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TOXICITY OF THE PYROLYSIS PRODUCTS OF SPACECRAFT MATERIALS

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TABLE OF CONTENTS

TABLE OF CONTENTS, cont. Page(s)

TABLE OF CONTENTS, cont. Page(s)

I. INTRODUCTION

In the selection of materials for construction, whether for an apartment house, office building, airplane, submarine, spacecraft, etc., the physical properties of the candidate materials are very important in that they must provide the necessary characteristics of strength or flexibility to mechanically accomplish their designed functions. If, however, a material is subjected to thermal stresses, either in its designed role or as the result of an accident or malfunction, another problem may arise from the liberation of noxious or toxic fumes. This feature may become even more critical when it is not feasible to exhaust the fumes from inhabited areas. While it may be impractical to select only materials which do not release toxic fumes when heated, it is highly desirable to have information concerning the relative toxicity of such pyrolysates in order to permit selection of those materials which pose the least toxic potential and still provide satisfactory mechanical and physical properties.

Issuance of NASA Contract (NAS 9-13617) to the Materials Science Toxicology (MST) Laboratories was for the purpose of evaluating the relative toxicity of various materials selected and submitted by NASA to the MST Laboratories. Some of these samples were evaluated by two distinctly different protocols, while others were tested by only one of these procedures. While information from one method does not quantitatively extrapolate to the other, it does permit a relative ranking of potential toxicity of thermally generated fumes under different conditions of

temperature and oxygen availability.

Carbon dioxide and carbon monoxide are almost universally produced by thermal decomposition of organic materials, and many materials will produce other noxious or toxic gases depending on their compositions. Ammonia, cyanide, and chlorine are just a few of the substances which may be generated from materials such as those included in this study. Fluorinated polymers or copolymers may also produce HF or other fluoride-containing compounds. Additionally, many other compounds, representing various stages of breakdown and/or alteration of the components of the material, may occur in the fumes generated by heat. Such fumes are generally of a very complex nature, and complete identification and quantitation is a very expensive and time-consuming procedure; thus, it is of questionable value for screening tests of such materials. Even if the complete composition were known, additional information would be needed before an assessment of animal or human toxicity could rationally be made. For example, one would need to know the toxicity of each compound and if, and to what degree, the toxicity of the individual components would be potentiated or antagonized by the presence of other substances in the gaseous fumes. Exposure of animals to the fumes, however, tends to provide the net result of such interactions and permits an evaluation of the relative toxicity of the overall effects of such mixture of ingredients, even though the exact composition of the mixture is unknown.

In addition to the relative toxicity of pyrolysates of materials from acute exposure, a number of other factors may need to

be considered before final selection of a candidate material. Some or all of the following may be of concern: (1) Do repeated exposures to sublethal quantities of these fumes produce a cumulative toxic effect? (2) Does exposure to sublethal levels of the pyrolysate adversely affect mental performance or discriminatory behavior? (3) Does the pyrolysate generated pose a significant explosive hazard? (4) What would be a highly efficient filter or trap to remove noxious or toxic chemical species from the pyrolysate? (5) If the material is decomposed by heat, in addition to its potential biological toxicity, does the pyrolysate contain any particular chemical species which would be especially detrimental (e.g., a corrosive substance which may contact delicate instruments, or HF which might etch glass and impair visibility, etc.) to items with which it may come into contact?

Toxicity tests on thermodegradation products of a material may, to some extent, be tailored to a particular application, and thus become more practical for that application when conditions affecting that application are known. For instance, if the use of a particular material is such that, even in case of an accident or malfunction, the temperature will not exceed 400°C, then the tests could be performed at 400°C rather than at some higher temperature. Since the toxicity of the pyrolysate often is a function of the degradation temperature, the 400°C limit would provide more practical information for this application, but at the same time would not necessarily be appropriate for another application where the maximum temperature might reach 700°C. In a similar manner, use of the material may dictate whether it is

less hazardous to use a flammable fabric which produces less toxic fumes upon pyrolysis or a flame-retarded fabric which may produce more toxic fumes if pyrolyzed.

It is not feasible to attempt to answer all such questions about a material during preliminary screening tests; many of these may be investigated once a material appears sufficiently promising for a specific application, and before a final decision is reached concerning which material to use. The experiments conducted under this contract may be considered a general approach to determination of the relative toxicity of pyrolysates from possible candidate materials under two rather severe sets of conditions.

II. MATERIALS AND METHODS

The purpose of this first year of the contract was principally evaluative testing of toxicity of the pyrolysis products from materials selected by NASA, rather than exploratory investigations. The original contract, plus approved modifications, specified (1) determination of thermal stability of the samples by thermogravimetric (TGA) procedures; (2) determination of the LD_{50}* of samples by the NASA procedure of pyrolysis and exposure; (3) determination of LD_{50}* of specified samples by the MST procedure of pyrolysis and exposure; (4) qualitative and/or quantitative tests for the presence of selected substances in the exposure chamber atmosphere; (5) determination of carboxyhemoglobin

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*LD_{50} is used in this report to mean the initial weight of sample required to kill 50% of exposed rats when the sample is pyrolyzed according to the specifications described in the respective procedures.

(COHb) content of the blood of rats that die in the exposure chamber; (6) gross autopsy of exposed rats; (7) histological examination of selected tissues (particularly the lungs) from exposed rats; and (8) determination of pyrolyzed hydrolyzable fluoride (for the fluoride-containing polymers or copolymers) from the quantity of sample required to kill 50% of the exposed animals.

All samples tested for this contract were submitted directly by the NASA Technical Monitor or from NASA contractors on instruction of the Technical Monitor. A list of these materials, a brief description of each, and any identifying information (such as code number, manufacturer, etc.) which we have concerning the samples are presented in this report (Table 1). Male rats of the Sprague-Dawley strain, obtained from the barrier-sustained colony of Harlan Industries (Cumberland, Indiana), were test animals in these experiments. A Bethlehem hyperbaric chamber (Model 1836) was purchased from the Bethlehem Corporation (Bethlehem, Pennsylvania), and modified in accordance with the specifications in Attachment 1 of the original contract (June 28, 1973). These modifications were necessary to provide a test system which is comparable to that employed by the NASA group (Houston) working with these types of materials.

Two procedures were used in evaluating the relative toxicity of thermodegradation products from specified materials: (1) the NASA procedure, as described in detail in the original contract (except for pyrolysis temperature, which is discussed in the Amendment), and (2) the MST procedure, which was incorporated into the "Amendment of Solicitation/Modification of Contract."

Because of the marked differences in the two protocols, one would anticipate that the LD_{50} values obtained would also be quite different. The reasons for including both testing procedures will be discussed subsequently. Brief protocols of these two methods are:

1. NASA Procedure. The test sample is weighed and placed around the vertically-movable tube with the sample in the upper position. Pressure within the modified hyperbaric chamber is reduced to 600 torr and the system checked for leaks. Then air (100 ml/min) is passed through the sample-holding tube until completion of pyrolysis. The furnace is placed around the combustion tube with the lower part of the tube in the hottest portion of the furnace. The electric furnace is allowed to reach its preset temperature (requiring about 10 minutes), after which the movable tube containing the test sample is lowered into the hottest part of the combustion tube. A thermocouple mounted adjacent to the sample provides a continuous readout of the temperature in this area, and regulation of furnace temperature is based upon this temperature. The standard pyrolysis temperature was 600°C. Pyrolysis was allowed to proceed for 30 minutes, after which time the furnace was removed and air continued to flow into the chamber until it reached 750 torr. Ten 225-250-gram rats were introduced into the chamber through the air-lock system. Immediately upon introduction of the rats, a gas sample was taken from the chamber (0 time); other samples were obtained 15 and 30 minutes later. Thirty minutes after introduction of the rats, the chamber was opened by an operator, wearing a Scott mask connected to

a cylinder of breathing-quality air, and the rats were removed.

Surviving rats were caged in a fume hood for the following 2 hours and observed for evidence of toxicity. Blood was obtained from each dead rat for COHb analysis, and then those rats were autopsied; the lungs and other selected organs were preserved in 10% buffered formalin for histopathologic examination. After 2 hours under the hood, surviving rats were returned to the animal quarters where they were checked daily. Mortalities were recorded and an LD_{50} calculated for each material for animals which died in the chamber, and at 48 hours and 7 days post-exposure. At the conclusion of the observation period, the surviving rats were sacrificed, autopsied, and selected organs from a representative sample of these animals were examined histologically.

The chamber atmosphere was screened for the presence of certain substances, including CO and HCN, using gas detector tubes. Representative samples of chamber atmospheres were tested for concentration of O_2, CO_2, and CO by gas chromatography.

Special tests were conducted upon certain samples, such as volatilized (pyrolyzed) hydrolyzable fluoride or cyanide production from pyrolysis.

2. MST Procedure. Four male rats (150-180 gm) in small individual cages were placed in the rectangular, glass-walled exposure chamber (volume = approximately 63 liters). The chamber was equipped with an external light for illumination, an internal thermister probe for monitoring chamber temperature, and a magnetically-driven fan to prevent air stagnation and layering of pyrolysis products. The test sample was placed in a Vycor tube

inside a programmed electric furnace, with a thermocouple adjacent to the sample which controls heating of the furnace; thermocouple temperature (and chamber temperature, when desired) was recorded on a strip-chart recorder. One liter per minute of air was introduced at the end of the Vycor tube, passed over the pyrolyzing sample, cooled somewhat, and passed into the exposure chamber. Prior to entering the chamber, 0.5 liter/minute of fresh air was added to the effluent. The furnace was heated at 10°C/minute until it reached the temperature of maximum decomposition (as determined by TGA data) plus 50°C. At this point, the furnace was turned off and air-flow continued for an additional 60 minutes; then the chamber was opened and the animals removed. A gas sampling port was located immediately upon exit of the fumes from the chamber prior to their being exhausted.

3. COHb Determination. Carboxyhemoglobin determinations were conducted spectrophotometrically, using a modification of the method of Commins and Lawther (Brit. J. Ind. Med., 22:139, 1965). Preliminary experiments with this and other methods indicated that excessive dilution of the blood prior to analysis yielded lower calculated percentages of COHb in the blood; presumably this is due to dissociation of COHb in the dilute solution ($COHb \rightleftharpoons CO + Hb$). Thus, the modifications employed were to permit spectrophotometric analysis without undue dilution of the blood sample. The Commins and Lawther method was used with the following modifications: (a) 40 µl of blood (rather than 10 µl) was used per 10 ml of reagent (rather than 25 ml), and (b) a cell path of 0.1 cm (rather than 1.0 cm) was employed. Thus, the solution for analysis was 10 times more concentrated, and the cell

path for analysis was one-tenth that recommended by Commins and Lawther. Analyses were performed at a wave-length of 421 nm, with readings at 414 and 428 nm for purposes of correction. Concurrent COHb and O_2Hb controls were tested, in which rat's blood was saturated with gas from tanks of CO and O_2, respectively.

Using this procedure, we found that rats killed by exposure to CO in air typically exhibit 60-70% relative COHb. Also, the rats exposed only to CO in air either died while in the CO environment, or else survived a 2-week observation period; there were no delayed deaths.

4. Hydrolyzable Fluoride Determination. As specified in the contract, the hydrolyzable fluoride content of the pyrolysis products was determined for the fluorinated polymers; only one of the samples included in this study (Y-1794, Fluorel) was a fluorinated polymer. The LD_{50} weight (5.82 gm) of this polymer was thermally degraded at 600°C under the same conditions used in the toxicity studies; however, the pyrolysate was passed directly from the pyrolysis tube into a series of 6 traps containing 0.01 M sodium carbonate. Each of the first 5 traps contained 750 ml and the last 400 ml of the sodium carbonate solution. These were appropriately diluted, buffered, and analyzed for fluoride with the Orion ion-selective electrode. Five replications were conducted.

5. Cyanide Determination. Because of the relatively high levels of HCN detected in some pyrolysates, particularly Y-2000 (Modified Nomex HT-4), some exploratory studies were conducted to determine the total quantity of cyanide generated in the thermal degradation of the LD_{50} weight of this sample, in a fashion similar

to that used for hydrolyzable fluoride. The method used was essentially that of Vickroy and Gaunt (Tobacco Science, 16 (25): 22-25, 1972 ["Determination of Cyanide in Cigarette Smoke by a Cyanide Ion-Selective Electrode"]), in which the sample was pyrolyzed, as for the LD_{50} studies, but the pyrolysate was passed through a series of traps containing Ascarite® (Arthur H. Thomas Co., Philadelphia, Pa.). The cyanide was extracted from the Ascarite with distilled water in a markedly basic medium (~ pH 12-13), adjusted to a pH of 12.7, and quantitated using the Orion ion-selective electrode for cyanide. Due to the relatively large quantity of Ascarite used (about 230 gm), sequential extracts were made to ensure more complete extraction of cyanide. The final Ascarite-filled tubes in the traps were checked to see that they did not contain a significant quantity of cyanide, to be sure the previous traps had retained essentially all of the cyanide produced by the pyrolysis procedure.

6. Calculation of the LD_{50}. From the pyrolysis toxicity data, the initial weight of the sample which should produce death in 50% of a similarly-exposed population of rats is calculated. Cornfield and Mantel's modification of Karber's method was used in these calculations (Am. Stat. Assoc. J., 45:181, 1950).

Additionally, data are presented for a calculation of "lethal sample weight for 2 of the 4 exposed rats" ($LSW_{2/4}$). (The concept was also extended to the NASA procedure, which actually represented 5/10 deaths, but the $LSW_{2/4}$ designation was retained.) The $LSW_{2/4}$ value, essentially equivalent to the LD_{50}, is determined by a computer fit of the following equation to the rat mortality

data by means of the Marquardt Algorithm ("An Algorithm for Least-Squares Estimation of Nonlinear Parameters," J. Soc. Indust. Appl. Math., 11:431, 1963):

$$\% \text{ Mortality} = 0.555 \text{ ATAN } [\text{Slope} \times \frac{\pi}{100} (\ln \text{SW} - \ln \text{LSW}_{2/4})] + 50$$

where: ATAN = Arc Tangent, in degrees

ln SW = Natural logarithm of sample weight

$\ln \text{LSW}_{2/4}$ = Natural logarithm of lethal sample weight which kills 2 out of 4 rats (50% mortality)

Slope = Slope of the curve at the inflection point where % mortality is plotted vs. natural logarithm of sample weight

The $\text{LSW}_{2/4}$ value and its 95% confidence interval is calculated from the computer program.

7. Gas Analyses by Detector Tube. This involved sampling chamber atmosphere using the Unico Gas Detector, Model 400, in conjunction with the commercially-available Bendix Gas Detector Tubes. (A brief description of these is presented in catalogs of "Linde Specialty Gases," from Union Carbide Corporation, pp. 94-95, 1971, and in "Matheson Gas Products, General Catalog 27," from Matheson Gas Products, pp. E-54 and -55, 1969.) One of the problems encountered with this procedure is the "relative specificity" of the detector tubes, since many of these are affected by sufficient concentrations of interfering substances. Since the complete compositions of the pyrolysates are unknown, it is possible that interfering substances may be affecting some of the readings. Also, because of the difficulty in obtaining an exact concentration, we tend to refer to these data as approximate concentrations.

III. RESULTS AND DISCUSSION

Pyrolysis Toxicity Testing, the NASA Method and the MST Method.

Evaluation of the toxicity of pyrolysis products from a group of materials is, of necessity, an evaluation of relative toxicity during screening tests. Since the actual toxicity of the fumes from the thermally degraded material is influenced by a number of factors (such as quantity and location of material, temperature profile and maximum temperature [causing the degradation], the degree of oxidation of degradation products, confinement or dispersal of fumes generated, etc.), the actual in-use situations may differ markedly from any established laboratory protocol design and, in fact, would probably differ from one in-use situation to another.

Use of these two methods provides an assessment of relative toxicity from thermodegradation of the material under significantly different sets of conditions. The NASA procedure produced a rapid temperature rise up to the predetermined level (600°C), and during degradation of the material there may be a significant oxygen deficit within the pyrolysis tube*. All of the fumes generated by this method are retained within the exposure chamber until the end of the animal exposures; there is no loss to the atmosphere. Due to the relative static nature of the chamber atmosphere, however, there may be a tendency toward "layering" of gases of different densities.

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*A comparison of the residue weights obtained from the NASA pyrolysis procedure with those obtained by TGA in an air atmosphere, and a nitrogen atmosphere, suggests thermodegradation of many samples by the NASA procedure occurs in an oxygen-deficient (or oxygen-poor) state.

The MST procedure, on the other hand, produces a more gradual temperature rise and to a greater maximum temperature, with a potentially more oxygen-rich pyrolysis environment (1,000 ml of air/minute as opposed to 100 ml/minute), and an overall longer exposure time. The fumes generated are partially cooled, mixed with more air, and passed into the animal exposure chamber; a magnetically-driven fan prevents "layering" of the fumes in the chamber. After mixing with the chamber atmosphere, the fumes are exhausted from the chamber at the rate of entrance of additional fume-air mixtures into the chamber (1.5 L/minute). This provides a more dynamic system of fluctuating pyrolysate concentrations in the chamber, when compared with the NASA chamber static environmental conditions.

The previous discussion of "oxygen-poor" or "oxygen-rich" conditions referred only to the site of thermal degradation. Numerous checks of the chamber atmosphere (to which the rats were exposed) indicated that there was always sufficient oxygen in this environment; in no instance was there a serious depletion of oxygen in the atmosphere which the rats were breathing.

The volumes of the two exposure chambers are also quite different: the one used in the NASA procedure has a volume of approximately 150 liters; that used for the MST procedure has a 63-liter volume.

These factors must be kept in mind when evaluating the results from these thermodegradation-toxicity studies.

Thermogravimetric Analyses.

Before initiating animal tests, each material was subjected

to thermogravimetric analysis. This provided information concerning anticipated degradation temperature(s), completeness of thermodegradation, etc. Two basic patterns of TGA determinations were employed: (1) that used by MST Laboratories, in which the air-flow rate was 200 ml/min and the heating rate was 10°C/min, and (2) that used by NASA-Houston, in which the sample was heated at 20°C/min with an air-flow of 20 ml/min. In addition, some of the samples were tested using method 1 or 2 (above), or both, in which the air was replaced with nitrogen, thus providing some information relative to how the degradation picture was changed under anaerobic conditions. A summary of these data are presented in Table 2, and computer plots of the TGA information for these experiments are given in Figures 1-20.

