NASA TECHNICAL
MEMORANDUM

NASA TM X-53106

AUGUST 13, 1964

NASA TM X-53106

CROSS CHECK STUDY OF THERMAL-VACUUM WEIGHT LOSS DETERMINATIONS FOR SELECTED POLYMERS

by C. T. EGGER AND J. B. GAYLE
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XEROX $ 2.00 FS
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ABSTRACT

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MATERIALS DIVISION
PROPULSION & VEHICLE ENGINEERING LABORATORY
RESEARCH AND DEVELOPMENT OPERATIONS

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SUMMARY

Results of a cross check study of the vacuum compatibility of selected polymers are presented. MSFC data for polystyrene and polymethylmethacrylate are compared with results obtained by the National Bureau of Standards. The major finding was that temperature measurement methods are subject to unexpected errors and, thus, require careful evaluation.

INTRODUCTION

Thermal-vacuum weight loss studies are important tools in the development and evaluation of materials for launch and space vehicles. However, experimental test results for nominally similar materials under supposedly similar conditions frequently disagree, and actual behavior in space has been known to differ from that predicted, e.g., Echo I. Thus, a method of test standardization and comparison is needed.

As a part of a recent investigation of polymeric materials made on a crash basis, it was necessary to determine vacuum compatibility at temperatures appreciably in excess of those normally used at this Center. Comparison of the results with literature values suggested major discrepancies. Therefore, the extent and cause of these discrepancies were investigated by a series of cross check determinations carried out in cooperation with the National Bureau of Standards.

Polymers initially selected for this study were polymethylmethacrylate, polystyrene, and polytetrafluoroethylene. Test temperatures were 260°C for polymethylmethacrylate; 330°, 340°, and 360°C for polystyrene; and 520°C for polytetrafluoroethylene. These temperatures were in the range where difficulties had been encountered in the previous MSFC investigation but were within the range of normal operating temperatures for the Bureau of Standards. The polymers selected were known to have thermal degradation rates at these temperatures which permit precise measurement in a reasonable time period.

The project was initiated early in 1963, and the first series of determinations was completed in November 1963; the second series was completed in December. This report presents a detailed discussion of the results. Also included are the results of a related kinetics simulation study of the effects of heatup mode and temperature fluctuations on weight loss histories.

PRELIMINARY STUDIES

The first series of determinations was made without major modifications of the test apparatus or procedures.

Table I summarizes the experimental guidelines and the material characteristics. Fifteen experimental runs, consisting of triplicate determinations for each polymer/temperature combination, were planned for the first series of tasks. As indicated above, the temperatures selected were higher than those normally used for materials weight loss studies in this laboratory, and, as a result, equipment was repeatedly damaged, especially the electrobalance assembly. Subsequently, it was learned that erroneous temperature readings (and, consequently, erroneous weight loss rates) resulted from these attempts to operate at temperatures above those at which the equipment was designed to operate. This experience and also safety considerations dictate that this division forego the polytetrafluoroethylene (520°C) studies.

Equipment

The apparatus and procedure used by the Bureau of Standards have been described previously (Ref. 1-6). A brief comparison of the equipment and procedures with those of MSFC are included in Table II.

With the exception of polymethylmethacrylate run number 1, which was made on a similar system, all MSFC runs were made on the "4D" vacuum system shown in FIG 1 and 2.

As shown in FIG 1, the Cahn Model RA electrobalance was mounted within the bell-jar chamber. The Sargent Thermonitor, normally used for temperature control, was replaced by a Variac for these studies. A recorder, the Cahn balance console, the ionization gauge controller, and the thermocouple gauge were housed in a separate cabinet. Auxiliary recorders, as required, were employed for the test. The liquid nitrogen fill line for the cold trap was barely visible. The bell-jar safety shroud was not in place for the photograph.

The bell-jar contents are shown in greater detail in FIG 2. The photocell assembly of the Cahn balance was damaged by overheating during calibration, causing a delay in the program. The small cup contained ballast, and finer counterbalancing was accomplished by adding milligram-range weights to right-hand pan. An aluminum hemisphere containing the sample, illustrated by the foam specimen in the photograph, was placed in a central location in the lava oven. (A split lid with a 1/4-inch diameter orifice was placed over the oven during the tests.) The permanent heat shields were supplemented with several pieces of strategically placed aluminum foil, and thermocouples were placed near critical points to permit continuous monitoring of balance and bell-jar temperatures. Balance arrestment was provided by the caging shaft, and the pumping port was visible just below the right-hand balance pan.

The vacuum system consisted of a 13 cfm Welch mechanical pump and a NRC H-4-P four-inch diffusion pump rated at 340 liters per second maximum pumping speed.

Weight was monitored continuously by means of a Cahn Model RA electrobalance having a capacity of some 200 grams and a rated sensitivity of 0.1 mg at 100 milligrams. Calibration to a combined recorder-balance accuracy of more than 0.5 milligram for a full-scale deflection of 100 milligrams (0-10 mv.) was maintained.

The Vacuum Instruments Company ionization gauge for low pressure measurement was operated at the emission current recommended by the tube manufacturer. The ionization gauge tube was attached to the system between the bell-jar and the cold trap, approximately six inches below the pumping port.

Temperature control and measurement are discussed in the following section. The oven interior was closely represented by a 2-1/2" ID x 2-1/2" cylinder, and the thermocouple used for temperature control and measurement during the first series was located on the oven axis about 1/4-inch below the specimen holder. Power to the 250-ohm oven was supplied at a constant voltage to maintain the desired temperature level.

The system was originally designed to achieve 10^{-8} torr with metal gaskets and bakeout of a stainless steel bell-jar. Without bakeout and with a Pyrex bell-jar and neoprene gaskets, the ultimate vacuum is approximately 5×10^{-7} torr.

Experimental Procedure

Before initiation of the MSFC experimental runs, all environment-sensing elements of the apparatus were calibrated. Recorders were calibrated by Minneapolis-Honeywell personnel and were checked in the laboratory against a potentiometer to maintain an accuracy and precision of better than ± 0.5 percent. No calibration beyond use of the emission current recommended by the manufacturer was available for the ionization gauge tubes used for these tests. The electrobalance was calibrated using standard analytical weights according to the manufacturer's instructions, and the ± 0.5 milligram calibration tolerance for 100 milligrams full-scale was allowed to drift until periodic rechecking showed that it had reached 1.0 mg, at which point recalibration was required. (Experience has shown balance/recorder linearity to fall within this accuracy.) The sensing thermocouples, checked periodically and found to have little drift, were last calibrated approximately three months before the studies and were found to be within 2°C of the standard EMF table values up to 300°C, which was the highest temperature checked.

Meanwhile, all specimens were stored away from heat and light in colored bottles, in nitrogen, within nitrogen-filled plastic envelopes, just as they were received. During transfer to the apparatus, they were exposed to ambient conditions (40-55% humidity at 75-80°F) for less than five minutes.

No special fabrication or modification of specimens was required; additions were made to the weighed, 1/2-inch diameter, 0.010-inch aluminum foil hemispherical sample cup (FIG 3) until 65 milligrams were exceeded. The sample and cup then were weighed and placed within the oven on a 16-gauge silver solder wire planar spiral which served as the specimen support (FIG 3). This support was suspended from the balance through the 1/4-inch diameter hole in the oven lid by an extension of the spiral material.

After the specimen was in place, the bell-jar and safety shield were added, and the system was evacuated. When the pressure dropped below 10^{-5} torr, the five-minute temperature elevation cycle was initiated so that the oven temperatures slowly approached the preconditioning temperature, where it was held for a minimum of 12 hours. Table II describes the time and temperature conditions required for the various specimens. Weight changes during the initial heatup and the preconditioning periods were insignificant in relation to the balance/recorder error tolerances stated above.

Near the end of the preconditioning period, liquid nitrogen was added to the cold trap to aid in rapid removal of volatilized fragments during the subsequent weight loss study at elevated temperature. After the trap was filled, the recorder speed was increased, and the balance zero was checked. Then the temperature was raised to the specified run temperature as rapidly as possible; usually less than five minutes were required for reasonable stabilization. Frequent Variac adjustments were required to maintain the desired temperature level, particularly during the early phase of the run.

At the end of the run, the oven was cooled, and the system was vented to the atmosphere. The sample cup and any remaining specimen material were removed and weighed, providing a before-and-after check on the electrobalance accuracy.

Results

The experimental results obtained at this laboratory are shown as the dot-short dash (.-.-) lines and those by the Bureau of Standards as solid lines in FIG 4 through 7. Inspection of these figures indicates that the MSFC data exhibited weight loss rates greatly in excess of those determined by the Bureau of Standards. The unexpectedly large error in this laboratory's data appears to have been due to one predominant factor, temperature measurement. Although the slopes for duplicate runs were similar, a number of factors in addition to disagreement with Bureau of Standards results indicated that the data should be questioned. Thus, the temperature for the polystyrene 340° and 360°C runs did not reach the desired levels until most of the material had vaporized. The rapid evaporation rate frequently caused the ion gauge to shut off, resulting in such rapid weight changes that the recorder printed only about three points between 20% and 80% weight loss. Furthermore, the molecular conductance from the oven into the bell-jar probably restricted the rate of loss for cases where such rapid evaporation occurred. Finally, no reasonable activation energy calculation was possible using the polystyrene data.

