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FINAL REPORT

Lunar Sample Contracts Covering Period

from 7/1/68 thru 1/31/71

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Submitted by:

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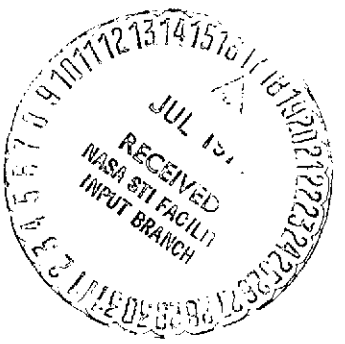


May 1974

Table of Contents

I. INTRODUCTION

Preparation for the study of lunar samples at Washington University began in July of 1968 when a contract was received from the National Aeronautics and Space Administration. Work on the actual samples started in September of 1969 when they were first received in our laboratory; this work has continued to the present day. Until January 31, 1971, the work was supported under a contract; since that time we have operated under a grant. This report summarizes our accomplishments as of January 31, 1971.

II. DESCRIPTION OF THE LUNAR SAMPLE LABORATORY AT WASHINGTON UNIVERSITY

A number of experimental techniques were employed in the period described. These included nuclear track measurements by optical and electron microscopy, thermoluminescence measurements, x-ray diffraction measurements, and differential thermal analysis (DTA) measurements. Subsequent to 1971 we have done little with either DTA or x-rays and have concentrated our efforts on the first two techniques. We have also added the capability, mainly through N.S.F. funds, for rare gas analysis.

The original contract provided for the purchase of a scanning electron microscope which has proved invaluable in our lunar work. As we correctly predicted, the track densities in many samples proved far too high to be studied by optical techniques. The original contract also made it possible to develop a high sensitivity thermoluminescence facility.

III. MAJOR SCIENTIFIC ACCOMPLISHMENTS THROUGH 1971

Our major research accomplishments up to 1/31/71 are detailed in the following nine papers:

G. Crozaz, U. Haack, M. Hair, M. Maurette, R. Walker and D. Woolum (1970), "Nuclear Track Studies of Ancient Solar Radiations and Dynamic Lunar Surface Processes," Proc. Apollo 11 Lunar Sci. Conf., Geochim. Cosmochim. Acta Suppl. 1, 3, 2051-2080, MIT Press.

H. P. Hoyt, Jr., J. L. Kardos, M. Miyajima, M. G. Seitz, S. S. Sun, R. M. Walker and M. C. Wittels (1970), "Thermoluminescence, X-ray and Stored Energy Measurements of Apollo 11 Samples," Proc. Apollo 11 Lunar Sci. Conf., Geochim. Cosmochim. Acta Suppl. 1, 3, 2269-2287, MIT Press.

D. S. Burnett, M. Monnin, M. Seitz, R. Walker, D. Woolum and D. Yuhas (1970), "Charged Particle Track Studies in Lunar Rock 12013," Earth Planet. Sci. Lett. 9, 127-136.

G. Crozaz and R. M. Walker (1971), "Solar Particle Tracks in Glass from the Surveyor 3 Spacecraft," Science 171, 1237-1239.

D. Burnett, M. Monnin, M. Seitz, R. Walker and D. Yuhas (1971), "Lunar Astrology - U-Th Distributions and Fission-Track Dating of Lunar Samples," Proc. Second Lunar Sci. Conf., Geochim. Cosmochim. Acta Suppl. 2, 2, 1503-1519, MIT Press.

G. Crozaz, R. Walker and D. Woolum (1971), "Nuclear Track Studies of Dynamic Surface Processes on the Moon and the Constancy of Solar Activity," Proc. Second Lunar Sci. Conf., Geochim. Cosmochim. Acta Suppl. 2, 3, 2543-2558, MIT Press.

H. P. Hoyt, Jr., M. Miyajima, R. M. Walker, D. W. Zimmerman, J. Zimmerman, D. Britton and J. L. Kardos (1971), "Radiation Dose Rates and Thermal Gradients in the Lunar Regolith: Thermoluminescence and DTA of Apollo 12 Samples," Proc. Second Lunar Sci. Conf., Geochim. Cosmochim. Acta Suppl. 2, 3, 2245-2263, MIT Press.

G. Crozaz, U. Haack, M. Hair, H. Hoyt, J. Kardos, M. Maurette, M. Miyajima, M. Seitz, S. Sun, R. Walker, M. Wittels and D. Woolum (1970), "Solid State Studies of the Radiation History of Lunar Samples," Science 167, 563-566.

R. Walker (1969), "Special Report on Potential Safety Hazard Due to Stored Energy Release in Lunar Samples," Lab. for Space Physics, Washington University, St. Louis.

There have been three major themes in our work: (a) the study of modern and ancient energetic particles in space as recorded in the lunar surface, (b) the study of lunar surface dynamics, and (c) the measurement of U and Th distributions in lunar rocks and the application of these data to the mechanisms of formation and ages of lunar rocks.

In the study of lunar surface dynamics we have measured surface exposure ages for lunar rocks (typically 1 to 10 my for 1 to 10 kg rocks), surface turnover rates (layers 6 cm thick can last undisturbed for $\gtrsim$20 my), absolute rates of microcratering (3 to 8 craters/cm^2/my >500μ in size), and rock erosion rates (typically 0.5 to 1.5 mm/my).

In the study of the radiations themselves we found solar flare tracks in lunar rocks and in a sample of glass returned from the Surveyor III mission. This allowed us to establish both the long- and short-term characteristics of solar flare heavy ions. This work covered a region of energy (<10 MeV/nuc) never before studied. We have further established the long-term constancy of heavy galactic cosmic rays.

We were one of only two groups out of some five or six that tried that proved capable of measuring thermoluminescence in lunar samples. This capability subsequently led up to the establishment of the properties of solar flare protons averaged over the last 5×10^3 y. It has also opened up the possibility of getting lunar heat flow data from lunar core samples.

Extensive mapping of the uranium distributions in lunar rocks led us, with several other groups, to the identification of the small, trace mineral phases in which uranium and thorium were concentrated. We were the only group to measure U and Th concentrations in these trace phases. The finding that Th/U ratios vary widely from one type of mineral to the next has important implications for the theory of nucleosynthesis. Extinct isotope effects were shown to be absent in samples returned from the Apollo 11 and 12 missions. (However, we were the first to find such effects in the Apollo 14 samples.)

The combination of different experimental techniques available in our laboratory made it possible for us to give a definitive negative answer to the possibility raised by several people that the Apollo 12

astronauts (and later astronauts) might be endangered by the lunar landings. These findings were given to NASA in a special report late in 1969.

IV. SUMMARY

The support provided by the NASA contracts during the period 1968 to January 1971 made it possible for us to equip a laboratory that was able to take immediate advantage of the returned lunar samples to obtain important new scientific information. Although our emphasis has naturally changed as time has gone on, we are still vigorously engaged in generating new scientific knowledge from the splendid collection of Apollo samples.

We feel highly privileged to have been a part of the Apollo program and are deeply grateful for the NASA support that made it possible.

NUCLEAR TRACK STUDIES OF ANCIENT SOLAR RADIATIONS AND DYNAMICS LUNAR SURFACE PROCESSES
G.CROZAZ, U HAACK■ M HAIR, M MAURETTE R WALKER, AND D WOOLUM
A70-41648 WASHINGTON UNIV.

CHARGED PARTICLES TRACK STUDIES IN LUNAR ROCKS 12013
D. S. BURNETT, M MONNIN, M SEITZ, R WALKER, D WOOLUM AND D YUHAS
UNIV. OF WASHINGTON A71-14216

RADIATION DOSE RATES AND THERMAL GRADIENTS IN THE LUNAR REGOLITH: THERMOLUMINESCENCE AND DTA OF APOLLO 12 SAMPLES
H P HOYT, JR. M MIYAJIMA, R M WALKER WASHINGTON UNIV.
A71-43776

LUNAR ASTROLOGY U-TH DISTRIBUTIONS AND FISSION TRACK DATING OF LUNAR SAMPLES
D. BURNETT, M MONNIN, M SEITZ, R WALKER AND D YUHAS WASHINGTON UNIV.
A71-43712

SOLAR PARTICLE TRACKS IN GLASS FROM THE SURVEYOR 3 SPACECRAFT
G CROZAZ AND R M WALKER
A71-23655

NUCLEAR TRACK STUDIES OF DYNAMICS SURFACE PROCESSES ON THE MOON AND THE CONSTANCY OF SOLAR ACTIVITY G CROZAZ WASHINGTON UNIV.
A71-43799

THERMOLUMINESCENCE, X-RAY AND STORED ENERGY MEASUREMENTS OF APOLLO 11 SAMPLES
H P HOYT, JR. J L KARDOS, M MIYAJIMA, M G SEITS
S. S. SUN, R M WALKER AND M C WITTELS WASHINGTON UNIV.
A70-41665

Preface

The work described in this report was performed by a team of students, research associates, and professors who have come together specifically to work with the principal investigator, R. Walker on the study of the radiation history of lunar samples. The individuals who are responsible for different areas of work are as follows:

Differential Thermal Analysis--Prof. John Kardos, Dept. of Chem. Engineering, Washington University.

Thermoluminescence-- M. Miyajima, Res. Assoc. Lab. for Space Physics, (Waseda Univ., Japan)
H. Hoyt, Grad. Student, Physics Dept. Washington University.

Fossil Track Studies--G. Crozaz, Res. Assoc. Lab for Space Physics, (Free Univ. of Brussels, Belgium)
U. Haack, Visiting Prof. and Res. Assoc. Lab. for Space Physics (Univ. of Gottigen, Germany)
D. Wollum, Res. Assoc. Lab. for Space Physics, Washington University.
M. Hair, Grad. Student, Physics Dept. Washington University
M. Maurette, Visiting Prof, and Res. Assoc. (University of Paris)

X-Ray Studies-- M. Wittels, visiting Prof. and Res. Assoc. Lab. for Space Physics. Washington University(Atomic Energy Comm. Wash.D. C.
M. Seitz, Grad. Student, Physics Dept. Wash. Univ.

These people have been greatly aided in their work by M. Marberry, A. Jones, and P. Swann.

SPECIAL REPORT ON POTENTIAL SAFETY HAZZARD DUE TO STORED ENERGY RELEASE IN LUNAR SAMPLES

This report is submitted in confidence to N.A.S.A. with the understanding that the material is not to be disseminated until after the Lunar Principal Investigators Meeting in January, 1970, or unless special permission is given by the Principal Investigator.

The work reported herein was performed by the following persons working in the Laboratory for Space Physics, Washington University, St. Louis, Missouri:

G. Crozaz	Research Associate	Free University of Brussels, Belgium
U. Haack	Visiting Professor-Dept. of Earth Sciences	University of Gottigen
M. Hair	Graduate Student in Physics	Washington University
J. Kardos	Ass't. Professor of Chemical Engineering	Washington University
M. Maurette	Visiting Professor	University of Paris, France
M. Miyajima	Research Associate	Waseda University, Japan
M. Seitz	Graduate Student in Physics	Washington University
R. Walker	Professor of Physics	Washington University
M. Wittels	Visiting Professor	U.S.A.E.C.--Washington, D.C.
D. Wollum	Research Assistant	Washington University

Special NASA Report on Possible Heat Release of Lunar Samples

Section I: Introduction:

This report concerns the possibility that lunar materials may heat up spontaneously due to the release of stored energy from the recombination of lattice defects produced by solar and cosmic ray nuclear particle irradiations of the surface.

The sudden release of stored energy is a well known phenomenon in nuclear reactor technology. If heavily irradiated graphite is heated to the point where the stored lattice defects become mobile, the ensueing recombination of interstitials and vacancies leads to release of energy. This energy release in turn raises the temperature of the material. Because of this increase in temperature, the recombination proceeds even faster leading to still more energy release. Under certain conditions a sudden rise in temperature occurs.

If this effect were present in lunar materials it would in itself constitute a potential hazzard. A doubly dangerous situation could conceivably arise if the increase in temperature led to the release of hydrogen gas that had been injected into the lunar materials by solar wind and solar flares.

Although it is always difficult to prove that no conceivable danger exists from a hypothetical set of circumstances, the measurements described in this report lead us to the conclusion that the danger in this case is very remote.

In the next section we describe measurements on stored energy release, thermoluminescence, and fossil track backgrounds in lunar fine materials. Both bulk fines and samples from the longer of the two core tubes were studied. One interesting sidelight of this work has been the demonstration that the longer core tube probably does represent a true vertical sampling of the lunar surface.

Following the description of the experimental results we present a brief theoretical discussion of the effects that might be expected. In the following section we discuss the implications of our results concerning the possible hazzards for lunar missions. The final section summarizes our major results and conclusions.

Section II: Experimental Results:

A) Differential thermal analysis.

The most direct way to test the question of a possible sudden release of stored energy is to warm the samples and measure the amount of heat released. We have made these measurements using a Dupont model 900 differential thermal analyzer. In this apparatus the sample is heated rapidly and its temperature rise is compared constantly to the temperature rise in a reference sample. The results are plotted as a temperature difference ΔT (y axis) between the unknown and the reference sample versus the temperature itself (x axis).

