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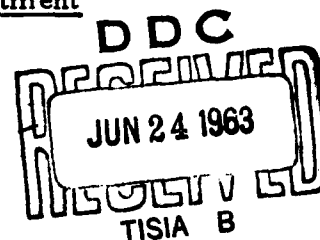
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PREDICTING THERMAL HAZARDS IN PROPELLANTS UTILIZING COOK-OFF TECHNIQUES

by

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ABSTRACT. From experimental data obtained with standardized cook-off ovens, the relationship between log time-to-deflagration and the reciprocal of the absolute temperature was established as a straight line. The experimental cook-off technique was applied to the complex configuration of the ASROC grain, and the characteristic cook-off curve established. An application in stabilizer studies is presented, and cook-off data are given for various propellants.

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U. S. NAVAL ORDNANCE TEST STATION

China Lake, California

March 1963

U. S. NAVAL ORDNANCE TEST STATION

AN ACTIVITY OF THE BUREAU OF NAVAL WEAPONS

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FOREWORD

Since February 1961, the Test and Evaluation Division has inaugurated a continuing project at the Naval Ordnance Test Station that has as its goal the prediction of the critical temperature (temperature that, if exceeded, will result in spontaneous combustion) and the thermal behavior of large-diameter propellants during storage.

In this report, the surveillance and cook-off data on presently developed propellant systems are summarized, and applicable applied research techniques for predicting the safe-life of newly-developed propellant systems are evaluated.

The work was performed in the Product Evaluation Branch of the Propulsion Development Department under Bureau of Naval Weapons Special Projects No. SP 71402-10.

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CONTENTS

Introduction	1
Methods of Stability Testing	1
150°F Test	1
Exothermic Process	3
Differential Thermal Analysis	3
Brass Block Ignition Temperature	5
Cook-off Temperature	5
Cook-off Oven Design	6
Applications of Cook-off Testing.....	7
X-12 and X-12 High Energy Propellants.....	7
Stabilizer Studies	7
Aerodynamic Heating	8
Cook-off Temperatures for Propellants	12
Prediction of Time to Deflagration	20
ASROC Tests	23
Oven Design	24
Test Procedure.....	24
Test Results	24
Summary and Conclusions	27

INTRODUCTION

By 1950 surveillance methods had been fairly well-stabilized for existing nitrate ester propellants. Pyro powders could be held in ambient storage for forty years, and there were good data on JPN double-base propellants indicating a similar long and useful life. The Naval Propellant Plant completed 3556 standard 150°F (65.5°C) surveillance tests with JPN. The average chemical safe-life at that temperature was 533 days; minimum was 315 days; and maximum was 700 days. Figure 1, adapted from Ref. 1, shows how JPN data could be extrapolated to give an estimate of 50 to 100 years for safe-life at 86°F. Two samples of JPN had been stored at 122°F for 8 years. Using an ample margin of safety for chemical safe-life, BuWeps instruction to all ships in 1952 were that rocket ammunition exposed to 130°F or above should be disposed of in deep water.

JPN records served as good reference data, and thus a determination of the 150°F surveillance life would be acceptable for new double-base propellants, if it proved to be close to the 533 days established for JPN. N-5, a mesa-burning propellant, was developed and was shown to have a 150°F surveillance life of over 500 days. On the basis of these findings no further surveillance studies were required for N-5.

METHODS OF STABILITY TESTING

150°F TEST

Propellant stability could be evaluated in different ways. One method employed with nitrate esters, as previously indicated, was to store 45-gram samples in glass bottles until red fumes appeared with the oven atmosphere maintained at 150°F (65.5°C). This sometimes required 2 years or more. The following table shows time to fume-off at 150°F of five different propellants:

Propellant	Time to fume-off
High Energy X-12	20 months
Standard X-12	1 sample 24 months
	2 samples--no fume-off after 24 months.
N-5	No fume-off after 24 months
	Test discontinued.
X-9	26 months
N-4	22 months

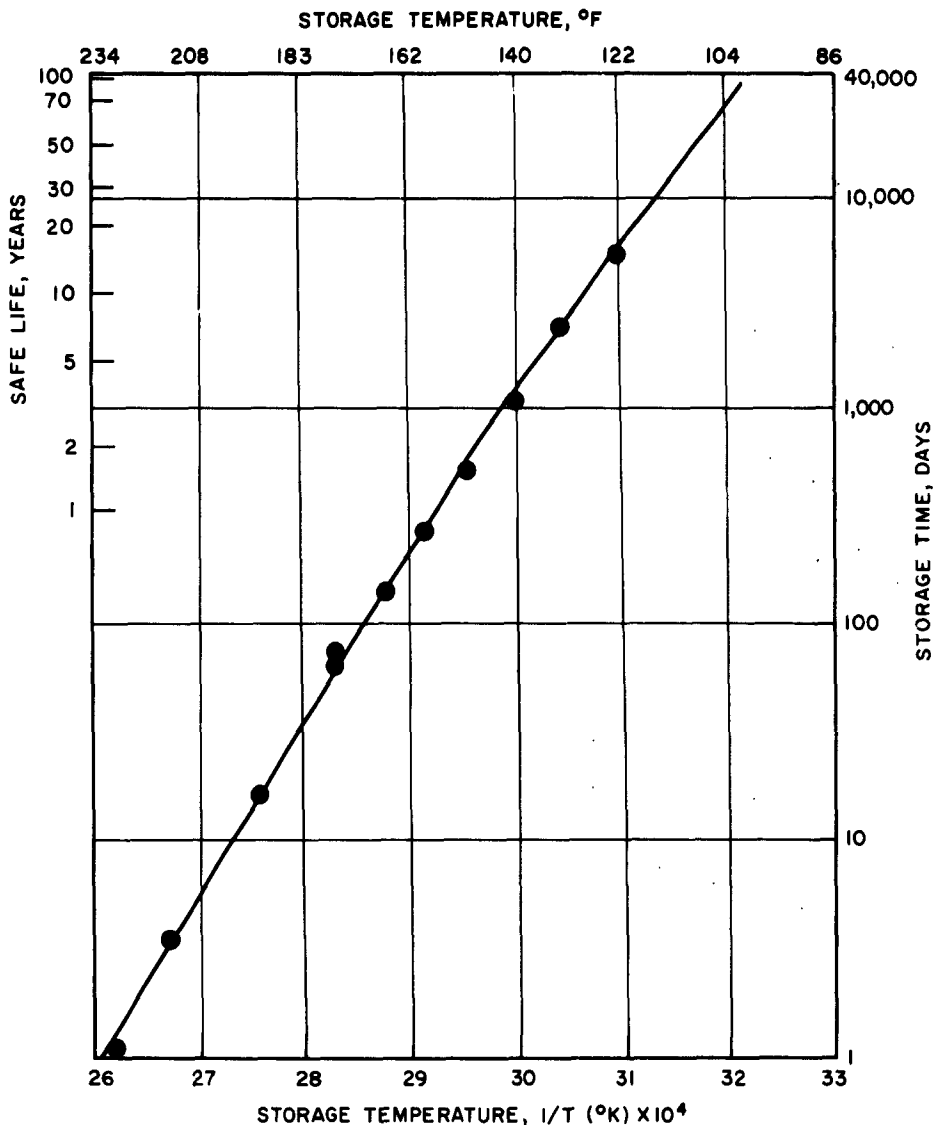


FIG. 1. Safe-Life of JPN Propellant Stored as Small-Web (< 3-Inch) Samples.

Higher storage temperatures decreased the time required to complete the tests. From ambient temperature up to a somewhat indeterminate boundary near 200°F, decomposition of nitrate esters in double-base propellants is controllable by stabilizers. Oxides of nitrogen, liberated in the decomposition process of the nitrate esters, are absorbed by the stabilizer and the reaction does not become autocatalytic. Reaction rates increase as the propellant is heated. If the effect of the

stabilizer is overcome by high temperature, or if the stabilizer becomes depleted, an increasingly rapid evolution of heat may be expected.

EXOTHERMIC PROCESS

The exothermic process has been studied by use of the adiabatic heating oven. The procedure used by Amster (Ref. 2) was to raise the oven temperature until a sensible indication of exothermic heat was established. The oven control was then programmed to follow the sample temperature with no interchange of heat between sample and oven air; thus, heating of the sample was adiabatic. With a sample of cast double-base propellant 2 inches in diameter and 2 inches long, the time required for adiabatic heating from 212°F to ignition was 10 hours. The following tabulation is a typical rate of temperature increase at various intervals as a sample heated exothermically:

Sample temperature, °F	Adiabatic heating rate, °F/min
220	0.05
240	0.36
260	0.68
280	2.70
300	8.64

DIFFERENTIAL THERMAL ANALYSIS

Another way to observe exothermic reactions is to subject small samples to differential thermal analysis (DTA). An inert sample and a propellant sample (weighing 20 mg, or less) are monitored by two thermocouples connected in series while the assembly is heated at a uniform rate. An exothermic reaction, developing within the propellant sample, causes its thermocouple to indicate a temperature above that of the inert sample. Figure 2 shows thermograms of several nitrate ester plasticizers and of two double-base propellants. The curves all show very similar exotherms, increasing rapidly at temperatures above 300°F. The DTA tests have characteristically been made with sample sizes of 5 to 60 milligrams and are performed in the laboratory without need for barricading. The sample is heated at rates varying from 0.1 to more than 10°F per minute, and graphs obtained show exotherms and endotherms. Points of interest are the "ONSET POINT" (Fig. 3), the first detectable evidence of a reaction, and the "autocatalytic point", which is not yet well defined. A tentative definition of autocatalytic point is the point at which the reaction shows an exponential rise, with a given configuration, and thus quickly goes to completion. The maximum departure of the sample temperature from the control temperature is also noted and is named a "PEAK" for a given exotherm or endotherm.

