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THE PREDICTION OF THERMAL HAZARDS IN PROPELLANTS BY A NOMOGRAPHICAL TECHNIQUE

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ABSTRACT. A nomographic technique developed for predicting thermal hazards in explosives was applied to double-base and cast composite propellants. Chemical and physical property data, together with differential thermal analysis or an adiabatic auto-ignition determination on the material under investigation, are required in order to solve the nomographs. Predicted time to deflagration for representative propellants was compared with experimental data obtained in cook-off tests. The need for improved chemical and physical property data, extension of the nomographs to account for changes occurring with time and temperature and more cook-off data on larger masses of propellant was indicated. This work is continuing and reference is made to NavWeps Report Nos. 7769 (U) and 7775 (C).

U.S. NAVAL ORDNANCE TEST STATION
China Lake, California
9 October 1961

U. S. NAVAL ORDNANCE TEST STATION

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FOREWORD

During the period January-June 1961, an attempt was made to apply the nomographical technique for rapidly predicting thermal hazards in explosives and propellant systems. The nomographical technique was developed by Professors Paul A. Longwell and Frank A. Conrad, Station Consultants, in conjunction with Dr. C. D. Lind of the Naval Ordnance Test Station.

This first attempt demonstrated the value of the nomographs, but also pointed out some limitations in attempting to predict safe-life of large-sized propulsion masses. This initial effort is being expanded and results will be published in subsequent reports. The work was performed in the Product Evaluation Branch of the Propulsion Development Department under Bureau of Naval Weapons Special Projects No. 71401-1.

This report is transmitted for information only and does not represent the official views or final judgment of this Station. It presents information released at the working level that is still subject to modification and withdrawal. This report was reviewed for technical accuracy by Drs. C. M. Anderson, and C. D. Lind.

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INTRODUCTION

The accurate prediction of the time-to-deflagration or the time-to-explosion under a given thermal environment is essential in the testing, processing, handling, and storage of propellants. In the past, extra precautions were taken in any operations area where a thermal hazard was known or suspected to exist. As advances, present and future, are made in the state-of-the-art it becomes apparent that new methods are desirable for the prediction of thermal hazards especially for the growing number of new and exotic propellants.

The application to propellant systems of a rapid method for the prediction of thermal hazards in explosive systems developed by P. A. Longwell (Ref. 1), is one approach to this problem and is the subject of this report. Longwell's report gives the background, development, and operation of a nomographical technique for solving the basic rate equations leading to a time-to-explosion number. C. D. Lind (Ref. 2) reports the use of the technique with high explosives and also discusses the interpretation and significance of the results. Since each nomograph can indicate but one additive operation, a sequence of operations through a series of nomographs is required to solve each rate equation. The nomographs are reproduced from NavWeps Report 7646 (Ref. 1); Fig. Nos. 1, 2, and 3 are the nomographs. The required sequence of operations is given in these figures.

A list of the symbols and their respective definitions as used in the nomographs and in this report follows:

a = radius or half thickness, cm

ρ = density, g/cm^3

Q = heat of decomposition, cal/g

Z = collision frequency, sec^{-1}

E = activation energy, cal/mole

K = specific rate constant

λ = thermal conductivity, cal/°K cm sec

R = gas constant, 1.987, cal/mole °K

α = axes on nomographs

β = axes on nomographs

δ = shape factor = $\frac{\rho Q Z E a^2}{\lambda R T_o^2} \cdot e^{-E/RT_o}$

T_o = initial temperature of the propellant

T_m = critical temperature, °C

E/T^* = nomographical operator, cal/mole°K

c = heat capacity, cal/g °K

T_1 = surface temperature, °C

$T^* = \frac{1}{(1/T_m - 1/T_1)}$, °C

T_e = time-to-explosion or deflagration

$\frac{\rho Q Z E}{\lambda R}$ = nomographic operator, $(\frac{°K}{cm})^2$

$\frac{\rho c}{\lambda}$ = nomographic operator, sec/cm^2

The following numerical quantities are necessary to enter the nomographs for the complete calculation of critical temperature and time-to-"decomposition":

a, ρ, Q, Z, E, λ, R, c and δ.

These values can be determined by chemical and physical laboratory tests, along with either a differential thermal analysis or an adiabatic auto-ignition determination on the material under investigation.

PURPOSE

The need for theoretically predicting what a propellant will do upon heating becomes obvious when one considers the hazards and expense involved in a large motor. It is reasonable to determine the thermal behavior in propellants experimentally up to about five inches in diameter. However, scaling up these cook-off tests exponentially raises the hazards and expense involved.

Because scaling operations of high energy propellant systems are extremely hazardous, as well as costly; maximum efforts have been directed toward seeking rapid, reliable methods for predicting thermal

hazards. This report utilizes the nomographs of (Ref. 1) and makes a comparison of predicted times-to-deflagration with experimental cook-off data of two typical propellant systems.

MATERIALS

The first comparisons were made using a double base and a cast composite propellant. The two propellants used were X-14 and C-509. Their respective formulations may be found in Solid Propellant Information Agency reports.

EXPERIMENTAL

In predicting thermal decomposition, certain basic parameters must be obtained experimentally. These thermal parameters include heat capacity (c), thermal conductivity (λ), heat of decomposition (Q), the QZ product, and the activation energy (E). Heat capacity (c) and thermal conductivity (λ) are relatively easy to determine with an acceptable degree of accuracy. It should be noted that these two values do not enter into the exponential term of the Arrhenius equation ($k = Ze^{-E/RT}$), hence variations do not affect the final results to the same degree as does the activation energy. The activation energy (E) is in the exponential term and this factor gives the greatest amount of variation in the prediction of thermal decomposition.

This argument holds only if E and QZ are known to the same accuracy. Determinations of E can be more accurate than QZ. Activation energy (the energy barrier opposing a reaction) is generally defined as the additional energy average molecules must acquire for reaction. From the Arrhenius equation:

$$\frac{d \ln k}{dT} = \frac{E}{RT^2}$$

or its integrated form:

$$\ln k = -\frac{E}{RT} + \text{const.}$$

the plot of ln k against 1/T will give a straight line with a slope of -E/R. Thus, activation energy can be determined from rate measurements at two or more temperatures.

Determination of activation energy from adiabatic autoignition.

The basic principle for this determination is derived from the D. A. Frank-Kemenetskii (Ref. 3) relation which is:

$$-\lambda\nabla^2 T + \rho c \frac{\partial T}{\partial t} = \rho Q Z e^{-E/RT}$$

where ∇^2 is the Laplacian operator and definition of the other terms has been previously listed.

Rearranging the equation:

$$\rho c \frac{\partial T}{\partial t} = \lambda\nabla^2 T + \rho Q Z e^{-E/RT}$$

or,

$$\begin{array}{c} \text{self} \\ \text{heating} \end{array} = \begin{array}{c} \text{heat gained} \\ \text{by} \\ \text{conduction} \end{array} + \begin{array}{c} \text{heat generated} \\ \text{by} \\ \text{chemical reaction} \end{array}$$

The adiabatic requirement sets the heat gained by conduction ($\lambda\nabla^2 T$) equal to zero giving the simplified equation:

$$\rho c \frac{dT}{dt} = \rho Q Z e^{-E/RT}$$

and,

$$\frac{dT}{dt} = \frac{QZ}{c} e^{-E/RT}$$

Taking the logarithms of both sides

$$\ln \frac{dT}{dt} = \ln \frac{QZ}{c} - E/RT$$

Thus, plotting $\ln \frac{dT}{dt}$ against $1/T$, a straight line with a slope of $-E/R$ and an intercept of $\ln \frac{QZ}{c}$ is produced. From this determination, activation energy and the quantity $\frac{QZ}{c}$ are obtained. Multiplying $\frac{QZ}{c}$ by the heat capacity (c) gives the term, QZ, which is used in the term $(\frac{\rho QZE}{\lambda R})$ on Nomograph I.

Determination of activation energy by a variable heating rate method.

This method is based upon the fact that in differential thermal analysis, the temperature at which a peak temperature occurs will vary with heating rate in certain kinds of reactions. An expression was derived relating this variation with the kinetics of the reaction. Kissinger (Ref. 4 and 5) develops the complete theory for this type of determination, and Pakulak (Ref. 6) discusses its application to a double-base propellant (X-14). Two basic relations that Kissinger developed are:

$$(1) \quad \frac{d\left(\ln \frac{\phi}{{}'T_m^2}\right)}{d\left(\frac{1}{{}'T_m}\right)} = -\frac{E}{R}$$

$$(2) \quad \frac{E\phi}{R\,{}'T_m^2} = An\,(1 - X)_m^{n-1}\, e^{-E/R\,{}'T_m}$$

Where:

A = frequency factor, sec^{-1} for first order reactions

ϕ = heating rate, °K/sec

${}'T_m$ = temperature at point of maximum reaction, °K

E = activation energy, cal/mole

R = gas constant, 1.987 cal/mole °K

x = fraction reacted

n = order of reaction

From equation (1), plotting $\ln \frac{\phi}{{}'T_m^2}$ against $\frac{1}{{}'T_m}$, a straight line results whose slope is equal to (-E/R). After the activation energy is obtained, the quantity "A" may be calculated from equation (2).