If there is no take up or release of energy by the unknown, and if the heat capacity of the unknown is exactly matched to that of the reference sample, then a flat horizontal line results. If there is no energy release but the heat capacities are different (as a practical matter this is usually the case) then a sloping straight line, referred to as the base line, results. Energy release in the unknown is signaled by a sudden change in slope of the line in the positive direction during the time the sample is releasing energy, followed by a return to the extrapolated base line when the energy release is finished. Thus energy release gives rise to a positive peak in the curve of ΔT vs. T. Changes in heat capacity vs. T will also give rise to changes in the slope of the base line. As a rule however, these slope changes stay fixed as the temperature increases further

and no peak is observed. In any event the effect of changing heat capacity can be taken into account by running a cooling curve and a re-heat of the sample. An irreversible exothermic process of the type we are looking for here shows up as a peak in the original curve; this peak is missing when the sample is cooled or re-heated.

In Fig. 1 we show a DTA run on a 13.6 mg sample of as-received bulk lunar fines. This run was made using an intermediate temperature cell with a heating rate of 30°C/min. It can be seen that there is evidence for a broad exothermic peak in the region between 450°C and 710°C. That this is a real peak can be seen in Fig. 2; the re-heat curve shows no evidence for the peak observed in the original curve. Several repeat runs using different samples give essentially the same results.

Although there is a definite effect, the total amount of energy released is small, amounting to $\lesssim$ 20 cal/gm. This estimate is based on a calibration using the α-β transition in quartz and is good to $\pm$ 20%. This amount of energy release could not cause a catastrophic temperature rise in the lunar material, even if it occurred over a narrow temperature interval. If we further consider the fact that the energy release is very broad then it may be concluded that the observed energy release does not constitute a safety hazard.

The origin of the observed energy release is not known but could be due to either a radiation effect or a chemical reaction. In Figs. 3 and 4 we show previous data obtained in our laboratory on terrestrial hypersthene.

In Fig. 3 a noticeable peak of about 15 cal/gm is observed between 550° and 750°C in a sample that was irradiated with 3.7×10^{15} p/cm^2 of 30 MeV alpha-particles. The peak is missing in the non-irradiated control shown in Fig. 4.

From the safety point of view, the low temperature region immediately above 20°C is of more interest than the higher temperature regions. We have therefore made a series of runs at higher sensitivity using a low-temperature cell that extends to 500°C. In Fig. 5 we show a high sensitivity run on as-received lunar bulk fines. There is evidence for a very weak exothermic peak between about 250° and 450°C. This peak, which is approaching the sensitivity detection limits of the apparatus, represents < 4 cal/gm. Because of its low value and its breadth, this peak does not constitute a safety hazard.

In the last few days we have made additional measurements in the 0 to 500°C range using samples removed from the following distances from the top of the longer of the two core tubes: 0 cm, 3 cm, 6cm, 9 cm, 12 cm. The results are shown in Figs. 6-10. In all cases there is evidence for the broad low temperature exotherm previously seen in the bulk sample. In addition there appears to be a considerable variation from the top to the bottom of the core although the variation is not strictly monatonic. Specifically, the following values were found for the 250° - 400°C peak: ocm, <0.5 cal/gm; 3cm, 1.9 cal/gm; 9cm, 1.4 cal/gm; 12cm, 5.7 cal/gm.

The sample at 6 cm. is clearly anomalous. There is a sudden slope change at $\sim 275^{\circ}C$ which persists to high temperature. On cooling, the new slope persists to low temperature indicating an irreversible change in the material. To investigate this core position further we ran a new sample in the 0° to $800^{\circ}C$ range using maximum sensitivity. As can be seen in Fig. 11, both the low temperature and the high temperature behavior are anomalous (see for comparison Fig. 1). There is a very broad exotherm extending from $\sim 250^{\circ}$ to $\sim 700^{\circ}$ C. with a total energy release of $\lesssim 6$ cal/gm. As we shall show later the 6 cm core sample also gives an anomalous glow curve. It is tempting to speculate that these anomalies may be correlated with the visual anomaly that was observed at about this depth by Fryxell in his original examination of this core.

In Figs. 12 and 13 we show high sensitivity runs on samples of terrestrial feldspar and pyroxene that were irradiated with 3.7×10^{15} α particles/cm^2, The temperature of the samples during irradiation was not measured in these experiments. In Fig. 14 we show a sample of bulk lunar fines that was irradiated with 2×10^{14} α -particles/cm^2. In this latter experiment we estimate from thermoluminescence measurements that the temperature of the samples did not exceed $90^{\circ}C$. during the irradiation.

In none of these runs do we see any evidence for a large stored energy release induced by the irradiations.

B) Thermoluminescence measurements.

We have also made glow curve measurements on the bulk fines and more recently on the core tube samples described in Section II (A). In Fig. 15 we show the curve of light output vs. temperature for a sample of lunar bulk fines. The heating was accomplished in 150 sec. using a programmed oven with a nichrome heating element. The temperature was monitored with chromel-alumel thermocouples and the light output was measured with an EMI 9635QB photo multiplier.

The excess of light output in the first heating of the lunar material over that in a second subsequent heating measures the thermoluminescence of the lunar samples. The obvious interpretation of the observed (weak) thermoluminescence is that it represents recombination of trapped electrons that were produced during the prolonged irradiation of the material on the lunar surface.

Before accepting this conclusion, however, we considered other possible explanations for the observed glow curve. We first considered the possibility that the observed glow was produced by the exposure to room light during the handling. This was checked by exposing a sample of as-received lunar material within 3 cm of a fluorescent light for a period of 70 hrs. The sample was subsequently mixed with silicone oil and heated. We observed no appreciable difference with the glow curve of the sample that had not been exposed extensively to light. To check this point we pre-heated a sample of bulk fines in an atmosphere of argon gas to 500°C and then exposed it again to the flouresent light. Once more no appreciable effect was observed.

We also considered the possibility that the glow was produced by triboluminescence--the production and storage of trapped electrons by mechanical deformation. To test this possibility we put a sample of an as-received bulk fine in a small beaker and then shook it vigorously by hand during one hour. To complete this series of tests we also vibrated a sample of pre-heated lunar material. No significant effect was observed in either case.

There rests the final possibility that the observed glow curve is caused by a chemical reaction of the lunar material as the sample is heated. Since the sample is heated in an inert atmosphere, we considered this an unlikely explanation. There is further supporting evidence that this is not the cause of the observed glow curve. When a lunar sample is exposed to ionizing radiation from a Sr^{90} source and subsequently heated, a considerably larger amount of light is emitted than in the case of a non-irradiated sample (see Fig. 15). Thus the lunar material is intrinsically thermoluminescent.

Any lingering doubts about the authenticity of the observed glow curves would appear to be removed by the runs that we have just completed on the core tube samples. As shown in Fig. 16 there is a striking difference in the glow curves of samples removed from different depths. The top surface sample shows the smallest effect and the bottom sample the largest. This experiment shows two things:

A) The core tube has not been mixed; it is a true vertical section

of the lunar surface and B) the thermoluminescence that we observe is an intrinsic property of the lunar material on the lunar surface. The increase with depth is not, again strictly monotonic. As mentioned before, the fact that the 6 cm and 9 cm samples are rather similar may be associated with the thin line of lighter colored material noted by Fryxell in his original examination of this core tube. This is an interesting direction for future research.

The observed depth variation could arise in a number of different ways. It might for example be due to a different sensitivity of the material as a function of depth; this in turn could be due to a vertical variation in the radiation damage or in the chemical composition. More likely it reflects the normal vertical temperature profile of the surface; those samples near the top get drained more and those samples deeper down are colder and are drained less. Also playing a role is the variation of ionization density with depth which increases for galactic cosmic ray bombardment.

C) Fossil track studies.

When heavy nuclear particles traverse a solid, they disarrange the atoms and leave a permanent trail of radiation-damaged material. This trail can be revealed for study by selective chemical etching. If the sample is heated prior to etching, the radiation damage recovers and no tracks are found.

In a survey of over twenty samples each of olivine, pyroxene, and feldspar we have found that every sample studied had a background of

fossil tracks. The densities were very high, ranging from $\sim 1 \times 10^7/cm^2$ to $> 5 \times 10^8/cm^2$ (see for example Fig. 17). Although we have also studied numerous glass spherules, the results are quite variable and cannot yet be simply interpreted.

Preliminary annealing measurements show that the tracks in both the feldspar and olivine are erased almost completely by heating for 30 min. at 550°C. Since the track densities are initially extremely high in all the grains studied, the effect is very striking. We conclude from these observations that the vast majority of the crystals in the as-received bulk fines have not been severely heated in recent times.

D) X-ray measurements.

As a preliminary check on the state of lattice disorder in the core tube samples, we chose six crystal fragments at random from the 12 cm fraction and measured their Laue x-ray transmission characteristics In Fig. 18 we show a typical pattern. As far as we can tell from these photographs the crystals are not severely disordered as they would have to be if they contained large amounts of stored energy.

Section III: Some Theoretical Considerations of Expected Energy Release:

In this section we discuss briefly the theoretical calculation of expected stored energy release. It is included simply to demonstrate that there is no pressing theoretical reason to expect a large effect.

It is possible to show by an a priori calculation that the lunar materials could conceivably contain enough lattice defects to produce a significant stored energy release. Such a calculation involves the assumption that the lunar materials store lattice defect energy as efficiently as graphite. The calculation also requires rather maximizing assumptions about the way in which the samples have been irradiated.

It is possible to show however that the irradiation history of the actual lunar samples is probably incompatible with a large stored energy release. For example, our track results indicate a probable average track concentration of $\sim 10^9/cm^2$. Such a track concentration could give at most 0.5 cal/gm of stored energy. Similarly it is possible to show that the maximum possible stored energy produced by solar wind particles is $\sim$ 1.5 cal/gm. It is also possible to show that the general point defect concentration, not visible in the form of tracks, probably contributes less than 5 cal/gm. (this part of the calculation is the least certain). Thus we consider it uncertain that even the small amount of stored energy that we observed is due to radiation damage.

The reasoning sketched out briefly above can get quite complicated when the details are taken into account. The arguments are also not above criticism; for example, the track densities are so high that it

is difficult for us to measure a reliable "average" value. The major burden of resolving the question of a potential large stored energy release must therefore rest with our direct experimental measurements. The direct experimental approach is also more desirable since there are potential sources of stored energy other than radiation damage that could conceivably affect the lunar samples.

Section IV: Discussion:

The most important single fact is this: In every sample that we studied, including those from the top to the bottom of a 12 cm. core tube, the observed stored energy release was < 20 cal/gm. Futhermore, the energy release pattern was very broad. Such material is not hazardous.

An independent arguement on the stored energy question can be based on the track observations alone. The essential point is the following: To have extensive stored energy release, the solid must be at a temperature where lattice defects are mobile; but fossil nuclear particle tracks are known to anneal out when the point defects become mobile. Hence the presence of fossil nuclear particle tracks indicate that no extensive release of stored energy occured prior to our reception of the samples in the laboratory. The only exception to the above line of reasoning is the ilmenite fraction; as yet we do not know how to reveal tracks in this material.

Before concluding that there is no possible danger of stored energy release there is an important point that must be considered. The glow curves shown in Fig. 16 can be interpreted to mean that the sample has been partially heated, perhaps to as high as 200°C to 250°C.

Because of the observed variation with depth it is extremely unlikely that this could be caused by an internal spontaneous heating due to a release of stored energy. However, the question might be

raised whether the samples have been partially heated by an external source such as the LEM rocket exhaust. If this were the case, would it not be possible to have had a large, sharp stored energy peak below 250°C that was simply missed in the current set of investigations? And if this hypothetical stored energy had been there, might it not constitute a hazzard for future Apollo missions where fine materials would be collected from locations remote from the rocket exhaust?

In the short time that we have had available it is very difficult to prove that such a hypothetical stored energy peak did not exist. However, we think that its existence is unlikely. Firstly, we do not expect extensive defect migration in this temperature range. Our previous experience with terrestrial feldspars, pyroxenes, olivines, and glasses indicates that structural lattice defects anneal out at higher temperatures. Secondly, we have previously pointed out that the large densities of tracks observed in every grain indicates that extensive lattice defect migration has not occurred. Thirdly, we have shown (see Fig. 14) that a lunar sample irradiated with α-particles does not show a pronounced stored energy peak below 200°C. Ideally, we would have perferred to have had the time to perform more extensive irradiations on cryogenically cooled samples. However we believe that the above is sufficient to show that the hypothetical stored energy release is unlikely.

As regards the safety question, however, there is an even more telling point; even if the samples did contain a hypothetical stored energy peak that was removed by the rocket exhaust, this energy release was not sufficient to heat the sample substantially above 250°C.

This is proved by the positive effects seen at 250°C and above in both the thermoluminescence and the D.T.A. measurements. Heating to this temperature in the sample box could probably be tolerated even in the unlikely event that it occurred.