Acute Toxicity of Pyrolysates.

A resumé of LD_{50} values and their 95% confidence intervals for both the NASA and MST procedures is shown in Table 3. Although the quantity of each sample required to kill 50% of the exposed rats differs considerably between the two methods, ranking of the samples in order of increasing toxicity tends to be similar by either method. Of the four samples tested by both methods, one inversion in ranking occurred; by the NASA method, Nomex was least toxic and Kevlar-29 was next, while by the MST method, Kevlar-29 was least toxic and Nomex was next. There was no constant ratio between the two LD_{50} values (NASA/MST) for these samples, with the ratio ranging from 20.3 to 1.9. Thus, if one knows the LD_{50} by one method, it is not possible to accurately estimate the LD_{50} produced by the other method, though

probably a reasonable estimate of its toxicity ranking would be possible.

It was slightly surprising that the LD_{50} by the MST method required a smaller sample than did the NASA method, since the MST procedure does not trap and retain all of the fumes generated by the sample. This result, however, may be due to temporarily larger concentrations of some toxicant (such as CO or HCN) at a particular phase of degradation. It could also be the consequence of using a higher maximum temperature, more complete oxidation of degradation products, the smaller chamber used, a longer exposure period, or any combination of these factors.

Table 4 presents a comparison of the LD_{50} data as a function of observation time following exposure (chamber deaths, deaths within 48 hours, and deaths within 7 days) by the NASA procedure. It may be noted that Nomex (Y-1796), Durette (Y-1797), and Kevlar-29 (Y-1856) produced all deaths in the chamber. The LD_{50} for Modified Nomex HT-4 (Y-2000) was changed only slightly by post-exposure deaths. Fluorel (Y-1794), on the other hand, caused chamber deaths only in one of the exposed groups, with the majority of deaths occurring within 48 hours of exposure. A subsequent death changed the LD_{50} only slightly at 7 days.

This table (Table 4) also presents a comparison of the two methods used to calculate the LD_{50}, i.e., the LD_{50} and the $LSW_{2/4}$. (The LD_{50} was calculated by Cornfield and Mantel's modification of Karber's method, and the $LSW_{2/4}$ was calculated by the arctangent procedure described previously.) As may be seen in this comparison, when comparable data are utilized for both calcula-

tions, the results are quite comparable. Because of differences in the mathematical models of these two systems, there were some instances in which additional data from the experiments were used in the $LSW_{2/4}$ calculation which could not be used with the LD_{50} model.

The Materials Science Toxicology Laboratories have been involved in other studies to evaluate the relative toxicity of a number of fabrics, yarns, and polyurethane foams by the MST procedure. There was a wide range of sample weights required to kill 50% of the exposed rats. The LD_{50} for the nylon samples was in the range of 4.5-5.2 gm; this compares with 0.33 gm for Nomex (Y-1796), 0.19 gm for Durette (Y-1797), and 0.14 gm for Modified Nomex HT-4 (Y-2000). The LD_{50} of rayon was in the range of 9.5-10.0 gm; of acetate about 3.5 gm; while acrylic and polyester were about 1.5-2.0 gm. Fortrel-cotton and wool-nylon blends yielded an LD_{50} in the range of 2.0 gm. The LD_{50} of various wool samples were in the range of 0.3-1.3 gm, and orlon was about 0.3 gm. Flame retardant-treated cottons exhibited an LD_{50} in the range of 1.5-3.2 gm, while untreated cottons were generally 4.0-6.5 gm. The polyurethane foams yielded LD_{50} values from a little more than 1.5 gm to a little over 3.5 gm. The NASA polyurethane foam (Y-2190) gave a tentative LD_{50} of 1.01 gm, but a few additional experiments are in progress to confirm or modify this value.

Examination of COHb levels from blood of rats dying in the chamber were of such a nature that they suggested that carbon monoxide was a probable cause of death, or a major contributor to mortality, in the nylon samples (excluding the NASA samples),

acetate, polyester, rayon, fortrel-cotton and wool-nylon blends, flame retardant-treated and untreated cottons, and polyurethane foams. There were several instances, especially in the polyurethane group, where COHb levels were marginal or on the low side of what would be expected for death from carbon monoxide alone. Conversely, COHb levels in rats exposed to pyrolysates from the NASA nylon samples (Y-1796, Y-1797, and Y-2000), orlon, acrylic, and wool samples strongly suggest that carbon monoxide was not the sole cause of deaths. Cyanide and various other substances were often found in the pyrolysates from many of these materials.

While the above facts tend to indicate some general magnitudes of toxicity from fumes generated by the MST testing procedure, these should be considered only general guides. For any given class of fabric/material, the toxicity of thermally-generated fumes may be significantly altered by types of processing the basic material receives when it is manufactured. In addition, treatments to provide flame retardant or other special properties might also significantly affect the toxicity of the pyrolysate. Thus, any specific finished fabric or other type of material may exhibit a markedly different toxicity profile from that mentioned above for that particular class.

Toxic Components of Pyrolysates

Thermal degradation or pyrolysis of a fabric or other material typically produces quite a number of products in the pyrolysate. Carbon monoxide and carbon dioxide are almost universally formed; the formation of others are largely dependent upon

structure of material and degradation temperature. Hydrogen cyanide (or other cyanides) was also found to be a frequent component of the pyrolysates, and on occasion other gases (chlorine, ammonia, organic vapors, oxide[s] of nitrogen, etc.) were tentatively identified. However, these are not all equally toxic, but some may act synergistically with, or tend to potentiate, the toxicity of other toxicants in the pyrolysate.

Chamber atmospheric concentrations of CO, CO_2, and O_2, as determined by gas chromatography, using the NASA pyrolysis procedure, are shown in Table 6. These are tabulated according to test sample, weight of sample, chamber deaths, and mean COHb values for chamber deaths. Similar data are presented in Table 7, in which the NASA chamber atmosphere is screened with gas detector tubes for selected components. Table 8 shows the results of similar screening tests conducted with gas detector tubes on the atmosphere of the exposure chamber used in the MST procedure.

An assessment of the relative importance or contribution of carbon monoxide to the observed deaths may be inferred from the COHb levels in rats dying in the exposure chamber. However, blood from animals surviving acute exposure to carbon monoxide rapidly loses its value for this purpose since there is a relatively rapid dissociation of COHb in the rat when it is allowed to breathe room air. This is readily seen in Figure 21, in which rats were exposed to pure CO in air, removed from the CO-environment, and simply allowed to breathe room air; frequent blood samples were obtained and plotted as % COHb vs. time after removal from the CO-air atmosphere. About 1 1/2 minutes after

removal from the chamber, there was approximately 68% COHb, which decreased to 60% COHb after 5 minutes, 49% after 10 minutes, 28% after 30 minutes, and only 14% after 60 minutes. This illustrates why COHb levels in surviving rats are not reliable indices of COHb levels in the exposure chamber, unless the blood samples are taken almost immediately after exposure of the animals. This is further complicated by the protocol of the MST procedure: during the last 60 minutes the rats are in the chamber before removal, there is a continuing flow of fresh air into the chamber, but the furnace is turned off and the pyrolysate tends to be flushed out.

Special Tests

Two samples were subjected to special tests to determine the quantity of "hydrolyzable fluoride" liberated from the LD_{50} quantity of Fluorel (NASA procedure) and the quantity of HCN liberated from the LD_{50} quantity (NASA procedure) of Modified Nomex HT-4. As described under "Methods," the LD_{50} quantity of the sample was pyrolyzed and the fumes passed into the respective traps. Both agents were quantitated using the appropriate Orion ion-selective electrode.

Five replicate determinations of F^- were made using 5.82 gm of Fluorel in each determination. The mean fluoride content was 2.65% based upon original sample weight pyrolyzed; the individual tests yielded a range of values of 2.33-2.92%.

Although not a part of the contract, 1.527 gm of Modified Nomex HT-4 was pyrolyzed and the fumes passed through Ascarite to trap HCN. Extraction of cyanide from the Ascarite and its subsequent analysis with the ion-selective electrode revealed this

quantity of Modified Nomex generated 6.2526×10^{-3} gm, or 0.409% of cyanide, calculated as HCN on the basis of initial sample weight.

Behavioral Responses of Rats During Exposure to Pyrolysis Products

General behavior of the rats in the chamber during exposure to the pyrolysates is given in Table 9 for each sample tested by the NASA procedure and in Table 10 for those samples tested by the MST method. Although differences were observed between samples, behavioral patterns were not highly specific or useful in assessing the cause of deaths.

Post Mortem Studies

Animals dying in the chamber were autopsied and selected tissues preserved in 10% buffered formalin for histopathological evaluation. Similarly, randomly selected surviving animals were sacrificed after 48 hours and at the end of the observation period, were autopsied, and tissues were examined histologically. These results are in Table 11 (NASA procedure) and Table 12 (MST method).

General Comments Concerning Materials Tested by NASA Procedure

Within the sample size range of the fabrics (Y-1796, Y-1797, Y-1856, and Y-2000) employed in these studies, there appears to be a general trend for COHb levels in the rats that expired in the chamber to increase with increasing sample weight, with the possible exception of Y-2000. Presuming a direct correlation between CO concentration in the chamber and COHb levels in expired rats, one could then conclude that, for a particular sample, as the sample size is increased more carbon monoxide is generated. Although there is considerable fluctuation in COHb levels when compared to sample size of Y-2000, the trend does not seem to be a

significant increase of COHb levels with increasing sample sizes (Figure 22); however, data for HCN concentration in the chamber (Table 7) also suggests an increase in HCN with increasing sample size.

If one similarly examines the relationship between percent mortalities in the chamber vs. COHb levels in the rats (Figure 23), a more uniform increase in COHb is noted with increases in mortality for Y-2000 (Modified Nomex HT-4). The relatively low mean COHb levels found (approximately 45-50%) would, however, suggest another toxicant (probably HCN) is responsible for, or a major contributing factor to, the deaths of these animals. The physiological responses to an increase in carbon dioxide (e.g., hyperventilation) may also serve to increase the susceptibility of the rats to the lethal effects of other gases (e.g., CO, HCN).

Examination of the other three curves (Figure 23) reveals the mean COHb levels for several of these groups are below 60% relative COHb. Preliminary experiments in which rats were exposed to only CO-air showed the COHb level could reach 65-70% (or sometimes more) and still the rats could survive if they were promptly removed from the chamber and allowed to breathe room air. This, then, would make it seem possible that many of these deaths were due to something more than simply carbon monoxide intoxication. Comparable data for Y-1794 (Fluorel) is not available since the exposed rats typically did not die for several hours after removal from the chamber; this, too, suggests that carbon monoxide was not the primary cause of death. When 10 rats were exposed to the pyrolysate of the LD_{50} weight of Fluorel, there were

no deaths in the chamber, but their mean COHb level was 53.3% (range of 47.8-59.4%; see Table 13).

The relationship between percent COHb and percent mortalities (Figure 23) for Durette (Y-1797) showed a much higher COHb value in the 10%-mortality group than in the higher-mortality groups. In that regard, this one is a single value (1 death from 10 exposed rats), while the others are mean values. Also, COHb values of this magnitude were occasionally observed in various other groups of exposed rats; thus it was not unique. (At this COHb level [79%], carbon monoxide could readily be the sole toxicant responsible for death.) The pyrolysate from this fabric seems to produce a decrease in COHb levels as mortality is increased, until the mortality reaches 60-70%, after which COHb levels increase with increased mortality.

Summary Comments

Studies were conducted to determine the relative toxicity of pyrolysis products from selected materials by the NASA procedure and the MST method. The data suggest that the quantity of CO or HCN produced may be related to the size of sample pyrolyzed; this is likely to be true for other toxic gases as well. Although studies were not undertaken to determine the number and concentrations of all toxic gases or vapors, one would anticipate the profile of such compounds would also probably be affected by the size of sample combusted, particularly in the NASA procedure where a relatively oxygen-poor state could readily develop in the pyrolysis tube with the larger samples, as compared to the smaller samples. Generally, COHb levels of animals expiring in

the chamber also suggest higher concentrations of CO are produced with the larger samples of most materials; this trend is shown in Figure 23.

COHb levels from chamber deaths imply that carbon monoxide is unlikely to be the sole cause of death (for example, see Modified Nomex HT-4 [Y-2000] in Figure 23), while the marginal nature of certain other COHb levels makes the role of CO questionable in the deaths. Since both CO and HCN exert their lethal action quite rapidly, the occurrence of delayed deaths (hours or days after removal from the chamber), such as in the case of Fluorel, indicates there is another toxicant(s) in the fumes which plays a major role in its lethality.

Another factor which markedly affects the LD_{50} value is the temperature of thermodegradation. In some exploratory experiments (data not included in this report), higher pyrolysis temperatures (for example, 800°C rather than 600°C) seemed to produce more toxic fumes from the same sample weight; this may be partially due to more complete decomposition (with less residue) as well as a possible difference in thermodegradation products produced. A major investigation into the influence of temperature was not a part of this contract. This could, however, help explain why the samples appeared to be more toxic by the MST procedure than the NASA method, since the MST procedure almost invariably utilized a higher maximum temperature.

IV. APPENDIX

As specified in the contract, the following is a list of the equipment purchased with funds from the contract costing $100 or more:

Pyrolysis furnace, Lindberg model #55031	$ 132.60
Temperature Controller, Lindberg model #59344	468.00
Vacuum Pump, Cole-Parmer, #7165-20	208.95
Orion Digital pH/mv Meter, Model #801	1,095.00
Digital Thermistor Thermometer, Cole-Parmer, #8501-60	395.00

TABLE 1

MATERIALS SUBMITTED FOR EVALUATION UNDER NASA CONTRACT

Code No.	Identification	Description
Y-1794	Fluorel	A co-polymer of vinylidene fluoride and hexafluoropropylene; small rectangular pieces of beige sheet of material. From Dr. V. L. Carter, NASA.
Y-1795	Mosites	A cured silicone potting material in the form of a black, rubbery sheet, #140140-D. From Dr. V. L. Carter, NASA.
Y-1796	Nomex	A natural, antique-white fabric; an aromatic type of nylon, without flame retardants added; Style HT-90-40; PC #115-7; Lot #2258 (Stern and Stern). From Dr. V. L. Carter, NASA.
Y-1797	Durette	A gold-colored fabric; supposedly the same basic fabric as Y-1796 except for addition of flame retardant and color. #400-2 (Monsanto Research Corp.). From Dr. V. L. Carter, NASA.
Y-1856	Kevlar-29	A heavy-weave webbing material; from sample #615-17. From Dr. V. L. Carter, NASA.
Y-2000	Modified Nomex HT-4	Dark olive-green fabric, flame retardant-treated; HT-4. From Dr. V. Carter, NASA.
Y-2170	Asbestos Foam	A light-weight gray spongy material. From Dr. H. L. Kaplan, NASA.
Y-2188	Viton Sheet*†	A sheet of the Viton material to be used in coating polyurethane foam, ADL 150A. Received directly from Arthur D. Little Co. on request of Dr. H. L. Kaplan, NASA.
Y-2189	Viton Foam*	A sheet (~ 0.5 cm thick) of firm, spongy material, gray; supposedly of the same material as Y-2188. ADL 150B. Received directly from Arthur D. Little Co. on request of Dr. H. L. Kaplan, NASA.
Y-2190	Urethane Foam*	A blue, spongy foam in 4-5" discs, varying in thickness of about 0.6-1.0". ADL 150C. Received directly from Arthur D. Little Co. on request of Dr. H. L. Kaplan, NASA.

*In a telephone conversation (on or about July 11, 1974) with John Howard of A. D. Little Co., he indicated that all of their samples (Y-2188, 2189, and 2190) included a flame retardant, though he did not identify it.

†This material was not entered in the testing program of this year's contract.

TABLE 2

RESULTS OF THERMOGRAVIMETRIC ANALYSIS (TGA) STUDIES

Sample # (Y-#)	Y-1794	Y-1794*	Y-1794	Y-1795	Y-1795	Y-1796
Identification	Fluorel	Fluorel	Fluorel	Mosites	Mosites	Nomex
TGA Run No.	98	148 & 149	108	100	109	97
Atmosphere	Air	Nitrogen	Air	Air	Air	Air
Flow Rate	200 ml/min	200 ml/min	20 ml/min	200 ml/min	20 ml/min	200 ml/min
Heating Rate	10°C/min	10°C/min	20°C/min	10°C/min	20°C/min	10°C/min
Sample Weight	18.42 mg	9.88 mg	10.64 mg	16.92 mg	10.27 mg	9.84 mg
Initiation of Decomposition	140°C	224.2°C	245°C	184°C	238°C	272°C
Completion of Decomposition	831°C	898°C	1077.3°C	696°C	734°C	583°C
Maximum TGA Temp.	832°C	898°C	1077.3°C	961°C	1086.6°C	707°C
Final Residue Wt.	6.63 mg	4.03 mg	3.60 mg	10.08 mg	5.90 mg	0.04 mg
Percent Final Residue	36.0%	40.8%	33.8%	59.6%	57.4%	0.4%
Temp. for 50% Degradation	423°C	666.7°C	476°C	> 961°C	> 1086°C	483.6°C
Percent Residue at 600°C	41.2%	50.6%	44.2%	61.2%	61.1%	0.4%

*Mean values of duplicate determinations.

TABLE 2, cont.

RESULTS OF THERMOGRAVIMETRIC ANALYSIS (TGA) STUDIES

Sample # (Y-#)	Y-1796	Y-1796	Y-1797	Y-1797	Y-1797	Y-1856
Identification	Nomex	Nomex	Durette	Durette	Durette	Kevlar-29
TGA Run No.	128	110	101	129	111	107
Atmosphere	Nitrogen	Air	Air	Nitrogen	Air	Air
Flow Rate	200 ml/min	20 ml/min	200 ml/min	200 ml/min	20 ml/min	200 ml/min
Heating Rate	10°C/min	20°C/min	10°C/min	10°C/min	20°C/min	10°C/min
Sample Weight	10.0 mg	9.50 mg	9.0 mg	10.22 mg	9.55 mg	9.36 mg
Initiation of Decomposition	365°C	410°C	308°C	303°C	381.4°C	452.7°C
Completion of Decomposition	1025°C	684°C	579°C	1084°C	610.5°C	526.2°C
Maximum TGA Temp.	1048°C	992.5°C	707°C	1084°C	981°C	1002°C
Final Residue Wt.	0.32 mg	0.0 mg	0.0 mg	2.02 mg	0.0 mg	0.16 mg
Percent Final Residue	3.2%	0.0%	0.0%	19.8%	0.0%	1.7%
Temp. for 50% Degradation	622°C	552°C	488.6°C	640°C	528°C	485°C
Percent Residue at 600°C	57.4%	24.4%	0.0%	63.4%	0.7%	1.7%

TABLE 2, cont.