NOVEMBER MEETING OF PARTICIPANTS

A meeting was held at the Bureau of Standards to discuss the problems which had arisen and to analyze the data. The consensus was that temperature measurement errors were the predominant cause for the extreme variability of the data. The magnitude of such errors was indicated by a brief kinetic analysis in which activation energies and frequency factors were calculated from NBS data. Using these values in the Arrhenius equation with the experimental weight loss data, it was estimated that actual sample temperatures differed by as much as 50°C for the laboratories.

ADDITIONAL STUDIES

A further result of the review of initial results was the decision to undertake a second series of studies using only polystyrene at 330° and 350°C. These studies were to be initiated only after remedial action to improve MSFC temperature measurement capability.

The equipment and techniques, except for temperature measurement, were practically identical to those of the first series. Before initiation of the studies, temperature gradients within the oven at elevated temperatures were studied and found to be appreciable, thus confirming indications drawn from comparison of initial results.

Experimental

In an effort to rectify thermal problems, the sample holder was changed to a suspended ring of 38-gauge stainless steel wire supporting a 1/4-inch diameter, 0.010-inch hemispherical aluminum cup which contained the specimen. Of greatest importance, however, is the fact that temperature for the second series of tests was measured by a 0.002-inch copper-constantan thermocouple placed so that when the sample melted it engulfed the junction during the test. A typical result from the second series was a reading of 330°C on the copper-constantan thermocouple on the molten sample; the adjacent thermocouple (used to indicate "oven temperature" during the first series) gave a corresponding value of 265°C, and a thermocouple near the bottom of the oven gave a value of 185°C. These thermocouples were calibrated in a sand bath against an ordinary laboratory thermometer and are believed to indicate actual temperature within ± 3°C based on standard EMF tables.

Results

The data obtained from the second series of studies (FIG 8 and 9) represent a vast improvement over first series results in terms of agreement with Bureau of Standards data, amenability to reasonable kinetic analysis, and reproducibility.

The slight variations in the slopes of the curves in FIG 8 and 9 probably resulted from brief temperature control lapses. It is particularly significant that smoothed-curve slopes after one-half hour are practically identical for each of the three 330°C runs and again for each of the three 350°C runs. Using these smoothed slopes, a zero order activation energy of 39 kilocalories per mole was calculated.

The above considerations plus the application of direct-contact, calibrated temperature measurement indicate that the second series of results, shown in FIG 8 and 9, probably are as accurate as possible for polystyrene under the conditions imposed by the guidelines used for this investigation. They also confirm the previous indication that initial difficulties were due solely to operation beyond the range of conditions for which the equipment and procedures were designated.

KINETIC SIMULATION STUDY

During the experimental phases of this investigation, the effects of rate and mode of heatup on the weight loss curves became a matter of concern. Thus, the fact that the time required for heating the sample from the conditioning temperature to the run temperature was always an appreciable portion of the total test time led to speculation as to the importance of this variable.

Also, the effects of fluctuations in sample temperature, particularly at the beginning of each test, were of interest. Because these problems did not lend themselves to direct solution with the experimental equipment on hand, it was decided to carry out a relatively simple kinetic simulation in which analog computation would be used to study the importance of these variables.

A general review of pyrolysis literature indicated that at low temperatures polystyrene tends to decompose by chain scission to produce monomer or dimer units from end units of the polymer and, thus, give zero order weight loss. As temperature increases, the order becomes higher and more complex. Near-linearity of the data, as plotted in FIG 8 and 9, suggested that under the conditions of this investigation the process could best be described by the use of a zero-order kinetic equation.

The procedure consisted of substituting selected temperature-time functions into the zero-order Arrhenius equation,

$$dw/dt = Ae^{-(E_a/RT)}$$

where:

dw/dt = Weight loss rate

A = Arrhenius frequency factor

E_a = Activation energy

R = Gas constant

T = Absolute temperature

and determining the corresponding weight loss-time histories. Values used for A and Ea were 4.8×10^{-11} percent per second and 39 kcal per mole, respectively, and were estimated from the experimental data given in FIG 8 and 9 after smoothing.

Figure 10 shows the effect of ± 3°C constant temperature errors for both 330° and 350°C runs. The deviations shown would more than mask the combination of all mechanical and non-thermal instrumentation errors expected, a situation which lends further credence to the error analysis of results for the first series of tests.

The effects of sinusoidal ± 5°C temperature oscillations having a period of one hour are shown in FIG 11. The dashed lines represent the weight loss behavior expected without these oscillations. It is noted that the solid lines lie about the dashed lines because integration of the exponential terms over a period of time gives greater weight to higher temperatures and yields a more rapid weight loss than would maintenance of strictly isothermal conditions.

Figure 12 shows results of four modes of heatup, all starting at room temperature and becoming constant at 330°C. Figure 13 is similar except that the final temperature is 350°C.

Mode 1 simulates instantaneous attainment of programmed run temperature and, thus, predicts immediate attainment of a steady rate of weight loss.

Mode 2 represents an asymptotic approach to run temperature at such a rate that the temperature is within 10°C of the programmed value in 150 seconds, thus representing the heatup mode expected from a proportional-type controller. The resulting weight loss curves lag behind those for mode 1 heating but attain the same weight loss rate upon reaching the programmed temperature.

Mode 3 features a linear heatup with a damped sinusoidal oscillation upon attainment of the desired temperature. The overshoot is initially 10°C and practically disappears after about five 1-minute periods. Only slight fluctuations result in the corresponding weight loss curves, and, again, the final slopes are the same as before when the temperature becomes stabilized at the equilibrium level.

Mode 4 is characterized by the same linear heatup as mode 3 but does not have the subsequent damped oscillations. The weight loss curves were similar to those for mode 3.

All calculations were performed on an electrical analog computer where the heatup curves were programmed as the corresponding differential equations; the temperature integral for these was used as input to the Arrhenius equation. Integration of the latter provided the weight-time curves shown in FIG 12 and 13.

From the foregoing, it can be seen that the heatup mode is important in determining the amount of weight that is lost after a given time but relatively unimportant in its influence on the weight loss rate and subsequent kinetic analysis. This treatment has provided an estimate of the influence of constant thermal errors and of fluctuations about the programmed temperature level. It is noteworthy that symmetrical fluctuations do not result in a cancellation of errors because the rate behavior is an exponential rather than linear function of temperature. Although all of these findings would be anticipated without the computer study, this technique provides a graphic, reasonably accurate picture of the magnitude of such effects.

SIGNIFICANCE OF MECHANISM OF WEIGHT LOSS TO THE METHOD OF REPORTING RESULTS

Because of the different methods of reporting weight loss data noted during review of recent literature, it is considered appropriate to restate this division's position on the matter.

Vacuum pyrolysis of polymeric materials of multimolecular or greater thickness involves three major mechanisms: degradation, diffusion, and evaporation. Further, for those materials degrading at a temperature above the melting point, convection can supplement diffusion in transporting material to the specimen surface. The rate of weight loss, measured in experiments such as the present study, is influenced by all three but may be controlled by any combination or permutation of the mechanisms, except that evaporation is always the final step in the process. The presence of plasticizers and/or solvents within the polymer further complicates the process.

For a degradation-controlled process, ordinary chemical kinetics often serves as an adequate mechanistic representation although diversity of fragmented species and inhomogeneity of the matrix introduce complications. Simple integral-order chemical kinetics probably is most often chosen for describing polymer pyrolysis, but such a treatment cannot be justified unless it can be shown that mass transport (diffusion and evaporation) offers negligible resistance to weight loss.

Diffusion-limited cases must be described in terms of overall specimen geometry, and their behavior is highly dependent on material thickness. Reporting weight losses as rates (weight change/unit time) in such instances is not sound practice because the compositions of the diffusing material and the surrounding matrix vary with time, particularly for heterogeneous materials. In this sense, polymers containing species of varying chain length must be regarded as inhomogeneous.

For evaporation-controlled weight loss, the rate is instantaneously surface-specific but can be reported in this manner only when the composition of the vaporizing material does not change with the fraction evaporated.

Various permutations and combinations of these mechanisms, where more than one appreciably influences weight loss rates, become more complex and less susceptible to adequate representation as area-specific or time-specific phenomena.

The kinetic simulation included in this report is not intended as an implication that the data from this study necessarily indicate a degradation-controlled process. Instead, the method of simulation was used to obtain a reasonable approximation of the thermal dependence of the degradation phase of weight loss.

It is for the above reasons that this laboratory has emphasized and continues to emphasize the importance of complete weight-loss histories as the most forthright form for reporting weight loss data. Representation in terms of degradation, diffusion, and evaporation steps would be equally appropriate and technically superior since it would provide direct scaling of results to other conditions. However, sufficient information usually is not available to permit quantitative determination of either relative or absolute rates associated with these different mechanisms.

