in decreased payload capability but additionally leads to excessive quantities of expensive composite material being required for such hardware.

It therefore follows that improved moisture resistant composite materials would lead to significant cost and weight reductions, as well as enhancing the survivability of composite structures throughout their required service life. The objective of this program was to develop graphite and boron reinforced organic matrix composite systems that exhibit outstanding environmental aging characteristics. The investigation was limited to commercially available resins in order to have maximum impact on existing and newly emerging Air Force programs.

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SECTION 2

TASK DESCRIPTION

The program was divided into three separate efforts or phases. Phase I dealt with materials screening, selection, and initial characterization. Phase II involved the development of prepreg and an evaluation of the compatibility and capability of the selected matrix resins. In Phase III, significant engineering and environmental aging data were to be generated on the final composite systems. The individual tasks are described in detail as follows:

PHASE I - INITIAL RESIN, PREPREG, AND COMPOSITE EVALUATION

Task 1 - Resin Criteria. A set of resin criteria shall be established. Such criteria deal with the handling, processability, mechanical properties, and all appropriate characteristics of candidate resins as they apply to prepreg production, composite fabrication, and laminate performance. Target goals shall be set for the various criteria.

Task 2 - Resin Selection. A series of candidate resins shall be selected from commercially available polymer systems. The criteria established in Task 1 shall be used for the selection. Commercial state-of-the-art epoxy resin systems shall be included to provide base line comparative data.

Task 3 - Initial Screening Tests. Castings and/or moldings of the candidate systems shall be prepared. Initial screening tests shall consist of TMA, water pickup, and dimensional change after 24-hour water boil exposure. Additional factors, i.e. the ability to meet the criteria as defined in Task 1, shall also be carefully considered. The most promising candidates shall be selected for continued evaluation.

Task 4 - Process and Cure Studies. Additional castings or moldings shall be fabricated from the selected candidates and evaluated at room and elevated temperatures before and after various environmental exposures. Optimum processing parameters, cure cycles, postcures, etc., shall be determined.

Task 5 - Prepreg Parameter and Compatibility Studies. The compatibility of the candidate resins with boron and graphite reinforcements shall be determined. Prepregging parameters shall be determined, and methods shall be adapted to state-of-the-art prepregging procedures.

Task 6 - Tape and Laminate Fabrication and Tests. Prepreg shall be produced and characterized, curing conditions established, and laminates fabricated. Limited mechanical property and environmental exposure testing shall be conducted.

Task 7 - Optimization Studies. The most promising materials shall be optimized through additional prepregging, cure, and fabrication studies. The two most promising systems shall be selected for Phase II studies.

Task 8 - Preliminary Specifications. Preliminary specifications for the prepreg, the prepregging operations, and the composite fabrication shall be prepared for the two candidate systems.

PHASE II - PREPREG AND COMPOSITE FABRICATION

Task 1 - Graphite and Boron Prepregging. Graphite and boron reinforced tape shall be produced on manufacturing equipment at a commercial prepregging facility. WRD personnel shall be available to observe and direct the production of tape. All deviations from standard epoxy practice shall be noted and the total impact on cost, quality, reproducibility, time, etc., shall be determined.

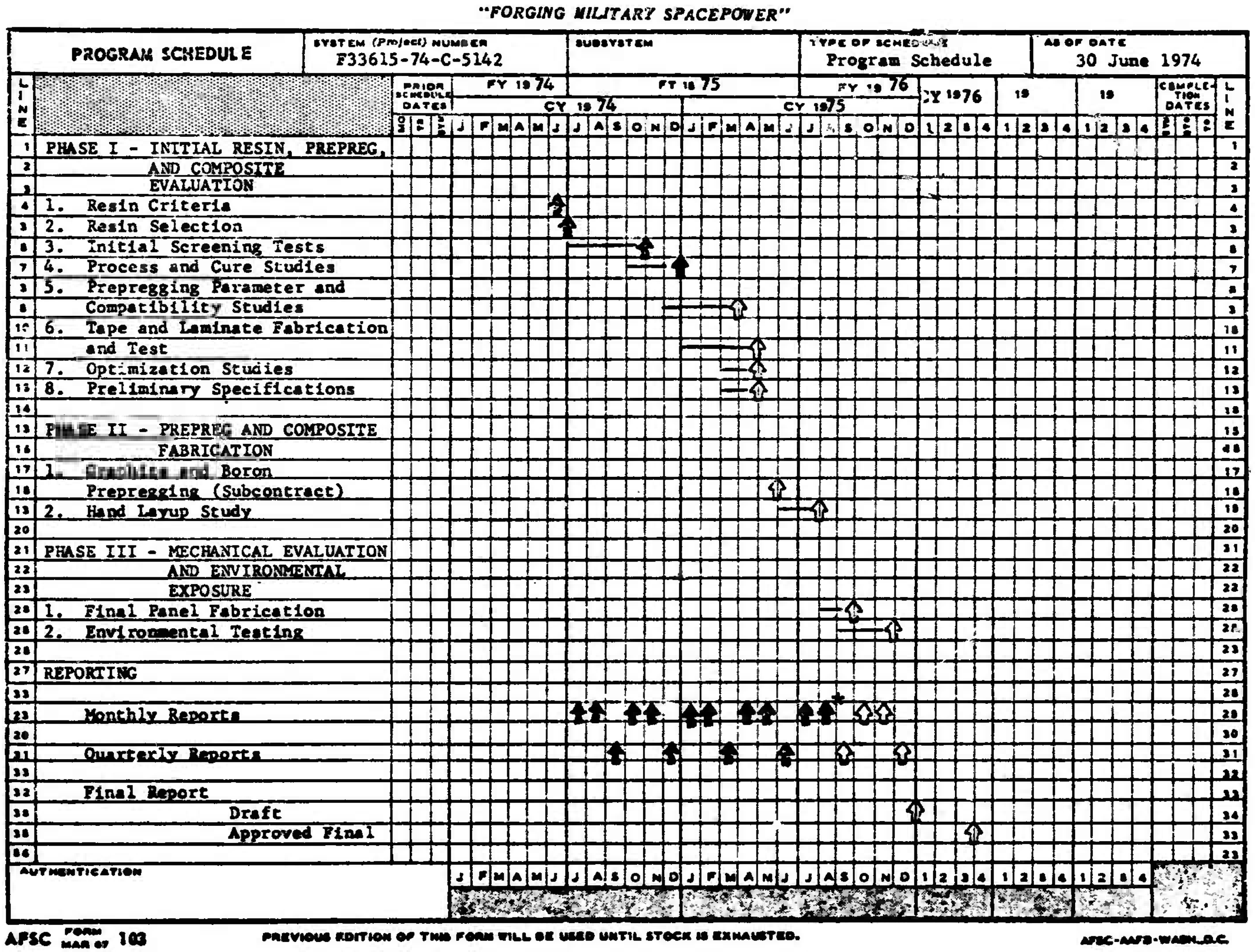
Task 2 - Hand Layup Study. The various material combinations shall be thoroughly evaluated through a hand layup study. All criteria from Phase I shall be considered and the materials and process specifications generated in Phase I shall be revised as appropriate.

PHASE III - MECHANICAL EVALUATION AND ENVIRONMENTAL EXPOSURE

Task 1 - Final Panel Fabrication. All panels required for the mechanical and environmental testing in Phase III shall be fabricated, using the material specifications produced in Phase II.

Task 2 - Environmental Testing. Various environmental exposures shall be carried out with the extensive generation of mechanical property data.

The milestone report showing the planned activities for the entire program as well as actual accomplishments is shown in Figure 2-1.



"FORGING MILITARY SPACEPOWER"

PROGRAM SCHEDULE

SYSTEM (Project) NUMBER F33615-74-C-5142

SUBSYSTEM

TYPE OF SCHEDULE Program Schedule

AS OF DATE 30 June 1974

1. PHASE I - INITIAL RESIN, PREPREG, AND COMPOSITE EVALUATION
4. 1. Resin Criteria
5. 2. Resin Selection
6. 3. Initial Screening Tests
7. 4. Process and Cure Studies
8. 5. Prepregging Parameter and Compatibility Studies
10. 6. Tape and Laminate Fabrication and Test
12. 7. Optimization Studies
13. 8. Preliminary Specifications
15. PHASE II - PREPREG AND COMPOSITE FABRICATION
17. 1. Graphite and Boron Prepregging (Subcontract)
19. 2. Hand Layup Study
21. PHASE III - MECHANICAL EVALUATION AND ENVIRONMENTAL EXPOSURE
24. 1. Final Panel Fabrication
25. 2. Environmental Testing
27. REPORTING
29. Monthly Reports *
31. Quarterly Reports
33. Final Report
34. Draft
35. Approved Final

AUTHENTICATION

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Figure 2-1. Milestone Report.

*Major Technical Summary prepared per Air Force request.

SECTION 3

CRITERIA SELECTION

A list of resin criteria was established to provide a basis for selecting and discriminating among the various commercially available polymer systems. Simultaneously, a target or goal was established for each criteria to identify the specific properties or performance requirements for the candidate resins. The list of resin criteria includes both necessary and desirable but not essential characteristics. While it may be unrealistic to expect one resin system to meet all criteria, the best balance or combinations of criteria met will determine the candidates selected for work in Phases II and III. For this reason, resins that meet the necessary criteria are included even though they will obviously be lacking in some desirable characteristics.

3.1 NECESSARY CRITERIA

(A) Superior Moisture Resistance and Environmental Stability - The goal is to achieve 80% or better retention of elevated temperature performance after extended ambient aging or accelerated high humidity testing.

(B) Excellent Mechanical Properties - Final laminates should exhibit properties equal to or exceeding present state-of-the-art T-300 reinforced epoxy systems. The goal will be to achieve at least the performance exhibited by T-300/5208 graphite epoxy at R.T. and 375°F.

(C) Low Void Content Composite Capability - The goal is to achieve 1% or less voids in the cured laminate.

(D) Compatibility with Reinforcements - The resins must wet the reinforcements well to achieve usable structural performance.

(E) Reasonable Shelf Life and Storage Requirements - The resin systems must have out-times and storage requirements compatible with prevailing industry shop capability to allow fabrication of large-production aerospace hardware.

(F) Compatibility with Prepregging Equipment - The resin must be compatible with present hot melt (preferably) or solvent prepregging equipment to allow large-scale commercial production of the final prepreg.

(G) Moderate Cure Temperatures - Cure cycles should be comparable to present epoxy matrix materials.

(H) Good Drape and Tack - The handling characteristics should be similar or equivalent to present epoxies. The combination of characteristics must allow the fabrication of complex/contoured structures.

(I) Cocurable - It is desirable that cocuring capability exist for the fabrication of honeycomb sandwich structures.

(J) Low Cost - The cost of the matrix resin and of the fabrication sequence must not significantly exceed that of present graphite/epoxy systems. Preferably, the cost of the final hardware should be lower than that of present parts.

3.2 DESIRABLE CRITERIA

(A) No Volatile Evolved During Cure - The goal is to achieve no evolution of volatiles. However, this may be compensated for if other mechanisms (i.e. a fusing to close voids and achieve <1% voids) are available to produce low void laminates.

(B) Toughness - The goal is to achieve elongation superior to present high temperature epoxy matrix systems. Transverse strain should be superior to that of 5208 or 3501 epoxy matrix systems.

(C) Low Shrinkage During Cure - Shrinking must be minimized and should be below that of 5208 or 3501 epoxy resins.

(D) Low Flow Capability - The goal is to minimize or even eliminate the need for complex bleeder systems. Inherent here is a prepreg resin content close to that of the finished composite.

(E) No Microcracking During Cure - Microcracking must be eliminated unless either a healing mechanism exists or it can be shown that mechanical properties, fatigue life, and environmental resistance are not significantly affected.

(F) Compatibility with Adhesives - The final composite surface should be suitable for adhesive bonding.

SECTION 4

CANDIDATE SELECTION

The candidate resin systems were selected from a broad spectrum of commercial polymer types. The following discussion covers the general classes of resins along with the specific commercially available material chosen:

4.1 BASELINE EPOXY RESINS

High performance state-of-the-art epoxy systems were selected to generate base line data for the screening program. The specific structure of the various commercial resins is proprietary and will not be investigated in this work. Recent studies (Ref. 1) have shown that the 3501B resin from Hercules and the 5208 resin from Whittaker Corporation, Narmco Materials Division, are superior composite matrix resins in their resistance to environmental aging. These two were therefore selected as standards for this study. Narmco 5209 was included as a typical example of a 250°F curing system for comparative purposes.

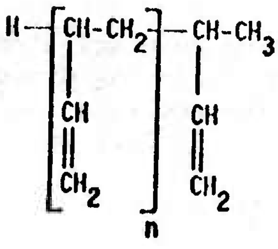
Epoxies selected: Hercules 3501
Narmco 5208
Narmco 5209

4.2 POLYBUTADIENE RESINS

A promising series of polymers that exhibit marked hydrophobic tendencies are the polybutadienes. In addition, the materials as a family exhibit low density, high chemical resistance, high U.V. resistance, good compressive strengths, and are generally easy to process. A general reaction is that of a diisocyanate with a 1,2-polybutadiene diol, followed by the free radical induced cyclization of the pendant vinyl groups. Unfortunately, one of the best sources for such resins, Firestone, discontinued this polymer line. However, good quality resins are available for this study.

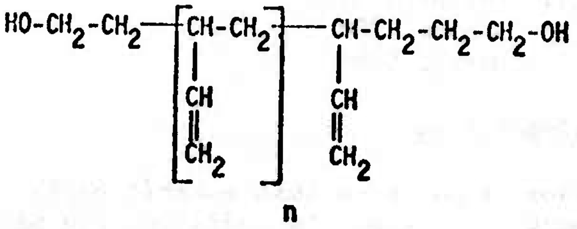
Polybutadiene resins selected: Ricon 100
Ricon 150
Ricon 431
Hystl G-1000
Hystl G-2000
Hystl G-3000
Hystl B-1000
Hystl B-2000
Hystl B-3000

Figure 4-1 gives the idealized structure for a B-series Hystl resin, which is a simple, unreactively terminated polybutadiene. The G-series resins are hydroxy terminated (see Figure 4-2), and may provide superior hydrolytic and oxidative stability, according to the manufacturer (Ref. 2).



$H-[CH(CH=CH_2)-CH_2]_n-CH(CH=CH_2)-CH_3$

Figure 4-1. Idealized Structure, Hystl B-Series Polybutadiene



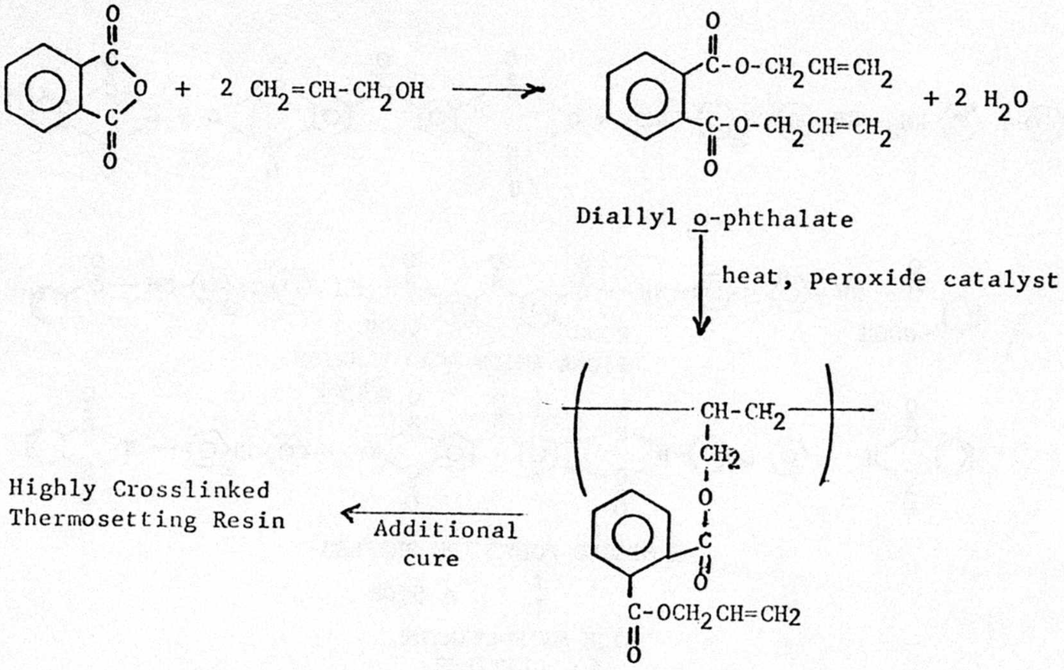
$HO-CH_2-CH_2-[CH(CH=CH_2)-CH_2]_n-CH(CH=CH_2)-CH_2-CH_2-CH_2-OH$

Figure 4-2. Idealized Structure, Hydroxy Terminated Polybutadiene

4.3 DIALLYLPHTHALATE (DAP) RESINS

Allylic resins are another promising family of materials. Much data has been generated since 1937 on these materials, which are formed by the copolymerization of unsaturated esters with other unsaturated monomers. A good review is given by Raech (Ref. 3). The general chemistry of the diallylphthalate is given in Figure 4-3. These resins are commercially available at a low cost (e.g., FMC Organic Chem. Div., Borden Chemicals, Sartomer Resins, Allied Chemical, etc., at ~$2.00 per pound).

The diallylisophthalate (DAIP) resin exhibits improved high temperature performance and is similarly available. The chemistry is the same, with the obvious isophthalate vs. o-phthalate structure present.



(1)

Figure 4-3. DAP Chemistry

The commercial products chosen for this study are

DAP	-	F.M.C.	DAP monomer Dapon 35 polymer
DAIP	-	F.M.C.	DAIP monomer Dapon M polymer

4.4 ADDITION POLYIMIDES

The addition type polyimides have been studied on numerous programs as an exciting development in high temperature resin chemistry. They have good thermal/oxidative stability, yet cure without the volatile evolution of conventional condensation polyimides. Perhaps the best known of them is the Ciba-Geigy P13N. This resin, however, does not lend itself to the fabrication of large parts, as a high heatup rate is required. The Ciba-Geigy P105A is an attractive alternative. As shown in Figure 4-4, no volatiles are released in cure. Additionally, slow heatup rates may be used. Unfortunately, recent OSHA regulations concerning the thiodianiline monomer required for P105A have resulted in Ciba-Geigy discontinuing this resin. It is therefore not included in the program.

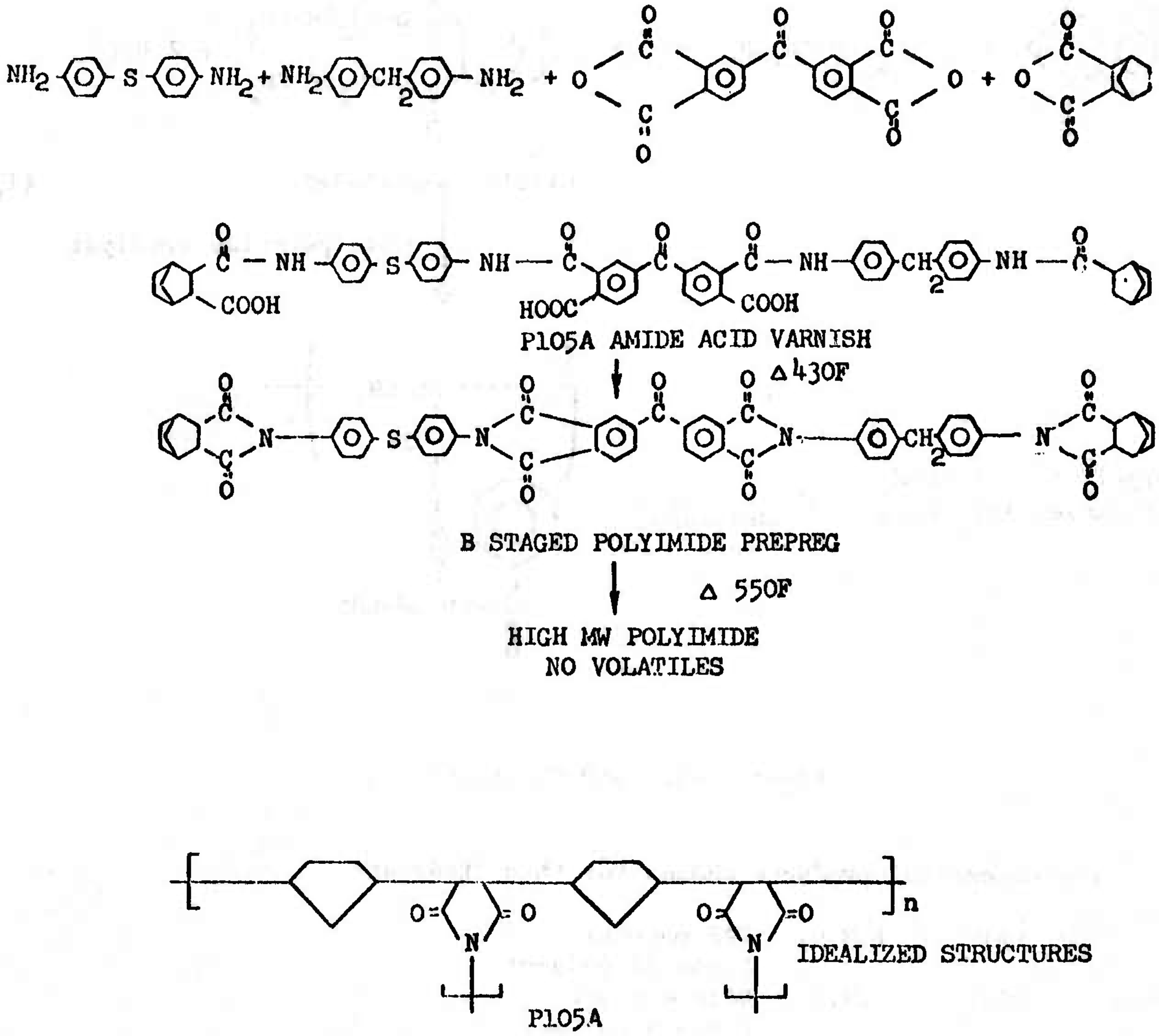
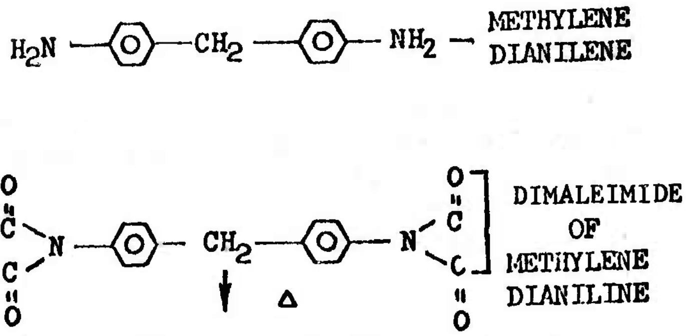


Figure 4-4. P105A Chemistry

Another candidate addition polyimide is the Kerimid 601, whose chemistry is given in Figure 4-5. This material exhibits many of the advantages of P105A, yet cures at more moderate temperatures. This resin was chosen for the program.

Additionally, Hexcel 580 was included. This is a proprietary material, but it is believed to be similar in nature to the 601 resin.

Addition Polyimides selected: Kerimid 601
Hexcel 580



CHAIN EXTENSION & CROSSLINKING VIA
VINYL POLYMERIZATION & MICHAELS
ADDITIONS; NO VOLATILES

Figure 4-5. Kerimid 601 Chemistry

4.5 THERMOPLASTIC POLYIMIDES

Another family of polyimides that avoids the problem of volatile evolution is the thermoplastic PI family. The disadvantage of high processing temperatures is more than compensated for by superb high temperature performance, excellent elongation and toughness, and postforming potential. Superior hydrolytic stability and low moisture sensitivity is claimed. Figure 4-6 shows NR150A, a candidate selected for this program.

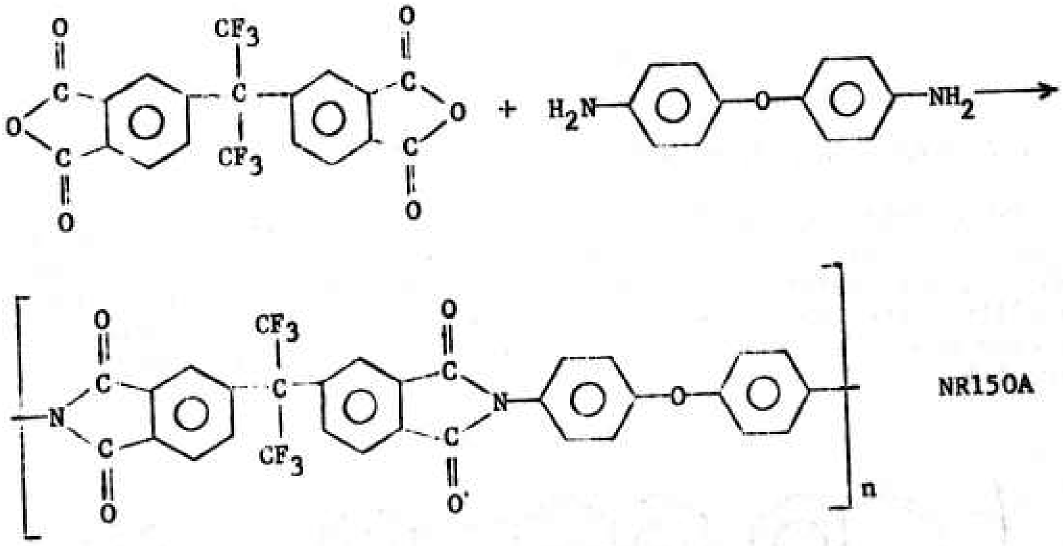


Figure 4-6. NR150A Chemistry

An additional proprietary resin, Upjohn 2080, was also selected as a candidate system.

Thermoplastic PI selected: Du Pont NR150A
Upjohn 2080

4.6 POLYSULFONES

These materials show great promise for applications in graphite and boron reinforced composite systems. Even though high processing temperatures are required, their thermoplastic nature opens the door to significant cost reductions. However, conventional processing methods are not suited to polysulfones; thermoplastic processing technology should be applied. Several commercially available resins were selected.

Polysulfones selected: Union Carbide P1700 polysulfone
ICI 200P polyethersulfone
ICI 300P polyethersulfone

All are inexpensive and readily available. The structure of the P1700 is given in Figure 4-7.

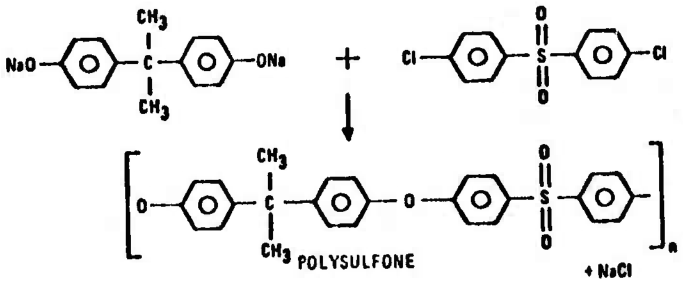


Figure 4-7. P1700

4.7 POLYPHENYLQUINOXALINE

The polyphenylquinoxaline (PPQ) resins are an attractive system. Outstanding thermal/oxidative stability, hydrolytic stability, elongation, toughness, and impact resistance are combined with thermoplastic processing capability. The PPQ 401 selected is commercially available from Whittaker Research and Development, and its structure is given in Figure 4-8.

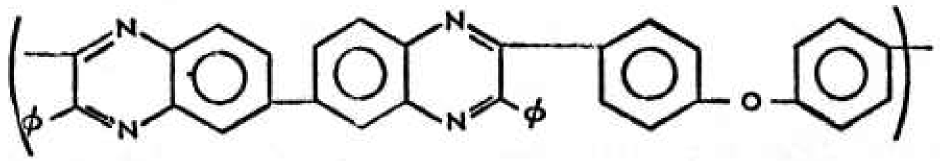


Figure 4-8. PPQ 401

4.8 AMIDE-IMIDE POLYMERS

These resins are commercially available from Amoco Chemicals. The manufacturer claims extremely long shelf life, excellent high temperature and environmental aging characteristics, and relatively low cost. Two proprietary candidates were obtained from Amoco for this study.

Polyamide-imide resins selected:	Amoco Torlon 2000
	Amoco Torlon 4000

4.9 POLYESTER RESINS

Numerous high performance, low cost polyesters have been developed for use with glass reinforcement. These have been largely overlooked by the aerospace industry as it concentrated on epoxy systems. Many potential candidates were found to be no longer available, but a significant number of high performance resins of varying chemistries were obtained for this study. The resins are proprietary and vary significantly in structure (i.e. chlorinated polyesters, triallylcyanurate polyesters, etc.), so no typical polymer can be shown.

Polyesters selected:	American Cyanamid Laminac 4232
	Hooker Chemical Co. Hetron 197
	Hooker Chemical Co. Hetron 197P
	Koppers V-7001-10
	Koppers V-7000-15

4.10 CYANATE ESTERS

Cyanate ester polymers are commercially available from Whittaker Corporation, Narmco Materials Division. These proprietary resins exhibit outstanding mechanical properties combined with excellent thermal/oxidative stability.

The XRS-10500 cyanate ester was selected as a candidate for this study.

4.11 PHENYL SILANE RESINS

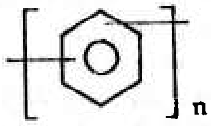
A commercially available phenyl silane resin, Resinox SC 1013, from Monsanto, was selected as a candidate. The phenyl silane family of resins show extremely low moisture pickup, as well as excellent high temperature performance.

4.12 FURFURYL ALCOHOL

A commercially available proprietary resin, Hetron 800, was obtained from Hooker Chemical for this study. The manufacturer claims excellent corrosion resistance, moisture resistance, and high temperature performance from this resin system. It is formulated as a low cost laminating resin, and is therefore an interesting candidate for the study.

4.13 POLYPHENYL RESINS

Another proprietary system, DP25-10, was obtained from Ironsides Resins, Inc. Excellent elevated temperature properties and environmental resistance are claimed. The polyphenyl structure,



should certainly be stable and generally hydrophobic in nature.

4.14 DIPHENYL OXIDE RESINS

Another proprietary candidate from Ironsides Resins, Resin 6481 (formerly DP4-94), was selected as a candidate. This laminating resin is said to be a diphenyl oxide system and should exhibit low moisture sensitivity and good thermal/oxidative performance.

In summary, a large and varied assortment of candidate systems were selected. All are commercially available and require no resin development efforts. Table 4-1 summarizes all candidate systems for the study.

TABLE 4-1

CANDIDATE RESINS

Resin	Type	Manufacturer
3501	baseline epoxy	Hercules, Inc.
5208	baseline epoxy	Narmco Materials Inc.
5209	baseline epoxy	Narmco Materials Inc.
Ricon 100	polybutadiene	The Richardson Co.
Ricon 150	polybutadiene	The Richardson Co.
Ricon 431	polybutadiene	The Richardson Co.
Hystl B-1000	polybutadiene	Hystl Development Co.
Hystl B-2000	polybutadiene	Hystl Development Co.
Hystl B-3000	polybutadiene	Hystl Development Co.
Hystl G-1000	polybutadiene	Hystl Development Co.
Hystl G-2000	polybutadiene	Hystl Development Co.
Hystl G-3000	polybutadiene	Hystl Development Co.
DAP	diallyl phthalate monomer	FMC
Dapon M	DAIP polymer	FMC
DAIP	diallyl isophthalate monomer	FMC
Dapon 35	DAP polymer	FMC
Dapon M/DAIP, 70/30 Blend	DAIP monomer/polymer	FMC
Kerimid 601	addition polyimide	Rhodia, Inc.
Hexcel 580	addition polyimide	Hexcel
NR-150A	thermoplastic polyimide	Du Pont
2080	thermoplastic polyimide	Upjohn
P1700	polysulfone	Union Carbide
200P	polyethersulfone	ICI
300P	polyethersulfone	ICI
Torlon 2000	amide-imide	Amoco
Torlon 4000	amide-imide	Amoco
PPQ 401	polyphenylquinoxaline	WRD
Laminac 4232	polyester	American Cyanamid
Hetron 197	polyester	Hooker Chemical
Hetron 197P	polyester	Hooker Chemical
V-7001-10	polyester	Koppers
V-7000-15	polyester	Koppers
XRS-10500	cyanate ester	Narmco Materials Inc.
Resinox SC 1013	phenyl silane	Monsanto
Hetron 800FA	furfuryl alcohol	Hooker Chemical
DP 25-10	polyphenyl	Ironsides Resins, Inc.
Resin 6481	diphenyl oxide	Ironsides Resins, Inc.

SECTION 5

RESIN SCREENING

Work was initiated on screening 35 candidate resins, with several resins added as they became available. Moldings or castings of the neat resins were made, using manufacturers' recommendations. The fully cured resin specimens were then sectioned into specimens for thermomechanical analysis (TMA) and dimensional change/moisture pickup investigations.

5.1 DIMENSIONAL CHANGE/MOISTURE PICKUP

Duplicate 1.00 in. by .25 in. by .25 in. sections were machined for dimensional change determination. Dimensional measurements were taken along with percent moisture gain by weight before and after a 24-hour water boil on the duplicate specimens. Table 5-1 summarizes the effects of a 24-hour water boil on all specimens.

5.2 THERMOMECHANICAL ANALYSIS (TMA)

The TMA screening test measures the compression properties of the specimen as a function of temperature. A small diameter weighted probe is placed in contact with a resin sample which is heated at a uniform rate. Vertical probe displacement as a function of temperature is recorded. This test simulates actual load/temperature couples found in service, and is therefore a useful tool for the quick, efficient, and inexpensive screening of candidate resins. The TMA is a plug-in module for the Du Pont 900 Thermal Analyzer, which is widely used for differential thermal analysis (DTA) and thermal gravimetric analysis (TGA) of polymers. The probe makes up the core of a differential transformer which provides a DC output proportional to linear displacement of the core resulting from the specimen expanding or deflecting under the load. This deflection versus temperature is plotted by an x-y recorder. Figure 5-1 is a schematic of the TMA apparatus. During this study, the maximum possible weight (100 g) was used to load the sample. Since the probe is about .025 in. in diameter, each sample was subjected to about 450 psi. Duplicate cubes, .25 in. on each side, were used for TMA specimens. The cubes were cut to final size before boil, which is the more severe method of evaluation.

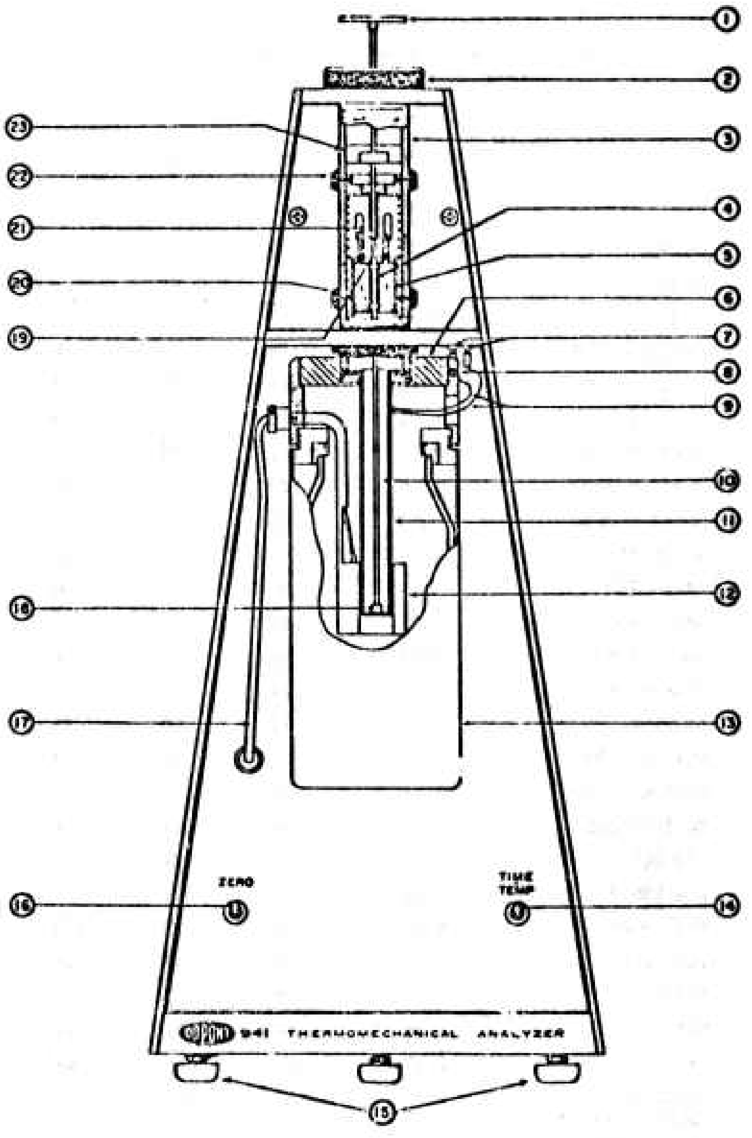
Initially, both specimens were run within 24 hours of boil exposure (storage under water at room temperature until testing). However, during the course of the study it was found that no change could be detected on the TMA curve after days or weeks of such storage. At least one specimen of each duplicate pair was tested as soon as possible after boil exposure to insure the accuracy of the screening efforts. The second replicate was tested after varying intervals of underwater room temperature storage as scheduling on the TMA instrument permitted. By following this procedure, the total time required for the screening portion of this program is significantly reduced. Excellent reproducibility of TMA curves was an assuring characteristic of these tests.

TABLE 5-1

RESIN WATER ABSORPTION AND DIMENSIONAL CHANGE (24-HOUR WATER BOIL)

Resin	Water Absorption (%)	Length Change (%)	Width Change (%)	Height Change (%)
3501B	3.39	.59	1.1	1.1
5208	1.93	.36	.44	.51
5209	3.20	.40	1.15	1.15
Hystl B-1000	.14	-.04	-.04	-.04
Hystl B-2000	.185	.01	.00	.02
Hystl B-3000	.22	.00	-.02	-.08
Hystl G-1000	.29	-.06	-.14	.00
Hystl G-2000	.49	.02	.01	.07
Hystl G-3000	.30	-.04	.02	.02
Ricon 100	.13	.01	-.07	.04
Ricon 150	.16	.00	.02	-.02
Ricon 431	.098	.00	.06	.04
Kerimid 601	3.90	.66	.73	.68
Hexcel 580	4.51	.77	.70	.73
NR-150A	2.02	.17	.20	.25
Torlon 2000	2.44	.52	.62	.61
Torlon 4000	2.06	.51	.43	.62
Torlon 4000	1.86	.76	.40	.29
PPQ 401	1.67	.10	.24	.38
XSR-10500	1.68	.19	.44	.46
ICI 200P	1.69	.35	.29	.26
ICI 300P	1.81	.28	.39	.40
P1700	.87	.19	.15	.15
DAIP	2.24	.54	.92	.72
DAP	1.98	-.09	.64	.45
Dapon M (DAIP polymer)	1.72	.35	.41	.42
Dapon M/DAIP, 70/30 blend	1.31	.36	.35	.25
Resinox SC 1013	1.21	.17	.20	.32
Resinox SC 1013 (high temp. cure)	4.78	.42	.20	.81
DP 25-10	1.34	.21	.15	.30
Resin 6481	1.64	.30	.20	.30
Hetron 197	1.19	.16	.20	.25
Hetron 197P	1.08	.21	.20	.20
Hetron 800FA (furfuryl alcohol, HCl cure)	3.12	.31	.35	.30
Hetron 800FA/800L catalyst	4.10	.30	.56	.48
Laminac 4232	4.14	1.38	1.45	1.10
V-7000-15	1.64	.47	.43	.60
V-7001-10	2.89	.97	.95	.85

NOTE: All values are the average of duplicate determinations.



Item	Description	Item	Description
1	Weight tray	12	Heater assembly
2	Retainer nut	13	Dewar
3	Head assembly	14	Time-Temp. switch
4	LVDT core	15	Adjustable legs
5	LVDT	16	Zero switch
6	Dewar cap	17	Heater lead wire
7	Thermocouple connectors	18	Sample
8	Set screws	19	Shaft assembly (upper)
9	Thermocouple wires	20	Transducer Position control
10	Shaft assembly (lower)	21	Zero spring
11	Sample holder tube	22	Probe Position control
		23	Upper Bearing guide

Figure 5-1. Thermomechanical Analyzer

Table 5-2 summarizes the results of the TMA testing. A curve for each specimen (before and after 24-hour water boil) is included in the Appendix.

5.3 DISCUSSION OF SCREENING TEST RESULTS

Most impressive are the polybutadiene systems, which appear totally unaffected by water boil exposure. It is also interesting to observe that the PPQ 401, NR-150A polyimide, P1700 polysulfone, Hetron 197P polyester, and the two Koppers polyesters, V-7000-15 and V-7001-10, absorb measurable moisture but remain apparently unaffected in mechanical performance at elevated temperature. They exhibit performance significantly superior to the baseline epoxy systems included in the program.

The Hetron 197 performed well, with a slightly higher TMA failure than the 197P version. The Hetron 800FA (furfuryl alcohol), HCl cure, is another promising polymer type. This resin picked up moisture, but elevated temperature performance was unaffected. The 800FA/800L performed in a similar manner. Some porosity was found and is tentatively believed to be the cause of the relatively high moisture pickup. However, as with the 800FA/HCl cure, no change of elevated temperature performance could be found.

The high temperature cure required to get good performance from the phenyl silane (Resinox SC 1013) precluded its use as a candidate for further study. In addition, this cure resulted in increased porosity and a high level of water absorption.

Perhaps the most unexpected results came from the Dapon M/DAIP based polymer. Castings from the DAIP monomer did not exhibit outstanding performance, but those from the Dapon M polymer exhibited much lower moisture pickup, a 50°C higher TMA failure point, and were unaffected by 24-hour boil. Evidently a significantly different final polymer is formed when Dapon M is used.

A spectacular test resulted when the ICI 200P and 300P polyethersulfones were run after water boil. The specimens literally "popped" like popcorn with the results shown in Figure 5-2. There appears to be a real problem of moisture sensitivity with these materials.

The evaluation of the 70/30 mix of Dapon M/DAIP monomer was also run, since this combination exhibited extremely attractive handling characteristics (i.e. drape, tack, etc.) when used for the production of prepreg materials. Here again the results were outstanding, and essentially as good as those obtained with Dapon M only. Evidently the final polymer more closely resembled that obtained from Dapon M rather than from DAIP monomeric starting material.

TABLE 5-2

EFFECT OF 24-HOUR WATER BOIL ON TMA FAILURE TEMPERATURE

Resin	Initial Failure Temperature (°C)	Failure Temperature After 24-Hour Water Boil (°C)
3501B	210	180
5208	220	195
5209	170	60
Hystl G-1000	~350	No change observed.
Hystl G-2000		
Hystl G-3000		
Hystl B-1000		
Hystl B-2000		
Hystl B-3000		
Ricon 100	~350	No change observed.
Ricon 150		
Ricon 431	375 - 390	375 - 380
Kerimid 601	355	160 - 240
Hexcel 580	370	95 - 110
NR-150A	280	No change observed.
Torlon 2000	265	200
Torlon 4000	250	195 - 220
PPQ 401	265	No change observed.
XSR-10500	245	150 - 160
ICI 200P	175	Blows up at 175°C when loaded after boil.
ICI 300P	200 - 220	Blows up at 203°C after boil.
P1700	160	No change observed.
DAP	300	240 - 260
DAIP	305	220 - 265
Dapon M	355	No change.
Dapon M/DAIP, 70/30 Blend	355	350
DP 25-10	210	185
Resin 6481	255	225
Hetron 197P	310	305
Hetron 197	320	310 - 320
Hetron 800FA (HCl Cure)	235	No change.
Hetron 800FA/800L Catalyst	258	No change.
Laminac 4232	305 - 310	115 - 125
V-7000-15	355	340 - 350
V-7001-10	325 - 340	315 - 320
Resinox SC 1013	120	120
Resinox SC 1013 High Temp. Cure	425 - 440	425 - 430

NOTE: All values are the average of duplicate determinations.

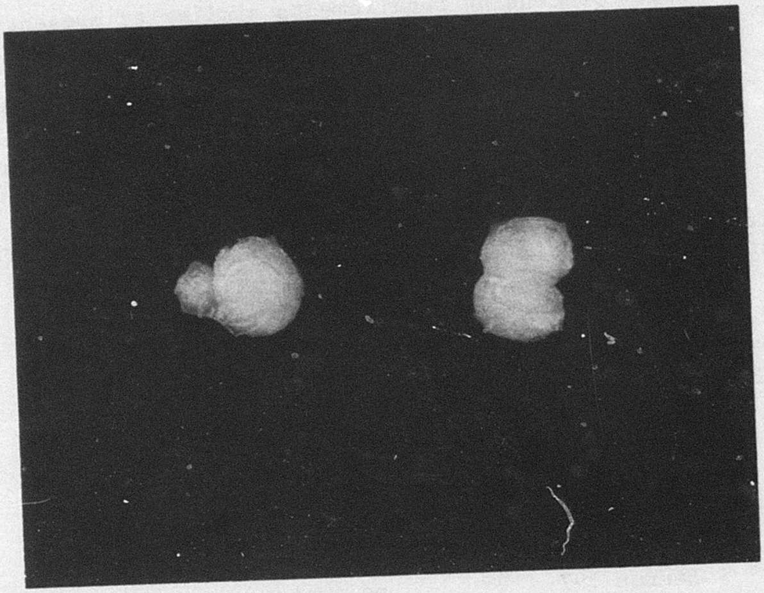


Figure 5-2. 200P and 300P Polyethersulfone, 1/4 in. Cubes after Water Boil and TMA Testing

SECTION 6

SELECTION OF CANDIDATES FOR PROCESS AND CURE STUDIES

A series of candidate resin systems were selected for process and cure parameter studies. In order to qualify, a resin must have exhibited:

(1) Moderate processing requirements similar to present state-of-the-art epoxies.

(2) Moderate to low moisture pickup and dimensional change.

(3) Little or no shift of the TMA failure temperature after 24-hour water boil.

Table 6-1 lists the candidates initially selected for further investigation.

TABLE 6-1

INITIAL CANDIDATE RESINS,
PROCESS AND CURE PARAMETER STUDIES

Resin	Type
Hetron 197	polyester
Hetron 197P	polyester
Koppers V 7000-15	polyester
Hystl B-3000	polybutadiene, unreactive termination
Hystl G-3000	polybutadiene, OH terminated
Dapon M	diallylisophthalate
Dapon M/DAIP, 70/30 Blend	diallylisophthalate
Hetron 800FA	furfuryl alcohol
Hetron 800FA/800L Catalyst	furfuryl alcohol

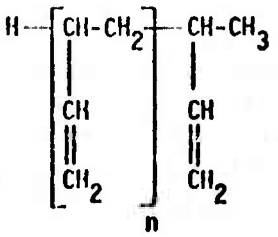
The selection of most of these candidate resins was quite straightforward after a review of the results of the screening program. The following is a brief discussion of the candidate systems.

6.1 POLYESTER RESINS

The three polyesters, Koppers V 7000-15, Hetron 197, and Hetron 197P, all exhibited great promise in screening. High TMA failure temperature, low moisture pickup, low cost, and moderate curing requirements are attributes shared by this series of materials. In addition, they are designed for use in composites (i.e. glass reinforcements) and should be readily applied to graphite composites. The potential shortcomings are shrinkage (microcracking) and transverse characteristics. Both the 197 and 197P were included in initial cure and process parameter studies. The intention was to select the resin showing superior handling, with the alternate to be held in reserve.

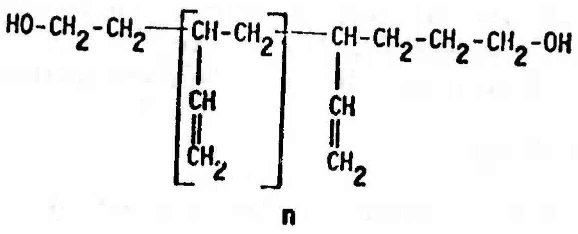
6.2 POLYBUTADIENE RESIN SYSTEMS

The choice of the Hystl B-3000 and G-3000 polybutadiene systems was based on a number of considerations. The polybutadiene polymers as a family are hydrophobic, process readily at moderate temperatures and pressures, and exhibit excellent electrical, mechanical, and thermal properties. The Hystl materials were selected as having good commercial availability and a fairly well defined chemistry. The B-series (unreactively terminated as shown in Figure 6-1) and the G-series (hydroxyl-terminated as shown in Figure 6-2) were selected to permit the greatest range of curing and processing options for future work. The high molecular weight (3000) type polymer was chosen to minimize the sometimes excessive tack of these systems, and to obtain greater control over molecular weight and the course of the chemical reaction.



$$\mathrm{H}-\left[\begin{array}{l}\mathrm{CH-CH_2}\\ | \\ \mathrm{CH}\\ \| \\ \mathrm{CH_2}\end{array}\right]_n-\begin{array}{l}\mathrm{CH-CH_3}\\ | \\ \mathrm{CH}\\ \| \\ \mathrm{CH_2}\end{array}$$

Figure 6-1. Hystl B-Series (unreactively terminated) Idealized Chemical Structure.



$$HO{-}CH_2{-}CH_2{-}\left[\underset{\underset{CH_2}{\|}}{\underset{|}{CH}}{-}CH_2\right]_n{-}\underset{\underset{CH_2}{\|}}{\underset{|}{CH}}{-}CH_2{-}CH_2{-}CH_2{-}OH$$

Figure 6-2. Hystl G-Series (hydroxyl-terminated) Idealized Chemical Structure.

The Ricon 431 polybutadiene exhibited the virtues of the Hystl resins but avoided their handling problems. Good drape and tack were apparent in contrast to the excessively sticky and tacky Hystl polybutadienes. The Ricon 431 also exhibited a stable, low viscosity melt phase, making it extremely attractive for hot melt coating techniques. It proved an extremely attractive resin and was therefore included in the program.