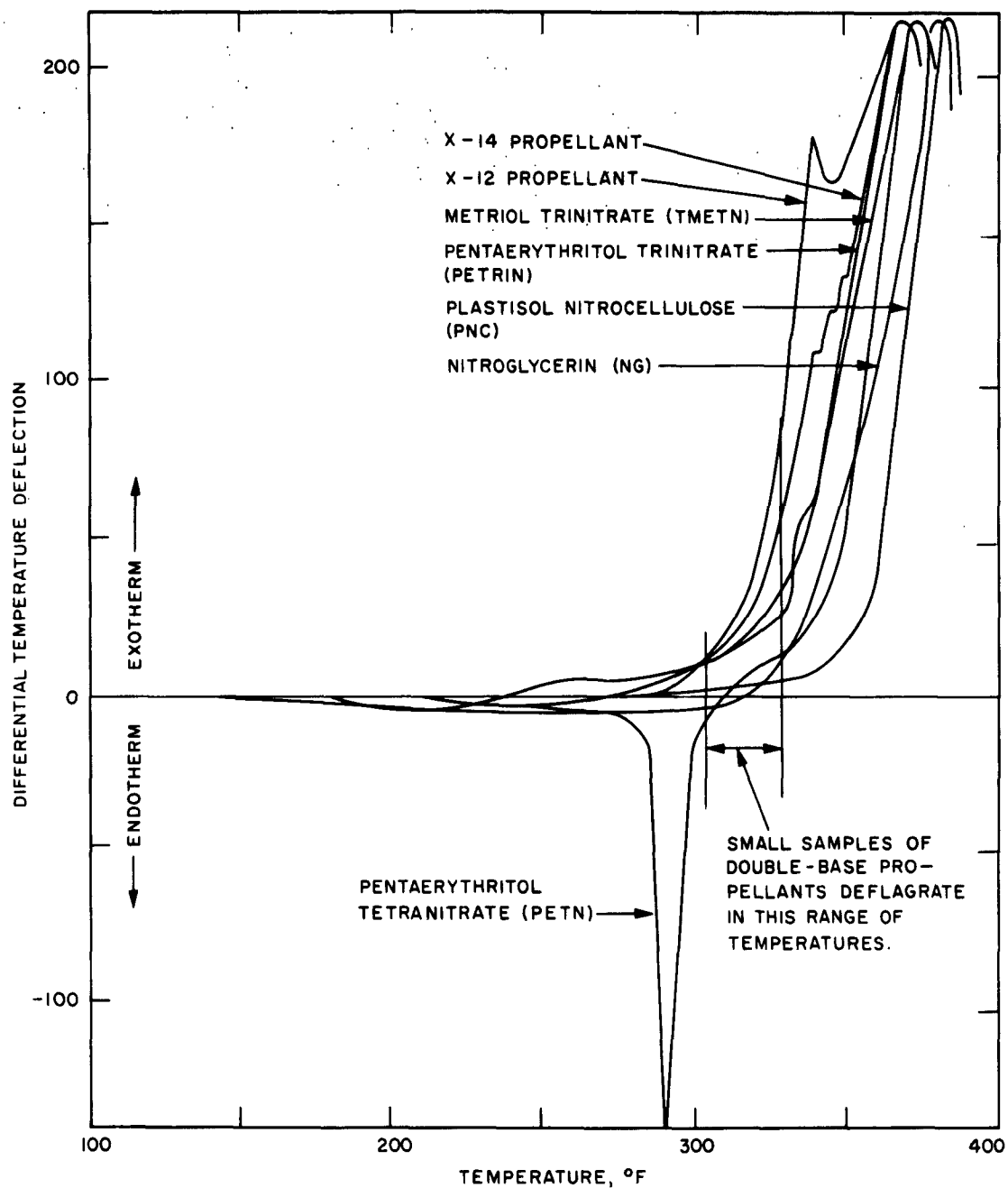


FIG. 2. Differential Thermogram of Double-Base Propellants and of Certain Plasticizers for Rate of Temperature Rise of 1° to 5°F/min.

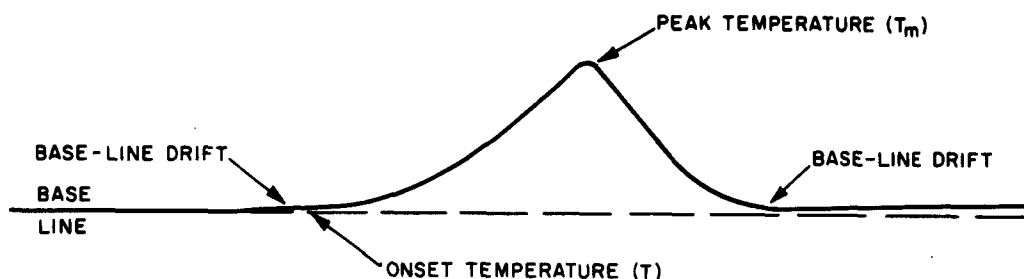


FIG. 3. Representative Thermogram.

The exotherms, as determined by DTA, enable the experimenter to select a temperature for the first cook-off test of larger samples. With nitrate esters, the cook-off temperatures for 1- to 5-inch diameter samples have been 70° to 100°F below the DTA "ONSET POINT". The exact difference is dependent on the geometry of the test specimen.

BRASS BLOCK IGNITION TEMPERATURE

In another small-sample technique, samples weighing 1 to 3 grams are heated in a brass block and the ignition temperatures recorded. The apparent autoignition point for double-base propellants, determined in this manner, ranges from 310°F to 350°F depending on the heating rate. Thus for a given test geometry, the exact temperature of deflagration is dependent on the heating rate. It becomes apparent from these studies that small samples of double-base propellants may be expected to ignite within a few minutes with oven temperatures above 300°F.

Both thermograms from the DTA and adiabatic heating records are valuable information to have in hand prior to planning cook-off tests where appreciable weights of propellants are involved.

COOK-OFF TEMPERATURE

The cook-off characteristics of propellants are of interest when storage at elevated temperatures is anticipated and data are required in order to set safe limits. Similar applications may be made for design of ovens used in heat-cracking tests and for surveillance. Autoignition temperatures may be of interest in this regard; however, with grains of various configurations, it is more important to establish with actual cook-off tests the residence time in the ovens at which deflagration occurs. This has been determined for certain propellants over a range of temperatures. The temperature ranges for various areas of interest can be classified as follows:

Double-base propellants		
Area of interest	Temperature range, °F	Probable safe-life, or elapsed time when cook-off occurs
Normal magazine storage	50 to 120	8 years to 100 years
Conditioning ovens, surveillance ovens, cast curing	120 to 200	10 days to 8 years
Cook-off studies	200 to 250	3 hours to 10 days
Screw extrusion	250 to 300	10 minutes to 3 hours
Extrusion inhibiting, aerodynamic heating	300 to 400	1 to 10 minutes
Ignition studies	Over 400	Less than one minute

COOK-OFF OVEN DESIGN

Ovens were built that would withstand repeated deflagrations of propellant samples weighing up to five pounds, and cook-off tests were made with samples of various sizes and configurations.

Standard cook-off ovens have been 7.65-inch ID and 4 feet long made from 8-inch steel pipe. They are designed to test 6-inch OD samples weighing up to 10 pounds. Lightweight fiberglass plugs are used in each end of the oven, made so that they are a tight fit to prevent convection heat losses and yet give quick release for the hot gases during deflagration.

In a standard test, the oven is brought to the desired temperature with a dummy set-up. The test specimen, implemented with thermocouples, is then placed in the oven and the test is monitored remotely until deflagration occurs.

The general practice has been to select such storage temperatures that deflagration occurs in one to five days. Plots of data obtained from these tests show a straight line relationship between the log of the time to deflagration and the reciprocal of the absolute temperature. In exploratory tests, five samples are generally required to establish a curve, and the testing of a propellant is completed in approximately 15 days. A preliminary curve may be established with three samples within 6 days with a judicious choice of oven temperatures. Size of

sample may be 100 grams during the development phase and 3.0 kilograms at later stages in the testing of a specific propellant formulation. This covers the range of grain dimensions (1 to 5 inches) normally used in development work.

Since deflagration occurs to terminate each test and occasional detonations may be expected, the ovens must be monitored by remote control and must be properly barricaded.

The cook-off techniques have been applicable and important in propellant research and development; e. g., in studying the effects of stabilizers on safe-storage life, in determining aerodynamic heating effects on propellant and explosive systems, in the development of high-temperature resistive propellants, and in predicting possible thermal hazards in scaling high-energy propulsion systems.

APPLICATIONS OF COOK-OFF TESTING

X-12 AND X-12 HIGH ENERGY PROPELLANTS

Typical cook-off data and the method of treating the data are illustrated by two NOTS developed propellants: X-12 standard and X-12 high-energy.¹ Both are typical of the nitrocellulose-nitroglycerin double-base systems. The cook-off data are summarized in Table 1. The warm-up time indicates the time required for the sample to reach the preset oven temperature. The remaining cook-off time shown in this tabulation is the time to deflagration after the sample reaches the oven temperature. All samples indicated an exothermic rise in temperature to cook-off.

A representation of cook-off data as residence time in the oven to deflagration versus the oven temperature settings is shown in Fig. 4. The typical straight line representation, when plotted as the logarithmic time to deflagration versus the reciprocal absolute temperature, is shown in this figure.

STABILIZER STUDIES

One area in which the cook-off techniques have become a useful tool is that of chemical stabilizers. In the nitrasol propellant system

¹ Solid Propellants Information Agency Manual M2, July 1961 for Compositions.

TABLE 1. Cook-off Data on Double-Base Propellant
Cylindrical sections (4-in. length, approx. 4-lb sections)

Oven temperature, °F	Time to cook-off, hours	
	X-12 standard	X-12 high-energy
221	8.3 (warmup) 10.3 (cook-off)	11.25 (warmup) 6.75 (cook-off)
219	--- 10.2 (cook-off)	
215	8.6 (warmup) 38.4 (cook-off)	---
210	---	11.0 (warmup) 45.0 (cook-off)
207	8.2 (warmup) 78.5 (cook-off)	---

Warmup = time required to bring sample to oven temperature.

Cook-off = additional time required to cook-off.

(plastisol nitrocellulose), the effectiveness of stabilizer A and stabilizer B is aptly demonstrated with cook-off data presented in Fig. 5. The data were determined in this comparison with 5.0-inch solid cylinders 6.0 inches in length. Figure 6 is a summarization of cook-off data using 5.0-inch diameter cylinders within the nitrasol propellant system, wherein smaller variations within the stabilizer system made only minor changes in the thermal stability characteristics.

AERODYNAMIC HEATING

Another area in which the cook-off techniques have been used in developmental procedures is that of aerodynamic heating. As the speed of missile carrying planes increases, the aerodynamic heating problem becomes more severe. Questions have been raised concerning the ability of propellants and explosives to endure repetitive aerodynamic heating cycles representing several missions of such planes.

Since propellants may ignite spontaneously at elevated temperatures, it is necessary to explore the conditions under which deflagration occurs. This information is essential as rocket motors are being exposed to higher and higher temperatures. Aerodynamic heating becomes a critical factor above Mach 2.5.