("A", which is often cited in the literature as the term "Z", is called the collision frequency and has the units of sec^{-1} for first order reactions. This "A" or collision frequency should not be confused with the heat generation coefficient term (A) used by chemists in recent reports (Ref. 7, 8, and 9).

In these earlier reports (A) had the units of cal/sec. cm^3 and in the present symbols is defined as the product, ρQZ).

In Ref. 6, Pakulak's calculations indicate an order of reaction of 0.95 to 1.0; and $n(1-x)_n^{n-1}$ is approximately one for all values of n. Thus, equation (2) may be simplified to:

$$\frac{E\phi}{RT_m^2} = A\, e^{-E/RT}$$

It is difficult to say which method is the most reliable since little comparison with experimental data from the two has been made. Adiabatic autoignition, however, does have the advantage of being able to determine the necessary data from one test, and in obtaining the product (QZ) directly; thus eliminating the necessity of a separate determination for Q.

TABLE I. SOURCE OF EXPERIMENTAL DATA REQUIRED FOR NOMOGRAPHIC CALCULATIONS.

Symbol	Units	Propellant X-14	Source of Data	Propellant C-509					
				Test 1	Source of Data	Test 2	Source of Data	Test 3	Source of Data
c	cal/g °K	0.338	1	0.2	4	0.2	4	0.2	4
λ	cal/sec cm °K	0.000527	2	0.00107	4	0.00107	4	0.00107	4
E	cal/mole	59	3	47.42	5	78.08	5	96.98	5
Z	sec^{-1}	3.3×10^{27}	3	--	-	--	-	--	-
Q	cal/g	1154	1	--	-	--	-	--	-
QZ	cal/g sec	--	-	6.07×10^{18}	5	3.59×10^{32}	5	1.42×10^{41}	5

Source of data:

1. SPIA/M2, Unit 550
2. Reference 7
3. Reference 6
4. Code 4558, NOTS
5. Code 4541, NOTS

The question arises as to why the activation energy, which directly affects QZ, varies over such a wide range in adiabatic autoignition tests. Three samples from the same lot and run in the same manner gave these results. Amster (Ref. 7) reports the same with ANP2639AF, which is a similar aluminized polyurethane type of propellant. This situation is difficult to understand but can probably best be answered by the fact that these propellants are extremely autocatalytic in nature. As will be seen later, this difference in activation energy, is somewhat compensated for by QZ and does not greatly affect the prediction of critical temperature. It does have, however, a slightly larger effect on the prediction of time-to-deflagration.

In order that comparison could be made between predicted and experimental results, data were needed closely simulating the actual situation. The data available that fitted this qualification closest were cook-off tests. In a cook-off test, a sample is held at a preset oven temperature, and the time-to-deflagration of a given size and shape of propellant is determined. The following table is a tabulation of cook-off data for various sizes of X-14 and C-509.

TABLE II. EXPERIMENTAL COOK-OFF RESULTS FOR X-14 AND C-509

Size (dia.)	T(°F)	$\frac{1}{T}(°K) \times 10^4$	Time-To-Deflagration (Hrs.)
X-14			
1"	261	24.98	3.7
1"	261	24.98	7.2
1"	254	25.22	12.8
1"	242	25.65	88.0 (up to 252°F and went at 95 hrs.)
5"	226	26.25	8.5
5"	223	26.37	12.2
5"	221	26.44	16.9
5"	220	26.48	19.5
5"	227	26.21	21.1
5"	215	26.68	37.0
C-509			
1"	384	21.33	4.78
1"	370	21.69	19.50 (no cook-off)
1"	370	21.69	10.30
1"	360	21.96	6.77
1"	356	22.07	6.93
1"	350	22.23	9.08
1"	350	22.23	8.13
1"	350	22.23	8.00
5"	360	21.96	18.57
5"	348	22.29	25.08
5"	330	22.79	50.85
5"	300	23.69	240.00 (no cook-off)

RESULTS AND DISCUSSION

The results obtained from the nomographic solutions of the thermodynamic equations for the time-to-"decomposition" predictions of X-14 and C-509 are listed in Table III. All results, both predicted and experimental, are for solid cylinders of various diameters.

TABLE III. PREDICTED DECOMPOSITION RESULTS FOR X-14 and C-509

X-14					C-509				
a Radius cm	T_m °C	T_l °C	$\frac{1}{T}(°K) \times 10^4$ T_l	T_e Time to explosion (Hr.)	a Radius cm	T_m °C	T_l °C	$\frac{1}{T}(°K) \times 10^4$ T_l	T_e Time to explosion (Hr.)
1" dia. cylinder					1" dia. cylinder				
1.27	116	117	25.62	0.85	1.27	225	227	19.99	0.30
1.27	116	118	25.55	0.70	1.27	225	230	19.87	0.19
1.27	116	119	25.49	0.55	1.27	225	235	19.68	0.13
1.27	116	120	25.43	0.50	1.27	225	240	19.49	0.11
1.27	116	125	25.12	0.35	1.27	225	250	19.11	0.10
1.27	116	130	24.80	0.30	1.27	225	260	18.76	0.08
1.27	116	135	24.50	0.20					
5" dia. cylinder					5" dia. cylinder				
6.35	99	102	26.64	10.00	6.35	193	195	21.36	4.80
6.35	99	105	26.44	8.00	6.35	193	200	21.13	2.50
6.35	99	110	26.10	6.70	6.35	193	210	20.70	2.00
6.35	99	115	25.76	4.30	6.35	193	220	20.28	1.70
6.35	99	120	25.43	3.20	6.35	193	230	19.87	1.10
6.35	99	125	25.12	1.80	6.35	193	240	19.49	0.80
6.35	99	130	24.80	1.00					
12" dia. cylinder					12" dia. cylinder				
15.24	90	95	27.16	42.00	15.24	176	178	22.16	27.00
15.24	90	100	26.80	35.00	15.24	176	180	22.07	19.50
15.24	90	105	26.44	28.00	15.24	176	185	21.83	13.50
15.24	90	110	26.10	18.00	15.24	176	190	21.59	11.40
15.24	90	120	25.43	5.50	15.24	176	200	21.13	8.70
15.24	90	130	24.80	1.30	15.24	176	220	20.28	3.70

TABLE III. PREDICTED DECOMPOSITION RESULTS FOR X-14 AND C-509 (Cont'd).

X-14					C-509				
a Radius cm	T_m °C	T_l °C	T_l $\frac{1}{T(°K)} \times 10^4$	T_e Time to explosion (Hr.)	a Radius cm	T_m °C	T_l °C	T_l $\frac{1}{T(°K)} \times 10^4$	T_e Time to explosion (Hr.)
54" dia. cylinder					54" dia. cylinder				
68.58	78	85	27.92	700	68.58	152	154	23.40	380
68.58	78	90	27.54	490	68.58	152	155	23.36	340
68.58	78	100	26.80	190	68.58	152	160	23.09	280
68.58	78	110	26.10	50	68.58	152	165	22.82	193
68.58	78	120	25.43	10	68.58	152	170	22.56	170
68.58	78	130	24.80	2	68.58	152	180	22.07	120
					68.58	152	190	21.59	71
					68.58	152	200	21.13	39
					68.58	152	220	20.28	8

In Fig. 4, predicted values of time-to-decomposition, surface temperature, and critical temperature data are shown for various sizes of X-14 propellant. Fig. 5 is identical in scope to Fig. 4 and shows the predicted data for C-509. Fig. Nos. 6 through 9 are comparisons between the experimental and predicted values for 1" and 5" samples of X-14 and C-509. Fig. Nos. 10 and 11 show an interesting correlation which is also shown in Fig. Nos. 4 and 5 in a different manner. This relationship is the variation in critical temperature for different sizes of propellant from infinitesimal mass to the mass of very large propellant grains. In practice it is found that small samples ranging from ten to twenty milligrams when decomposed give decomposition temperatures which fall near the infinitesimal mass asymptote. Data on these small samples were derived from differential thermal analysis. This report represents the initial effort in predicting thermal hazards using the nomographical technique. Considerable efforts are indicated for improvement in experimental chemical and physical property data, e.g., Q, ρ, c and λ. Extension and modification of the nomographs is also indicated. For example, the basic equations on which the nomographs are constructed do not account for changes in E and QZ with time.

The transition from the testing of pure compounds of simple shapes to composite propellants of complex composition and cross section (Ref. 8) poses many problems. Not only are there problems with the autocatalytic nature of certain materials and the effects of stabilizers,

there also exists problems in heat transfer that are very complex. The experimental cook-off data on hand does not show the predicted asymptotic approach to the critical temperature. The limited experimental cook-off data has been linearly represented.

This discussion has been presented to show the present state of the problem of predicting decomposition of propellants, to indicate what has been accomplished and in what areas further work is needed.

This report indicates the difficulties involved in predicting thermal decomposition of propellant systems. The data fit reasonably well in some cases. With improved chemical and physical property data, extension of the cook-off data to larger masses with improved instrumentation techniques, and investigations in the extension and modification of the nomographs to account for changes occurring with time and temperature, it is hoped to more closely predict critical temperatures and times for advanced, large-massed propellant systems.