6.3 DIALLYLISOPHTHALATE (DAIP) TYPE RESINS

The DAIP based systems have many attractive characteristics, including long out-time capability, good handling, low cost, and potential high temperature performance. The initial evaluation based on DAIP monomer proved disappointing, in that significant moisture pickup and TMA peak shift occurred. Surprisingly, as noted above, when the starting material was Dapon M, a DAIP based polymer, outstanding moisture resistance was eshibited. A 70/30 blend of Dapon M/DAIP monomer showed similar moisture resistance as well as outstanding handling characteristics. Both forms of DAIP based systems were carried in the program through initial process and cure studies.

6.4 FURFURYL ALCOHOL RESINS

The Hetron 800FA, cured with HCl or with Hetron 800L catalyst, also showed outstanding moisture resistance. Porosity was higher than desired in resin castings, but optimized processing may minimize the problem. Both catalyzed versions were included in early stages of process and cure studies with the intention of selecting the version with better handling, flow, etc.

SECTION 7

PROCESS AND CURE PARAMETER STUDY

7.1 INITIAL SPECIMEN FABRICATION

The original work statement called for a study involving specimens prepared from large castings and/or moldings of neat resins. It soon became apparent that this approach was not viable. Attempts at fabricating the castings met with little success, as it was nearly impossible to avoid excessive flaws. Such flaws rendered the specimens useless for mechanical property testing. Additional investigations revealed that large, high quality castings or moldings had never been prepared from some of the candidate materials. Rather than devote large amounts of time and funding to a task of such dubious value, an alternate approach was adopted with the approval of the Air Force program monitor. Laminates filled with 181 E glass Volan finish reinforcement were fabricated.

The first goal was to define processing and cure cycles that allowed fabrication of well consolidated, high performance laminates. The target was to achieve a fiber volume of approximately 60% with good wetting and consolidation. It was recognized that some difficulties could arise concerning resin/glass interface effects and/or sizing compatibility. Should this have proved the case with any resin, work with that candidate was to be shifted to Thornel 300 reinforced panels. Every attempt was made, however, to utilize glass reinforcement for the task in order to minimize the material costs.

The following discussion summarizes the initial handling and fabrication characteristics of the various candidate materials.

7.2 GLASS LAMINATE FABRICATION STUDIES

7.2.1 Hetron 197 and 197P Polyesters - Both resins handled well, with the 197 wetting more readily. Good quality laminates were made with no major difficulty. Prepreg was quite tacky, drape was high. Some B-staging work was done to reduce the tack. The 197 in general produced superior laminates by a small but definite margin. At this point, further efforts concentrated on the 197 only; the 197P was held as a backup system in the event unforeseen difficulties arose with the 197.

7.2.2 Koppers V 7000-15 Polyester - The above remarks for the Hetron 197 resins also describe the V 7000-15. Good laminates were readily fabricated; wetting, handling, etc., were excellent. The prepreg was excessively tacky and, as with the 197, some work on B-staging was done.

7.2.3 Hystl B-3000 and G-3000 Polybutadiene Resins - Good laminates were fabricated, but with more difficulty than with the polyesters. Wetting appears adequate with sufficient time allowed for the process. Both systems are quite tacky, with G-3000 displaying less tack and drape. Further reduction by B-staging is still desirable. The G-3000 also cures more readily, but both resins presently require a long, slow heat-up, cure, and cool-down to give high quality laminates. Significant shrinkage does occur. Even with these difficulties, however, the extremely hydrophobic nature of these two resins makes them good candidates for continued work.

7.2.4 In contrast to the Hystl PBD polymers, the Ricon 431 exhibited extremely attractive handling characteristics. Prepreg with ideal drape and tack was readily prepared.

7.2.5 Dapon M Diallylisophthalate (DAIP) Polymer - The Dapon M wets and impregnates readily with solvent present. Once the solvent is removed, the prepreg becomes stiff and exhibits essentially no tack at R.T. Moderate heating restores adequate drape and tack. A more serious problem is the limited flow and "gummy" nature of the polymer during cure. The retention of solvent would alleviate the flow problem as well as improving drape and tack. However, this is an unacceptable approach, since volatiles (solvent) would be evolved during cure, making the entire system difficult to use in the fabrication of thick and/or complex hardware. A more promising approach was to blend 70 parts Dapon M with 30 parts DAIP monomer. The resulting prepreg had ideal drape and tack as well as excellent flow at RT.

7.2.6 Hetron 800FA, HCl Catalyst and 800L Catalyst, Furfuryl Alcohol - The 800FA showed good wetting and handling with both catalyst systems. However, it was far easier to control catalyst levels and achieve uniform mixing with the 800L catalyst. The HCl catalyzed version proved tricky to handle and exhibited extremely limited pot life. As a result, further work will concentrate on the 800L catalyzed version. Good prepreg with somewhat excessive drape and tack was achieved with the 800FA/800L system, and high quality laminates were fabricated.

Glass laminates were satisfactorily fabricated with all the candidate systems. Water pickup, dimensional change, shear strength, and flexural strength were determined before and after 24-hour water boil and 30-day, 120°F, 95% relative humidity exposures. The flexural strength and modulus determinations were run at room temperature and 350°F.

The results of these tests and the preliminary cure schedules used are given in the following sections.

7.3 MOISTURE RESISTANCE TESTING

Two environmental exposures were run on all candidate systems. The first was a 24-hour boil, the second a 30-day, 120°F, 95% relative humidity exposure. Initial physical and mechanical properties were determined for all laminates. After environmental exposure, the water pickup, dimensional change, flexural strength, and flexural modulus were determined, the latter two mechanical properties at both room temperature and 350°F.

Table 7-1 gives physical properties, water pickup, and dimensional change, while Table 7-2 summarizes the results of the 24-hour boil and the 30-day humidity testing on the glass reinforced laminates. It appears that interface problems related to the glass reinforcement, glass sizing, etc., were significant and cannot be discounted. Note the increased water pickup on all laminates as opposed to neat resin data; this may be due to wicking, etc.

7.4 CURE SCHEDULES

The following preliminary cure schedules were used in fabricating the glass reinforced laminates discussed above. At this stage of development, press cures were used exclusively to fabricate all laminates. This proved far quicker, cheaper, and convenient than autoclave processing. The cures are as follows.

7.4.1 Hetron 197 - The laminate was fabricated in a 6 in. x 6 in. aluminum tray mold, using 181 E glass (Volan) reinforcement and Hetron 197 resin catalyzed with 0.5 phr BZQ-50 peroxide catalyst. The prepreg was placed under contact pressure and heated to 150°F. After a 35-minute hold, 100 psi was applied and the temperature raised as follows:

Total Cure Time	Temperature
45 minutes	225°F
75 minutes	300°F
105 minutes	350°F.

The part was held 1 hour at 350°F, 100 psi, and cooled.

7.4.2 Koppers V 7000-15 - As with Hetron 197, a 6 in. x 6 in. tray mold was used; the resin catalyzed with 0.5 phr BZQ-50 peroxide catalyst. The laminate was taken under constant pressure to 150°F. After 45 minutes, 100 psi was applied. The heat-up was as follows:

Total Cure Time	Temperature
1 hour	225°F
1½ hours	300°F
2 hours	350°F

Hold 1 hour at 350°F, 100 psi, then cool slowly.

7.4.3 Hystl B-3000 - The resin was catalyzed with 0.5 phr L-101 catalyst. The cure in the aluminum tray mold went as follows:

Contact pressure applied, raised to 275°F,
hold 25 minutes at 275°F,
75 minutes to 330°F, apply 100 psi,
hold 75 minutes at 330°F, cool

Postcure:

Heat-up to 465°F in 8 hours,
hold 16 hours at 465°F,
cool slowly.

7.4.4 Hystl G-3000 - The resin was catalyzed with 0.5 phr L-101 catalyst. The cure proceeded as follows:

Contact pressure, heat from R.T. to 275°F in 20 minutes,
after 1 hour, apply 100 psi,
1 hour 20 minutes to 330°F,
hold an additional 90 minutes at 330°F,
and 100 psi pressure.

Postcure:

Hold 2 hours at 365°F,
18 hours at 465°F,
cool slowly.

7.4.5 Ricon 431 - The resin was catalyzed with 3 phr L-101 catalyst. The following cure was used:

Heat from RT to 250°F under 100 psi,
Hold 15 min at 250°F, 200 psi
Hold 20 min at 325°F, 200 psi
Hold 25 min at 350°F, 200 psi, cool slowly

7.4.6 Dapon M - The resin was catalyzed with 2 phr L-101 catalyst. The cure proceeded as follows:

Total Time		Temperature	
15 minutes	to	150°F	under 100 psi pressure,
30 minutes	to	200°F,	
45 minutes	to	225°F,	
60 minutes	to	350°F,	

hold 1 hour at 350°F, 100 psi, then cool slowly.

7.4.7 Dapon M/DAIP, 70/30 Blend - The system was catalyzed with 2 phr L-101 catalyst; 100 psi was applied; the resin was heated to 350°F in 1 hour, held 1 hour at 350°F, 100 psi, and cooled slowly.

7.4.8 Hetron 800FA/800L - The resin was mixed, 800FA with 4 phr 800L catalyst. The layup was cured as follows:

R.T. to 150°F under contact pressure,
hold 10 minutes at 150°F, apply 100 psi,
heat as follows:

Total Time		Temperature
30 minutes	to	200°F,
75 minutes	to	300°F,
105 minutes	to	350°F,

cure 1 hour at 350°F, 100 psi pressure; cool slowly.

The cure cycles given above are those that produced high quality laminates that were used in the study. In some cases, two or three previous attempts were unsuccessful. These unsuccessful cycles are not included in the report; only those cure schedules yielding acceptable laminates have been presented.

In summarizing the results of the testing, the following observations can be made: As might be expected, the 24-hour boil tests is more severe than the 30-day humidity exposure. The latter, however, may correlate more accurately to actual service conditions. All resins exhibited less water pickup, etc., than would state-of-the-art epoxies. Fiber volumes and physical properties in general were quite satisfactory.

The mechanical properties, while generally low, are encouraging. Loss of 350°F properties after boil or humidity aging was generally no worse (and in a number of cases better) than loss of RT properties. The latter loss can be generally atributed to finish/interface problems encountered with the particular resin and the Volan/glass reinforcement. No attempt was made to deal with this problem since only the resin/graphite or resin/boron problems are of interest, and these were scheduled to be investigated in later tasks.

The main objective of Task 4 was quite satisfactorily accomplished in that:

(1) Good laminates of appropriate fiber volume were fabricated.

(2) Initial process and cure parameters were identified and defined.

(3) Relatively good resistance to environmental aging was exhibited by the candidate resins. Low moisture pickup was shown; the effect of moisture on mechanical properties was relatively independent of test temperature, indicating that plasticization of the matrix is not a problem.

TABLE 7-1

PHYSICAL PROPERTIES OF 181 E GLASS LAMINATES

Resin	Specific Gravity	Fiber Volume (%)	Resin Content (%)	24-Hour Boil Test				30-Day, 120°F, 95% RH Test			
				ΔLength (%)	ΔWidth (%)	ΔHeight (%)	Water Pickup (%)	ΔLength (%)	ΔWidth (%)	ΔHeight (%)	Water Pickup (%)
Hetron 197	1.98	55.3	27.1	.01	.06	.80	1.43	.00	.05	.25	.44
Koppers V 7000-15	1.90	61.0	22.4	.04	.03	-.03	2.71	.00	.03	.45	.28
Hystl B-3000	1.78	54.3	24.8	.10	.16	.60	1.52	.00	.04	.35	1.10
Hystl G-3000	1.87	57.8	22.1	.00	.01	-.05	1.92	.00	.02	.35	1.52
Dapon M	1.92	60.8	24.1	.00	.03	.45	4.80	.00	-.03	.10	2.00
Dapon M/DAIP, 70/30 Blend	1.92	57.4	27.0	.00	.02	.55	3.84	.00	-.01	.25	1.05
Hetron 800FA/800L Catalyst	1.92	55.0	26.8	.00	.04	.55	4.65	.06	.00	.45	2.27
Ricon 431	1.85	57.5	22.8	.002	.02	.07	0.57	.00	.00	.35	.04

TABLE 7-2

ENVIRONMENTAL AGING TESTS, GLASS REINFORCED LAMINATES

Resin	Test Temp.		Initial Values		24-Hour Boil		30-Day Humidity	
			Flexural Strength (ksi)	Flexural Modulus (msi)	Flexural Strength (ksi)	Flexural Modulus (msi)	Flexural Strength (ksi)	Flexural Modulus (msi)
Hetron 197	RT		52.2	4.00	24.7	3.31	33.7	3.67
			51.8	3.97	32.9	3.07	39.8	3.58
			56.7	3.85	31.2	3.08	40.2	3.68
		Average	53.6	3.94	29.6	3.15	37.9	3.64
	350°F		9.13	1.93	8.35	.89	9.31	1.26
			10.10	2.17	8.13	.80	8.61	1.22
			9.65	1.78	8.15	1.00	8.87	1.29
		Average	9.63	1.96	8.21	.90	8.93	1.26
Koppers V 7000-15	RT		55.8	3.41	20.6	2.95	33.2	3.24
			52.5	3.30	19.2	3.06	31.0	3.33
			54.2	3.74	19.8	2.76	26.3	3.22
		Average	54.2	3.48	19.9	2.92	30.2	3.26
	350°F		15.1	2.16	11.9	1.70	11.4	1.82
			14.6	2.09	11.3	1.53	11.3	1.82
			15.4	2.03	12.1	1.58	11.5	1.58
		Average	15.0	2.09	11.8	1.60	11.4	1.74
Hystl R-3000	RT		36.6	3.13	23.6	2.91	18.2	3.25
			39.0	3.14	22.2	2.80	18.4	2.38
			40.1	3.09	22.7	2.71	17.6	2.40
		Average	38.6	3.12	22.8	2.81	18.1	2.68
	350°F		18.3	2.35	16.8	1.77	22.5	2.26
			18.1	2.38	14.2	1.97	17.7	1.28
			16.3	2.18	14.2	1.89	18.6	1.64
		Average	17.6	2.30	15.1	1.88	19.6	1.73
Hystl G-3000	RT		40.4	3.57	24.6	3.19	22.4	3.11
			46.8	3.42	29.5	3.23	22.4	2.98
			40.0	3.56	24.3	3.09	24.8	2.86
		Average	42.4	3.52	26.1	3.17	23.2	2.98
	350°F		18.9	2.57	17.7	1.12	24.8	2.62
			20.1	2.46	14.2	1.86	17.3	2.24
			21.2	2.62	10.9	1.33	16.3	1.81
		Average	20.1	2.55	14.3	1.44	19.5	2.22
Dapon M	RT		30.0	3.14	15.9	1.61	21.9	2.53
			32.5	3.57	23.4	3.46	22.2	2.90
			31.6	3.35	21.8	3.23	23.5	3.10
		Average	31.4	3.35	20.4	2.77	22.5	2.84
	350°F		15.5	1.88	16.6	2.25	18.1	2.19
			20.4	2.89	18.1	2.67	18.7	2.65
			18.8	2.94	17.5	2.60	20.6	2.87
		Average	18.2	2.57	17.4	2.54	19.1	2.57
Dapon M/DAIP, 70/30 Blend	RT		36.9	3.26	11.6	2.07	25.3	2.99
			32.2	3.24	12.2	2.12	25.0	2.92
			34.0	3.22	14.1	2.25	24.6	2.95
		Average	34.4	3.24	12.6	2.15	25.0	2.95
	350°F		20.4	2.62	11.1	1.48	18.6	2.54
			20.2	2.56	11.7	1.66	19.8	2.35
			20.9	2.62	12.2	1.50	19.6	2.48
		Average	20.5	2.60	11.7	1.55	19.3	2.46
Hetron 800FA/800L Catalyst	RT		27.6	2.74	23.3	2.71	12.4	2.06
			28.1	2.76	23.3	2.88	12.3	2.06
			29.4	2.78	23.9	2.99	12.4	2.25
		Average	28.4	2.76	23.5	2.86	12.4	2.12
	350°F		9.92	1.53	19.3	2.26	12.0	1.60
			10.20	1.71	20.2	2.37	11.5	1.62
			11.90	1.62	20.3	2.44	11.0	1.71
		Average	10.67	1.62	19.9	2.36	11.5	1.64
Ricon 431	RT		60.1	3.79	38.7	3.48	44.6	3.40
			58.3	3.92	41.3	3.38	44.9	3.58
			55.4	4.07	38.4	3.60	44.9	3.75
		Average	57.9	3.93	39.5	3.55	44.8	3.58
	350°F		3.24	0.27	2.62	--	2.31	--
			3.60	0.32	2.61	--	2.61	--
			2.97	0.38	2.59	--	2.99	--
		Average	3.27	0.32	2.61	--	2.64	--

SECTION 8

PREPREGGING PARAMETER AND COMPATIBILITY STUDY

The primary objective of the process and cure parameter study was to learn to process the candidate resins; i.e., to screen handling problems, etc., and to determine the type of processing required to obtain sound laminates with good fiber volume, resin content, etc. In order to obtain maximum mechanical properties, however, problems of prepregging, compatibility with reinforcements, sizings, etc., had to be defined and understood. As seen in the previous section, optimum performance was not necessarily obtained with initial laminates using glass reinforcements. Rather than solve compatibility problems with that reinforcing material, the program proceeded directly into work with graphite (Thornel 300) reinforcements.

The processing parameters and cure cycles developed in Task 4 were used to fabricate Thornel 300 reinforced laminates with all candidates. A problem was immediately identified with the Koppers V 7000-15 polyester, which proved to be widespread as will be discussed in detail in the following section. In general, the prepregging went very well on T-300 reinforcement. No compatibility problems were identified at this point.

Laminates reinforced with 4-mil boron filaments were then fabricated from Hystl G-3000 resin. A cure schedule similar to that used for graphite reinforced prepreg yielded encouraging early mechanical properties:

	Flexural Strength RT	Flexural Modulus RT	Short Beam Shear RT
Hystl G-3000/Boron:	186 ksi	22.5 msi	12.7 ksi
	224 ksi	26.6 msi	11.6 ksi
Average	205 ksi	24.6 msi	12.2 ksi

Further work on 4 mil boron was, however, suspended as severe problem areas arose in the graphite reinforced laminates. These are more appropriately discussed in the following section.

SECTION 9

TAPE AND LAMINATE FABRICATION AND TESTS

The processing parameters and cure cycles developed in Task 4 were used to fabricate Thornel 300 reinforced laminates with all candidates. A problem was immediately identified with the Koppers V 7000-15 polyester. Unidirectional laminates could not be satisfactorily fabricated without severe cracking in the 0° direction as shown in Figure 9-1. This difficulty, attributed to excessive shrinkage during cure and the brittle nature of the resin, could not be eliminated, even with exceedingly slow cure and cool-down schedules. Not surprisingly, this does not appear with woven reinforcement or with cross-plied laminates. However, hardware layups with extra unidirectional plies would prove difficult to fabricate with such a system. After discussions with the Air Force Program Monitor, Koppers V 7000-15 polyester was dropped from the program. In addition, the Dapon M was not included. The Dapon M/DAIP blend exhibited all attributes of Dapon M with greatly improved drape, tack, and handling. Additional work with Dapon M was to be scheduled if unforeseen difficulties arose with the Dapon M/DAIP blend. The Ricon 431 was also included in this work, using cure parameters developed on the glass reinforced version.

Tables 9-1 and 9-2 give the results obtained on the Thornel 300 reinforced panels before and after environmental aging. The excellent performance of Hetron 800FA (furfuryl alcohol) came as a pleasant surprise, considering that it was one of the poorest performing systems on glass. In general, the results, especially at elevated temperature, indicated that a significant amount of optimization was required to achieve desired levels of mechanical performance. A particularly difficult goal was to achieve high mechanical strengths at service temperature at 375°F. Work on other aspects of the program, i.e., boron reinforced laminates, etc., was suspended as efforts were devoted to improving elevated temperature mechanical properties.

Higher temperature and/or extended duration cures or postcures were evaluated for laminates fabricated from the following matrix resins reinforced with Thornel 300 fibers:

Hystl G-3000
Hystl B-3000
Dapon M/DAIP
Hetron 197
Hetron 800FA
Ricon 431
Ricon 431 + 3% Borden triallyl cyanurate (TAC).

Satisfactory laminates were fabricated in the following cases:

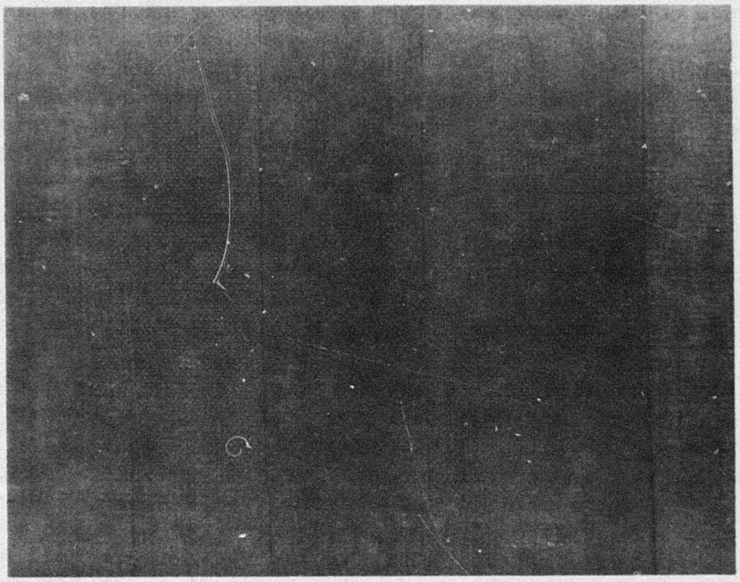


FIGURE 9-1. T-300/V 7000-15 Laminate

TABLE 9-1

PHYSICAL PROPERTIES, THORNEL 300 REINFORCED LAMINATES
ENVIRONMENTAL AGING

Resin	Specific Gravity	Fiber Volume (%)	Resin Content (wt%)	24-Hour Boil				30-Day, 120°F, 95% RH			
				ΔLength (%)	ΔWidth (%)	ΔHeight (%)	Water Pickup (%)	ΔLength (%)	ΔWidth (%)	ΔHeight (%)	Water Pickup (%)
Hetron 197	1.52	60.6	30.9	0.00	0.21	0.20	2.73	0.00	0.02	-0.20	0.33
Hetron 800FA/800L	1.49	55.2	36.6	0.00	0.06	0.15	0.67	0.04	0.28	0.05	0.56
Hystl B-3000	1.40	58.7	29.1	0.00	0.00	0.20	0.50	0.00	0.03	0.00	0.38
Hystl G-3000	1.41	50.0	28.0	0.00	0.00	0.10	1.03	0.00	0.03	0.50	0.23
Ricon 431	1.41	54.7	33.1	0.00	-0.03	-0.10	0.13	0.00	0.00	0.11	0.04
Dapon M/DAIP	1.44	60.3	32.6	0.00	0.02	0.55	3.83	0.00	-0.01	0.21	1.05

TABLE 9-2

ENVIRONMENTAL AGING TESTS, THORNEL 300 REINFORCED LAMINATES

Resin	Test Temp.		Initial Values			24- Hour Boil			30-Day Humidity		
			Flexural Strength (ksi)	Flexural Modulus (msi)	Short Beam Shear (ksi)	Flexural Strength (ksi)	Flexural Modulus (msi)	Short Beam Shear (ksi)	Flexural Strength (ksi)	Flexural Modulus (msi)	Short Beam Shear (ksi)
Hetron 197	RT		93.2	16.3	8.46	96	14.8	6.14	106	15.8	7.88
			62.7	17.2	8.50	111	16.5	6.64	191	15.9	7.50
			-	17.8	8.71	-	-	7.19	-	-	6.99
		Avg.	78.0	17.1	8.50	104	15.7	6.66	149	15.8	7.46
	375°F		16.5	6.39	1.36	15.6	5.65	1.07	14.2	4.59	1.38
			17.6	7.51	1.25	17.2	5.47	1.02	17.4	5.96	1.36
			16.7	6.18	1.19	17.5	5.67	1.07	17.4	5.29	1.19
		Avg.	16.9	6.69	1.27	16.8	5.60	1.05	16.3	5.28	1.30
Hystl B-3000	RT		106	18.5	6.98	107	16.2	6.15	124	18.1	6.89
			111	18.6	6.57	112	17.3	6.15	117	18.9	6.83
			108	18.3	7.39	106	16.6	5.75	125	19.4	6.54
		Avg.	108	18.5	6.98	108	16.7	6.02	122	18.8	6.75
	375°F		20.5	6.98	1.58	21.9	7.41	1.69	23.4	8.20	1.61
			20.9	7.50	1.69	20.5	6.71	1.61	20.9	8.18	1.43
			21.1	7.47	1.61	21.9	6.87	1.66	22.4	7.66	1.64
		Avg.	20.8	7.32	1.63	21.4	7.00	1.65	22.2	8.01	1.56
Hystl G-3000	RT		146	20.4	7.04	142	18.8	6.25	148	19.4	6.21
			154	20.1	6.79	148	18.3	5.68	154	19.3	6.82
			148	19.4	7.07	138	17.9	5.75	143	18.7	6.50
		Avg.	149	20.0	6.97	142	18.3	5.89	148	19.1	6.51
	375°F		23.1	11.7	2.00	21.0	11.9	1.82	22.1	10.7	1.79
			26.1	10.9	1.79	19.6	10.1	1.79	22.4	11.3	1.86
			23.7	10.8	1.82	19.5	10.0	1.91	22.9	9.7	1.91
		Avg.	24.3	11.1	1.87	20.0	10.7	1.84	22.5	10.8	1.85
Dapon M/DAIP	RT		134	17.3	5.86	128	15.9	4.91	129	15.0	5.74
70 30			121	17.0	6.00	118	16.4	4.16	145	16.3	9.52
			139	16.7	5.39	113	14.8	5.83	112	15.1	5.08
		Avg.	131	17.0	5.75	119	15.7	4.97	129	15.5	6.69
	375°F		51.0	14.7	2.50	42.9	12.6	2.28	44.5	12.6	2.43
			48.4	13.5	2.57	54.1	14.2	2.52	42.8	12.5	2.52
			50.6	14.6	2.50	44.3	13.0	2.52	47.9	13.7	2.45
		Avg.	50.0	14.3	2.52	47.1	13.3	2.44	45.1	12.9	2.47
Hetron 800FA/800L	RT		184	17.2	11.1	158	14.7	9.02	182	14.0	9.82
			182	17.3	12.0	145	14.2	8.50	168	14.6	11.10
			185	16.2	11.2	145	13.4	8.71	173	14.4	9.38
		Avg.	184	16.9	11.4	149	14.1	8.75	174	14.3	10.00
	375°F		83.7	13.6	4.11	80.0	13.9	4.03	73.6	12.8	4.22
			74.8	15.2	4.60	85.5	13.1	3.84	83.2	12.7	4.11
			79.9	14.7	4.27	83.9	12.9	3.88	71.9	13.3	4.25
		Avg.	79.5	14.5	4.33	83.1	13.3	3.96	76.2	12.9	4.19
Ricon 431	RT		150	16.6	8.50	141	16.7	8.07	135	18.5	7.40
			158	16.3	8.43	140	17.5	6.03	135	17.1	6.65
			145	16.8	8.15	-	-	6.07	-	-	6.92
		Avg.	150	16.6	8.36	141	17.1	6.08	135	18.8	6.99
	375°F		9.5	2.75	0.85	9.33	3.01	0.85	9.85	2.79	0.51
			11.4	2.65	0.64	8.84	2.80	0.51	8.28	2.32	0.51
			8.0	2.39	0.68	7.05	2.30	0.85	8.31	2.27	0.51
		Avg.	9.6	2.60	0.72	7.74	2.70	0.59	8.81	2.46	0.51

Resin	High Temperature Cure Variations
Hystl G-3000	Postcured 24 hours at 500°F
Dapon M/DAIP	Cured 2 hours at 450°F

In all other cases, high temperature and/or extended cure or postcure resulted in excessive cracking of unidirectional laminates. This cracking was so severe as to preclude the machining of usable flexure specimens from the panels.

Table 9-3 compares the performance of the high temperature cured panels to the performance of Hystl G-3000 and Dapon M/DAIP as originally cured.

TABLE 9-3

EFFECT OF HIGH TEMPERATURE CURE CYCLES

Resin	Cure	Test Temp.	Flexural Strength (ksi)	Flexural Modulus (msi)	Short Beam Shear (ksi)
Hystl G-3000	Regular	RT	149	20.0	6.97
		375°F	24.3	11.1	1.87
	High Temp.	RT	158	18.5	7.38
		350°F	49.0	15.0	3.10
		375°F	45.2	14.4	2.92
Dapon M/DAIP	Regular	RT	131	17.0	5.75
		375°F	50.0	14.3	2.52
	High Temp.	RT	188	15.9	11.8
		350°F	62.3	13.5	2.89
		375°F	56.5	13.3	2.67

NOTE: All values are an average of the results of three test specimens.

Note that although performance was significantly improved by the use of a high temperature cure or postcure, it is still far from adequate at elevated temperatures. The probable reasons for these difficulties will be discussed in the following section as will promising approaches to circumvent these problem areas.

SECTION 10

CONCLUSIONS AND RECOMMENDATIONS

As shown in the preceding section, a major difficulty developed concerning the candidate resin systems. The difficulty involves achieving an advanced state of cure which allows good mechanical properties to be generated at 350 - 375°F while simultaneously avoiding the cracking of unidirectional laminates during cure. Such cracking cannot be tolerated, as the typical layups used in aerospace hardware are not only pseudoisotropic, but in many cases are highly directional in nature. The cracking observed in the unidirectional laminates would manifest itself as the directional character of a particular piece of hardware increased.

The problem appears to be inherent in the structure of these resins. The polybutadiene, diallylisophthalates, etc., are quite moisture resistant, probably due to the aliphatic, nonpolar characteristics of their structure. In order to achieve high temperature performance, a high state of cure or crosslinking must be reached. Unfortunately, the higher the degree of crosslinking results in increased shrinkage during cure. As the layup becomes more directional in nature (a unidirectional laminate being the worst case), the increased shrinking manifests itself in cracking, as observed in the program. If the degree of cure is reduced to avoid this shrinkage and subsequent cracking, the matrix loses rigidity at elevated temperature and poor mechanical performance results. Note that cross-plied laminates or those with woven reinforcement can accommodate a far greater amount of shrinkage than unidirectional laminates and are not subject to this problem.

WRD believed that a number of approaches to circumvent the problem are possible; some more attractive than others.

(1) Reduced Elevated Temperature Requirements

One approach is to define the useful temperature ranges for the candidate resins where adequate performance levels are reached and cracking is avoided. This approach is unattractive and in general unacceptable. First, this approach abandons one of the prime goals of the program; i.e. to find an alternate to the moisture sensitive, high temperature (i.e. 350°F service) modified epoxies currently in use. Secondly, although the high temperature epoxies, such as 3501 or 5208, do lose some performance at 350°F due to moisture pickup, their remaining mechanical properties coupled with good handling characteristics, moderate cost, and moderate curing parameters make them more attractive than the alternates being developed on the program.

(2) Modification or Blending of Resin Systems

A number of polymer based approaches utilizing available candidate resins would offer potential solutions to the problem. For example, an agent such as TAC could be added to DAIP or PBD systems. The subsequent cure involving cyclization and the formation of aromatic species would certainly increase elevated temperature performance and perhaps avoid an accompanying increase in shrinkage and cracking. Alternately available linear, boardy, moisture insensitive (as previously determined in Task 3), high temperature resins such as NR-150 polyimides, PPQ, polyarylsulfones, etc., could be blended with compatible, high tack materials such as DAIP monomer, various PBD resins, etc., to achieve a tough, yet high temperature system with adequate drape and tack, etc.

Unfortunately, interesting as this approach may be, it involves significant development of matrix materials and may be beyond the scope of this program as presently conceived and authorized.

(3) Use of Woven Reinforcement

An attractive alternative, less exotic than (2), but certainly with a high probability of success, is to continue development of the candidate materials but utilizing woven graphite and woven Kevlar 49 reinforcements. The candidate systems have proven 350°F performance on woven glass or quartz. The use of such woven reinforcements accommodates and is compatible with a far higher degree of shrinkage during cure. Woven graphite and Kevlar 49 are becoming increasingly important reinforcements destined to see far greater use in future aerospace hardware. Their ease of application, handling, etc., make them extremely attractive. The development of the candidates into viable systems for use with woven high performance reinforcements would provide the Air Force with useful, novel systems exhibiting significant advantages over modern high temperature epoxy matrix materials. Note that the use of woven reinforcements with epoxy systems brings us right back to the same problem of moisture resistance.

(4) Alternate Matrix Resins

A number of resin systems screened in Task 3 looked extremely attractive from the standpoint of moisture resistance but were discarded since their processing requirements differ significantly from those of current epoxy systems. These are the high performance thermoplastics, specifically polysulfone, NR-150A2, and PPQ. However, these differences in processing may actually prove an advantage. Although not readily compatible with conventional autoclave techniques, if processing is tailored to the requirements of these systems, they are proving simpler, quicker, and cheaper to use than present state-of-the-art epoxies. Present programs geared to achieve significant cost savings through the use of these thermoplastics are presently being funded by various government agencies. These resins would prove attractive candidate systems and the program would generate sorely needed environmental aging data which would facilitate the use of these materials in actual Air Force applications.

It is recommended that this work be continued. In addition, we believe either alternative 3 or 4 listed above or a mix of these approaches is an extremely attractive low risk, and potentially highly rewarding alternative and should be pursued if further funding becomes avilable.

APPENDIX

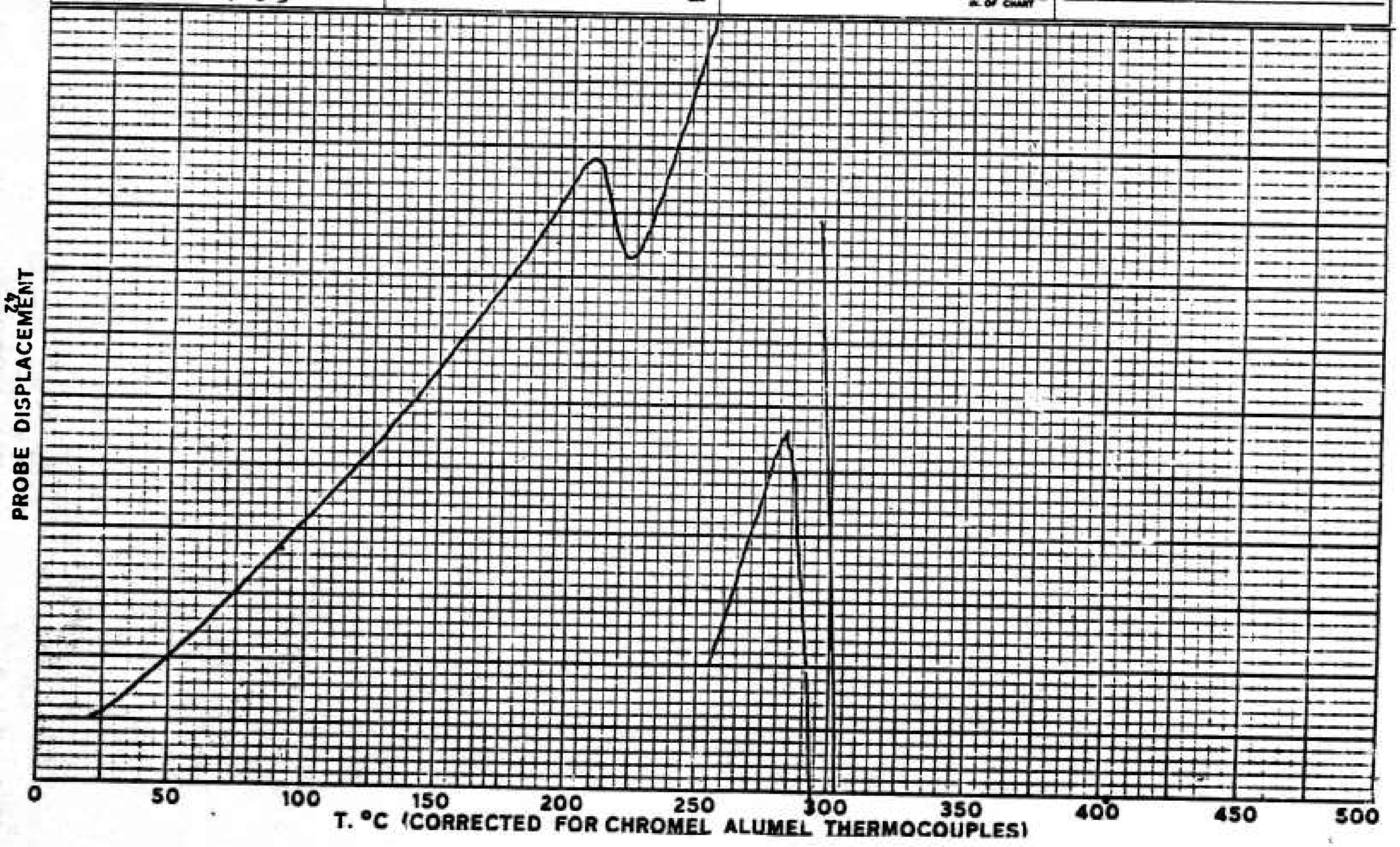
TMA CURVES OF CANDIDATE RESINS

The following appendix includes thermomechanical analysis (TMA) curves for each candidate resin evaluated, before and after a 24-hour water boil exposure.

PART NO. 941017

DUPONT INSTRUMENTS

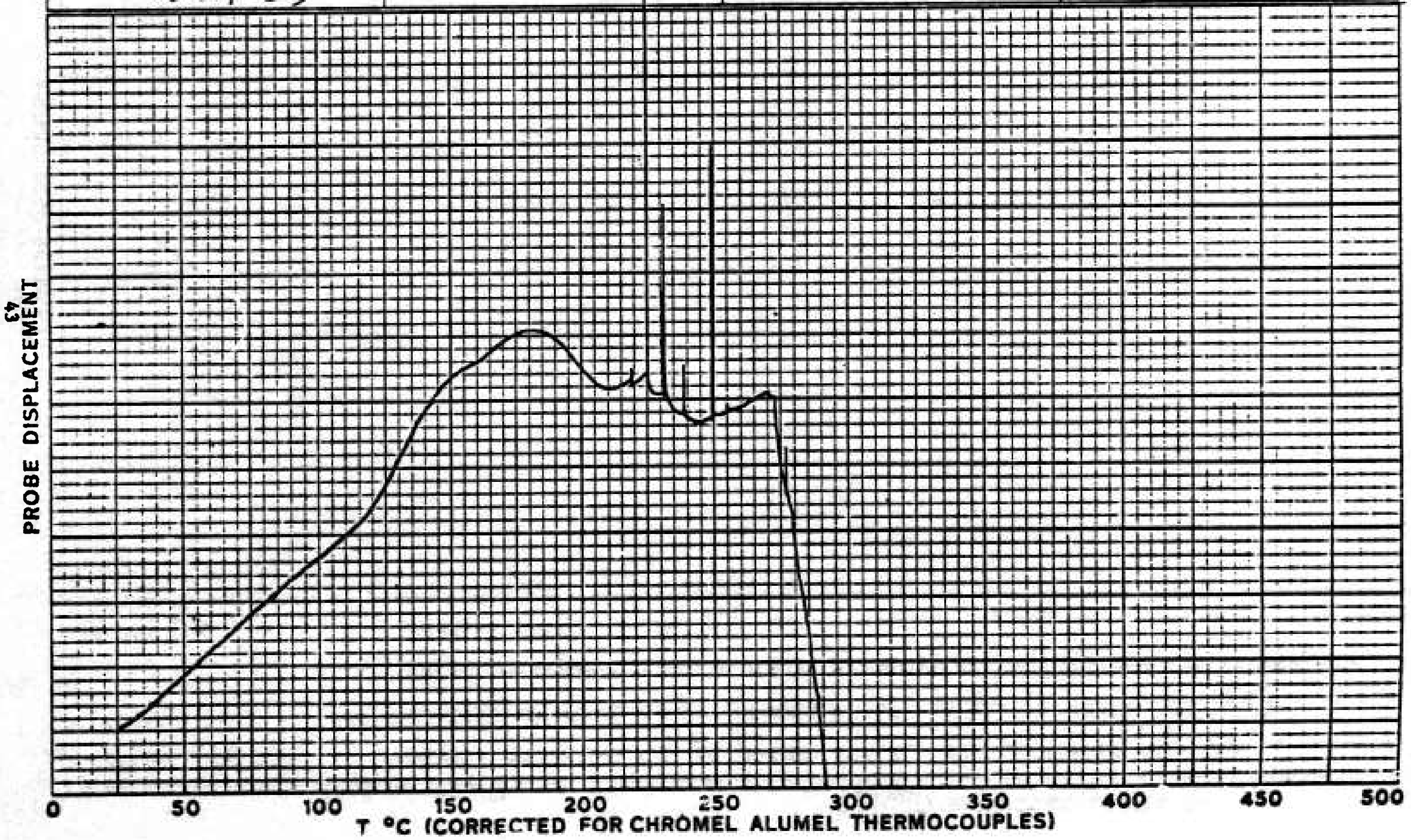
SAMPLE: 3501B
ORIGIN: DD-319-23
SAMPLE HEIGHT .25"x.25"x.25"
LOADING ON TRAY 100g
PROBE: EXPANSION/PENETRATION
HEATING RATE 5 °C/min
X-AXIS SCALE 50 °C/DIV.
Y-AXIS SCALE .02 MV/IN.
Y-AXIS SENSITIVITY ____ IN. PROBE DISPL./IN. OF CHART
RUN NO. 102
DATE 8-19-74
OPERATOR D. Dayton



DUPONT INSTRUMENTS

PART NO. 941017

SAMPLE: 3501 B 24 hr H₂O Boil 24 hr H₂O Soak	**SAMPLE HEIGHT** .25"x.25"x.25"	**X-AXIS SCALE** 50	**RUN NO.** 96
	LOADING ON TRAY 100g	**Y-AXIS SCALE** .02	**DATE** 8-15-74
	PROBE: EXPANSION/PENETRATION	**Y-AXIS SENSITIVITY**	**OPERATOR** D. Dayton
ORIGIN: DD-319-23	**HEATING RATE** 5	IN. PROBE DISPL. / IN. OF CHART	



DU PONT INSTRUMENTS

PART NO. 941017

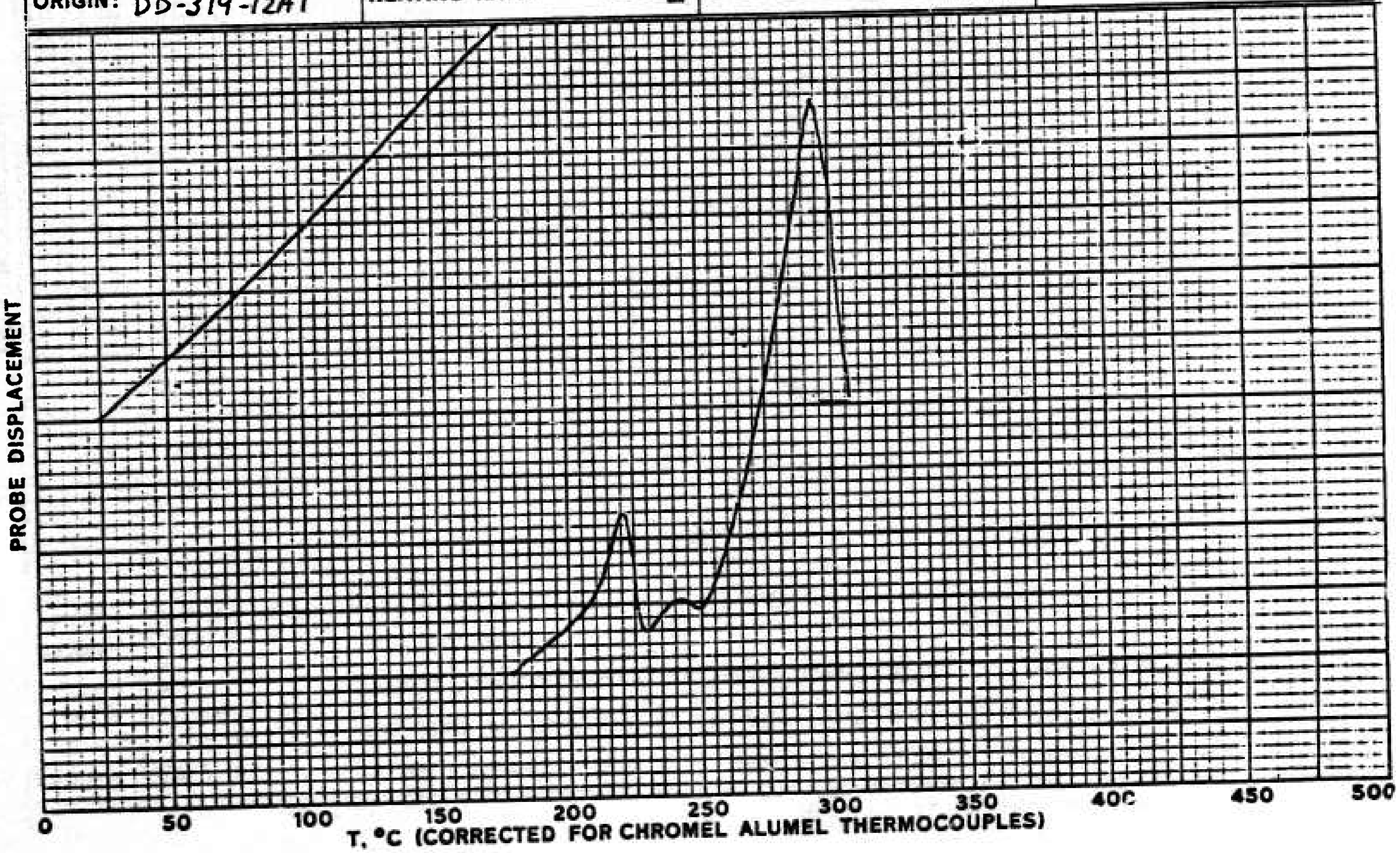
SAMPLE: 5208
Epoxy

ORIGIN: DD-319-12A1

SAMPLE HEIGHT .25"x.25"x.25"
LOADING ON TRAY 100g
PROBE: EXPANSION/PENETRATION
HEATING RATE 5 °C/min

X-AXIS SCALE 50
Y-AXIS SCALE .02
Y-AXIS SENSITIVITY 0.0343 in
IN. PROBE DISPL. / IN. OF CHART

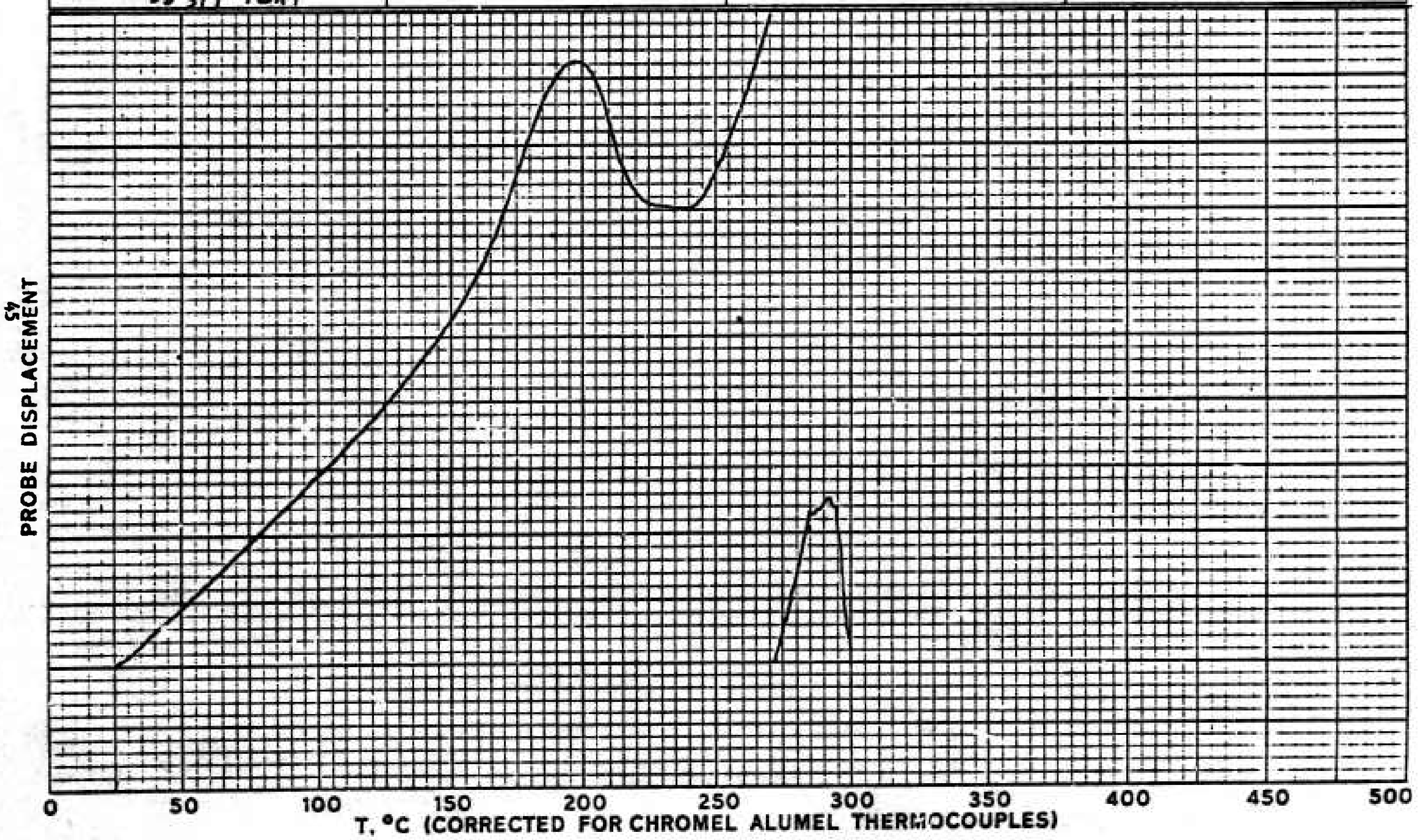
RUN NO. 66
DATE 8-1-74
OPERATOR DDayton



PART NO. 941017

DU PONT INSTRUMENTS

SAMPLE: 5208 Epoxy 24 hr H2O Boil 24 hr H2O Soak	SAMPLE HEIGHT 2.5"x.25"x.25"	X-AXIS SCALE 50	RUN NO. 68
	LOADING ON TRAY 100g	Y-AXIS SCALE .02	DATE 8-2-74
	PROBE: EXPANSION/PENETRATION	Y-AXIS SENSITIVITY	OPERATOR D Dayton
ORIGIN: DD-319-12A1	HEATING RATE 5		



DUPONT INSTRUMENTS

PART NO. 941017

SAMPLE: 5209 (EPOXY)
ult@250°F dry stored in desiccator

ORIGIN: DD-319-1281

SAMPLE HEIGHT .25"x.25"x.25"

LOADING ON TRAY 100g

PROBE: EXPANSION/PENETRATION

HEATING RATE 5 °C/MIN

X-AXIS SCALE 50 °C/DIV.