RESULTS OF THERMOGRAVIMETRIC ANALYSIS (TGA) STUDIES

Sample # (Y-#)	Y-1856	Y-1856	Y-2000*	Y-2000*	Y-2000*
Identification	Kevlar-29	Kevlar-29	Modified Nomex	Modified Nomex	Modified Nomex
TGA Run No.	150	106	130 & 132	133 & 134	131 & 135
Atmosphere	Nitrogen	Air	Air	Nitrogen	Air
Flow Rate	200 ml/min	20 ml/min	200 ml/min	200 ml/min	20 ml/min
Heating Rate	10°C/min	20°C/min	10°C/min	10°C/min	20°C/min
Sample Weight	8.50 mg	10.62 mg	10 mg	9.65 mg	9.94 mg
Initiation of Decomposition	523°C	417°C	285.7°C	280.5°C	298°C
Completion of Decomposition	933.7°C	667°C	725°C	977°C	797°C
Maximum TGA Temp.	933.7°C	1001°C	852.6°C	996°C	1019.5°C
Final Residue Wt.	0.30 mg	0.34 mg	0.0 mg	0.0 mg	0.0 mg
Percent Final Residue	3.5%	3.2%	0.0%	0.0%	0.0%
Temp. for 50% Degradation	644.6°C	529°C	490.5°C	696.8°C	524.5°C
Percent Residue at 600°C	63.5%	11.9%	8.5%	66.0%	11.7%

*Mean values of duplicate determinations.

TABLE 2, cont.

RESULTS OF THERMOGRAVIMETRIC ANALYSIS (TGA) STUDIES

Sample #(Y-#)	Y-2000*	Y-2189*	Y-2190*
Identification	Modified Nomex	Viton Foam	Urethane Foam
TGA Run No.	136 & 137	144 & 145	146 & 147
Atmosphere	Nitrogen	Air	Air
Flow Rate	20 ml/min	200 ml/min	200 ml/min
Heating Rate	20°C/min	10°C/min	10°C/min
Sample Weight	10.15 mg	10.06 mg	7.6 mg
Initiation of Decomposition	264.5°C	258°C	233.9°C
Completion of Decomposition	980°C	543.2°C	611.1°C
Maximum TGA Temp.	980°C	939°C	900.5°C
Final Residue Wt.	0.0 mg	1.26 mg	0.0 mg
Percent Final Residue	0.0%	12.5%	0.0%
Temp. for 50% Degradation	633.8°C	447°C	302°C
Percent Residue at 600°C	57.2%	12.5%	2.1%

*Mean values of duplicate determinations.

TABLE 3

RESUMÉ OF TOXICITY OF PYROLYSIS PRODUCTS OF NASA SAMPLES

TEST SAMPLE		LD_{50} VALUES (95% CONFIDENCE INTERVAL)[a]	
		NASA Procedure (7-day)[b]	MST Procedure (14-day)
Y-1794	Fluorel	5.78 gm (5.53-6.03)	-----
Y-1795	Mosites	> 50.0 gm[c]	d
Y-1796	Nomex	7.32 gm (6.70-8.00)	0.36 gm (0.31-0.41)
Y-1797	Durette	1.83 gm (1.75-1.91)	0.205 gm (0.18-0.24)
Y-1856	Kevlar-29	3.12 gm (2.90-3.36)	1.67 gm (1.58-1.76)
Y-2000	Modified Nomex	1.53 gm (1.40-1.67)	0.15 gm (0.14-0.15)[e]
Y-2170	Asbestos Foam	Not tested by either procedure. TGA data from NASA (Houston) indicated that there was no thermal degradation in the temperature range employed in these studies. Therefore, upon direction of the Technical Monitor for the contract, this material was not entered into the testing procedure.	
Y-2189	Viton Foam	Not scheduled to be tested by this procedure.	
Y-2190	Urethane Foam	Not scheduled to be tested by this procedure.	

[a]Expressed as weight of sample necessary to kill 50% of the exposed rats when pyrolyzed according to respective protocols. LD_{50} calculations were by the Cornfield and Mantel modification of Karber's method

[b]Most of these animals were observed for 14 days for mortalities, but there were no deaths in these groups in the 8- to 14-day period. Thus, LD_{50} sample weight differences between these 2 methods appear to be due to the method rather than a difference in observation periods.

[c]Lethality was not determined. Different sample weights, up through 50 gm (about the maximum the pyrolysis tube would accomodate), were combusted with the standard pyrolysis procedure. No rats died from exposures.

[d]Preliminary tests with this sample in this particular apparatus indicated formation of an explosive gas (possibly silicon hydrides or silanes) during this type of thermodegradation. Since the facility was not equipped to safely handle potentially explosive gases, no further tests were made on this sample by this pyrolysis method.

[e]This is an $LSW_{2/4}$ value, which is quantitatively comparable to the LD_{50}. See Table for additional comparisons of the two types of values.

TABLE 4

COMPARISON OF LD_{50} AND $LSW_{2/4}$ VALUES AND THEIR 95% CONFIDENCE INTERVALS*

SAMPLE	PYROLYSIS TEST & OBSERVATION TIME	LD_{50} & 95% Confidence Interval	$LSW_{2/4}$ & 95% Confidence Interval
Y-1794 Fluorel	NASA (chamber)	***	***
	NASA (48-hour)	5.82 gm (5.56-6.08)	5.96 gm (5.64-6.31)
	NASA (7-day)**	5.78 gm (5.53-6.03)	5.91 gm (5.62-6.22)
Y-1796 Nomex	NASA (chamber)	7.32 gm (6.70-8.00)	7.31 gm (6.53-8.19)
	NASA (48-hour)	7.32 gm (6.70-8.00)	7.31 gm (6.53-8.19)
	NASA (7-day)**	7.32 gm (6.70-8.00)	7.31 gm (6.53-8.19)
	MST (14-day)	0.36 gm (0.31-0.41)	0.33 gm (0.24-0.47)
Y-1797 Durette	NASA (chamber)	1.83 gm (1.75-1.91)	1.84 gm (1.71-1.99)
	NASA (48-hour)	1.83 gm (1.75-1.91)	1.84 gm (1.71-1.99)
	NASA (7-day)**	1.83 gm (1.75-1.91)	1.84 gm (1.71-1.99)
	MST (14-day)	0.205 gm (0.18-0.24)	0.19 gm (0.17-0.21)
Y-1856 Kevlar-29	NASA (chamber)	3.12 gm (2.90-3.36)	3.02 gm (2.51-3.63)
	NASA (48-hour)	3.12 gm (2.90-3.36)	3.02 gm (2.51-3.63)
	NASA (7-day)**	3.12 gm (2.90-3.36)	3.02 gm (2.51-3.63)
	MST (14-day)	1.67 gm (1.58-1.76)	1.63 gm (1.52-1.76)
Y-2000 Modified Nomex	NASA (chamber)	1.57 gm (1.41-1.71)	****
	NASA (48-hour)	1.55 gm (1.42-1.69)	1.53 gm (1.35-1.74)
	NASA (7-day)**	1.53 gm (1.40-1.67)	1.52 gm (1.34-1.72)
	MST (14-day)	****	0.15 gm (0.14-0.15)
Y-2189 Viton Foam	MST (14-day)	2.35 gm (2.14-2.57) (75% of mortalities were delayed deaths)	2.51 gm (2.33-2.69)
Y-2190 Urethane Foam	MST (14-day)	1.03 gm (0.87-1.22) (27% of mortalities were delayed deaths; tentative values. Additional tests will be conducted.)	1.01 gm (0.66-1.54)

*The LD_{50} and 95% confidence intervals were calculated by Cornfield and Mantel's modification of Karber's method; the $LSW_{2/4}$ (lethal sample weight for 2 of the 4 exposed rats) and 95% confidence intervals were calculated by an arc-tangent procedure developed by L. J. Nunez, MST Labs. All calculations for the MST procedure are based upon a 14-day observation period.

**Many of the surviving rats from these exposures were observed for 14 days post-exposure; there were no deaths in the 8- to 14-day period.

***Could not calculate. Only 5 animals in 1 group died in chamber; the other deaths were outside the chamber, most within 48 hours.

****Not calculated.

TABLE 5

ATMOSPHERIC CONCENTRATION OF CO, CO_2, AND O_2 IN EXPOSURE CHAMBER AFTER THERMODEGRADATION OF TEST SAMPLE, NASA METHOD (DETERMINED BY GAS CHROMATOGRAPHY)

Sample Weight	Mortality in Chamber	Mean COHb*	Time of Sample (min.)	Carbon Monoxide	Carbon Dioxide	Oxygen
Y-1794 Fluorel (48-hour LD_{50} = 5.82 gm)						
5.15 gm	0/10	NA	0	1.5 ppth	3.7 ppth	20.9%
			15	1.6 ppth	7.4 ppth	20.9%
			30	---	---	---
			Mean	1.6 ppth	5.6 ppth	20.9%
5.51 gm	0/10	NA	0	2.0 ppth	3.5 ppth	20.9%
			15	1.0 ppth	6.1 ppth	20.9%
			30	1.9 ppth	12.7 ppth	20.9%
			Mean	1.6 ppth	7.4 ppth	20.9%
6.31 gm	0/10	NA	0	1.5 ppth	3.6 ppth	20.9%
			15	0.9 ppth	6.6 ppth	20.9%
			30	0.9 ppth	12.2 ppth	20.9%
			Mean	1.1 ppth	7.5 ppth	20.9%
Y-1796 Nomex (48-hour LD_{50} = 7.32 gm)						
4.50 gm	0/10	NA	0	---	---	---
			15	1.5 ppth	11.7 ppth	normal
			30	2.7 ppth	12.1 ppth	normal
			Mean	2.1 ppth	11.9 ppth	normal
5.90 gm	2/10	55.8%	0	---	---	---
			15	2.8 ppth	11.0 ppth	20.9%
			30	---	---	---
			Mean	2.8 ppth	11.0 ppth	20.9%
6.75 gm	4/10	56.9%	0	1.7 ppth	7.0 ppth	---
			15	3.3 ppth	10.7 ppth	20.9%
			30	3.1 ppth	14.7 ppth	---
			Mean	2.7 ppth	10.8 ppth	20.9%
7.73 gm	6/10	59.9%	0	1.9 ppth	5.6 ppth	normal
			15	---	---	---
			30	1.9 ppth	13.2 ppth	normal
			Mean	1.9 ppth	9.4 ppth	normal
10.13 gm	10/10	63.4%	0	1.0 ppth	4.9 ppth	normal
			15	2.3 ppth	13.1 ppth	normal
			30	2.7 ppth	12.7 ppth	normal
			Mean	2.0 ppth	10.2 ppth	normal

*Of animals dying in the chamber.

TABLE 5, cont.

Sample Weight	Mortality in Chamber	Mean COHb*	Time of Sample (min.)	Carbon Monoxide	Carbon Dioxide	Oxygen
Y-1797 Durette (48-hour LD_{50} = 1.83 gm)						
1.53 gm	0/10	NA	0	2.8 ppth	13.7 ppth	normal
			15	2.5 ppth	19.6 ppth	normal
			30	2.8 ppth	23.7 ppth	normal
			Mean	2.7 ppth	19.0 ppth	normal
1.63 gm	1/10	79.0%	0	1.9 ppth	9.4 ppth	normal
			15	---	---	---
			30	2.1 ppth	19.8 ppth	normal
			Mean	2.0 ppth	14.6 ppth	normal
1.75 gm	5/10	64.1%	0	1.6 ppth	9.0 ppth	normal
			15	---	15.8 ppth	normal
			30	1.8 ppth	20.4 ppth	normal
			Mean	1.7 ppth	15.1 ppth	normal
2.00 gm	8/10	----	0	2.4 ppth	12.0 ppth	normal
			15	---	---	---
			30	---	19.0 ppth	normal
			Mean	2.4 ppth	15.5 ppth	normal
2.29 gm	9/10	64.9%	0	2.6 ppth	10.4 ppth	normal
			15	2.7 ppth	17.8 ppth	normal
			30	2.2 ppth	16.8 ppth	19.0%
			Mean	2.5 ppth	15.0 ppth	normal
Y-1856 Kevlar-29 (48-hour LD_{50} = 3.12 gm)						
3.00 gm	4/10	61.1%	0	0** ppth	4.5 ppth	21.5%
			15	0** ppth	---	19.5%
			30	0** ppth	11.0 ppth	19.5%
			Mean	---	7.8 ppth	20.2%
3.43 gm	7/10	64.0%	0	< 0.5 ppth	10.5 ppth	18.8%
			15	0** ppth	8.5 ppth	21.5%
			30	0** ppth	11.3 ppth	21.5%
			Mean	---	10.1 ppth	20.6%
Y-2000 Modified Nomex HT-4 (48-hour LD_{50} = 1.55 gm)						
1.53 gm	6/10	51.3%	0	1.4 ppth	3.8 ppth	20.9%
			15	1.8 ppth	8.6 ppth	20.9%
			30	2.0 ppth	10.1 ppth	20.9%
			Mean	1.7 ppth	7.5 ppth	20.9%
1.75 gm	6/10	41.3%	0	1.9 ppth	3.8 ppth	20.9%
			15	2.3 ppth	7.9 ppth	20.9%
			30	2.0 ppth	9.1 ppth	20.9%
			Mean	2.1 ppth	6.9 ppth	20.9%

**Data from gas detector tubes and COHb levels indicate these results are incorrect.

TABLE 6

ATMOSPHERIC CONCENTRATION OF CO, HCN, AND OTHER SELECTED GASES IN EXPOSURE CHAMBER AFTER THERMODEGRADATION OF TEST SAMPLE, NASA METHOD (DETERMINED BY BENDIX GAS DETECTOR TUBES)

Sample Weight	Mortality in Chamber	Mean COHb*	Carbon Monoxide	Hydrogen Cyanide	Others
Y-1794 Fluorel (48-hour LD_{50} = 5.82 gm)					
4.82 gm	0/10	NA	1.0 ppth	10 ppm	
5.15 gm	0/10	NA	---	---	
5.51 gm	0/10	NA	0.8 ppth	---	
5.90 gm	0/10	NA	1.3 ppth	15 ppm	
6.75 gm	5/10	60.9%	1.6 ppth	20 ppm	
7.22 gm	0/10	NA	1.5 ppth	16 ppm	
Y-1796 Nomex (48-hour LD_{50} = 7.32 gm)					
4.50 gm	0/10	NA	2.0 ppth	---	
5.15 gm	1/10	54.7%	2.7 ppth	---	
6.75 gm	4/10	56.9%	> 3.0 ppth	---	
7.73 gm	6/10	59.9%	> 3.0 ppth	---	
8.84 gm	6/10	57.8%	> 3.0 ppth	---	
10.13 gm	10/10	63.4%	> 3.0 ppth	---	
Y-1797 Durette (48-hour LD_{50} = 1.83 gm)**					
2.14 gm	9/10	60.5%	> 3.0 ppth	13 ppm	chlorine, 15 ppm
2.29 gm	9/10	64.8%	> 3.0 ppth	25 ppm	chlorine, 18 ppm
Y-1856 Kevlar-29 (48-hour LD_{50} = 3.12 gm)					
2.29 gm	0/10	NA	2.1 ppth	---	
2.65 gm	3/10	50.3%	2.3 ppth	---	
3.00 gm	4/10	61.1%	3.4 ppth	---	
3.43 gm	7/10	64.0%	4.5 ppth	---	
3.93 gm	9/10	63.5%	4.8 ppth	---	
4.50 gm	9/10	66.0%	4.2 ppth	---	acetone, 4 ppth
Y-2000 Modified Nomex, HT-4 (48-hour LD_{50} = 1.55 gm)					
1.02 gm	0/10	NA	1.4 ppth	20 ppm	
1.16 gm	2/10	46.1%	1.5 ppth	15 ppm	
1.33 gm	2/10	41.2%	1.6 ppth	25 ppm	
1.53 gm	6/10	51.3%	1.6 ppth	50 ppm	
1.75 gm	6/10	41.3%	1.6 ppth	50 ppm	
2.00 gm	8/10	50.5%	1.8 ppth	60 ppm	
2.29 gm	10/10	50.2%	1.9 ppth	40 ppm	

*Of animals dying in the chamber.

**These particular detector tubes had a maximum concentration of 0.3% CO.

TABLE 7

SCREENING OF RAT EXPOSURE CHAMBER ATMOSPHERE FOR PRESENCE OF CERTAIN GASES FOLLOWING THERMAL DEGRADATION OF NASA SAMPLES, MST PROCEDURE DETECTOR TUBE METHOD*

Sample	Identification	Positive Test for Presence	Negative Test for Presence
Y-1796	Nomex	Carbon Monoxide Carbon Dioxide Hydrogen Cyanide Chlorine	Ammonia Hydrogen Sulfide Sulfur Dioxide Organic Vapors
Y-1797	Durette	Carbon Monoxide Carbon Dioxide Hydrogen Cyanide	Ammonia Chlorine Hydrogen Sulfide Sulfur Dioxide Organic Vapors
Y-1856	Kevlar-29	Carbon Monoxide Carbon Dioxide Hydrogen Cyanide Ammonia Organic Vapors Ozone Nitrous Oxide and/or Nitrogen Dioxide	Chlorine Hydrogen Sulfide Sulfur Dioxide Phosphine Phosgene
Y-2000	Modified Nomex HT-4	Carbon Monoxide Carbon Dioxide Hydrogen Cyanide	Ammonia Chlorine Hydrogen Sulfide Sulfur Dioxide Organic Vapors
Y-2189	Viton Foam	Carbon Monoxide Carbon Dioxide Hydrogen Cyanide Organic Vapors	Ammonia Chlorine Hydrogen Sulfide Sulfur Dioxide Ozone Phosphine Nitrous Oxide and/or Nitrogen Dioxide Phosgene
Y-2190	Urethane Foam	Carbon Monoxide Carbon Dioxide Hydrogen Cyanide Organic Vapors	Ammonia Chlorine Hydrogen Sulfide Sulfur Dioxide

*A positive test for the presence of any of these gases/vapors should be considered presumptive evidence that the substance is present in the chamber atmosphere. However, since various other compounds may interfere with the test, and since there is an unknown mixture of gases present, confirmation of the presence of any compound of major interest would be necessary by some other method. The quantitative nature of this test is also subject to these interferences; the positive tests ranged in intensity from a trace to significant measurements by the detector tubes.