We are therefore of the opinion that the possible release of stored energy by the lunar materials is a very remote hazzard.

V: Summary:

The salient experimental observations are as follows:

1) DTA runs on as-received lunar fines show two broad exothermic peaks from 250° to 400°C and from 450° to 700°C.

2) DTA runs on core tube samples show these same peaks but with different intensities, the top giving the least and the bottom the most stored energy.

3) The stored energy peaks always amount to $\lesssim$ 20cal/gm and are very broad.

4) Alpha-irradiated terrestrial minerals and a sample of irradiated lunar dust show no large stored energy peaks below 200°C. However the temperature of irradiation was unknown for the terrestrial minerals.

5) Both bulk fines and core tube samples show measurable thermoluminescence curves. The output is very low at the top of the core but increases rapidly with increasing depth.

6) Feldspar, pyroxene, and olivine crystals removed from the bulk fines all contain very large fossil track densities ranging from $>1 \times 10^7$ to $>5 \times 10^8/cm^2$.

7) In laboratory heating experiments, the feldspars have the tracks almost completely erased at $\lesssim$ 550°C. (30 min. heating) Olivine recovers completely in 30 min. at $\sim$ 450°C.

8) Six crystals, chosen at random from the core sample at 12 cm, have sharp diffraction patterns and show no evidence of extensive lattice disorder.

Based on these experimental observations, we have reached the following conclusions:

1) Based on our current knowledge of the radiation history of the lunar surface, there is no pressing theoretical reason to expect a large energy release.

2) None of our measurements, including those made as a function of depth in the core tubes, show a hazzardous release of stored energy.

3) Thermo-luminescence measurements show that the core tube is a true vertical section of the moon's surface.

4) The thermoluminescence and D.T.A. measurements show that the lunar samples have not been heated substantially above about 250°C in recent times.

5) We cannot exclude the possibility that the samples were heated by the rocket exhaust and that there was a large stored energy release below 250°C.

6) However, for a variety of reasons stated in the report we consider the existence of such a hypothetical energy release below 250°C. as unlikely.

7) In any event it may be concluded that even if such a hypothetical energy release occurred it did not heat the material substantially above 250°C.

Given these observations it is our opinion that the danger from a catastrophic release of stored energy is slight.

Figure Captions

Fig. 1: DTA run on as-received lunar fines. See text for description of this technique. The total temperature span is 800°C. and the sensitivity in ΔT is 0.5°C/in.

Fig. 2: Second DTA run on same sample shown in Fig. 1. The broad peak seen between 450°C and 710°C in Fig. 1 is no longer present.

Fig. 3: High sensitivity (ΔT=0.2°C/in) DTA run on sample of irradiated terrestrial hypersthene. Total temperature span is 800°C.

Fig. 4: DTA run on a non-irradiated control sample of hypersthene shown after irradiation in Fig. 3.

Fig. 5: DTA run on as-received sample of lunar fines. The total temperature span is 500°C and the sensitivity in ΔT is 0.2°C/in. The difference between the first run (top) and the re-heat curve (bottom) indicates a broad exothermic peak between 275°C and 450°C.

Fig. 6-10: DTA runs on core tube samples taken respectively from top, 3 cm, 6 cm, 9 cm, and 12 cm, The total temperature span is 500°C and the sensitivity in ΔT is 0.2°C/in. The top sample shows little or no energy release while the 12 cm sample shows the largest energy release. The sample from the 6 cm level is quite different from all the others.

Fig. 11: DTA run on second sample from the 6 cm level. Total temperature span is 800°C and the sensitivity in ΔT is o.2°C/in. This run confirms the unusual nature of the 6 cm sample and suggests that there may be a discontinuity in the core tube at this level.

Fig. 12: DTA run on terrestrial hypersthene irradiated with 3.7 x 10^{15} p/cm^2 of 30 MeV α-particles. Temperature span 500°C and sensitivity 0.2°C/in. Irradiation temperature unknown.

Fig. 13: DTA run on terrestrial feldspar irradiated with p/cm^2 of 30 MeV α-particles. Temperature span 500°C and sensitivity 0.2°C/in. Irradiation temperature unknown.

Fig. 14: DTA run on lunar dust irradiated with 2×10^{14} p/cm^2 of 30 MeV. α-particles. Temperature span 500°C sensitivity 0.2°C/in. Irradiation temperature < 90 °C.

Fig. 15: Thermoluminescence curves of lunar fine materials. The curves marked A and B are natural glow curves. The curve A is a sample of the lunar bulk fines and B is from the 12 cm level of the drill core. Both these curves lie well above the background curve d which shows the output on a second heating of the lunar material. Curve C shows a sample of lunar fines after a one hour irradiation with a 1 curie Sr^{90} source. The abrupt break above 300°C is caused by a change of scales for the natural samples.

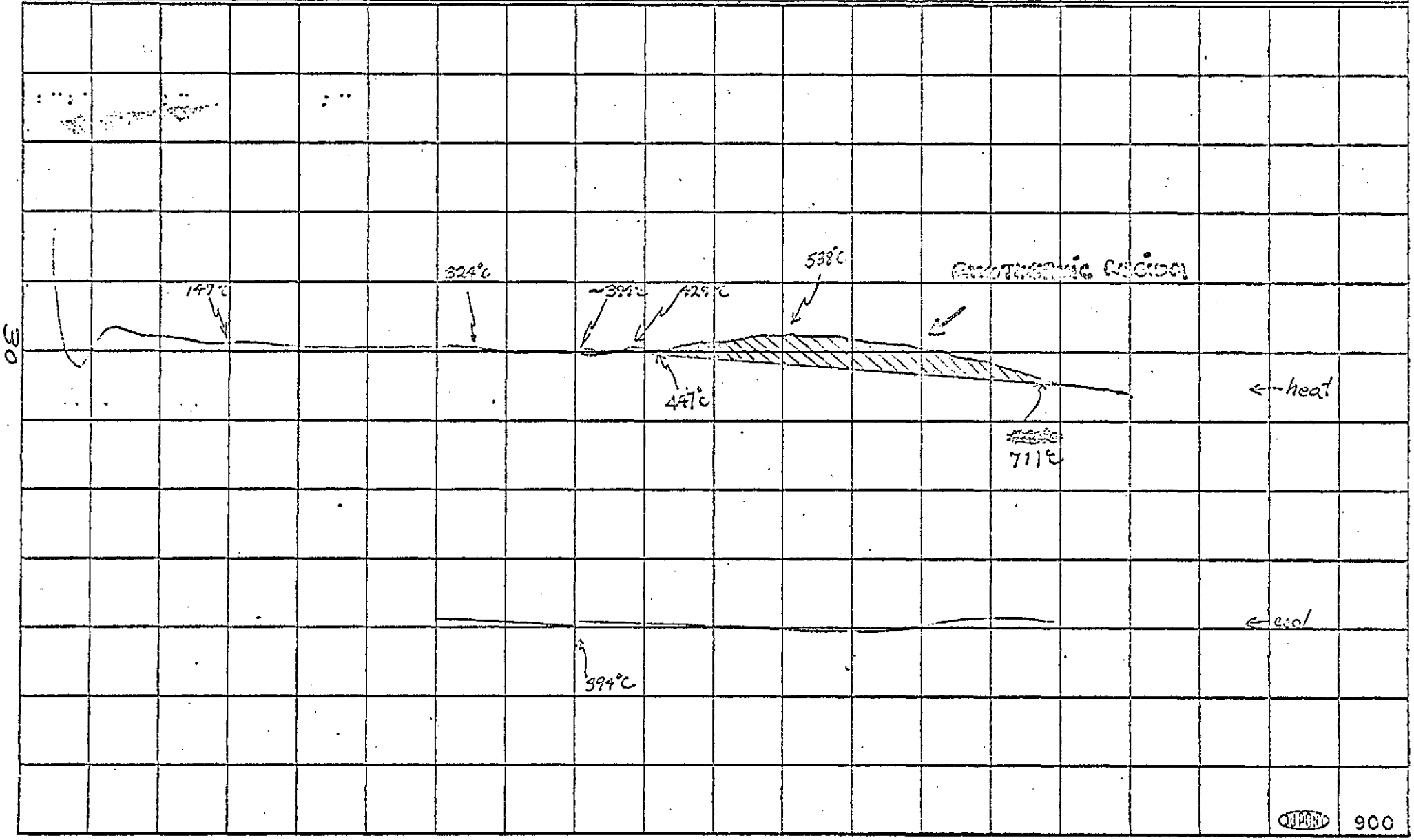
Fig. 16: Thermoluminescence curves of core tube samples. There is a striking increase in output as the depth increases. The 6 cm sample appears anomalous indicating possible structure in the core.

Fig. 17: Etched fossil nuclear tracks in lunar feldspar as seen in a stereo-scan electron microscope. Comparison with optical microscopy confirms that these pits are caused by nuclear particle tracks. The magnification is ~5000 and the track density is $\sim 1.5 \times 10^7$/cm^2. Most grains from the bulk fines contain higher track densities. These tracks would not be visible if the sample had been severely heated in recent times.

Fig. 18: X-ray photograph of one of six crystals picked at random from core tube sample at 12 cm. All six crystals gave sharp diffraction patterns of the type shown here indicating that they were not severely disordered.

FIGURE 6018

SAMPLE: DTA-1 from B-11 (Fines)	SIZE 2mm - 13.6 mg.	ATM. Air 1 atm MM	RUN NO. L-1
	REF. Al_2O_3	T / ΔT	DATE 9/24/69
	PROG. MODE Heat	SCALE 100 °C/IN. / 0.5 °C/IN.	OPERATOR J. L. Kaufer
ORIGIN: Lunar Soil - 3-1	RATE 30 °C/MIN, START Rm °C	SHIFT 0 IN. / IN.	BASE LINE SLOPE 0

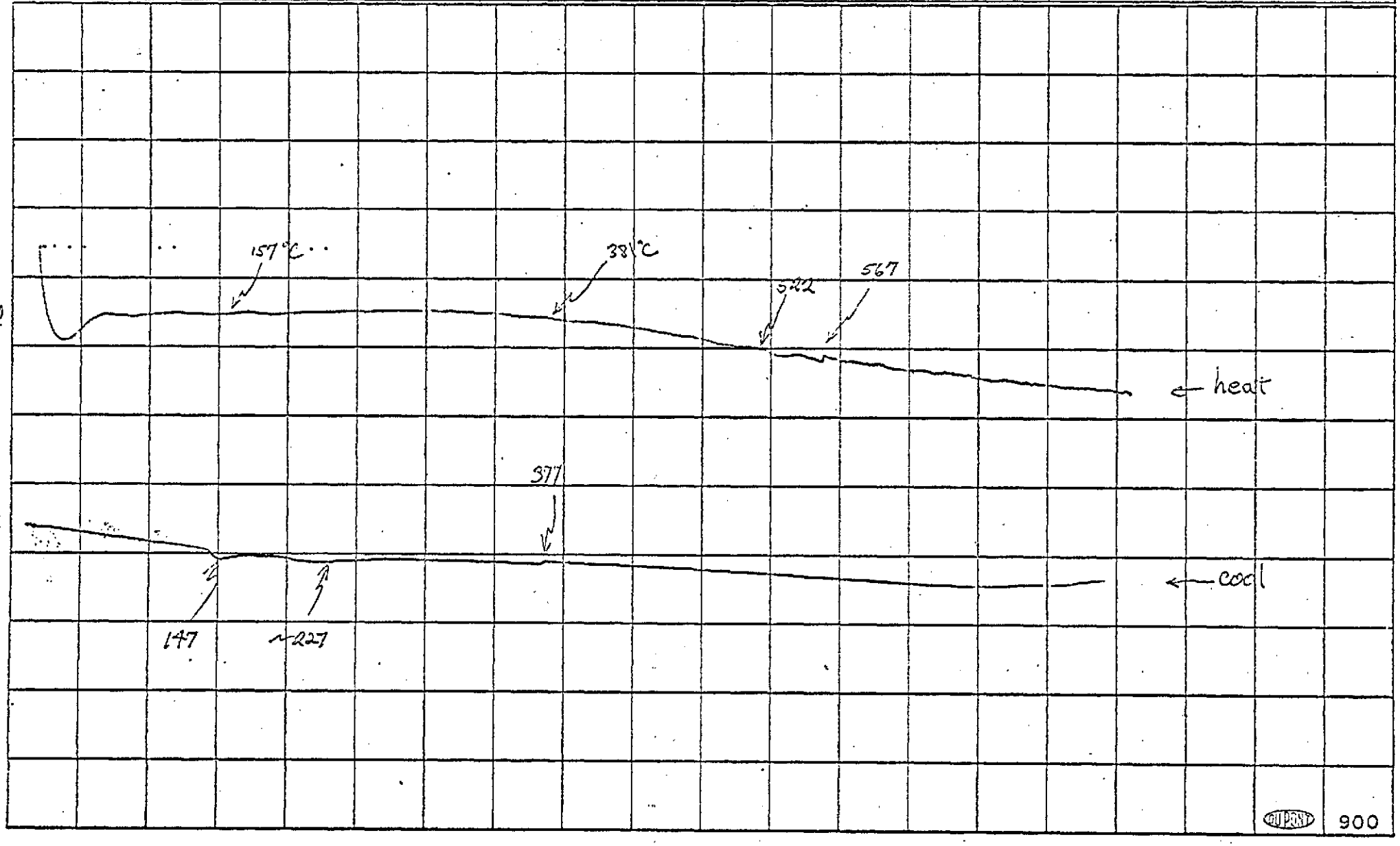


SAMPLE: DTA-1 (Fines) RUN NO.: L-1

JUPON 900

FIGURE T-103

SAMPLE: DTA-1 from B-11 Reheat of DTA L-1	SIZE 2mm - 13.6 mg	ATM. air, 1 atm MM	RUN NO. L-2
	REF. Al_2O_3	T / ΔT	DATE 4/25/69
	PROG. MODE Heat	SCALE 100 °/in / 0.5 °/in	OPERATOR J. L. Kardos
ORIGIN: Lunar Fines - S-1	RATE 30 °/min, START Rm °C	SHIFT 0 IN. / 0 IN.	BASE LINE SLOPE 0