Y-AXIS SCALE .02 MV/IN

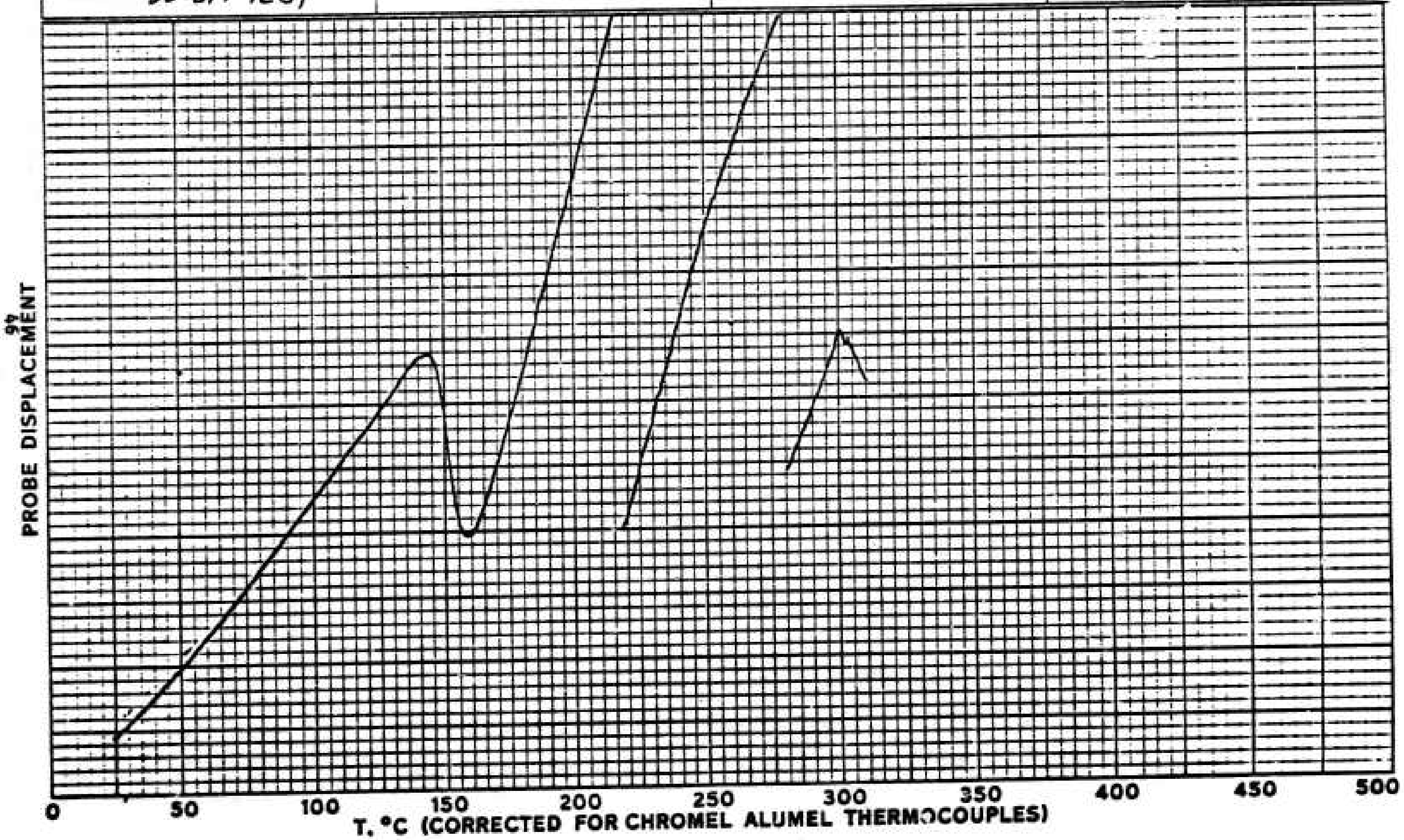
Y-AXIS SENSITIVITY

IN. PROBE DISPL./IN. OF CHART

RUN NO. 72

DATE 8-5-74

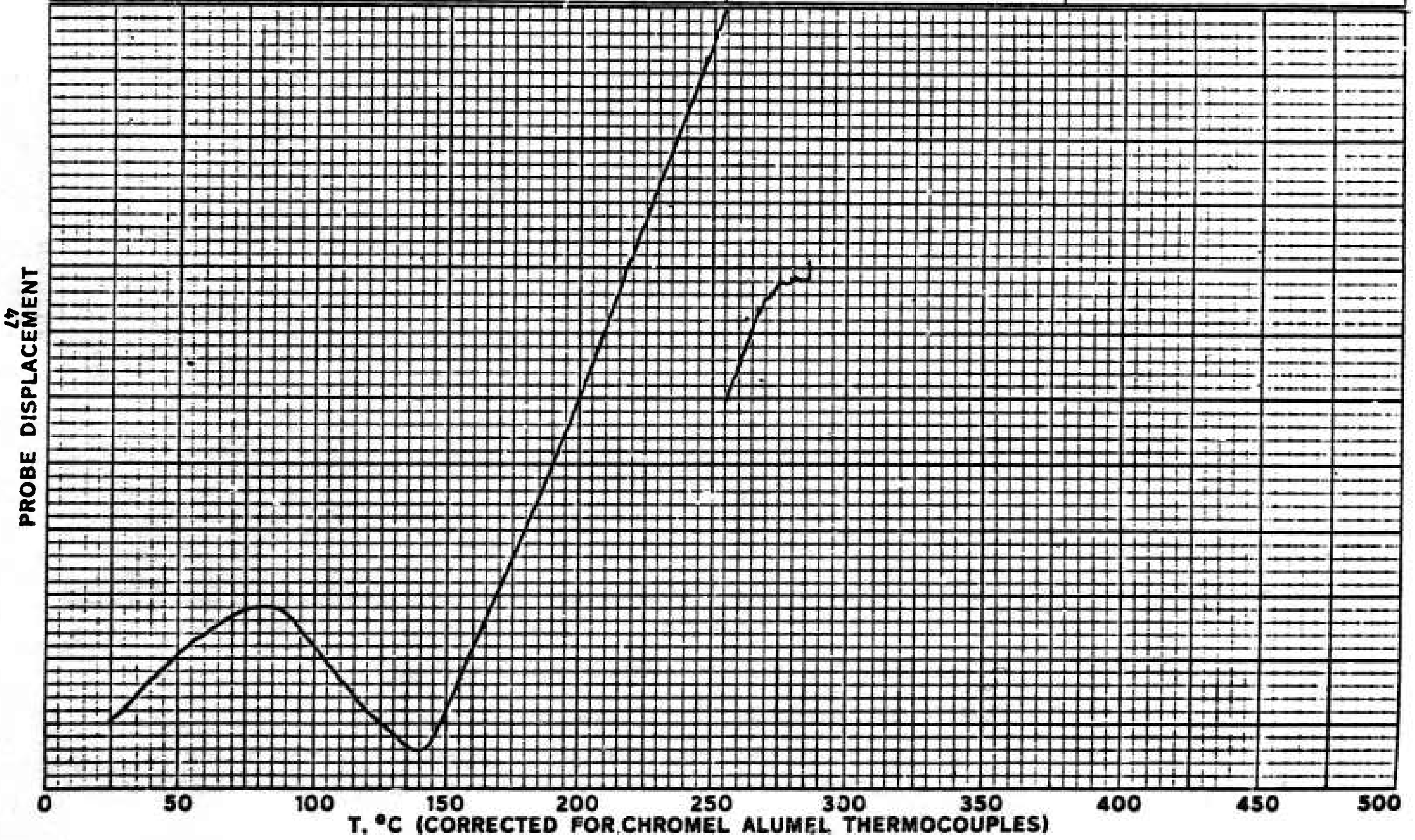
OPERATOR D.E. Dayton



PART NO. 941017

DUPONT INSTRUMENTS

SAMPLE: 5209 (EPOXY) 24 hr H_2O Boil 24 hr H_2O Soak	SAMPLE HEIGHT 25"x25x25"	X-AXIS SCALE 50 °C/in.	RUN NO. 69
	LOADING ON TRAY 100g	Y-AXIS SCALE .02 mV/in.	DATE 8-2-74
	PROBE: EXPANSION/PENETRATION	Y-AXIS SENSITIVITY	OPERATOR D Dayton
ORIGIN: DD-319-1281	HEATING RATE 5 °C/min	in. probe displ./in. of chart	



PART NO. 941017

INSTRUMENTS

SAMPLE: Hystl 6-1000

ORIGIN: 2249-4A-1

SAMPLE HEIGHT .25"x.25"x.25"

LOADING ON TRAY 100g

PROBE: EXPANSION/PENETRATION

HEATING RATE 5 °C/min

X-AXIS SCALE 50 °C/in.

Y-AXIS SCALE .02 mV/in.

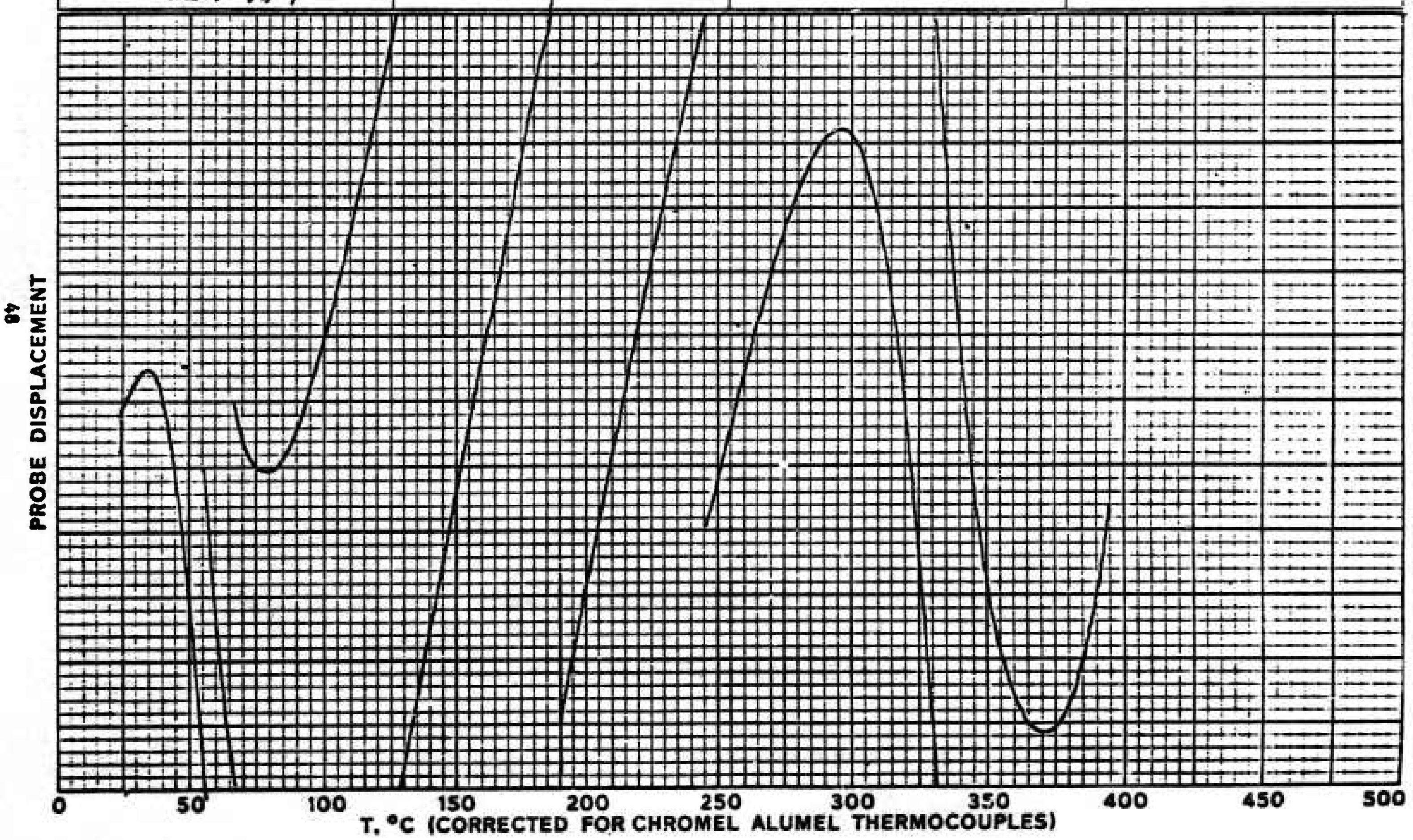
Y-AXIS SENSITIVITY ____

____ in. probe displ./in. of chart

RUN NO. 62

DATE 7-30-74

OPERATOR D Dayton



DUPONT INSTRUMENTS

PART NO. 941017

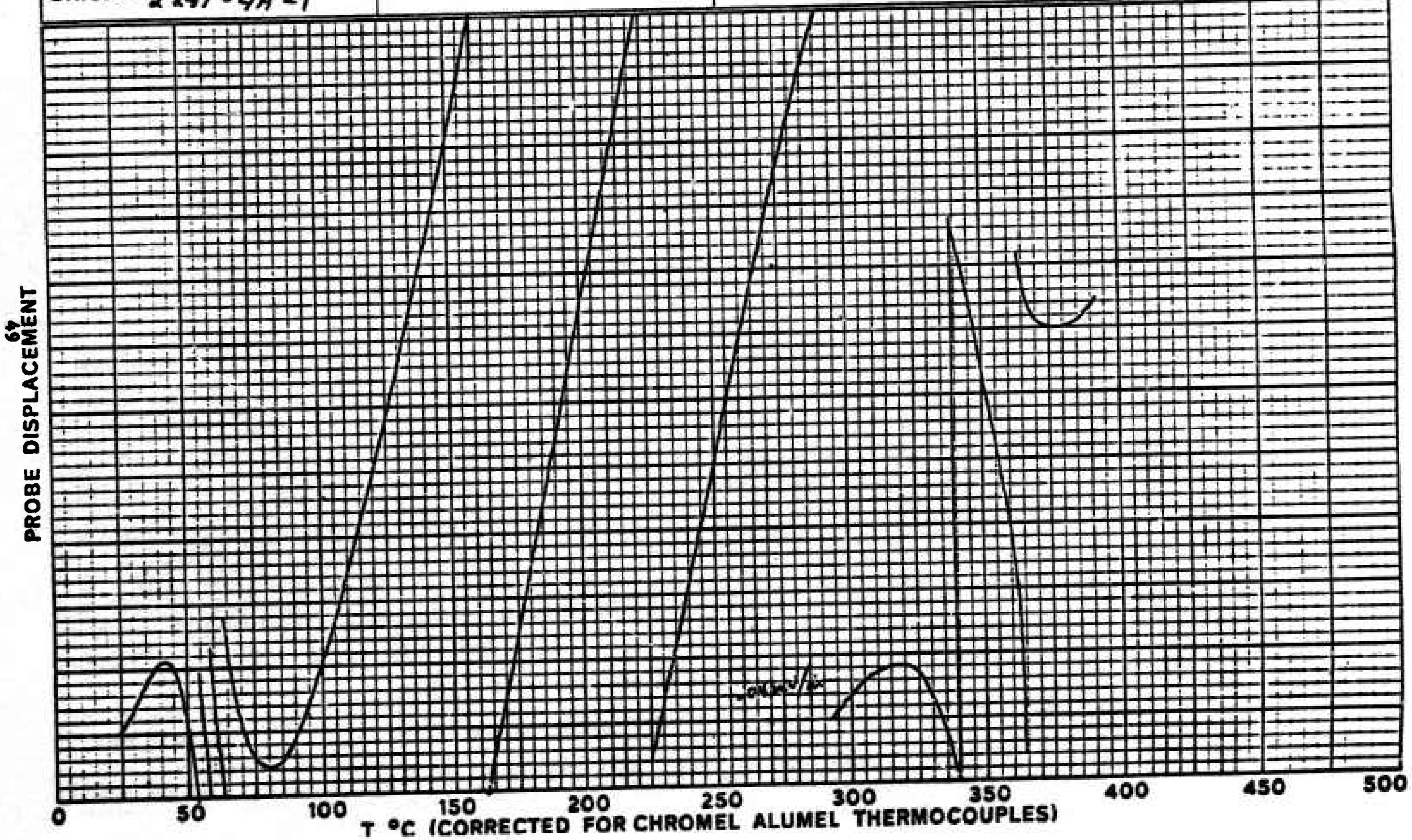
SAMPLE: HYSTL G-1000
25½ hr H₂O Boil
120 hr Soak

ORIGIN: 2241-4A-1

SAMPLE HEIGHT .25"x.25"x.25"
LOADING ON TRAY 100g
PROBE: EXPANSION/PENETRATION
HEATING RATE 5

X-AXIS SCALE 50
Y-AXIS SCALE .02
Y-AXIS SENSITIVITY

RUN NO. 58
DATE 2-29-74
OPERATOR D Dayton



DU PONT INSTRUMENTS

PART NO. 941017

SAMPLE: Hystl 6-2000
24 hr H2O Boil
24 hr H2O Soak

ORIGIN: 2249-5A-2 ~~[illegible]~~

SAMPLE HEIGHT .25 x .25 x .25"

LOADING ON TRAY 100 g

PROBE: EXPANSION/PENETRATION

HEATING RATE 5 °C/min

X-AXIS SCALE 50 °C/in.

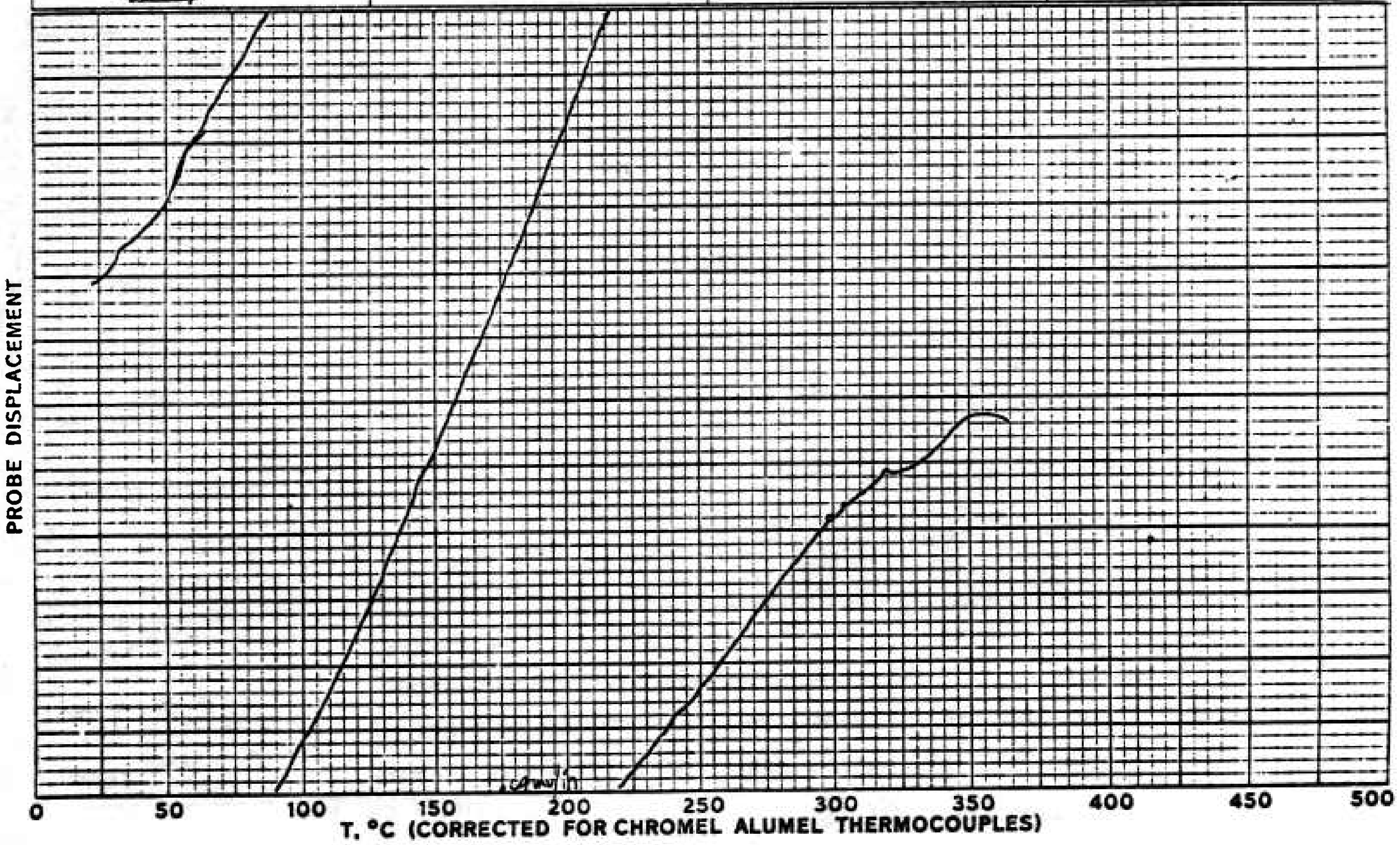
Y-AXIS SCALE .02 mv/in

Y-AXIS SENSITIVITY ______ in. PROBE DISPL./in. OF CHART

RUN NO. 64

DATE 7-31-74

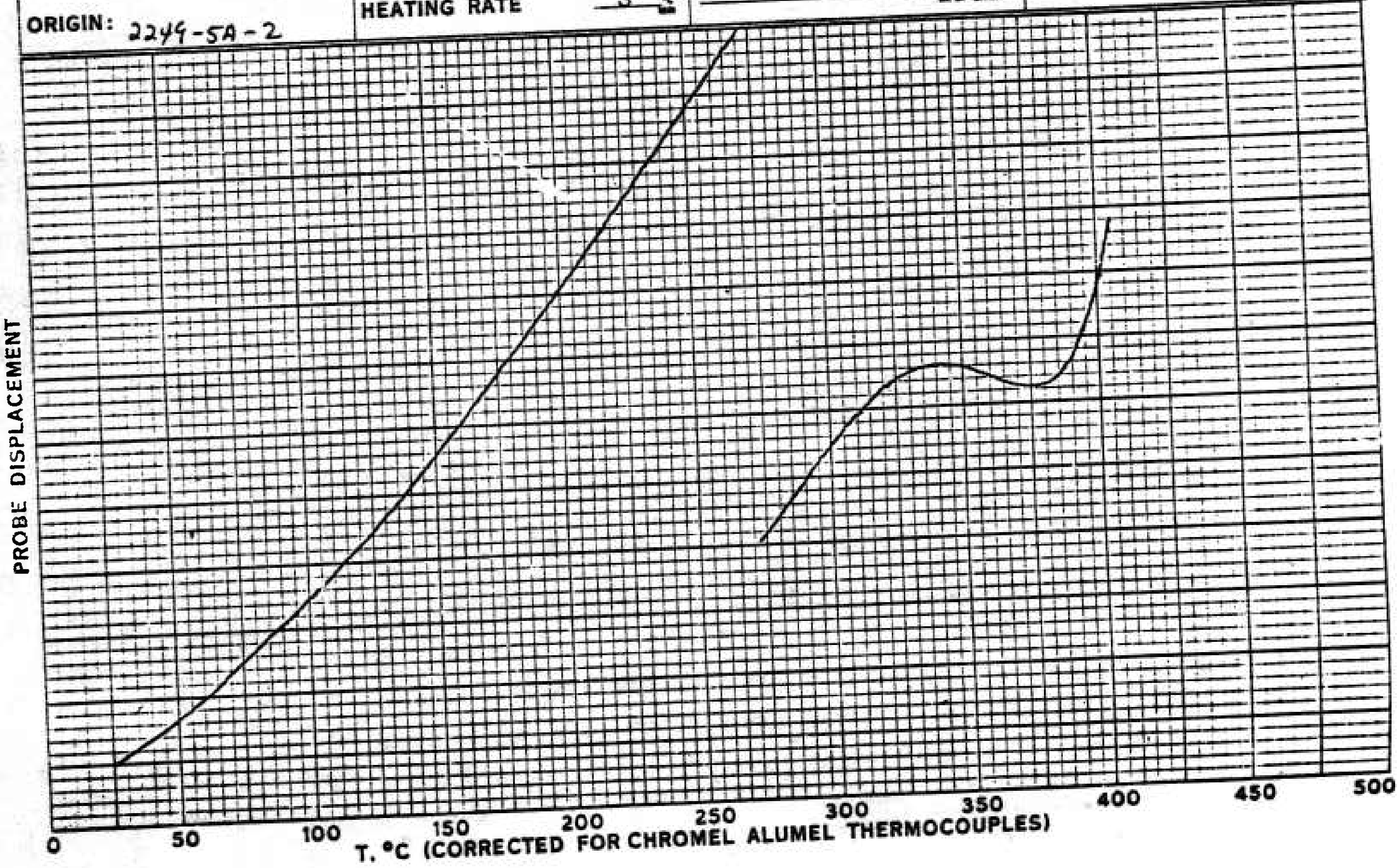
OPERATOR D Dayton



DU PONT INSTRUMENTS

PART NO. 941017

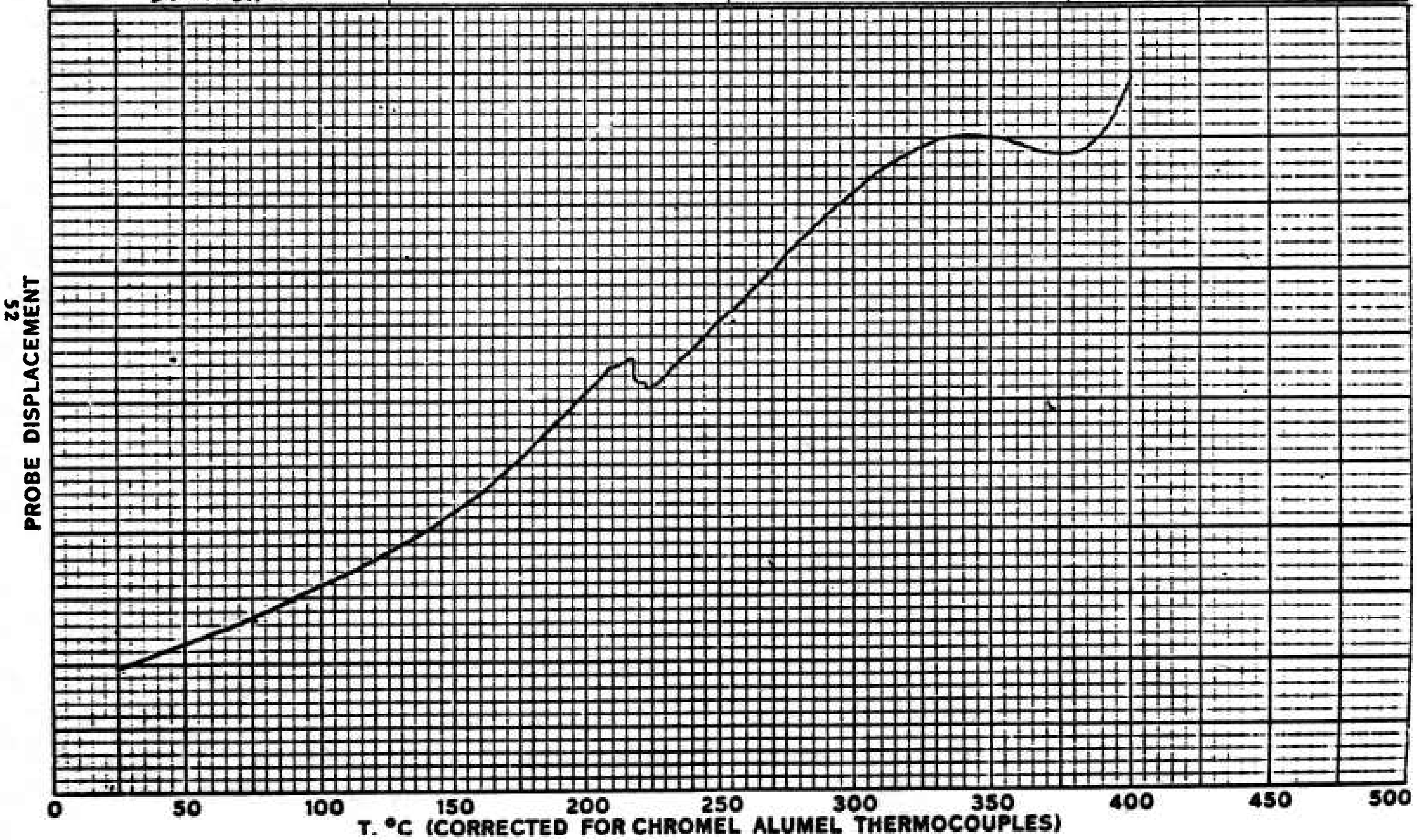
SAMPLE: HystL G-2000	SAMPLE HEIGHT .25x.25"x.25"	X-AXIS SCALE 50	RUN NO. 76
	LOADING ON TRAY 100g	Y-AXIS SCALE .04	DATE 8-6-74
	PROBE: EXPANSION/PENETRATION	Y-AXIS SENSITIVITY	OPERATOR D.Dayton
ORIGIN: 2249-5A-2	HEATING RATE 5	in. probe displ. / in. of chart	



PART NO. 941017

DUPONT INSTRUMENTS

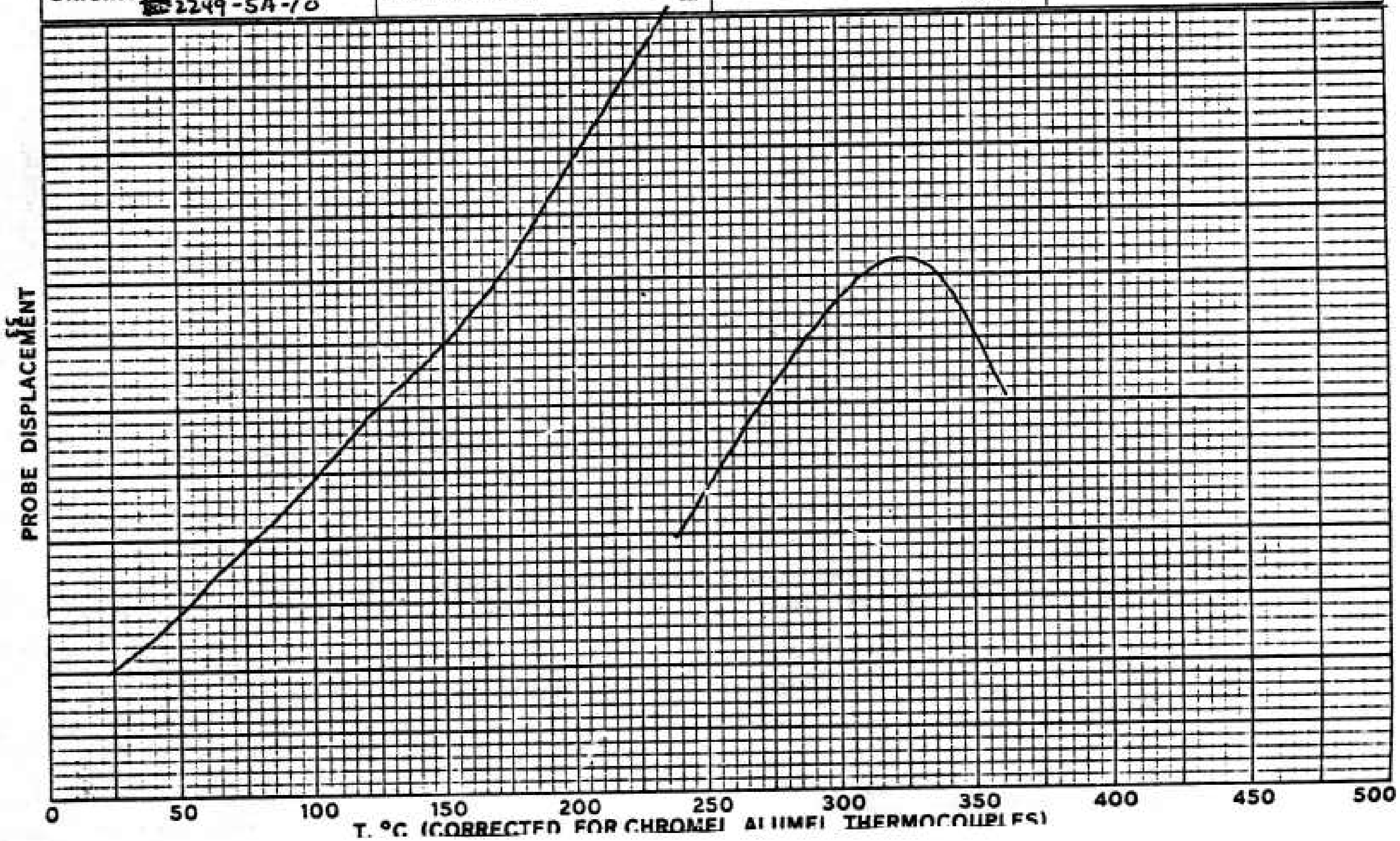
SAMPLE: Hystl G-3000	SAMPLE HEIGHT .25x.25x.25	X-AXIS SCALE 50 °C/in.	RUN NO. 75
	LOADING ON TRAY 100g	Y-AXIS SCALE .04	DATE 8-6-74
	PROBE: EXPANSION/PENETRATION	Y-AXIS SENSITIVITY	OPERATOR D E Dayton
ORIGIN: 2249-5A-10	HEATING RATE 5 °C/min	in. probe displ./in. of chart	



DUPONT INSTRUMENTS

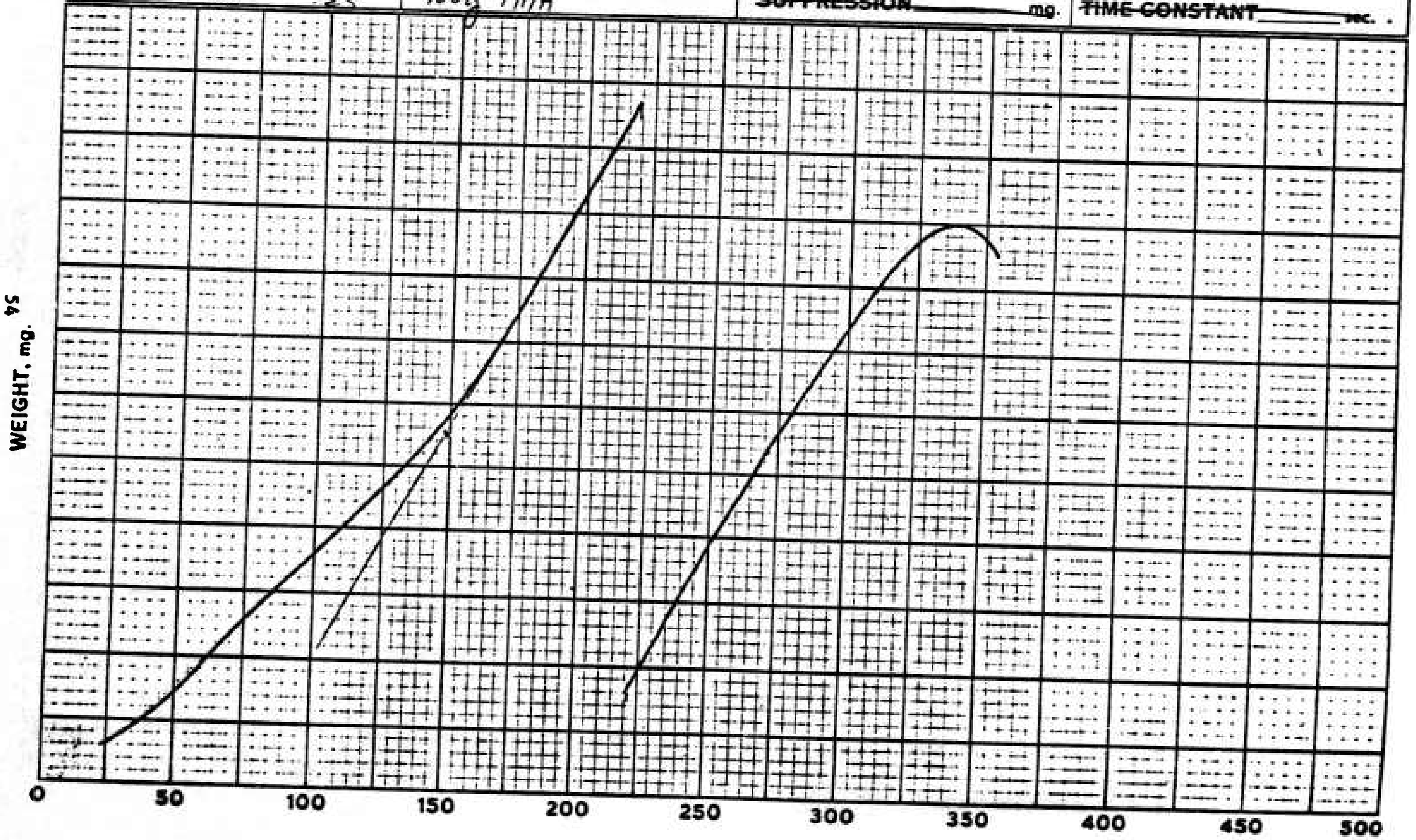
PART NO. 941017

SAMPLE: HysTL G-3000 24 hr H2O Boil 29 day H2O Soak	SAMPLE HEIGHT .25"&.25"/.25"	X-AXIS SCALE 50	RUN NO. 128
	LOADING ON TRAY 100g	Y-AXIS SCALE .04	DATE 8-28-74
	PROBE: EXPANSION/PENETRATION	Y-AXIS SENSITIVITY	OPERATOR D. Dayton
ORIGIN: 2249-5A-10	HEATING RATE 5		



DUPONT INSTRUMENTS

SAMPLE: Hystl B-1200 (2249-5A-4) Dried 250°F, 1hr, stored in Dessicator.	**X-AXIS**	**Y-AXIS**	RUN NO. 36 DATE 6/25/79
	TEMP. SCALE 50 °C/inch	SCALE mg./inch .04 mil/in (SCALE SETTING X 2)	OPERATOR JHa
	SHIFT 0 inch		HEATING RATE 5 °C/min.
SIZE mg. .25 x .25 x .25	100g 7mA	SUPPRESSION mg.	ATM. N2
			~~TIME CONSTANT~~ sec.



54

DUPONT INSTRUMENTS

PART NO. 941017

SAMPLE: Kystl B 1000
25½ hr H2O Boil
H2O Soak
33 DAYS

ORIGIN: 2249-5A-4

SAMPLE HEIGHT .25x.25x.25

LOADING ON TRAY 100 g

PROBE: EXPANSION/PENETRATION

HEATING RATE 5 °C/MIN.

X-AXIS SCALE 50 °C/DIV.

Y-AXIS SCALE .02 MV/IN.

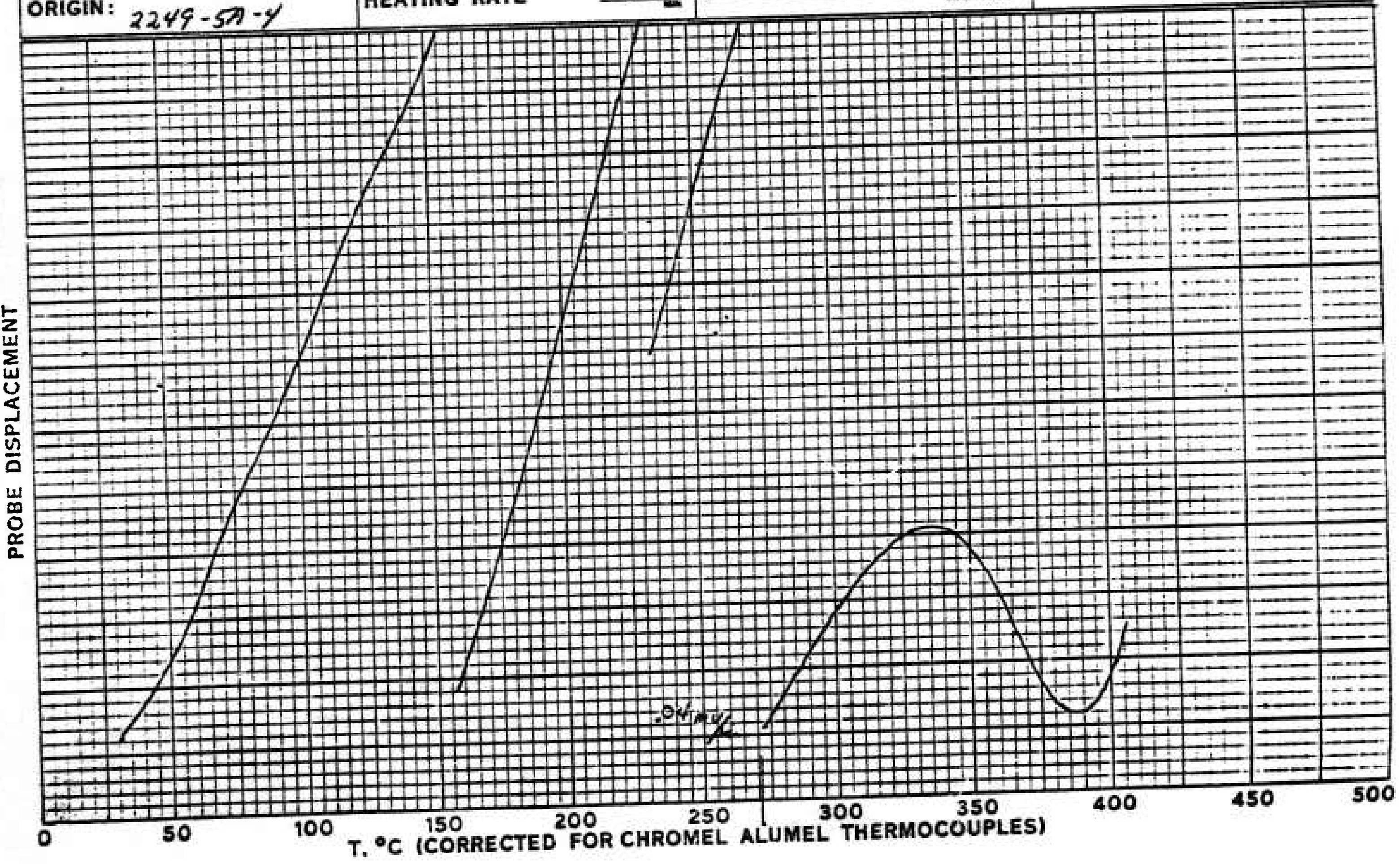
Y-AXIS SENSITIVITY

IN. PROBE DISPL./IN. OF CHART

RUN NO. 56

DATE 7-29-74

OPERATOR D Dayton



DU PONT INSTRUMENTS

SAMPLE: Hyst/ B-2000 (2219-5A-6-) Dried 250°F, stored in dessicator

SIZE .25 X .25 x 25

X-AXIS

TEMP. SCALE 50 °C/inch

SHIFT 0 inch

100g TMA

Y-AXIS

SCALE .04 mv/in (SCALE SETTING x 2)

SUPPRESSION ______ mg.

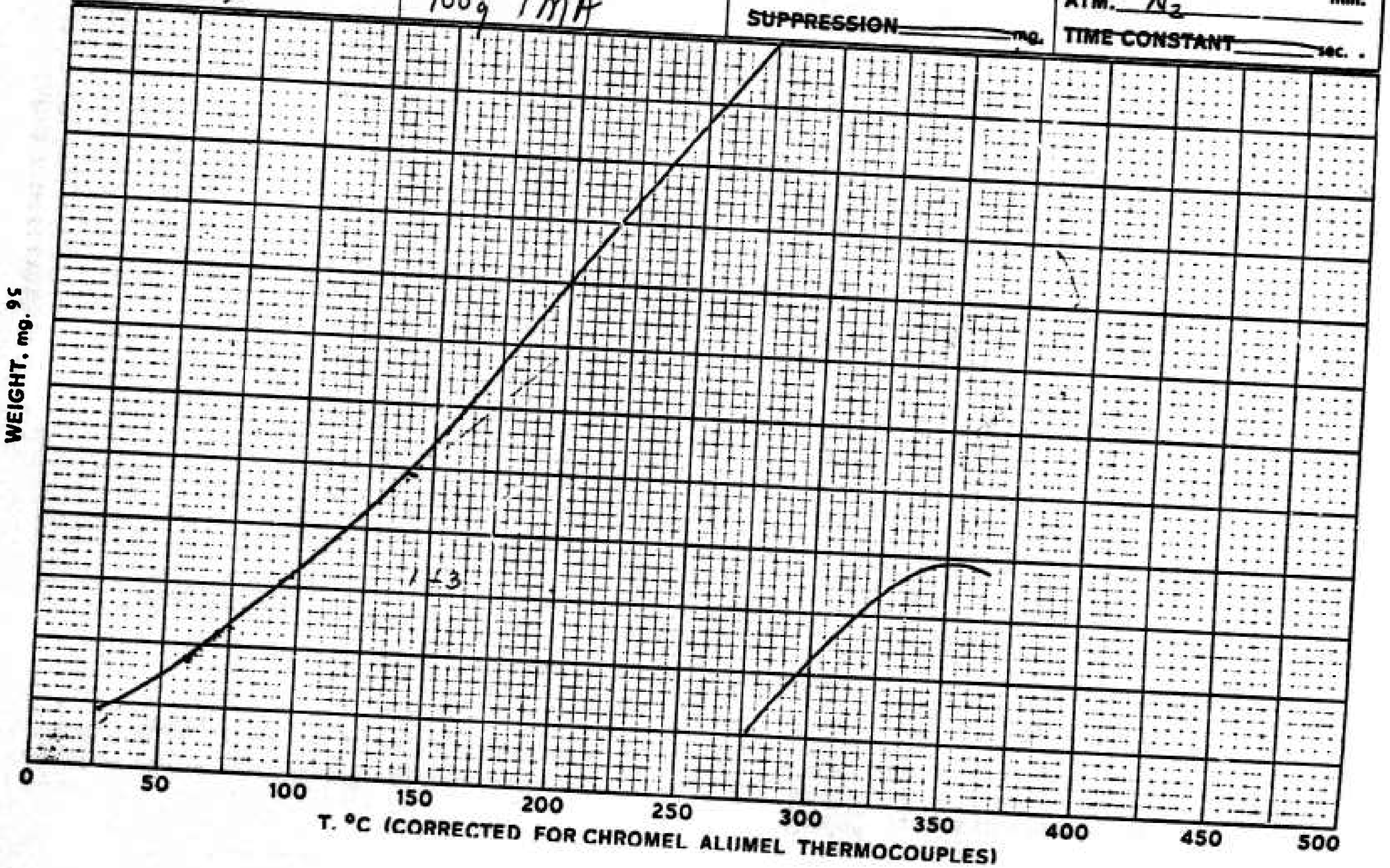
RUN NO: 38 DATE 6/25/74

OPERATOR JHa

HEATING RATE 5 °C/min.

ATM. N2

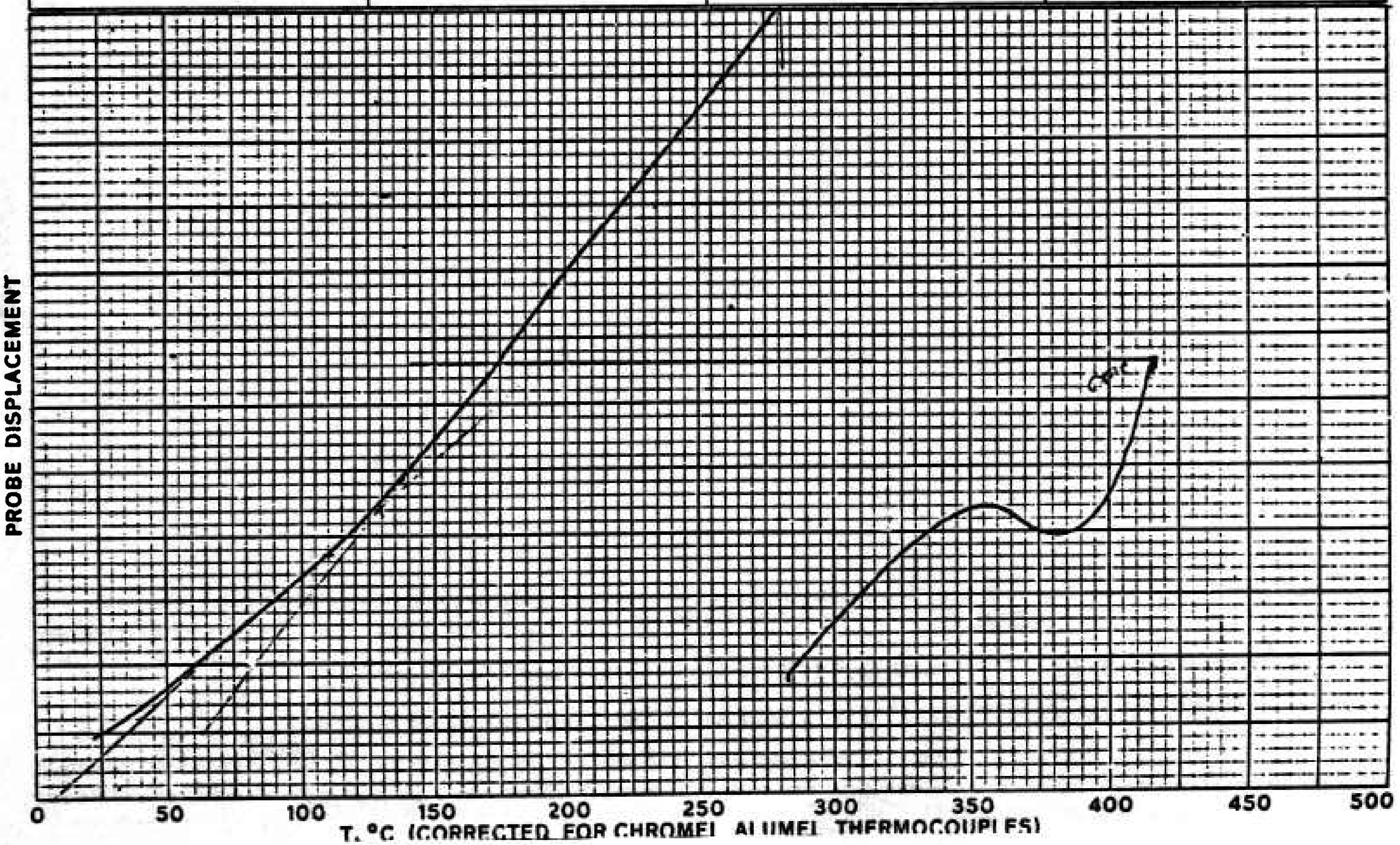
~~TIME CONSTANT~~ ______ sec.