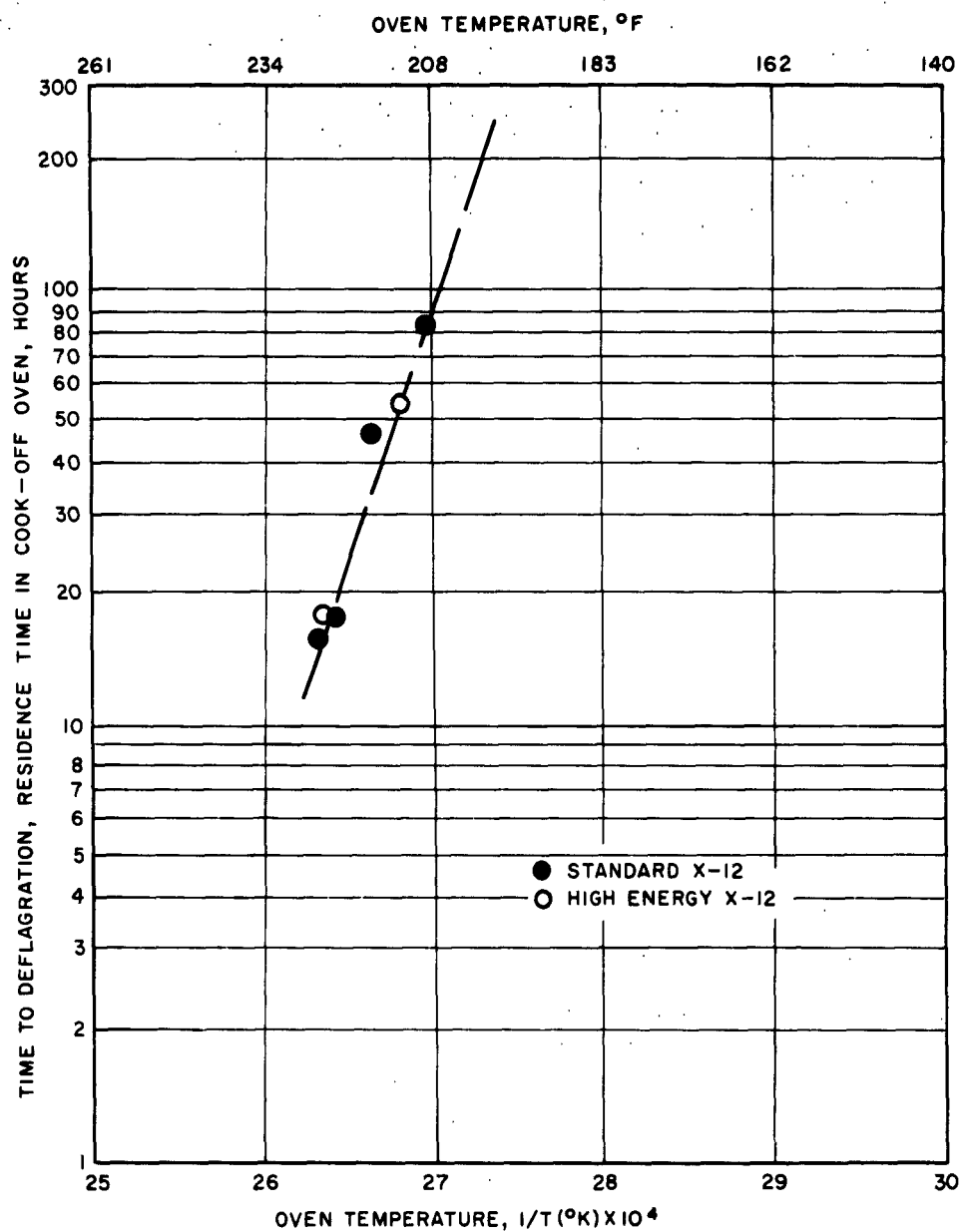
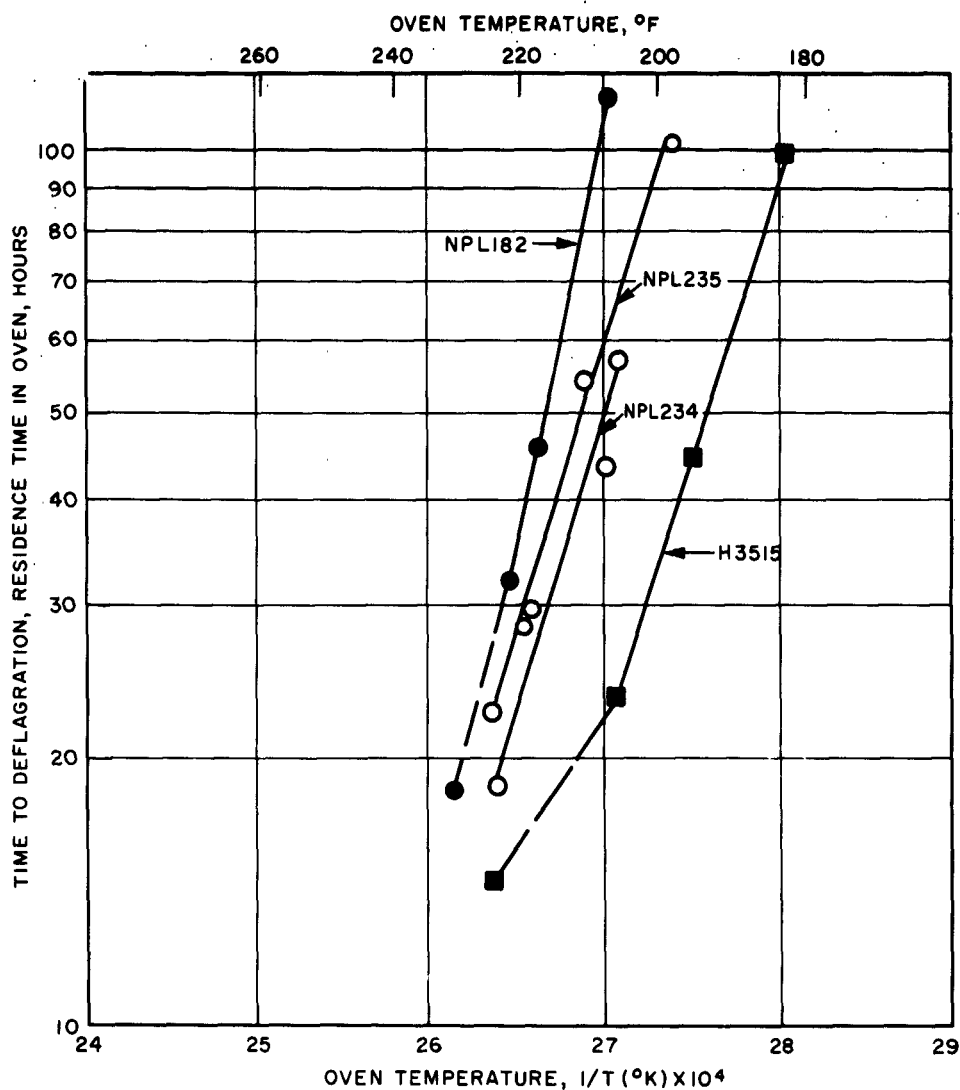


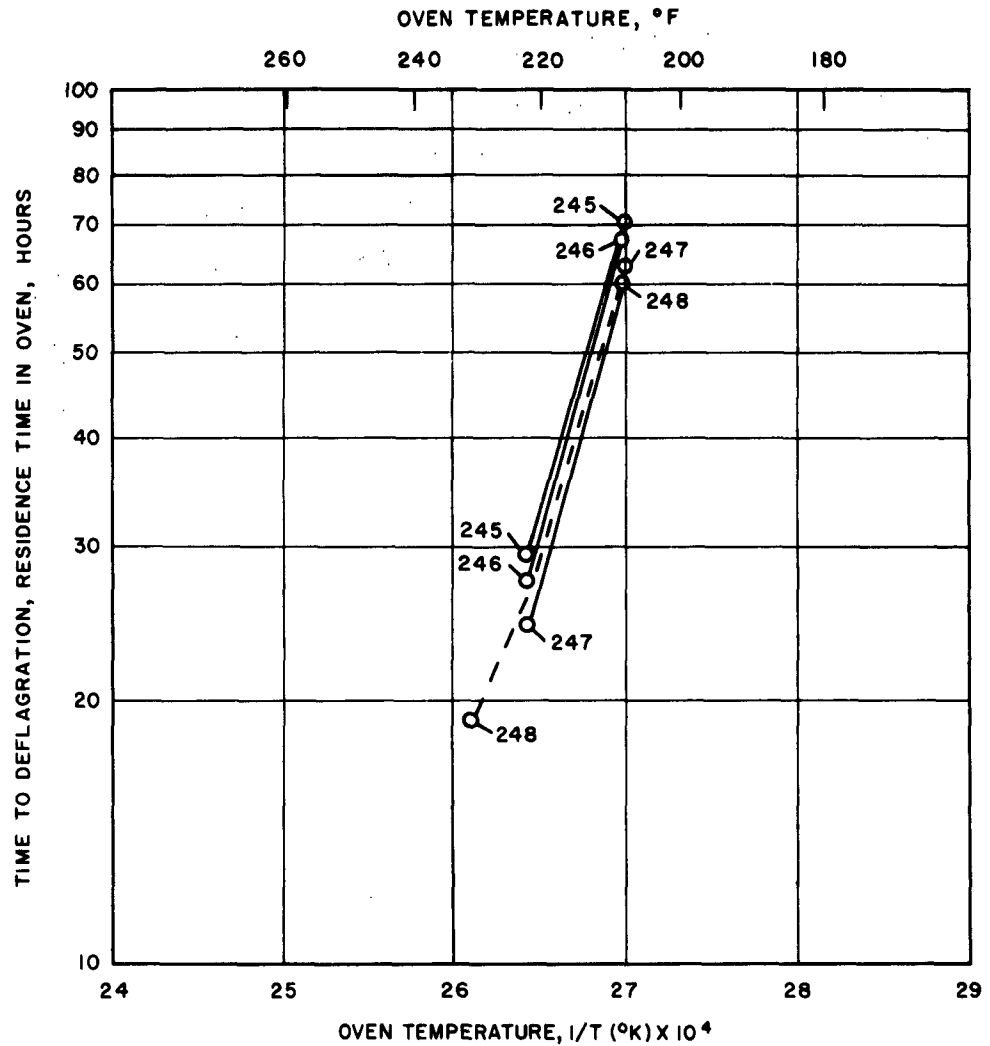
FIG. 4. Cook-off Chart.



Formulation	Time to cook-off*, hours	Percent of stabilizers		
		A	B	C
H3515	21	none	none	0.75
NPL234	43	0.3	0.3	0.75
NPL235	53	0.4	0.6	none
NPL182	90	1.0	none	0.75

*With oven temperature at 210°F.

FIG. 5. Effect of Stabilizers on Cook-off of Nitrasol Propellant (Solid Cylinders 5.0-Inch OD and 6.0-Inch Long).



Formulation	Percent of stabilizers			
	A	B	C	D
NPL245	0.3	0.3	0.8	none
NPL246	0.4	0.6	none	none
NPL247	0.3	0.3	0.8	5.0
NPL248	0.4	0.6	none	5.0

FIG. 6. Cook-off of Four Formulations of Nitrasol Propellant (Solid Cylinders 5.0-Inch OD and 6.0-Inch Long).

Figure 7 is illustrative of the motor tube temperatures caused by aerodynamic heating of the Sidewinder 1C missile during cruising and acceleration of an aircraft carrying these missiles.

Radiant heat ovens were specifically designed to simulate the temperature cycle of Fig. 7. A description of the oven design and the procedures are reported in NAVWEPS Report 7762 (Ref 3). Figure 8 is a 5-cycle thermal profile of a Sidewinder 1C sustainer grain.

Figure 9 depicts the thermal profile of a PBX explosive warhead to cook-off. Figure 10 indicates the extent of the exotherm developed in a PBX charge with two-day storage above 300°F (in this case the charge was cooled before deflagration could occur).