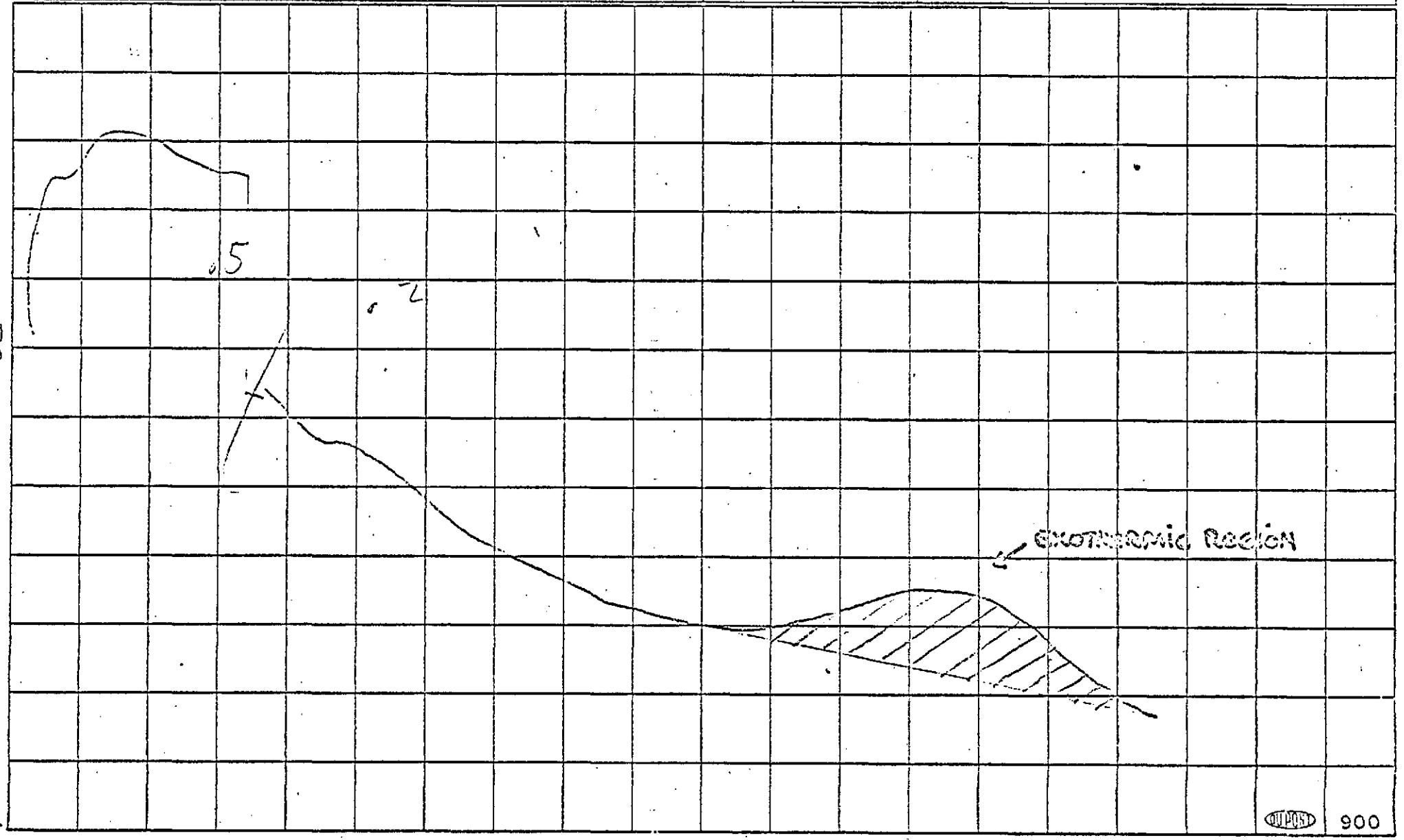


DUPONT 900

SAMPLE: DTA-1 RUN NO.: L-2

FIG. 3

SAMPLE: Hyperchromic IF	SIZE 10.3 mg	ATM. 955 Torr MM	RUN NO. 216
rad. dose – 3.7×10^{15} e/cm²	REF. Al_2O_3	T / ΔT	DATE 9-12-64
ORIGIN:	PROG. MODE Heat	SCALE 100 °C/IN. / 0.2 °C/IN.	OPERATOR RLC
	RATE 30 °C/MIN. START 10 °C	SHIFT 0 IN. / ½ IN.	BASE LINE SLOPE 0



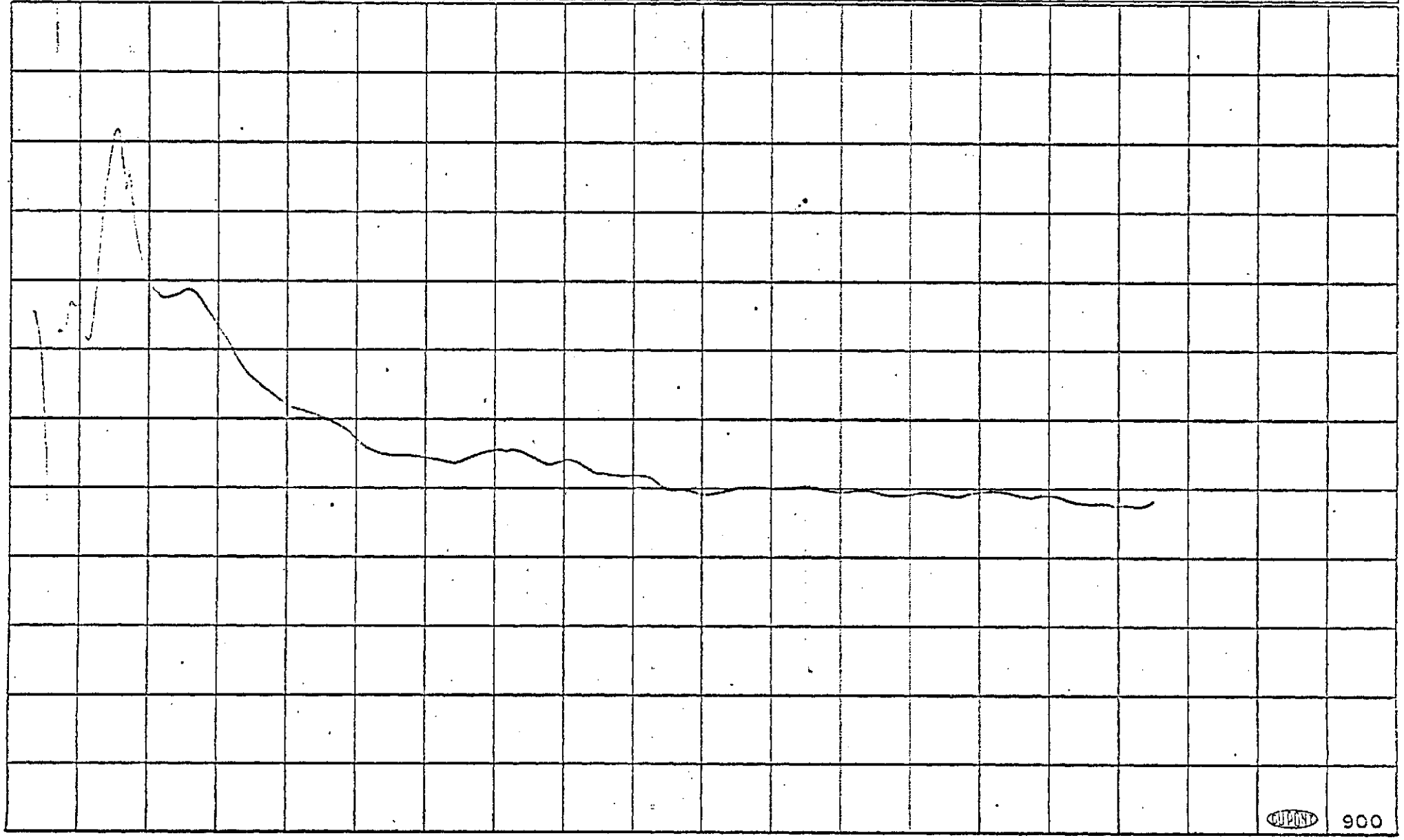
32

SAMPLE: RUN NO.:

T °C (CHROMEL-ALUMEL)

FIG. 4

SAMPLE: Hyperthane F non-irradiated	SIZE 10.3 mg	ATM. 1000 /	MM	RUN NO. 213	
	REF. Al O₃		T	ΔT	DATE 9-12-69
	PROG. MODE Heat	SCALE	100 °C/IN.	0.5 °C/IN.	OPERATOR [illegible]
ORIGIN:	RATE 30 °C/MIN, START 20 °C	SHIFT	0 IN.	1/2 IN.	BASE LINE SLOPE 0

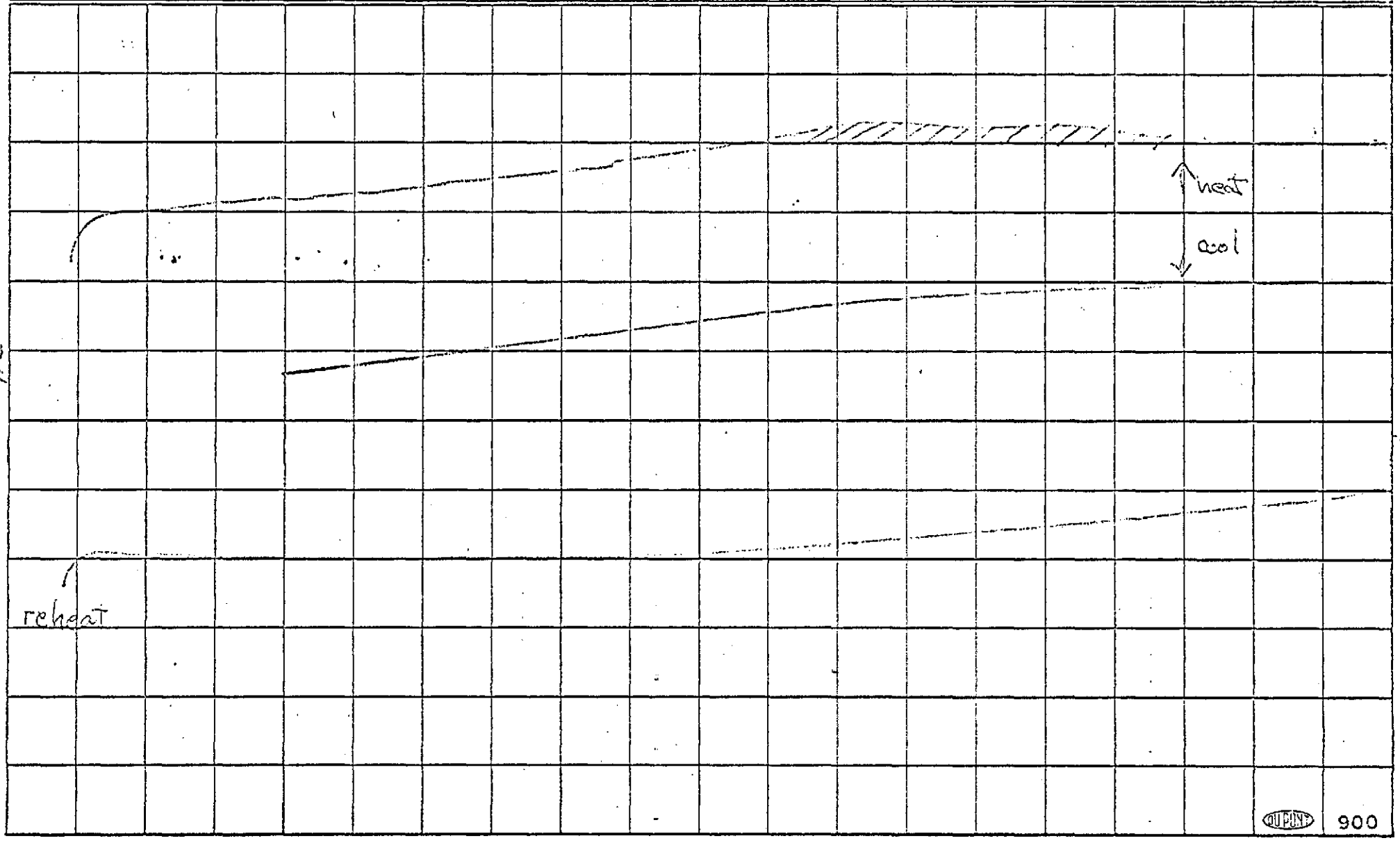


T °C (DUPONT) (UNITS)*

INSTRUMENT PRODUCTS DIVISION

FIG. 5

SAMPLE: DTA-3 from B-11	SIZE 2mm, 13.3mg	ATM. air, 1 atm. MM	RUN NO. L-4
	REF. glass beads	T / ΔT	DATE 10/23/69
	PROG. MODE Heat	SCALE 50 / 0.2 °C/IN. / 0.2 °C/IN.	OPERATOR J. L. Kaufman
ORIGIN: Lunar Fines, S-1	RATE 20 °C/MIN, START 24 °C (547)	SHIFT 0 IN. / 0 IN.	BASE LINE SLOPE 0