DU PONT INSTRUMENTS

PART NO. 941017

SAMPLE: Hust! B-2000 24 hr H2O boil - 24 hr soak	SAMPLE HEIGHT .25 x .25 x .25	X-AXIS SCALE 50	RUN NO. 48
	LOADING ON TRAY 100g	Y-AXIS SCALE .04	DATE 2/3/74
	PROBE: EXPANSION/PENETRATION	Y-AXIS SENSITIVITY	OPERATOR JHa
ORIGIN: 2219-3A-6	HEATING RATE 5		



DUPONT INSTRUMENTS

PART NO. 941017

SAMPLE: Hystl B-3000

ORIGIN: 2249-5A-8

SAMPLE HEIGHT .25"x.25"x.25"

LOADING ON TRAY 100g

PROBE: EXPANSION/PENETRATION

HEATING RATE 5

X-AXIS SCALE 50

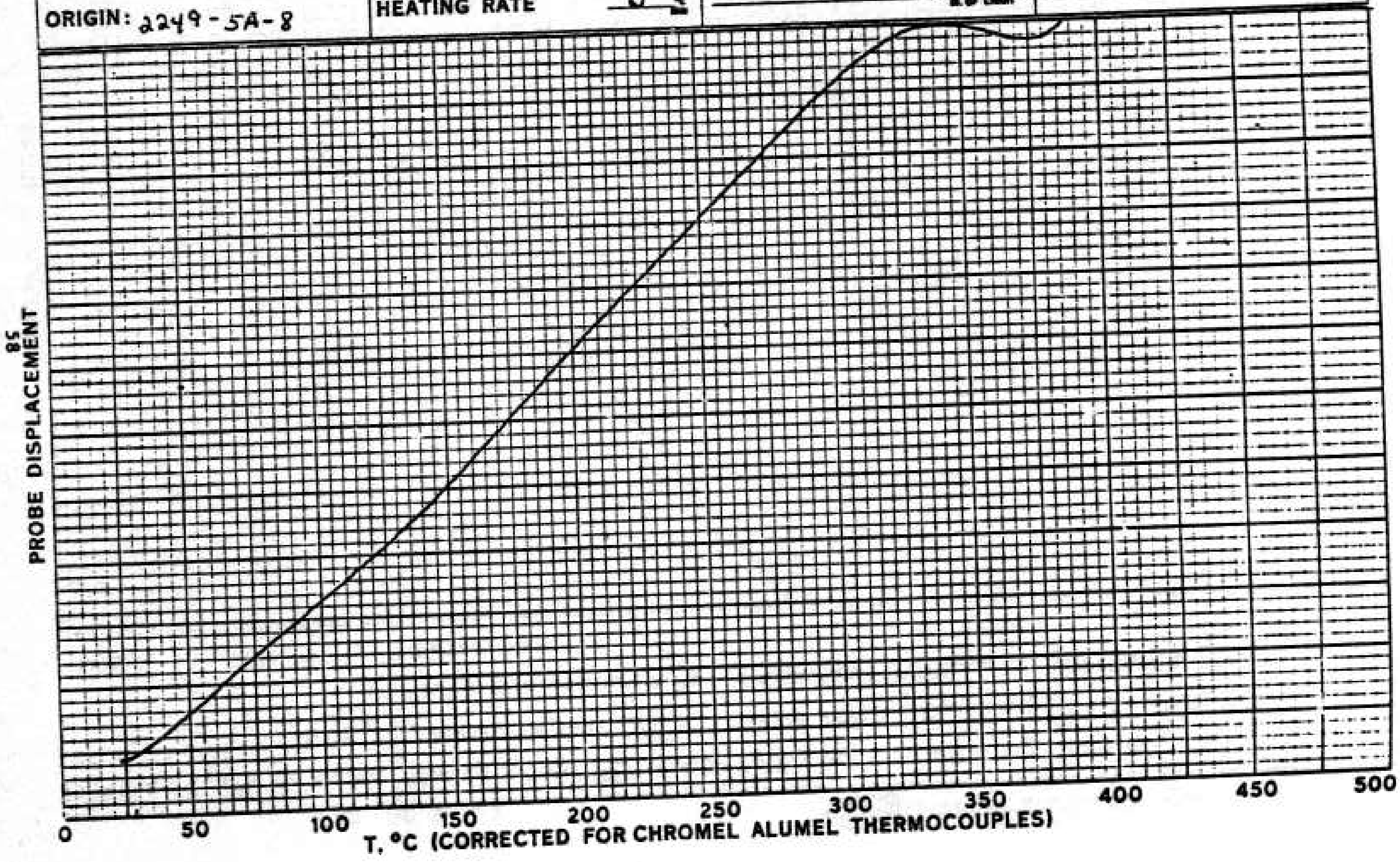
Y-AXIS SCALE .04

Y-AXIS SENSITIVITY

RUN NO. 77

DATE 8-6-74

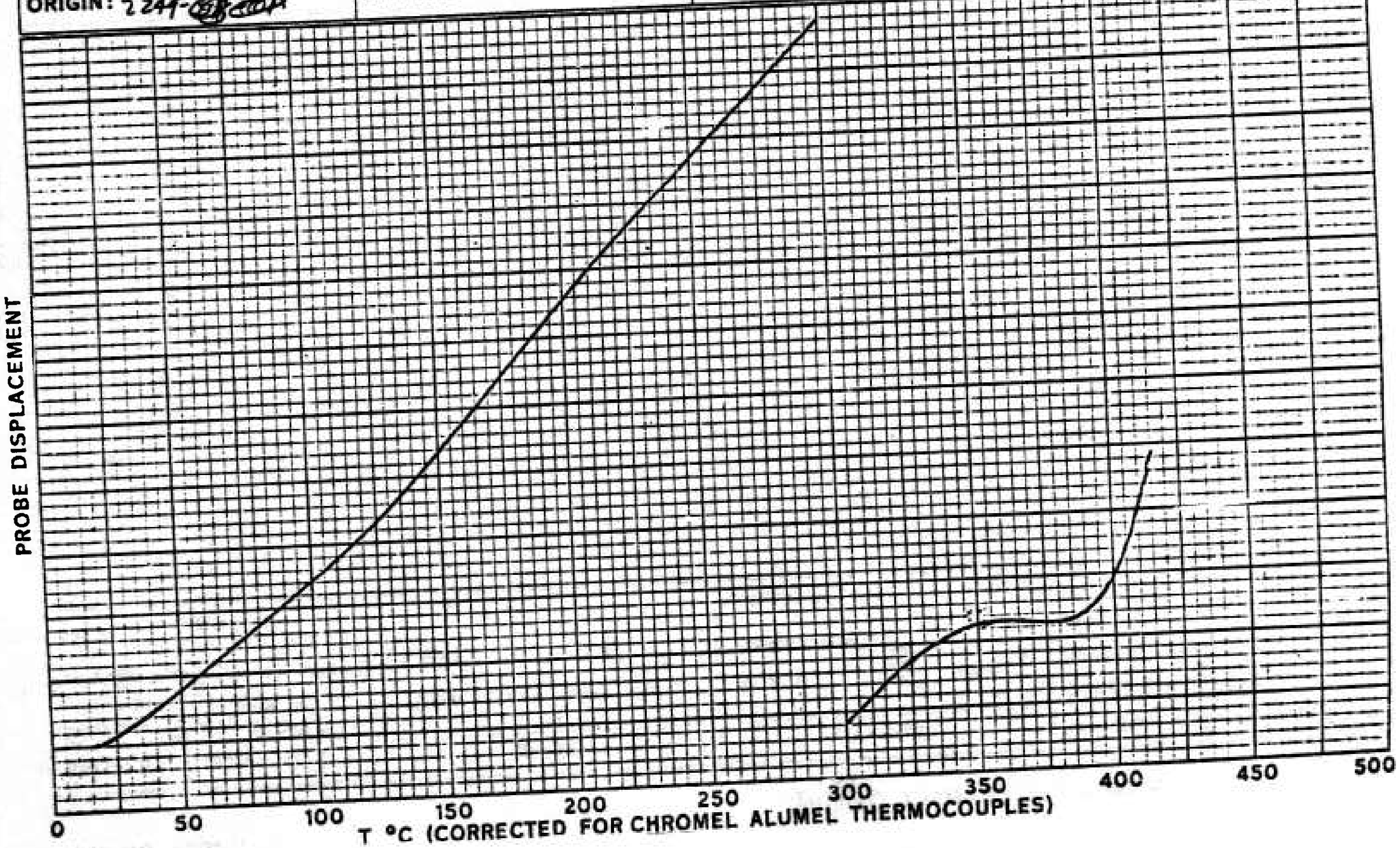
OPERATOR D E Dayton



DUPONT INSTRUMENTS

PART NO. 941017

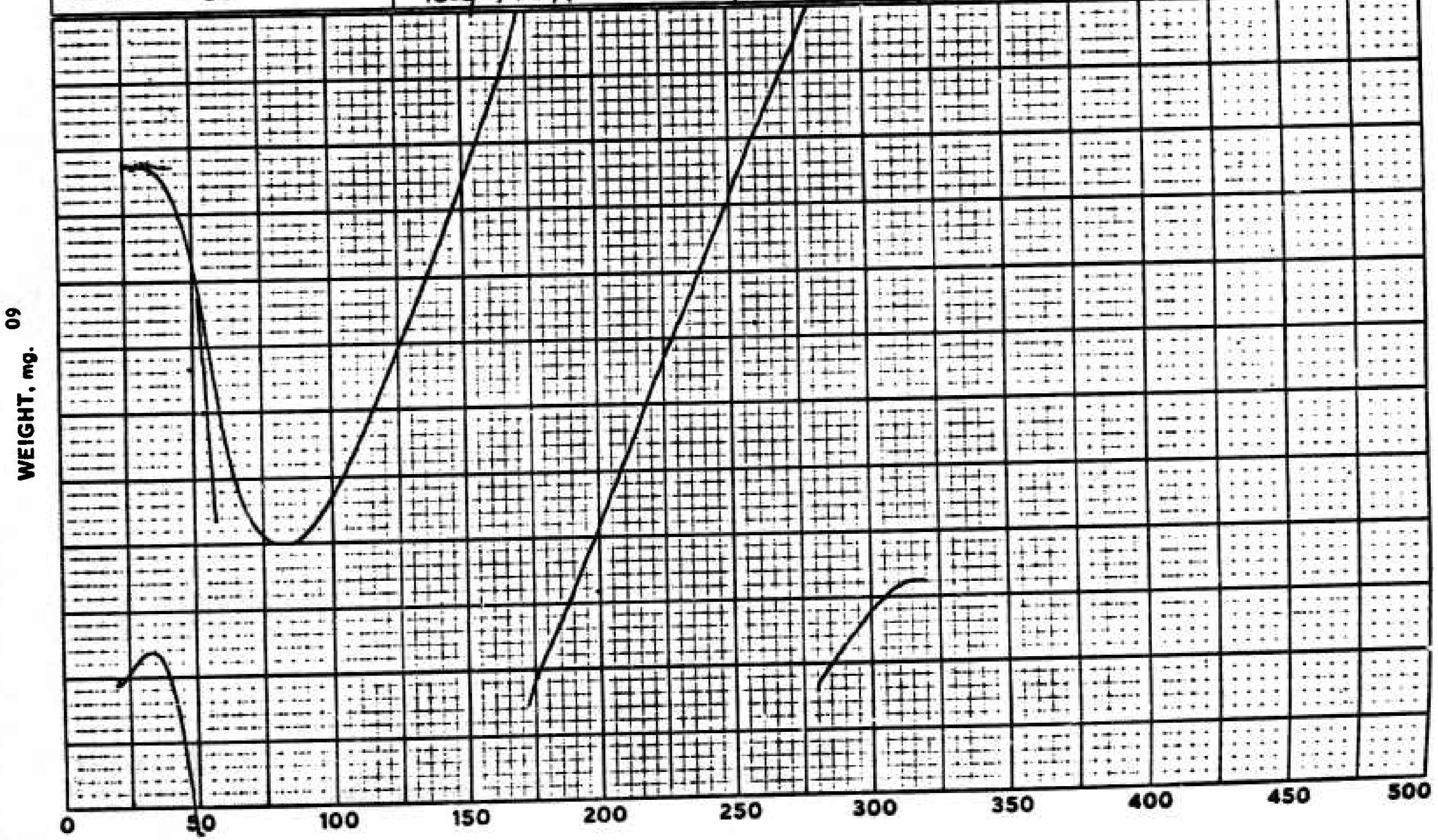
SAMPLE: Hysl B-3000 24 Hr H$_2$O boil, 24 hr soak	SAMPLE HEIGHT .25 x .25 x .25	X-AXIS SCALE 50 °C/in.	RUN NO. 50
	LOADING ON TRAY 100g	Y-AXIS SCALE .04 in./in.	DATE 9/3/74
	PROBE: EXPANSION/PENETRATION	Y-AXIS SENSITIVITY	OPERATOR
ORIGIN: 2244-SA-8	HEATING RATE 5 °C/min	in. probe displ./in. of chart	



PART NO. 990231

DUPONT INSTRUMENTS

SAMPLE: Ricon 100 Source = B27-R100B (24 hr, 450°F PC) (dried @ 250°F, stored in desiccator) .25 x .25 x .25 SIZE ~~80~~	X-AXIS TEMP. SCALE 50 °C/inch SHIFT 0 inch 100g TMA	Y-AXIS SCALE ~~[illegible]~~ .04 mil/in (SCALE SETTING X 2) ~~SUPPRESSION~~ ___ mg.	RUN NO. 33 DATE 6/29/78 OPERATOR JHa HEATING RATE 5 °C/min. ATM. N2 TIME CONSTANT ___ sec.



DUPONT INSTRUMENTS

PART NO. 941017

SAMPLE: Ricon 100B 24 hr H2O boil, 24 hr H2O soak

ORIGIN: 2249-27R100-B

SAMPLE HEIGHT .25 x .25 x .25

LOADING ON TRAY 100g

PROBE: EXPANSION/PENETRATION

HEATING RATE 5

X-AXIS SCALE 50

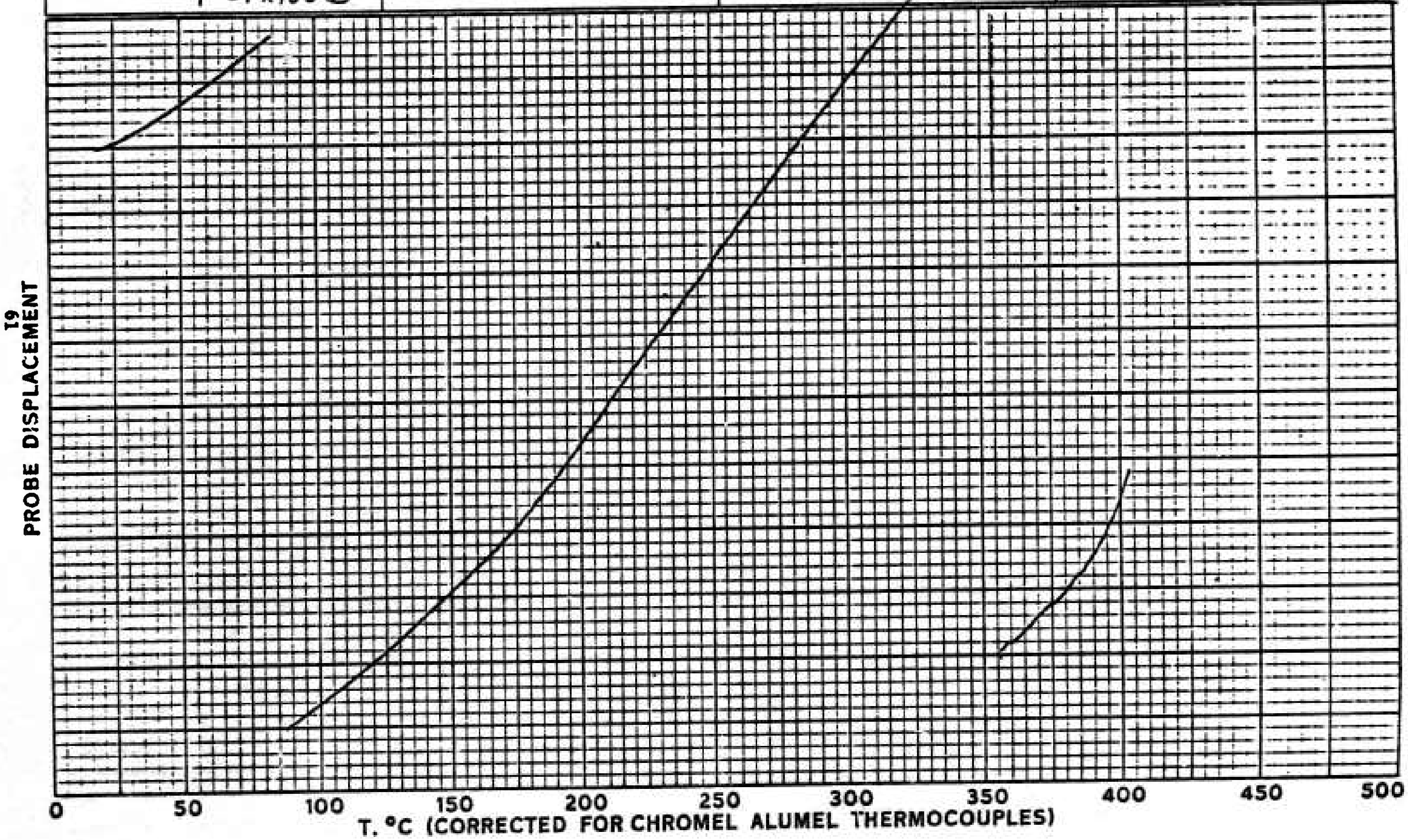
Y-AXIS SCALE .04

Y-AXIS SENSITIVITY (.043)

RUN NO. 41

DATE 6/26/74

OPERATOR



DUPONT INSTRUMENTS

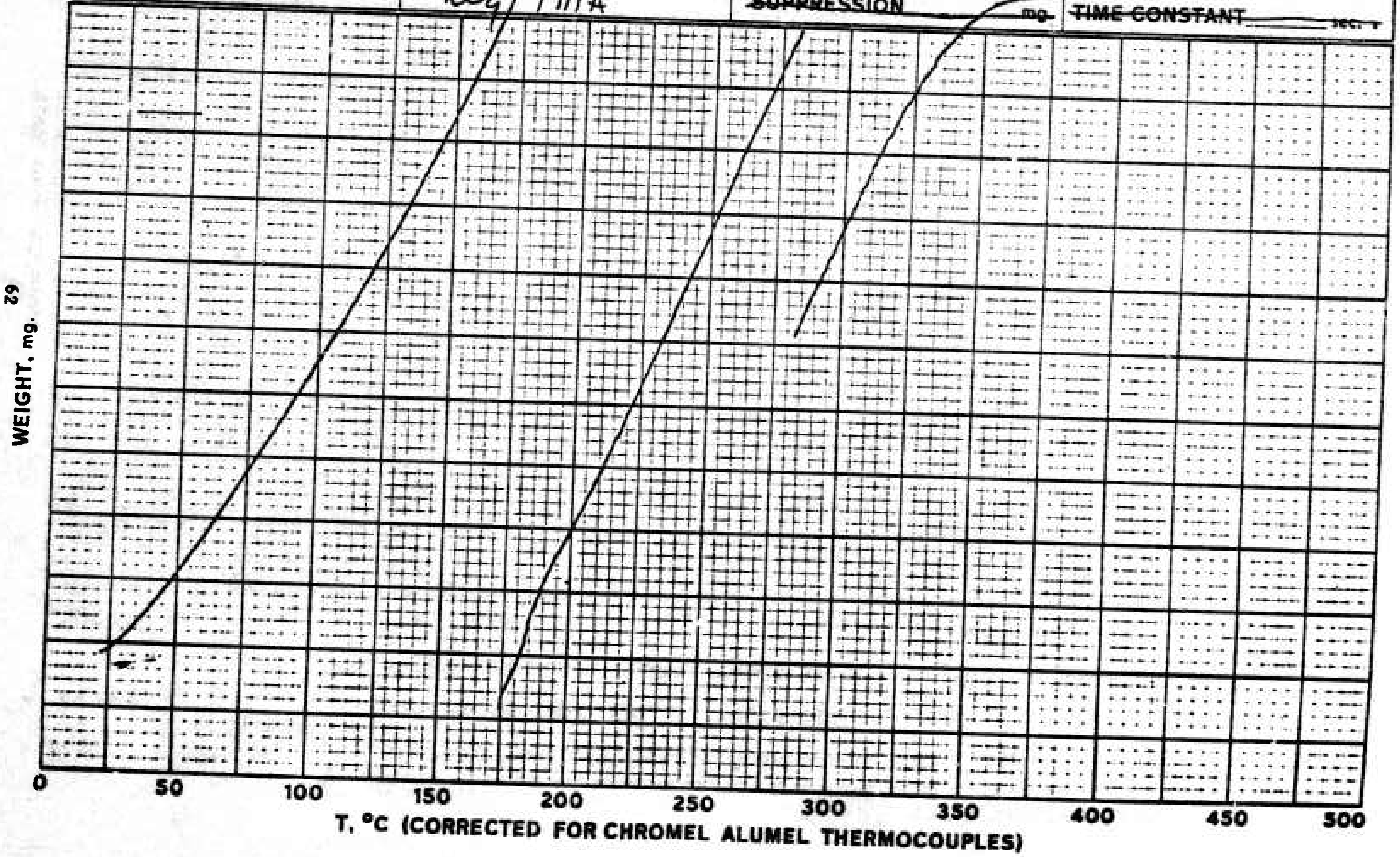
SAMPLE: Ricon 150
250°F dry -
Source - DD2299-8
SIZE ____ ~~mg.~~ .25×.25×.25

X-AXIS
TEMP. SCALE 50 °C/inch
SHIFT 0 inch

100g/TMA

Y-AXIS
SCALE ~~mg/inch~~ .02 mv/in
(SCALE SETTING X 2)
~~SUPPRESSION~~ ____ mg.

RUN NO. 22 DATE 6/19/74
OPERATOR JHa
HEATING RATE 5 °C/min.
ATM. N2
~~TIME CONSTANT~~ ____ sec.



PART NO. 941017

DUPONT INSTRUMENTS

SAMPLE: Ricon 150
24 hr H_2O boil, 24 hr soak

ORIGIN: DD-2256-8A-4

SAMPLE HEIGHT 0.25" x .25 x .25

LOADING ON TRAY 100g

PROBE: EXPANSION/PENETRATION

HEATING RATE 5 °C/min

X-AXIS SCALE 50 °C/in.

Y-AXIS SCALE 04 mv/in

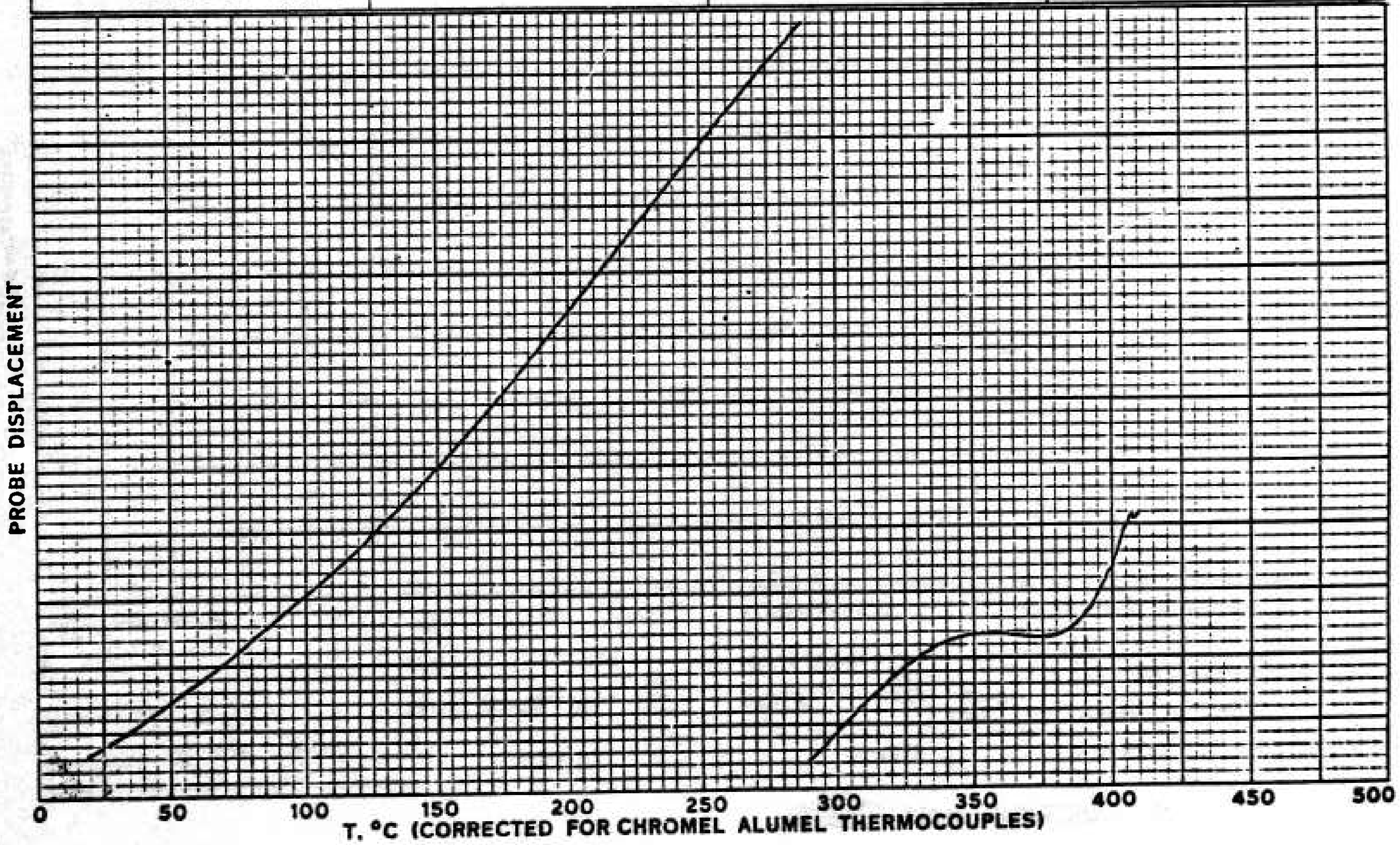
Y-AXIS SENSITIVITY .0343 in/mv

in. probe displ. / in. of chart

RUN NO. 40

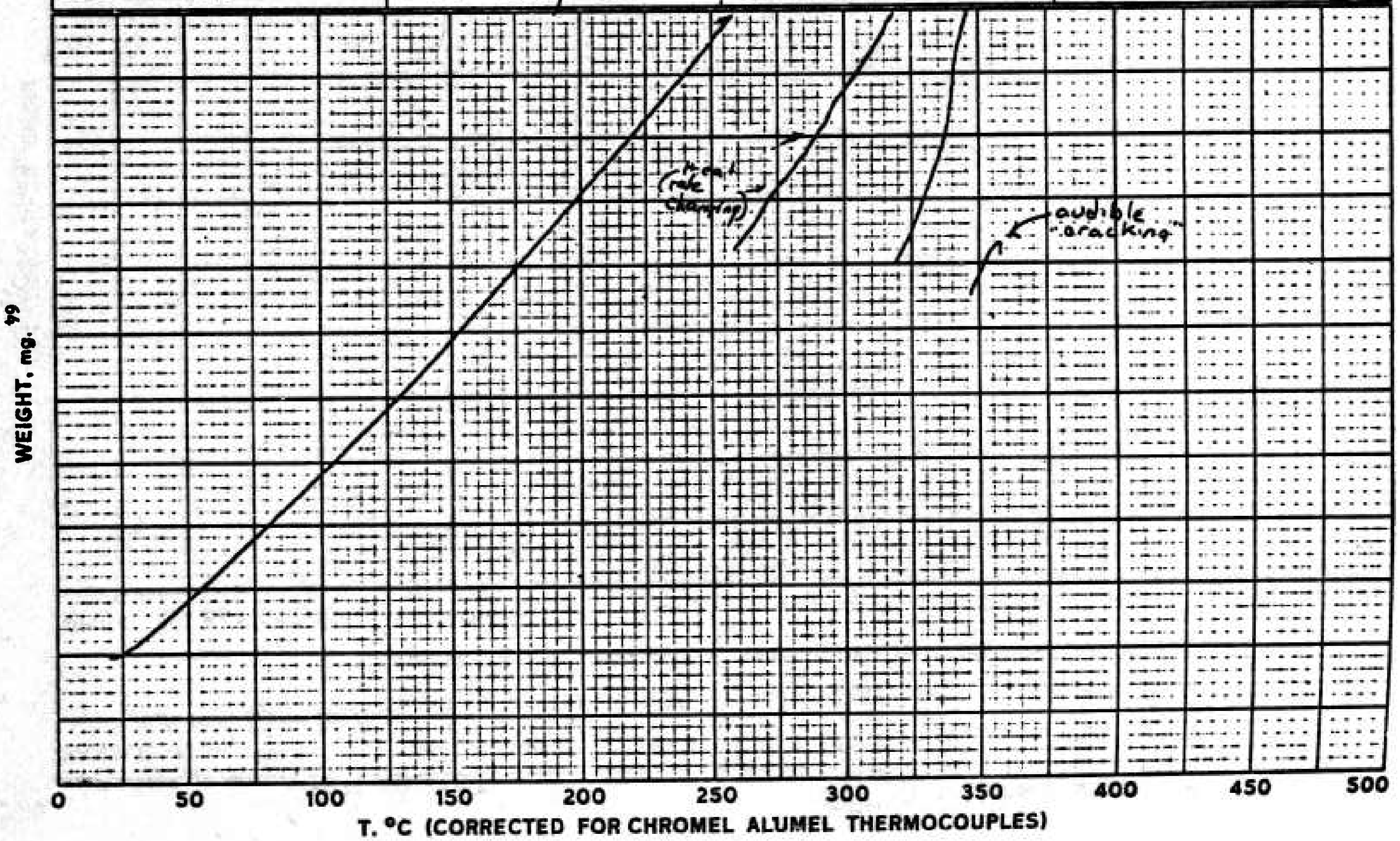
DATE 6/26/74

OPERATOR JWa



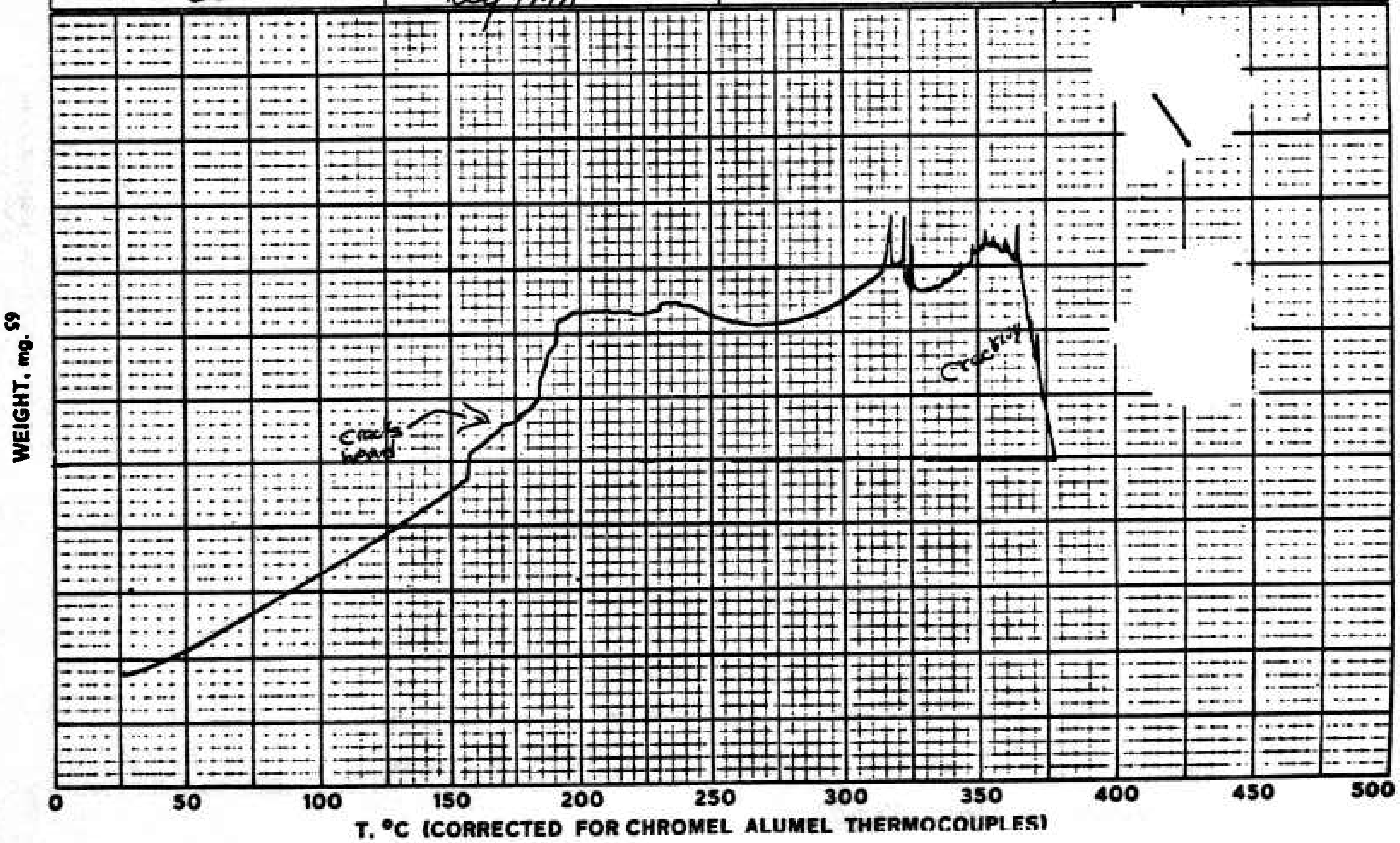
DUPONT INSTRUMENTS

SAMPLE: Keramid 601 From DD-0519-1 **SIZE** .2 × .2 × 2 **mg.**	**X-AXIS** **TEMP. SCALE** 50 **°C/inch** **SHIFT** 0 **inch** TMA 100 g load	**Y-AXIS** **SCALE** ~~mg/inch~~ .02 mil/in **(SCALE SETTING X 2)** **SUPPRESSION** ____ **mg.**	**RUN NO.** 16 **DATE** 6/18/79 **OPERATOR** JHa **HEATING RATE** [illegible].5 **°C/min.** **ATM.** N_2 ~~**TIME CONSTANT**~~ ____ **sec.**



DU PONT INSTRUMENTS

SAMPLE: Kerimid 601 24hr boil, 24 hr soak, DD -	X-AXIS	Y-AXIS	RUN NO. 31 DATE 6/21/78
	TEMP. SCALE 50 °C/inch	SCALE ~~mg/inch~~ .04 mil/in	OPERATOR [illegible]
	SHIFT 0 inch	~~(SCALE SETTING X 2)~~	HEATING RATE 5 °C/min.
			ATM. N_2
SIZE ~~mg~~ .25 x .25 x .25	100g TMA	~~SUPPRESSION mg.~~	~~TIME CONSTANT sec.~~



DUPONT INSTRUMENTS

PART NO. 941017

SAMPLE: HX-580

ORIGIN: DD-319-18B

SAMPLE HEIGHT .25"x.25"x.25

LOADING ON TRAY 100 g

PROBE: EXPANSION/PENETRATION

HEATING RATE 5

X-AXIS SCALE 50

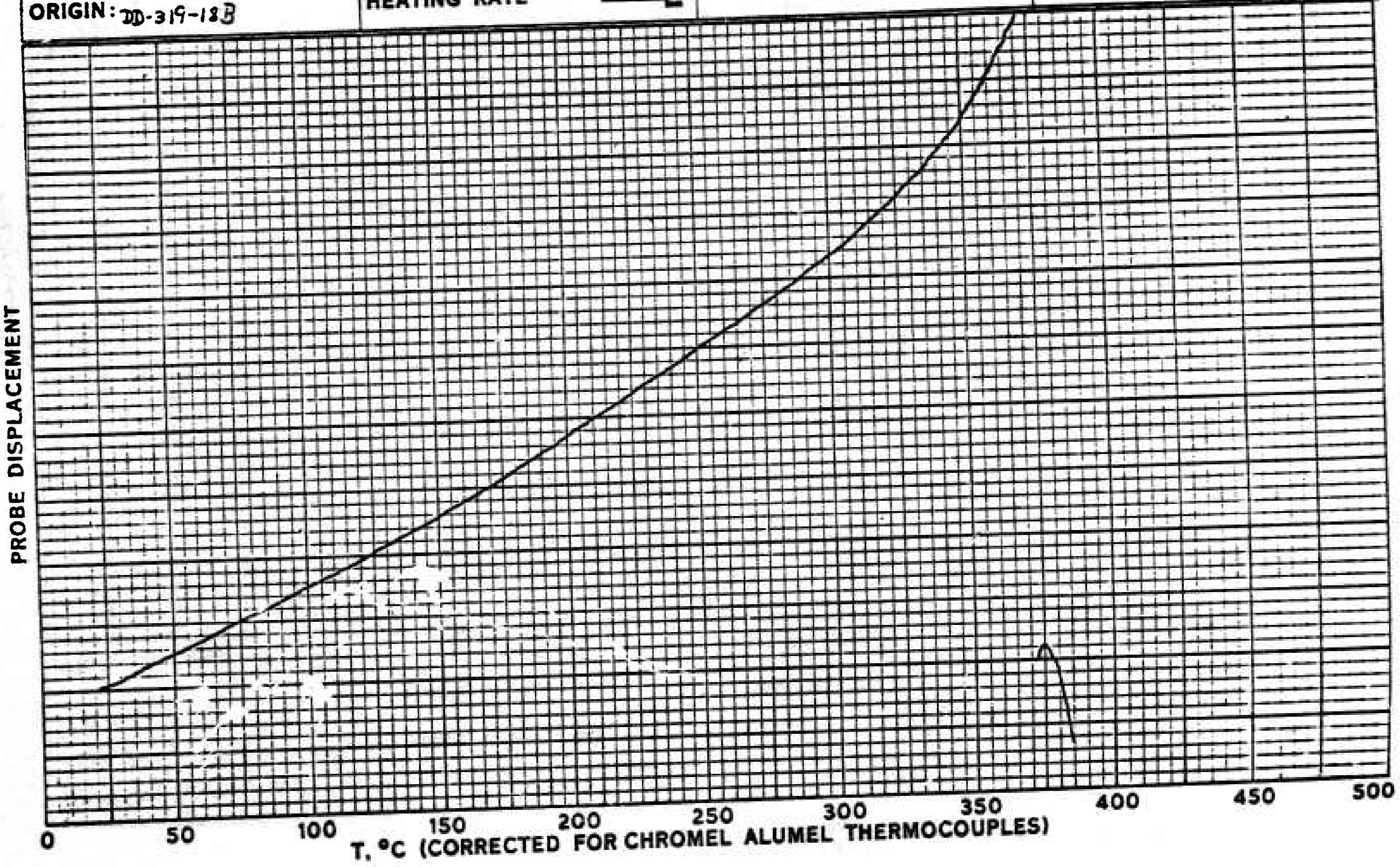
Y-AXIS SCALE .04

Y-AXIS SENSITIVITY

RUN NO. 78

DATE 8-7-74

OPERATOR DEDayton



PART NO. 941017

DUPONT INSTRUMENTS

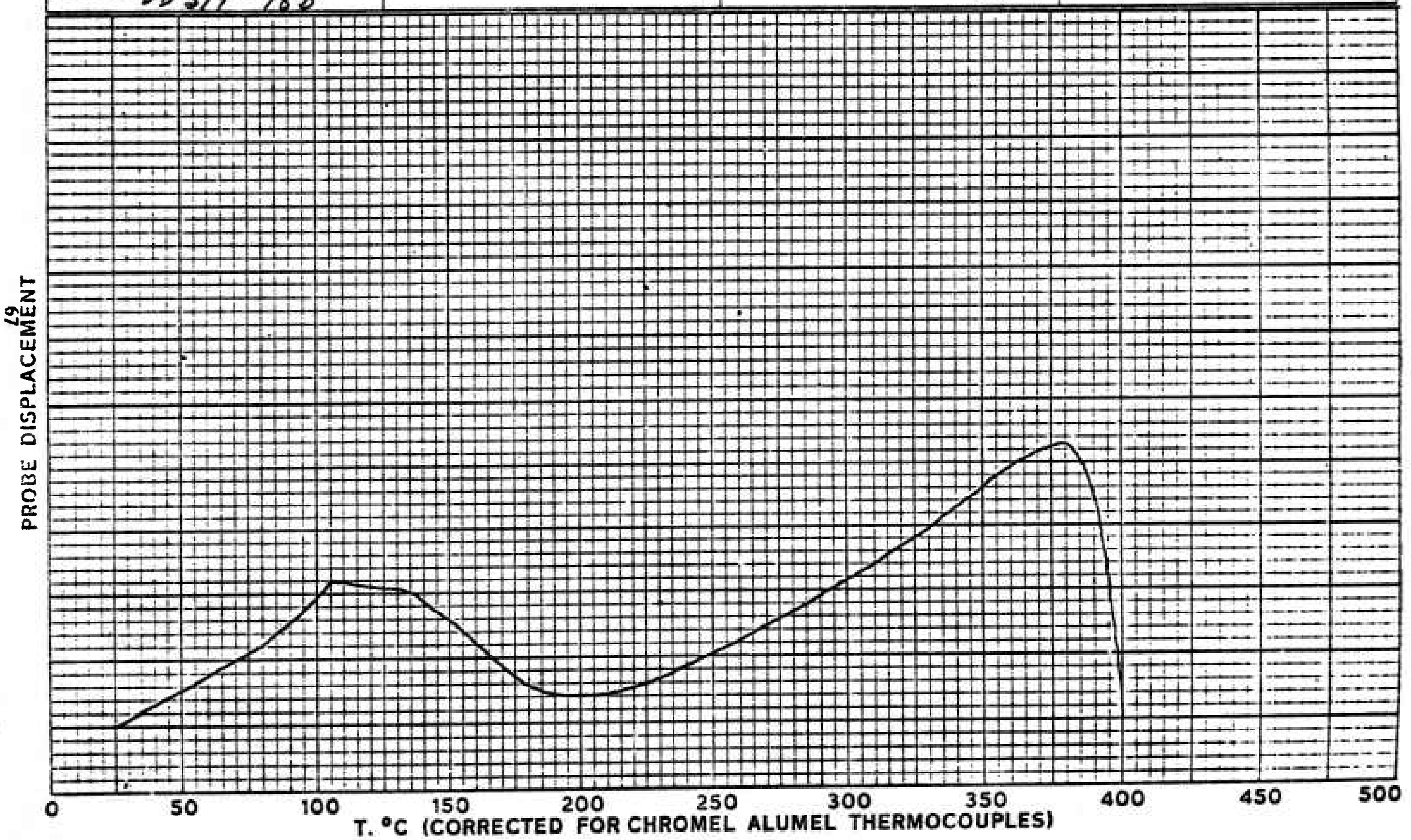
SAMPLE: HY-580
24 hr H_2O boil
30 hr H_2O soak

ORIGIN: DD-319-188

SAMPLE HEIGHT .25x.25x.25"
LOADING ON TRAY 100g
PROBE: EXPANSION/PENETRATION
HEATING RATE 5

X-AXIS SCALE 50
Y-AXIS SCALE .04
Y-AXIS SENSITIVITY

RUN NO. 80
DATE 8-8-74
OPERATOR D E Dayton



DUPONT INSTRUMENTS

SAMPLE: NR 150A
Dried @ 250°C - stored in dessicator

ORIGIN: 2253-16

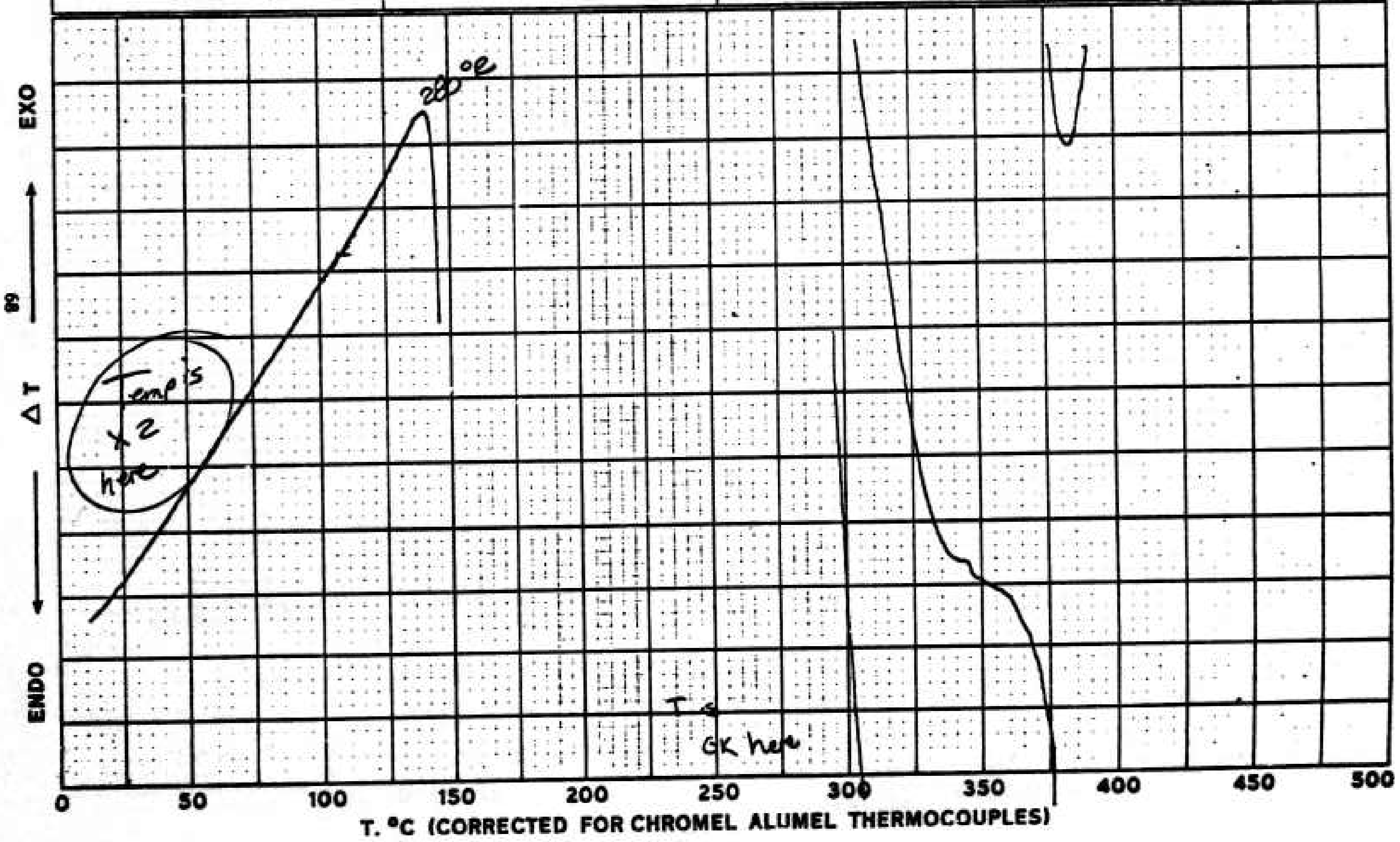
SIZE pre shapes
REF. 100g TMA
PROGRAM MODE Heat
RATE 10/~~5~~ °/min START RT °C

ATM. N_2

	T	ΔT
SCALE SETTING	~~50~~ °/in	.02 mV/in

pube pan

RUN NO. 6
DATE 6/12/79
OPERATOR 4He



DU PONT INSTRUMENTS

PART NO. 941017

SAMPLE: NR 150A - 24 hr boil, 24 hr soak

ORIGIN:

SAMPLE HEIGHT .25 x .25 x .25

LOADING ON TRAY 100g

PROBE: EXPANSION/PENETRATION

HEATING RATE 5 °C/MIN

X-AXIS SCALE 50 °C/DIV.