CONCLUSIONS

This investigation consisted of cross check studies on selected polymers by two laboratories in an effort to compare and to standardize weight loss measurement techniques under vacuum conditions. The initial results were compiled, discussed, and analyzed during a meeting of participants, and further work was planned. Subsequent experiments indicated that the error analysis of the initial results and subsequent equipment modifications rectified problems resulting from trying to operate under conditions normally not encountered in similar work at this Center.

The finding of singular importance from the project at this stage was the gross temperature deviations from levels thought to be maintained, primarily a consequence of an overextension of equipment capabilities. The fact that thermal problems can occur in the observed magnitudes should be of interest to every worker in the thermal-vacuum materials science area.

A need for more careful calibration and checkout in the always-difficult area of temperature control and measurement is apparent. However, these comments are not intended as an inference that data published previously should be questioned because of these findings. Data published earlier by this laboratory were obtained under conditions and for materials much more appropriate to the equipment and procedures than those involved in the current investigation.

Discussions of the results indicated a need for recognition that reporting of data in forms other than weight-time histories can lead to misinterpretation. Until full delineation of the weight loss mechanism operative for any given material/test environment combination is demonstrated, reporting of data on either an area-specific or time-specific basis is not justified.

The brief kinetic analysis showed that either constant or fluctuating thermal errors are indeed critical. However, heatup mode and rate probably have little influence on the ultimate weight loss rates if serious overheating does not result in a change in basic polymer structure. Analog simulation of the reaction kinetics provides a simple and flexible tool for investigating the effects of changing operating conditions.

With proper and thorough planning and preparation, comparison projects of this type are of great value in standardizing procedures. The results provide an indication of errors encountered and permit comparison and selection of equipment and methods.

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TABLE I

EXPERIMENTAL GUIDELINES

General:
1. Document all procedures, particularly those differing from the guidelines.
2. Minimize ambient exposure of specimen during preparation and installation in system.
3. Sample sizes shall range from 65 - 130 milligrams.
4. Time at conditioning temperature shall not be less than 12 hours.
5. The run at "isothermal decomposition temperature" shall terminate after six hours, or sooner if 90% of initial weight is lost.

Material	Form	Source	Conditioning Temperature, °C	Isothermal Decomp. Temperature, °C	No. of Runs
Polymethylmethacrylate	Granules	Rohm & Haas	140	260	3
Polystyrene	Pellets	NBS	180	330	3
Polystyrene			180	340	3
Polystyrene			180	360	3
Polytetrafluoroethylene (Teflon)	Powder	DuPont	250	520	-

TABLE II

COMPARISON OF EQUIPMENT AND TEST CONDITIONS

	MSFC	NBS
Sample Size and Special Preparation	65-130 mg None	Thin slices 6 mg
Time Required to Heat to Run Temperature	<5 min.	Very short
Pressure, Torr	10^{-3} - 10^{-6}	(0 on 504 gauge) $<10^{-5}$
Balance Type	Cahn Electrobalance, Type RA	Tungsten Helix
Temperature Control	Manual, const. voltage	On-off
Pump Fluid	Octoil	Mercury
Cold Trap	Sometimes	Yes
Pressure Gauge Type	V.I.C. Ionization	Pirani
Thermocouple:		
Type	Copper-Constantan	Chromel-Constantan
Changed	No	No
Moved	No	No