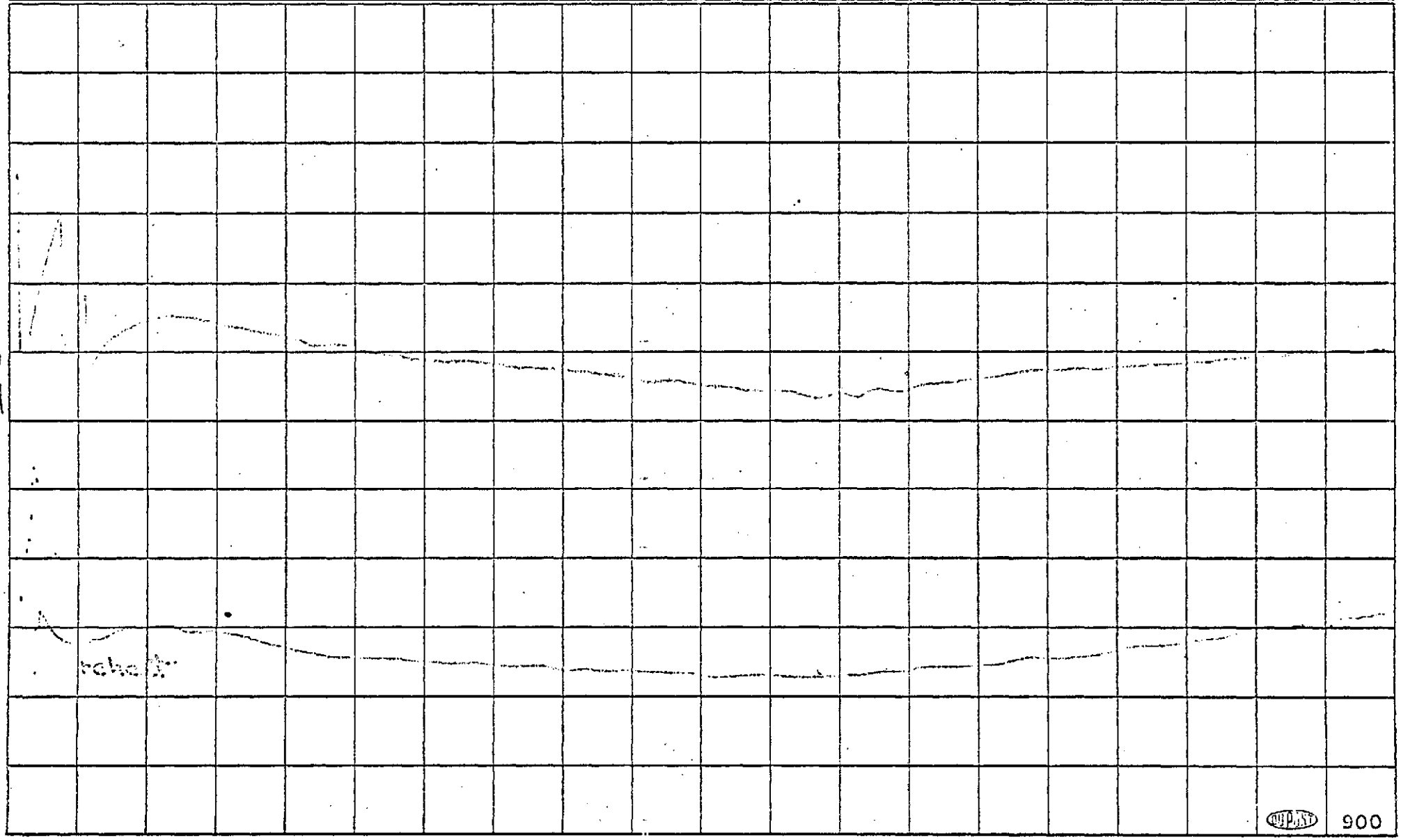


SAMPLE: RUN NO.:

DUPONT 900

F16.6

SAMPLE: DTA-4 Top of core (fines) Fines from ORIGIN: Lunar Core Sample	SIZE 2mm dia. 12.2 mg.	ATM. air . 1 atm MM	RUN NO. L-5
	REF. glass beads	T / ΔT	DATE 10/22/69
	PROG. MODE Heat	SCALE 50 °C/IN. 0.2 °C/IN.	OPERATOR J. L. Kaufos
	RATE 20 °C/MIN. START °C	SHIFT 0 IN. — IN.	BASE LINE SLOPE 0

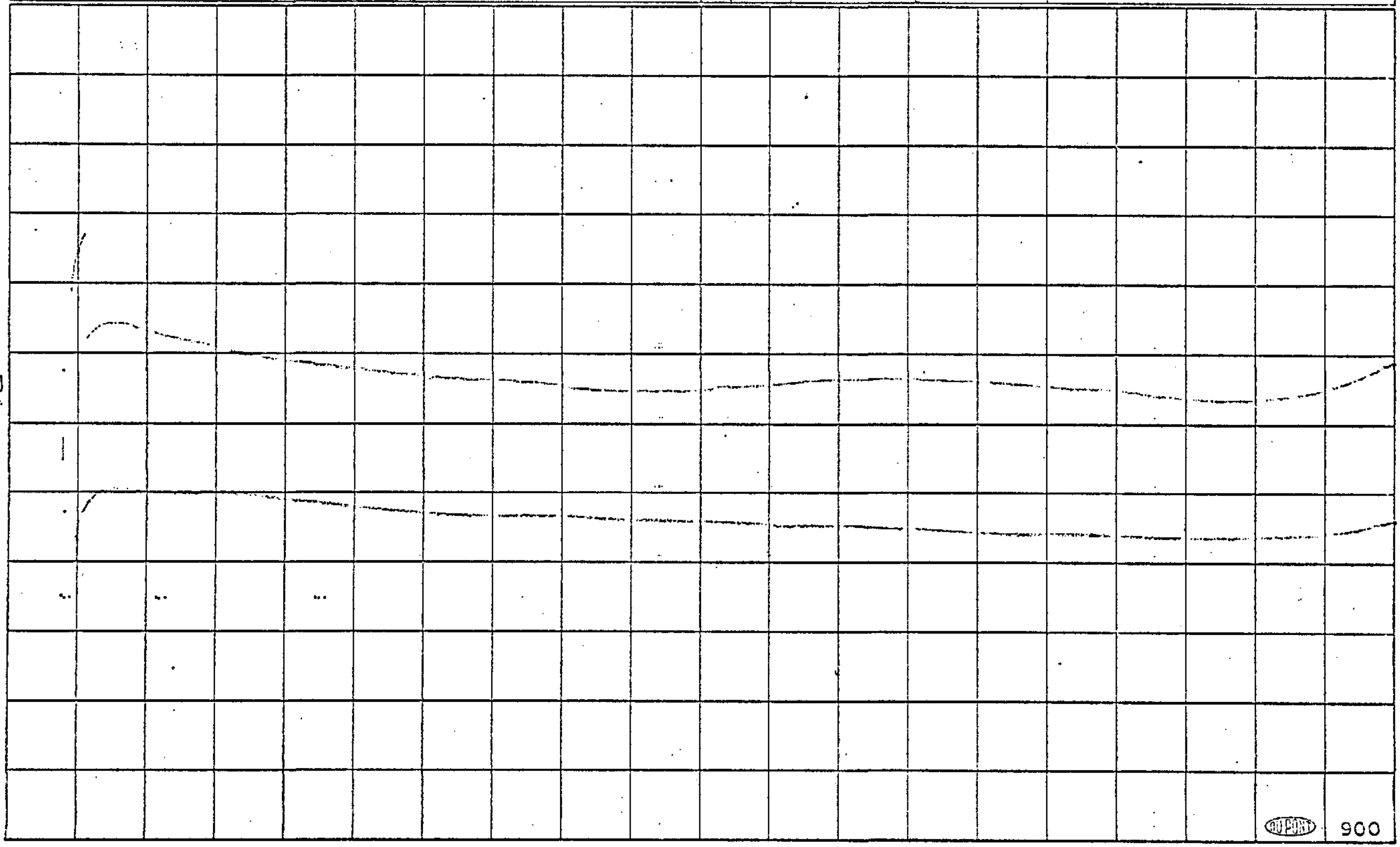


T °C (CHROMEL - ALUMEL)

FIG. 9

SAMPLE: DTA-10 1080-7, 22 3 cm from top	SIZE 2mm 12.8 mg. REF. glass beads PROG. MODE Heat	ATM. air, latm. MM	RUN NO. 2-8 DATE 10/30/69 OPERATOR
ORIGIN: Luna Core Sample	RATE 20 °C/MIN, START RM °C	SCALE T: 50 °C/IN., ΔT: 0.2 °C/IN. SHIFT T: 0 IN., ΔT: — IN.	BASE LINE SLOPE 0

36



SAMPLE: DTA-10 RUN NO.: 2-8

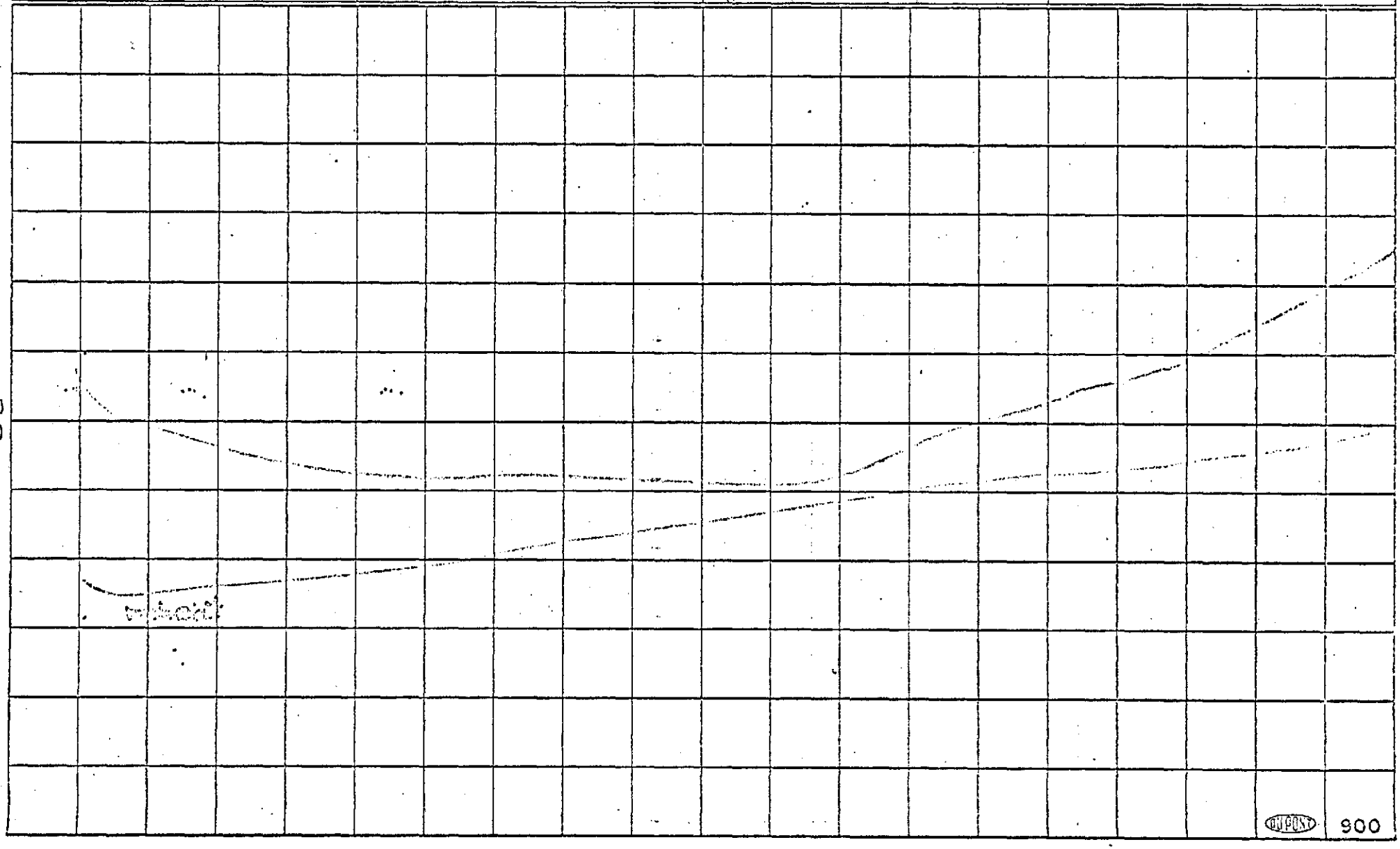
900

T °C (CHROMEL ALUMEL)*

DUPONT INSTRUMENT PRODUCTS DIVISION

FIG. 8

SAMPLE: DTA-8 6 cm from top of core 10004,23 ORIGIN: Lunar Core Sample	SIZE 2mm 13.0 mg REF. glass beads PROG. MODE Heat RATE 20 °C/MIN. START RM °C	ATM. air 1 atm. MM T / ΔT SCALE 50 °C/IN. 0.2 °C/IN. SHIFT 0 IN. — IN.	RUN NO. L-7 DATE 10/24/69 OPERATOR J. V. Kaiser, R. E. Lowe BASE LINE SLOPE 0