Y-AXIS SCALE .04 MV/IN

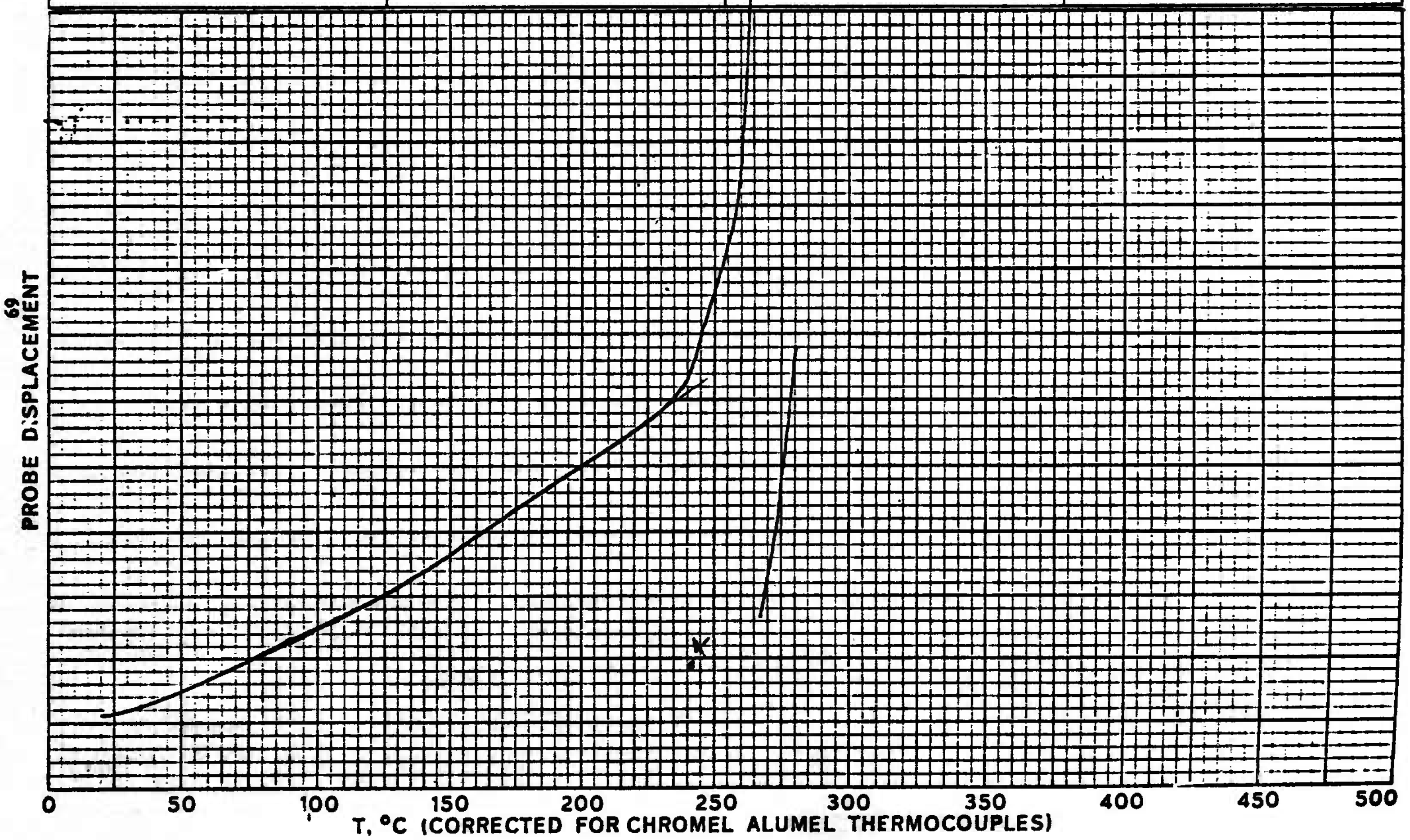
Y-AXIS SENSITIVITY

IN. PROBE DISPL. / IN. OF CHART

RUN NO. 44

DATE 6/27/79

OPERATOR 4Ha



DUPONT INSTRUMENTS

SAMPLE: Torlon 4000
Dried, 250°F 1hr

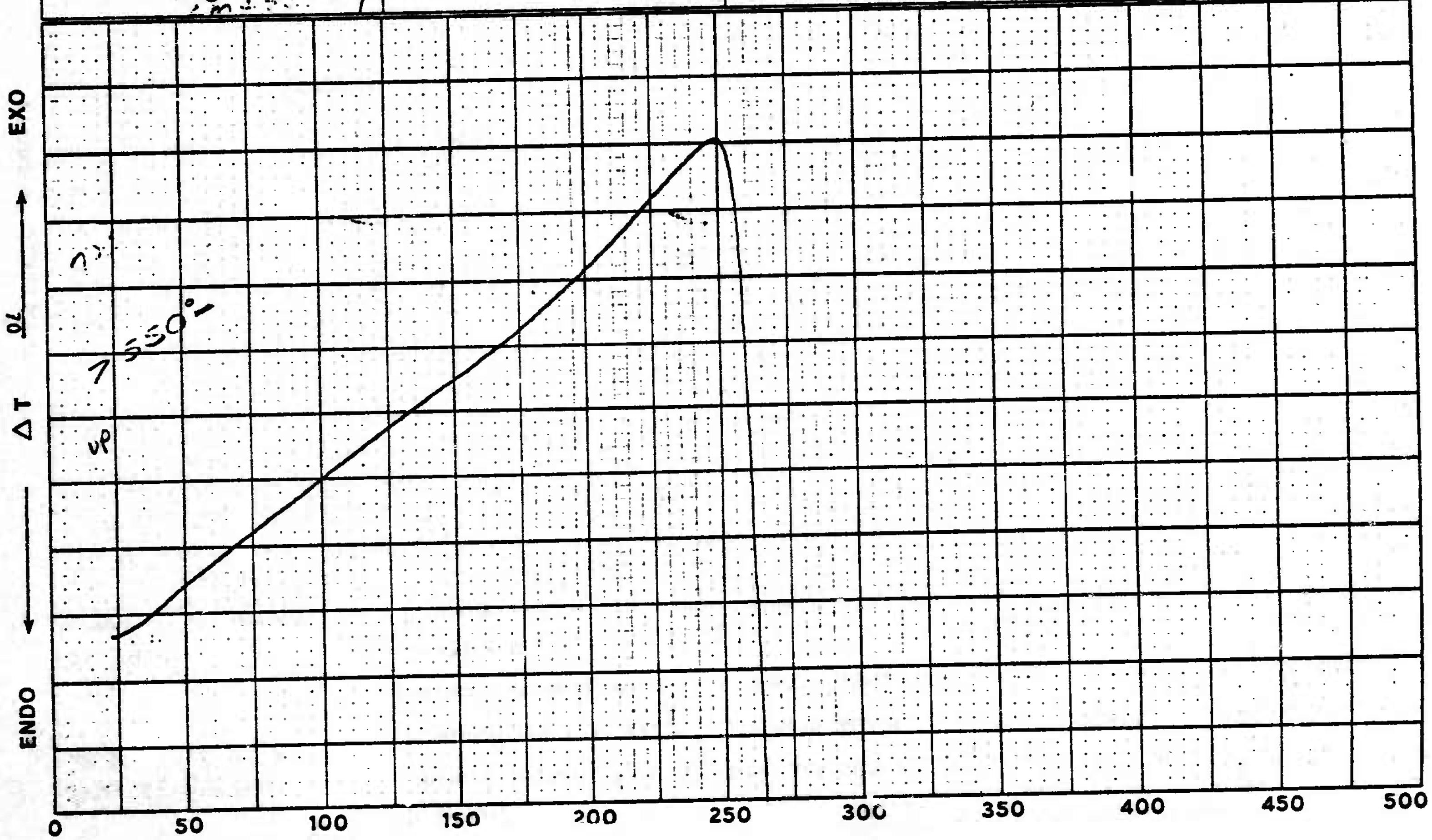
ORIGIN: Amoco Molding

SIZE cube x .2 x .2 x .2
REF. 100g load - TMA
PROGRAM MODE Heat
RATE 5 °/min, START RT °C

ATM. N_2

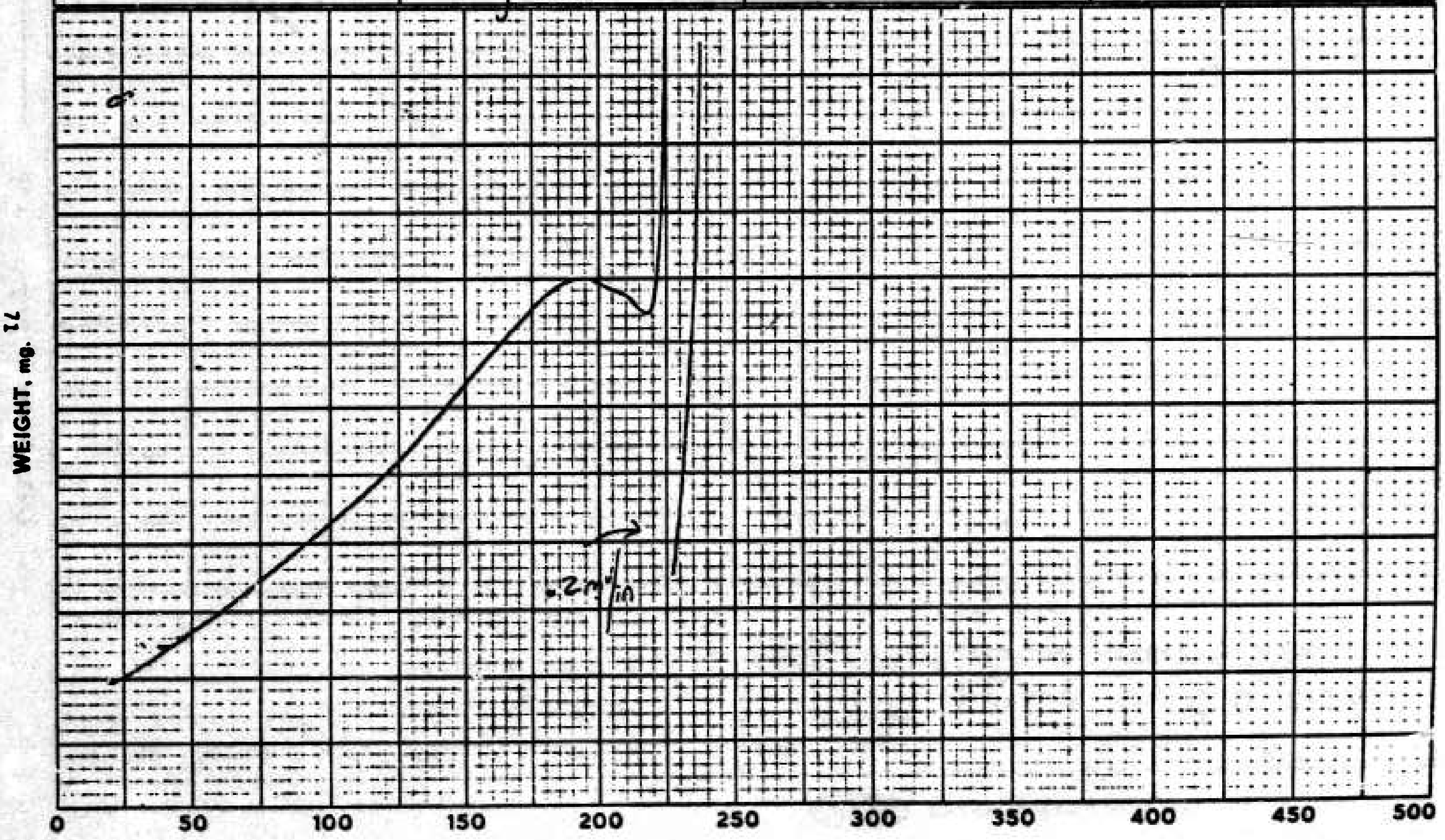
	T	ΔT
SCALE SETTING	50 °/in	.5 mil/ .2 in probe displacement

RUN NO. 3
DATE 6/11/74
OPERATOR



DU PONT INSTRUMENTS

SAMPLE: Torlon 4000 ㉓ 24 hr H_2O boil 24 hr soak LM1244 .254.3 x.2 **SIZE** ____ **mg.**	**X-AXIS** **TEMP. SCALE** 50 **°C/inch** **SHIFT** 0 **inch** 100g TMA	**Y-AXIS** **SCALE** ~~mg/inch~~ .02 mil/in **(SCALE SETTING X 2)** ~~**SUPPRESSION** ____ **mg.**~~	**RUN NO.** 24 **DATE** 6/20/74 **OPERATOR** [illegible] **HEATING RATE** 5 **°C/min.** **ATM.** N_2 ~~**TIME CONSTANT** ____ **sec.**~~



DUPONT INSTRUMENTS

SAMPLE: Torlon 2000
dried - 250°F - 1hr

ORIGIN: Amoco - Lm-321

SIZE .24 . 2x.2

REF. 100g - TMA

PROGRAM MODE heat

RATE 5 °/min START RT °C

ATM. N_2

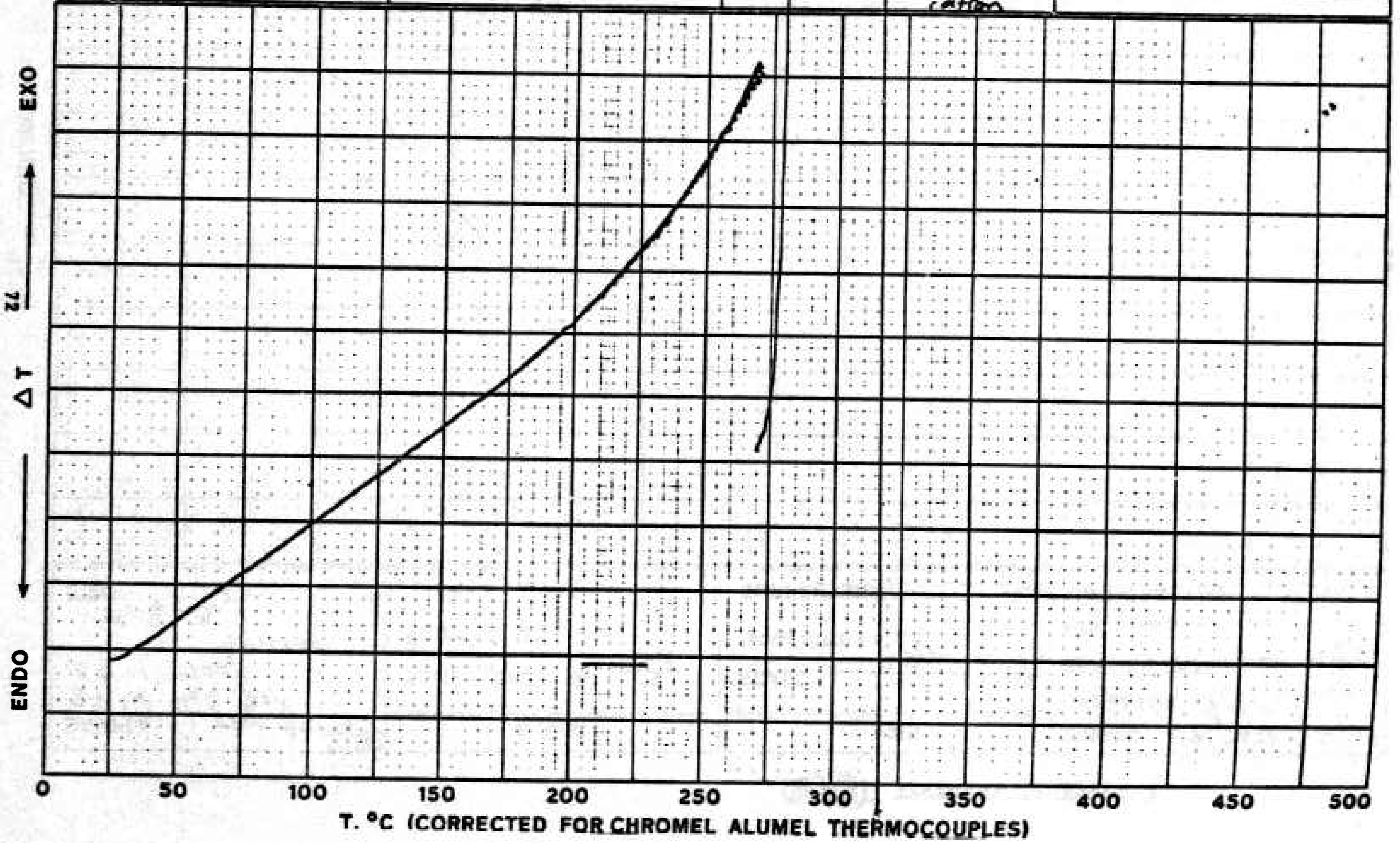
SCALE SETTING

	T	ΔT
SCALE SETTING	50 °/in	.02 [illegible] in [illegible]

RUN NO. 7

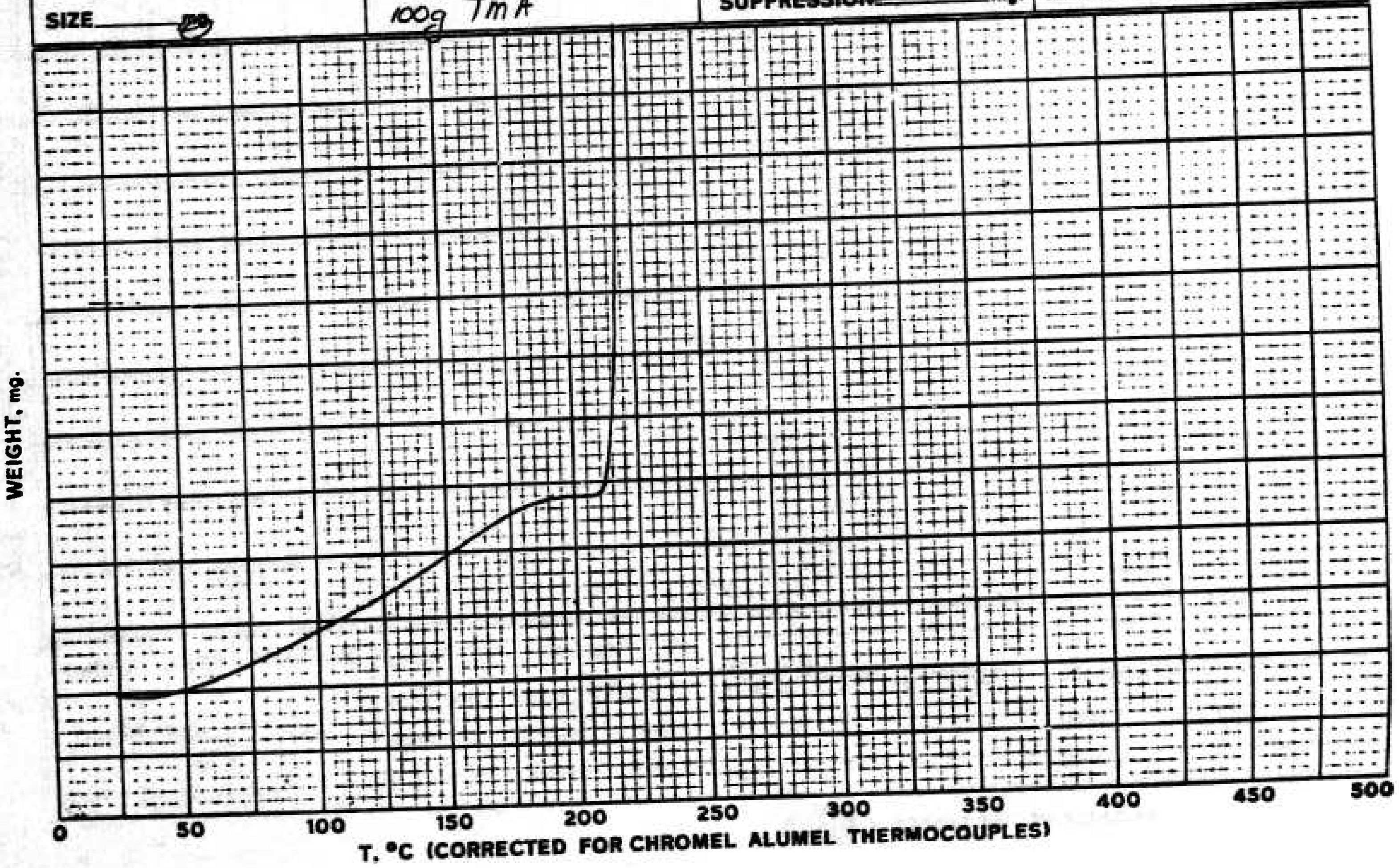
DATE 6/12/74

OPERATOR



DU PONT INSTRUMENTS

SAMPLE: Torlon 2000 (21) 24 Hr water boil, soak 24 hrs LM 932) SIZE	X-AXIS TEMP. SCALE 50 °C/inch SHIFT 0 inch 100g TMA	Y-AXIS SCALE ~~mg./inch~~ .02 mil/in (SCALE SETTING X 2) SUPPRESSION ____ mg.	RUN NO. 28 DATE 6/21/78 OPERATOR 4#a HEATING RATE 5 °C/min. ATM. N_2 TIME CONSTANT ____ sec.



DUPONT INSTRUMENTS

SAMPLE: PPG 401 #2
Dried @ 250°F, 1 hour

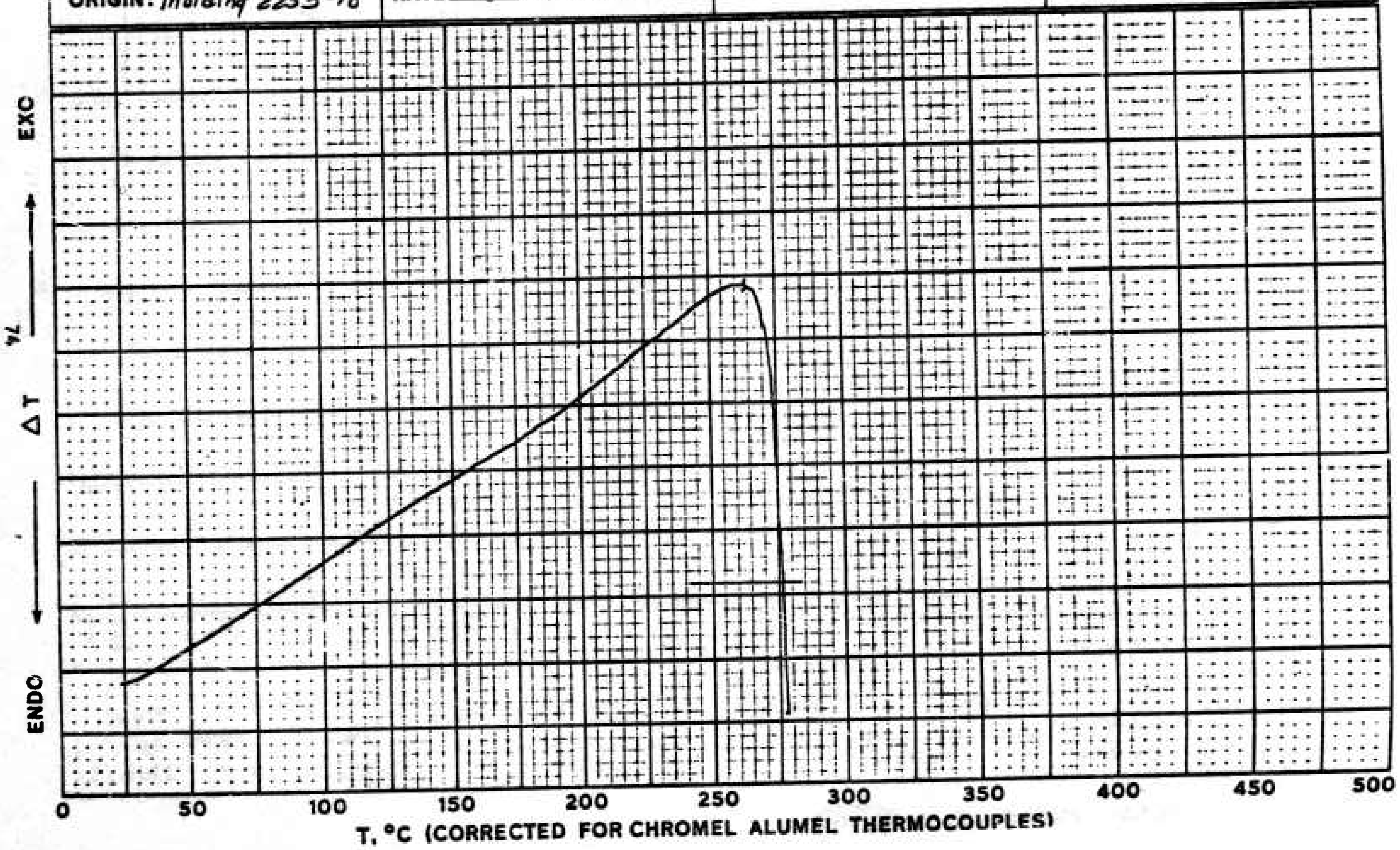
ORIGIN: Molding 2253-18

SIZE ______
REF. 100g load
PROGRAM MODE Head
RATE 5 °/min START ___ °C

ATM. ______

	T	ΔT
SCALE SETTING	50 °/in	~~.8~~ .02 mv/in

RUN NO. 2
DATE 6/10/74
OPERATOR JHG



DUPONT INSTRUMENTS

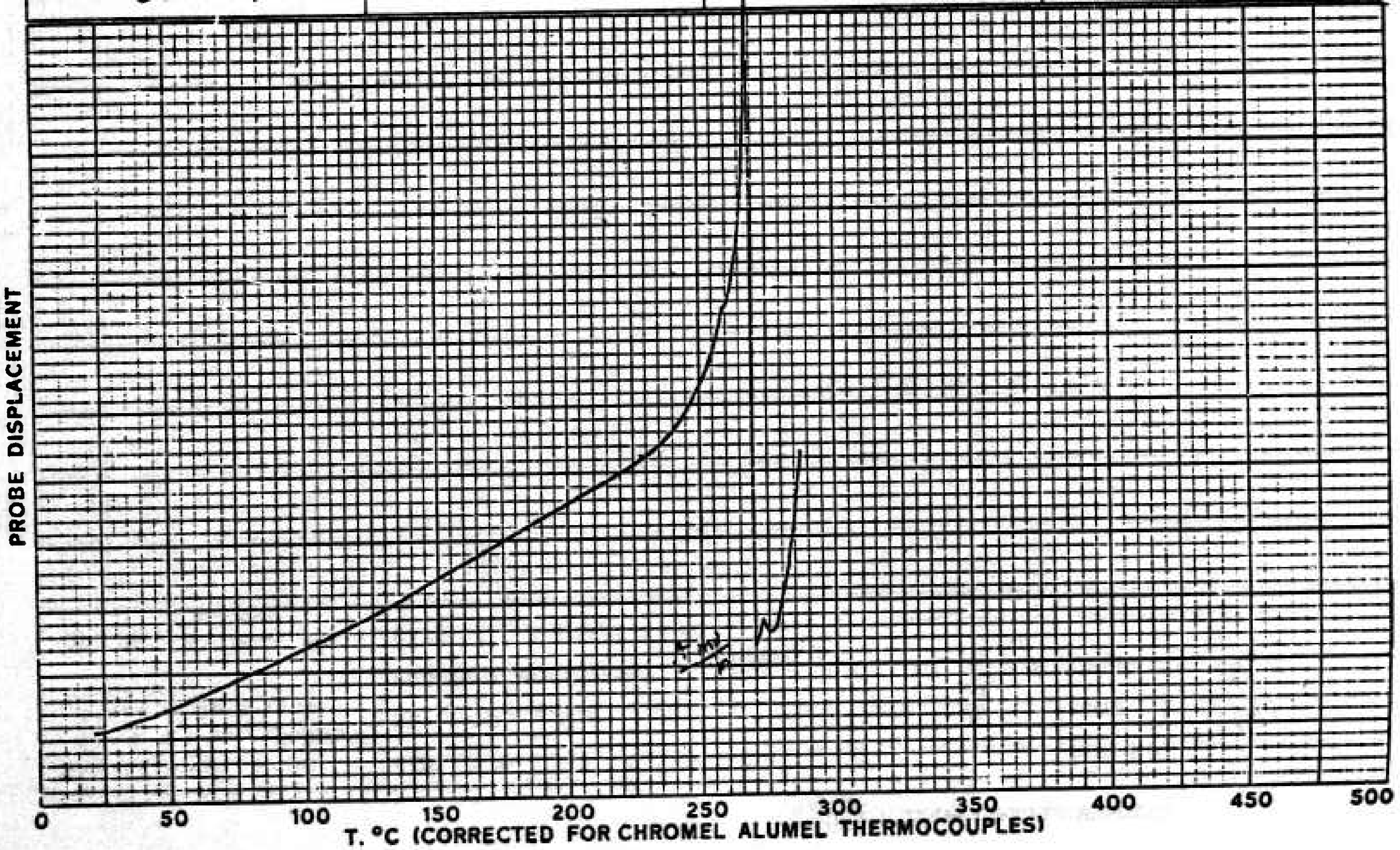
PART NO. 941017

SAMPLE: PPG 401 (27)
24 hr boil - no soak
9
ORIGIN: DD-3197

SAMPLE HEIGHT .25 x .25"
LOADING ON TRAY 100g
PROBE: EXPANSION/PENETRATION
HEATING RATE 5

X-AXIS SCALE 50
Y-AXIS SCALE .04
Y-AXIS SENSITIVITY

RUN NO. 46
DATE 6/22/74
OPERATOR JHa



DUPONT INSTRUMENTS

SAMPLE: XSR-10500
dried 23 hrs @ 250°F
stored in dessicator
(15 min only)

ORIGIN: Lee Powell from Chemistry

SIZE .2 × .2 × .2 in

REF. 100g TMA

PROGRAM MODE Heat

RATE 5 °/m, START R T °C

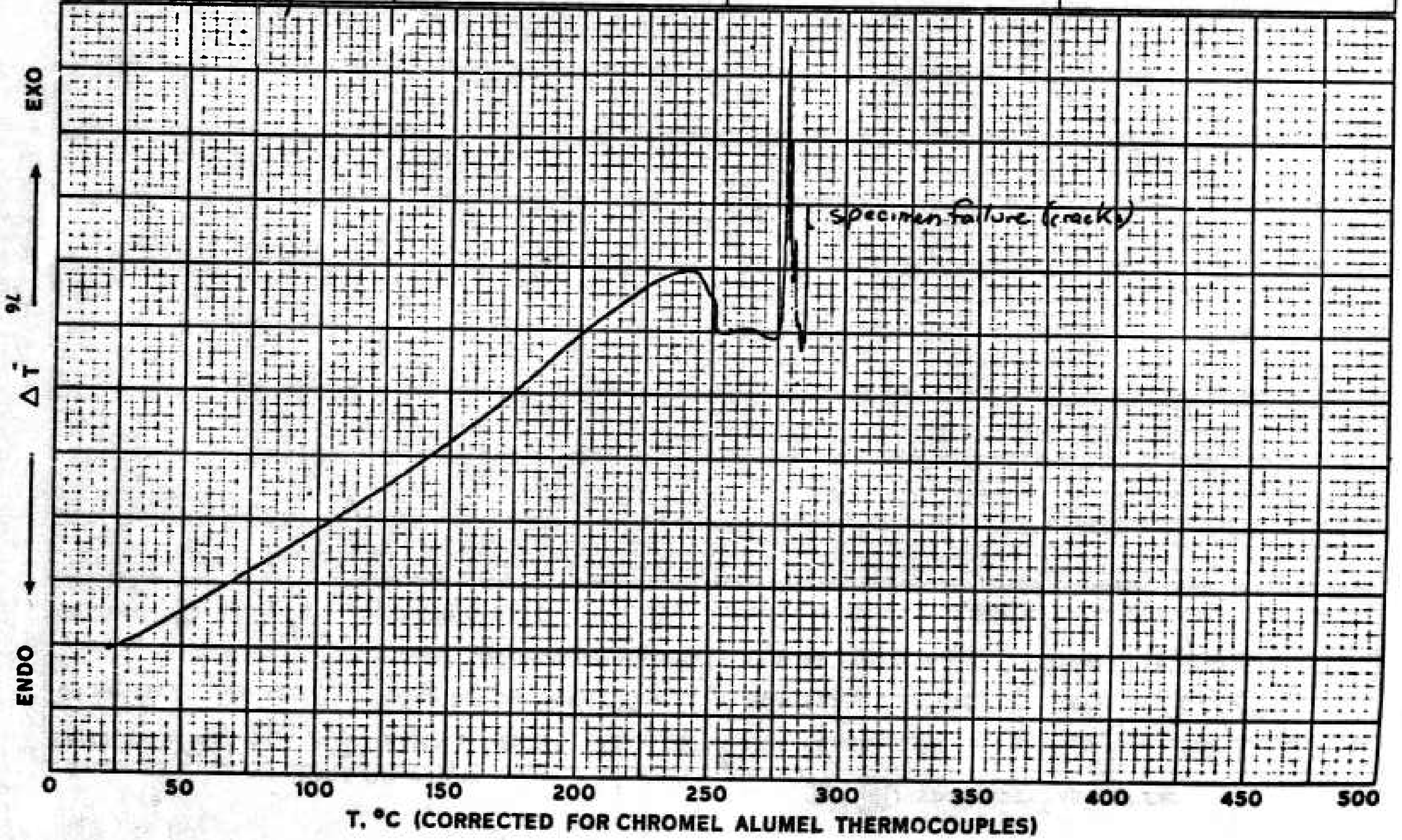
ATM. N2

	T	ΔT
SCALE SETTING	°K 20 °/in	.02 in/in °/in

RUN NO. 13

DATE 6/14/74

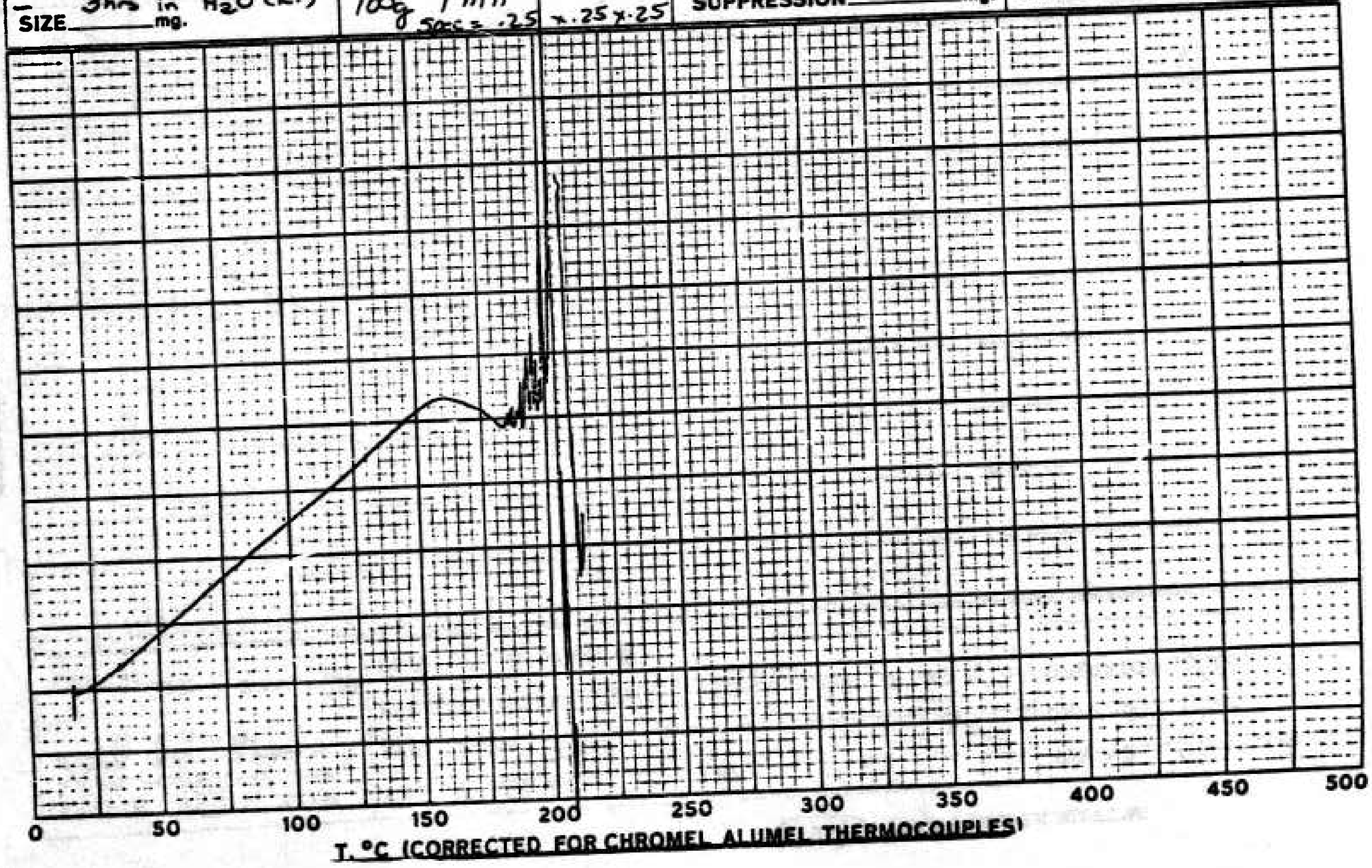
OPERATOR 4H a



PART NO. 990251

DUPONT INSTRUMENTS

SAMPLE: XSR-10500 From same panel as 13 + 14 - - 24 hr H_2O boil - 3hrs in H_2O (RT) SIZE ____ mg.	X-AXIS TEMP. SCALE 50 °C/inch SHIFT 0 inch 100g TMA Spec = .25 × .25 × .25	Y-AXIS SCALE ~~mg/inch~~ .2 .5 mv/in (SCALE SETTING X 2) SUPPRESSION ____ mg.	RUN NO. 18 DATE 6/18/78 OPERATOR JHa HEATING RATE 5 °C/min. ATM. N_2 ~~TIME CONSTANT~~ ____ sec.



DUPONT INSTRUMENTS

SAMPLE: ICI 300P Polyethersulfone - dried @ 250°F, 1hour - stored in dessicator

ORIGIN: DD - 2256 - 20A

SIZE .1 x .1 x .1

REF. 100g TMA

PROGRAM MODE Heat

RATE 5 *, START RT °C

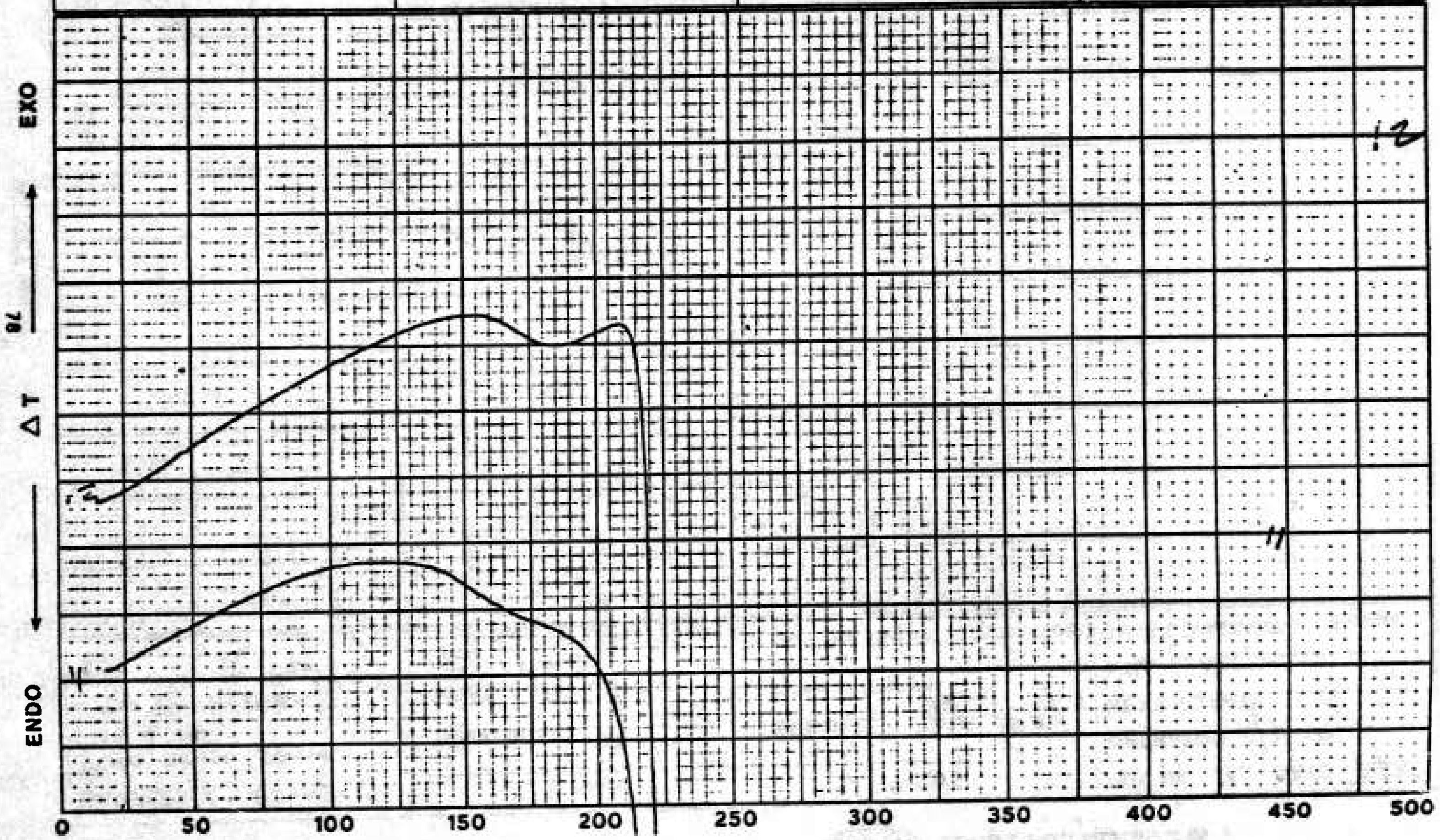
ATM. N2

	T	ΔT
SCALE SETTING	50 *	.02 ~~.05~~ mv/in *

RUN NO. 11,~~52~~ 12

DATE 6/13/74

OPERATOR JHW



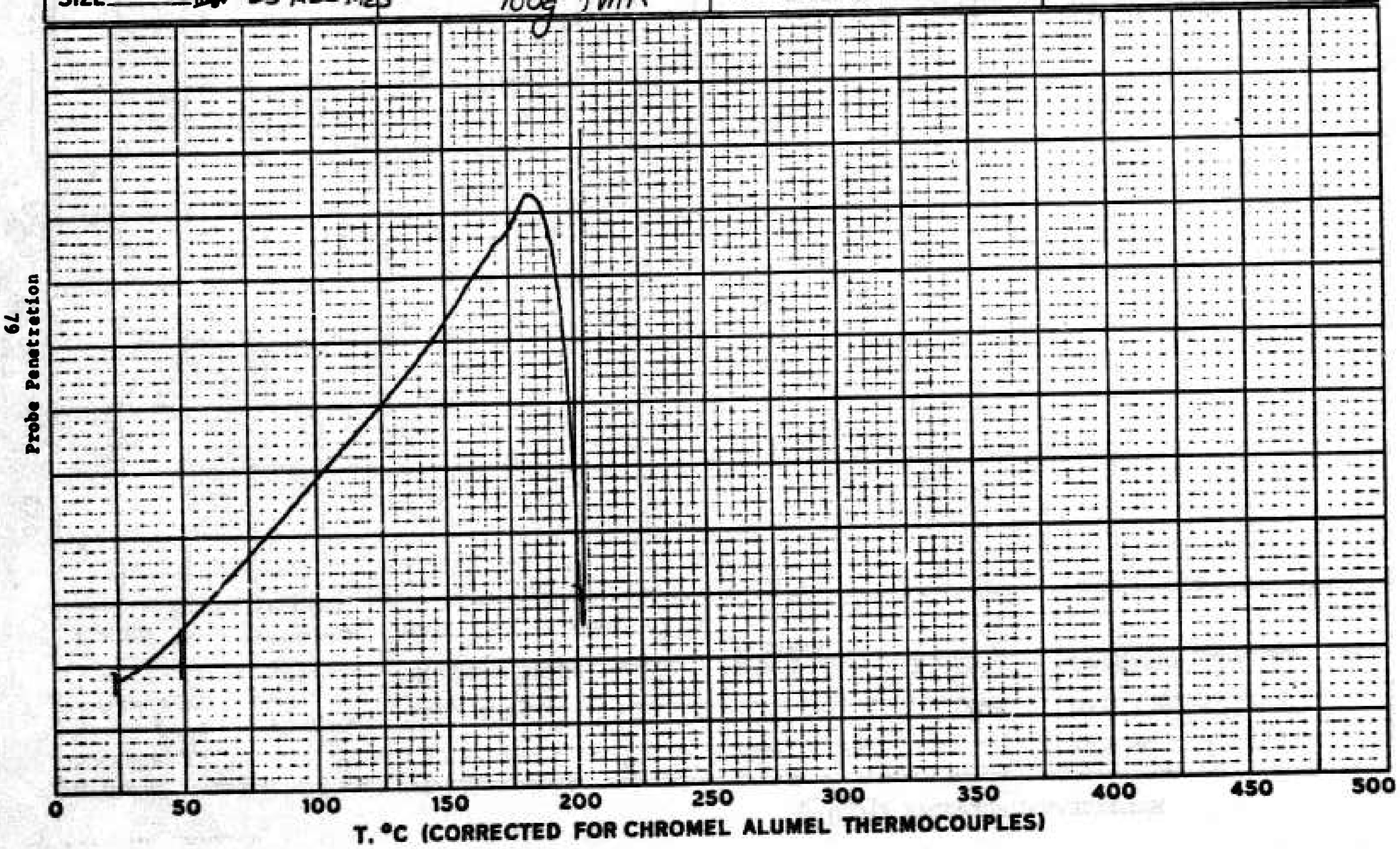
DU PONT INSTRUMENTS

SAMPLE: ICI 300P
24 hr H_2O boil
(in H_2O 24 hrs additional)
Source = DD2256-20A
SIZE ~~mg.~~ .25 x .25 x .25

X-AXIS
TEMP. SCALE 50 °C/inch
SHIFT 0 inch
100g TMA

Y-AXIS
SCALE ~~mg/inch~~ ~~0.5 mil~~ .02/in
(SCALE SETTING X 2)
SUPPRESSION —— mg.

RUN NO. 21 DATE 6/19/74
OPERATOR JHa
HEATING RATE 5 °C/min.
ATM. N_2
TIME CONSTANT —— sec.



PART NO. 900320

DU PONT INSTRUMENTS

SAMPLE: ICI 200P polyethersulfone (dried 250°F, 1hr)

ORIGIN: ICI furnished tensile coupon

SIZE .1 x .2 x .3

REF. 100g TMA

PROGRAM MODE Heat

RATE 5 °C/min. START RT °C

ATM. N_2

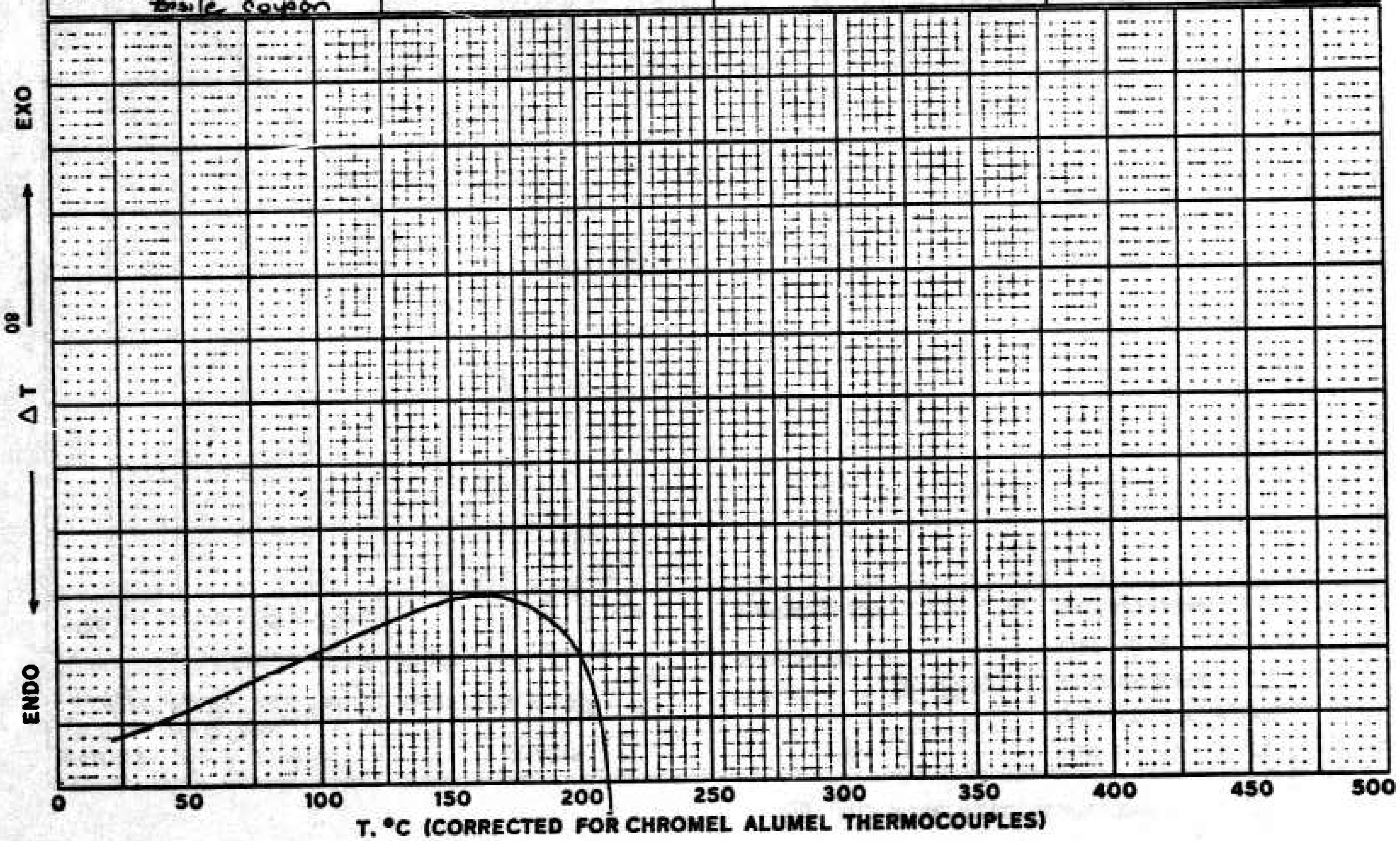
SCALE SETTING

T	~~ΔT~~
50 °C/in.	.05 mg/in.