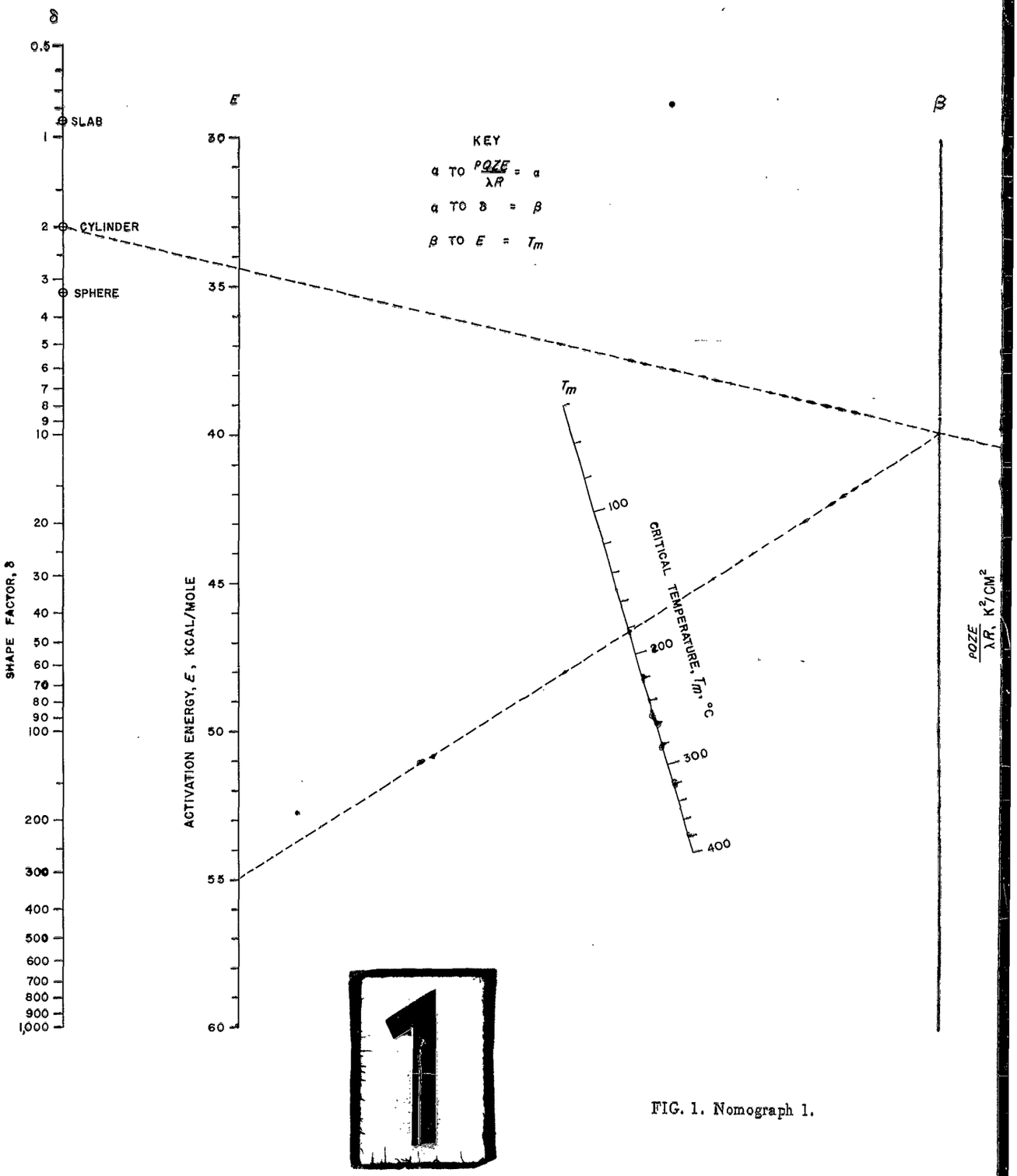

FIG. 1. Nomograph 1.

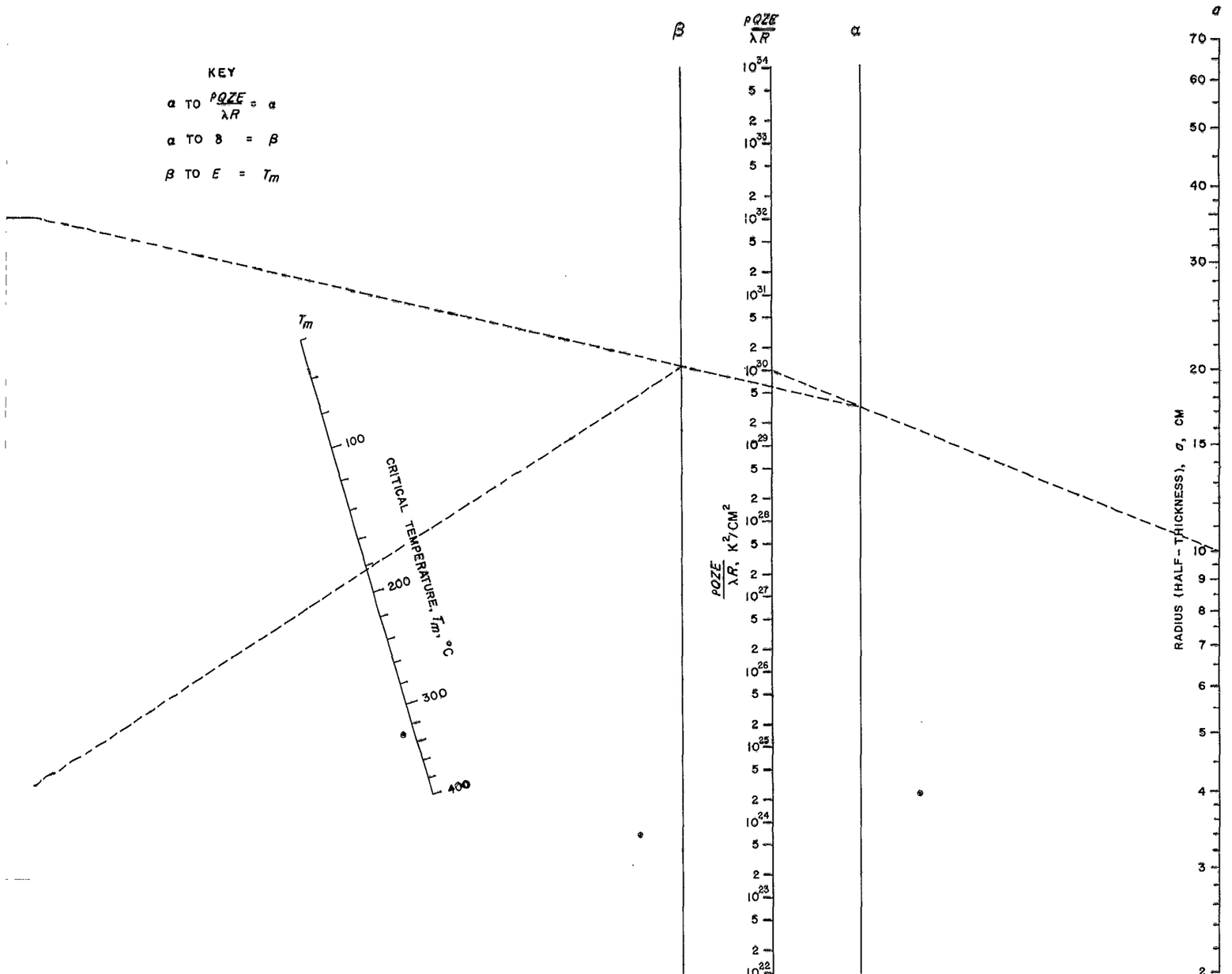

FIG. 1. Nomograph 1.