COOK-OFF TEMPERATURES FOR PROPELLANTS

The time to deflagration for various standard propellant grains has been measured for oven temperatures in the range 170° to 400°F. Various types of perforations were used with outside diameters of one to five inches. Samples were used with lengths greater than the diameter, and these samples were implemented with thermocouples and placed in simulated motor tubes. The tubes were placed in ovens at controlled temperatures until deflagration occurred. For oven storage times of 10 to 100 hours, the following results were obtained:

Propellant type	Range of cook-off temperatures, °F (For 5-inch diameter samples)
Polyurethane composite	300 to 400
Double-base	207 to 260

More than 200 deflagrations of double-base propellants ranging from slow fume-offs to sudden violent bursts of flame occurred in one oven without damage to the oven. This oven was destroyed in October 1958 (see Fig. 11), when a sample of polyurethane propellant weighing less than four pounds did not burn but exploded. Figure 12 shows the central cores of three cook-off ovens after explosions of 5-inch diameter 6-pound samples of cast-composite propellants.

The cook-off characteristics for a series of double-base propellant systems are presented in Fig. 13, and a summarization of cook-off characteristics of double-base and polyurethane-base propellants to 6-pound sample sizes is shown in Fig. 14.

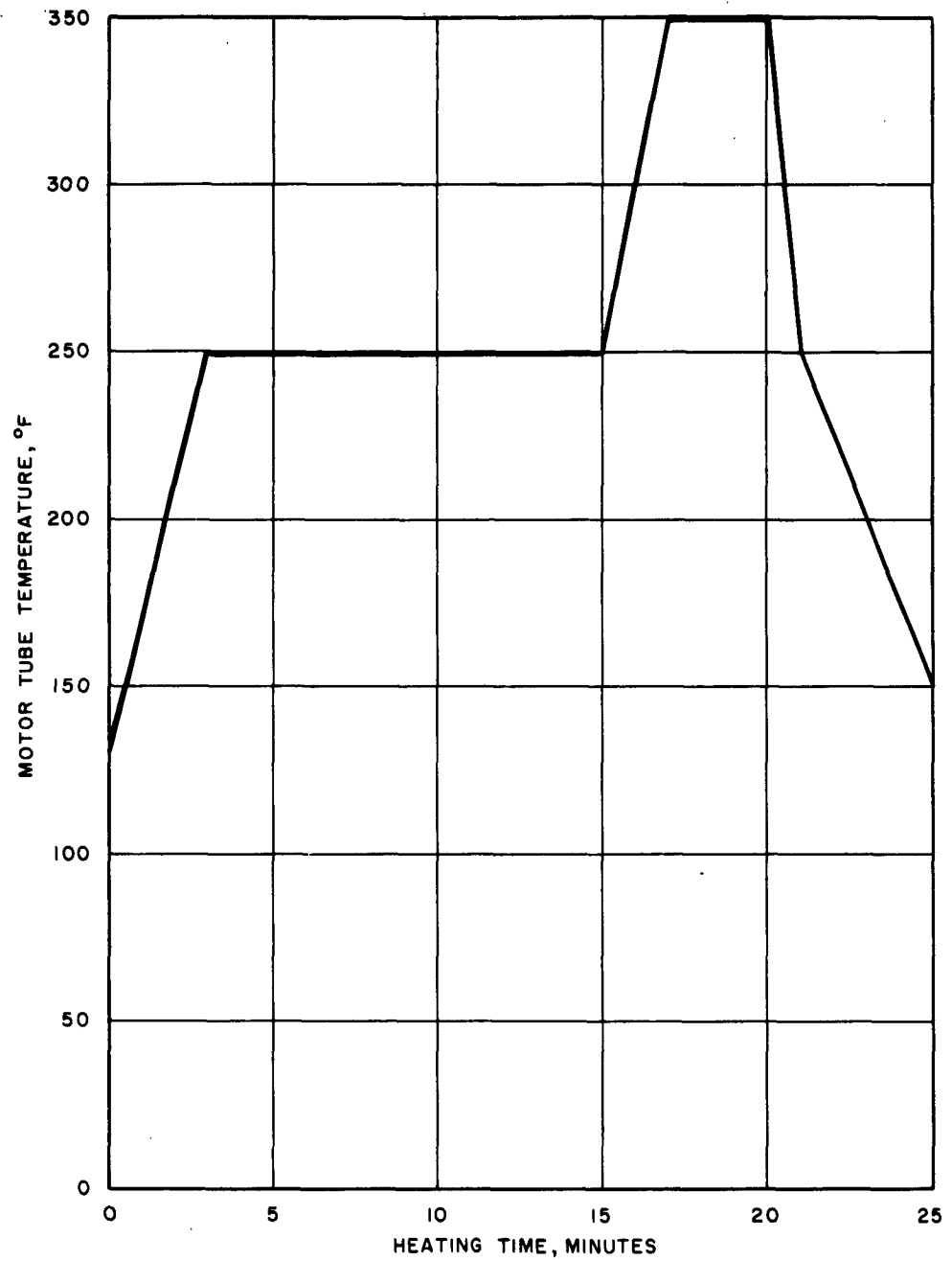


FIG. 7. Temperature-Time Profile Specification for Aerodynamic Heating Requirements.

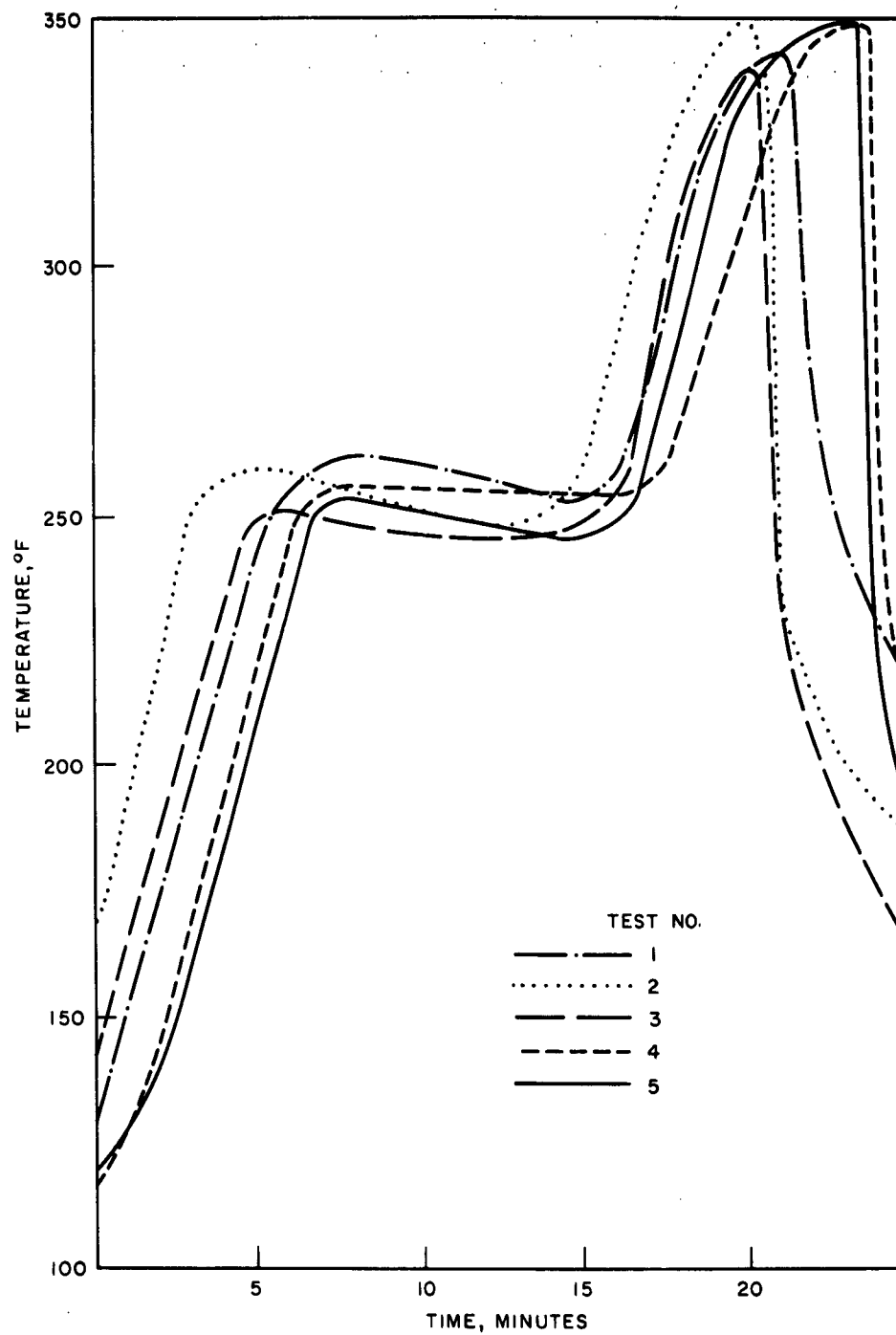


FIG. 8. Five-Cycle Thermal Profile of SW 1-C Sustainer Grain.

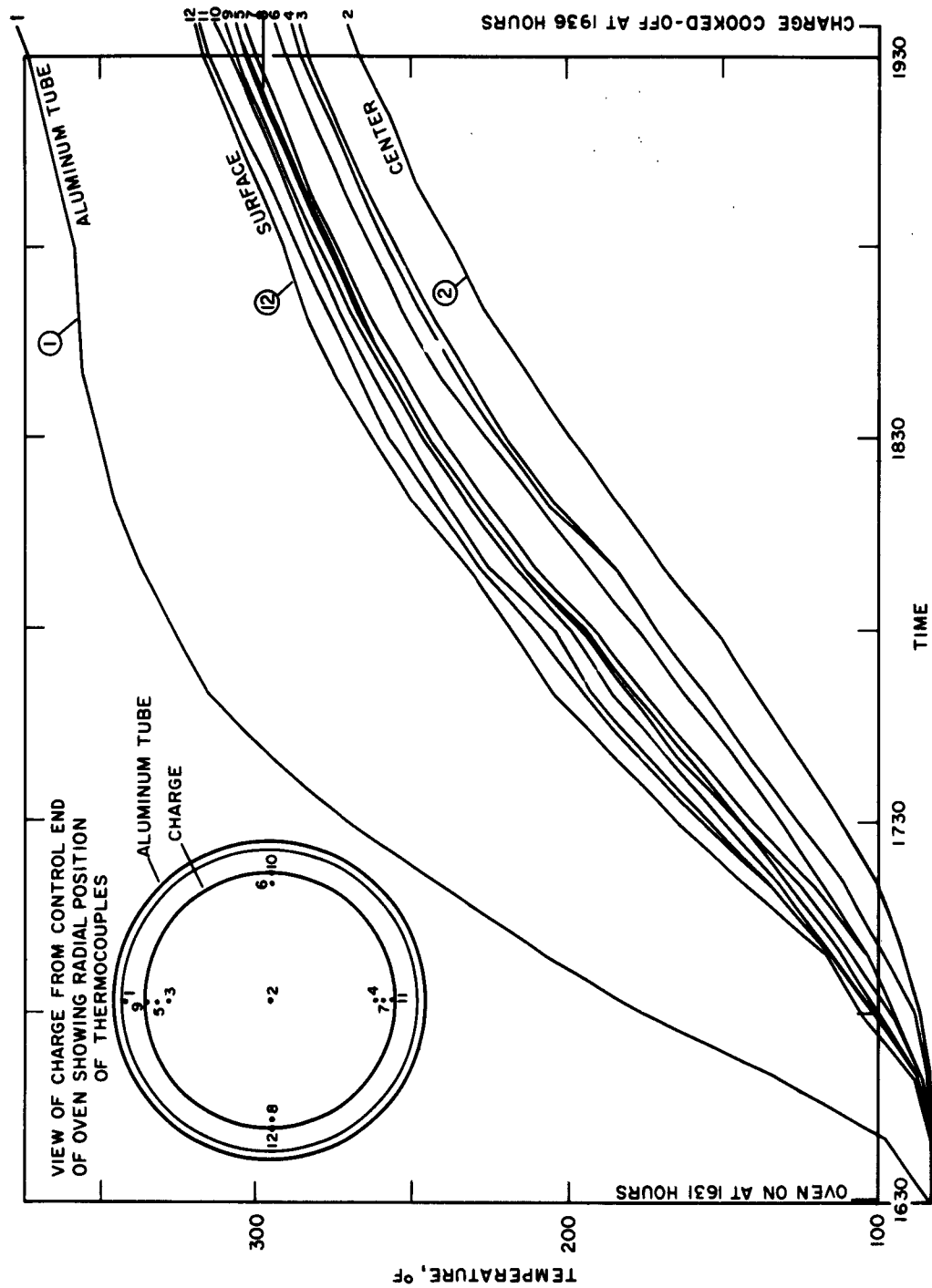


FIG. 9. Cook-off of PBX Charge. (4.0-Inch OD and 6.5-Inch Long Contained in 5-Inch Aluminum Tube. Twelve Thermocouples Located 1.625-Inch From End of Charge).