RUN NO. 9

DATE 6/13/74

OPERATOR JHa



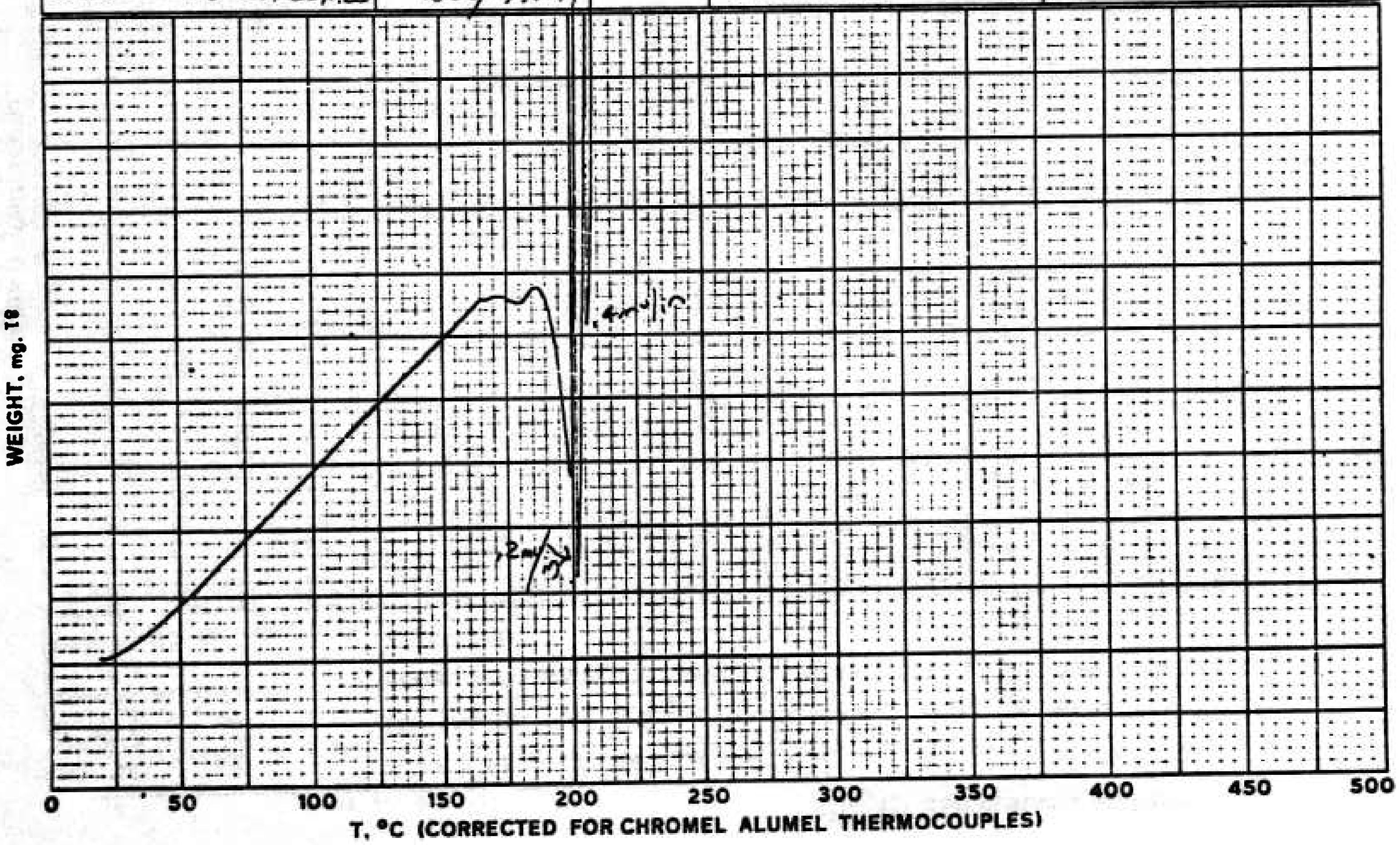
T, °C (CORRECTED FOR CHROMEL ALUMEL THERMOCOUPLES)

SAMPLE: PolyEther Sulfone 200P -
24hr boil, 24hr soak
~~Source~~.
Source - Tensile provided by ICI, remolded by WRD
SIZE______mg. .25x.25x.25

X-AXIS
TEMP. SCALE 50 °C/inch
SHIFT 0 inch
100g TMA

Y-AXIS
SCALE ~~mg/inch~~ .02 mv/in
(SCALE SETTING X 2)
~~SUPPRESSION______mg.~~

RUN NO. 25 DATE 6/30/77
OPERATOR JHa
HEATING RATE 5 °C/min.
ATM. He
~~TIME CONSTANT______sec.~~



DU PONT INSTRUMENTS

PART NO. 941017

SAMPLE: P-1700
Polysulfone
Dried 2 hrs @ 250°F
Stored in dessicator

ORIGIN: U.C. casting

SAMPLE HEIGHT .25"x.25"x.20"

LOADING ON TRAY 100g

PROBE: EXPANSION/PENETRATION

HEATING RATE 5

X-AXIS SCALE 50

Y-AXIS SCALE .02

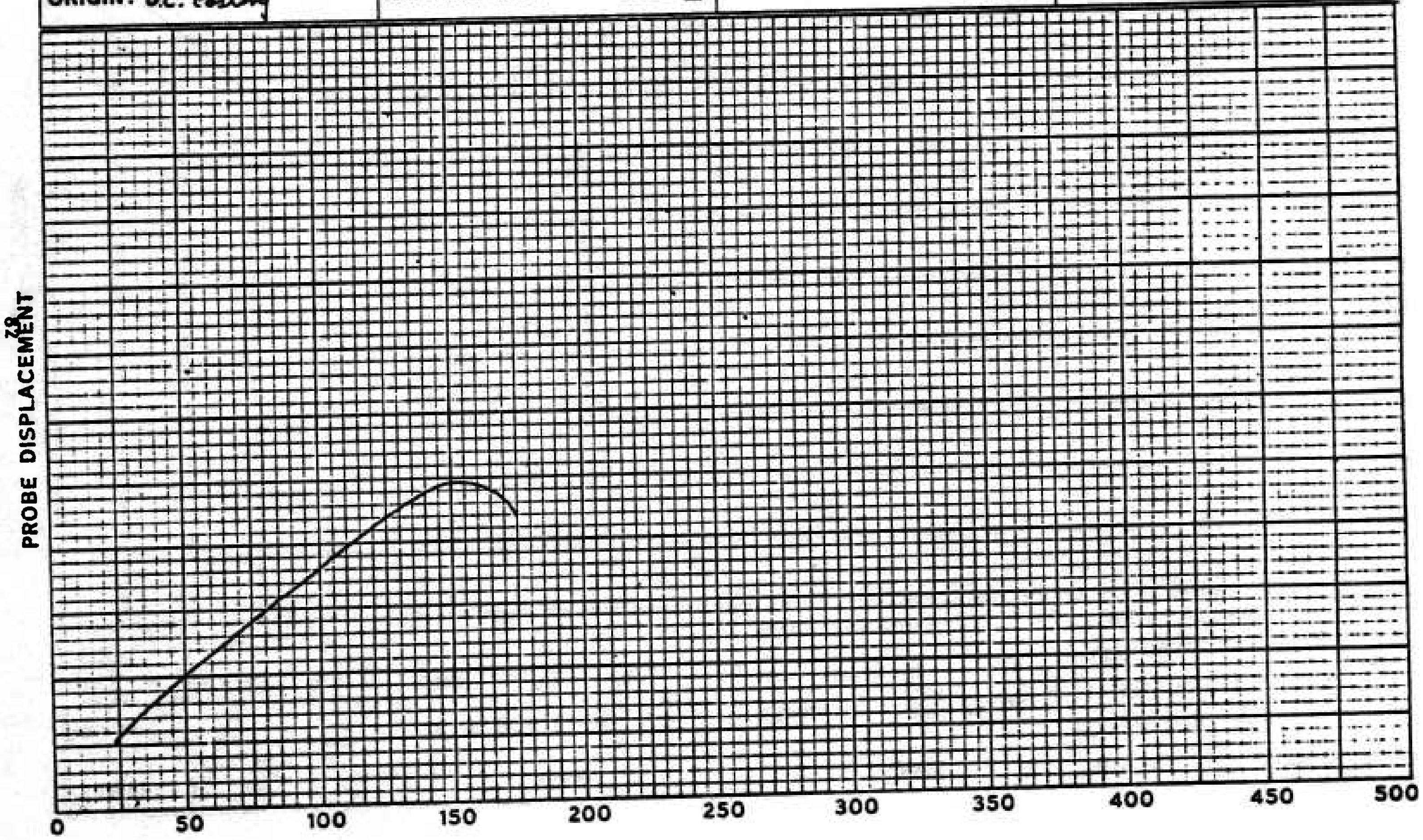
Y-AXIS SENSITIVITY

IN. PROBE DISPL. / IN. OF CHART

RUN NO. 61

DATE 7-30-74

OPERATOR D. Dayton



PART NO. 941017

DUPONT INSTRUMENTS

SAMPLE: P-1700
-Polysulfone
25½ hr H_2O BOIL
24 hr H_2O SOAK
Sof

ORIGIN: U.C. CASTING

SAMPLE HEIGHT .2"X.25"X.25"

LOADING ON TRAY/ 180g

PROBE: EXPANSION/PENETRATION

HEATING RATE 5 °C/MIN

X-AXIS SCALE 50 °C/DIV.

Y-AXIS SCALE .02 mv/in. MV/IN

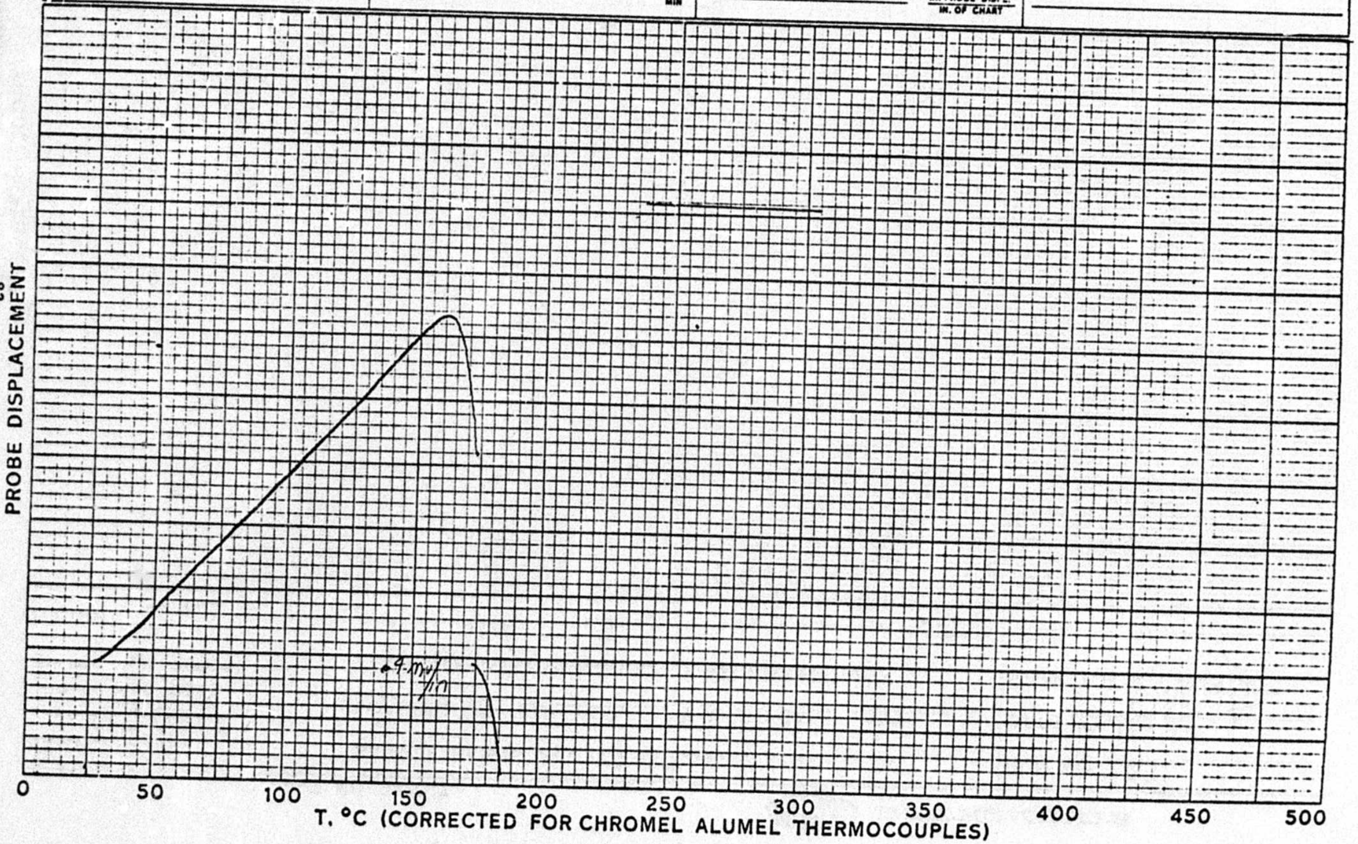
Y-AXIS SENSITIVITY

IN. PROBE DISPL. / IN. OF CHART

RUN NO. 54

DATE July 25, 1974

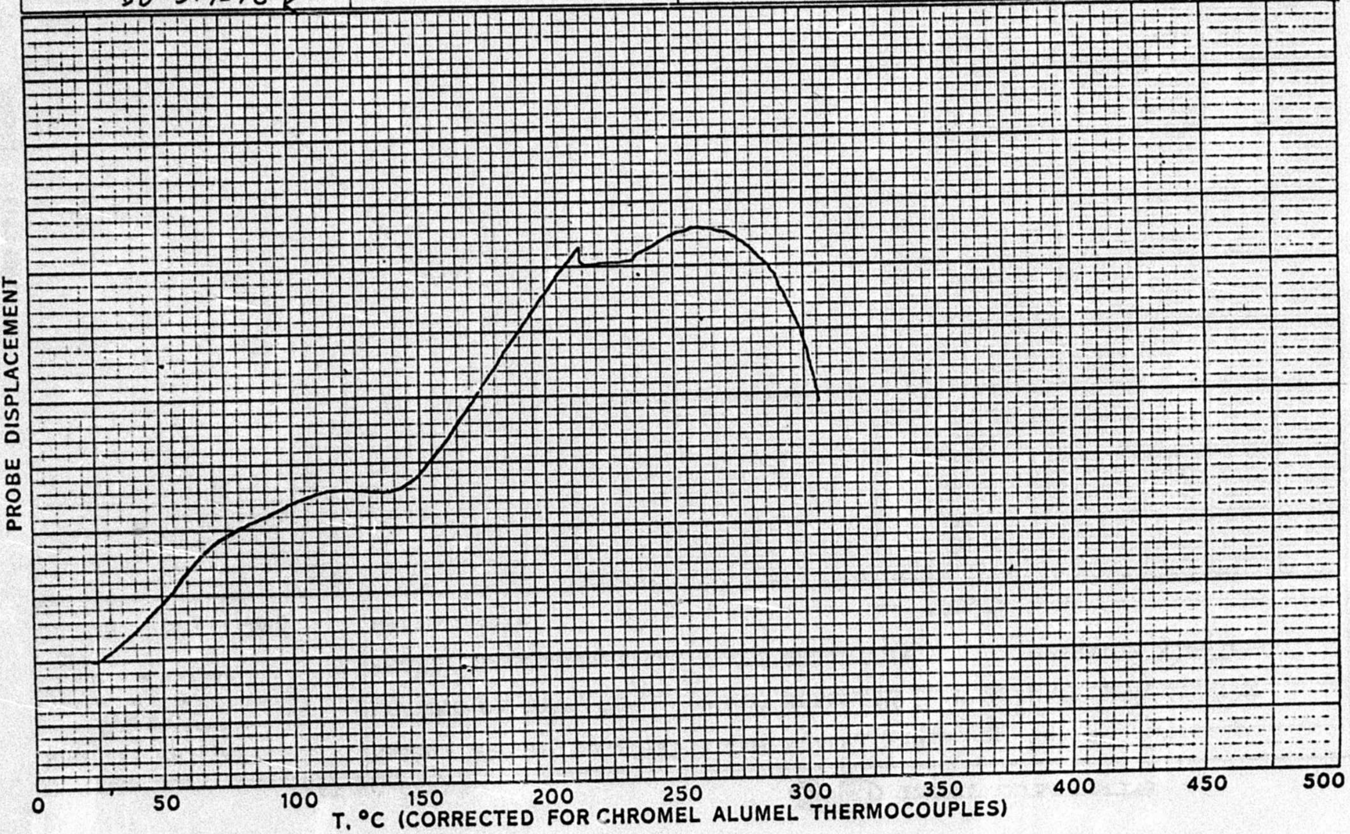
OPERATOR Dayton



PART NO. 941017

DU PONT INSTRUMENTS

SAMPLE: DAP diallyl phthalate 24 hr H_2O boil 26 day H_2O soak	SAMPLE HEIGHT .25" x .25" x .20"	X-AXIS SCALE 50 °C/DIV.	RUN NO. 123
	LOADING ON TRAY 100	Y-AXIS SCALE .04 MV/IN	DATE 8-26-74
	PROBE: EXPANSION/PENETRATION	Y-AXIS SENSITIVITY	OPERATOR D E Dayton
ORIGIN: DD-319-10	HEATING RATE 5 °C/MIN	IN. PROBE DISPL. / IN. OF CHART	



DU PONT INSTRUMENTS

PART NO. 941017

SAMPLE: DAP
Diallyl Phthalate
2 hr @ 250°F dry Stored in humidor

ORIGIN: DD-319-10 $\frac{S}{K}$

SAMPLE HEIGHT .25 x .25 x .25

LOADING ON TRAY 100g

PROBE: EXPANSION/PENETRATION

HEATING RATE 5

X-AXIS SCALE 50

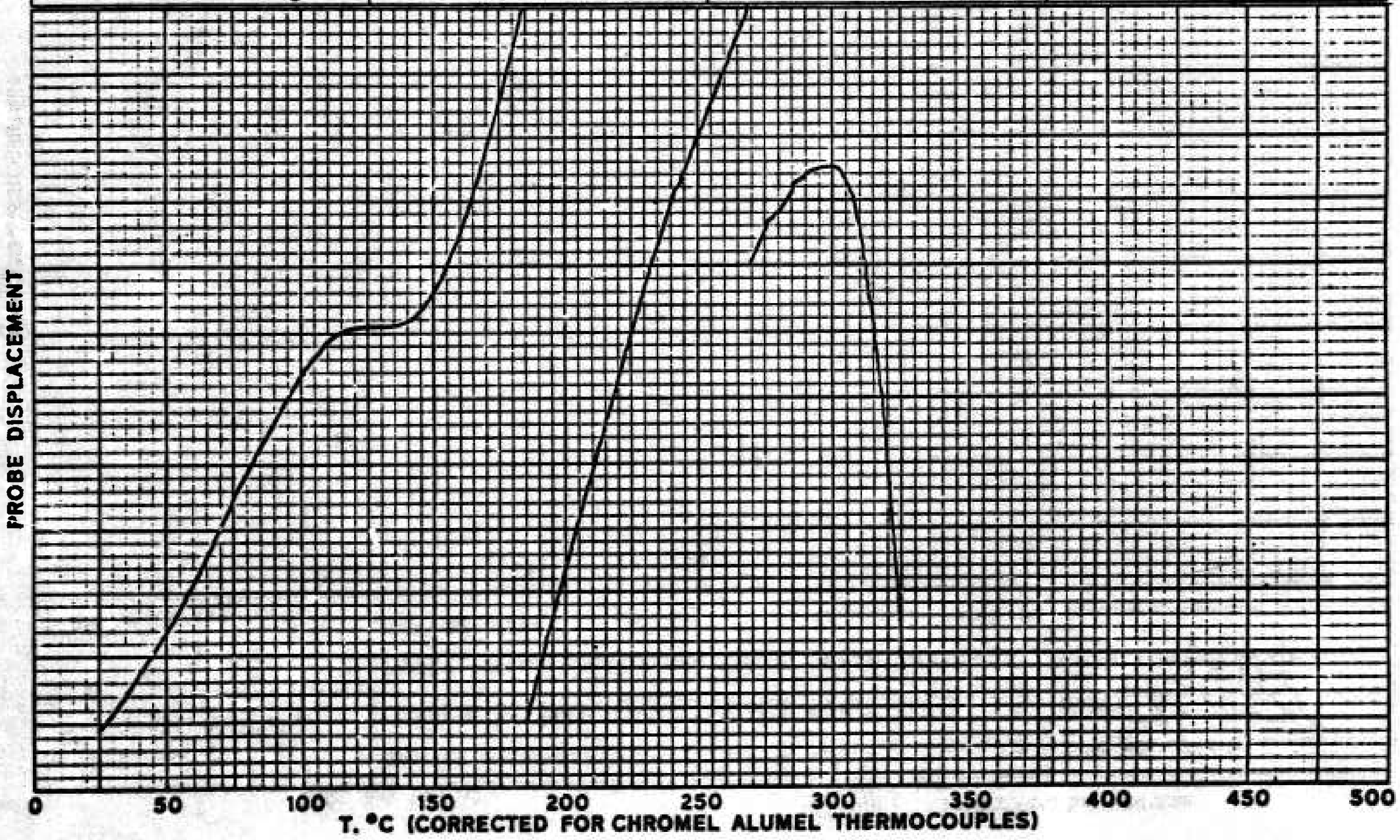
Y-AXIS SCALE .02

Y-AXIS SENSITIVITY

RUN NO. 74

DATE 8-5-74

OPERATOR D. Dayton



PART NO. 941017

DUPONT INSTRUMENTS

SAMPLE: DAIP
Diallyl Isophthalate
2hr 350°F dry desiccator storage

ORIGIN: DD-319-10 C/14

SAMPLE HEIGHT .25"x.25"x.25"

LOADING ON TRAY 10g

PROBE: EXPANSION/PENETRATION

HEATING RATE 5 °C/min

X-AXIS SCALE 50 °C/in

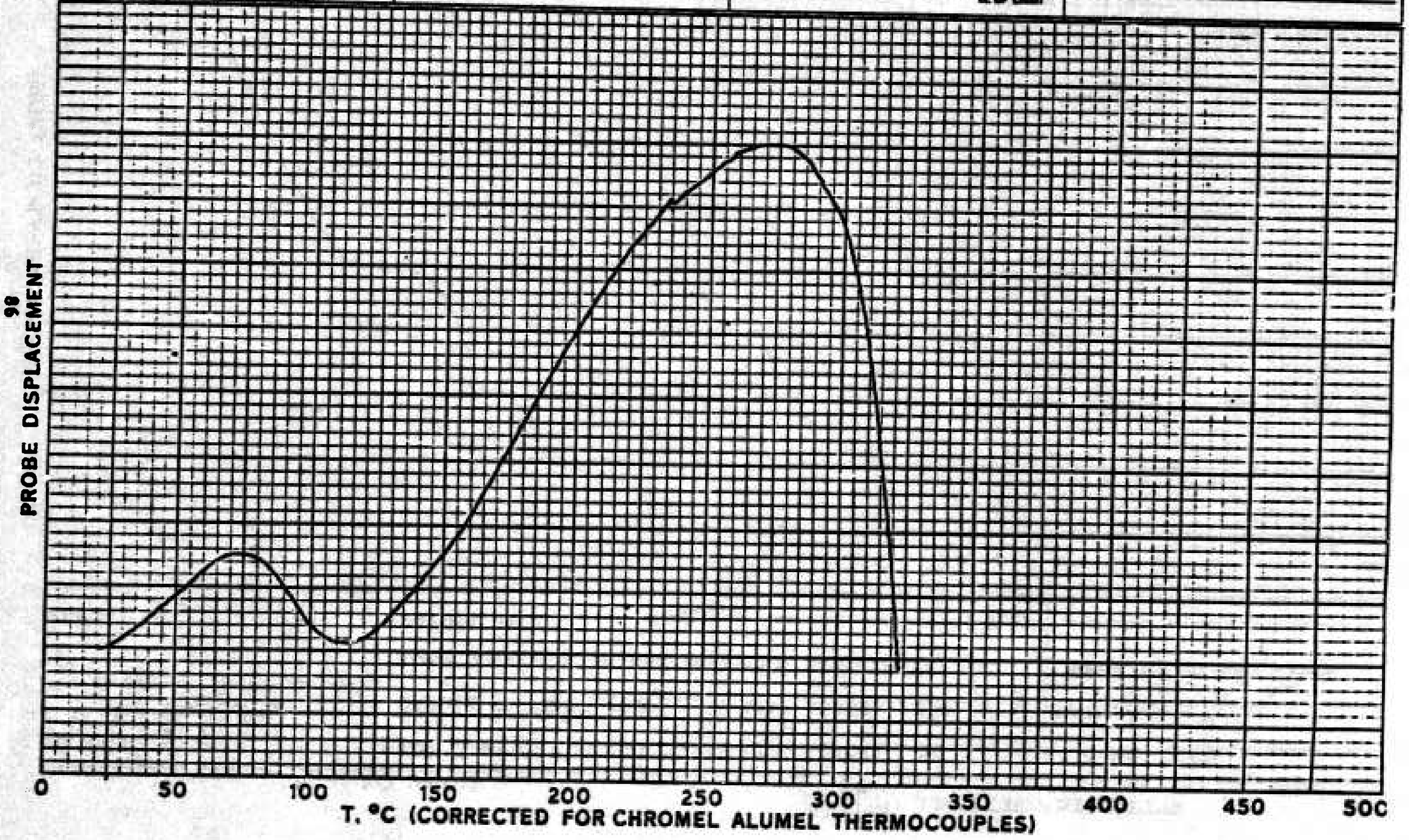
Y-AXIS SCALE .04

Y-AXIS SENSITIVITY

RUN NO. 126

DATE 8-27-74

OPERATOR



DU PONT INSTRUMENTS

PART NO. 941017

SAMPLE: DAIP
diallyl isophthalate
24 hr H2O boil
26 days H2O soak

ORIGIN: DD-319-10 6/4

SAMPLE HEIGHT .25" X .25" X .20"

LOADING ON TRAY 100g

PROBE: EXPANSION/PENETRATION

HEATING RATE 5

X-AXIS SCALE 50

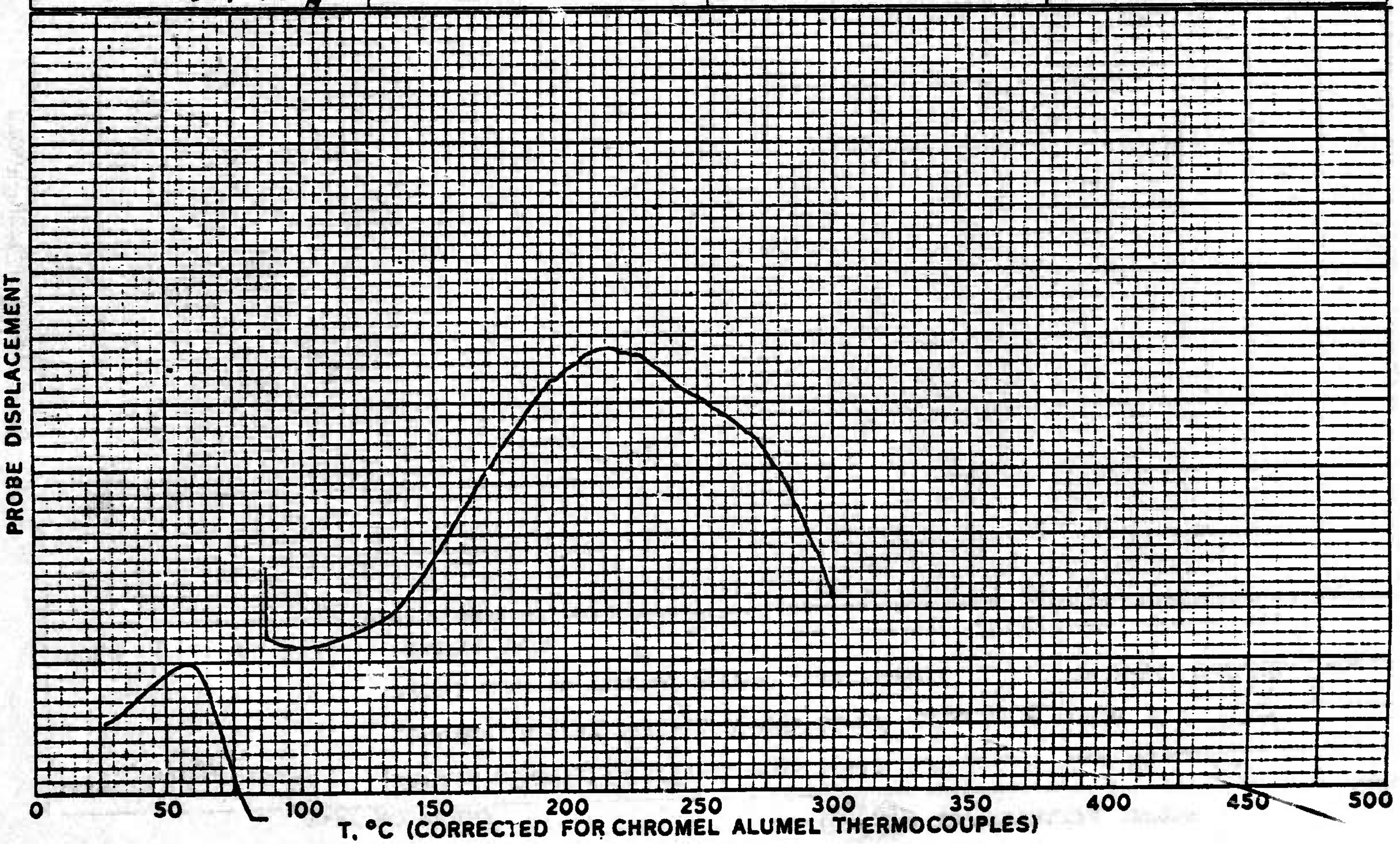
Y-AXIS SCALE .04

Y-AXIS SENSITIVITY

RUN NO. 122

DATE 8-26-74

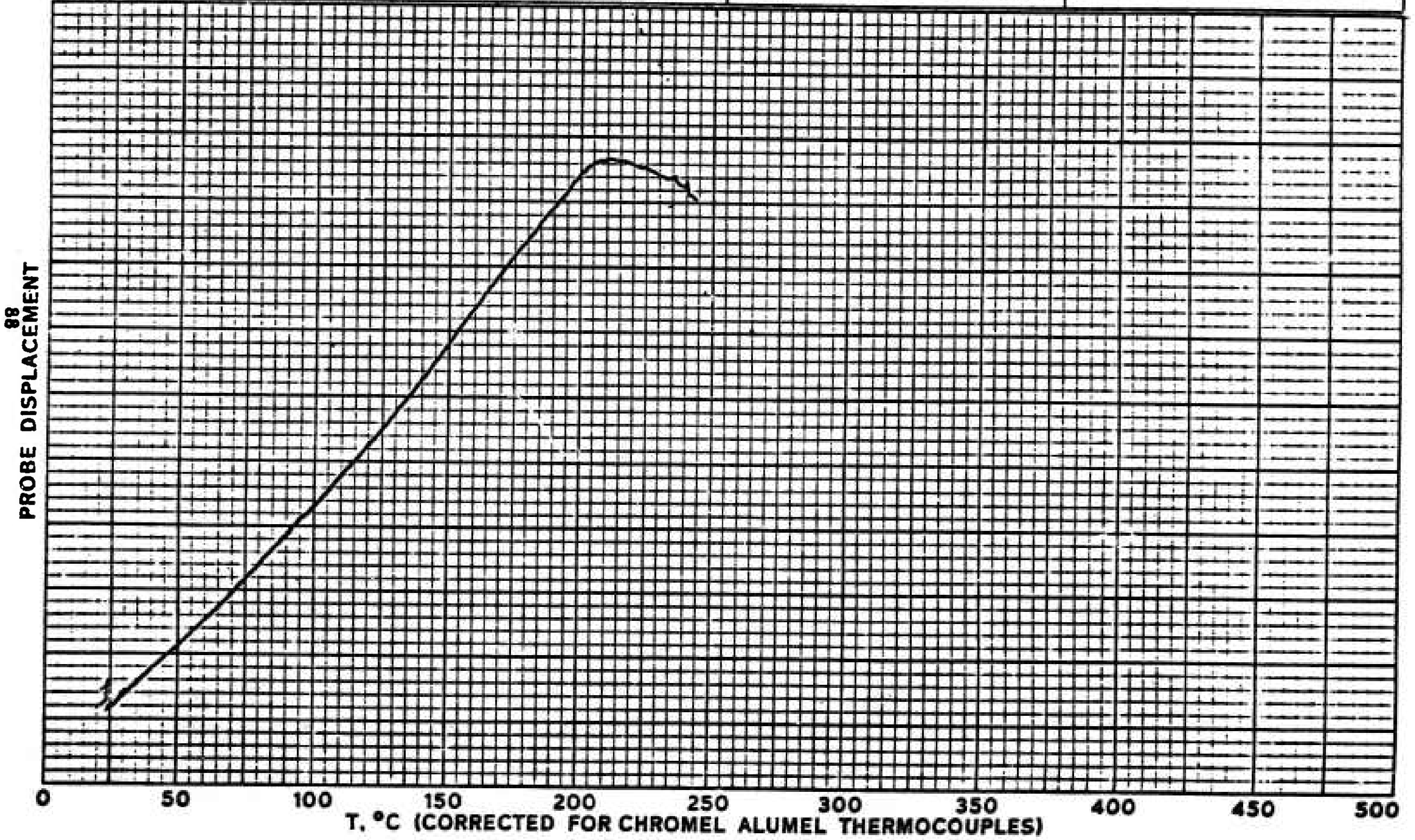
OPERATOR DeDayton



DU PONT INSTRUMENTS

PART NO. 941017

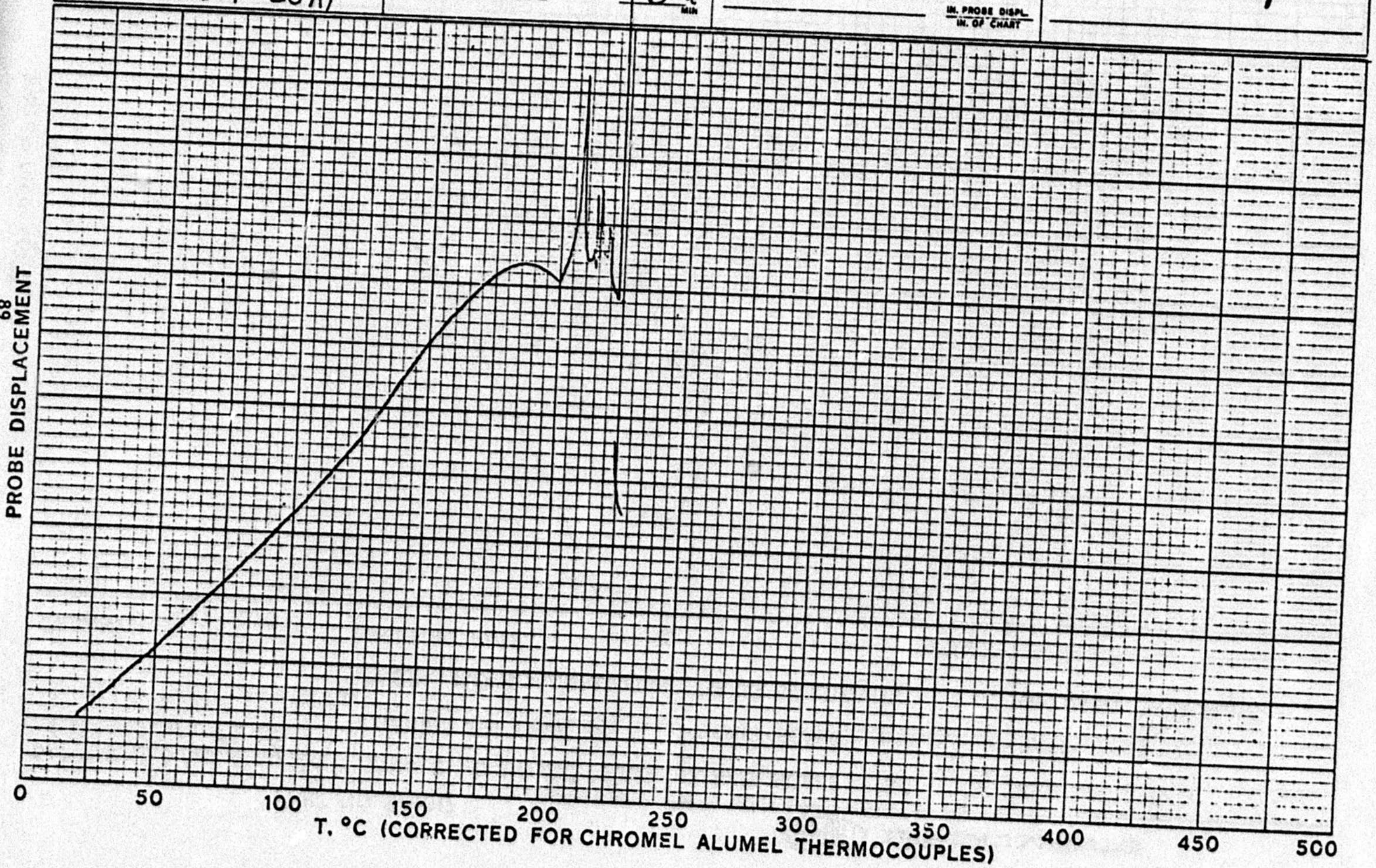
SAMPLE: Polyphenol DP 25 1,10	SAMPLE HEIGHT .25"x.25"x.25"	X-AXIS SCALE 50	RUN NO. 85
	LOADING ON TRAY 100 g	Y-AXIS SCALE .02	DATE 8-13-74
	PROBE: EXPANSION/PENETRATION	Y-AXIS SENSITIVITY	OPERATOR D Clayton
ORIGIN: DD-319-20A1	HEATING RATE 5		



PART NO. 941017

DUPONT INSTRUMENTS

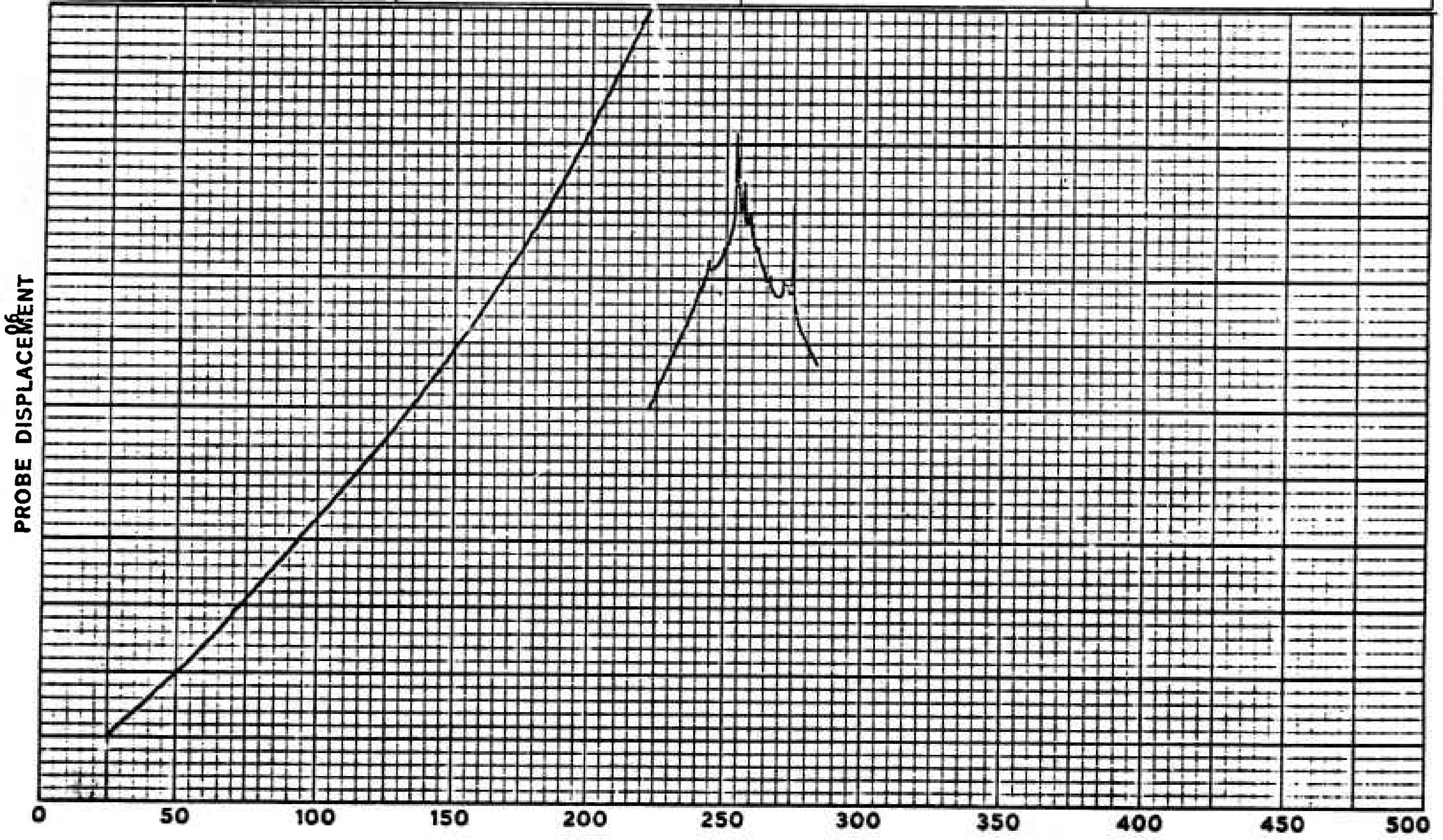
SAMPLE: Polyphenol DP25-10 24 hr H2O Boil 24 hr H2O Soak	SAMPLE HEIGHT .25"X.25"X.25"	X-AXIS SCALE 50 °C/DIV.	RUN NO. 91
	LOADING ON TRAY 100g	Y-AXIS SCALE .02 MV/IN	DATE 8-14-74
ORIGIN: DD-319-20A)	PROBE: EXPANSION/PENETRATION	Y-AXIS SENSITIVITY ____ IN. PROBE DISPL./IN. OF CHART	OPERATOR D.E. Dayton
	HEATING RATE 5 °C/MIN		



DUPONT INSTRUMENTS

PART NO. 941017

SAMPLE: Diphenyl Oxide (DP4-94N) R6481 Dried 2 hr @ 230°F stored in des.	SAMPLE HEIGHT .25"x.25"x.25"	X-AXIS SCALE 50 °C/IN.	RUN NO. 86
	LOADING ON TRAY 100g	Y-AXIS SCALE .02	DATE 9-13-74
	PROBE: EXPANSION/PENETRATION	Y-AXIS SENSITIVITY	OPERATOR D. Dayton
ORIGIN: DD-319-2082	HEATING RATE 5 °C/MIN	IN. PROBE DISPL. / IN. OF CHART	



PART NO. 941017

DU PONT INSTRUMENTS

SAMPLE: DIPHENOL OXIDE
£648' (DP4-94N)
24 hr H2O Boil
24 hr H20 Soak

ORIGIN: DD-319-2082

SAMPLE HEIGHT .25"X.25"X.25"

LOADING ON TRAY 100g

PROBE: EXPANSION/PENETRATION

HEATING RATE 5 °C/MIN

X-AXIS SCALE 50 °C/IN.

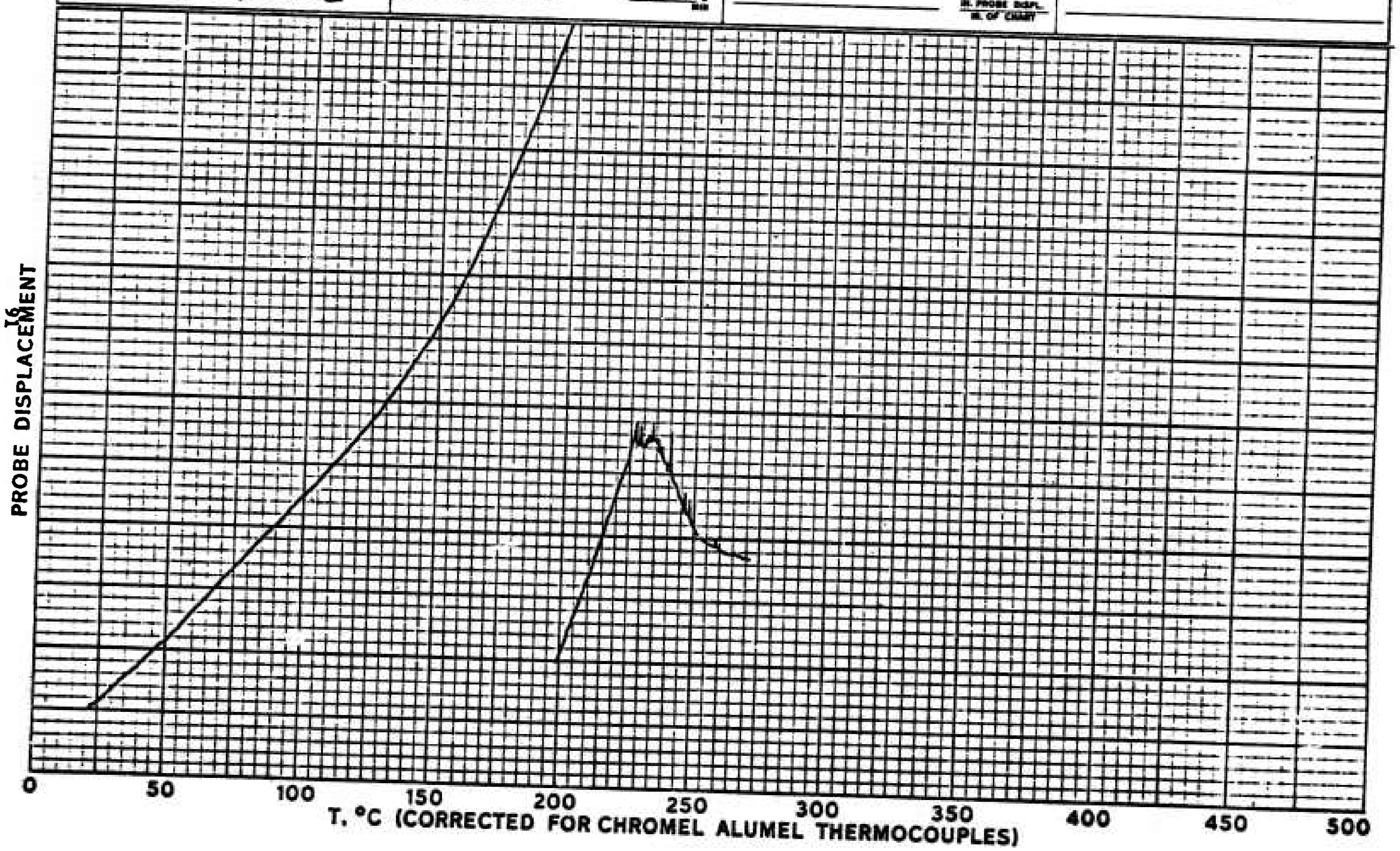
Y-AXIS SCALE .02 MV/IN.