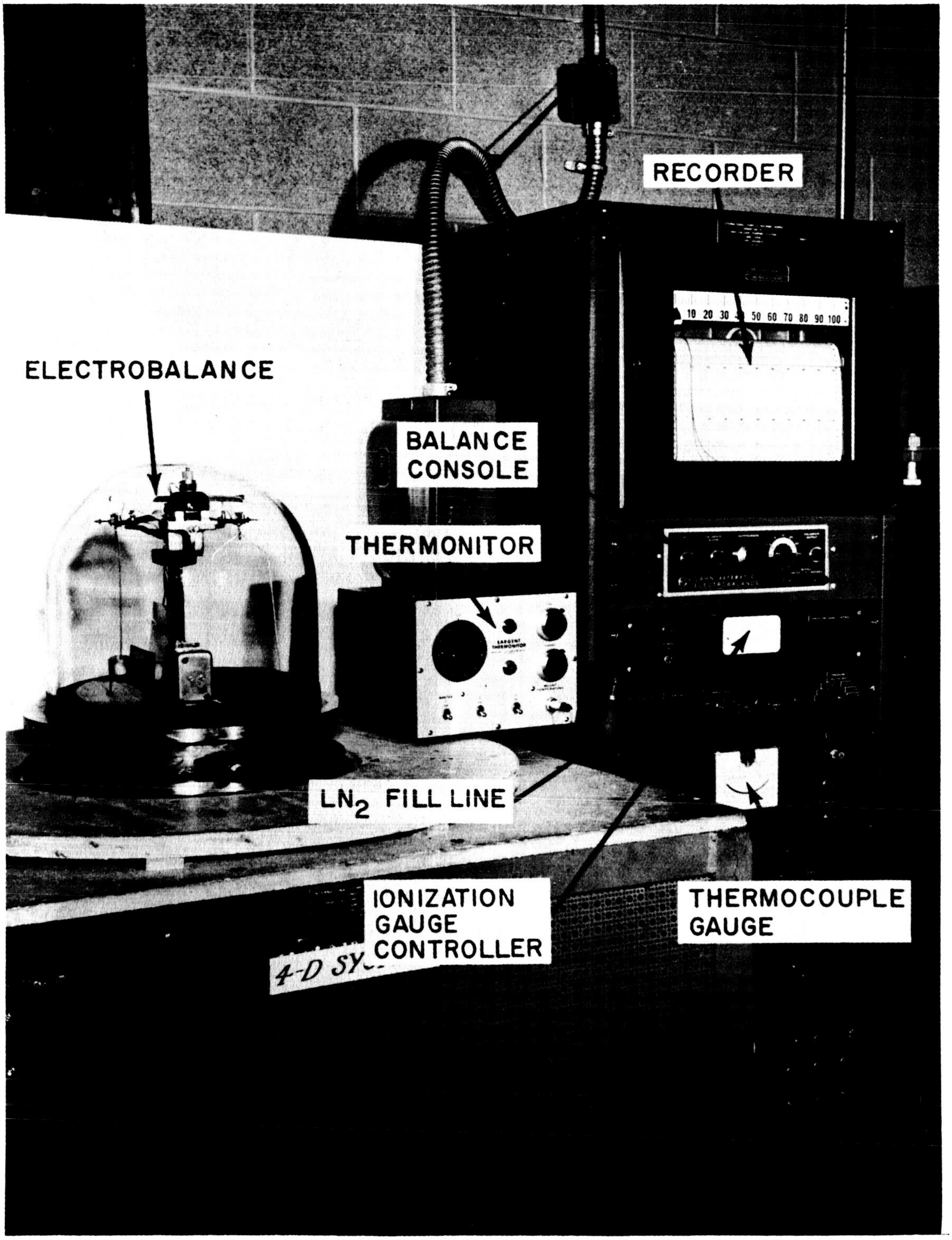

FIGURE 1. VACUUM SYSTEM USED FOR MSFC STUDIES

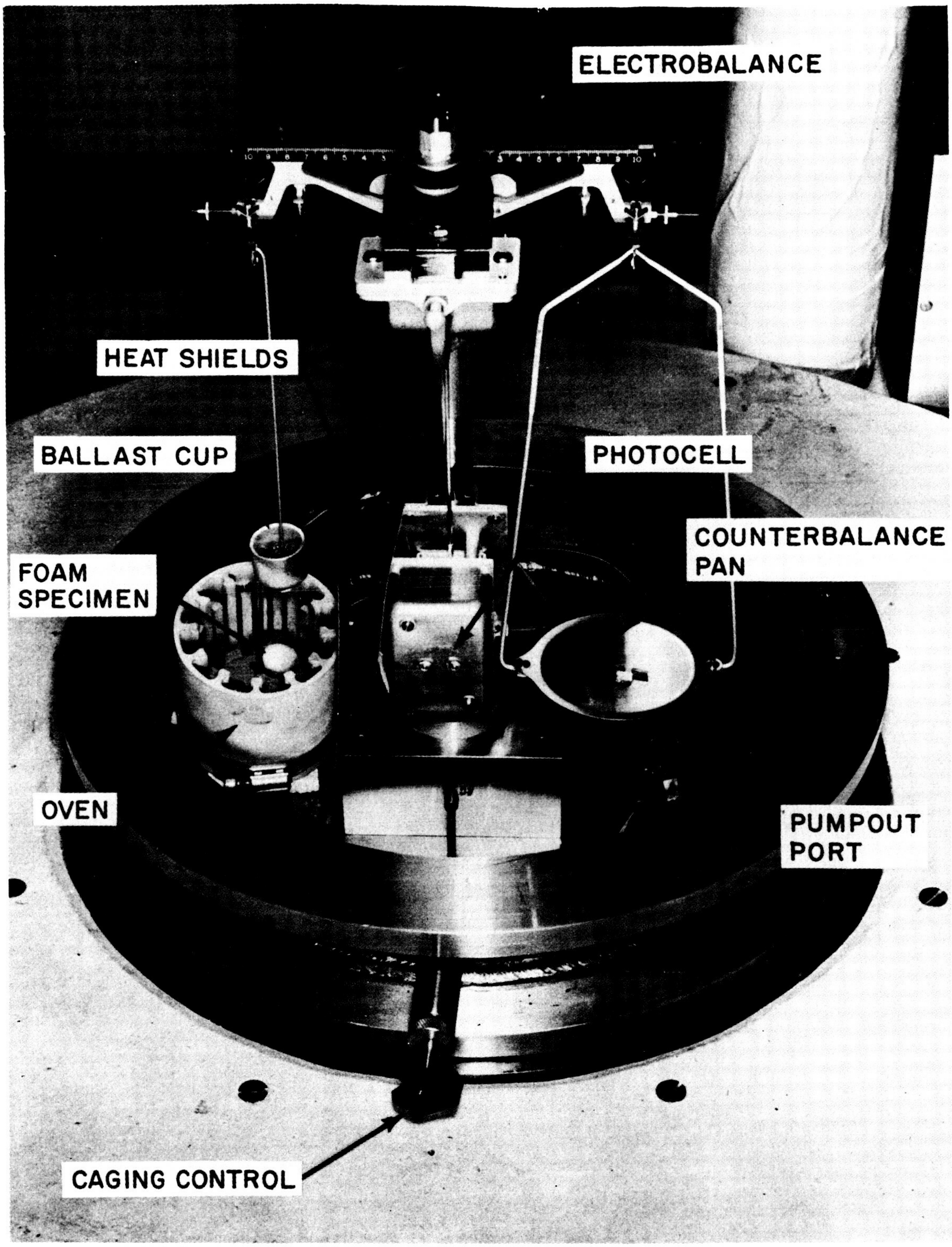

FIGURE 2. CONTENTS OF BELL-JAR FOR MSFC VACUUM SYSTEM

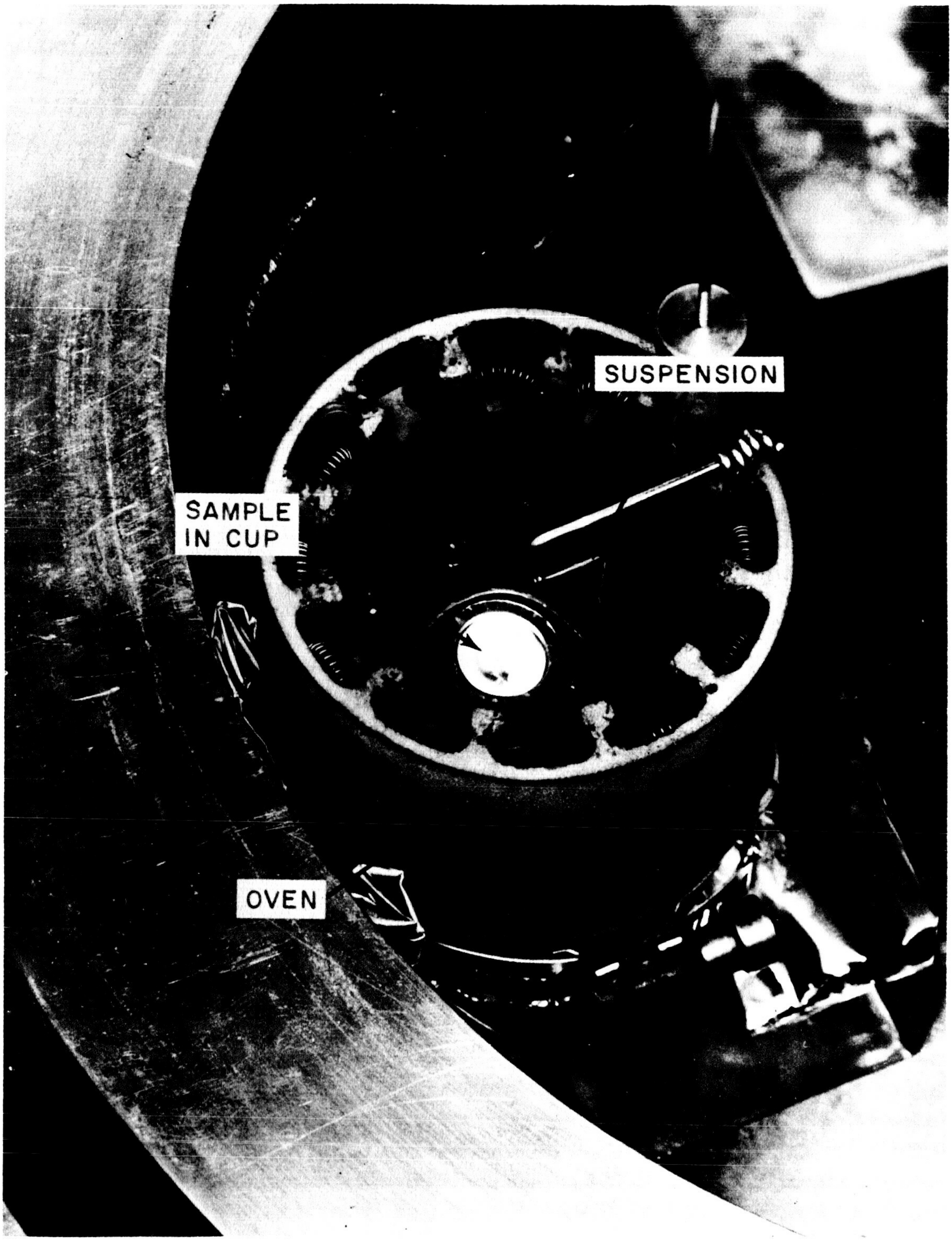

FIGURE 3. SAMPLE AND OVEN USED FOR MSFC TESTS

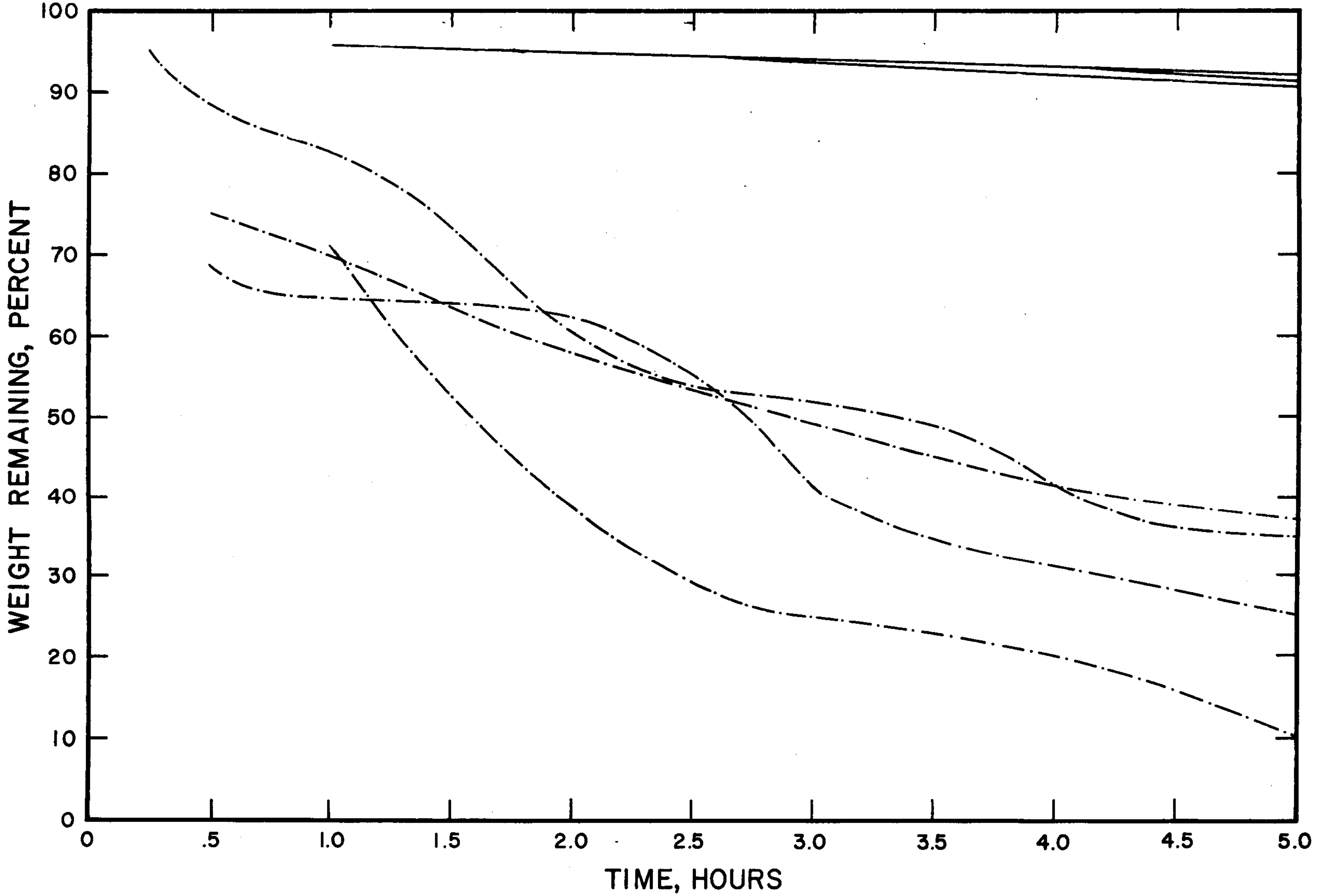

FIGURE 4. RESULTS FOR INITIAL POLYMETHYLMETHACRYLATE TESTS AT 260°C