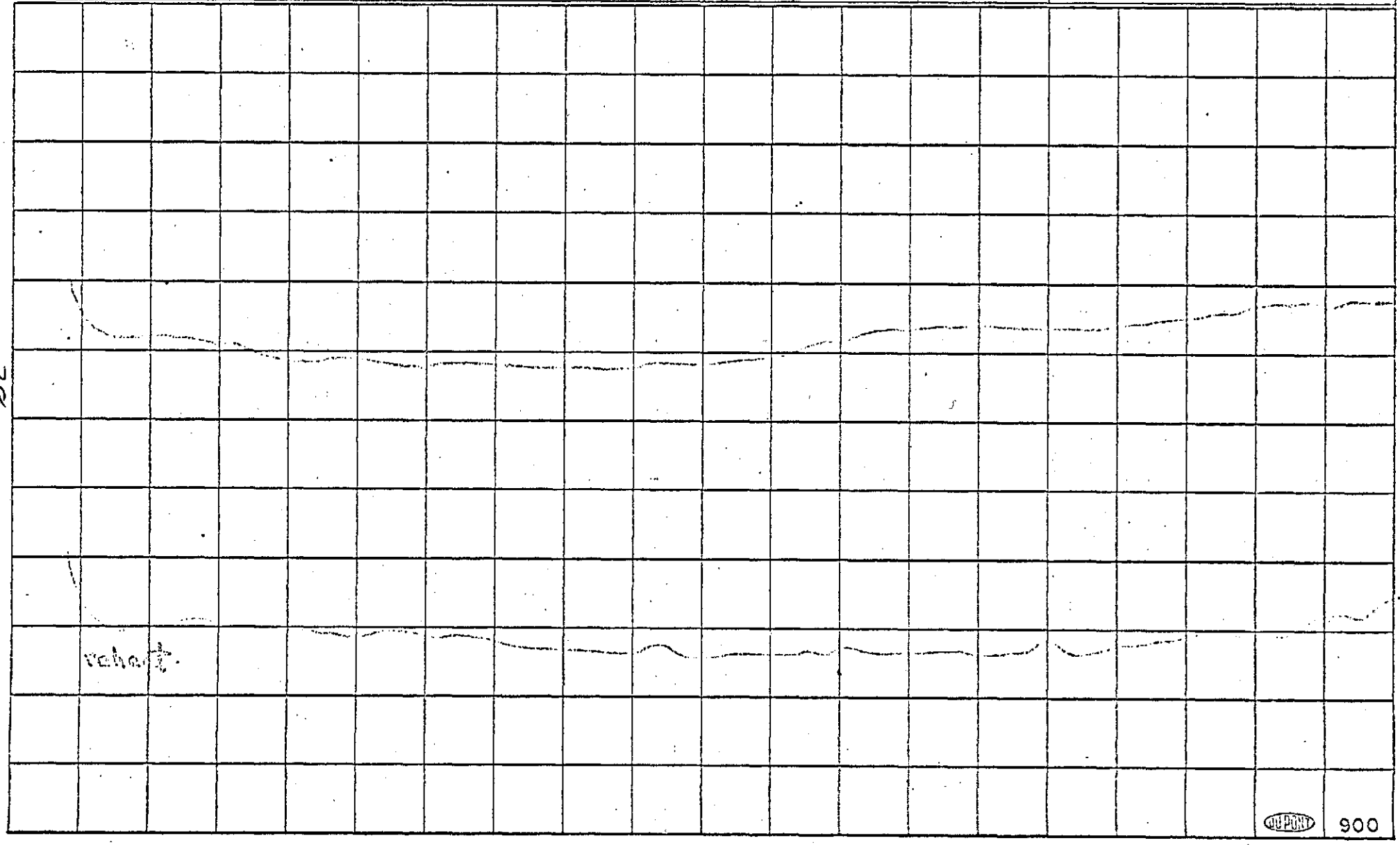


SAMPLE: _____ RUN NO.: _____

DUPONT 900

FIG. 9

SAMPLE: DTA-6 9cm from top of core	SIZE 2mm 13.6 mg	ATM. air 1 atm MM	RUN NO. L-6
	REF. glass beads	T / ΔT	DATE 10/24/69
	PROG. MODE Heat	SCALE 50 in. / 0.2 in.	OPERATOR J. L. Kardos
ORIGIN: Lunar Core Sample	RATE 20 °C/min, START 22 °C	SHIFT 0 IN. / — IN.	BASE LINE SLOPE 0



SAMPLE: _____ RUN NO.: _____

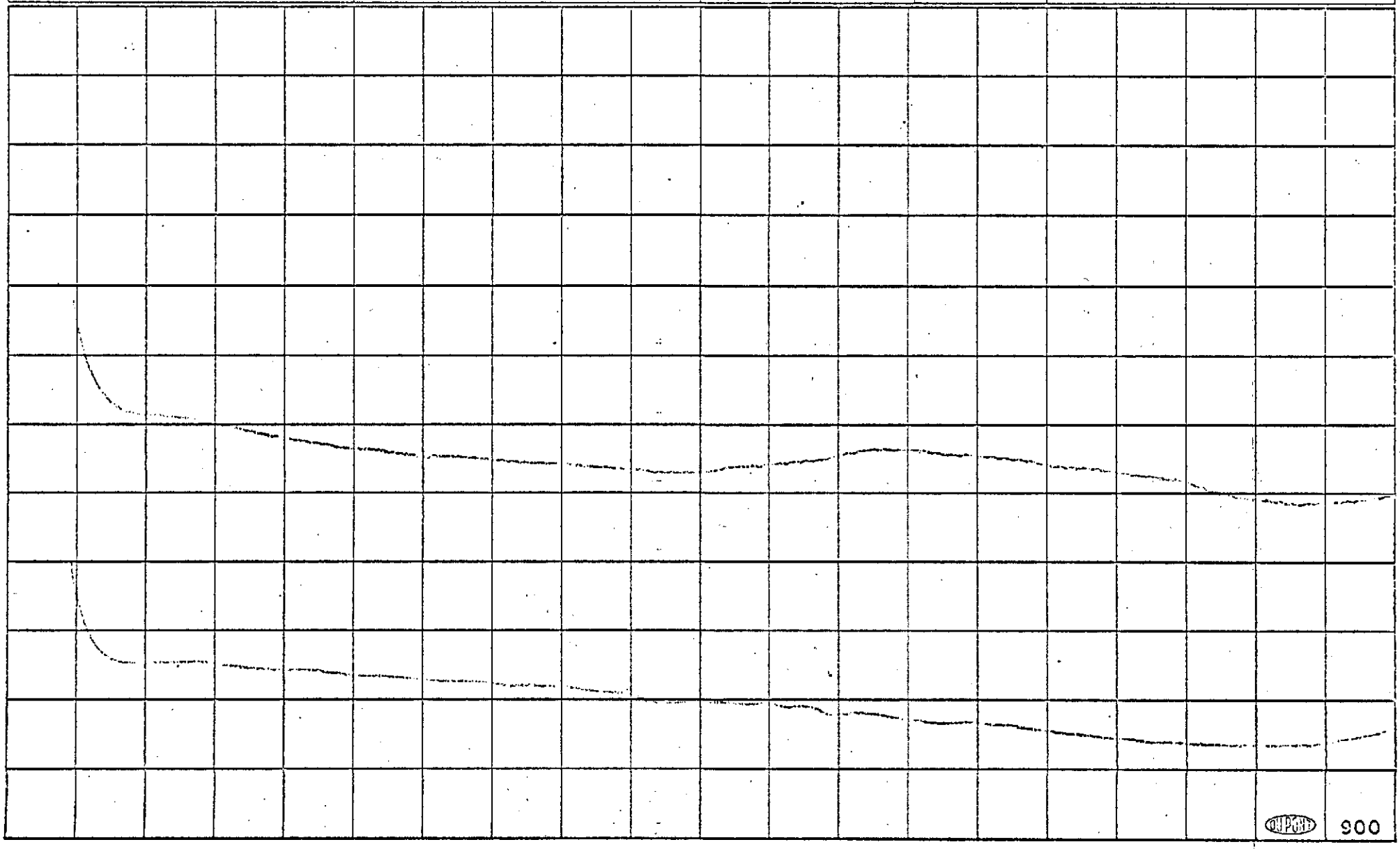
38

T °C (CHROMEL - ALUMEL)*

INSTRUMENT PRODUCTS DIVISION

FIG. 10

SAMPLE: DTA-12 10004,25 12cm from top	SIZE 2mm 13.9mg	ATM. air . 1atm. MM	RUN NO. L-9
	REF. glass beads	T / ΔT	DATE 10/25/69
	PROG. MODE Heat	SCALE 50 ° IN / 0.2 ° IN	OPERATOR J. L. Haules
ORIGIN: LUNAR CORE SAMPLE	RATE 20 °/MIN. START RM °C	SHIFT 0 IN. / — IN.	BASE LINE SLOPE 0