TABLE 8

GENERAL BEHAVIORAL RESPONSES OF RATS TO EXPOSURE TO THERMODEGRADATION PRODUCTS OF NASA SAMPLES, NASA PROCEDURE

Y-1794 Fluorel

Within the first minute after introduction into the chamber, the rats showed evidence of squinting and lacrimation. In about 5 minutes, breathing became somewhat labored. Later, during the exposure, the rats exhibited dyspnea, squinting, and ataxia. The rats' behavior returned to normal rather quickly after removal from the chamber except for ataxia. Signs of slight ataxia persisted for several minutes.

Y-1795 Mosites

Although thermodegradation of this sample (up to 50 gm initial weight) failed to kill any rats, mild behavioral responses were observed. Lacrimation was first to occur, within about 5 minutes of introduction into the chamber. This was followed later by labored breathing. Upon removal from the chamber, the rats became normal after a few minutes.

Y-1796 Nomex

Shortly after introduction into the chamber, the rats exhibited lacrimation and, shortly thereafter, labored breathing. Later they developed severe dyspnea and ataxia, and occasionally mild tremors were seen. After they were removed from the chamber, the rats' respiration became normal and ataxia disappeared within a few minutes.

Y-1797 Durette

Lacrimation and hyperactivity were noted shortly after the rats were introduced into the exposure chamber. Mild dyspnea soon developed and tended to become more severe during the latter part of the exposure period. Ataxia was commonly seen, and some rats exhibited tremors. Breathing became normal and ataxia disappeared a few minutes after removal from the chamber. No additional tremors were noted, and behavior returned to normal.

Y-1856 Kevlar-29

Early in the exposure period lacrimation and slightly labored breathing were noted in the rats. Later ataxia and severe dyspnea were seen. Breathing became normal promptly upon removal

TABLE 8, cont.

of the survivors from the chamber. After a brief period of ataxia, their behavior became normal.

Y-2000 Modified Nomex HT-4

Lacrimation and hyperactivity were observed soon after the rats entered the chamber. Shortly thereafter, severe dyspnea and ataxia (including loss of balance) were noted. At the same time many rats exhibited tremors. Ophthalmic irritation from the fumes was apparently pronounced, because squinting was common, especially during the latter part of the exposure period. When removed from the chamber, the rats still experienced labored breathing and, in some, it was quite irregular. Most of the rats showed a post-exposure period of ataxia of about 1 hour. Some rats were still inactive at the end of the 2-hour observation period.

TABLE 9

GENERAL BEHAVIORAL RESPONSES OF RATS TO EXPOSURE TO THERMODEGRADATION PRODUCTS OF NASA SAMPLES, MST PROCEDURE

Y-1794 Fluorel

Only a few preliminary tests were conducted on this polymer by the MST procedure. The fumes from this material caused squinting, mild to moderate dyspnea, ataxia, hyperactivity, convulsions, coma, and death. This material was not designated for complete evaluation by this method.

Y-1795 Mosites

Because of the explosive nature of the fumes generated by this procedure in preliminary experiments, Mosites was not evaluated by this method.

Y-1796 Nomex

The rats exposed to these fumes exhibited moderate to severe dyspnea, hyperactivity, ataxia, convulsions, tremors of extremities, coma, and death.

Y-1856 Kevlar-29

The rats showed mild to moderate dyspnea, hyperactivity, ataxia, coma, and death. Muscle tremors were also noted in some animals.

Y-2000 Modified Nomex HT-4

The rats revealed mild to severe dyspnea, hyperactivity, ataxia, convulsions, coma, and death. Sometimes muscle tremors were present.

Y-2189 Viton Foam

The signs noted in rats were: mild to moderate dyspnea, nasal discharge, hyperactivity, ataxia, coma, and death. Delayed deaths (i.e., after removal from the exposure chamber) were fairly common.

TABLE 9, cont.

Y-2190 Urethane Foam

Exposure of rats to fumes of this material produced mild to moderate dyspnea, hyperactivity, ataxia, coma, and death. Delayed deaths (after removal from the chamber) were fairly common.

TABLE 10

AUTOPSY FINDINGS IN RATS EXPOSED TO THERMODEGRADATION PRODUCTS OF NASA SAMPLES, NASA PROCEDURE

Sample Tested	Gross Autopsy	Histopathologic Examination
Y-1794 Fluorel	Lungs, heart, liver, kidneys, and spleen were congested; lungs contained bright red spots (hemorrhagic) on the surface of each lobe. Some hemorrhage of internal organs was noted. When the rats survived more than 48 hours, there was still some congestion of lungs present.	Rats dying within 24 hours of exposure appear to have similar morphological changes, the most outstanding features being pulmonary edema and varying degrees of congestion. Sometimes bronchopneumonia was also noted. The liver, heart, spleen, and kidneys showed mild to moderate congestion. Rats dying 24 hours or more after exposure exhibited pulmonary edema, diffuse severe congestion, focal abscesses, suppurative bronchitis, and moderate to severe acute bronchopneumonia. The liver, heart, spleen, and kidneys were also congested.
Y-1795 Mosites	No deaths were noted within 2 weeks of exposure to fumes generated from up to 50 gm (initial weight) of the silicone sample.	NA
Y-1796 Nomex	The liver, heart, and lungs were congested. Black spots were noted on surface of each lobe of the lungs. Liver was congested and enlarged.	Tissues of rats which died in the chamber revealed moderate to marked congestion (lungs, heart, liver, spleen, and kidneys). The lungs also showed edema, atelectasis, and sometimes focal severe hemorrhage. Tissues from rats sacrificed 48 hours after exposure showed mild, diffuse congestion of the heart; diffuse severe edema and congestion of lungs; diffuse mild congestion of liver; moderate medullary congestion of kidneys; and moderate perifollicular congestion of spleen.

TABLE 10, cont.

Sample Tested	Gross Autopsy	Histopathologic Examination
Y-1797 Durette	The heart, liver, and lungs were congested. Some bright red patches were noted on the surface of each lobe of the lung.	The primary acute toxic response appeared in the lungs. They exhibited moderate to marked edema and congestion. Moderate to severe atelectasis was also noted. In addition, one rat had bronchopneumonia. The rats sacrificed 48 hours or more after exposure revealed diffuse severe edema and congestion of lungs; some showed diffuse severe atelectasis, and some had acute bronchitis. Most rats had diffuse marked congestion of heart, liver, spleen, and kidneys.
Y-1856 Kevlar-29	Heart, liver, lungs, and kidneys of rats dying in the chamber were congested, and liver was enlarged. The spleen was bright red. Rats sacrificed 48 hours after exposure did not show marked congestion of heart, liver, lungs, and kidneys as seen in the chamber deaths. Brown spots, however, were noted on the surface of each lobe of the lungs.	The major acute toxic effects were found in the lungs. There was moderate to marked edema and congestion with mild to moderate focal hemorrhages. Also, one rat exhibited acute bronchiolitis. Varying degrees of congestion and petechiae were seen in the heart, liver, spleen, and kidneys. Animals sacrificed 48 hours post-exposure revealed pulmonary lesions consisting of atelectasis, hyperplasia of the alveolar lining, and a macrophage infiltrate. There was congestion in the heart, liver, spleen, and kidneys, but to a lesser degree than in those rats dying in the chamber.
Y-2000 Modified Nomex, HT-4	In the chamber deaths, the liver, heart, lungs, and kidneys were congested and bright red. The spleen was congested, but was dark in color in most rats. The lungs contained bright red patches on the surface of each lobe. All lobes of the livers were very fragile, with some internal hemorrhages.	Rats dying in the chamber showed varying degrees of edema and congestion of the lungs. Atelectasis, atypical hyperplasia, suppurative bronchitis, and sometimes bronchopneumonia were seen. Other than edema and congestion, lesions in the heart, liver, spleen, and kidneys were nonspecific. The rats sacrificed after 48 hours revealed diffuse marked congestion and mild focal edema of lungs, and diffuse marked congestion of liver, spleen, and kidneys.

TABLE 11

AUTOPSY FINDINGS IN RATS EXPOSED TO THERMODEGRADATION PRODUCTS OF NASA SAMPLES
MST PROCEDURE

Sample Tested	Gross Autopsy	Histopathologic Examination
Y-1794 Fluorel	Congestion was noted in heart, brain, and lungs. Liver and spleen were dark brown in some cases, and cherry-red in others. The trachea and kidneys were normal. (NOTE: This sample was not designated for complete evaluation by this method. Thus, these data are from a few preliminary experiments.)	Mild to severe diffuse congestion, focal hemorrhage, focal moderate atelectasis, and alveolar hyperplasia were noted in the lungs of the exposed rats. There was also mild diffuse congestion of the brain, heart, kidneys, liver, and spleen. Additionally, perifollicular hemorrhage of the spleen was present. (See note under "Gross Autopsy".)
Y-1795 Mosites	Because of the explosive nature of fumes generated by this procedure in preliminary experiments, Mosites was not evaluated by this method.	
Y-1796 Nomex	Kidneys and trachea appeared normal. Heart, brain, and lungs were congested. Liver and spleen were usually dark reddish-brown, but sometimes they were cherry-red.	Severe diffuse congestion, mild to moderate diffuse atelectasis, alveolar hyperplasia, and sometimes emphysema were noted in the lungs of the exposed rats. There was also mild to moderate congestion of the liver, heart, brain, spleen, and kidneys.
Y-1797 Durette	Brain, heart, and lungs revealed congestion. Liver and spleen were usually cherry-red, but sometimes the spleen was dark reddish-brown. Kidneys and trachea were normal.	Varying degrees of atelectasis and petechial hemorrhage of the lung present (mild to severe); petechial hemorrhagic meninges of the brain; diffuse focal hemorrhage and moderate ventricular dilation of the heart; moderate diffuse peripheral lobular congestion of liver; moderate red pulp congestion of spleen; and moderate diffuse congestion of kidneys.
Y-1856 Kevlar-29	Brain, heart, and lungs were congested. Liver and spleen were dark reddish-brown; kidneys and trachea were normal.	Varying degrees of congestion and edema of brain, heart, lung, liver, spleen, and kidneys (mild to severe). Some specimens revealed severe diffuse atelectasis and alveolar hyperplasia, and mucosal erosion of the trachea.

TABLE 11, cont.

AUTOPSY FINDINGS IN RATS EXPOSED TO THERMODEGRADATION PRODUCTS OF NASA SAMPLES
MST PROCEDURE

Sample Tested	Gross Autopsy	Histopathologic Examination
Y-2000 Modified Nomex HT-4	Congestion noted in heart, brain, and lungs. Hemorrhage seen in brain and lungs of one rat. Spleen and liver were cherry-red to dark reddish-brown; kidneys and trachea were normal.	Tissues from the exposed rats showed mild focal atelectasis and focal hemorrhage of lungs, and severe congestion of liver and spleen. No abnormalities were seen in the brain, heart, kidneys, or tracheal tissues.
Y-2189 Viton Foam	Trachea and kidneys were normal. Brain, heart, and lungs were congested, and lungs were edematous. Both liver and lungs were a dark reddish-brown.	Mild to severe diffuse congestion of the brain, heart, lungs, liver, kidneys, and spleen, with petechiae, were seen. Heart and brain were edematous. Trachea was normal. Moderate diffuse pneumonia was noted in those rats dying at various times after exposure (delayed deaths).
Y-2190 Urethane Foam	Trachea and kidneys appeared normal. Brain, heart, and lungs were congested. Liver and spleen were a dark reddish-brown.	Focal moderate to severe alveolar hyperplasia and atelectasis of lungs, and mild diffuse congestion of liver. Kidneys and spleen were normal. One rat exhibited mild diffuse congestion of heart and brain, while another showed focal erosion of the trachea.

TABLE 12

COMPARISON OF BLOOD CARBOXYHEMOGLOBIN LEVELS IN RATS FROM EXPOSURE TO FUMES OF THE LD50 SAMPLE WEIGHT OF NASA SAMPLES USING THE NASA PYROLYSIS AND EXPOSURE PROCEDURE*

Sample Number	Identification	48-Hour LD_{50} Wt.	Mean COHb Levels (and Range) in Rats Immediately After Exposure to LD_{50} Weight of Sample	
			Those Dying in Chamber [# of rats]	Those Living at Time of Removal from Chamber [# of rats]
Y-1794	Fluorel	5.82 gm	[none]	53.3% (47.8-59.4%) [10]
Y-1795	Mosites	> 50 gm	NA	NA
Y-1796	Nomex	7.32 gm	62.7% (55.1-75.1%) [4]	67.3% (58.1-76.1%) [6]
Y-1797	Durette	1.83 gm	56.8% (----------) [1]	59.4% (47.0-74.0%) [9]
Y-1856	Kevlar-29	3.12 gm	61.6% (50.6-75.0%) [6]	53.4% (46.1-64.4%) [4]
Y-2000	Modified Nomex	1.55 gm	59.5% (50.5-69.3%) [2]	54.2% (50.8-64.3%) [8]

*Combustion temperature = 600°C.

Figure 1

TGA Run No. 98

Y-1794 Fluorel
Atmosphere: Air
Flow Rate: 200 ml/min
Heating Rate: 10°C/min
Sample Weight: 18.42 mg

41.2% Residue @ 600°C
50.0% Residue @ 423°C
36.0% Residue @ 832°C

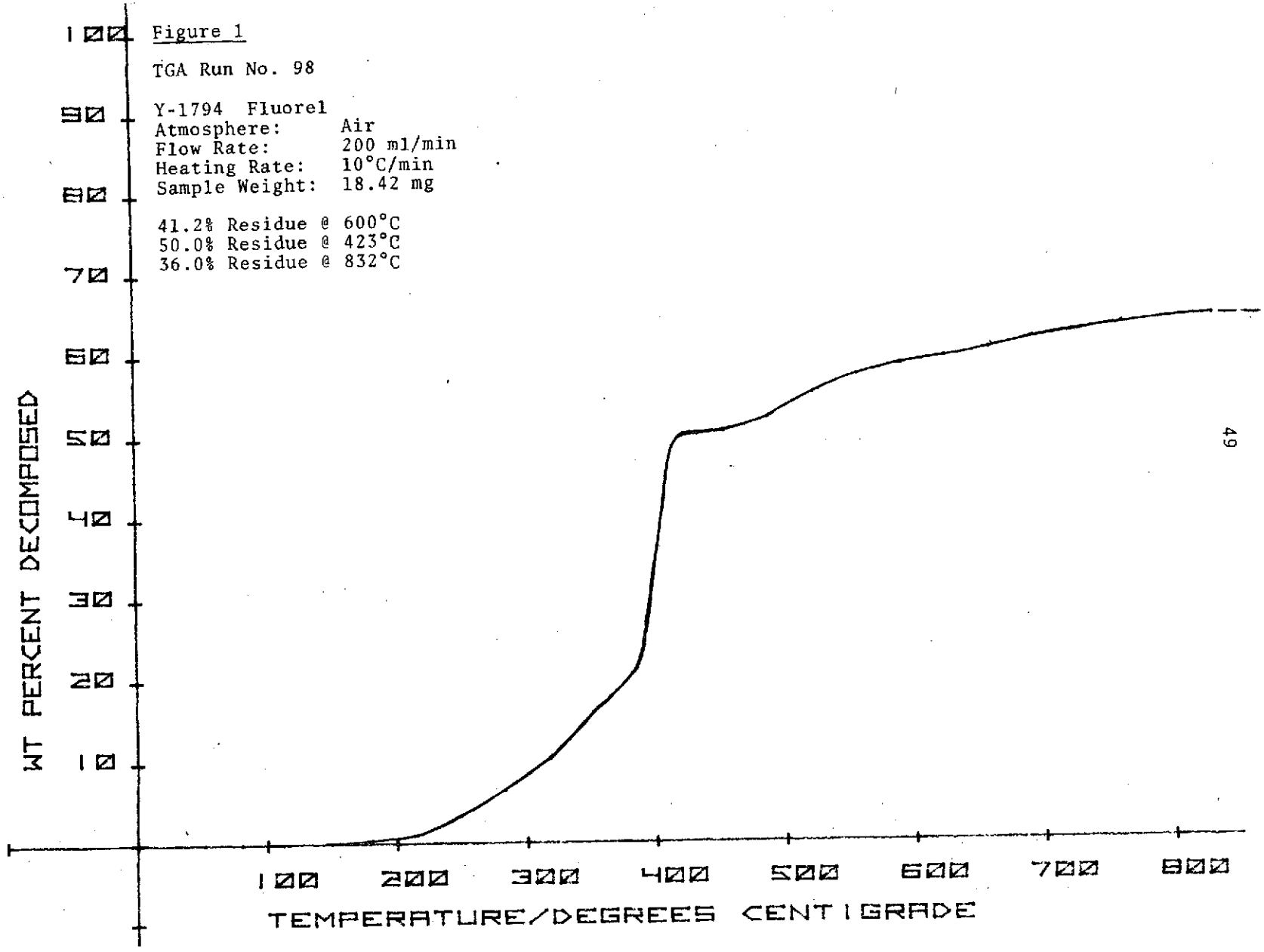


Figure 2

TGA Run No. 149

Y-1794 Fluorel
Atmosphere: Nitrogen
Flow Rate: 200 ml/min
Heating Rate: 10°C/min
Sample Weight: 9.88 mg