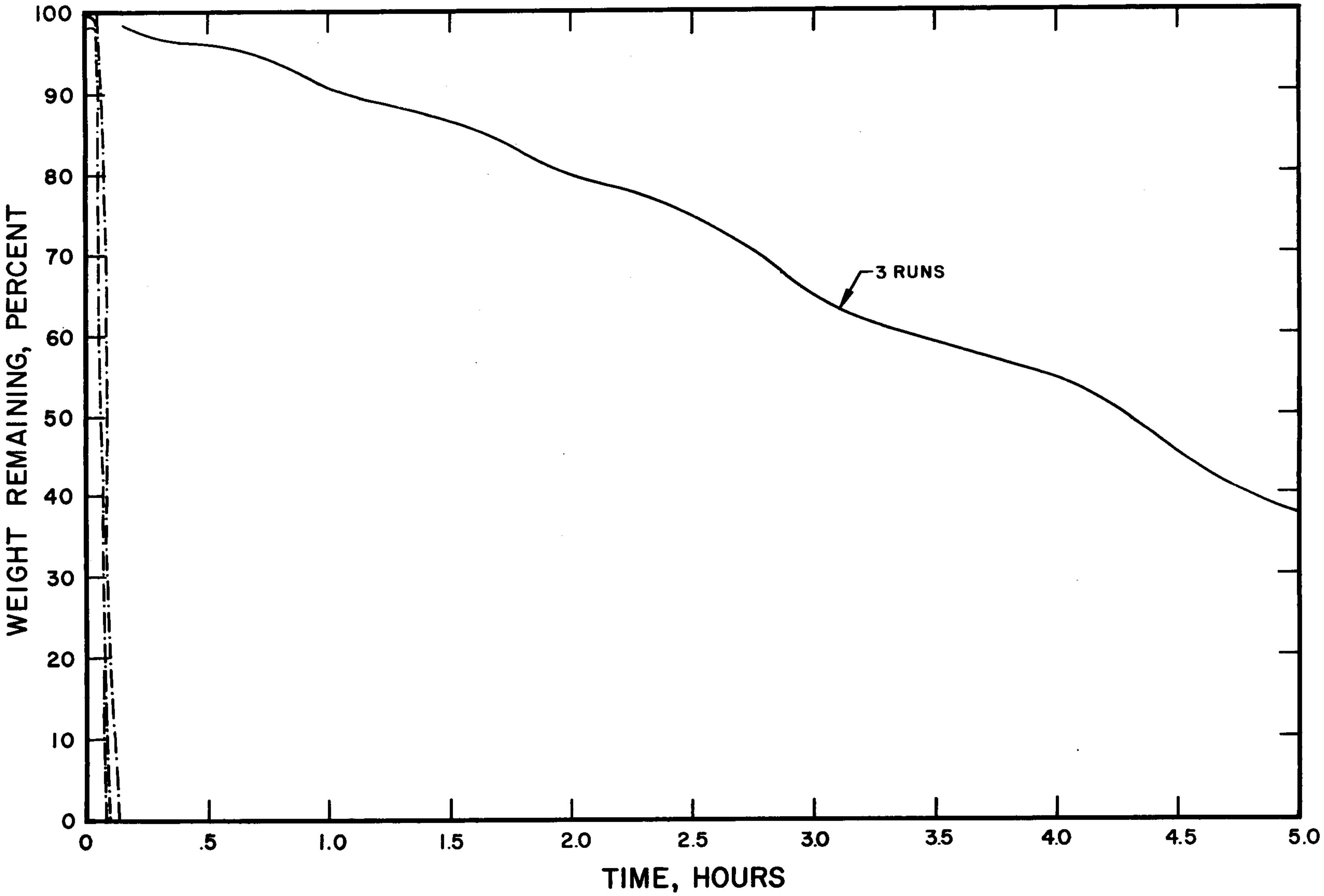

FIGURE 5. RESULTS FOR INITIAL POLYSTYRENE TESTS AT 330°C

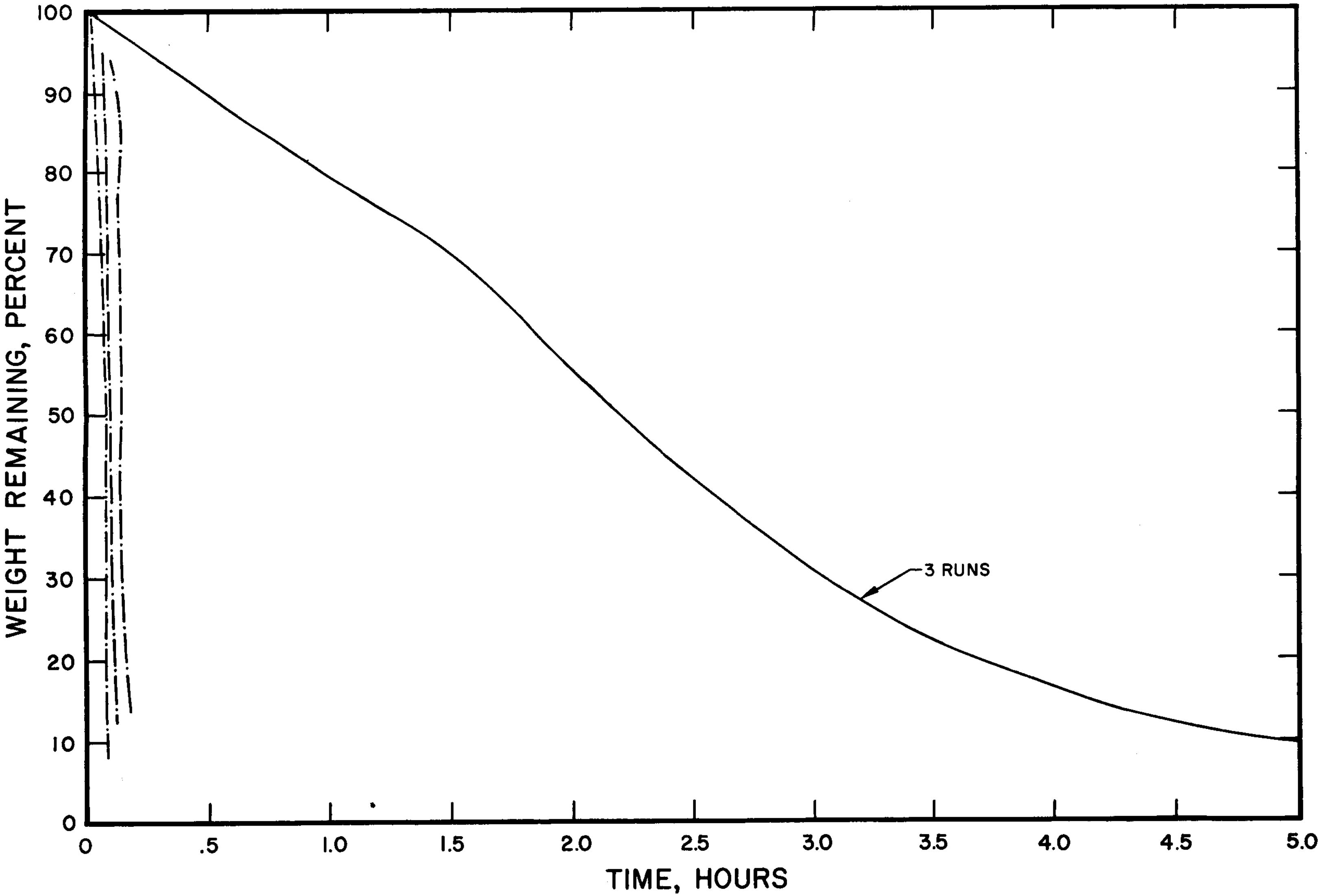

FIGURE 6. RESULTS FOR INITIAL POLYSTYRENE TESTS AT 340°C

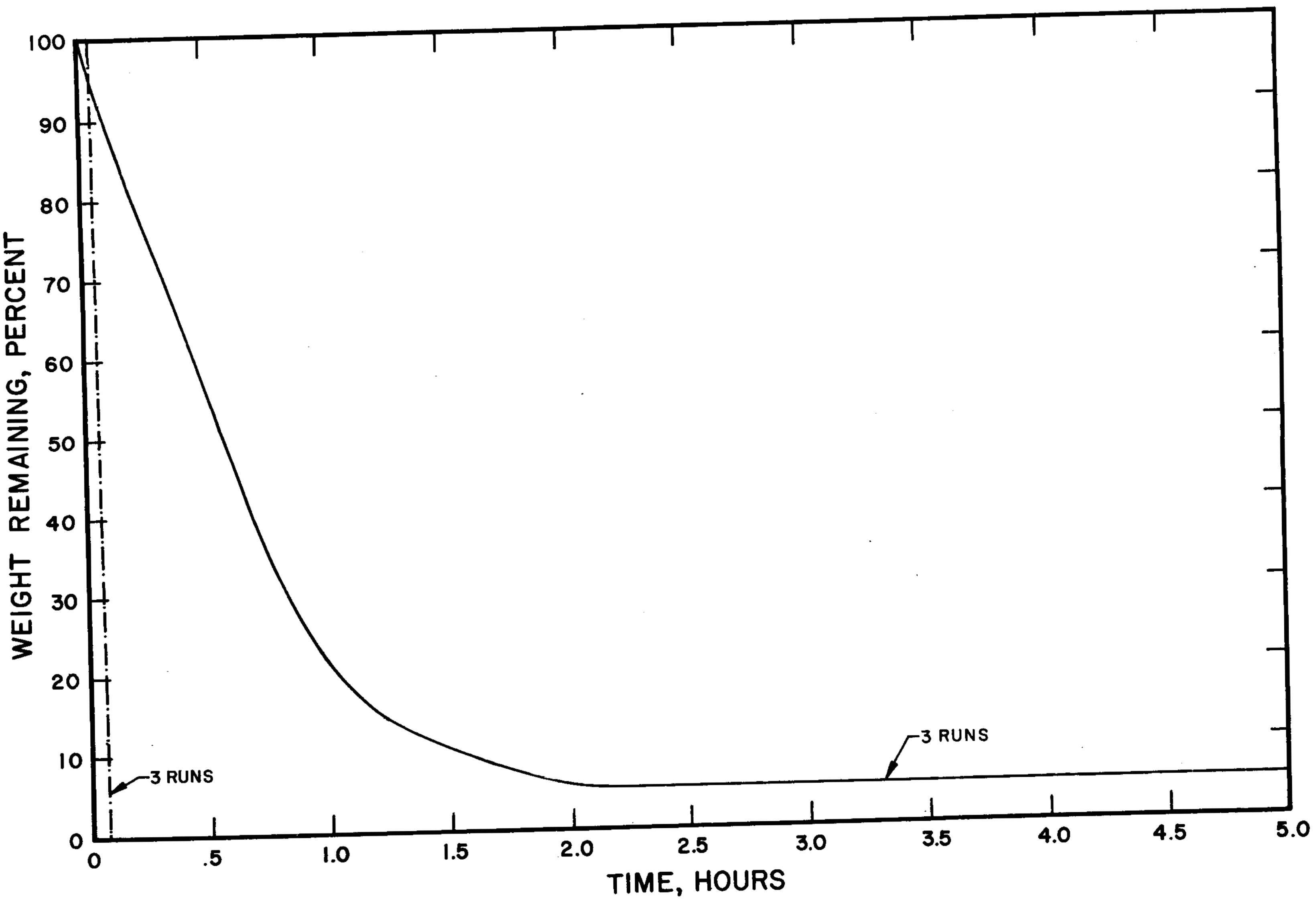

FIGURE 7. RESULTS FOR INITIAL POLYSTYRENE TESTS AT 360°C

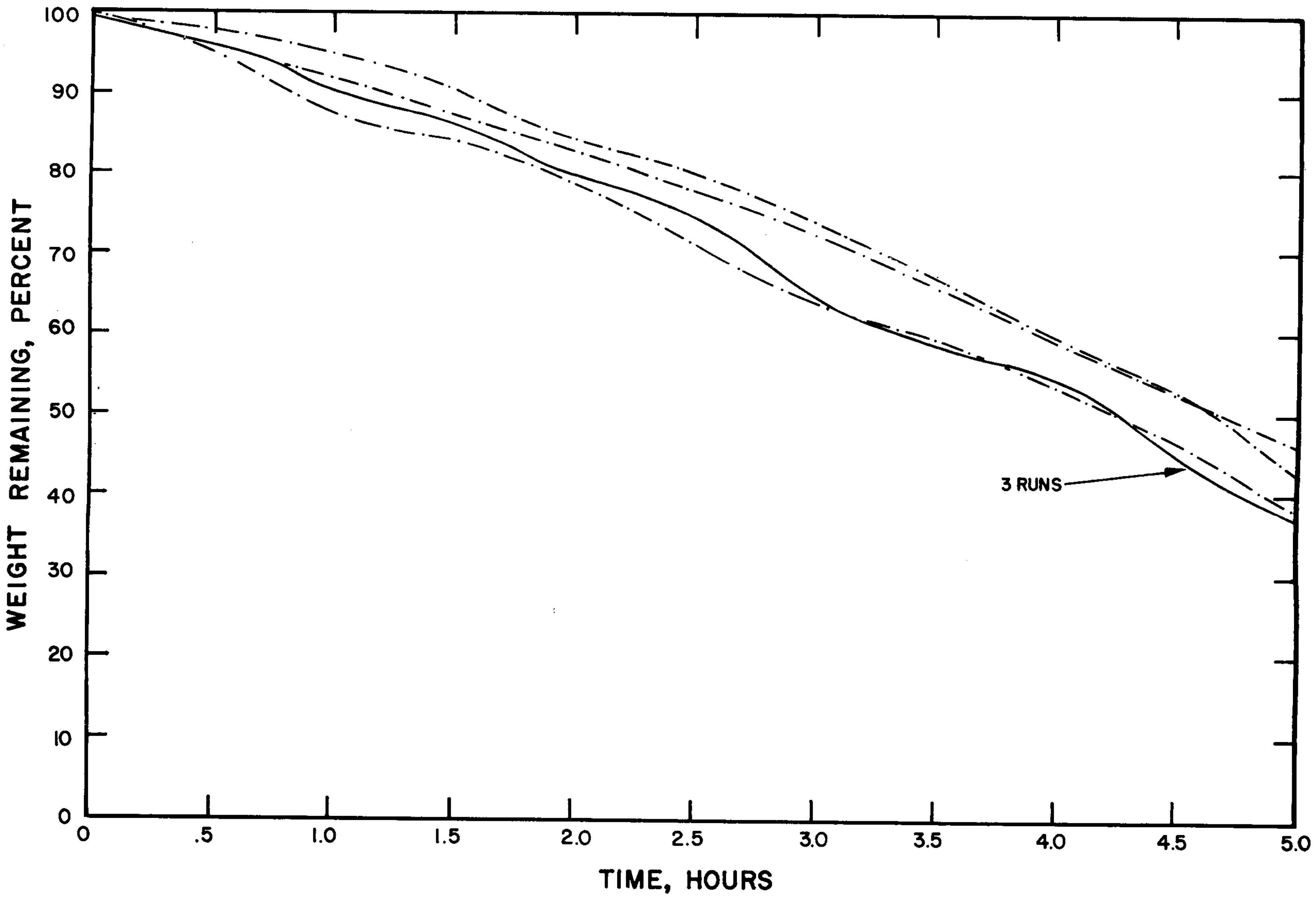