SAMPLE: DTA-12 RUN NO.: L-9

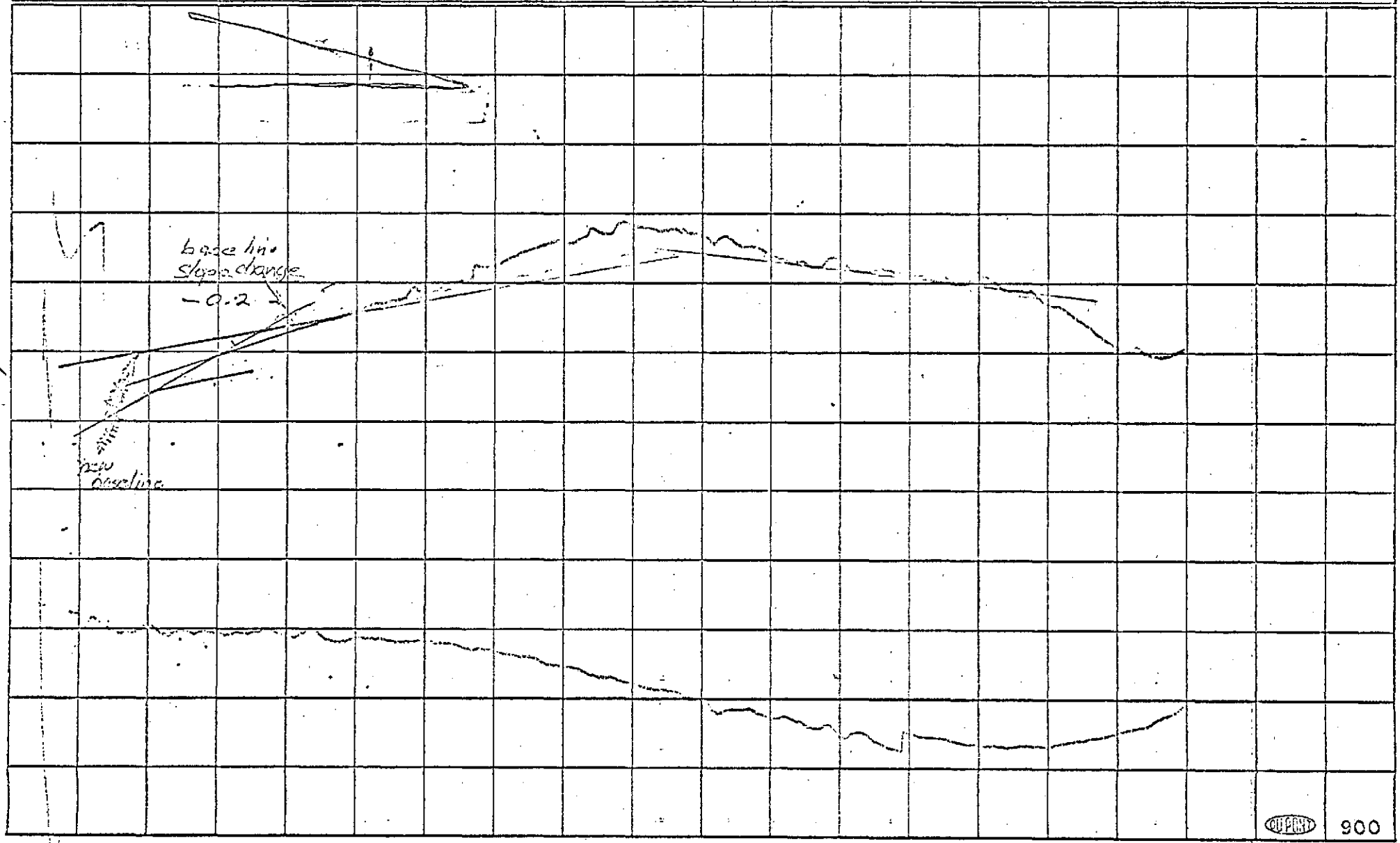
39

T °C (CHROMEL - ALUMEL)*

DUPONT 900

FIG. 11

SAMPLE: DTA-9 6 cm. from Top of core	SIZE 2mm 12.6 mg.	ATM. air 1 atm MM	RUN NO. L-10	
	REF. Al_2O_3	T / ΔT	DATE 10/26/69	
	PROG. MODE Heat	SCALE 100 °C/IN. 0.2 °C/IN.	OPERATOR J. L. Kendes	
ORIGIN: Lunar Core Sample 10004.23	RATE 20 °C/MIN, START RM °C	SHIFT 0 IN. — IN.	BASE LINE SLOPE 0.-0.2	



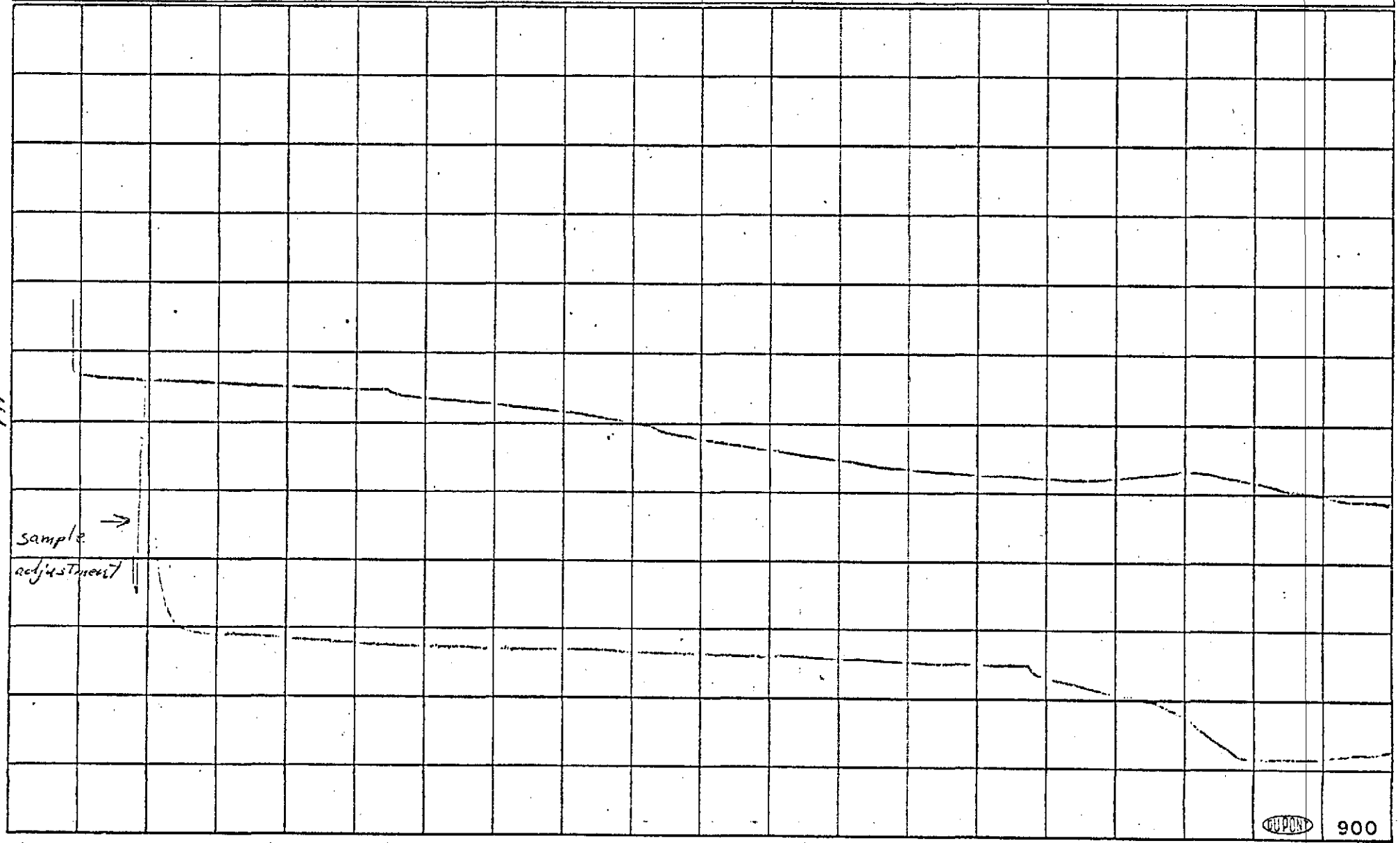
SAMPLE: DTA-9 RUN NO.: L-10

T °C (CHROMEL - ALUMEL)

900

FIG. 12

SAMPLE: Hypersthene Irradiated 3.7×10^{15} α/cm² DTA-14	SIZE 2mm 14.0 mg	ATM. air 1 atm MM	RUN NO. L-11
	REF. glass beads	T / ΔT	DATE 10/27/69
	PROG. MODE Heat	SCALE 50 °C/IN. / 0.2 °C/IN.	OPERATOR J. L. Kaufee
ORIGIN: M. Seitz	RATE 20 °C/MIN, START RM °C	SHIFT 0 IN. / — IN.	BASE LINE SLOPE 0



DUPONT 900

T °C (CHROMEL - ALUMEL)*

SAMPLE: Hypersthene RUN NO.: L-11

FIG. 13

SAMPLE: DTA-15
irradiated Labradorite B
dose – 3.7×10^{15} α/cm²

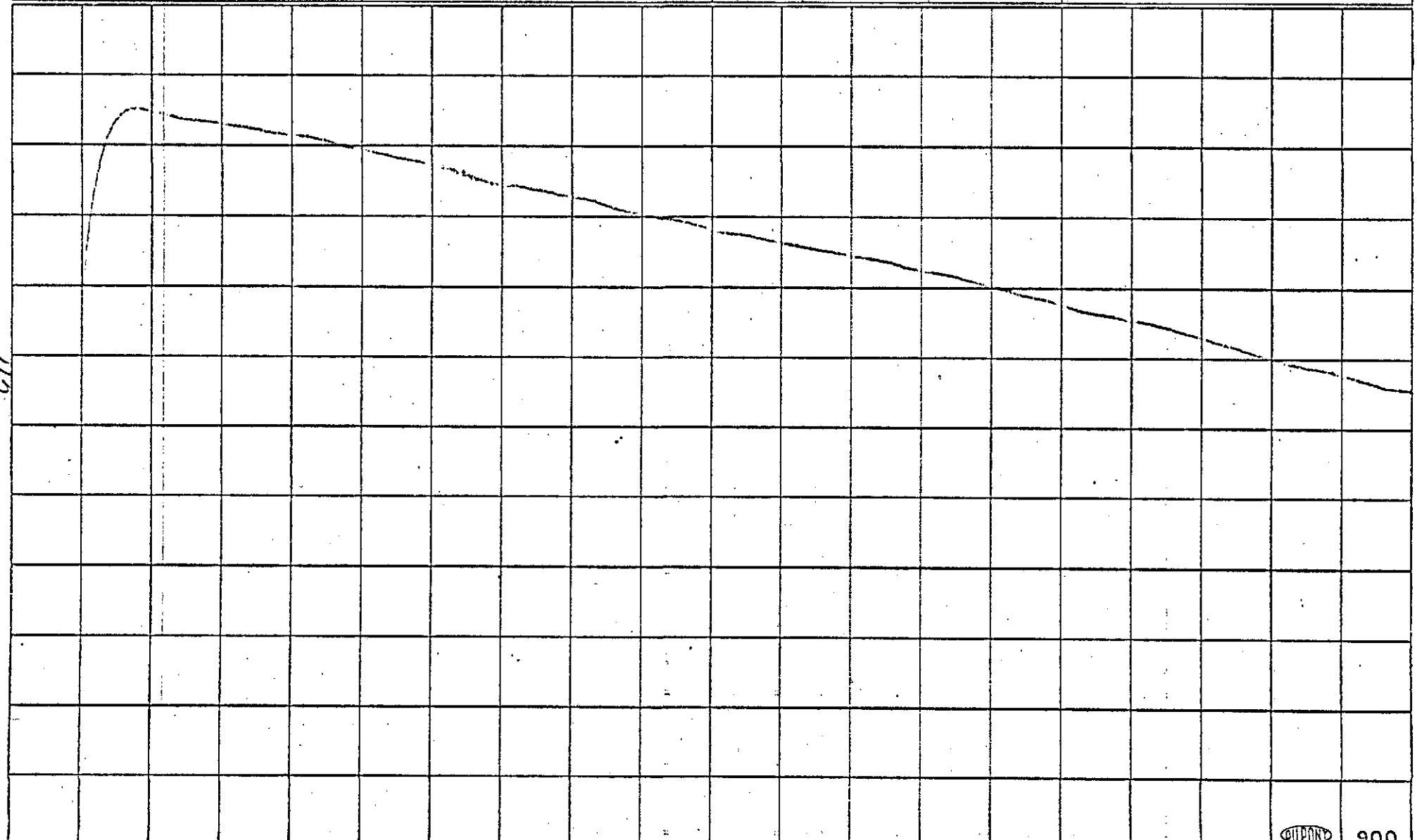
ORIGIN: M. Seitz

SIZE 2mm 14.0mg
REF. glass beads
PROG. MODE Heat
RATE 20 °C/MIN, START RM °C

ATM. air, 1 atm MM

	T	ΔT
SCALE	50 °C/IN.	0.2 °C/IN.
SHIFT	0 IN.	— IN.