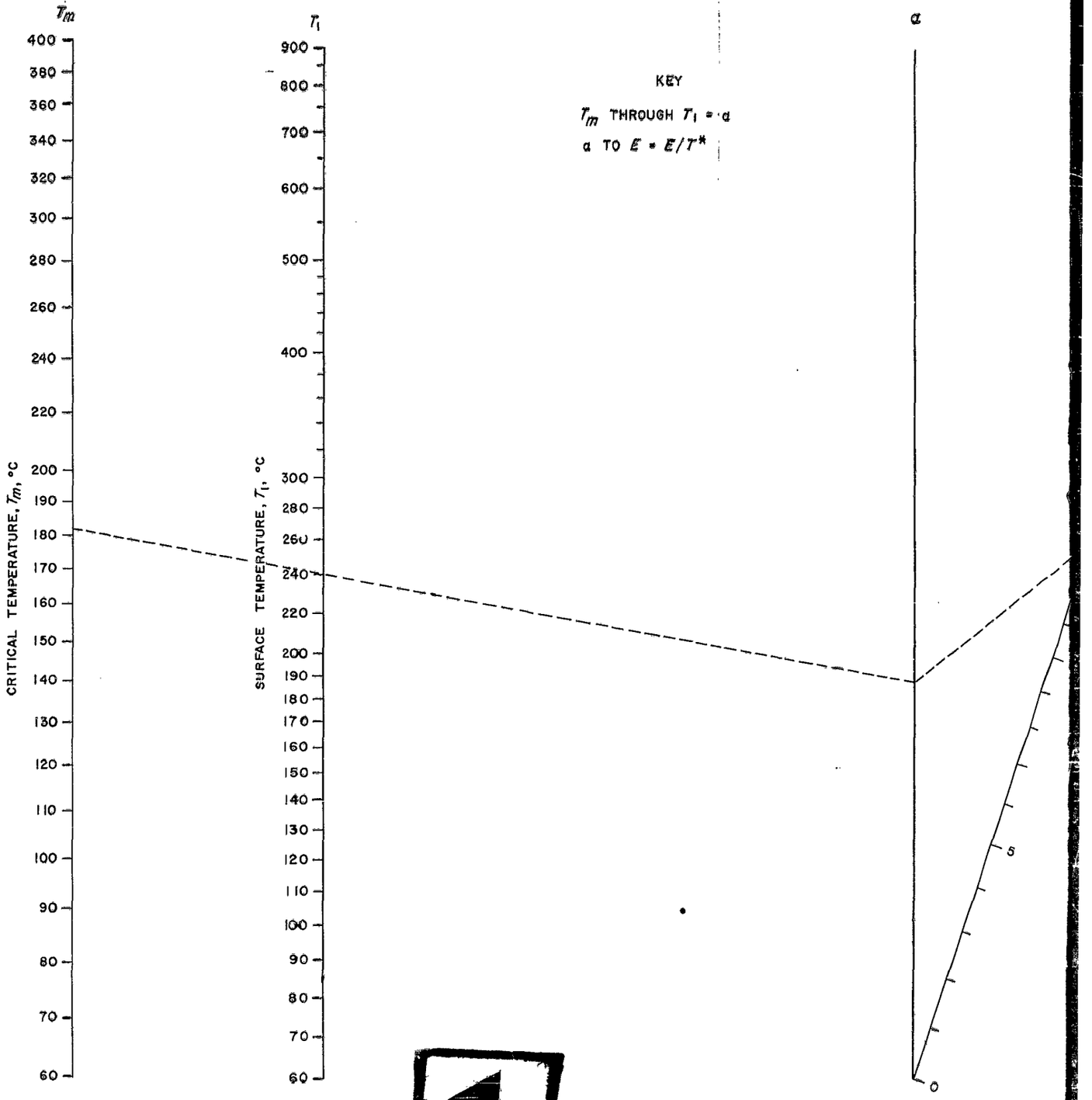

FIG. 2. Nomograph 2.

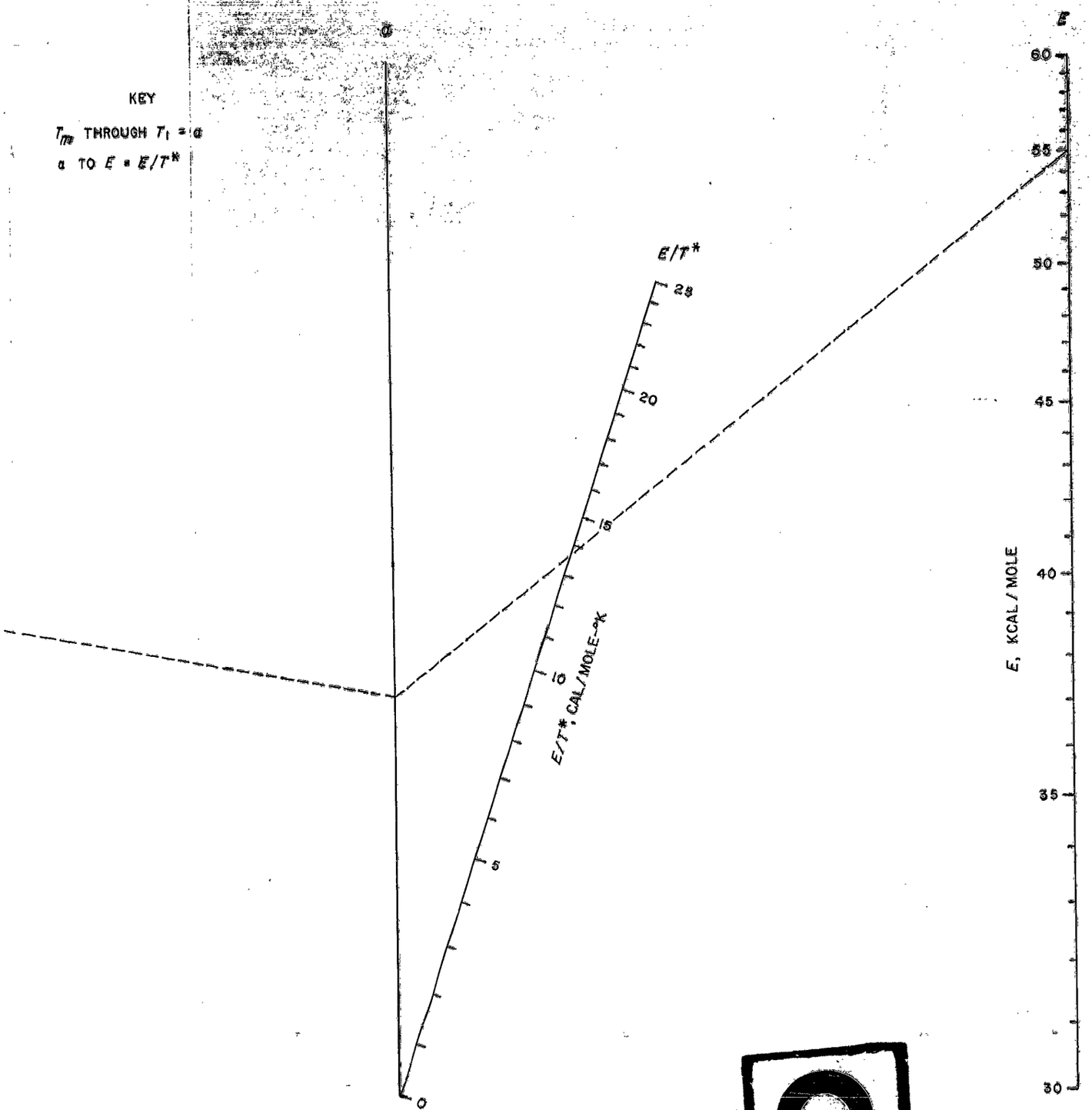

FIG. 2. Nomograph 2.

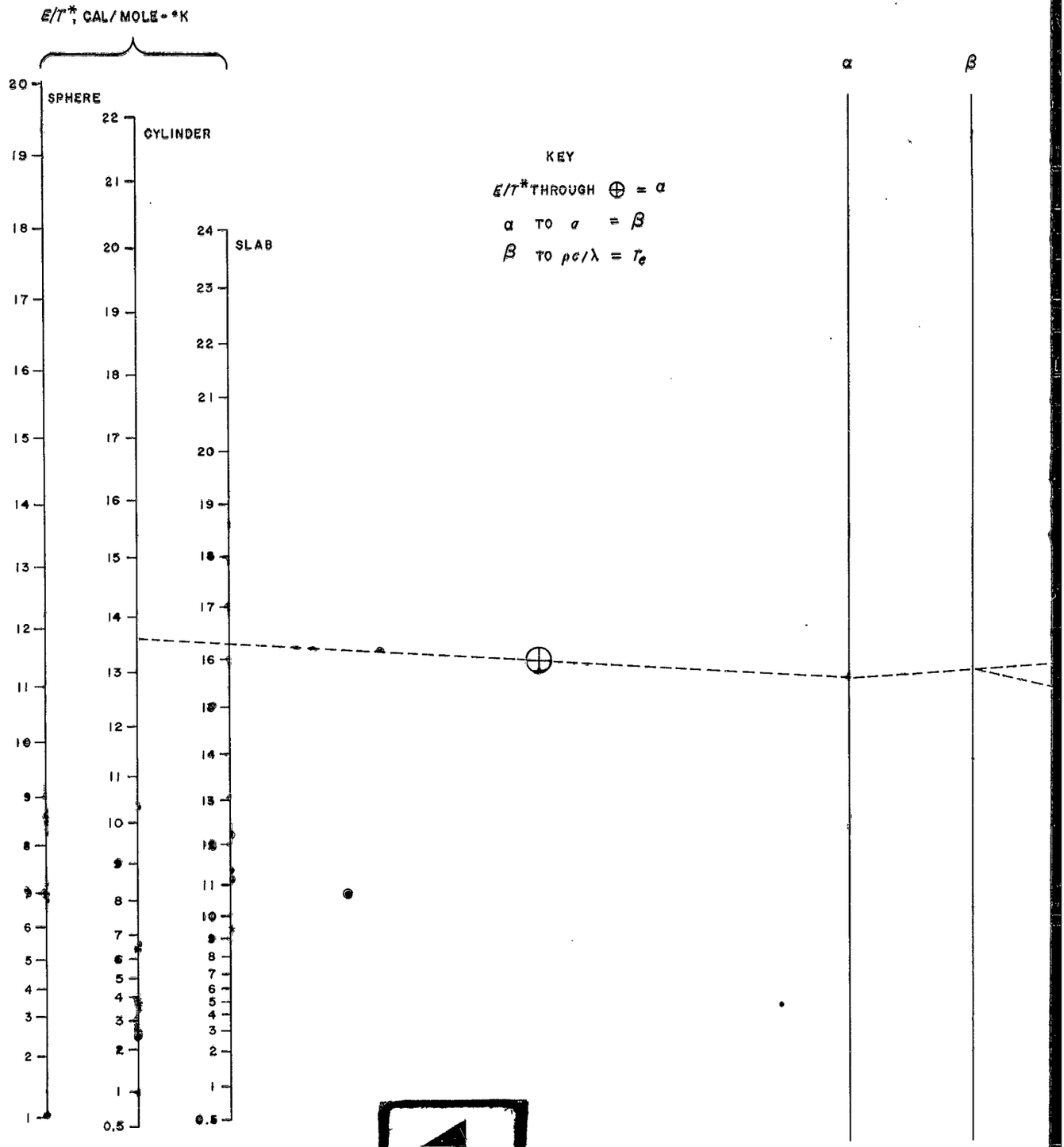

FIG. 3. Nomograph 3.