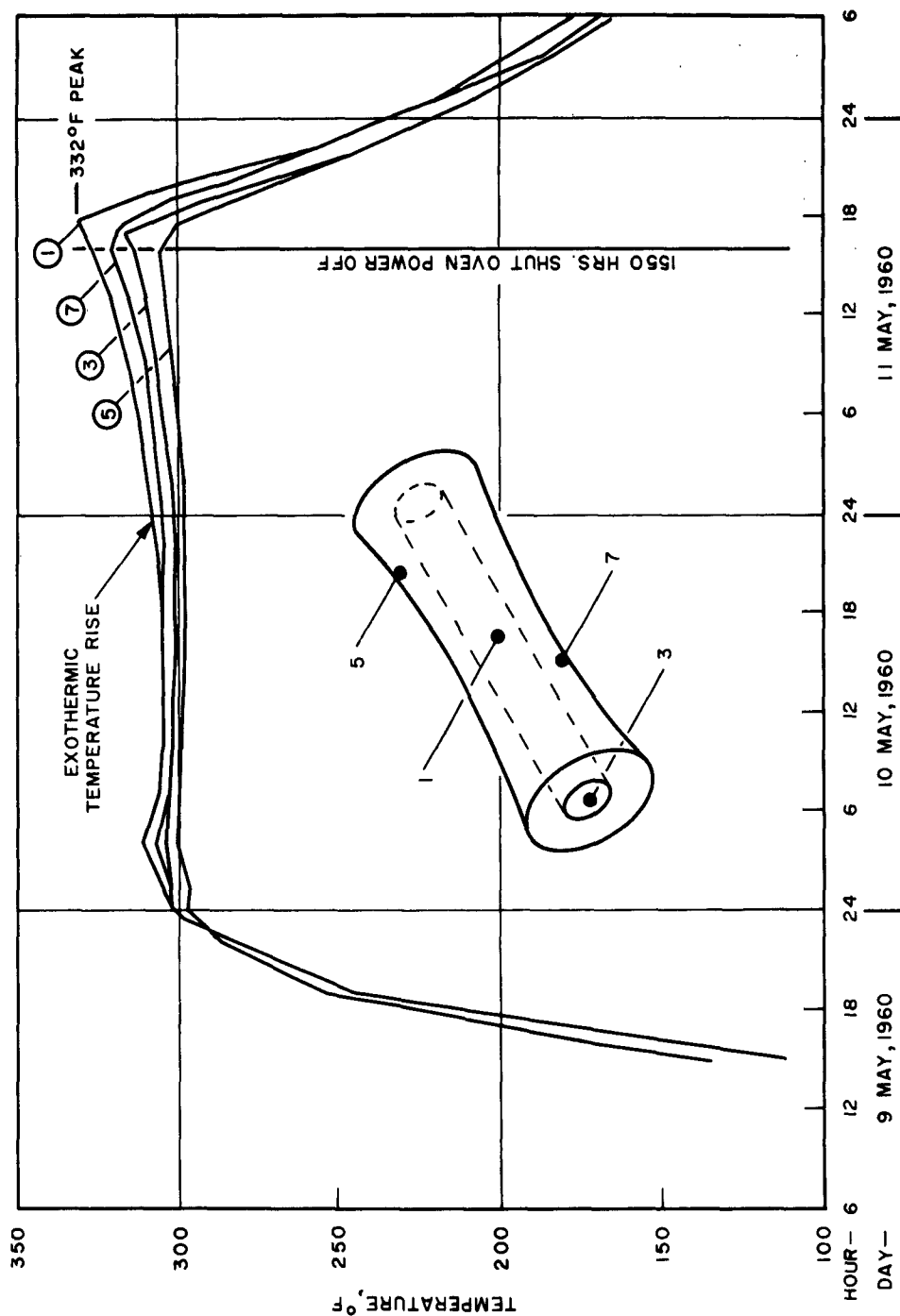


FIG. 10. Temperature Record of PBX Charge With Two Days Storage Above 300°F.



FIG. 11. Effect of Explosion in Cook-Off Oven,
24 October 1958.



FIG. 12. Central Cores of Three Cook-Off Ovens
After Explosions of 5.0-Inch Diameter 6-lb samples
of Cast-Composite Propellants.

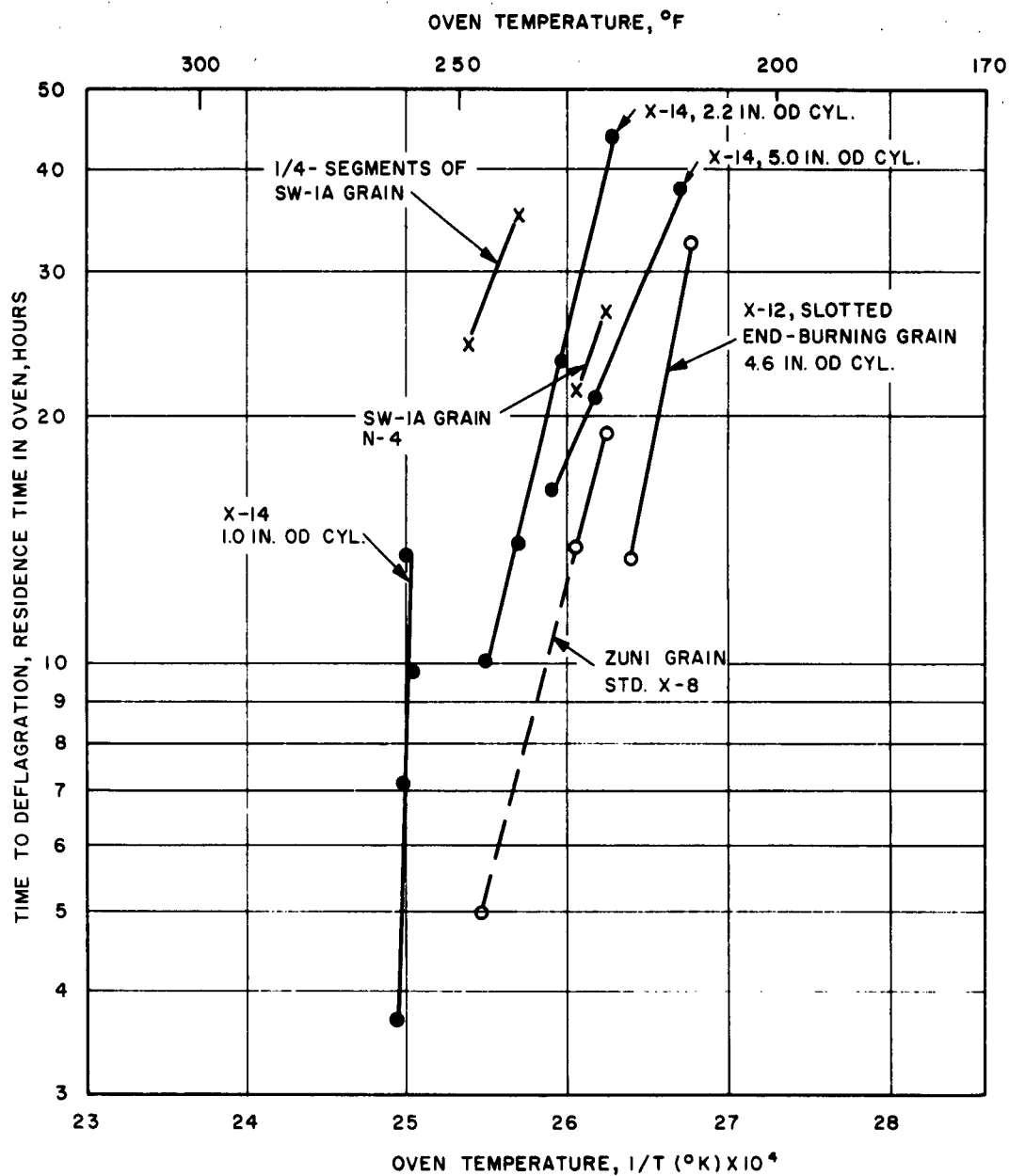


FIG. 13. Cook-Off Characteristics of Double-Base Propellants.