50.6% Residue @ 600°C
50.0% Residue @ 666.7°C
40.8% Residue @ 898°C

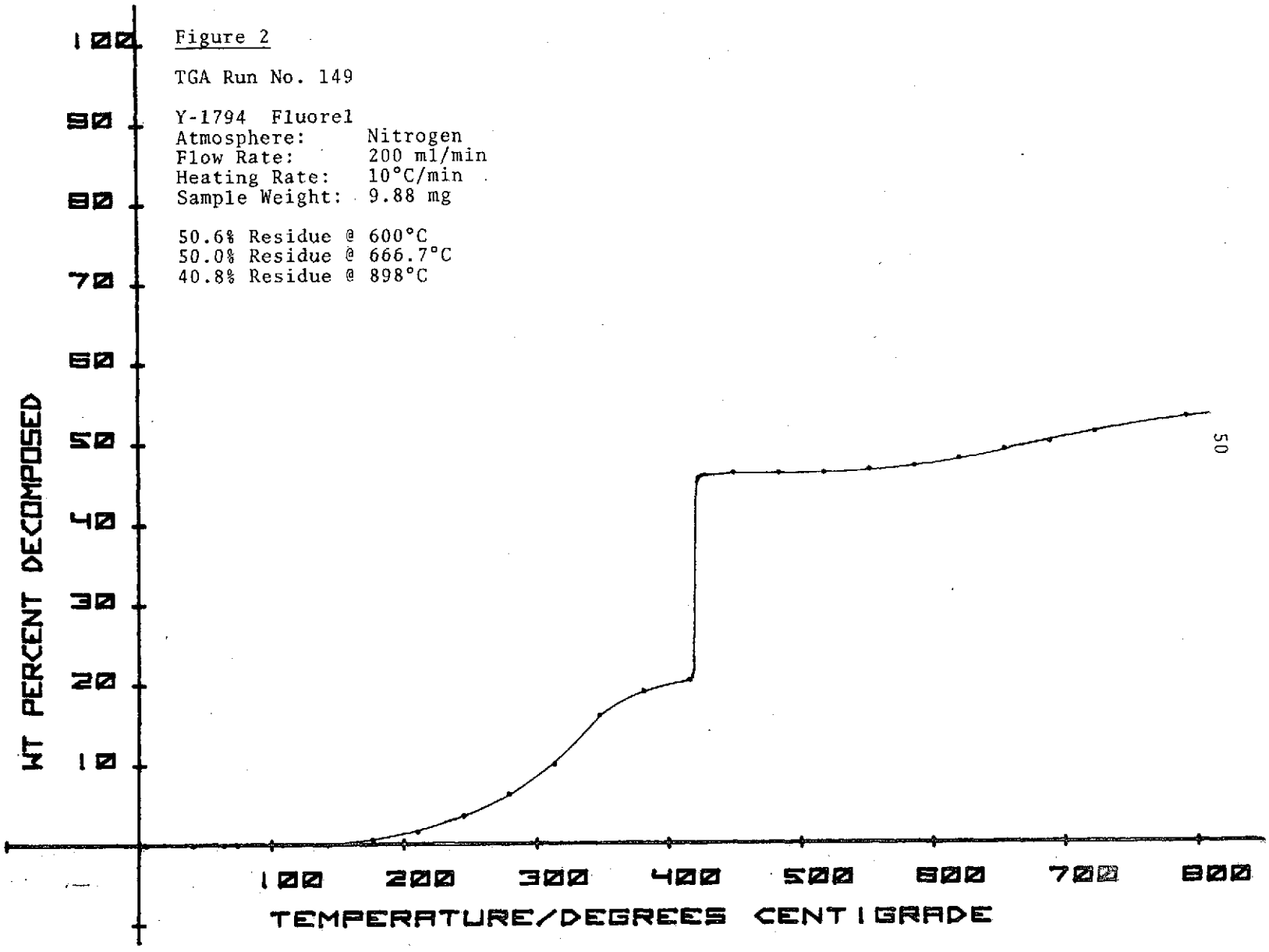


Figure 3

TGA Run No. 108

Y-1794 Fluorel
Atmosphere: Air
Flow Rate: 20 ml/min
Heating Rate: 20°C/min
Sample Weight: 10.64 mg

44.2% Residue @ 600°C
50.0% Residue @ 476°C
33.8% Residue @ 1077.3°C

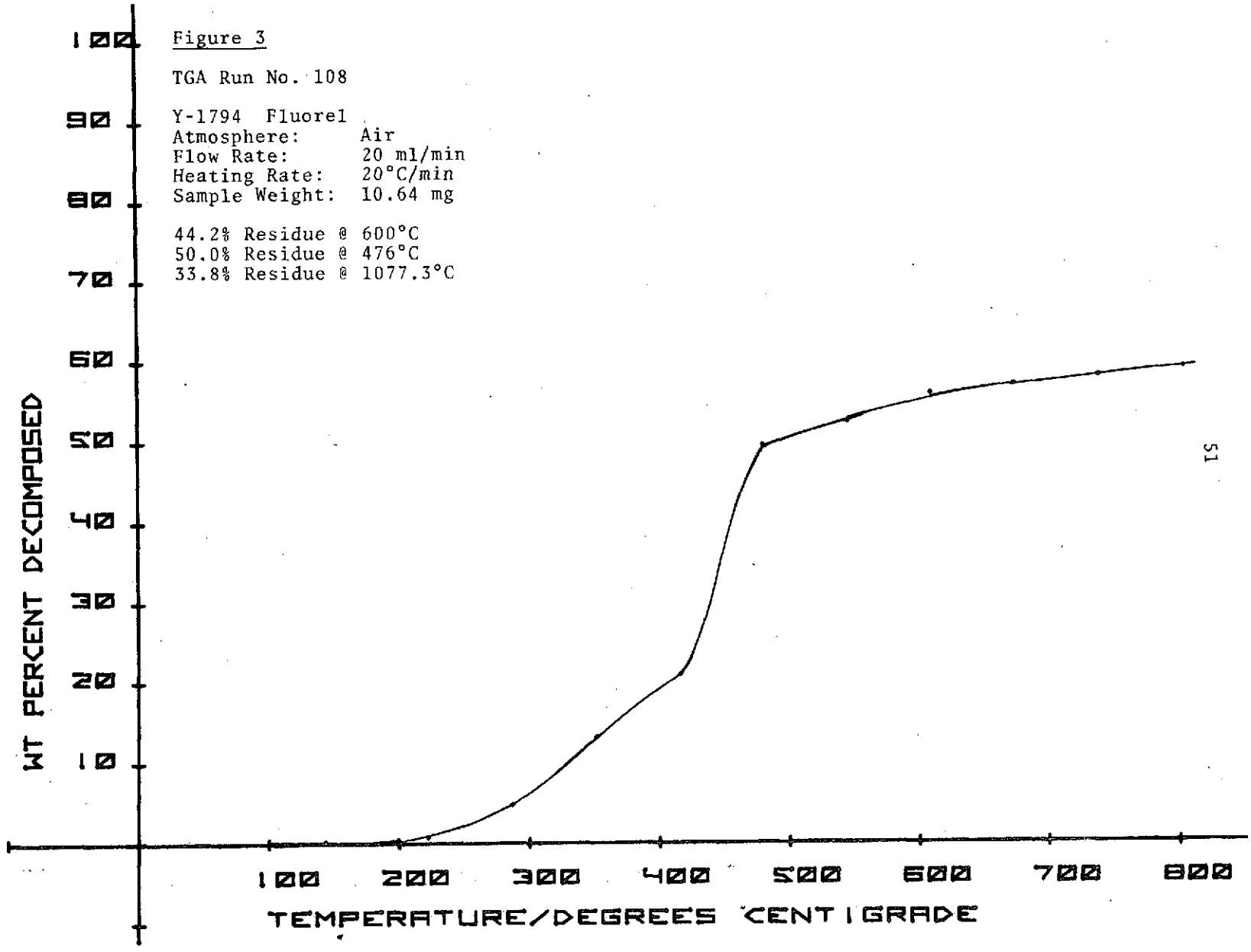


Figure 4

TGA Run No. 100

Y-1795 Mosites, cured
Atmosphere: Air
Flow Rate: 200 ml/min
Heating Rate: 10°C/min
Sample Weight: 16.92 mg

61.2% Residue @ 600°C
50.0% Residue @ > 961°C
59.6% Residue @ 961°C

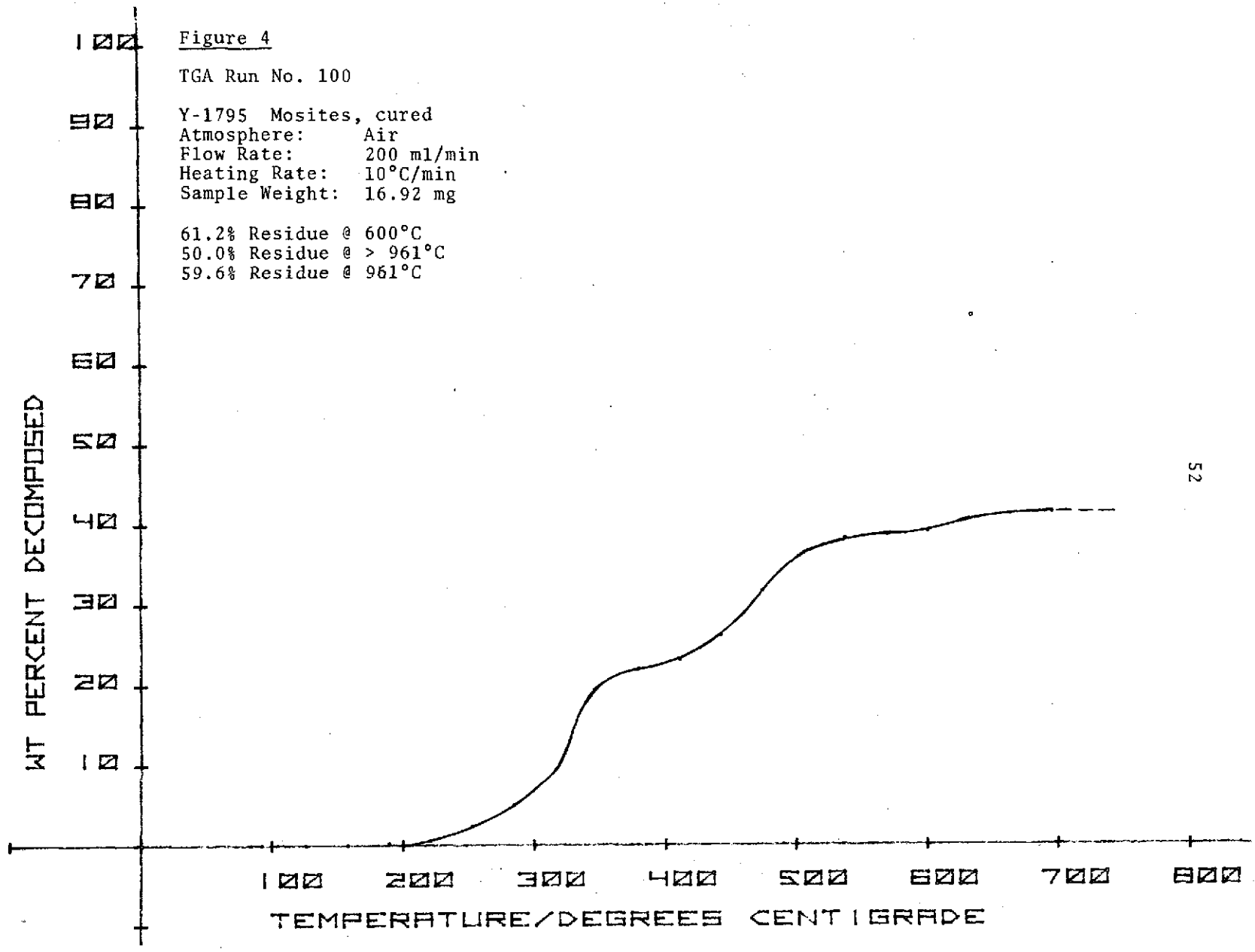


Figure 5

TGA Run No. 109

Y-1795 Mosites, cured
Atmosphere: Air
Flow Rate: 20 ml/min
Heating Rate: 20°C/min
Sample Weight: 10.27 mg

61.1% Residue @ 600°C
50.0% Residue @ > 1086°C
57.4% Residue @ 1086.6°C

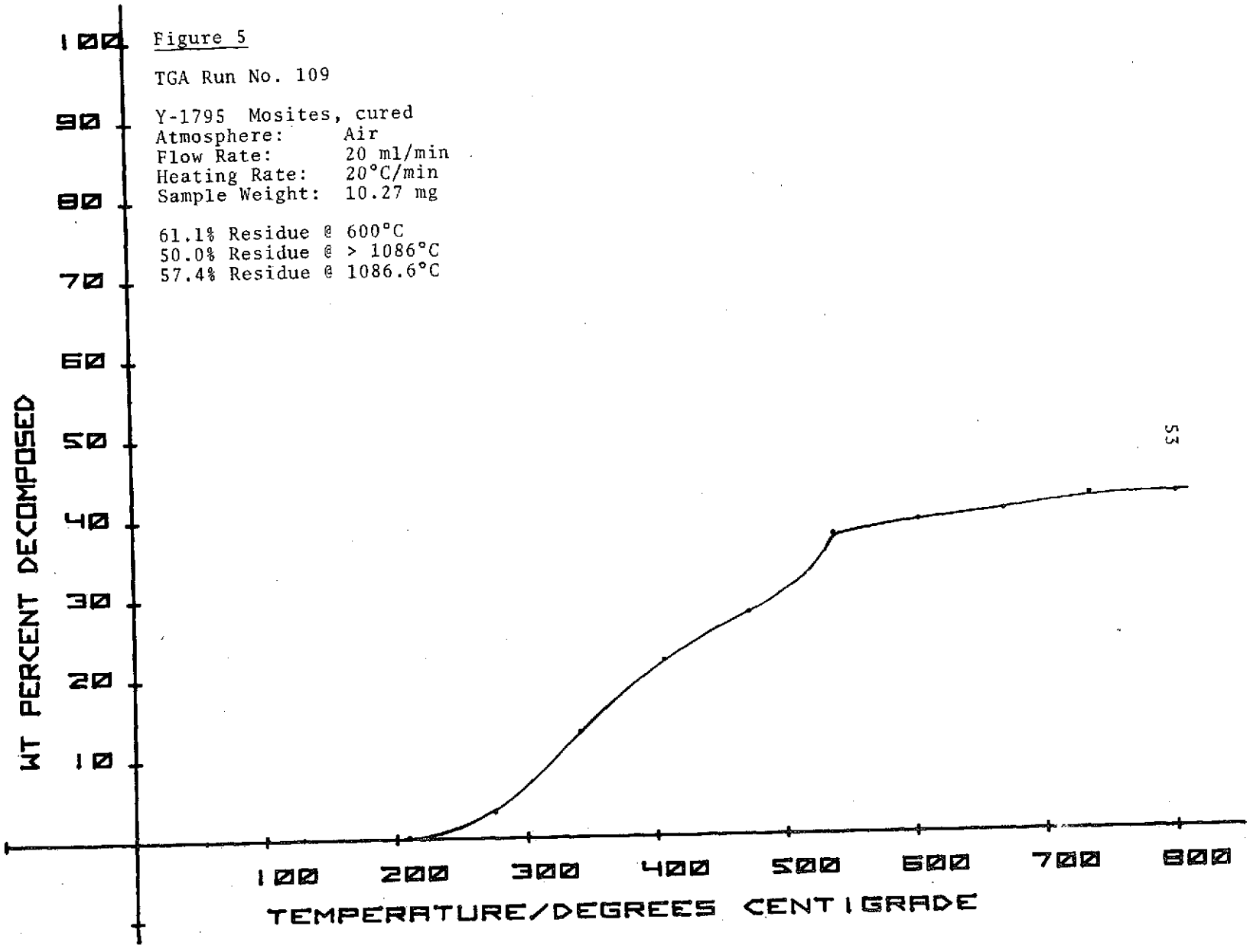


Figure 6

TGA Run No. 97

Y-1796 Nomex
Atmosphere: Air
Flow Rate: 200 ml/min
Heating Rate: 10°C/min
Sample Weight: 9.84 mg

0.4% Residue @ 600°C
50.0% Residue @ 483.6°C
0.4% Residue @ 707°C

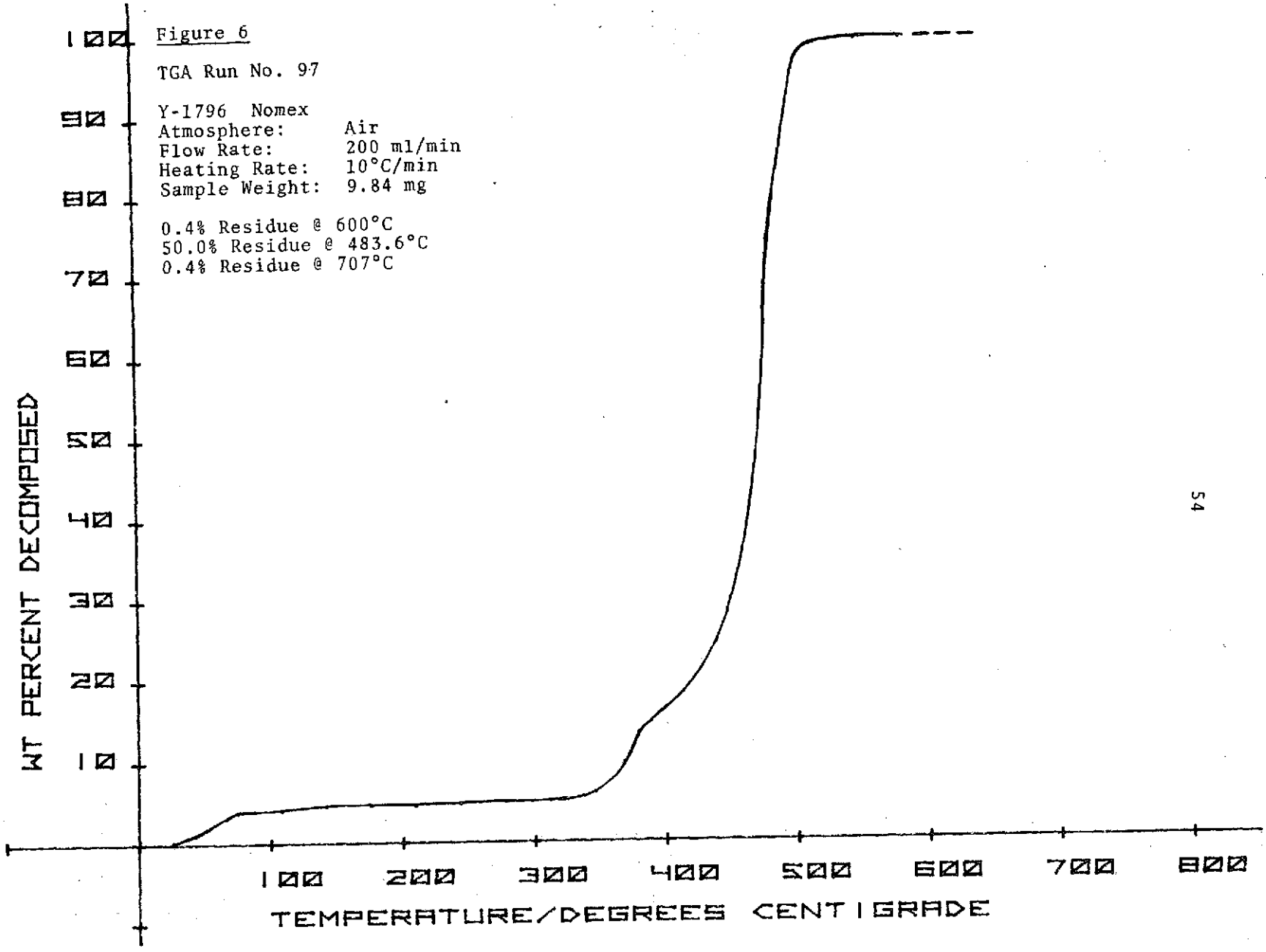


Figure 7

TGA Run No. 128

Y-1796 Nomex
Atmosphere: Nitrogen
Flow Rate: 200 ml/min
Heating Rate: 10°C/min
Sample Weight: 10.0 mg