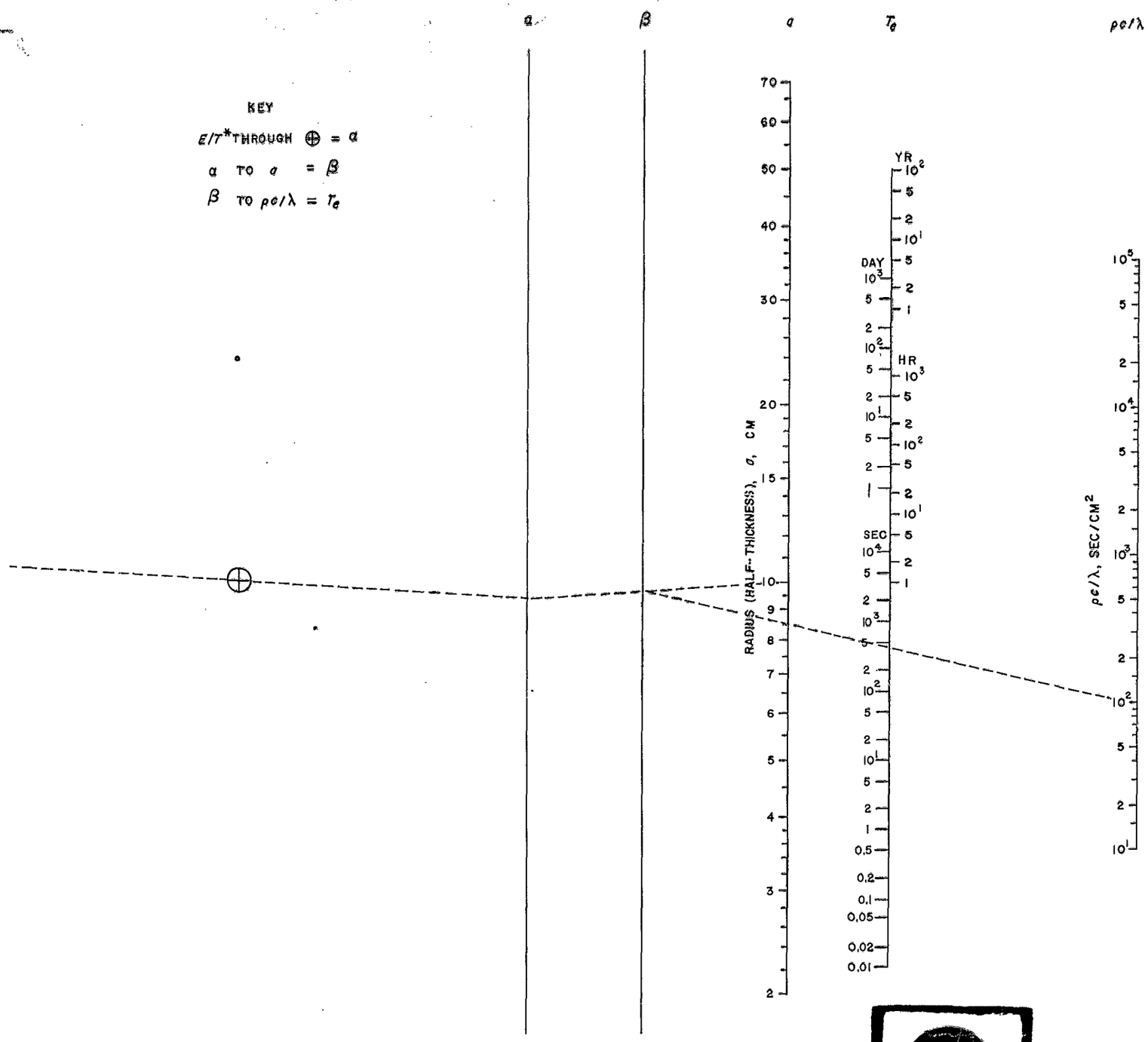

FIG. 8. Nomograph 8.