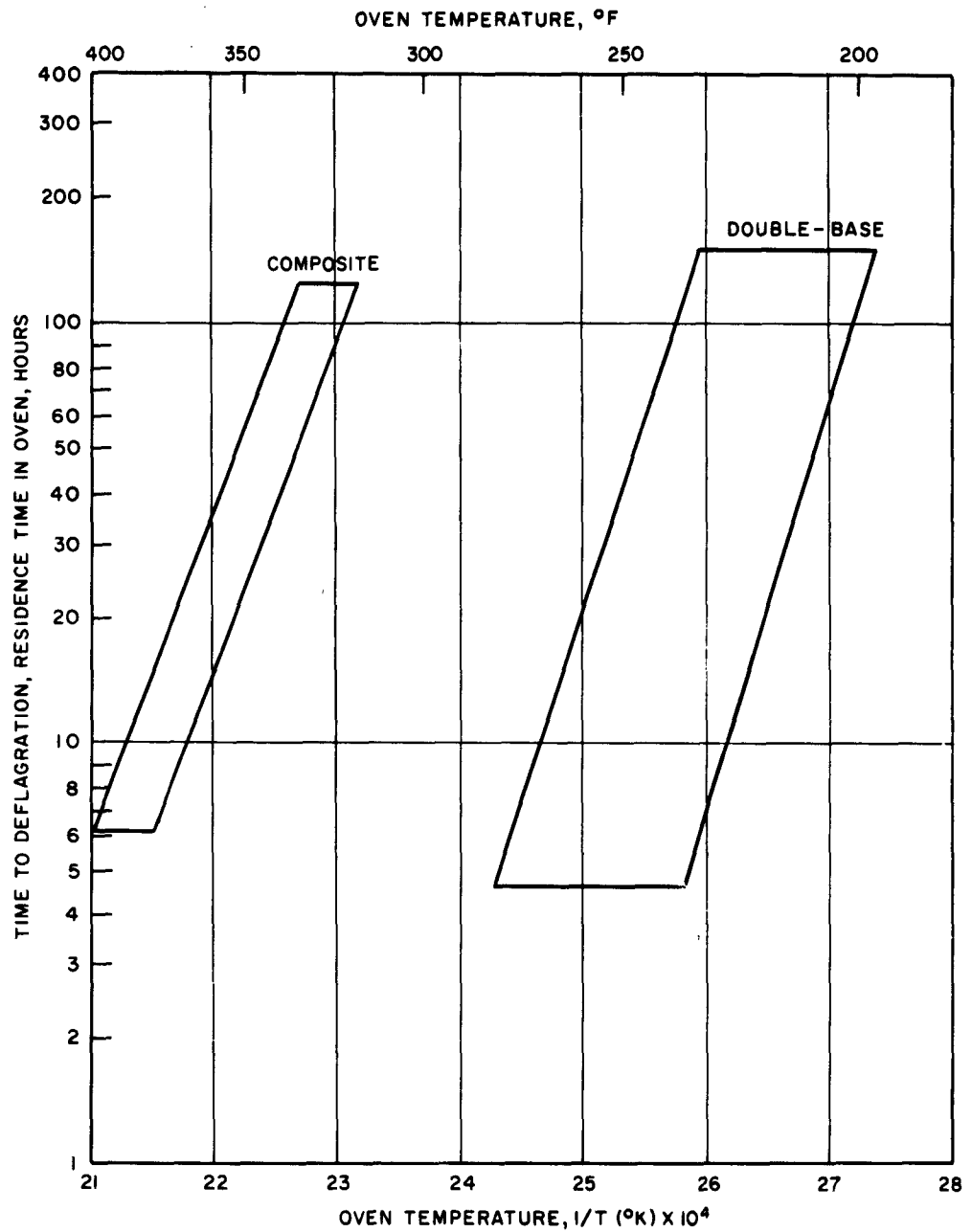


FIG. 14. Comparison of Cook-off Characteristics of Composite and Double-Base Propellants to 6-lb Sizes (5-Inch Diameter).

PREDICTION OF TIME TO DEFLAGRATION

As the size of solid propellant motors increases, it becomes less feasible to test destructively in order to learn characteristics of the propellant.

The temperature dependence of a given propellant system having been determined, then two approaches are available to apply the data to the prediction of residence-time safe-life at a particular temperature.

The first approach, recently elaborated by Longwell (Ref. 4), uses thermal parameters to predict the safe-life of a unit mathematically.

The second approach is more empirical and requires the study of various sizes of propellant charges at various temperatures.

The first correlation between theory and actual cook-off data is reported in NAVWEPS Report 7774 (Ref. 5). Figures 15 and 16 indicate the extent of the correlation.

Prediction of the critical temperature is predicated on the determination of activation energies of thermally-induced decompositions (DTA), together with other chemical and physical property data, by use of the theoretical calculations referenced.

DTA (differential thermal analysis) studies are presently made with small samples (milligram range) in which temperature measurement comparisons are made continually of the sample against a known reference while both are being heated at a constant rate.

Small changes in the sample temperature (endo- or exothermic) are easily detected in the differential thermocouple circuit and continuously recorded on a chart. DTA equipment is essentially a dynamic calorimeter, and the temperature at which heat effects (endo- or exothermic) occur and their relative intensities are characteristic of the materials being studied.

DTA techniques, method of analysis, etc., and data on several propellant systems and their components are reported in NAVWEPS Reports 7769 and 7914 (Ref. 6-7).

The data to date have shown promising correlation between theory and experiment as indicated in Fig. 17 where the points determined by experiment fall very close to the lines of prediction.

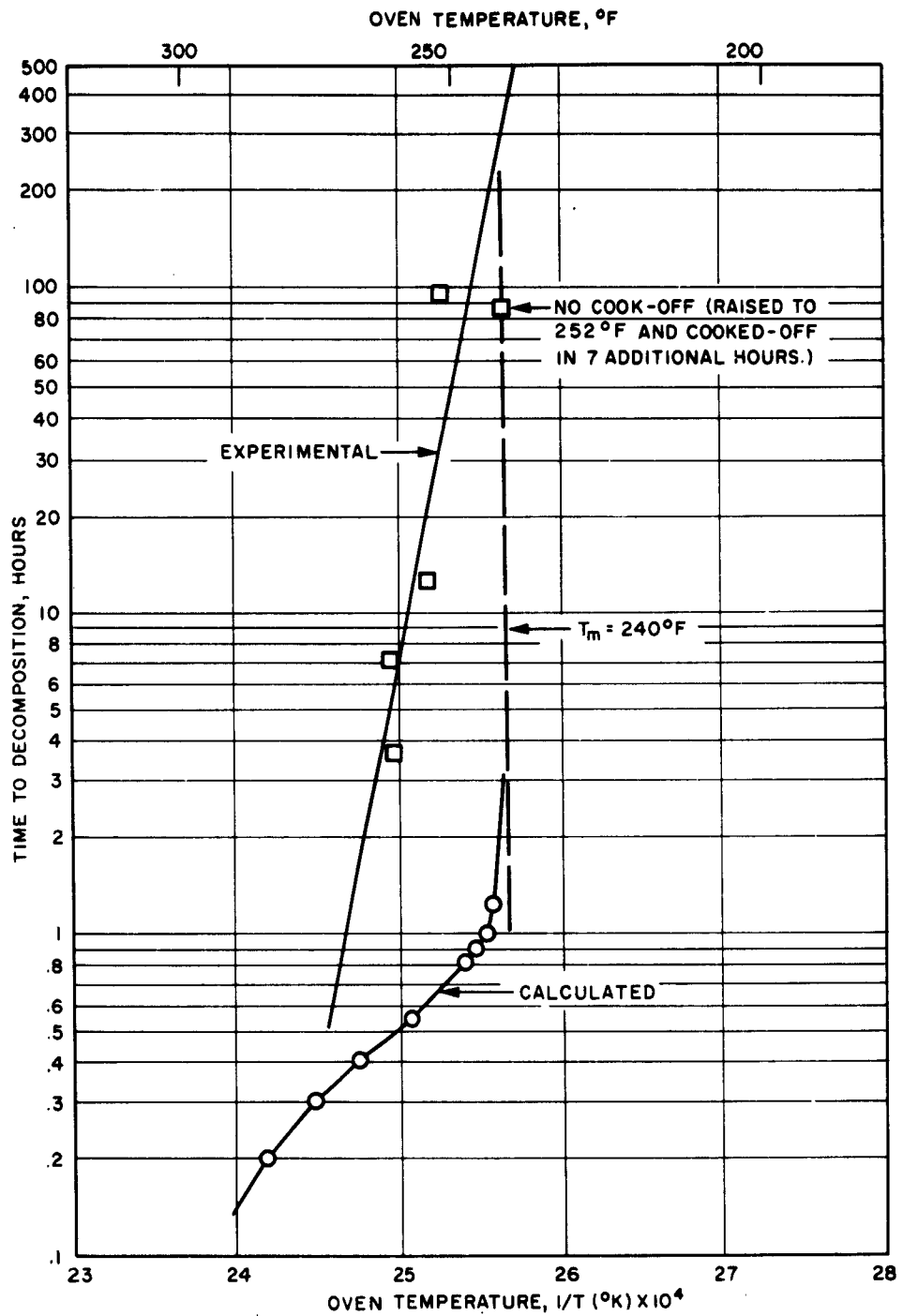


FIG. 15. Comparison of Predicted and Experimental Decomposition Data for 1.0-inch Cylinders of X-14.

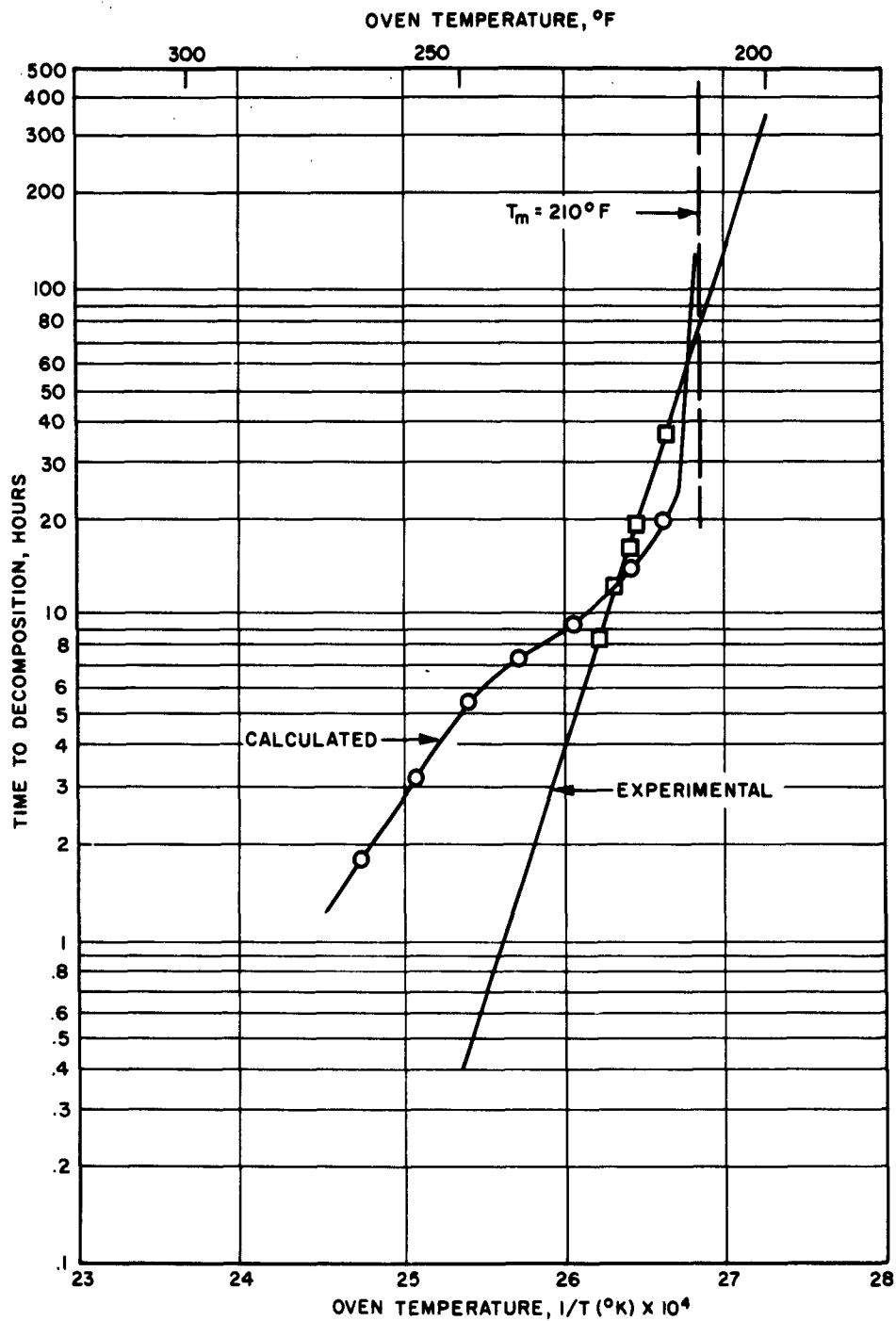


FIG. 16. Comparison of Predicted and Experimental Decomposition Data for 5.0-inch Cylinders of X-14.