Y-AXIS SENSITIVITY ______ IN. PROBE DISPL./IN. OF CHART

RUN NO. 92

DATE 8-14-74

OPERATOR DDDayton



DUPONT INSTRUMENTS

PART NO. 941017

SAMPLE: Polyester HETRON 197P
VAC DRIED

ORIGIN: DD-319-19B

SAMPLE HEIGHT .25"X.25"X.25

LOADING ON TRAY 100g

PROBE: EXPANSION/PENETRATION

HEATING RATE 5

X-AXIS SCALE 50

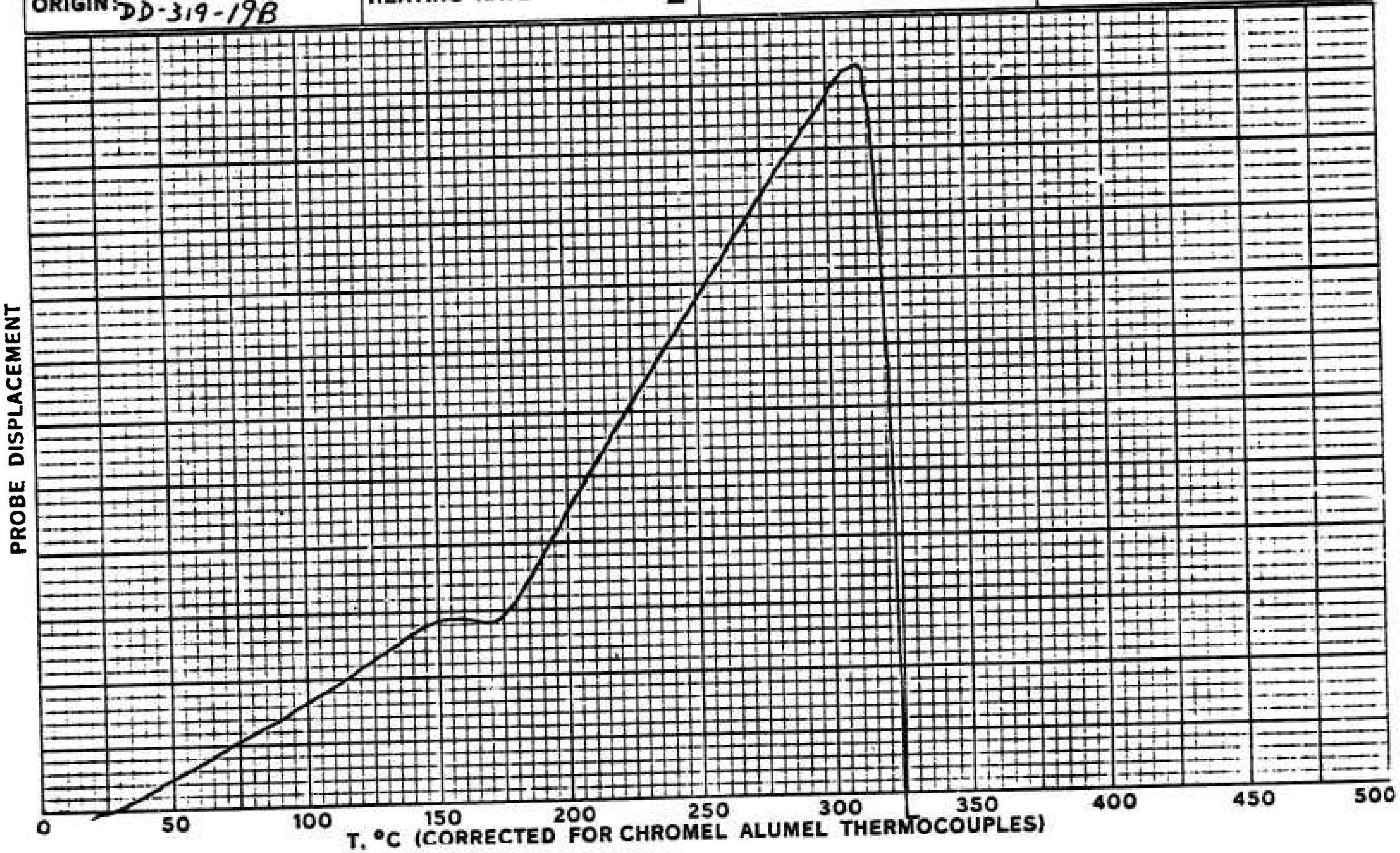
Y-AXIS SCALE .04

Y-AXIS SENSITIVITY

RUN NO. 119

DATE 8-23-74

OPERATOR D L Dayton



DU PONT INSTRUMENTS

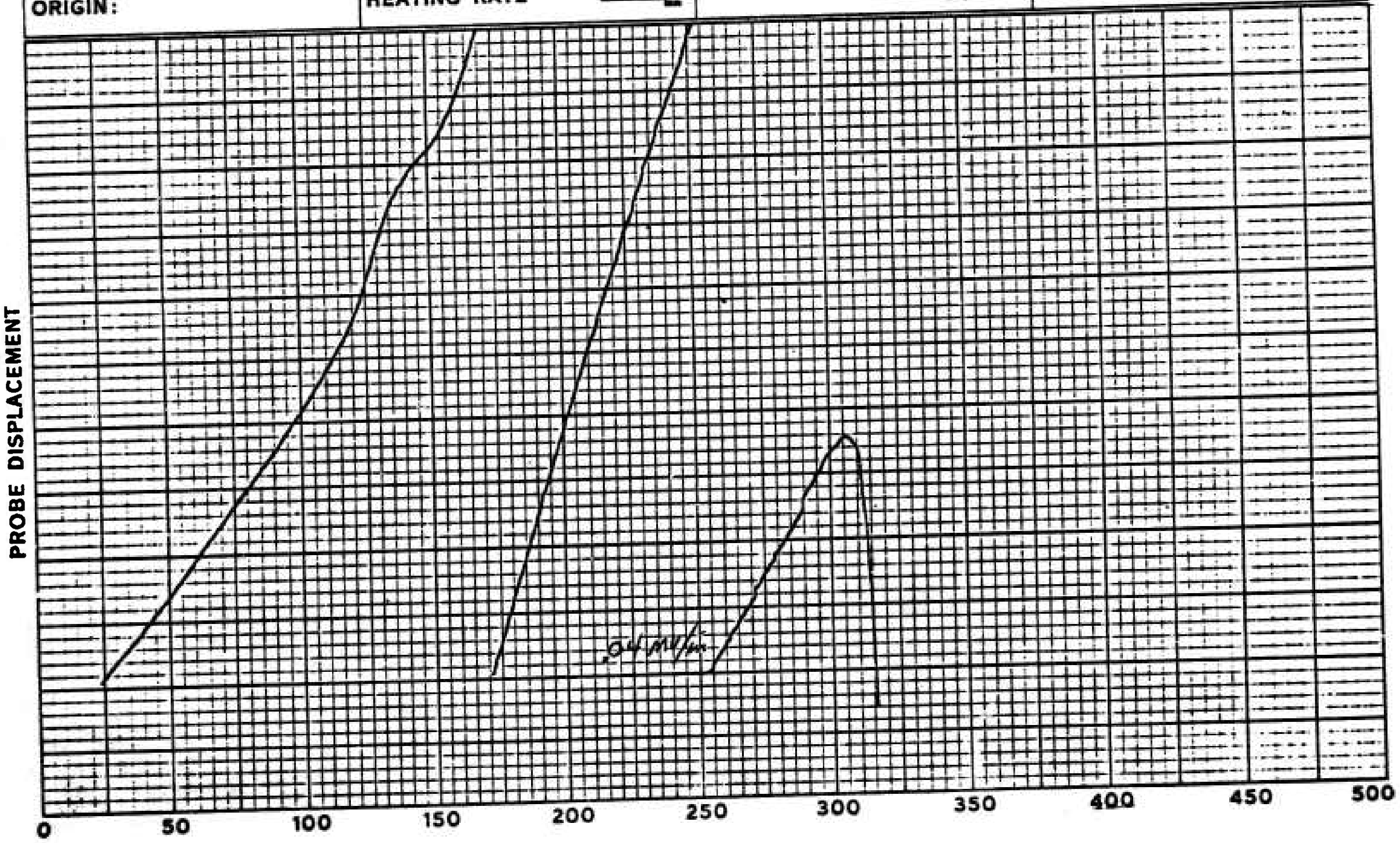
PART NO. 941017

SAMPLE: HETRON 197P
POLYESTER
24 hr H₂O Boil
24 hr H₂O Soak
ORIGIN:

SAMPLE HEIGHT .25"x.25"x.25"
LOADING ON TRAY 100g
PROBE: EXPANSION/PENETRATION
HEATING RATE 5 °C/min

X-AXIS SCALE 50 °C/in.
Y-AXIS SCALE .02 mV/in.
Y-AXIS SENSITIVITY
in. PROBE DISPL./in. OF CHART

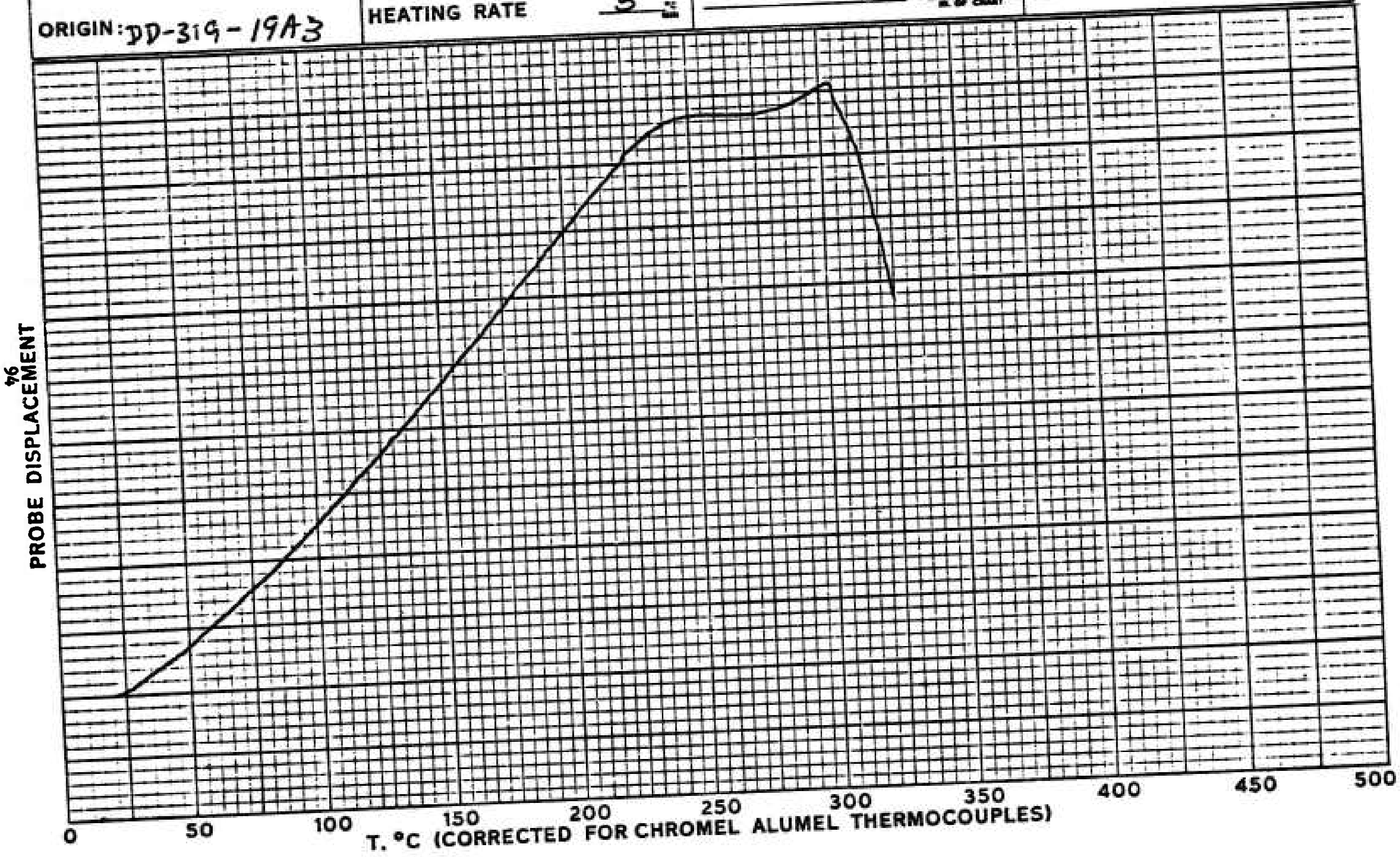
RUN NO. 81
DATE 8-8-74
OPERATOR D.E. Dayton



DU PONT INSTRUMENTS

PART NO. 941017

SAMPLE: LAMINAC 4232 POLYESTER VAC. DRIED @ 60°C STORED IN DESICCATOR	SAMPLE HEIGHT .25"x.25"x.20"	X-AXIS SCALE 50	RUN NO. 90
	LOADING ON TRAY 100g	Y-AXIS SCALE .04	DATE 8-14-74
	PROBE: EXPANSION/PENETRATION	Y-AXIS SENSITIVITY	OPERATOR D.E. Dayton
ORIGIN: DD-319-19A3	HEATING RATE 5		



DU PONT INSTRUMENTS

PART NO. 941017

SAMPLE: LAMINAC 4232 POLYESTER
24 hr H2O Boil
48 hr H2O Soak

ORIGIN: DD-319-19A3

SAMPLE HEIGHT .25" x .25" x .20"

LOADING ON TRAY 100g

PROBE: EXPANSION/PENETRATION

HEATING RATE 5 °C/min

X-AXIS SCALE 50 °C/in.

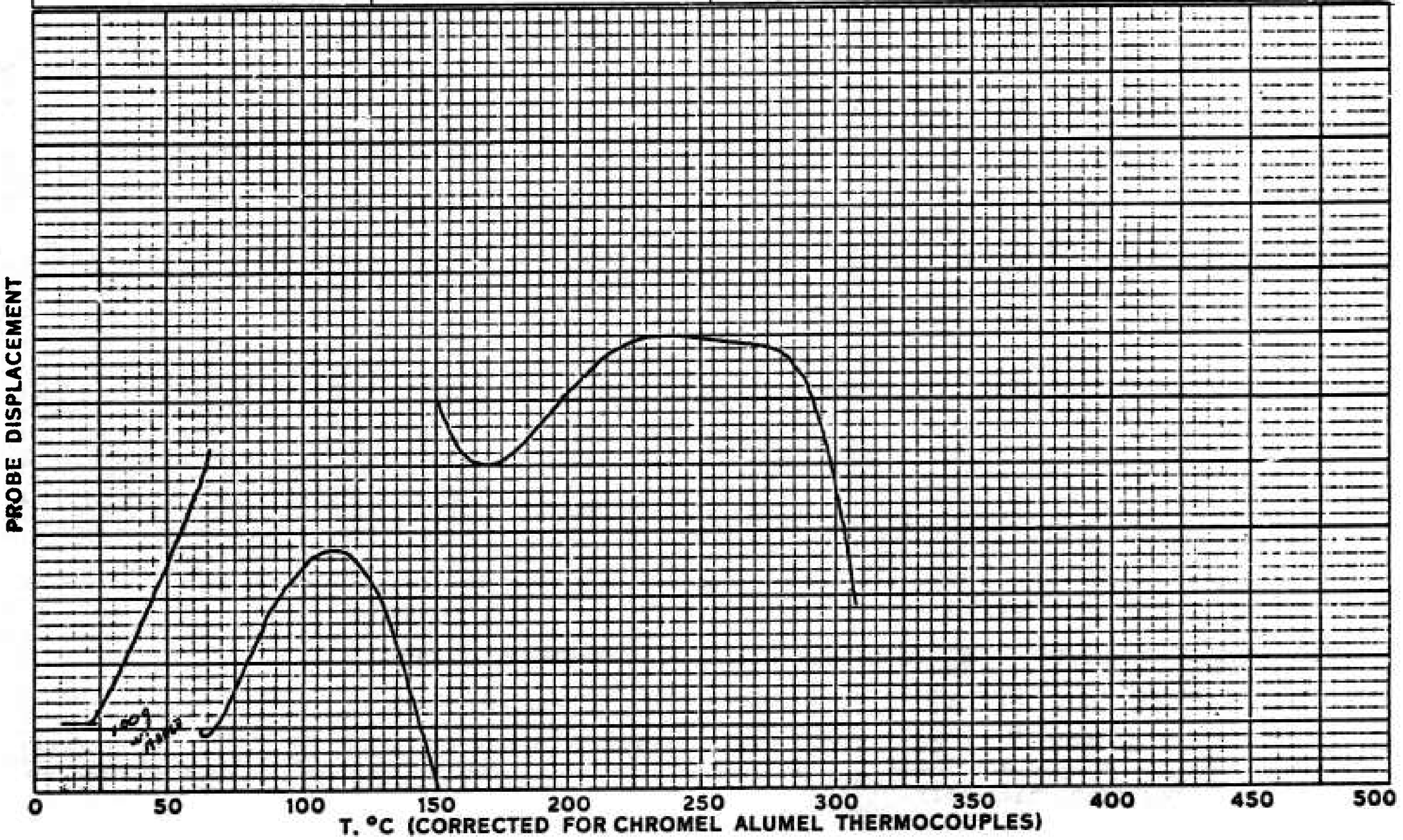
Y-AXIS SCALE .02 mV/in.

Y-AXIS SENSITIVITY ______ in. PROBE DISPL./in. OF CHART

RUN NO. 97

DATE 8-16-74

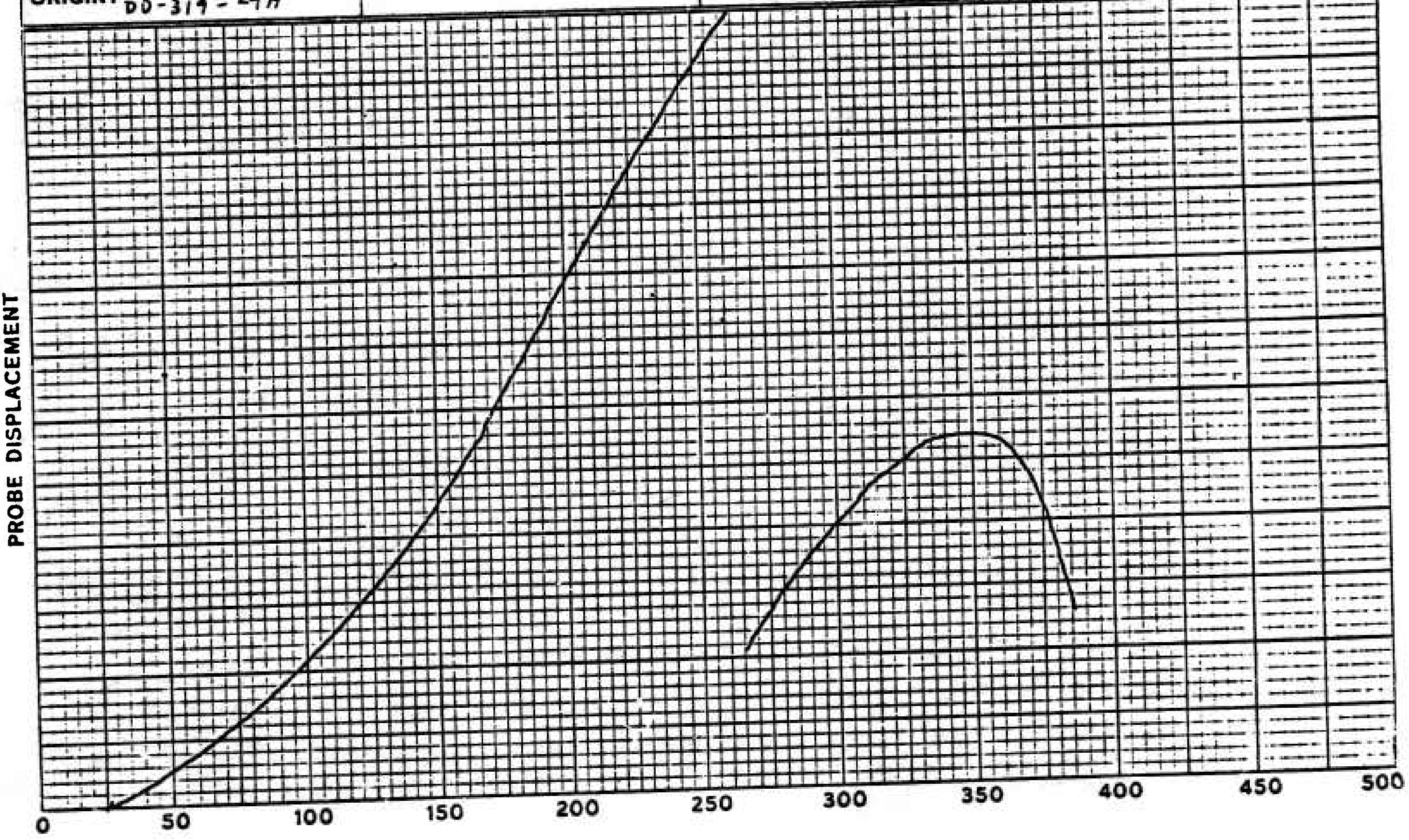
OPERATOR D. Dayton



DUPONT INSTRUMENTS

PART NO. 941017

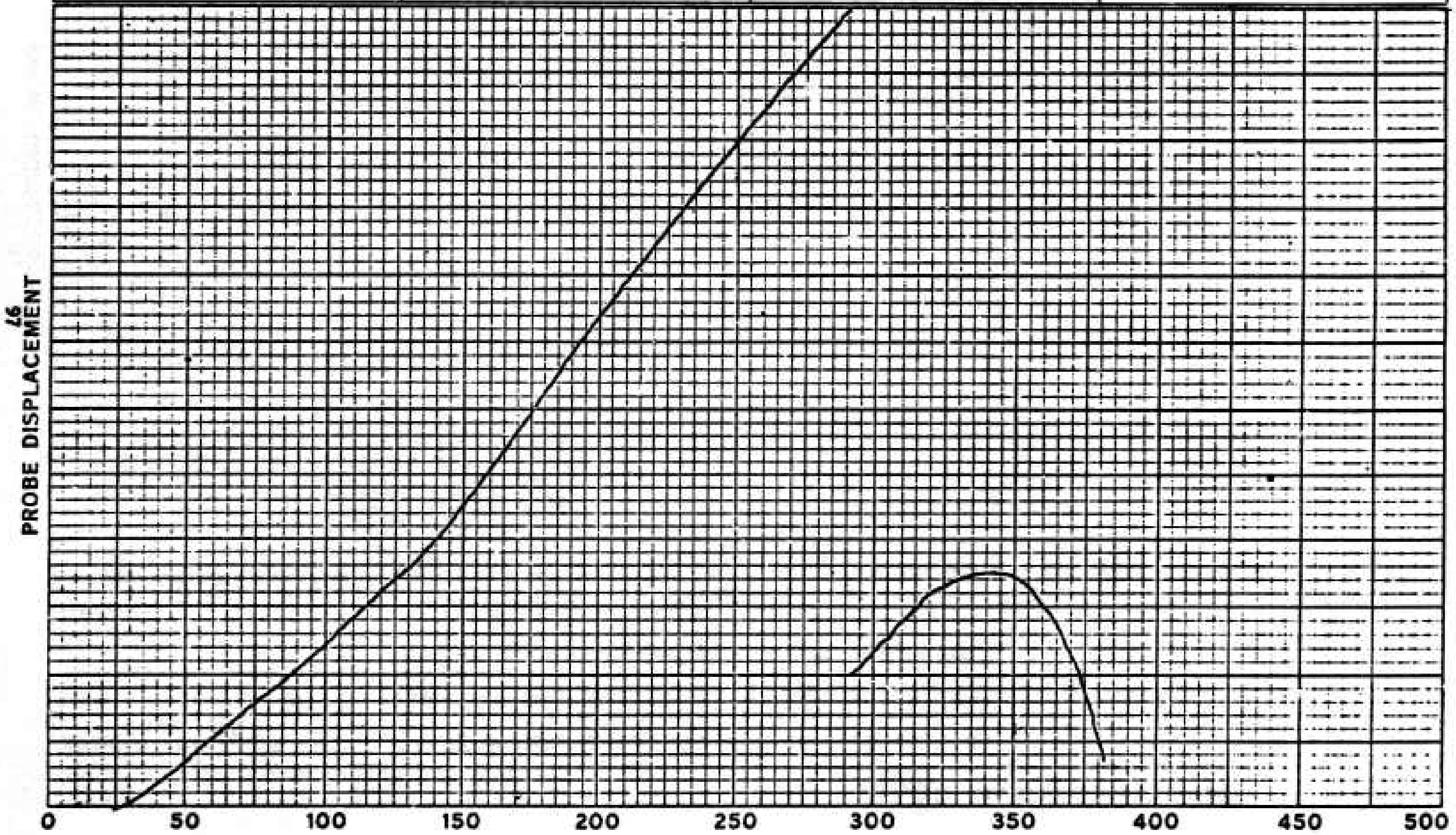
SAMPLE: Koppers Polyester Y-7000-15	SAMPLE HEIGHT .25" x .25" x .25"	X-AXIS SCALE 50	RUN NO. 117
	LOADING ON TRAY 100g	Y-AXIS SCALE .04	DATE 8-23-74
	PROBE: EXPANSION/PENETRATION	Y-AXIS SENSITIVITY 5	OPERATOR D.E. Dayton
ORIGIN: DD-319-24A	HEATING RATE 5		



PART NO. 941017

INSTRUMENTS

SAMPLE: Koppers Polyester V-7000-15 24 hr H2O Boil 24 hr H2O Soak	**SAMPLE HEIGHT** .25"X.25"X.25"	**X-AXIS SCALE** 50	**RUN NO.** 711
	LOADING ON TRAY 100	**Y-AXIS SCALE** .04	**DATE** 8-21-74
	PROBE: EXPANSION/PENETRATION	**Y-AXIS SENSITIVITY**	**OPERATOR** DEDayton
ORIGIN: DD-319-24A	**HEATING RATE** 5	IN. PROBE DISPL. / IN. OF CHART	



DUPONT INSTRUMENTS

PART NO. 941017

SAMPLE: POLYESTER
V-7001-10 Koppers

ORIGIN: DD-319-23 B

SAMPLE HEIGHT .05"x.25"x.25"

LOADING ON TRAY 100g

PROBE: EXPANSION/PENETRATION

HEATING RATE 5 °C/min

X-AXIS SCALE 50 °C/in

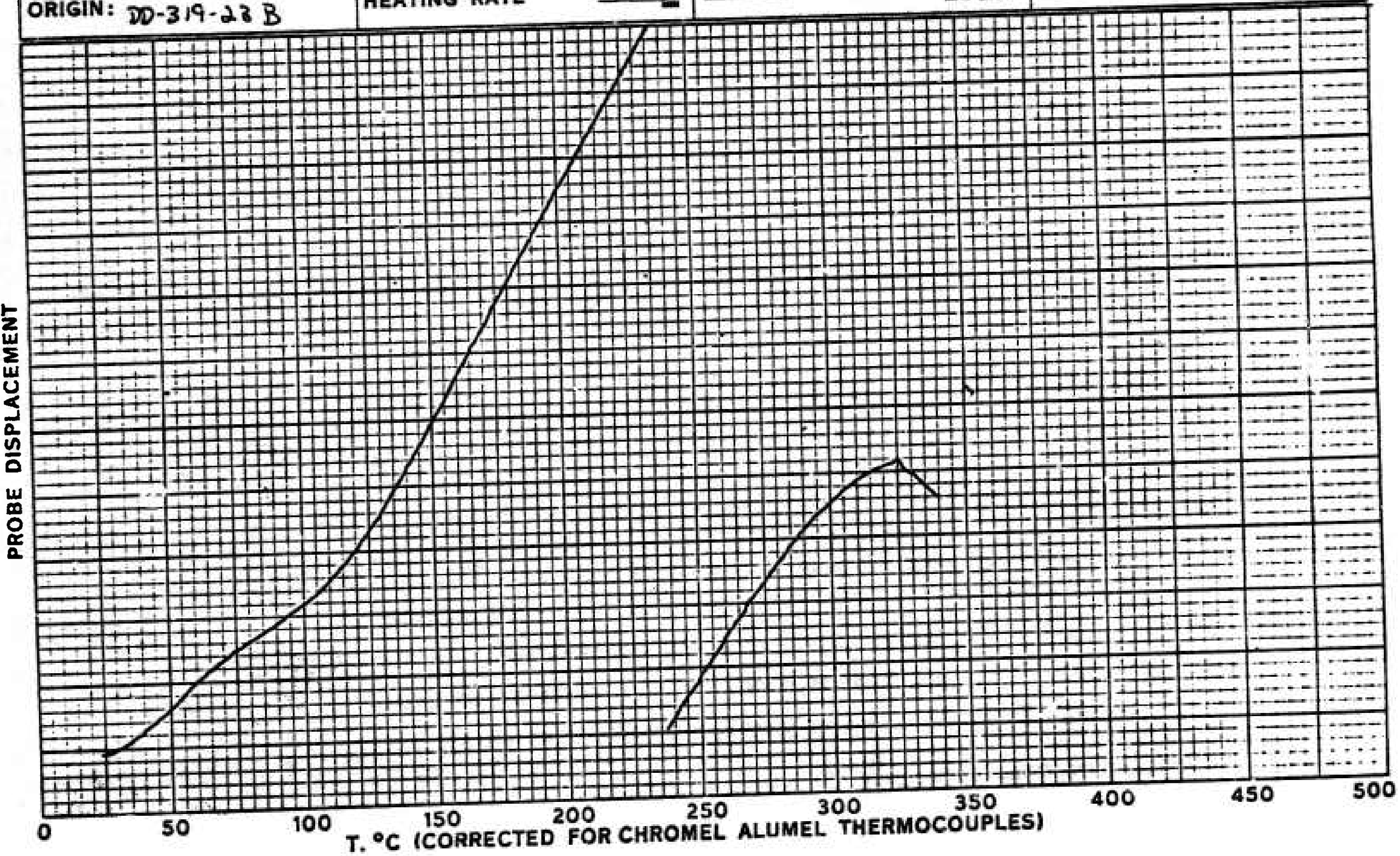
Y-AXIS SCALE .04

Y-AXIS SENSITIVITY

RUN NO. 109

DATE

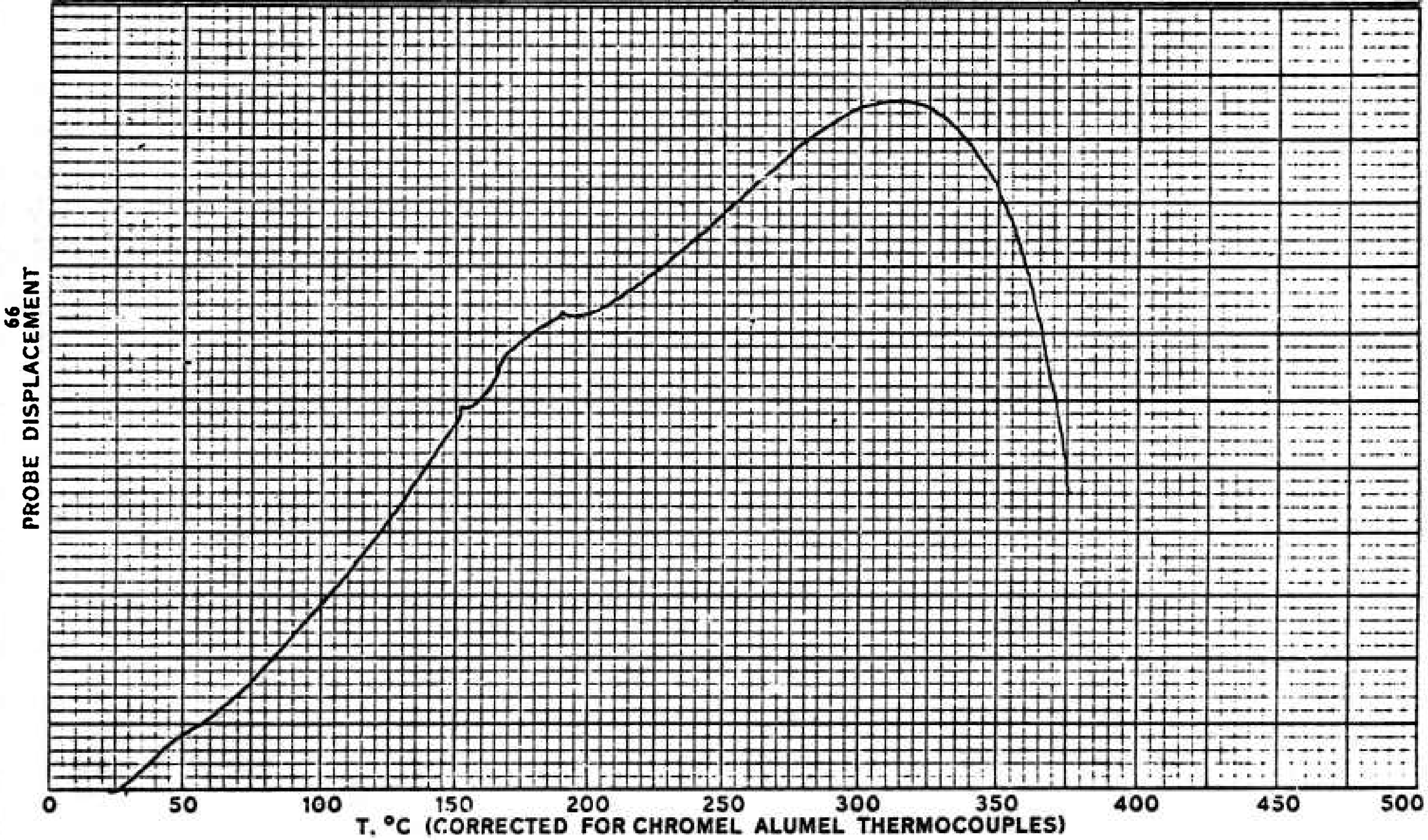
OPERATOR D C Dayton



DUPONT INSTRUMENTS

PART NO. 941017

SAMPLE: Koppers Polyester Y-7001-10 24 hr H2O Boil 24 hr H2O Cond	SAMPLE HEIGHT .25"x.25"x.25"	X-AXIS SCALE 50 °C/in.	RUN NO. 112
	LOADING ON TRAY 100	Y-AXIS SCALE .04 mV/in.	DATE 8-21-74
	PROBE: EXPANSION/PENETRATION	Y-AXIS SENSITIVITY	OPERATOR DWayton
ORIGIN: DD-319-24B	HEATING RATE 5 °C/min	in. probe displ. / in. of chart	



DUPONT INSTRUMENTS

PART NO. 941017

SAMPLE: Hytron 197 Polyester

ORIGIN: DD-319-28A

SAMPLE HEIGHT .25x.25"x.24

LOADING ON TRAY 100g

PROBE: EXPANSION/PENETRATION

HEATING RATE 5

X-AXIS SCALE 50

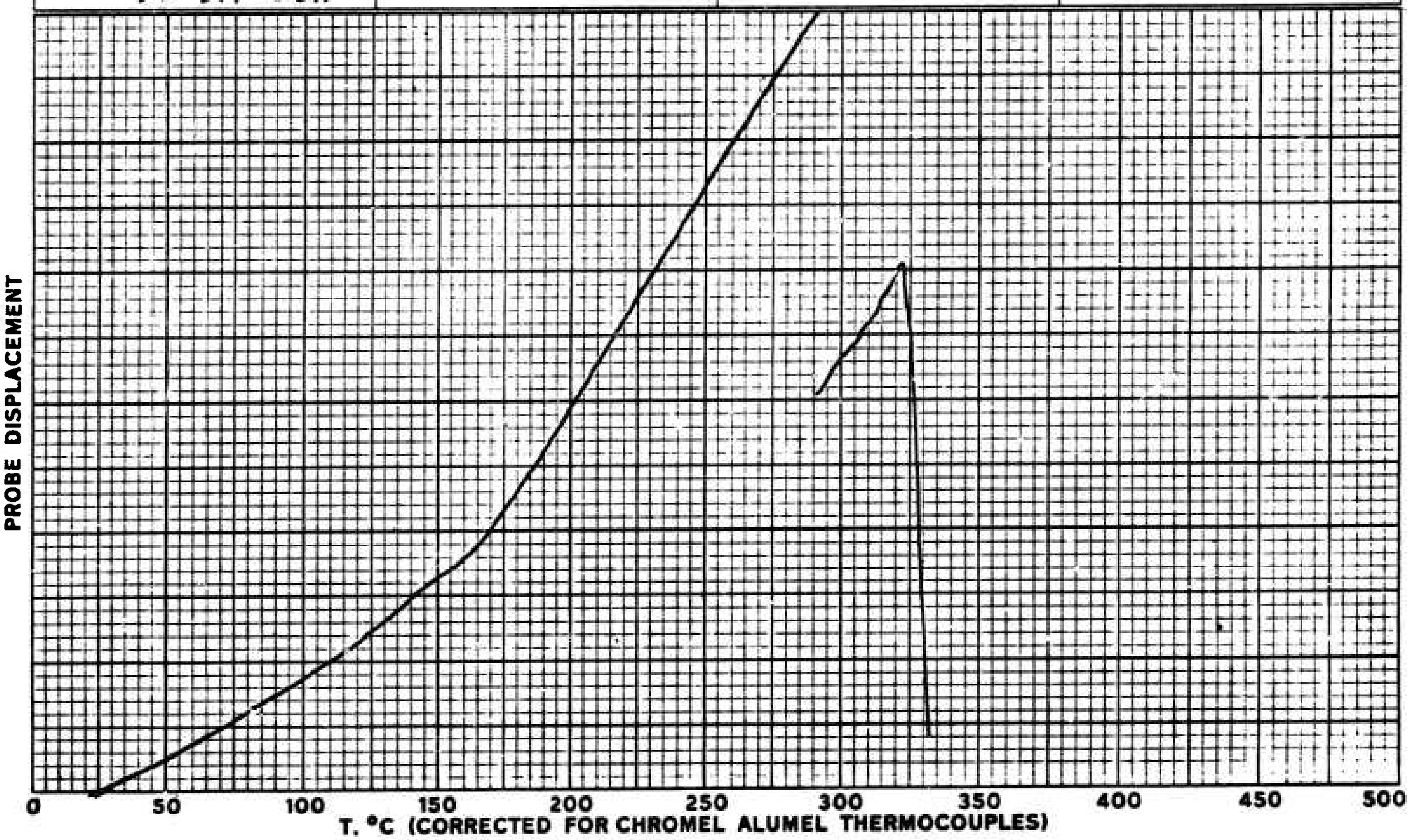
Y-AXIS SCALE .04

Y-AXIS SENSITIVITY

RUN NO. 135

DATE 9-12-74

OPERATOR DE Dayton



DU PONT INSTRUMENTS

PART NO. 941017

SAMPLE: METRON 197
Polyester
24 hr H2O Boil
24 hr H2O Soak

ORIGIN: PD-319-28A

SAMPLE HEIGHT .25 x .25 x .25

LOADING ON TRAY 100

PROBE: EXPANSION/PENETRATION

HEATING RATE 5 °C/min

X-AXIS SCALE 50 °C/in

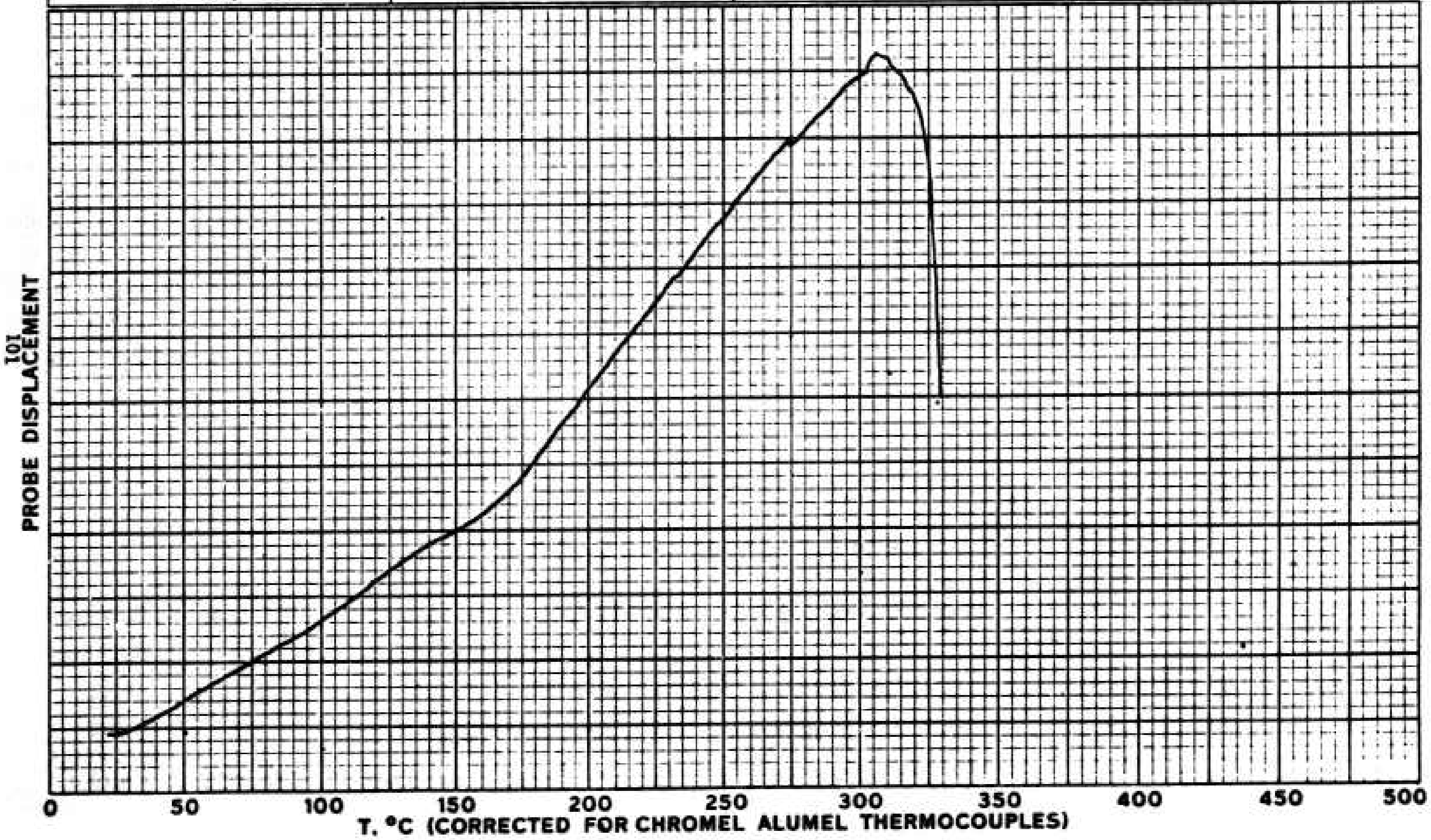
Y-AXIS SCALE .04 in/in

Y-AXIS SENSITIVITY ______ in. probe displ. / in. of chart

RUN NO. 141

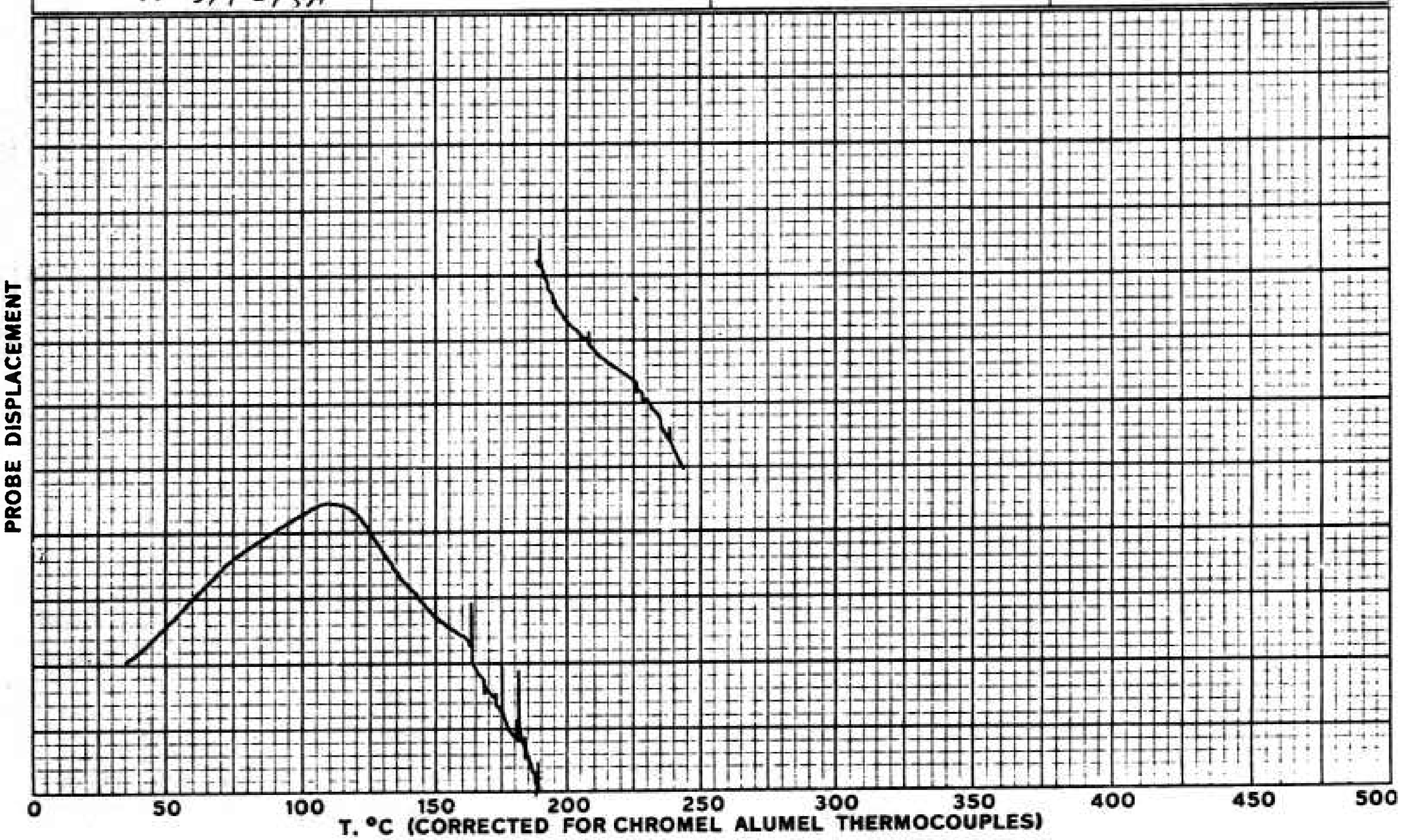
DATE 9-18-74

OPERATOR DHDayton

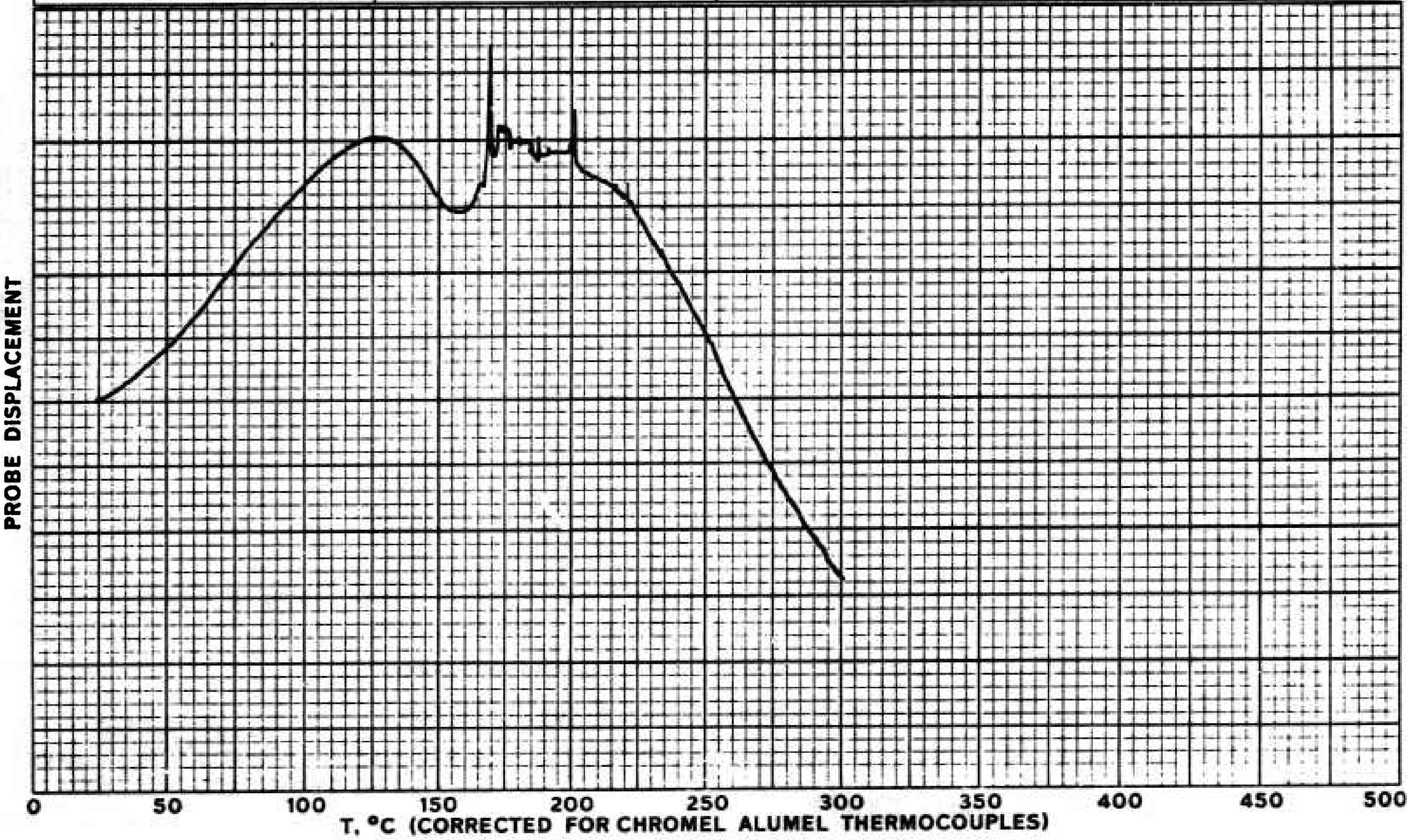


PART NO. 941017

SAMPLE: Phenyl Silane RESINOX [illegible] SC 1013 24 hr H2O boil 21 day soak	SAMPLE HEIGHT .25"x.25"x.25"	X-AXIS SCALE 50 °C/in.	RUN NO. 130
	LOADING ON TRAY 100g	Y-AXIS SCALE .04 in./in.	DATE 8-28-74
	PROBE: EXPANSION/PENETRATION	Y-AXIS SENSITIVITY	OPERATOR D.E. Dayton
ORIGIN: DD 319-157A	HEATING RATE 5 °C/min	in. probe displ./in. of chart	



SAMPLE: Phenyl Silane Resinox SC 1013 2 hr @ 250°F dry	**SAMPLE HEIGHT** .25 x .25 x .25	**X-AXIS SCALE** 50	**RUN NO.** 132
	LOADING ON TRAY 10 g	**Y-AXIS SCALE** .04	**DATE** 8-30-74
	PROBE: EXPANSION/PENETRATION	**Y-AXIS SENSITIVITY**	**OPERATOR** L E Dayton
ORIGIN: DD-319-151	**HEATING RATE** 5		



DUPONT INSTRUMENTS

PART NO. 941017

SAMPLE: Phenyl Silane Resinox SC1008 700°F cure

ORIGIN: DD-319-25R

SAMPLE HEIGHT .25 X .25 X .20

LOADING ON TRAY 100g

PROBE: EXPANSION/PENETRATION

HEATING RATE 5 °C/MIN

X-AXIS SCALE 50 °C/DIV.