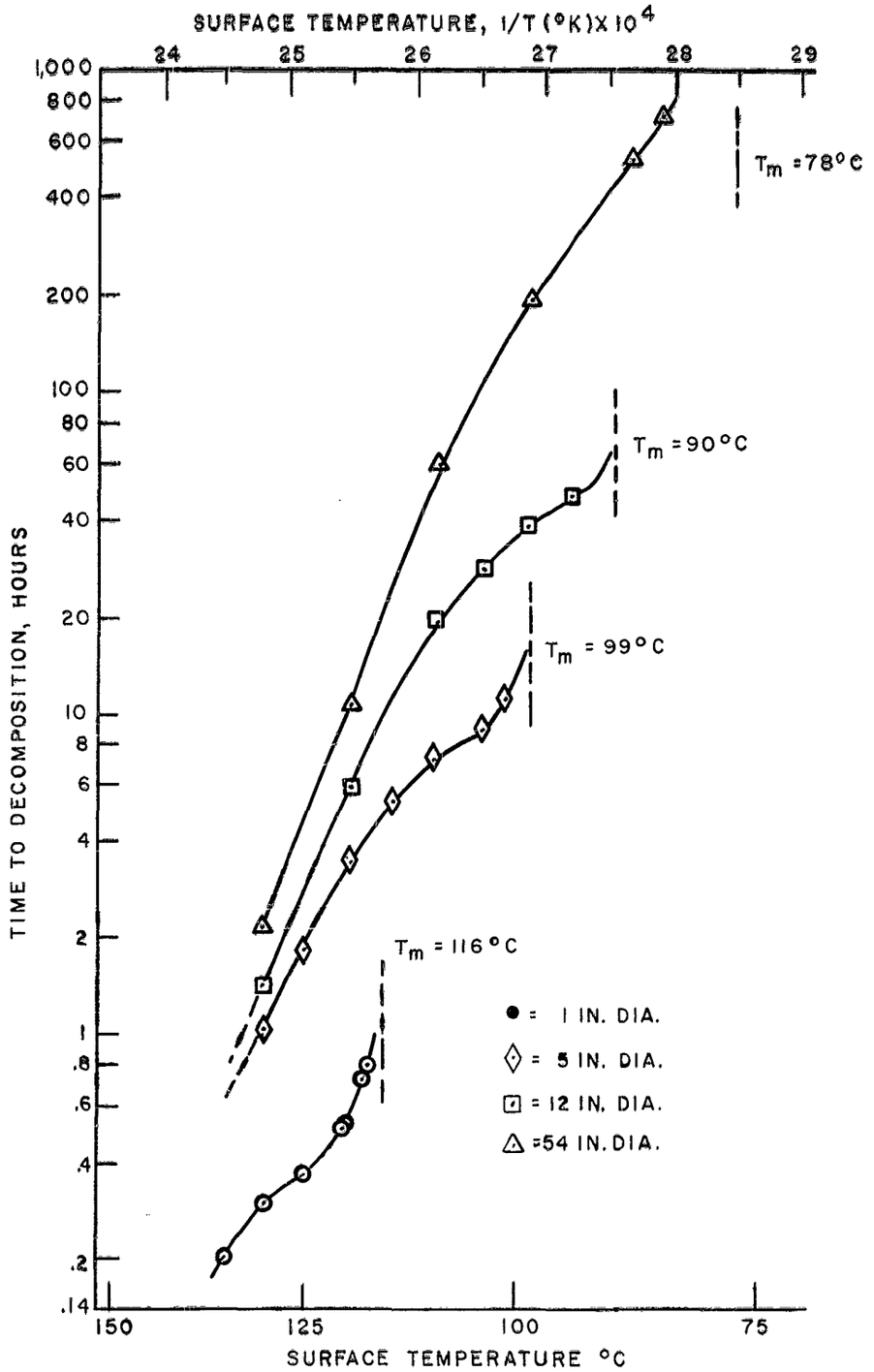

FIG. 4 PREDICTED DECOMPOSITION DATA FOR VARIOUS SIZED CYLINDERS OF X-14

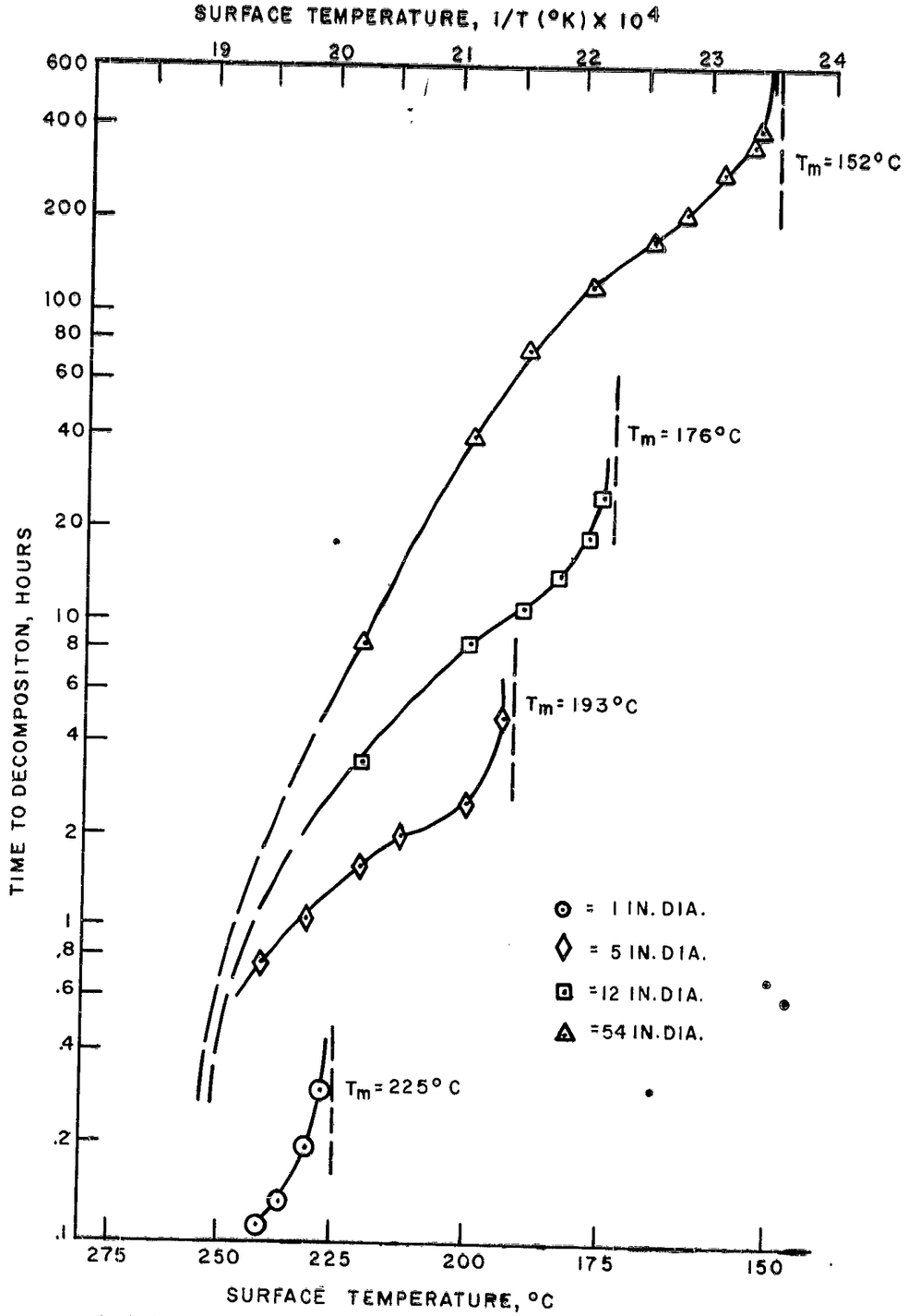

FIG. 5 PREDICTED DECOMPOSITION DATA FOR VARIOUS SIZED CYLINDERS OF C-509

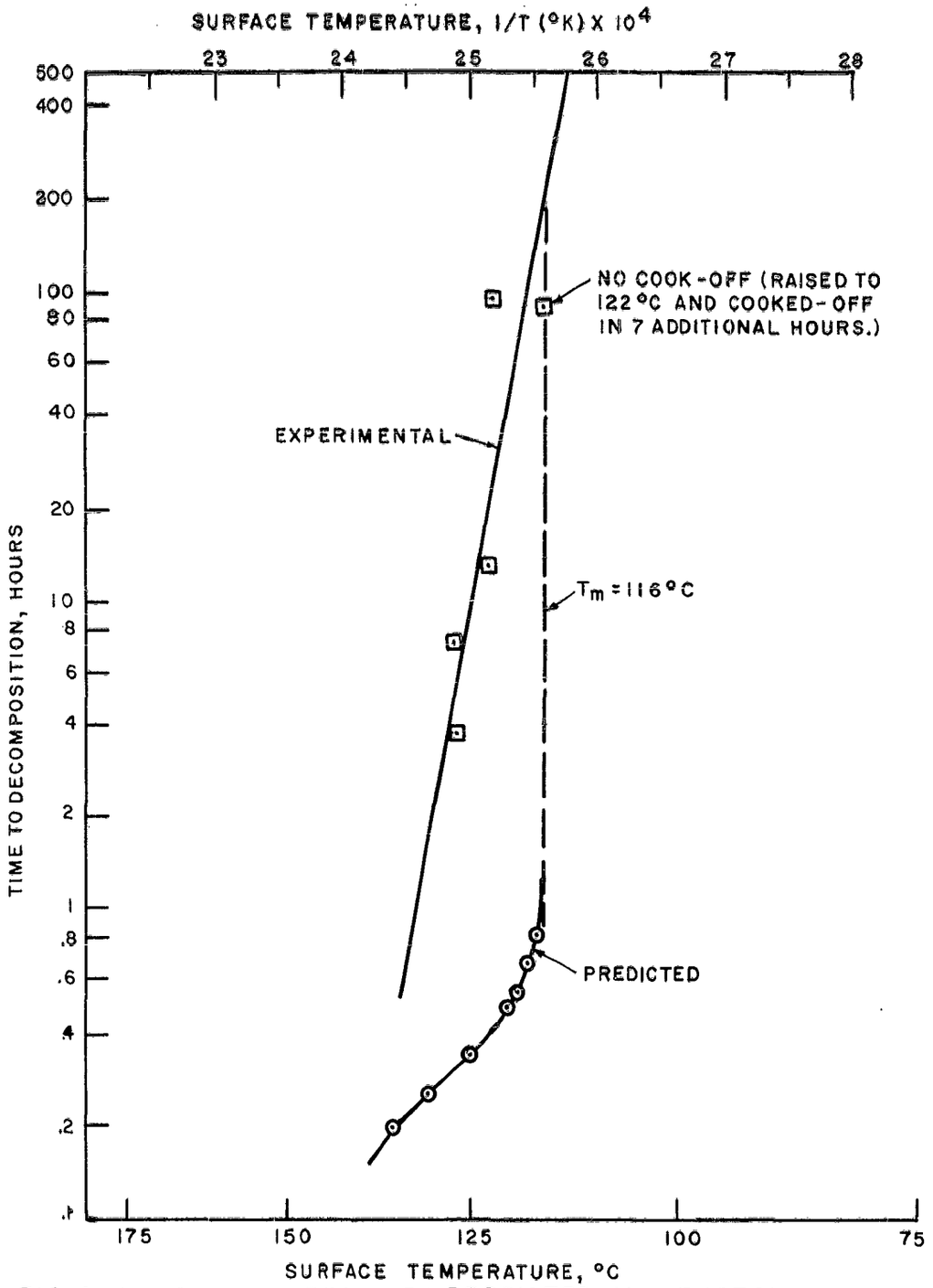

FIG. 6 COMPARISON BETWEEN PREDICTED AND EXPERIMENTAL DECOMPOSITION DATA FOR 1" CYLINDERS OF X-14