57.4% Residue @ 600°C
50.0% Residue @ 622°C
3.2% Residue @ 1048°C

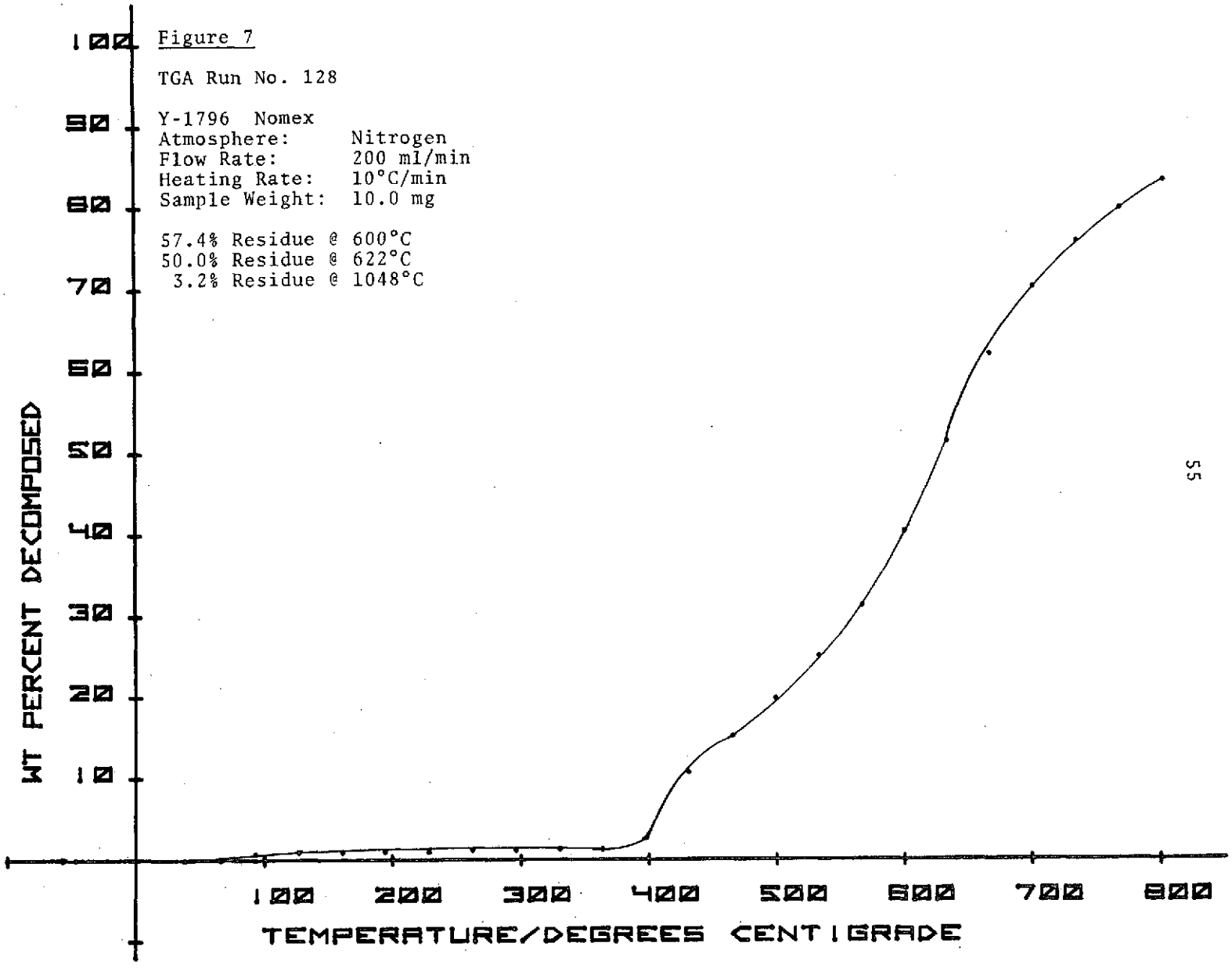


Figure 8

TGA Run No. 110

Y-1796 Nomex
Atmosphere: Air
Flow Rate: 20 ml/min
Heating Rate: 20°C/min
Sample Weight: 9.50 mg

24.4% Residue @ 600°C
50.0% Residue @ 552°C
0.0% Residue @ 992.5°C

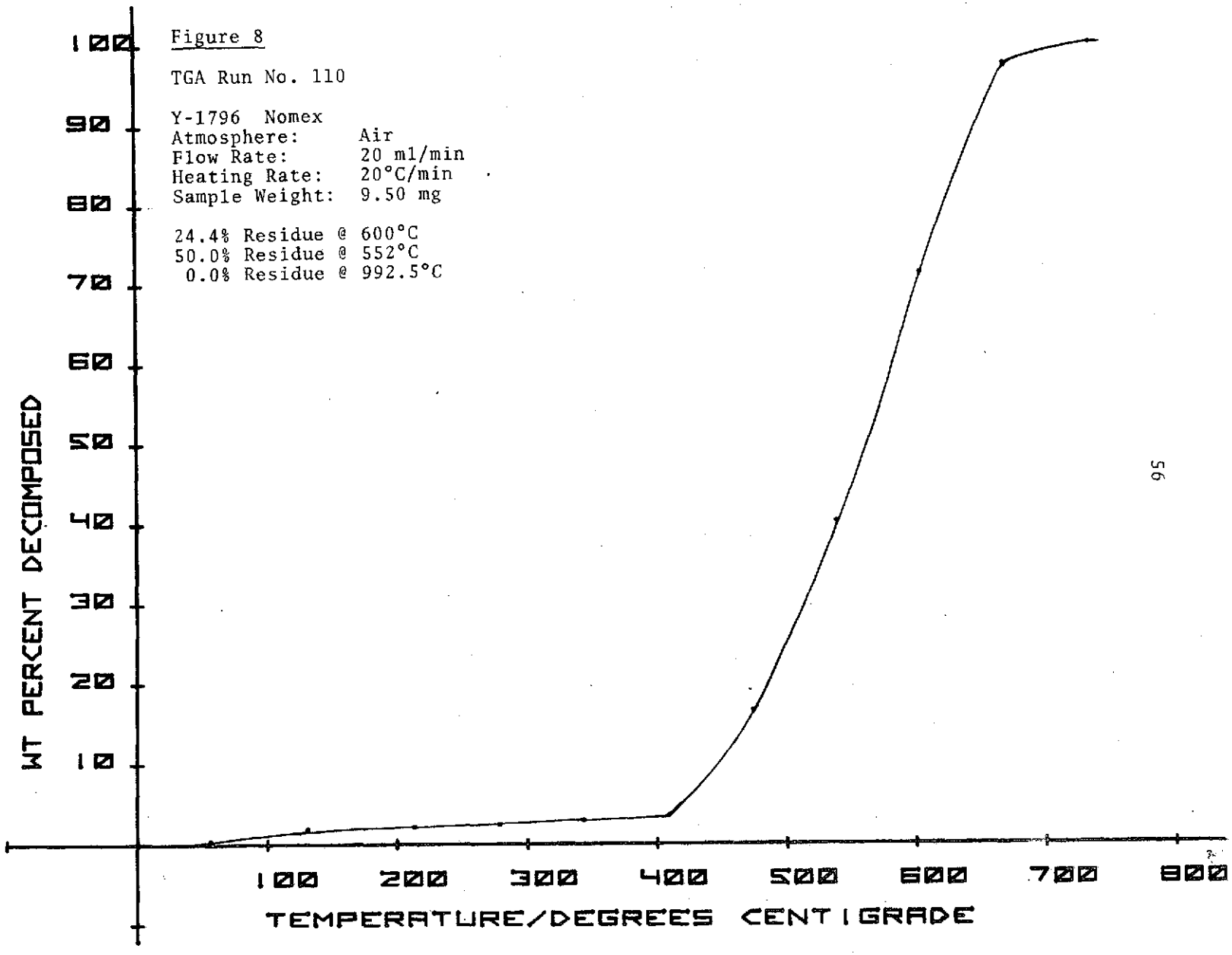


Figure 9

TGA Run No. 101

Y-1797 Durette
Atmosphere: Air
Flow Rate: 200 ml/min
Heating Rate: 10°C/min
Sample Weight: 9.0 mg

0.0% Residue @ 600°C
50.0% Residue @ 488.6°C
0.0% Residue @ 707°C

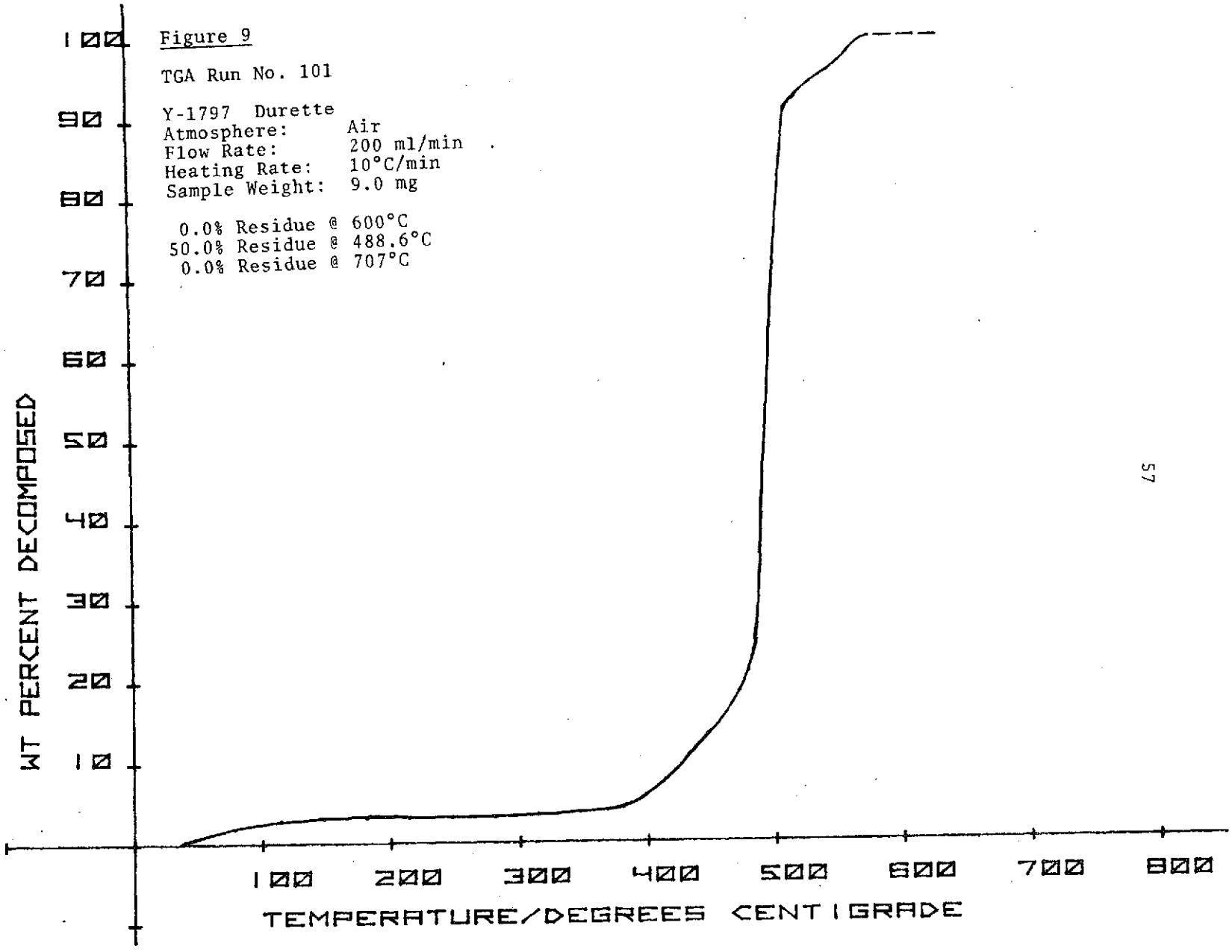


Figure 10

TGA Run No. 129

Y-1797 Durette
Atmosphere: Nitrogen
Flow Rate: 200 ml/min
Heating Rate: 10°C/min
Sample Weight: 10.22 mg

63.4% Residue @ 600°C
50.0% Residue @ 640°C
19.8% Residue @ 1084°C

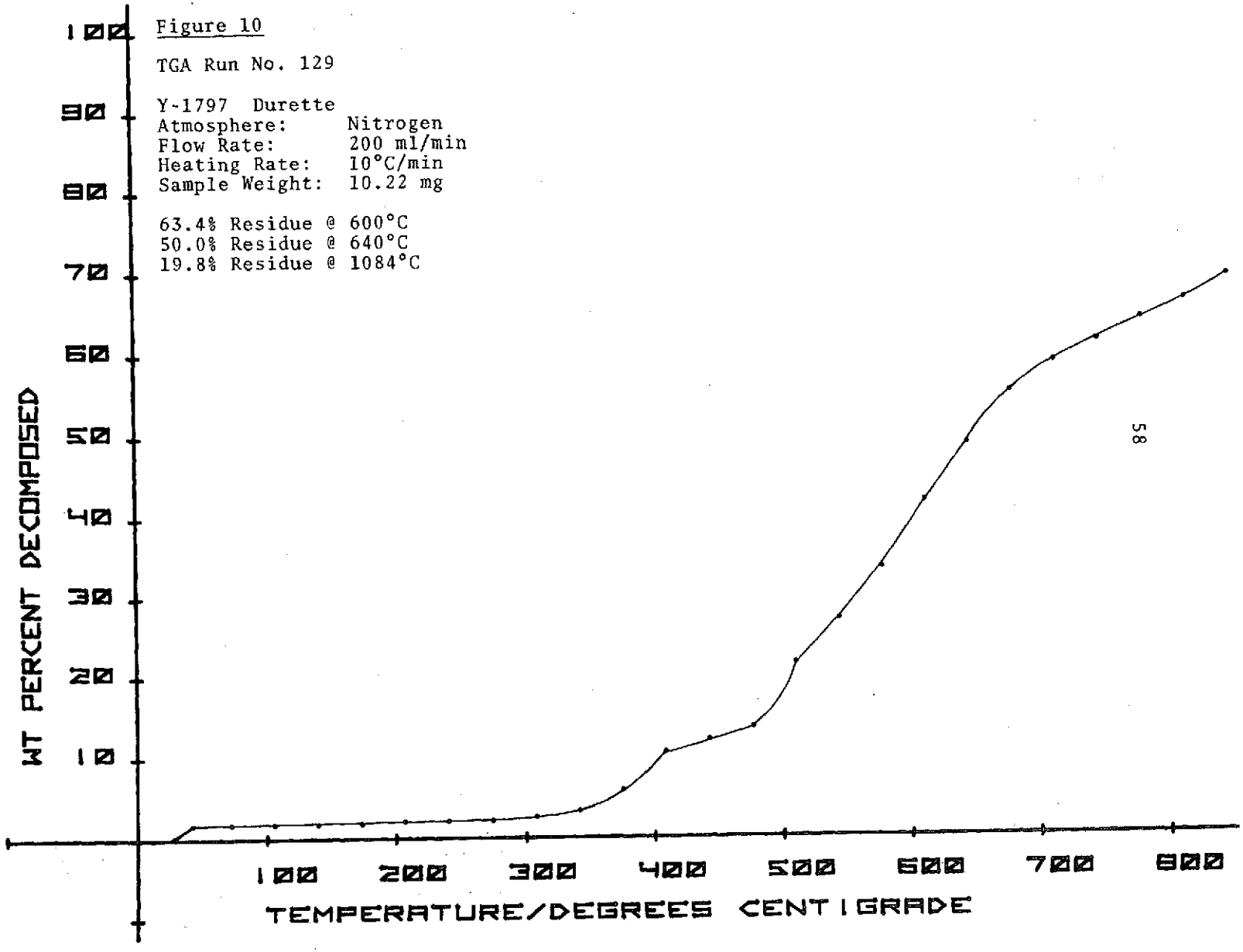


Figure 11

TGA Run No. 111

Y-1797 Durette
Atmosphere: Air
Flow Rate: 20 ml/min
Heating Rate: 20°C/min
Sample Weight: 9.55 mg