RUN NO. L-12
DATE 10/27/69
OPERATOR J. L. Kaufee
BASE LINE SLOPE 0



SAMPLE: _____ RUN NO.: _____

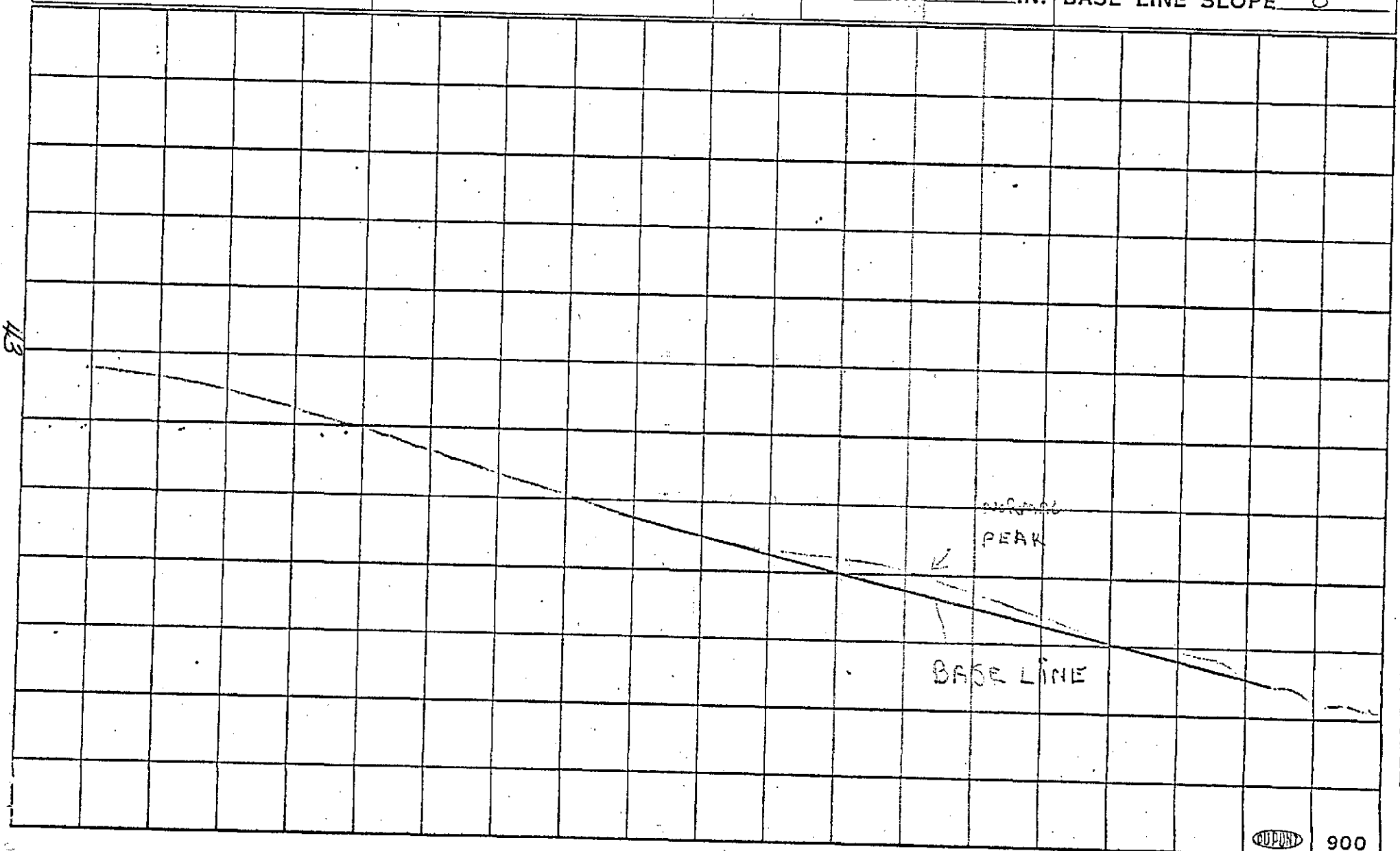
DUPONT 900

T °C (CHROMEL - ALUMEL)*

DUPONT INSTRUMENT PRODUCTS DIVISION

FIG. 14

SAMPLE: DTA-17 irradiated Lunar fines	SIZE 2mm 11.6 mg	ATM. air 1 atm MM	RUN NO. L-14
	REF. glass beads	T / ΔT	DATE 10/27/69
	PROG. MODE Heat	SCALE 50 °C/IN. 0.2 °C/IN.	OPERATOR J-L. Kardos
ORIGIN:	RATE 20 °C/MIN, START RM °C	SHIFT 0 IN. — IN.	BASE LINE SLOPE 0



SAMPLE: _____ RUN NO.: _____

DUPONT 900

FIG. 15

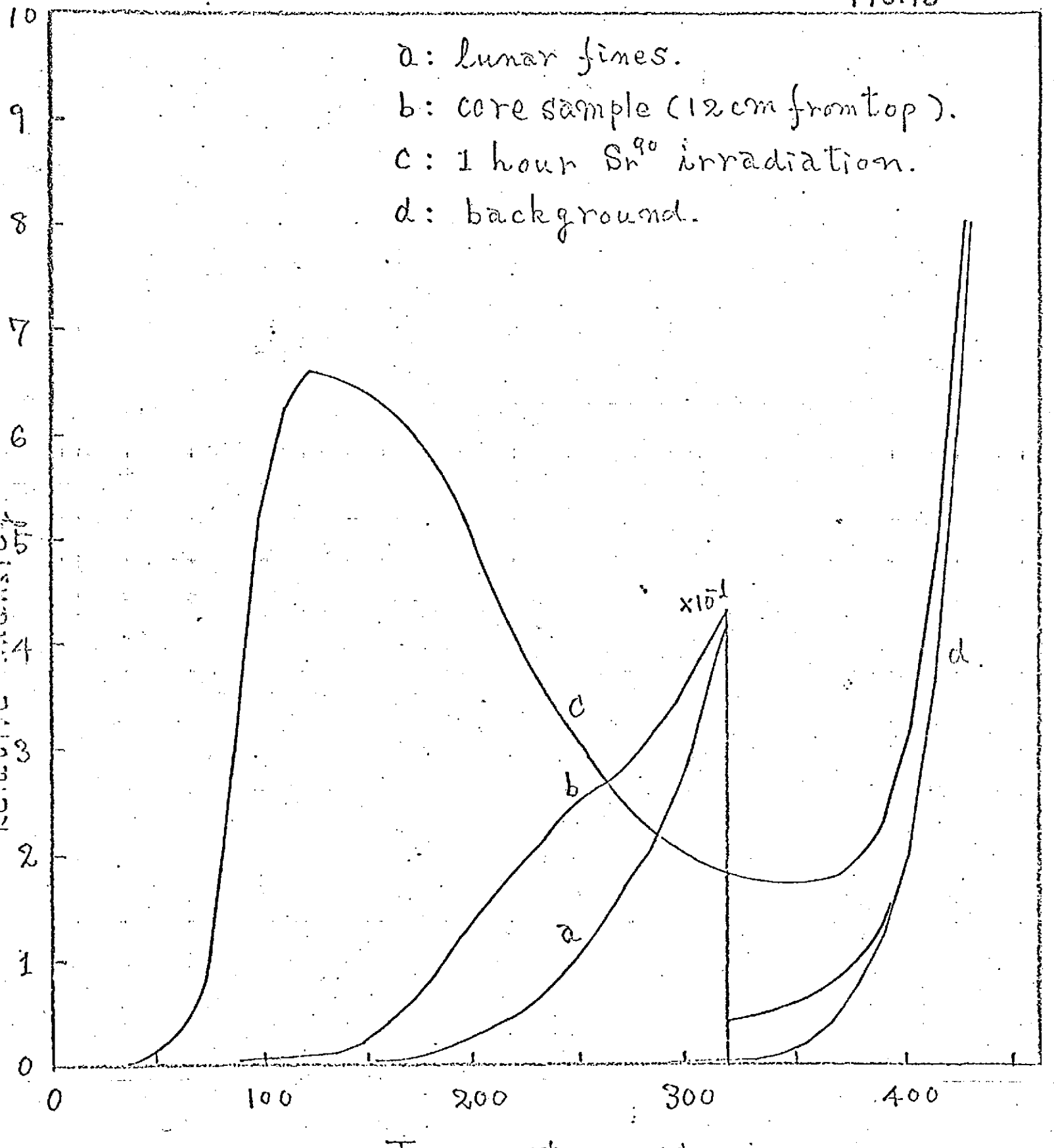

a: lunar fines.
b: core sample (12 cm from top).
c: 1 hour Sr^{90} irradiation.
d: background.
$\times 10^{-1}$
a
b
c
d
0
1
2
3
4
5
6
7
8
9
10
0
100
200
300
400
Temperature °C

FIG. 16

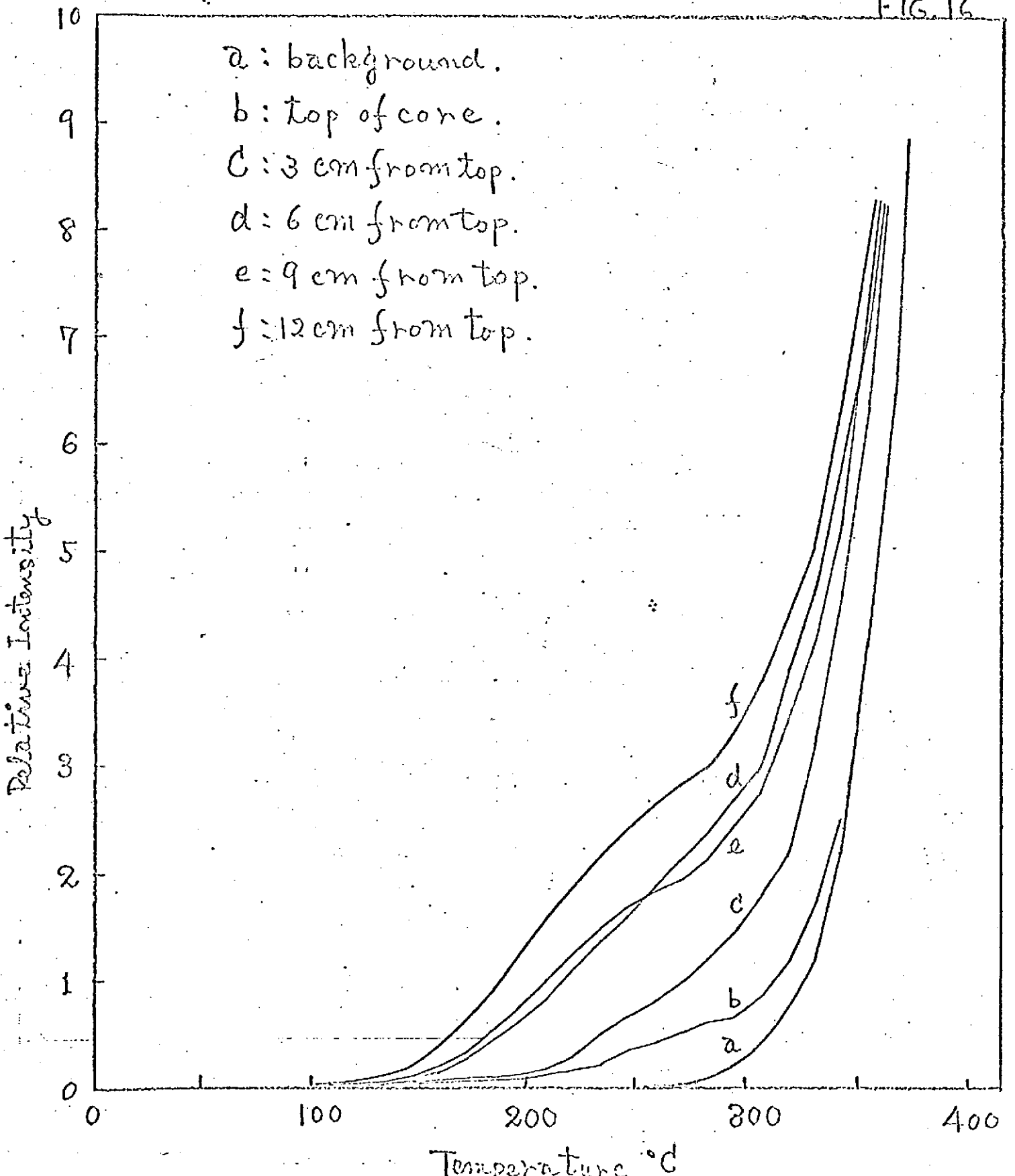

a : background.
b : top of core.
C : 3 cm from top.
d : 6 cm from top.
e : 9 cm from top.
f : 12 cm from top.
Relative Intensity
Temperature °C
0
1
2
3
4
5
6
7
8
9
10
100
200
300
400
a
b
c
d
e
f

FIG. 17

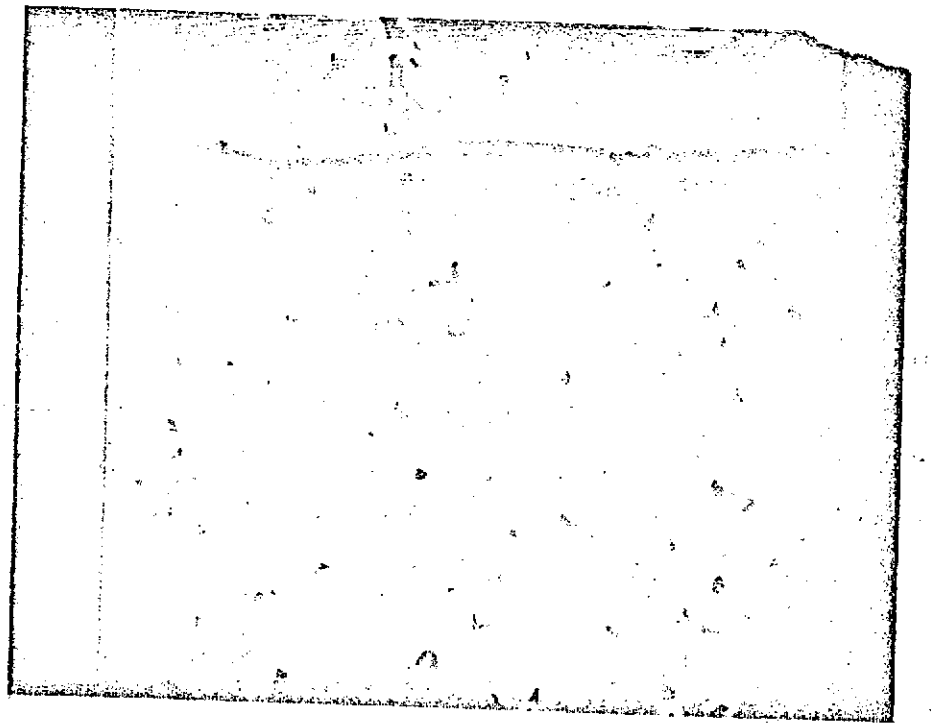


FIG. 18