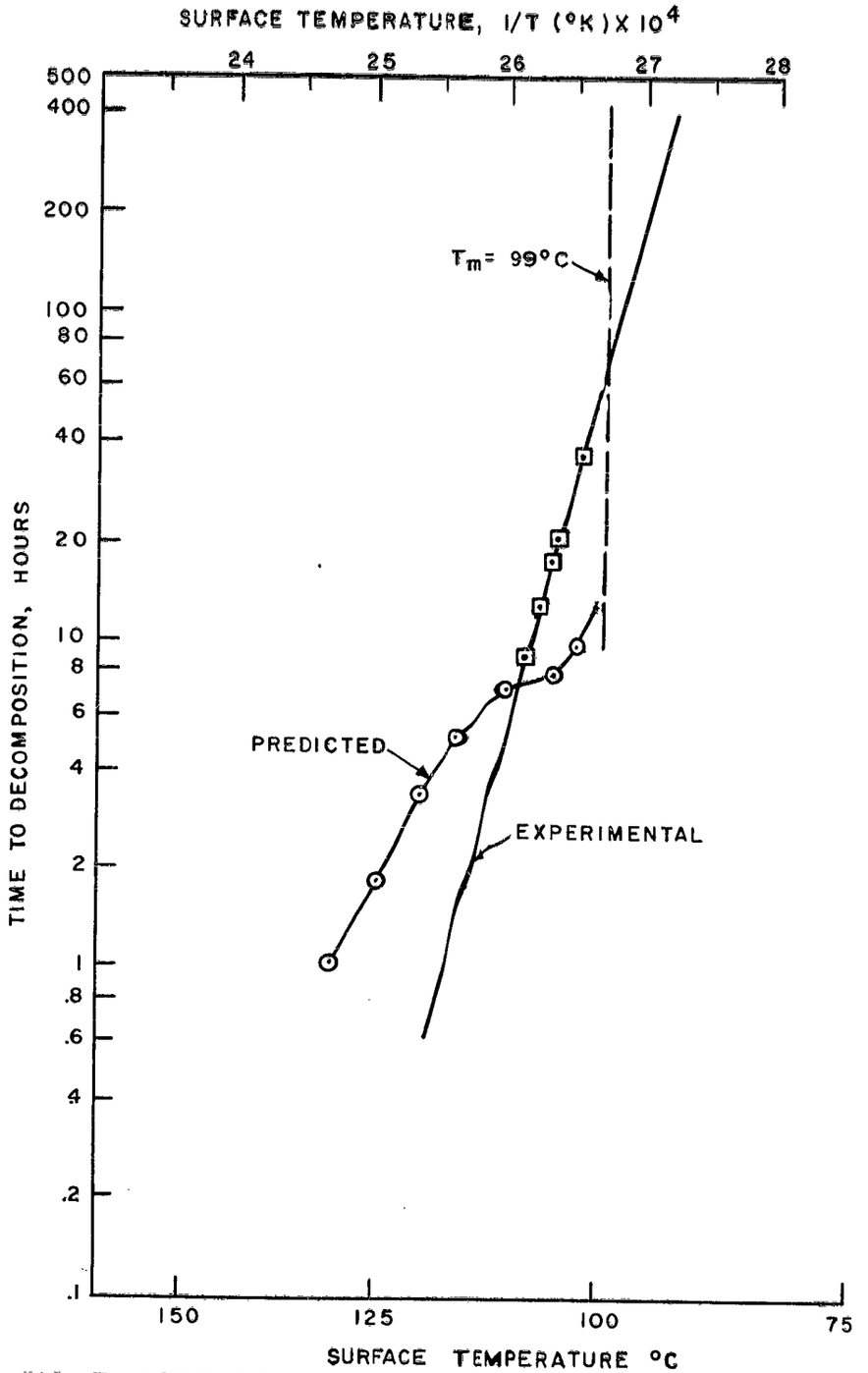

FIG. 7 COMPARISON BETWEEN PREDICTED AND EXPERIMENTAL DECOMPOSITION DATA FOR 5" CYLINDERS OF X-14

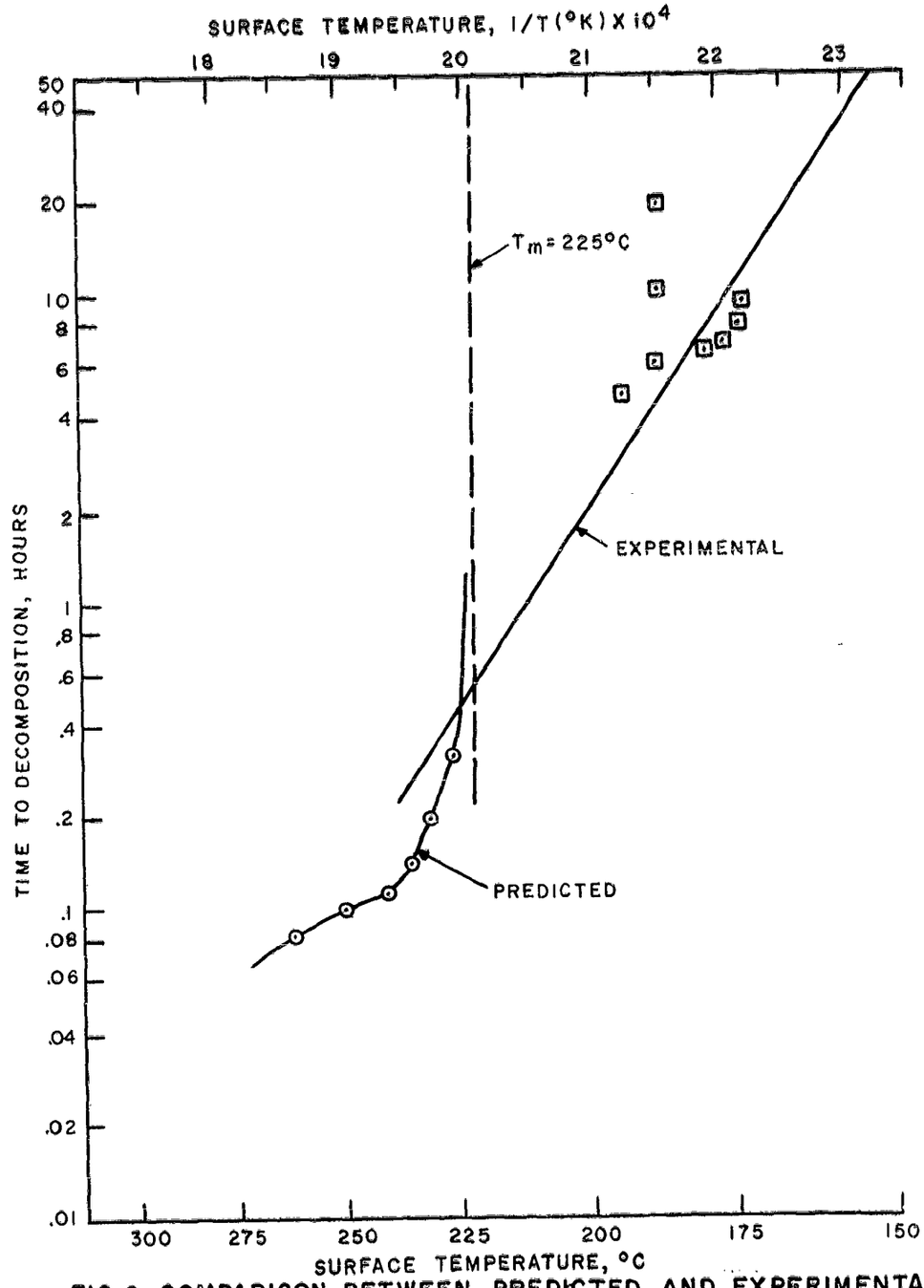

FIG. 8 COMPARISON BETWEEN PREDICTED AND EXPERIMENTAL DECOMPOSITION DATA FOR 1" CYLINDERS OF C-509

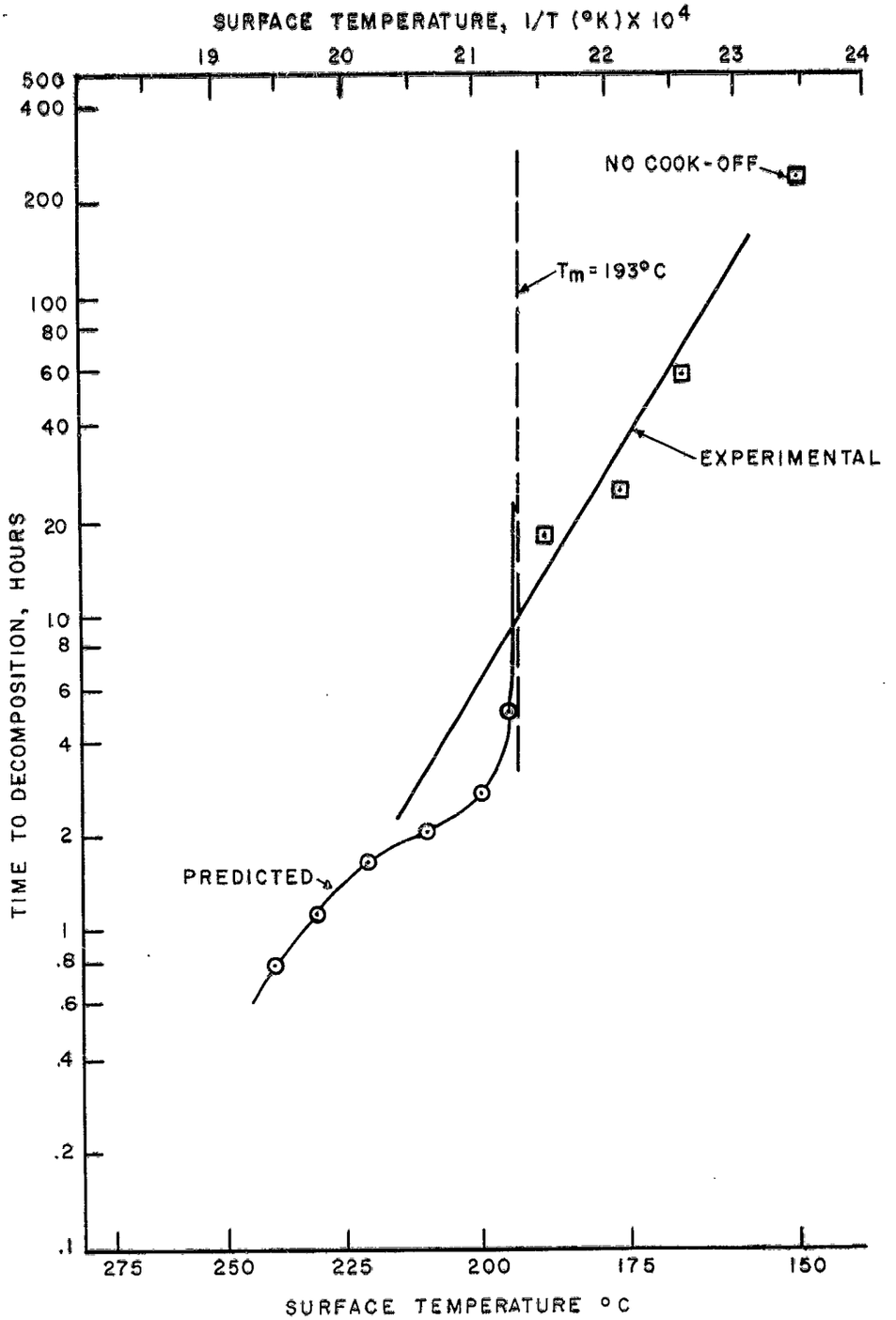