FIGURE 8. RESULTS FOR FINAL POLYSTRYENE TESTS AT 330°C

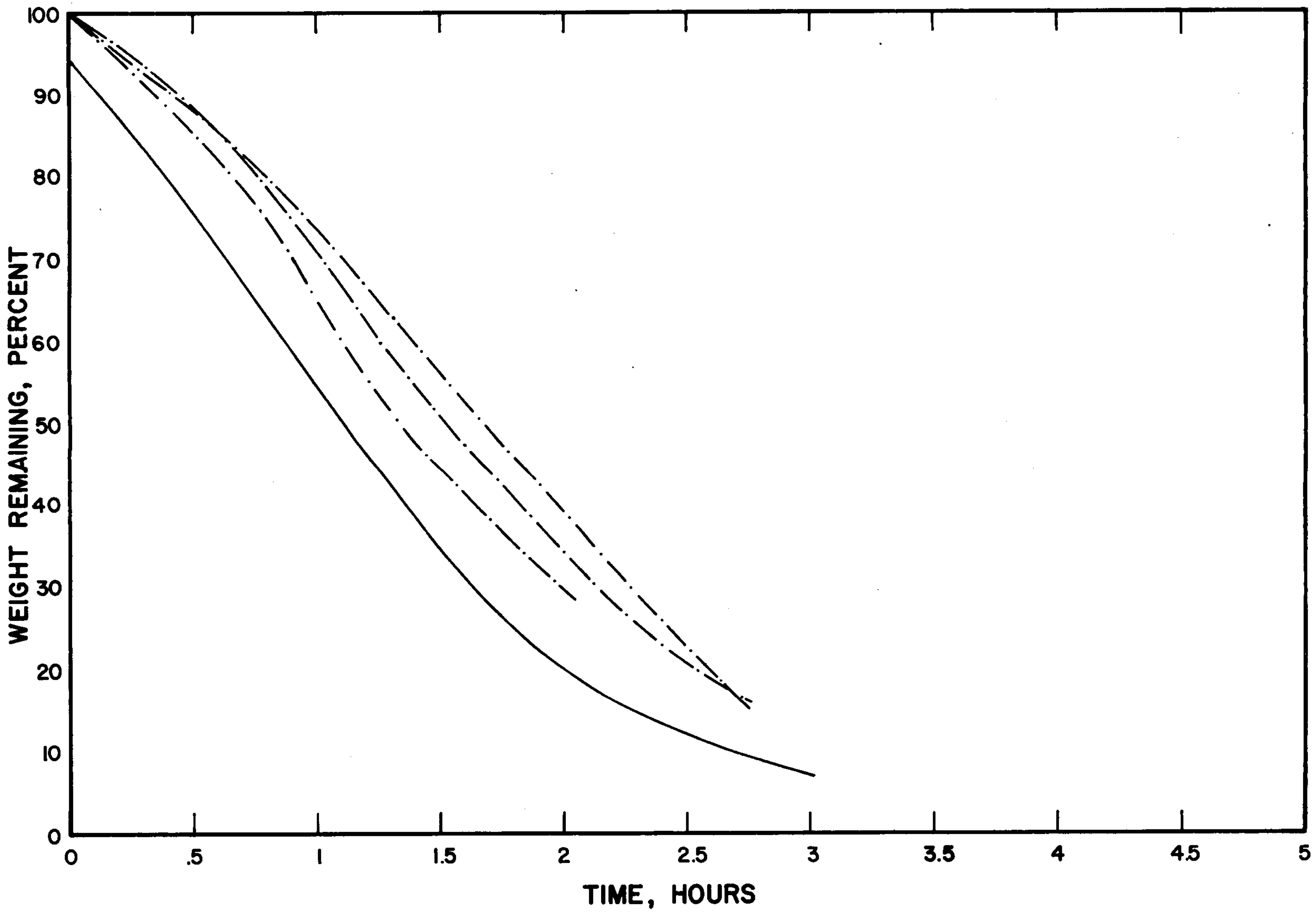

FIGURE 9. RESULTS FOR FINAL POLYSTYRENE TESTS AT 350°C

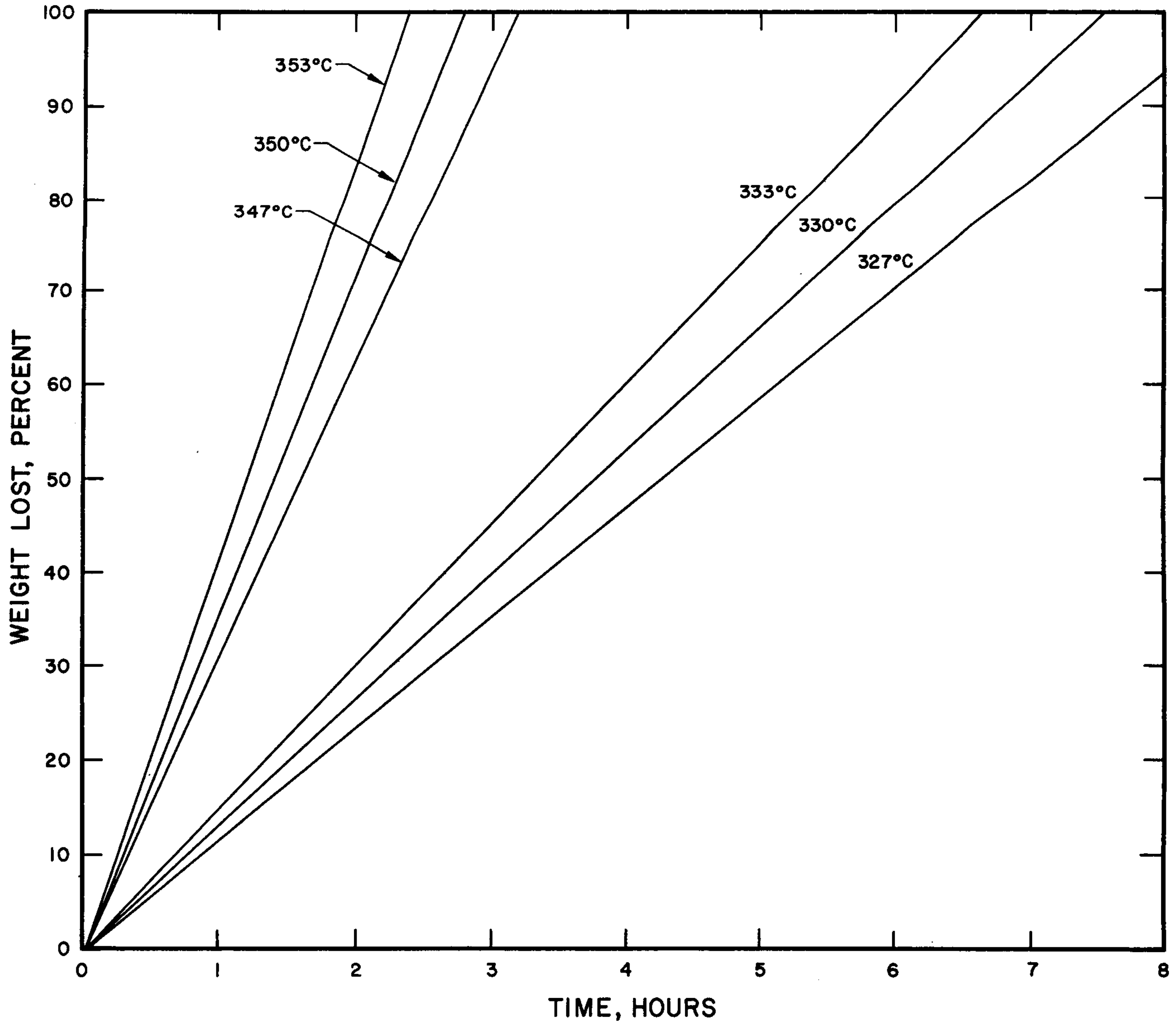

FIGURE 10. EFFECTS OF CONSTANT THERMAL ERRORS ON WEIGHT LOSS HISTORIES

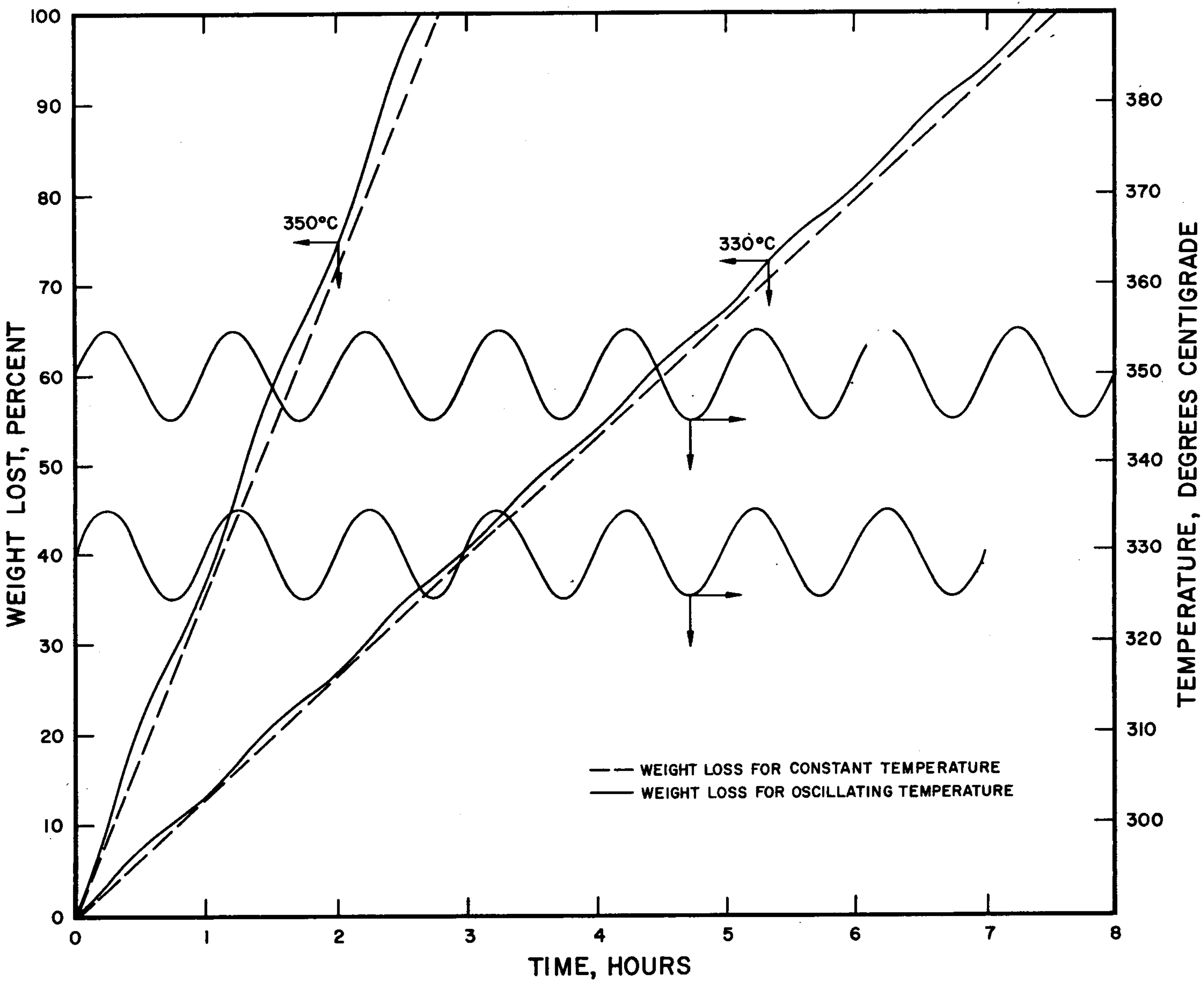

FIGURE 11. EFFECTS OF FLUCTUATING THERMAL ERRORS ON WEIGHT LOSS HISTORIES