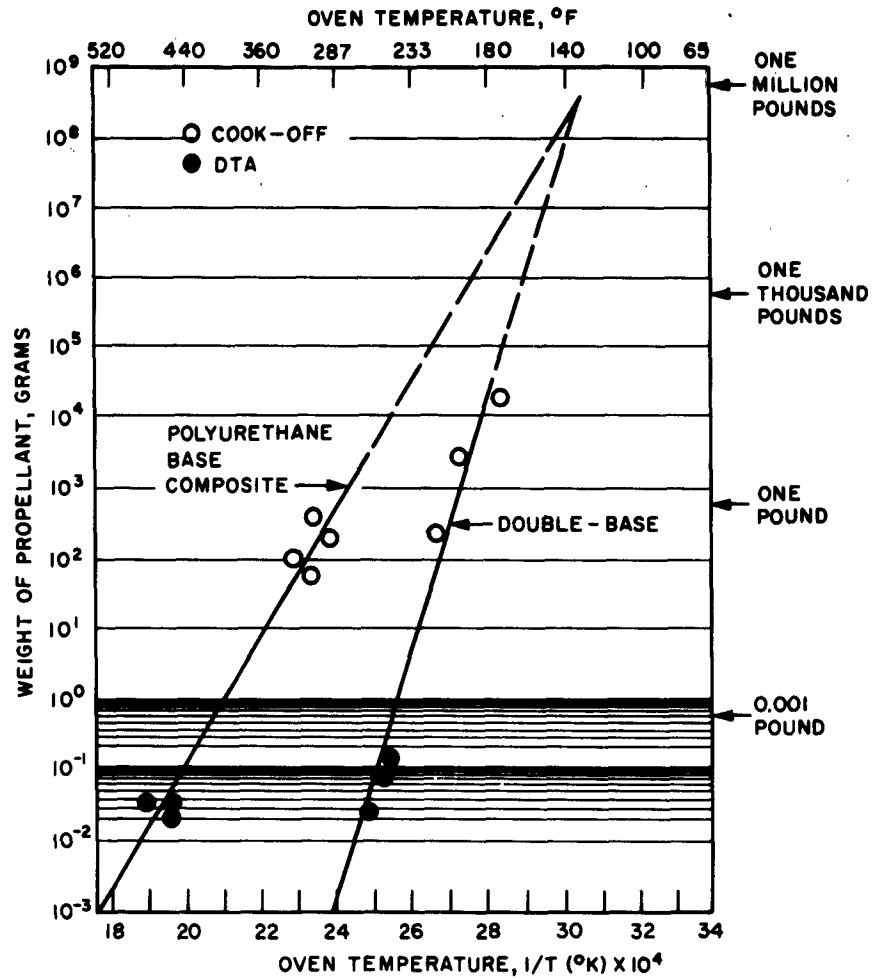


FIG. 17. Propellant Weight vs Critical Temperature for Cylinders with L/D Ratio Equal to 1.

ASROC TESTS

A program was set up to determine cook-off characteristics of the ASROC system. The grain diameter (11.25 inches) afforded the opportunity to scale-up the cook-off data, and to further check experimental data with theory.

OVEN DESIGN

An ASROC shell and cruciform grain combination were cut to make three sections, each 16 inches long. The weight of each "third" was 75 pounds. A tubular oven with inside diameter of 14 inches was wound with nichrome ribbon to make an oven core two feet long. This oven core was centrally mounted within a 55-gallon steel drum with insulation between the core and drum. A test pad was assembled from three 34-ton gun shields in a remote location. The oven was installed within the shelter of these gun shields. Recorder and controller for the oven were set along the power line 250 feet from the test pad. Figure 18 is typical of the barricade used.

TEST PROCEDURE

A "one-third" section of ASROC grain was taken to the test pad and fitted with 12 thermocouples. The assembly was then wrapped in three wraps of aluminum foil, 2.5 mils thick, and centered in the oven. Closure of the open end of the oven was made using vibradamp disks, one inch thick. Three vibradamp disks were placed in the inner core, and three in the end of the 55-gallon drum. The oven was about three-fourths buried in sand, but the end with the vibradamp disks was left free of sand in, the hope that the burning grain would be expelled from the oven.

TEST RESULTS

The controller was set to give an oven temperature of 222°F for Test No. 1. From previous oven tests, it was estimated that 72 hours would be required to bring the central part of the cruciform to within plus or minus two degrees of the oven temperature if no exothermic reaction developed. A plot from the first eight hours of this run yielded an estimate of 75 hours. Since this was the first ASROC cook-off test, no previous data were available to establish a temperature at which an exotherm could be expected for this configuration. During the course of Test No. 1, preliminary plotted data showed exothermic heat becoming evident at the 46th hour (Fig. 19) with the temperature of one thermocouple station at 212°F. The grain ignited and burned after 62.2 hours of continuous heating with the oven at 222°F, and the last print at the "high" station, No. 11, was 259°F. Since actual ignition requires a temperature above 310°F, obviously there were no thermocouples near the spot where the ignition occurred. From the evidence around the pad, it appeared that the ignition occurred at the back of the oven, thereby ejecting the burning grain from the oven.

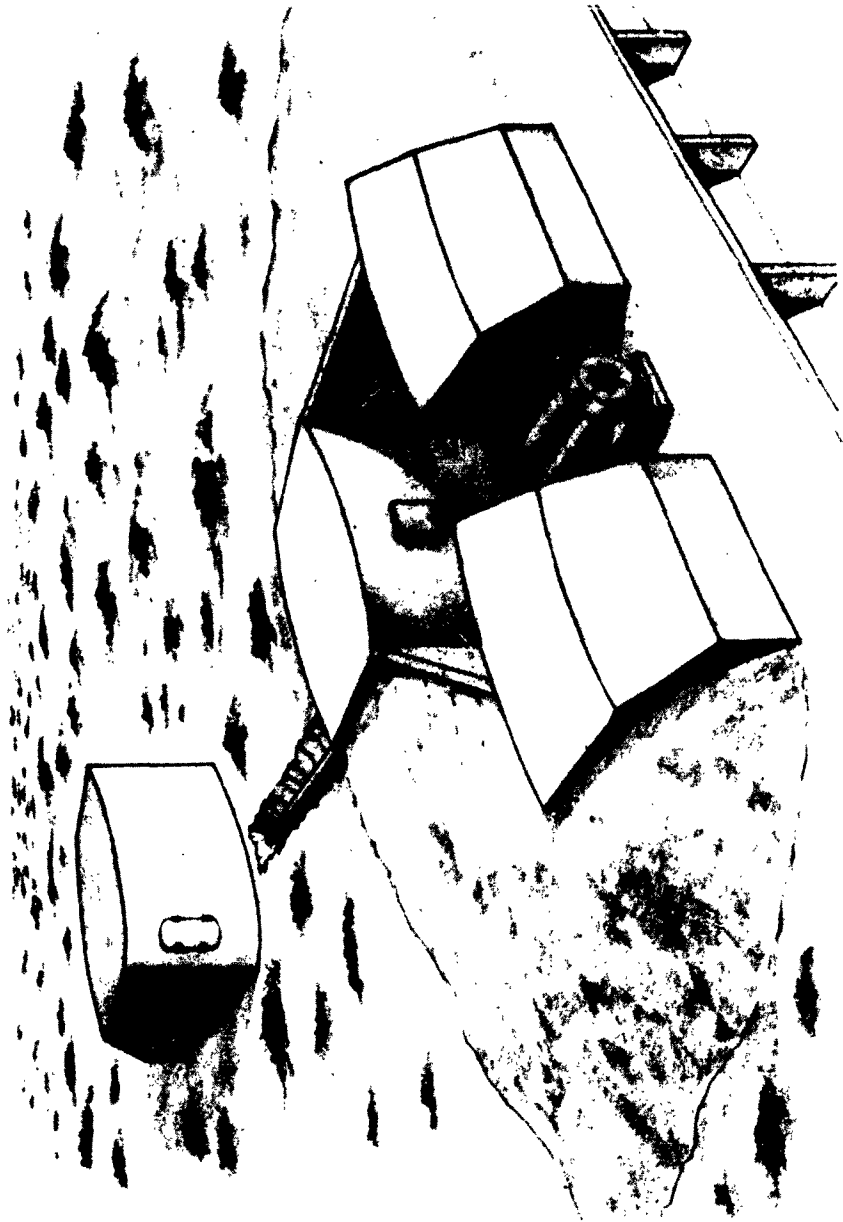


FIG. 18. Typical Barricade Used in ASROC Cook-Off Test.