FIG. 9 COMPARISON BETWEEN PREDICTED AND EXPERIMENTAL DECOMPOSITION DATA FOR 5" CYLINDERS OF C-509

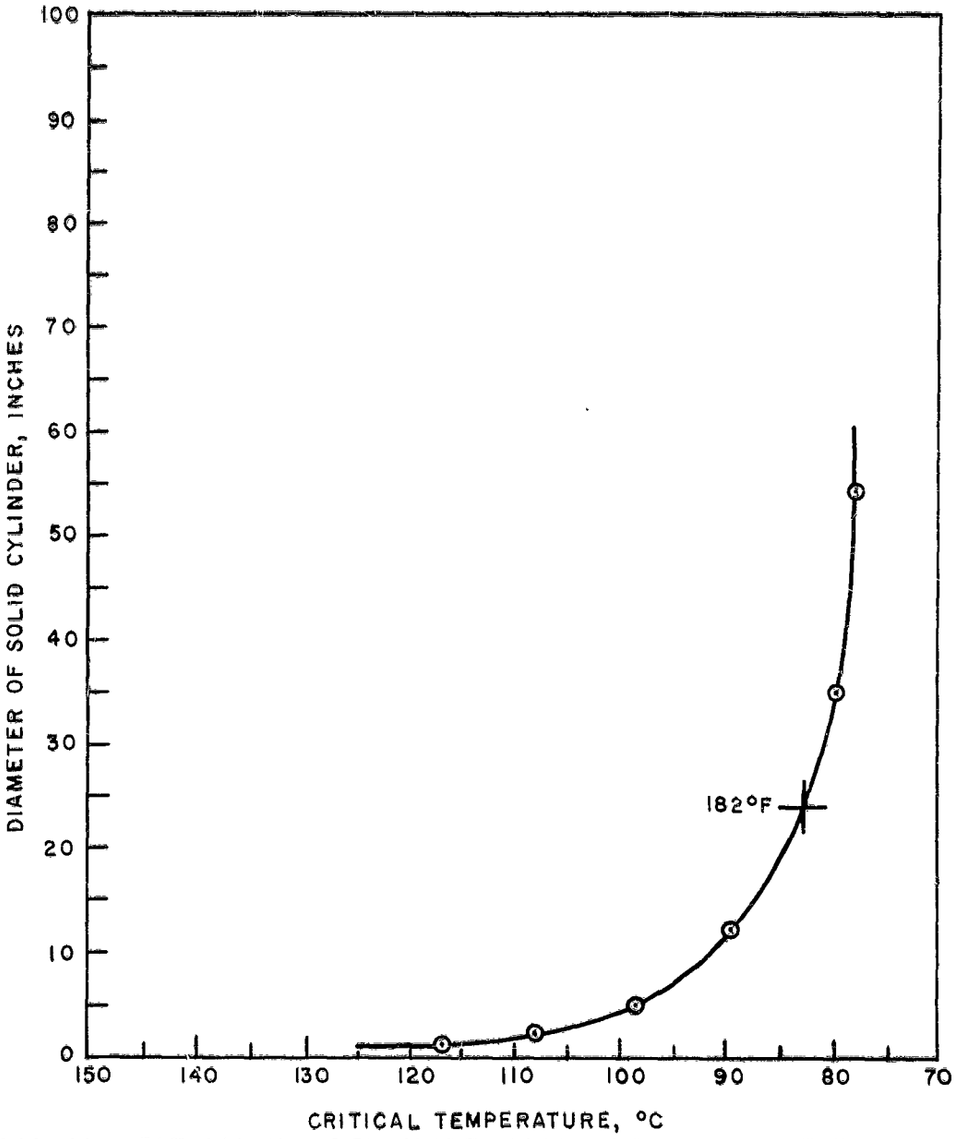

FIG. 10 CRITICAL TEMPERATURE VARIANCE WITH SIZE FOR X-14

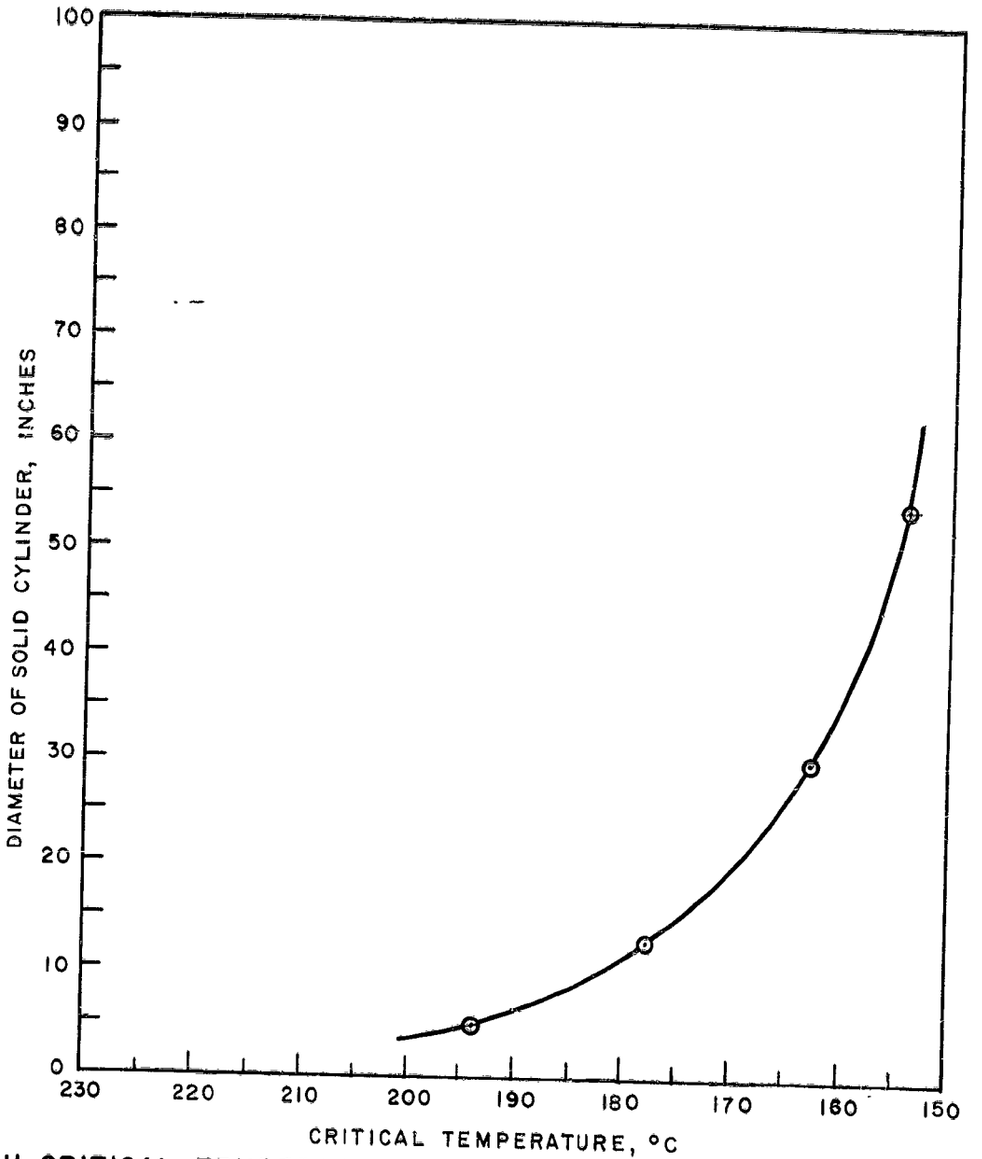

FIG. 11 CRITICAL TEMPERATURE VARIANCE WITH SIZE FOR C-509

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