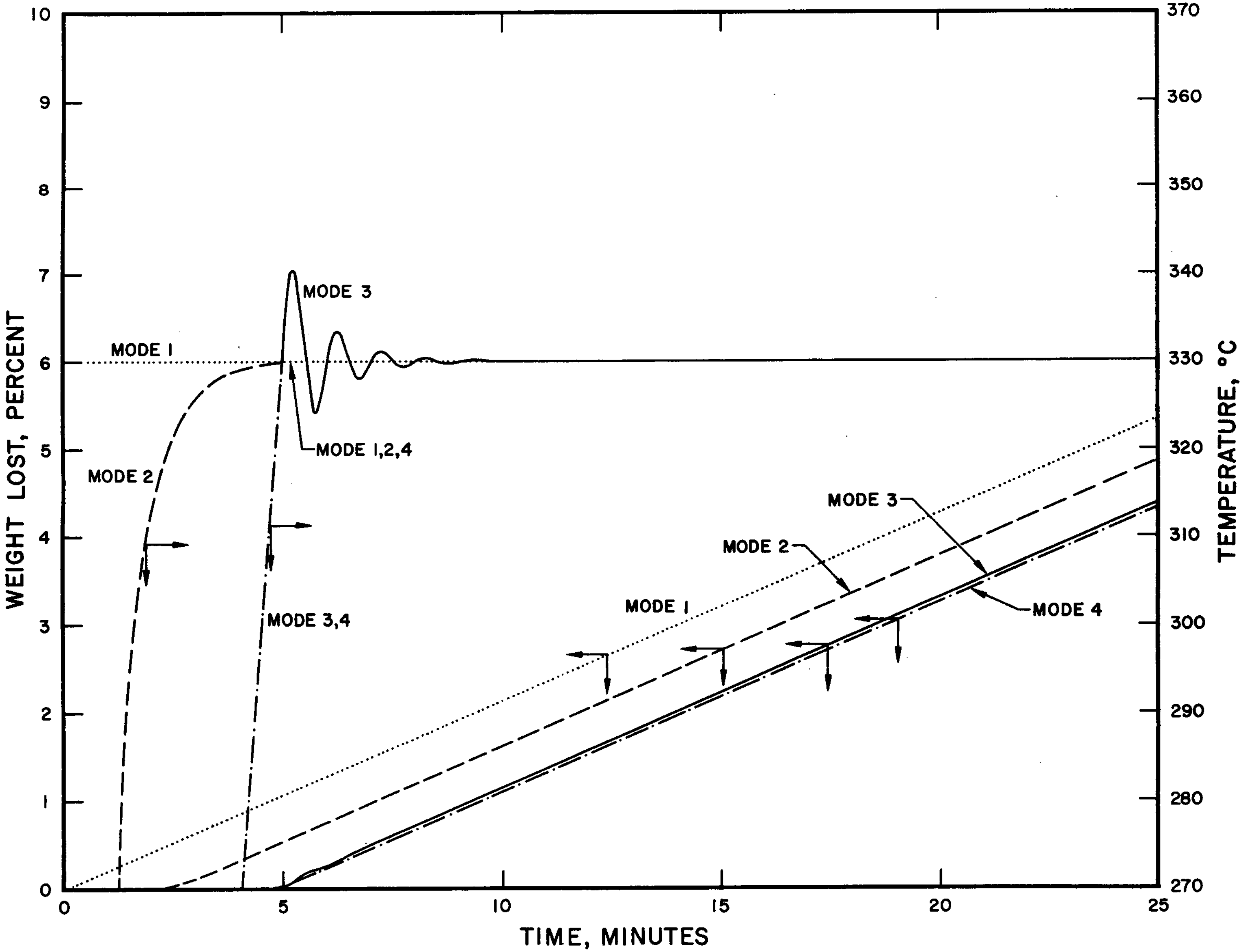

FIGURE 12. EFFECTS OF HEATUP RATE AND MODE ON WEIGHT LOSS HISTORIES AT 330°C

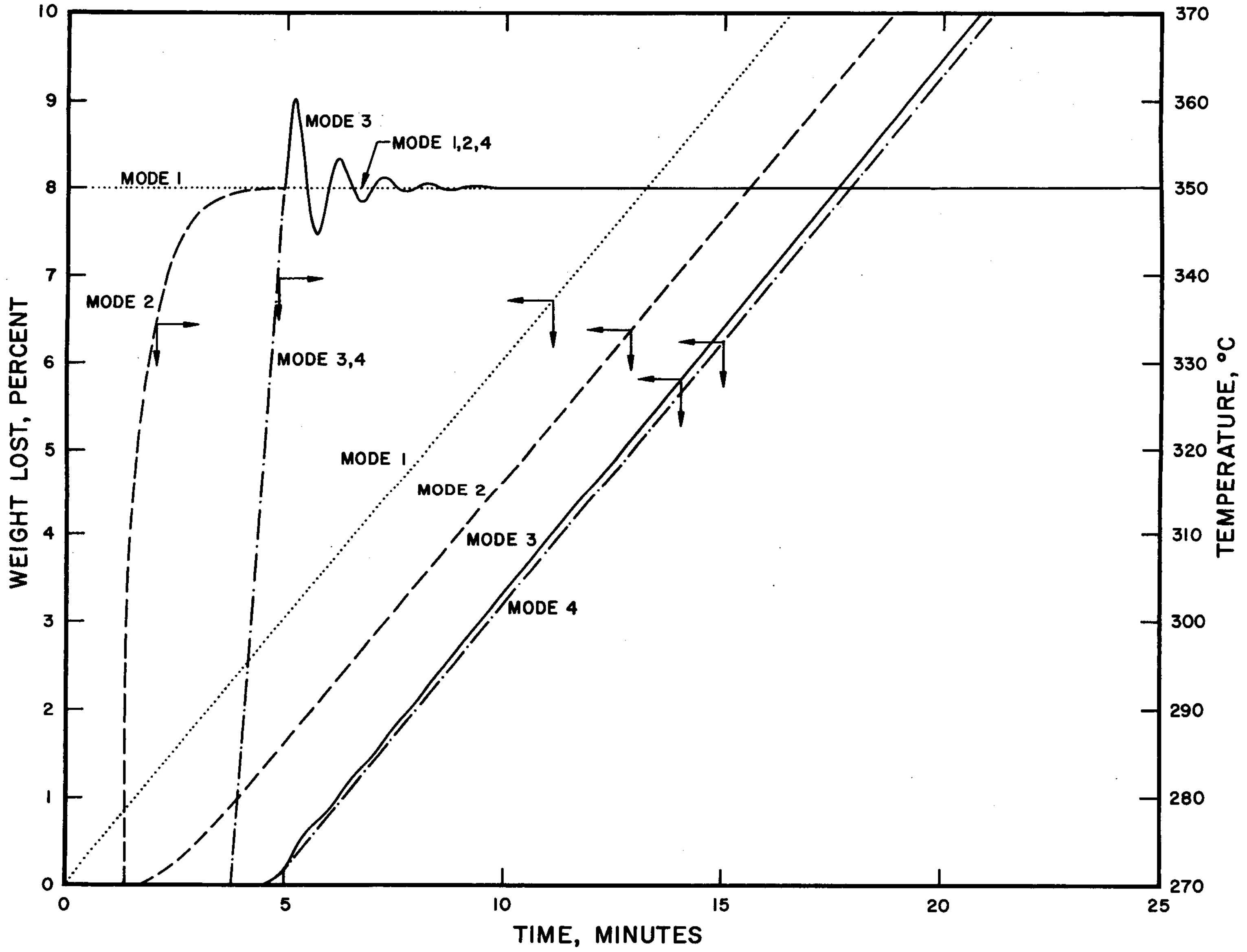

FIGURE 13. EFFECTS OF HEATUP RATE AND MODE ON WEIGHT LOSS HISTORIES AT 350°C

TM X-53106 APPROVAL August 13, 1964

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C. T. Egger and J. B. Gayle

The information in this report has been reviewed for security classification. Review of any information concerning Department of Defense or Atomic Energy Commission programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

This document has also been reviewed and approved for technical accuracy.

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