0.7% Residue @ 600°C
50.0% Residue @ 528°C
0.0% Residue @ 981°C

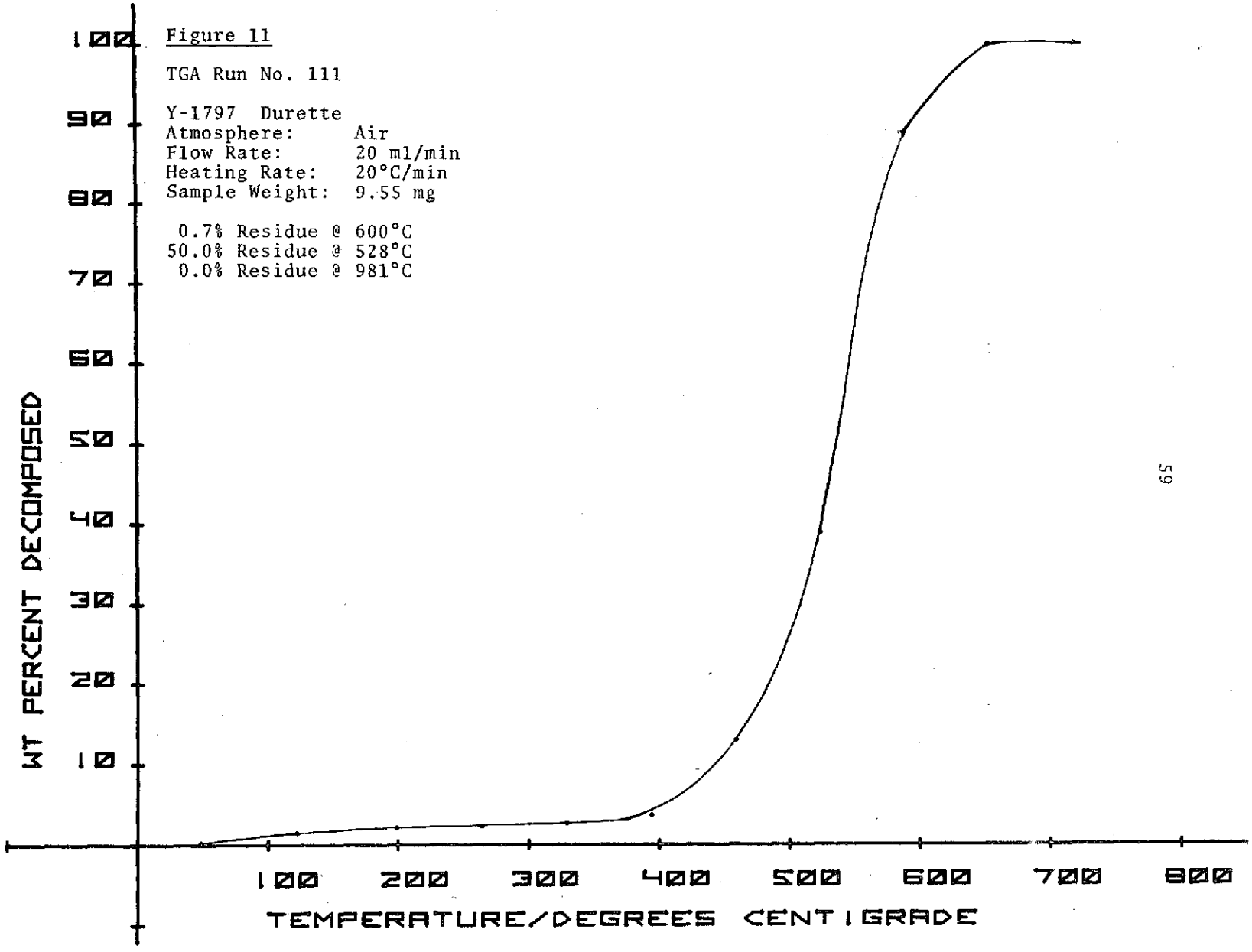


Figure 12

TGA Run No. 107

Y-1856 Kevlar-29
Atmosphere: Air
Flow Rate: 200 ml/min
Heating Rate: 10°C/min
Sample Weight: 9.36 mg

1.7% Residue @ 600°C
50.0% Residue @ 485°C
1.7% Residue @ 1002°C

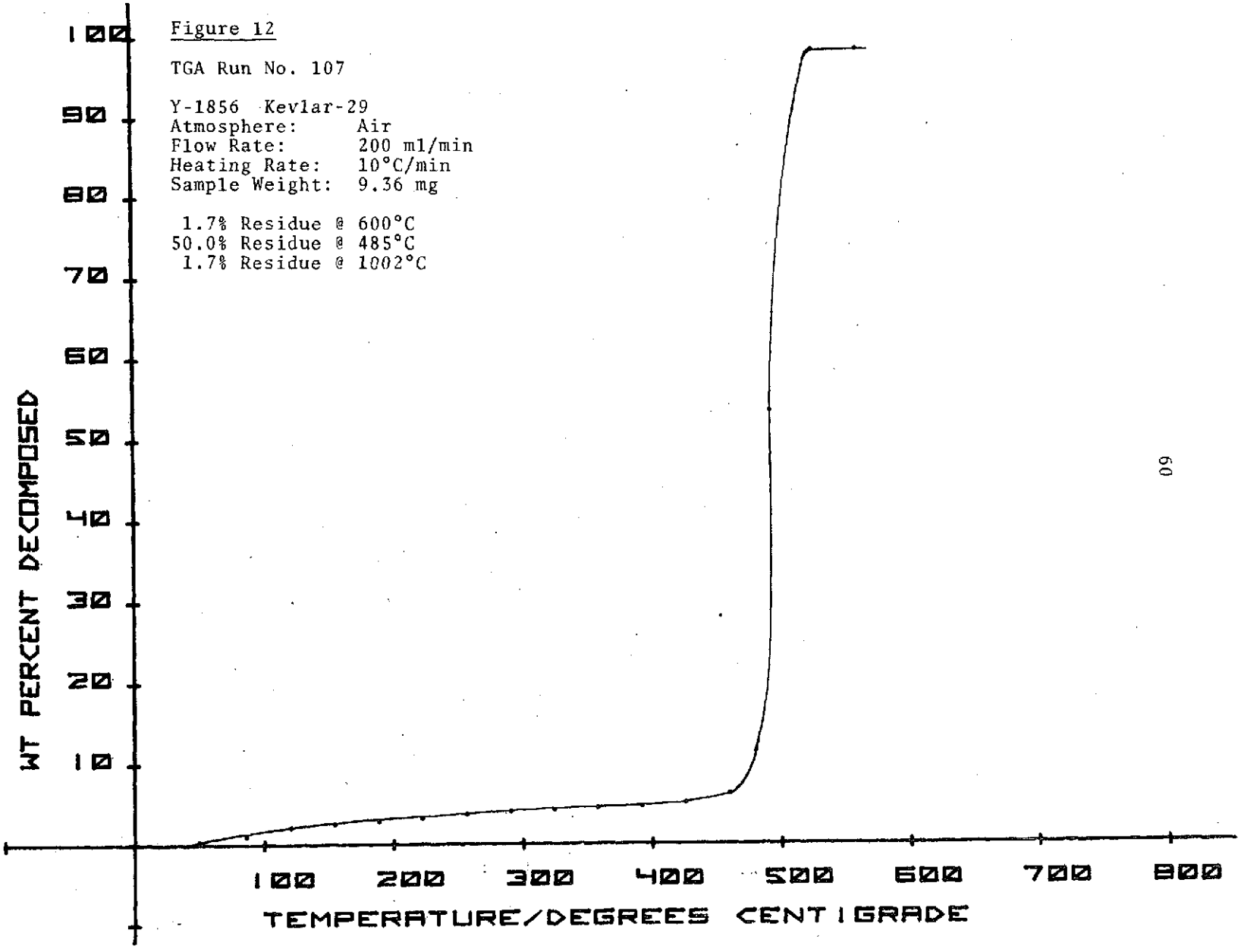


Figure 13

TGA Run No. 150

Y-1856 Kevlar-29
Atmosphere: Nitrogen
Flow Rate: 200 ml/min
Heating Rate: 10°C/min
Sample Weight: 8.50 mg

63.5% Residue @ 600°C
50.0% Residue @ 644.6°C
3.5% Residue @ 933.7°C

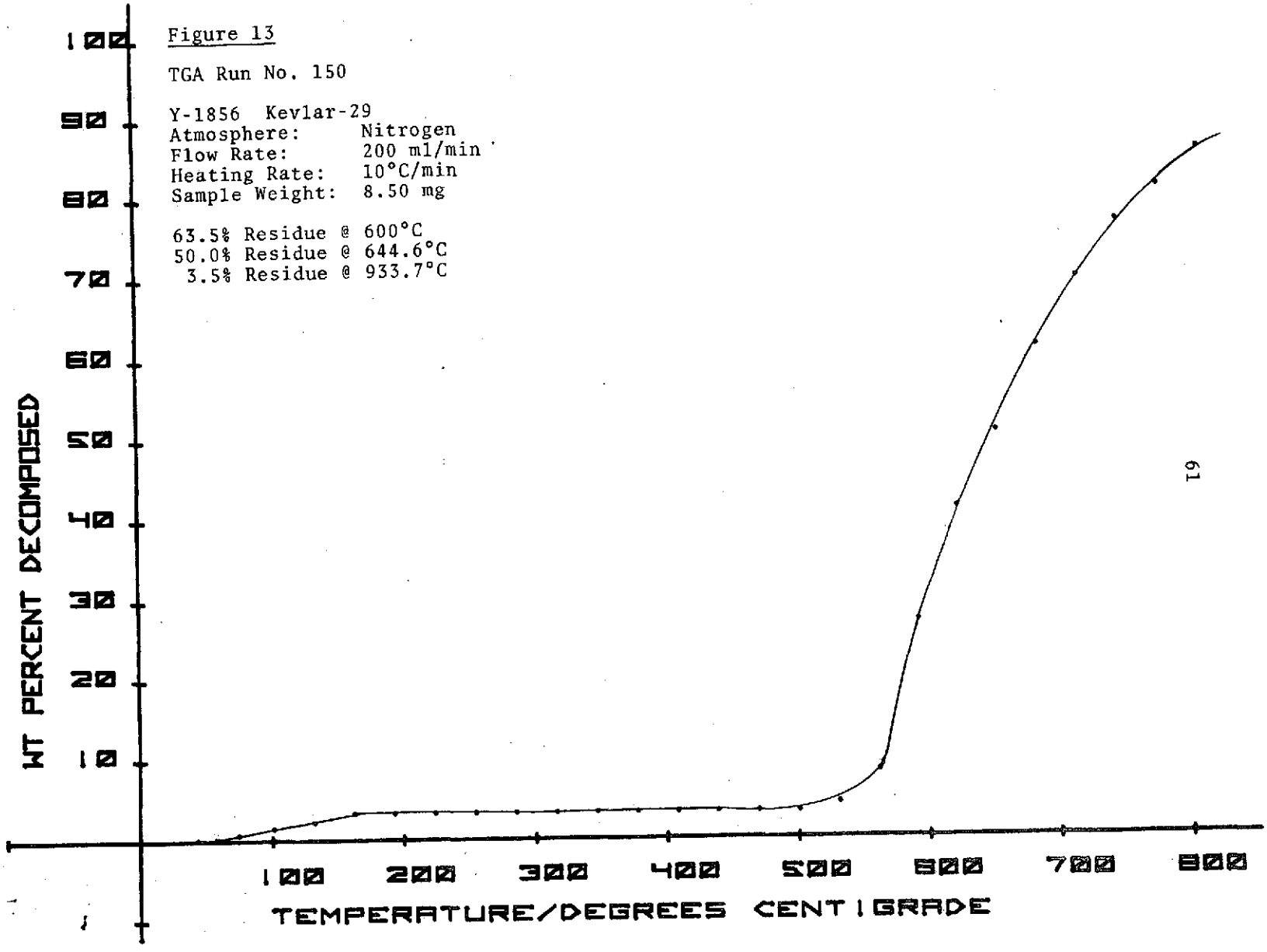


Figure 14

TGA Run No. 106

Y-1856 Kevlar-29
Atmosphere: Air
Flow Rate: 20 ml/min
Heating Rate: 20°C/min
Sample Weight: 10.62 mg

11.9% Residue @ 600°C
50.0% Residue @ 529°C
3.2% Residue @ 1001°C

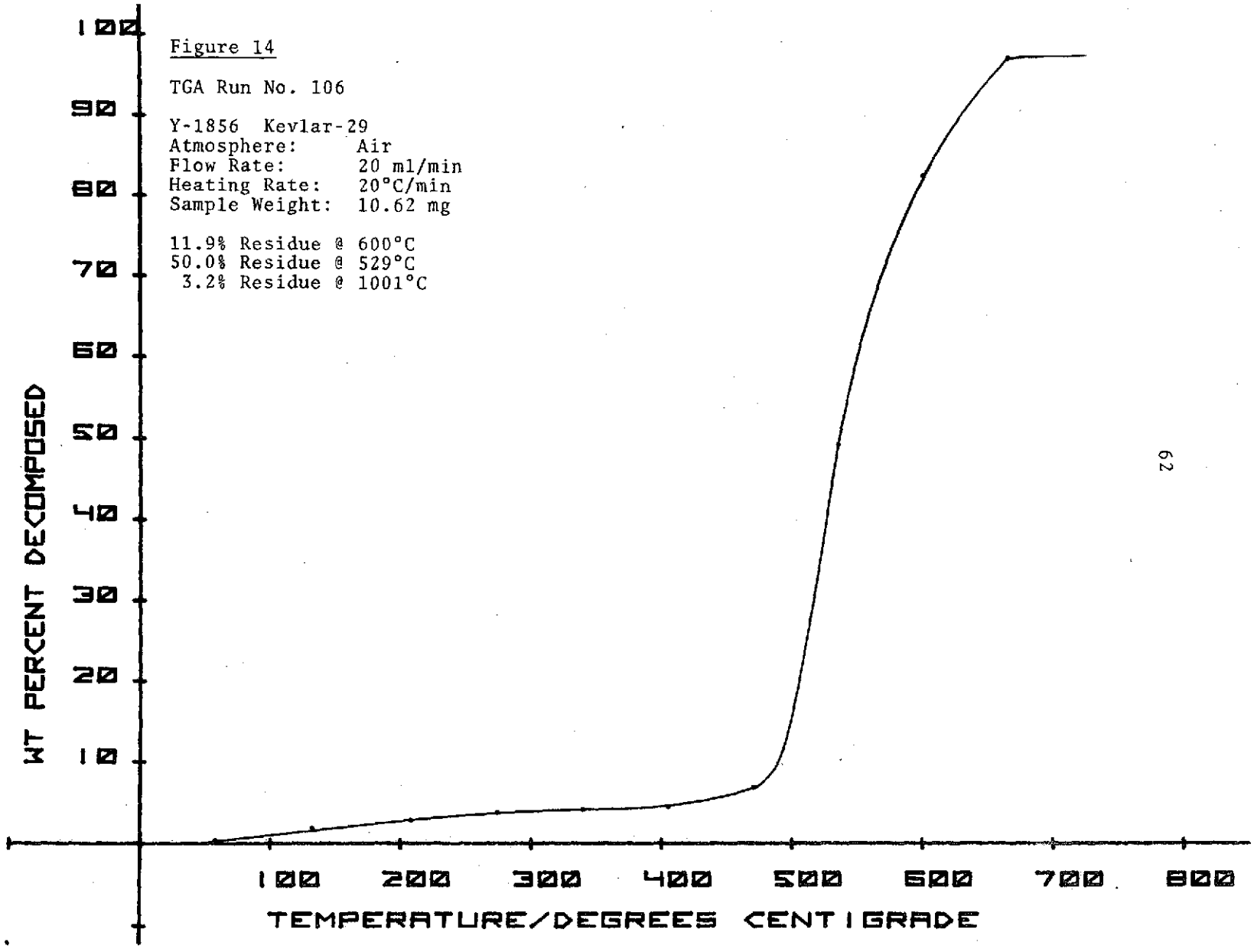


Figure 15

TGA Run No. 130 & 132

Y-2000 Modified Nomex
Atmosphere: Air
Flow Rate: 200 ml/min
Heating Rate: 10°C/min
Sample Weight: 10.0 mg*

8.5% Residue @ 600°C*
50.0% Residue @ 490.5°C*
0.0% Residue @ 852.6°C*

*Mean of duplicate determinations

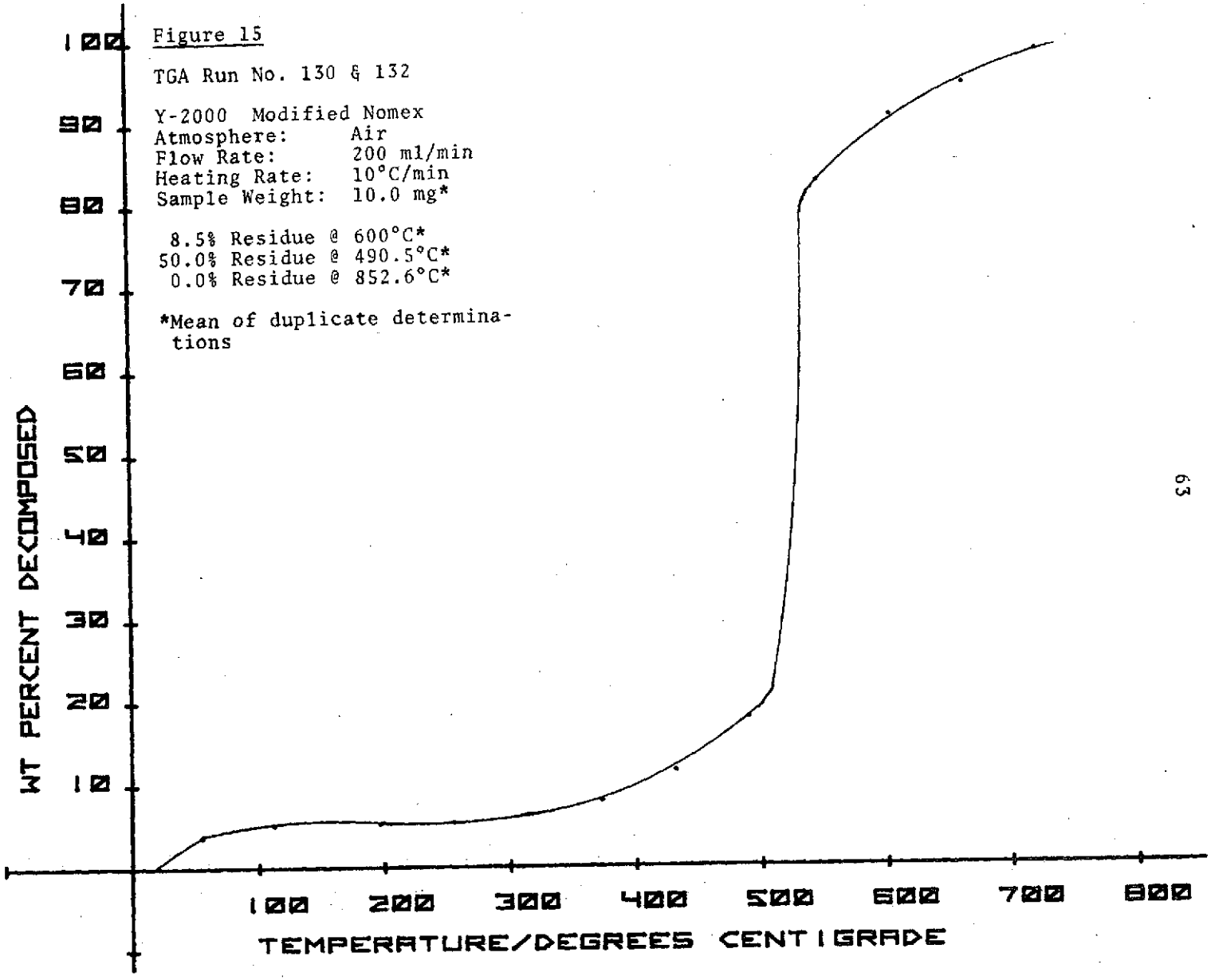


Figure 16

TGA Run No. 133 & 134

Y-2000 Modified Nomex
Atmosphere: Nitrogen
Flow Rate: 200 ml/min
Heating Rate: 10°C/min
Sample Weight: 9.65 mg*