Y-AXIS SCALE .04 MM/IN

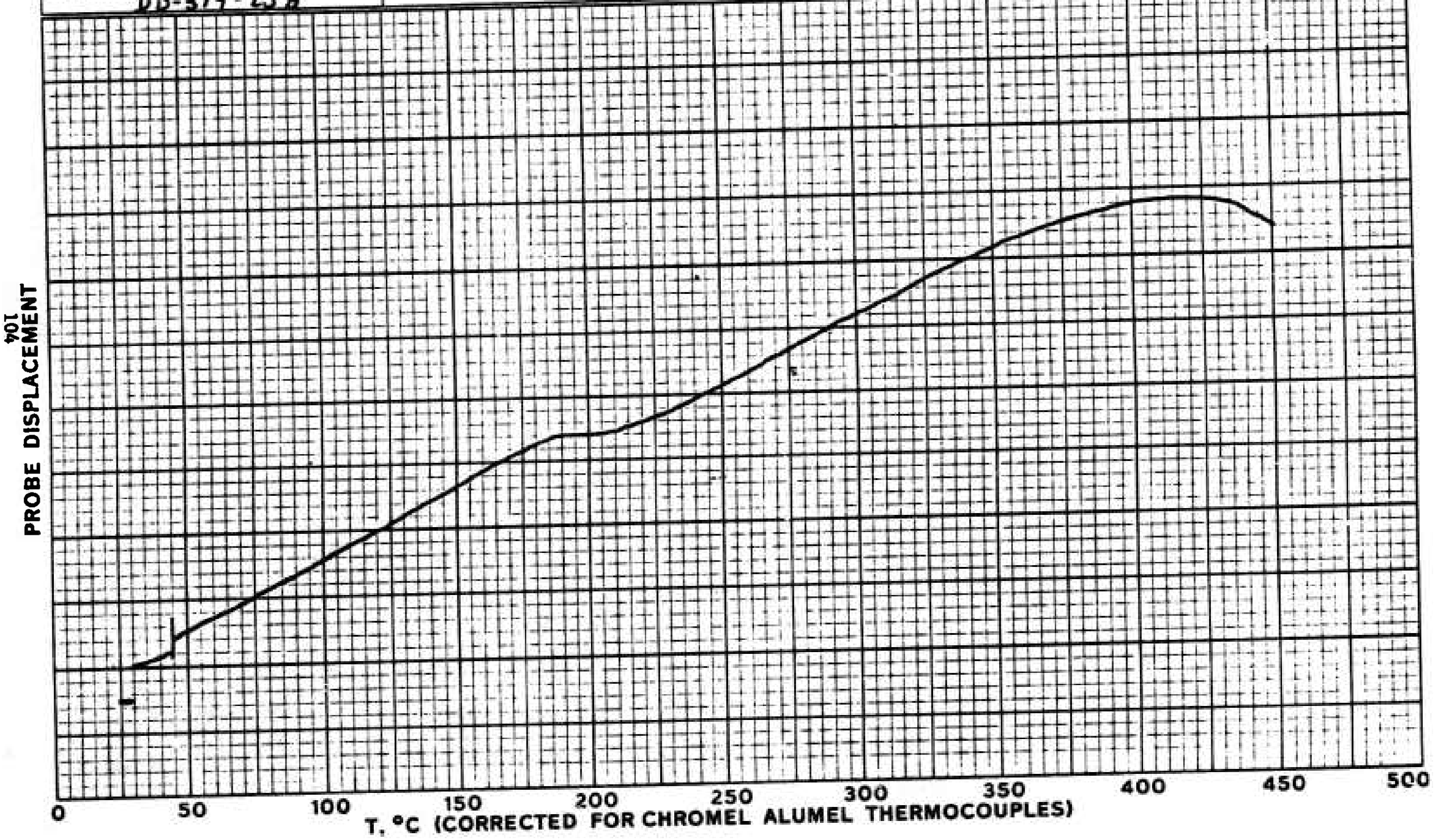
Y-AXIS SENSITIVITY

IN. PROBE DISPL./IN. OF CHART

RUN NO. 133

DATE 9-12-74

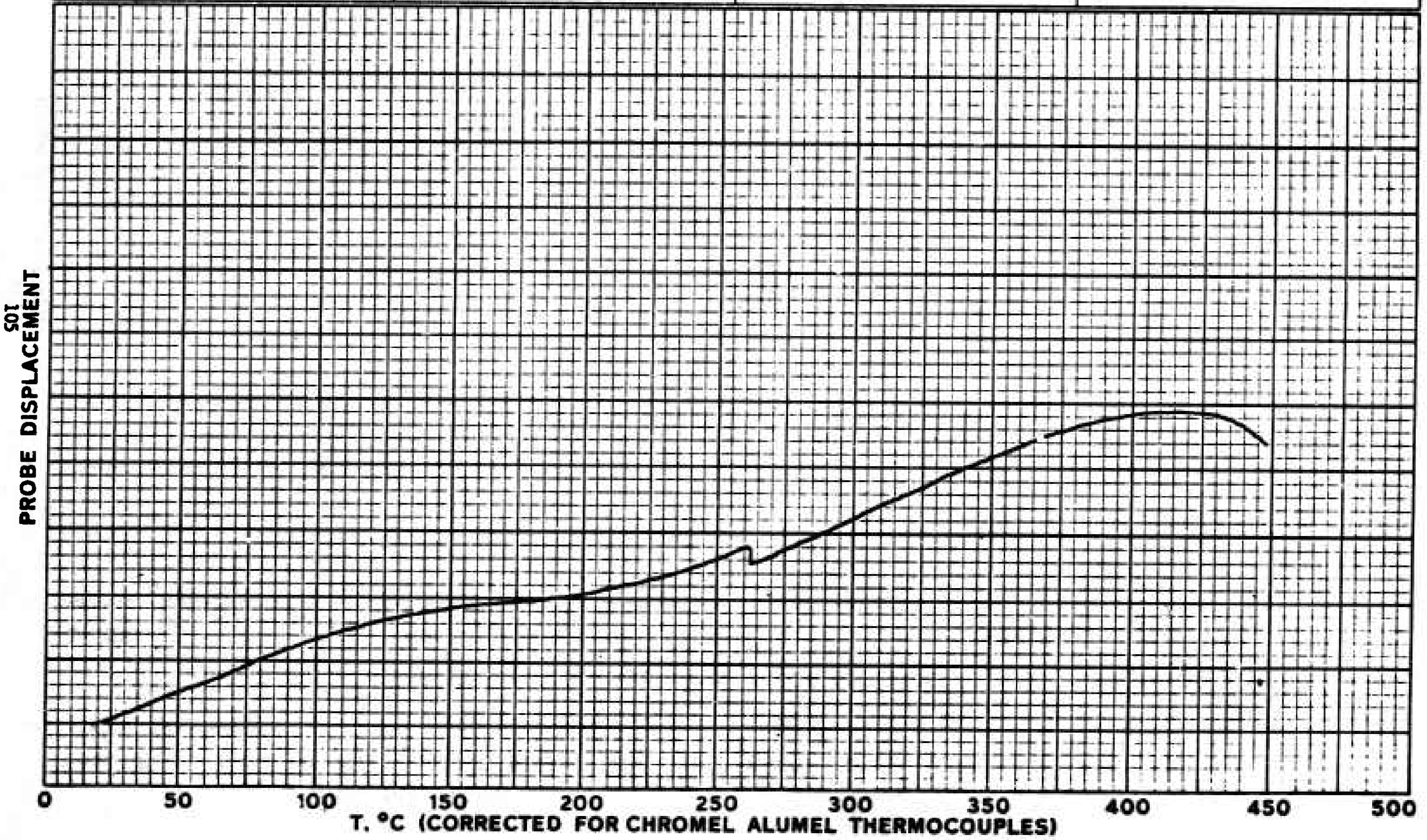
OPERATOR D.E. Dayton



PART NO. 941017

DU PONT INSTRUMENTS

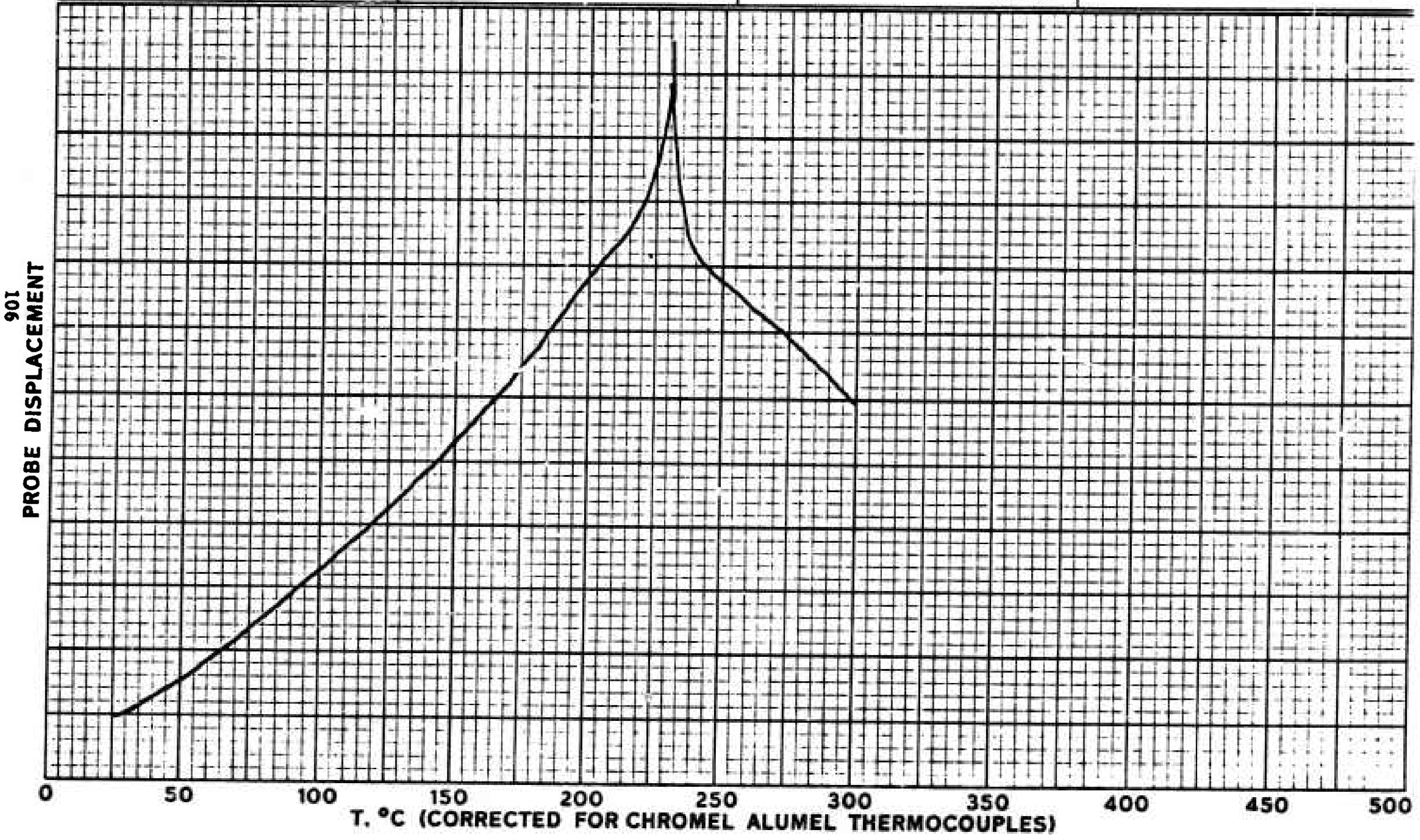
SAMPLE: Phenyl Silane RESINOX SC1013 700°F cure 24 hr H₂O Boil 24 hr H₂O Soak	SAMPLE HEIGHT .25 X .25" X 20"	X-AXIS SCALE 50 °C/DIV.	RUN NO. 14
	LOADING ON TRAY 100g	Y-AXIS SCALE 04 IN/IN	DATE 2-18-74
	PROBE: EXPANSION/PENETRATION	Y-AXIS SENSITIVITY	OPERATOR
ORIGIN: DD-319-25B	HEATING RATE 5 °C/MIN	IN. PROBE DISPL./IN. OF CHART	



PART NO. 941017

DUPONT INSTRUMENTS

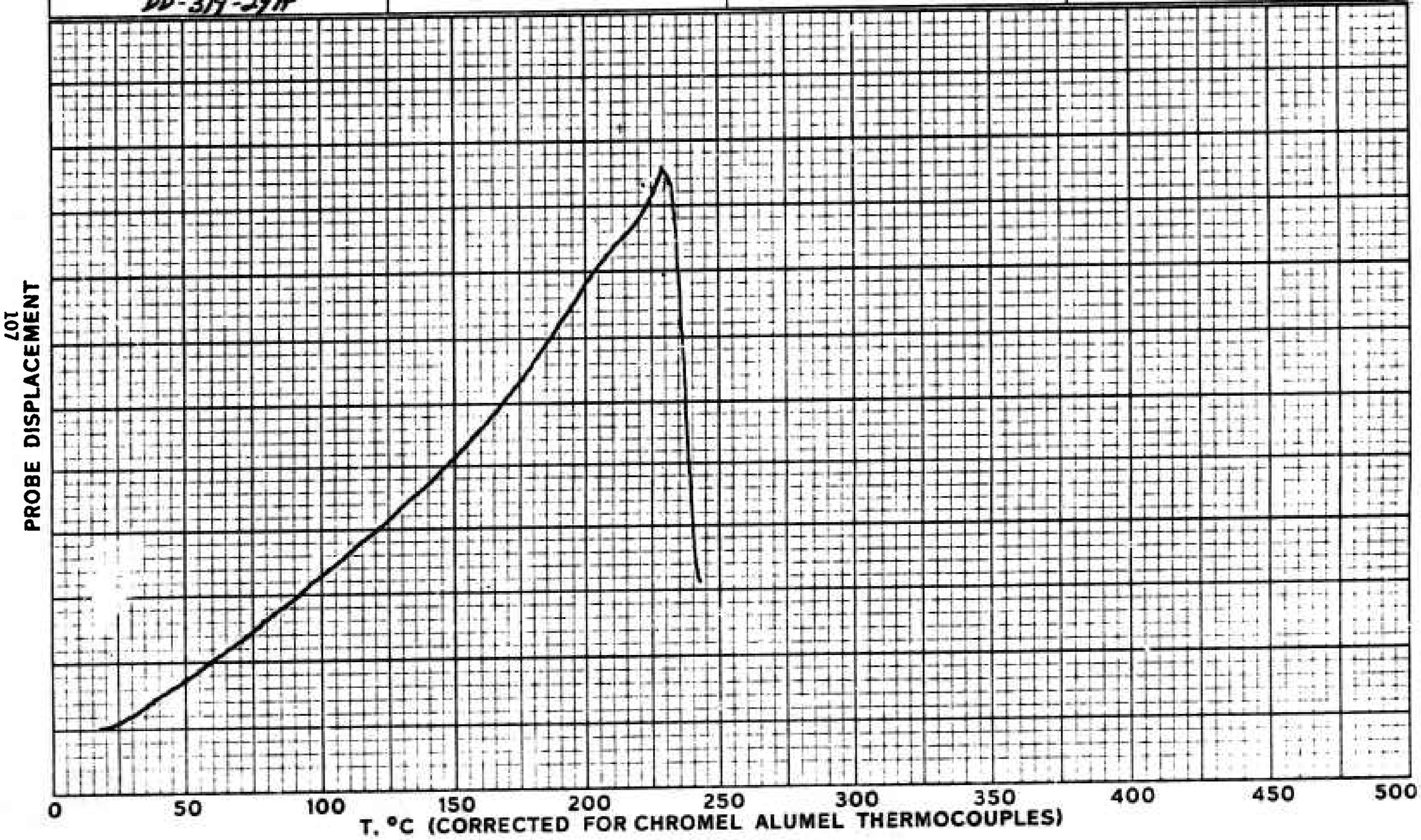
SAMPLE: HETRON 800FA 2 phr HCl catalyst	SAMPLE HEIGHT .25"x.25"x.25"	X-AXIS SCALE 50 °C/in.	RUN NO. 139
	LOADING ON TRAY 100g	Y-AXIS SCALE 04 mv/in.	DATE 9-16-74
	PROBE: EXPANSION/PENETRATION	Y-AXIS SENSITIVITY	OPERATOR
ORIGIN: DD-319-29A	HEATING RATE 5 °C/min	in. probe displ./in. of chart	



DUPONT INSTRUMENTS

PART NO. 941017

SAMPLE: HETRON 800 FA + 2 phr [illegible] catalyst 24 hr H2O Boil 24 hr H2O Soak	SAMPLE HEIGHT .25" X .25" X .25"	X-AXIS SCALE 50	RUN NO. #16
	LOADING ON TRAY 100g	Y-AXIS SCALE .04	DATE 9-19-74
	PROBE: EXPANSION/PENETRATION	Y-AXIS SENSITIVITY	OPERATOR DE Clayton
ORIGIN: DD-319-25A	HEATING RATE 5		



DUPONT INSTRUMENTS

PART NO. 941017

SAMPLE: HETRON 800FA
14 ply 800L
3 hr 210°F Dry Desiccator Storage

ORIGIN: DD-319-31A3

SAMPLE HEIGHT .25"x.25"x.25"

LOADING ON TRAY 100 g

PROBE: EXPANSION/PENETRATION

HEATING RATE 5

X-AXIS SCALE 50

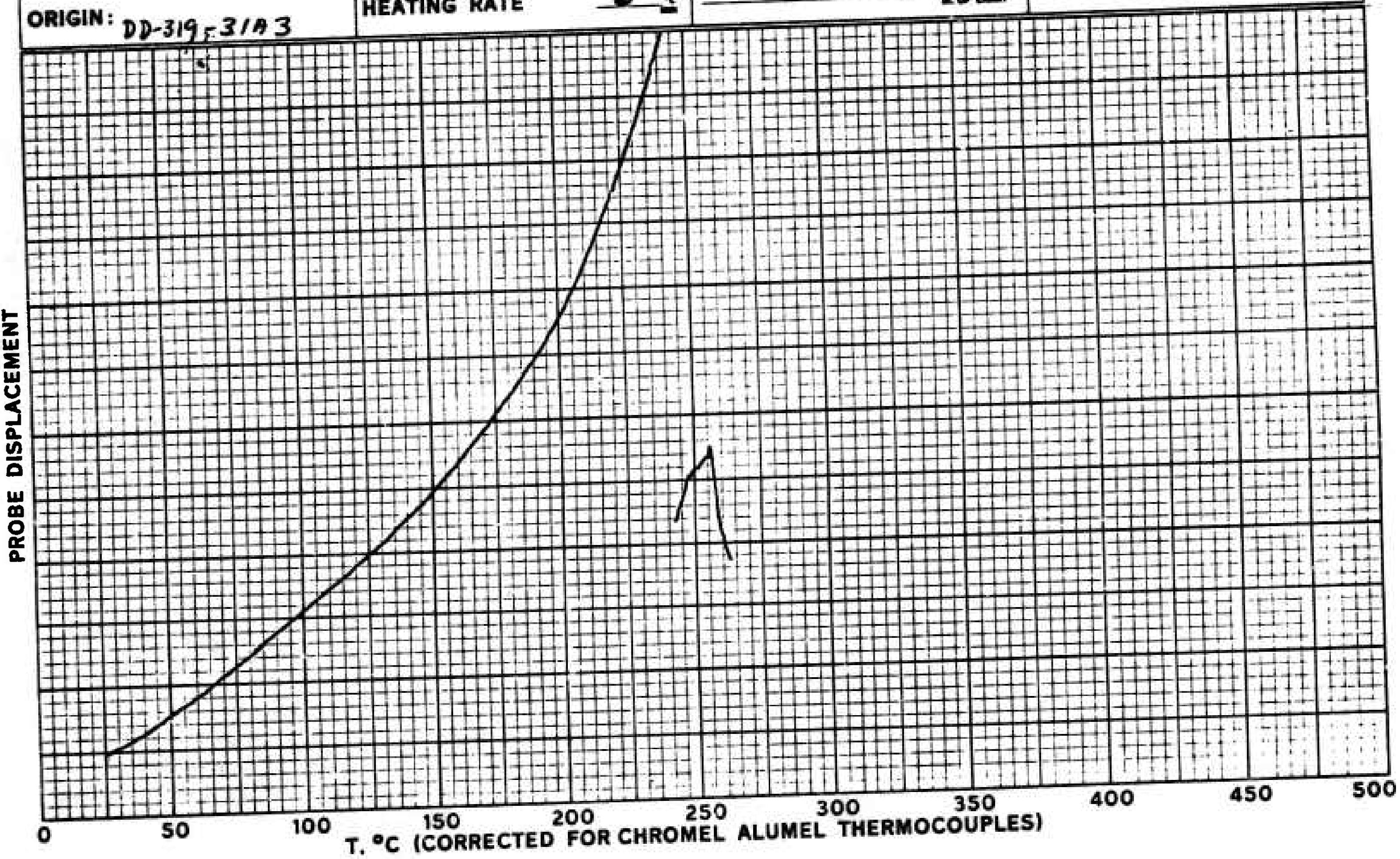
Y-AXIS SCALE .04

Y-AXIS SENSITIVITY

RUN NO. 149

DATE 10-4-74

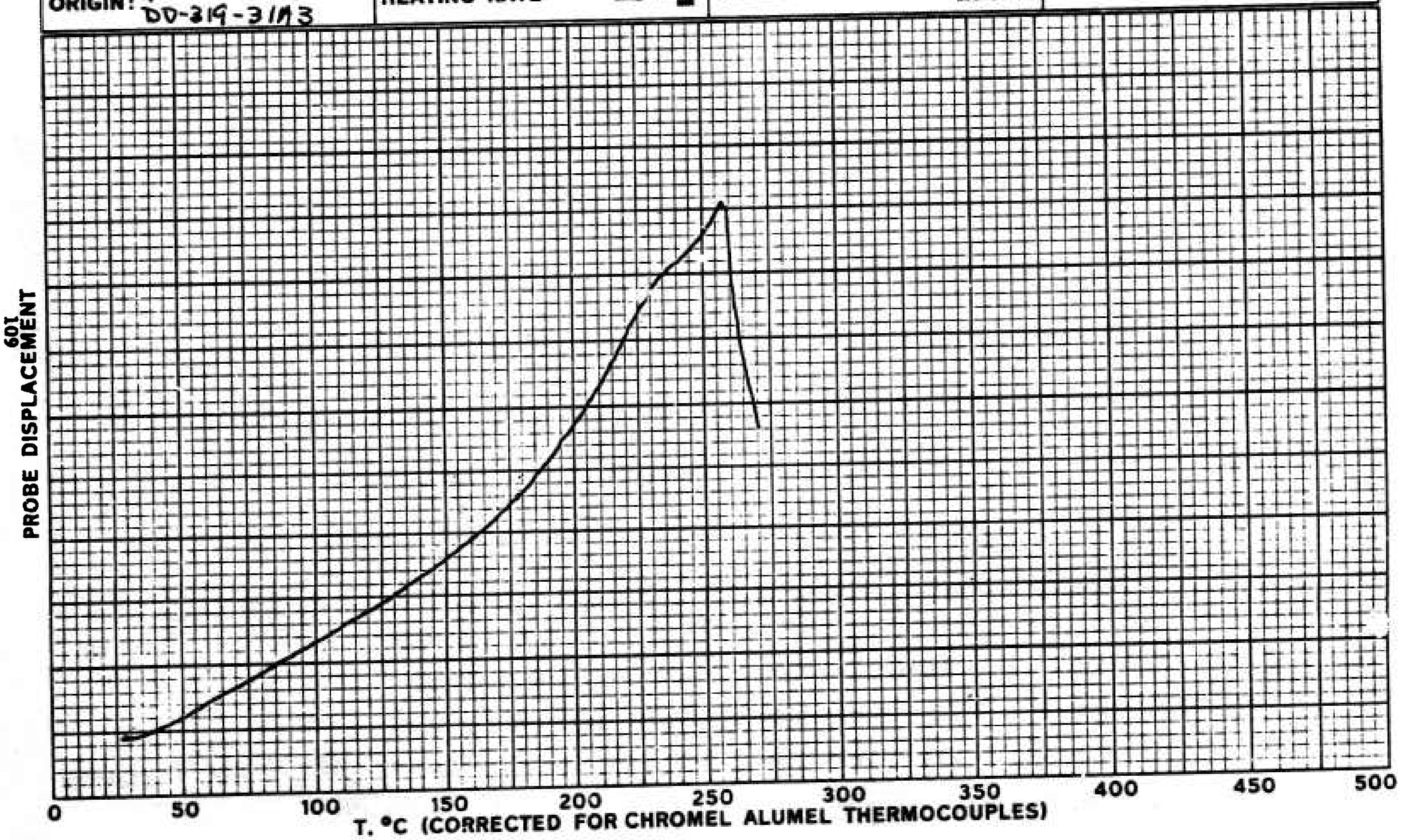
OPERATOR D. E. Dempton



DUPONT INSTRUMENTS

PART NO. 941017

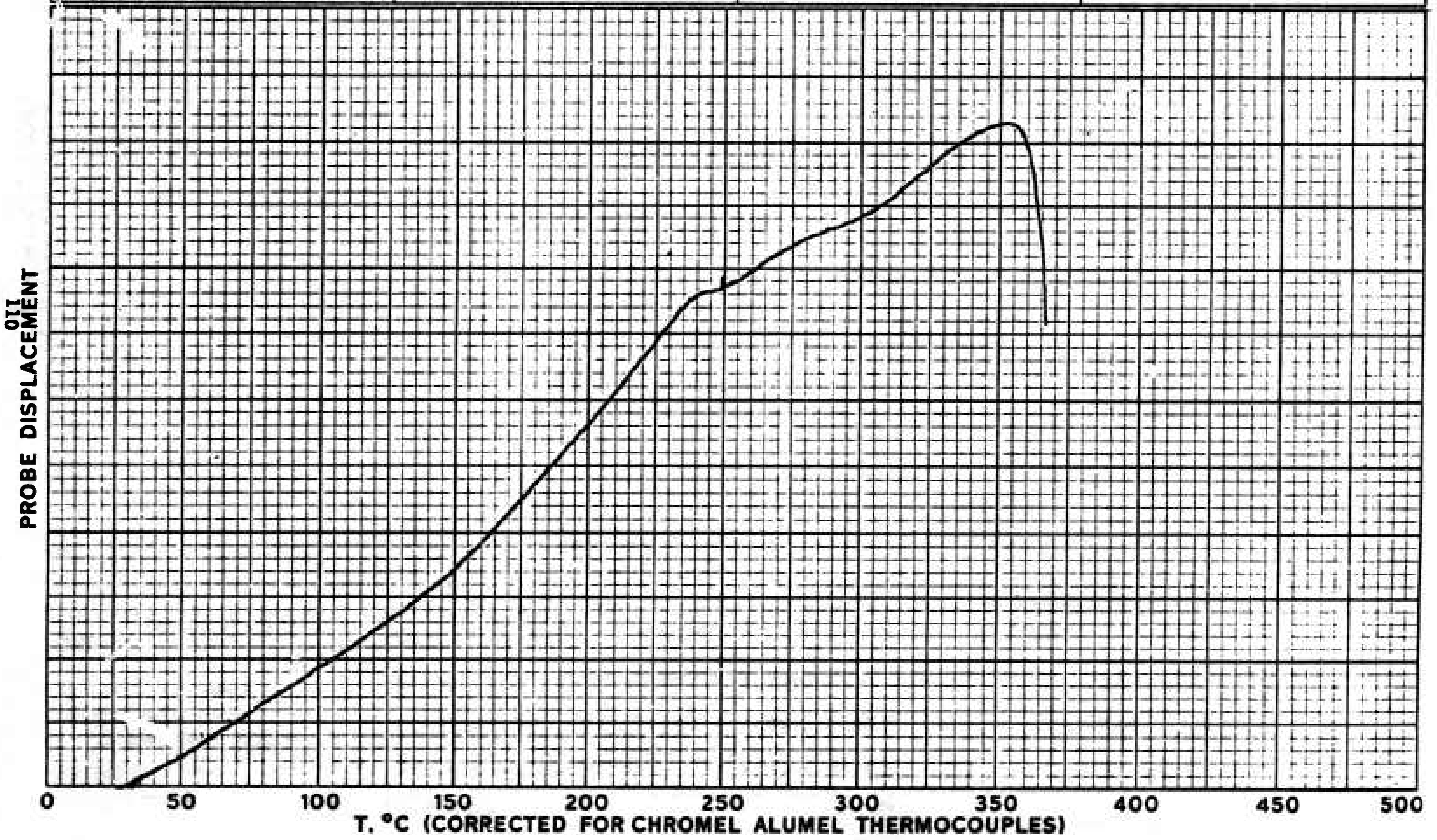
SAMPLE: HETRON 800 FA 1 HETRON 800L 24 hr H2O Boil 24 hr H2O Soak	SAMPLE HEIGHT .25"X.25"X.25	X-AXIS SCALE 50	RUN NO. 152
	LOADING ON TRAY 100g	Y-AXIS SCALE .04	DATE 10-9-74
	PROBE: EXPANSION/PENETRATION	Y-AXIS SENSITIVITY	OPERATOR D.E. Dayton
ORIGIN: DD-319-3113	HEATING RATE 5		



PART NO. 941017

DU PONT INSTRUMENTS

SAMPLE: Dapon M (DAIP)	SAMPLE HEIGHT .25" x .25" x .25"	X-AXIS SCALE 50 °C/in.	RUN NO. 138
	LOADING ON TRAY 100g	Y-AXIS SCALE .04 in/in	DATE 9-13-74
	PROBE: EXPANSION/PENETRATION	Y-AXIS SENSITIVITY	OPERATOR D Dayton
ORIGIN: DD-319-30A3	HEATING RATE 5 °C/min		



INSTRUMENTS

PART NO. 941017

SAMPLE: DAPON M (DAIP) (PDI)
24 hr H2O Boil
24 hr H2O Soak

ORIGIN: DD-319-30A3

SAMPLE HEIGHT .25"X.25"X.25"

LOADING ON TRAY 100g

PROBE: EXPANSION/PENETRATION

HEATING RATE 5

X-AXIS SCALE 50

Y-AXIS SCALE .04

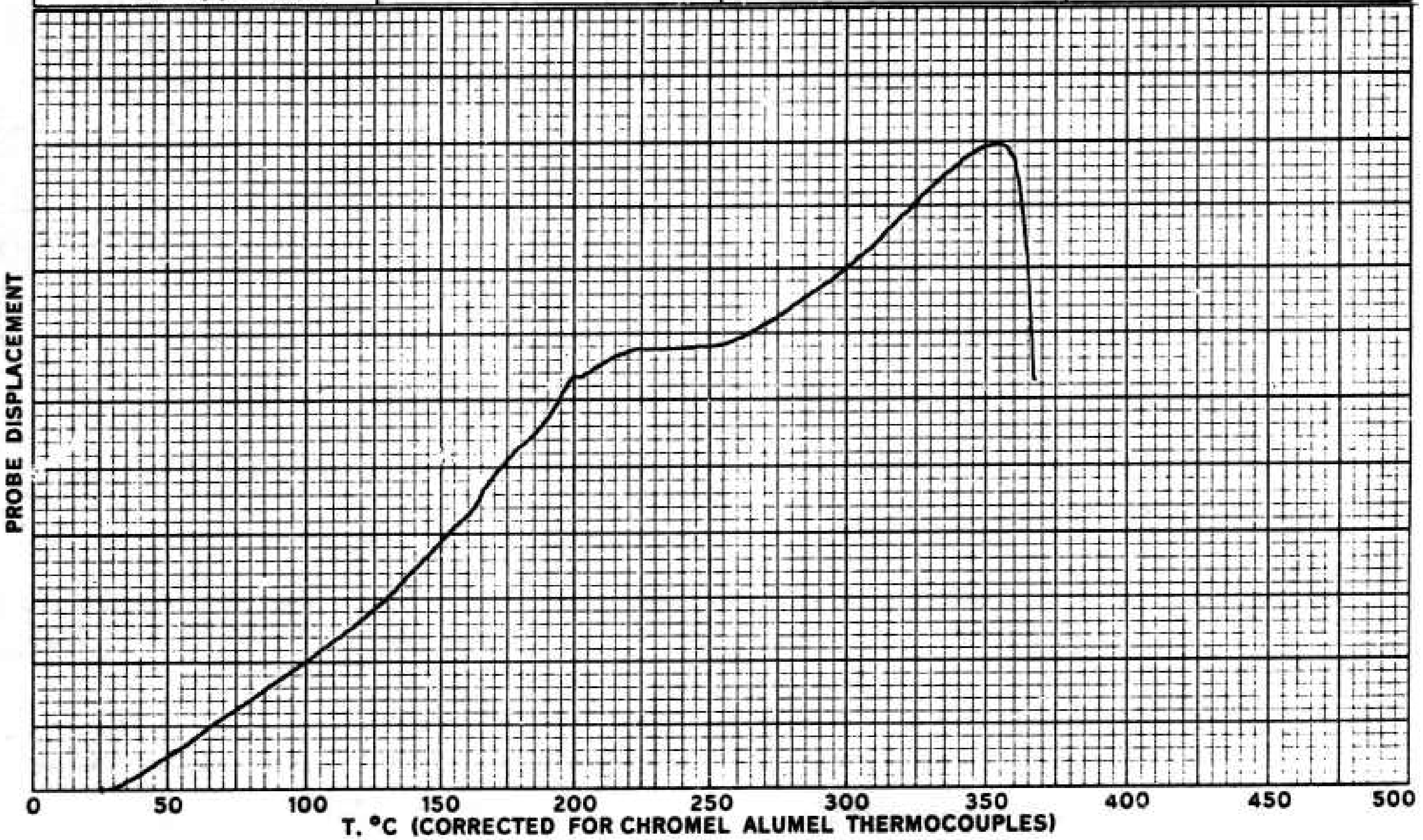
Y-AXIS SENSITIVITY

IN. PROBE DISPL. / IN. OF CHART

RUN NO. 147

DATE 9-20-74

OPERATOR DE Dayton



DU PONT INSTRUMENTS

PART NO. 941017

SAMPLE: DAPON M 70% / DAIP 30%
2 hr dry 250F

ORIGIN: 7D-319-558

SAMPLE HEIGHT .25" x .25" x .16"

LOADING ON TRAY 100g

PROBE: EXPANSION/PENETRATION

HEATING RATE 5 °C/min

X-AXIS SCALE 50 °C/in.

Y-AXIS SCALE .04 in./in.

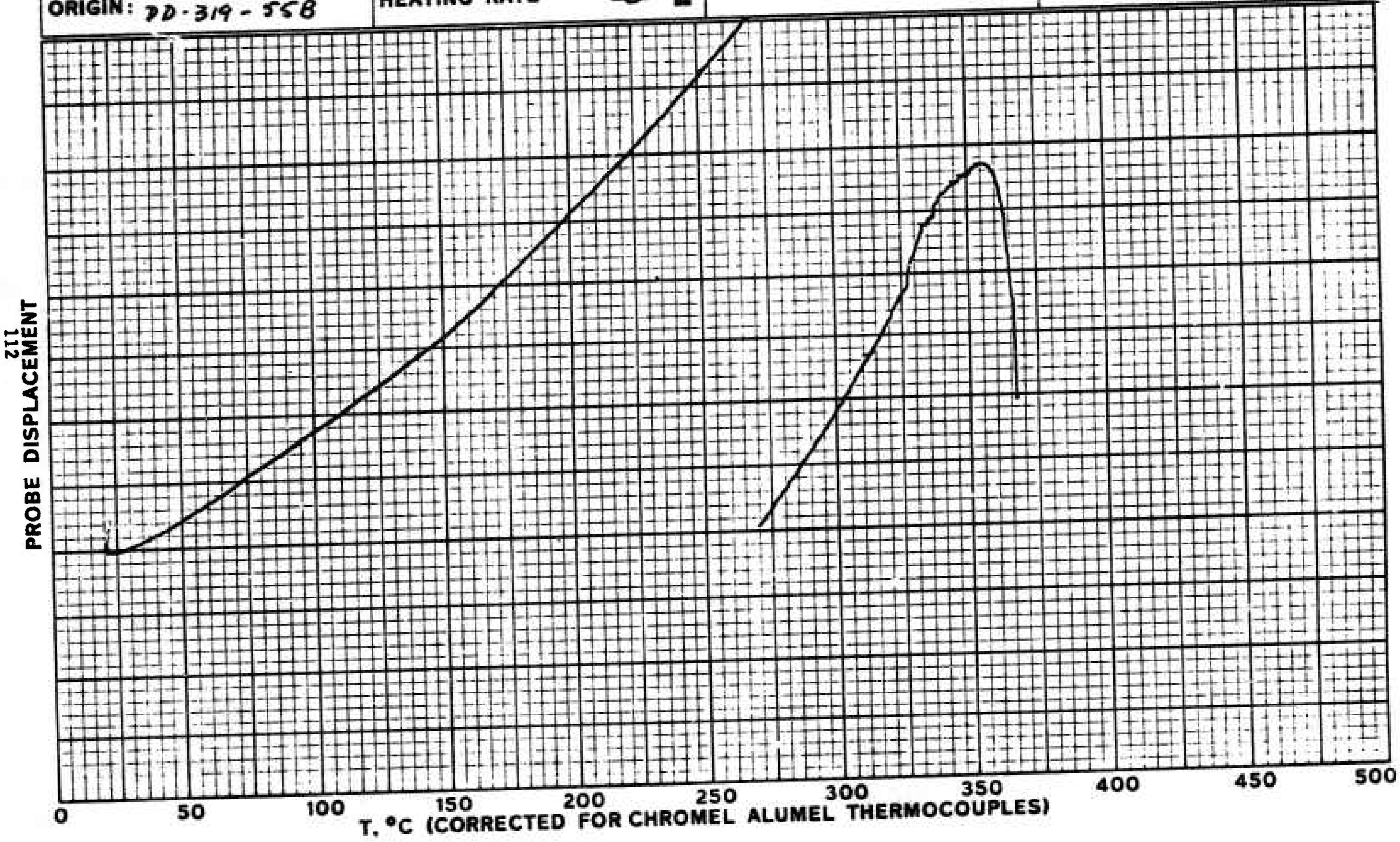
Y-AXIS SENSITIVITY

in. probe displ./in. of chart

RUN NO. 153

DATE 11-12-74

OPERATOR D E Dayton



DUPONT INSTRUMENTS

PART NO. 941017

SAMPLE: DAPON M / DAIP
70% 30%
24 hr H2O Boil
24 hr H2O Soak

ORIGIN: DD-319-55B

SAMPLE HEIGHT .25" X .25" X .25"

LOADING ON TRAY 100 g

PROBE: EXPANSION/PENETRATION

HEATING RATE 5

X-AXIS SCALE 50

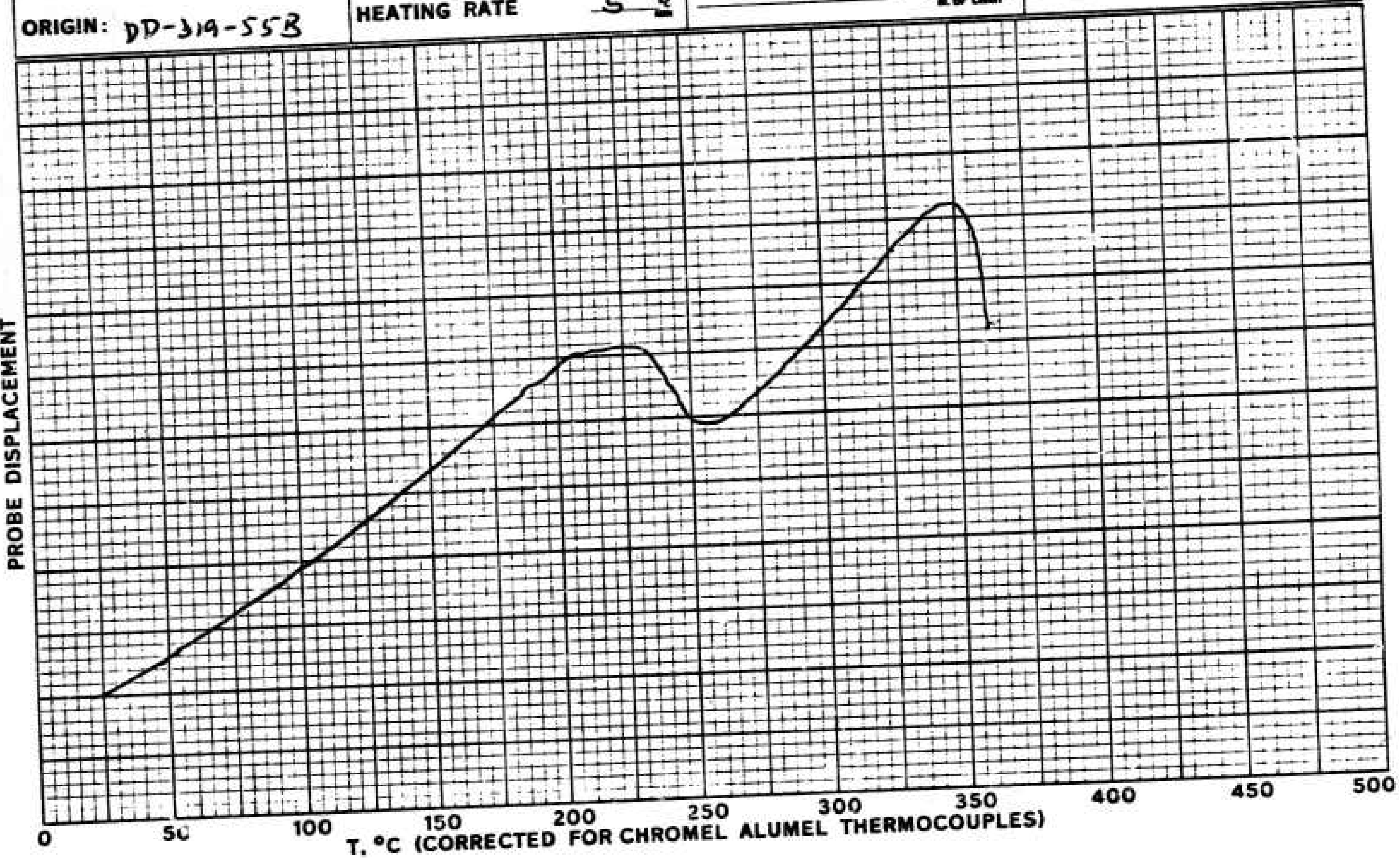
Y-AXIS SCALE .04

Y-AXIS SENSITIVITY

RUN NO. 156

DATE 11-13-74

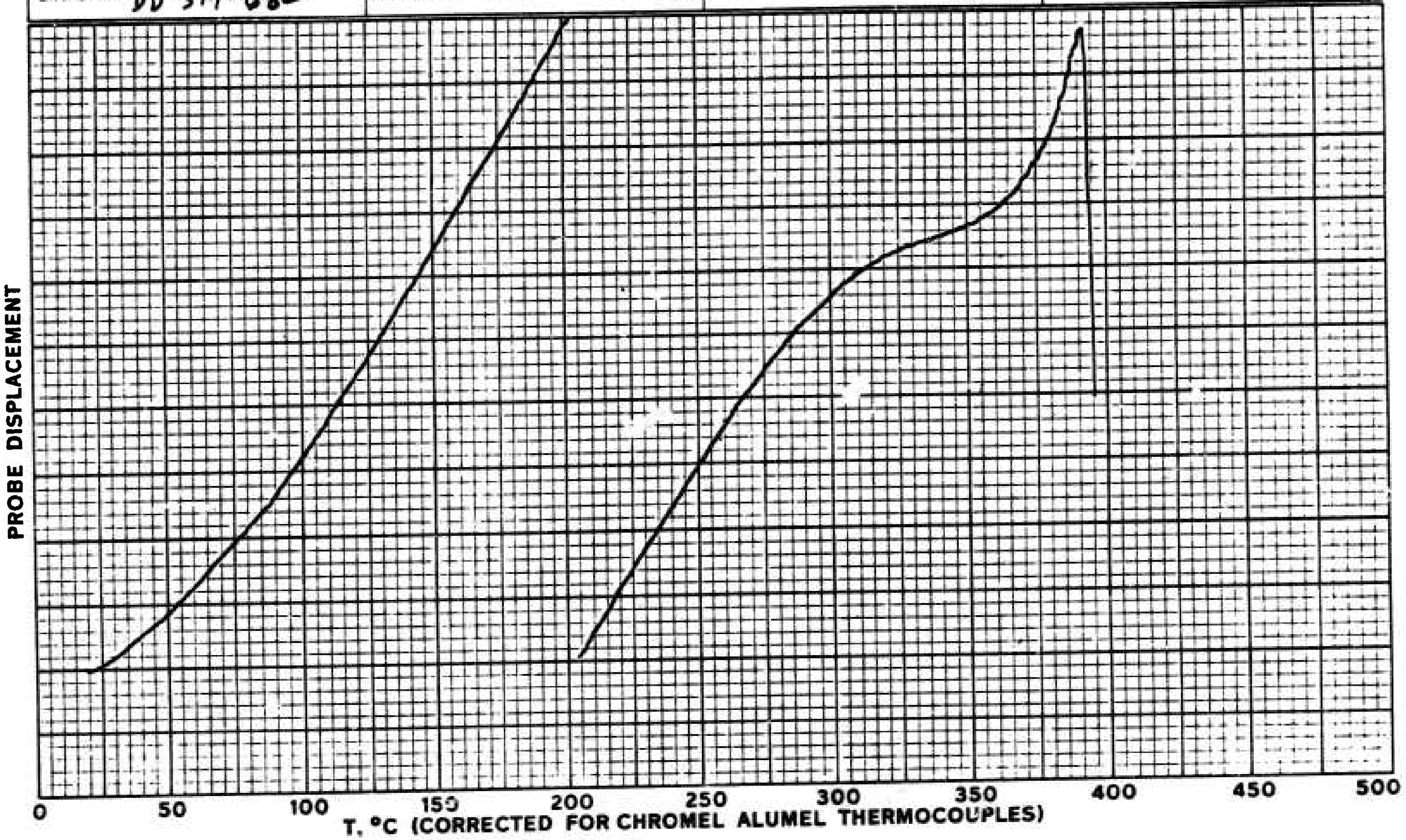
OPERATOR D E Dayton



DUPONT INSTRUMENTS

PART NO. 941017

SAMPLE: Ricon 431	SAMPLE HEIGHT .25x.25x.25"	X-AXIS SCALE 50	RUN NO. 157
	LOADING ON TRAY 100g	Y-AXIS SCALE .04	DATE Jan 21, 1975
	PROBE: EXPANSION/PENETRATION	Y-AXIS SENSITIVITY	OPERATOR DEDayton
ORIGIN: DD-319-68C	HEATING RATE 5		



DUPONT INSTRUMENTS

PART NO. 941017

SAMPLE: RICON 431
24 hr H₂O Boil
24 hr H₂O Soak

ORIGIN: ZD-319-68C

SAMPLE HEIGHT .25 X 2.5 X 20

LOADING ON TRAY 100g

PROBE: EXPANSION/PENETRATION

HEATING RATE 5

X-AXIS SCALE 50

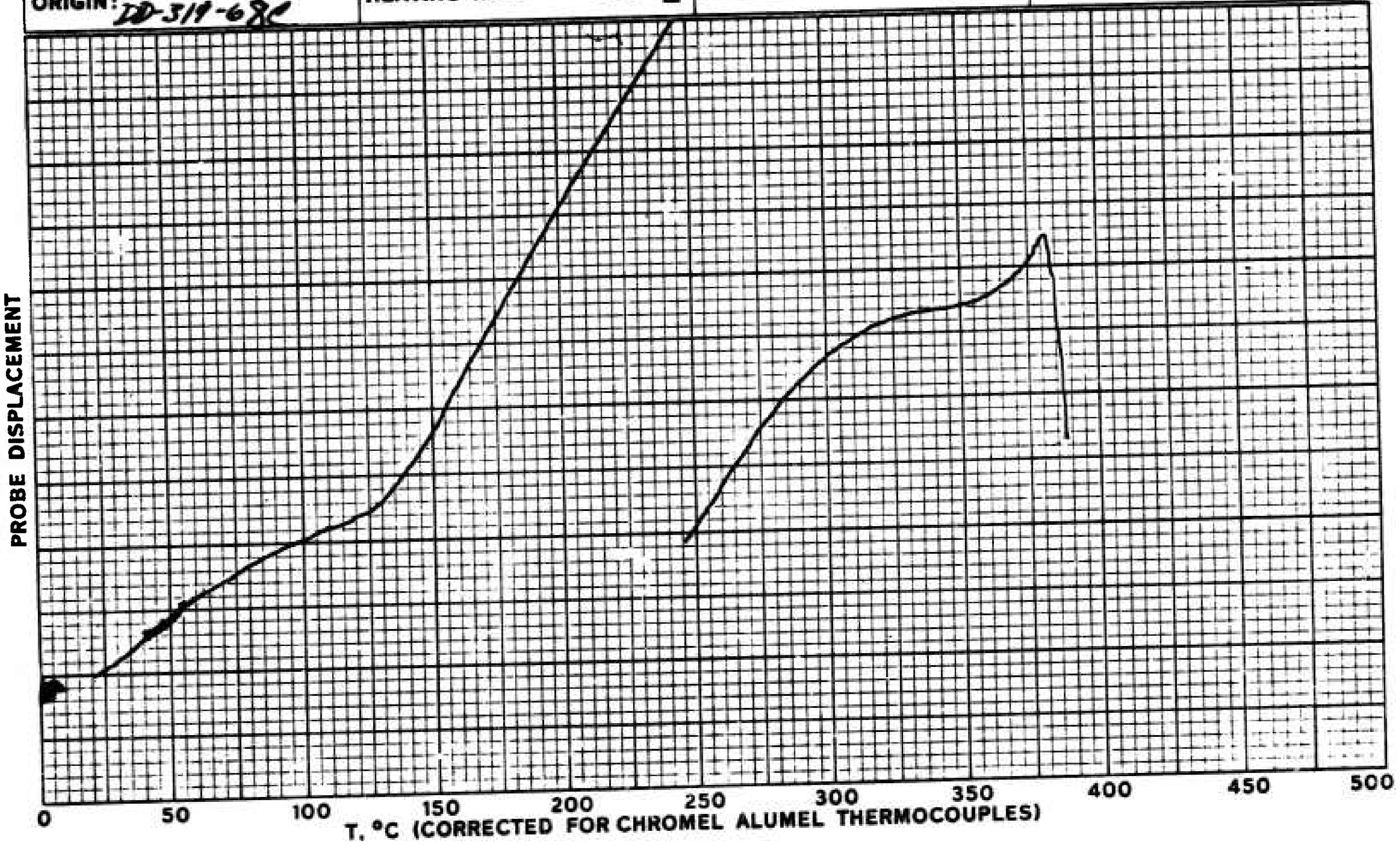
Y-AXIS SCALE .04

Y-AXIS SENSITIVITY

RUN NO. 169

DATE Jan 24 1976

OPERATOR



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