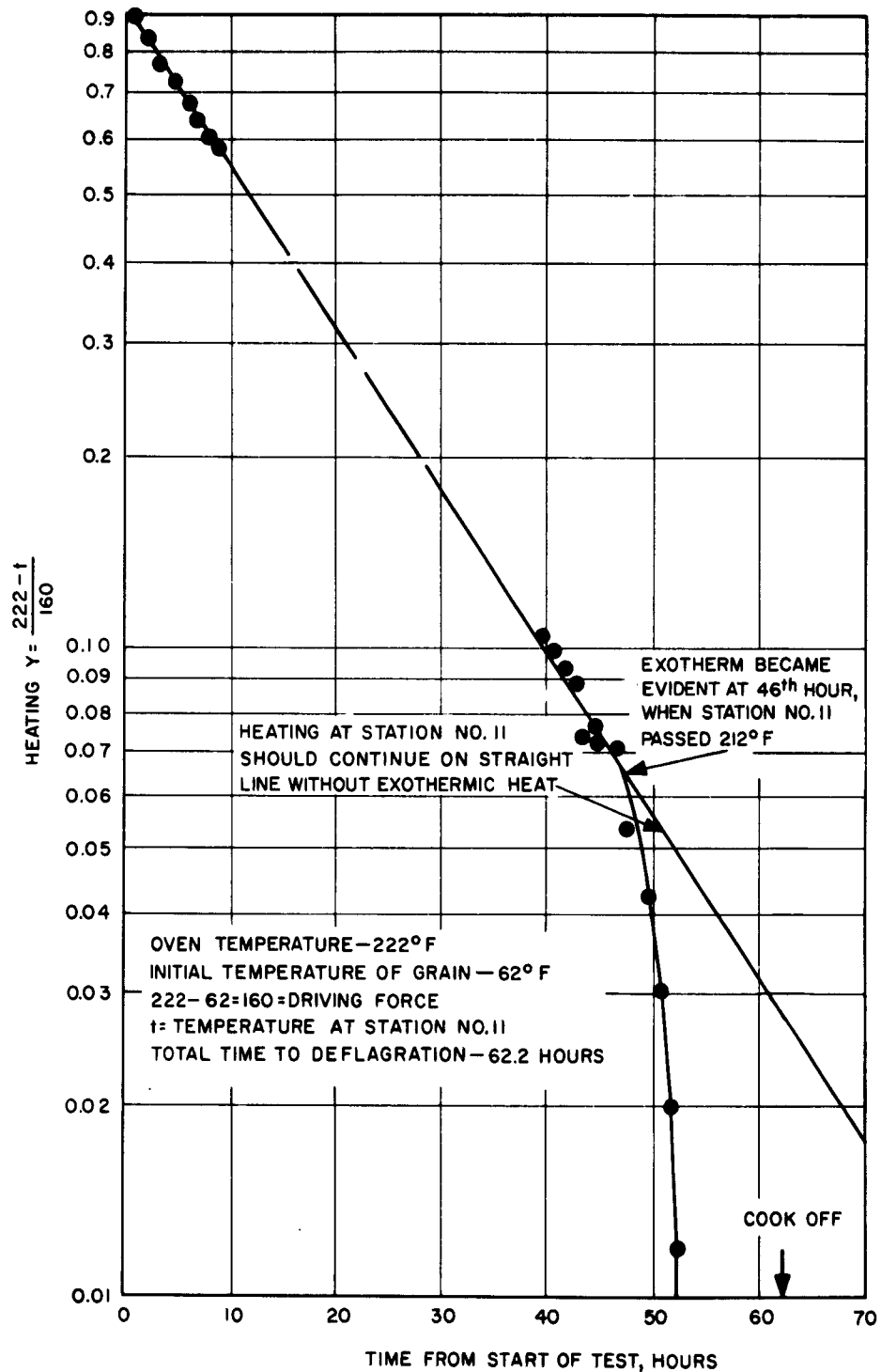


FIG. 19. Heating Curve for ASROC Test No. 1.

The oven was not damaged, and a new set-up was made with ASROC Test No. 2. Test No. 2 was similar to No. 1 in set-up, with some added precautions to avoid interference from stormy weather. Six inches of glass wool was introduced ahead of the vibradamp pad that closed the 55-gallon drum. A layer of asbestos was applied, then a layer of aluminum foil, and finally the whole assembly was buried with sand. For Test No. 2, the controller was set to yield an oven temperature of 250°F. This sample cooked off in 31.3 hours.

For Test No. 3, the controller was set to yield an oven temperature of 204°F, and the sample cooked off in 94.7 hours. Thirty-six thermocouples were used in Test No. 3 in order to get more detailed temperature data.

Figure 20 is a plot of the cook-off data showing time to cook-off in relation to the oven temperature. Figure 21 is a plot of temperature conditions at the center of the cruciform during Test No. 3 from the 25th to the 60th hour of heating. It shows evidence of an exotherm at the 42nd hour of heating, although the deflagration did not occur until 52.7 hours later, or at the 94.7 hour from start of test.

During the course of Test No. 3 at 204°F oven temperature, there was time for the center of the grain to develop the dominant exotherm. At 250°F oven temperature the center of the cruciform was relatively cool with respect to temperatures elsewhere in the grain when the deflagration occurred. From these data, and other experiments, Table 2 is presented as a temperature profile for the ASROC propellant grain system.

SUMMARY AND CONCLUSIONS

Cook-off ovens were designed capable of withstanding repeated deflagrations of samples weighing up to 5 pounds. Propellant samples were then tested at oven temperatures at which samples would deflagrate within 1 to 5 days. A plot of log time-to-deflagration versus the reciprocal of the absolute temperature proved to be a straight line. The experimental cook-off technique was used as a rapid means of evaluating the relative effect of various stabilizers in nitrasol propellant. Cook-off characteristics of various propellant types are reported. The effect on propellant and warhead explosive of the short-term, high-temperature, aerodynamic heating profiles was explored. The experimental method was applied to the complex configuration of the ASROC grain, and its characteristic cook-off curve was established.

Experimental data obtained through cook-off procedures gave experimental verification of theoretical predictions of critical temperatures based on results of differential thermal analysis.

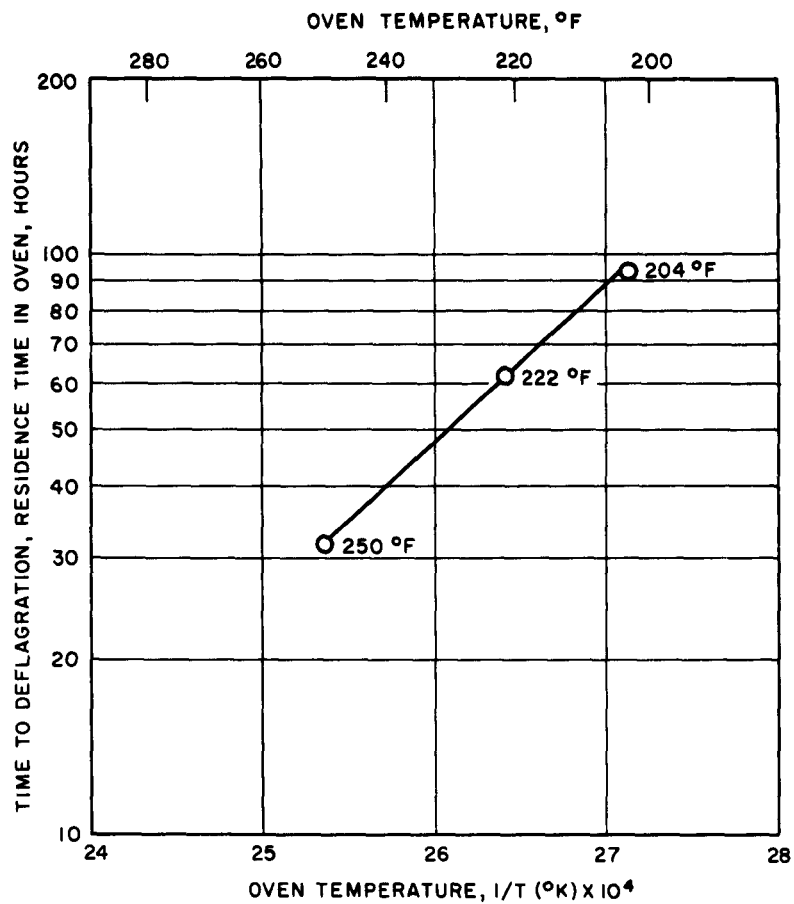


FIG. 20. Cook-off of ASROC Grain.

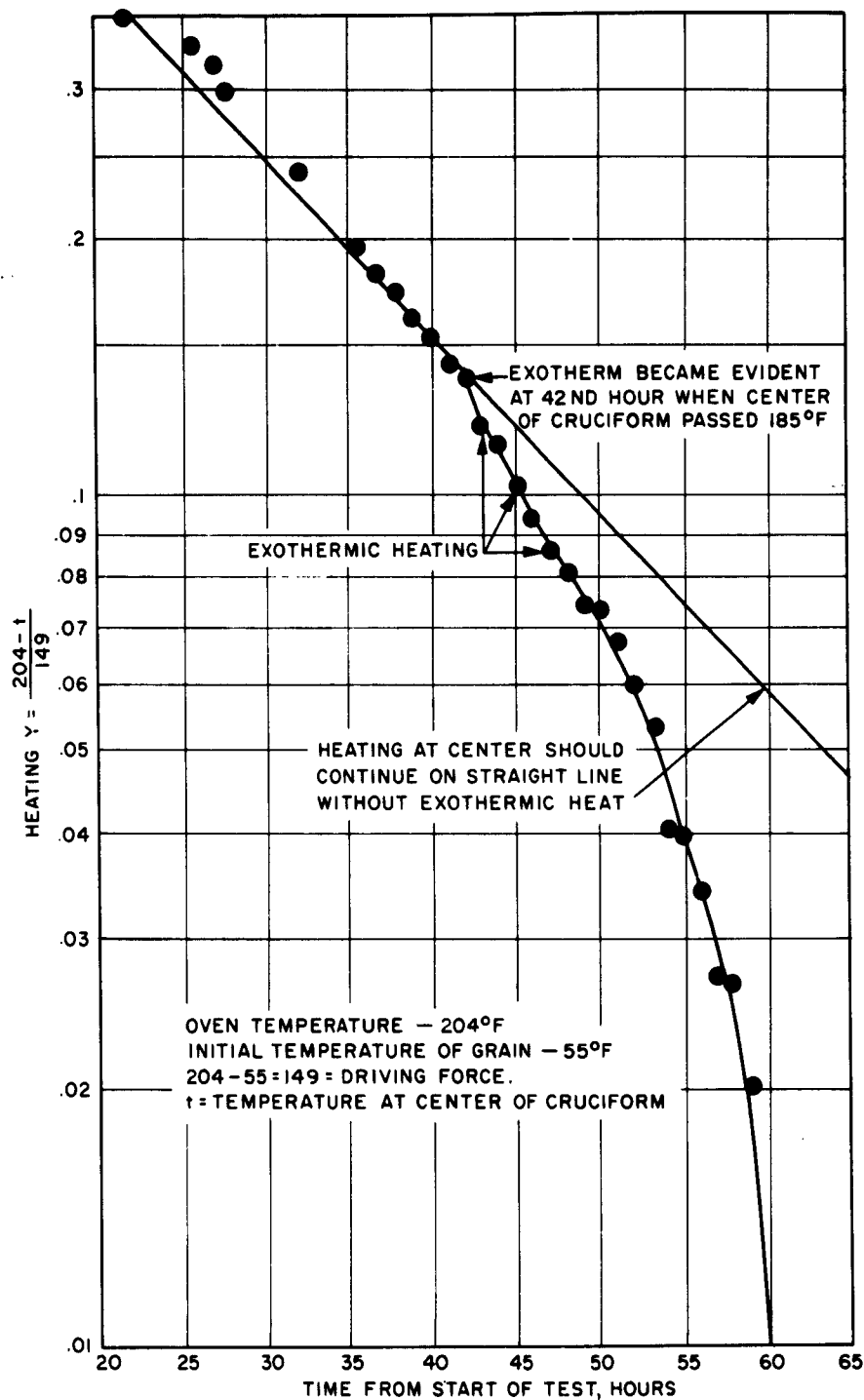


FIG. 21. Portion of Heating Curve at Center of Cruciform Grain in ASROC Configuration.

TABLE 2. Temperature Profile

Oven storage temperature, °F	Temperature at center of grain, °F	Exotherm at center of grain, °F	Time to deflagration, (t _e) hours	Remarks
40-112	---	---	---	Allowable launcher temperature limits
165	165	0	---	Internal gassing causes fissures in grain in 10 days
171	172	1	---	
183	186	3	---	
190	196	6	---	
195 ± 5	(a)	(a)	(b)	Critical temperature
204	---	---	94.7	
222	---	---	62.2	
250	---	---	31.3	

^aAt the critical temperature, the exothermic heat at the center of the grain will cause an exponential rise in grain temperature until deflagration occurs. The time to deflagration or explosion (t_e) decreases with increasing storage temperature.

^bThe critical temperature is estimated to be 195°F ± 5°F.

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