66.0% Residue @ 600°C*
50.0% Residue @ 696.8°C*
0.0% Residue @ 996°C*

*Mean of duplicate determinations

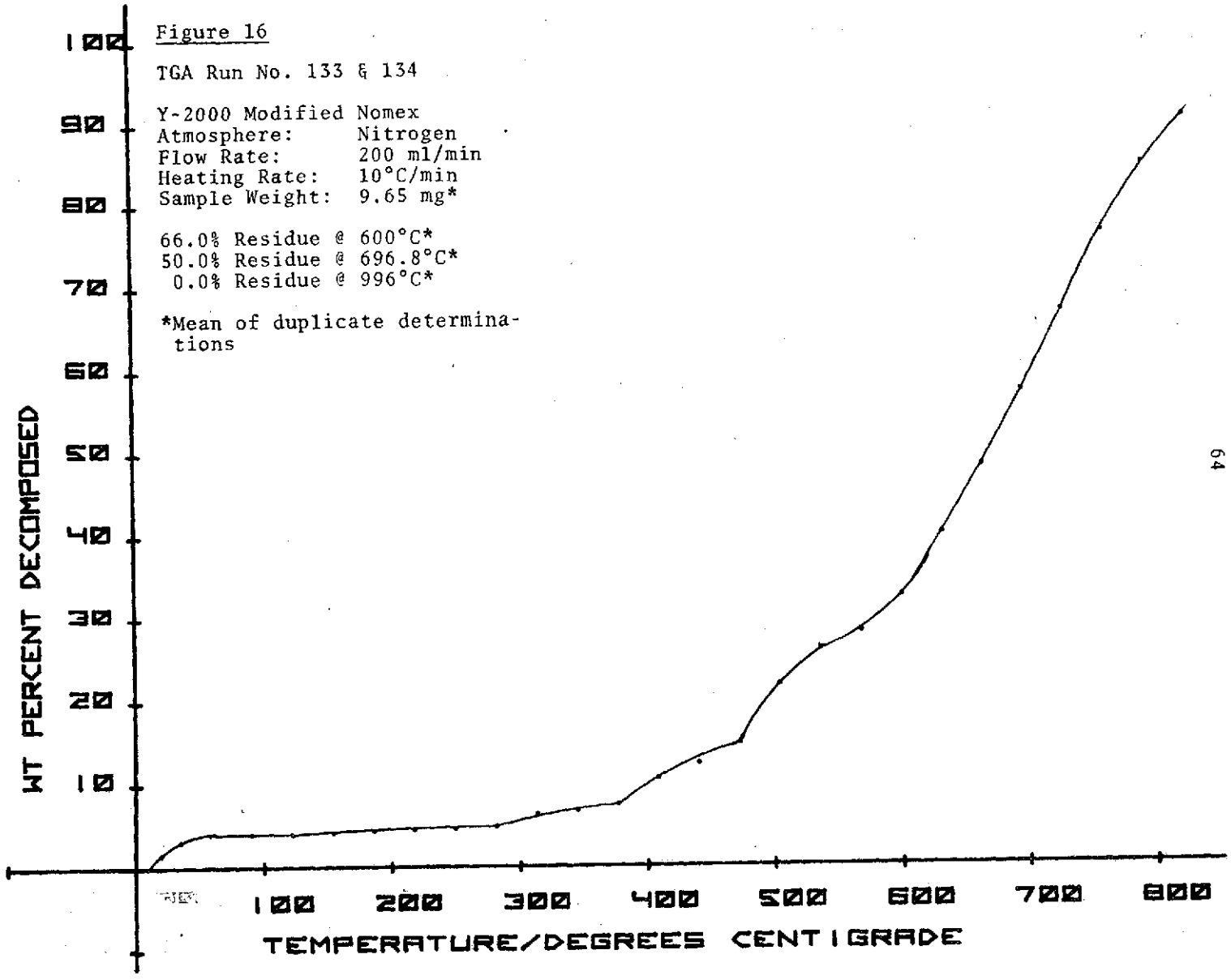


Figure 17

TGA Run No. 131 & 135

Y-2000 Modified Nomex
Atmosphere: Air
Flow Rate: 20 ml/min
Heating Rate: 20°C/min
Sample Weight: 9.94 mg*

11.7% Residue @ 600°C*
50.0% Residue @ 524.5°C*
0.0% Residue @ 996°C*

*Mean of duplicate determinations

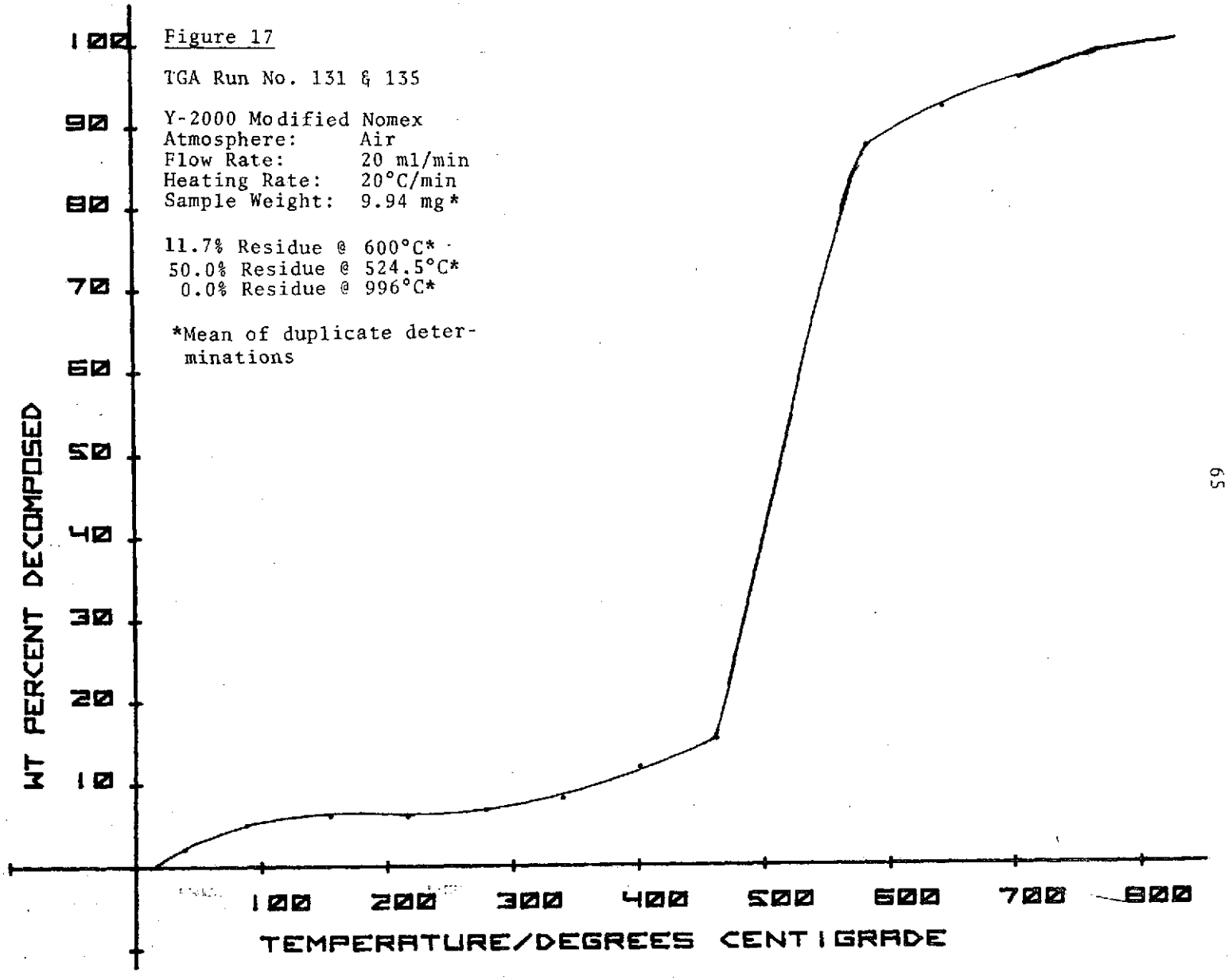


Figure 18

TGA Run No. 136 & 137

Y-2000 Modified Nomex
Atmosphere: Nitrogen
Flow Rate: 20 ml/min
Heating Rate: 20°C/min
Sample Weight: 10.15 mg*

57.2% Residue @ 600°C*
50.0% Residue @ 633.8°C*
0.0% Residue @ 980°C*

*Mean of duplicate determinations

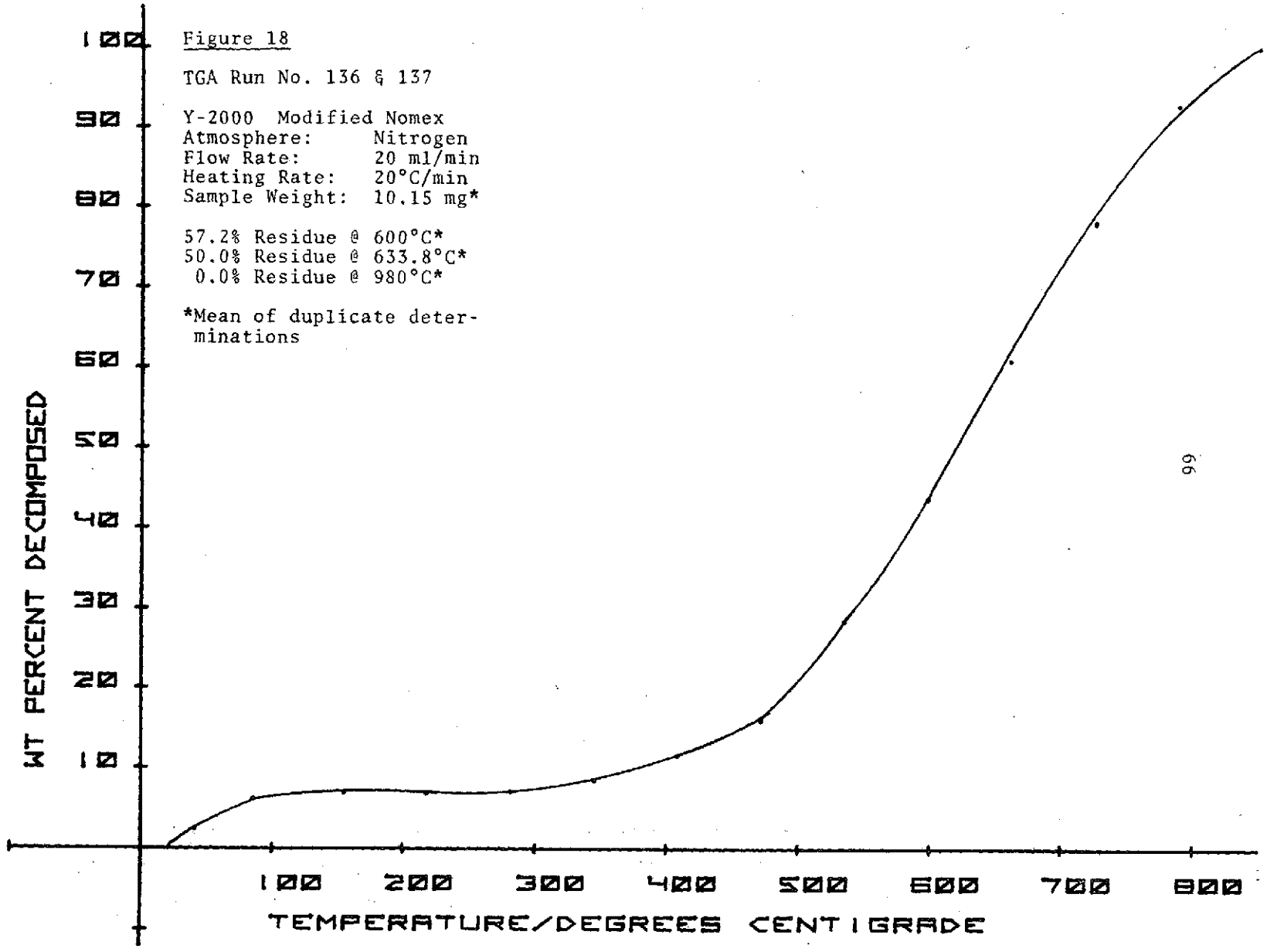


Figure 19

TGA Run No. 144 & 145

Y-2189 Viton Foam
Atmosphere: Air
Flow Rate: 200 ml/min
Heating Rate: 10°C/min
Sample Weight: 10.06 mg*

12.5% Residue @ 600°C*
50.0% Residue @ 447°C*
12.5% Residue @ 939°C*

*Mean of duplicate determinations

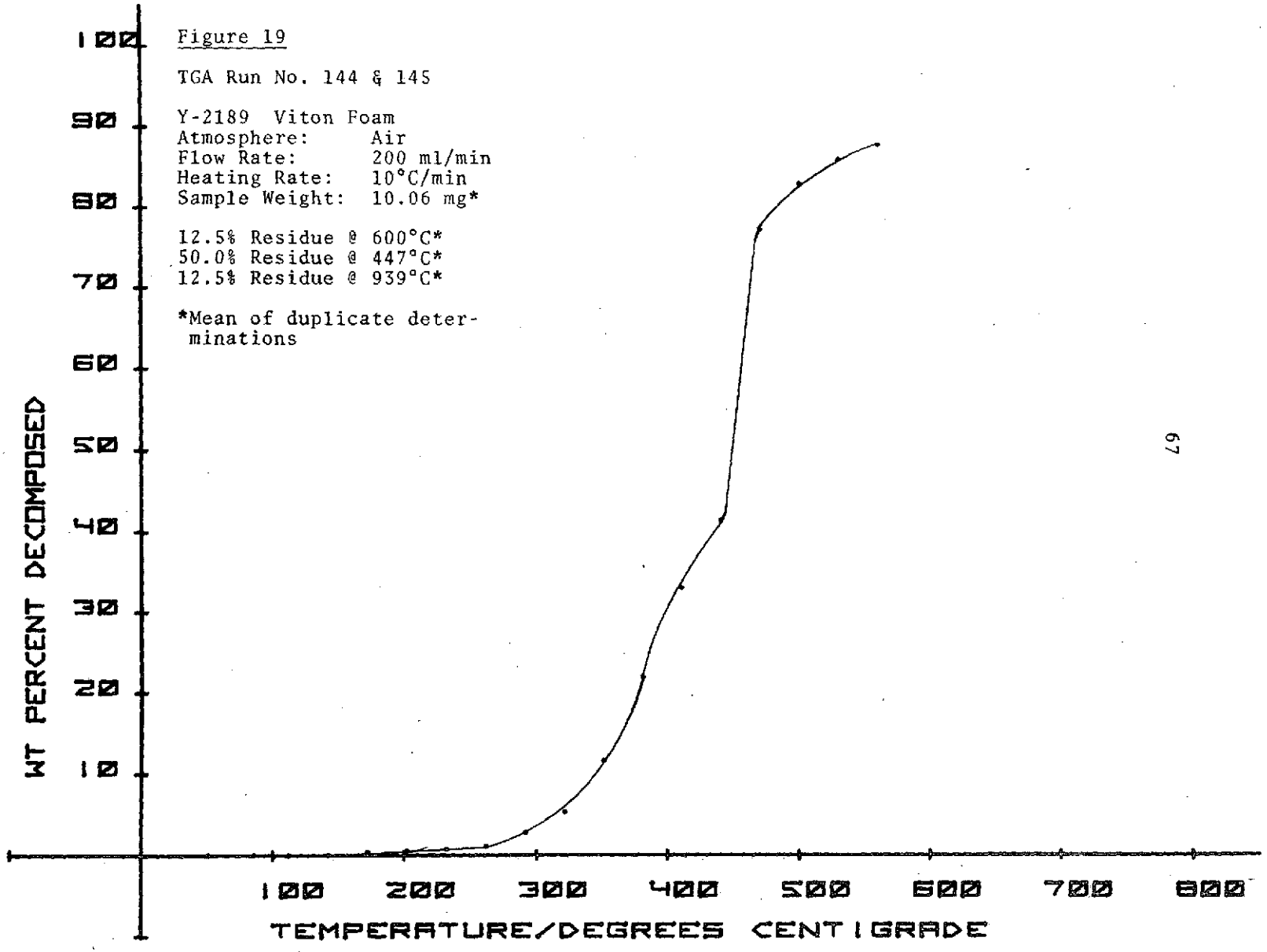


Figure 20

TGA Run No. 146 & 147

Y-2190 Urethane Foam
Atmosphere: Air
Flow Rate: 200 ml/min
Heating Rate: 10°C/min
Sample Weight: 7.6 mg*

2.1% Residue @ 600°C*
50.0% Residue @ 302°C*
0.0% Residue @ 939°C*

*Mean of duplicate determinations

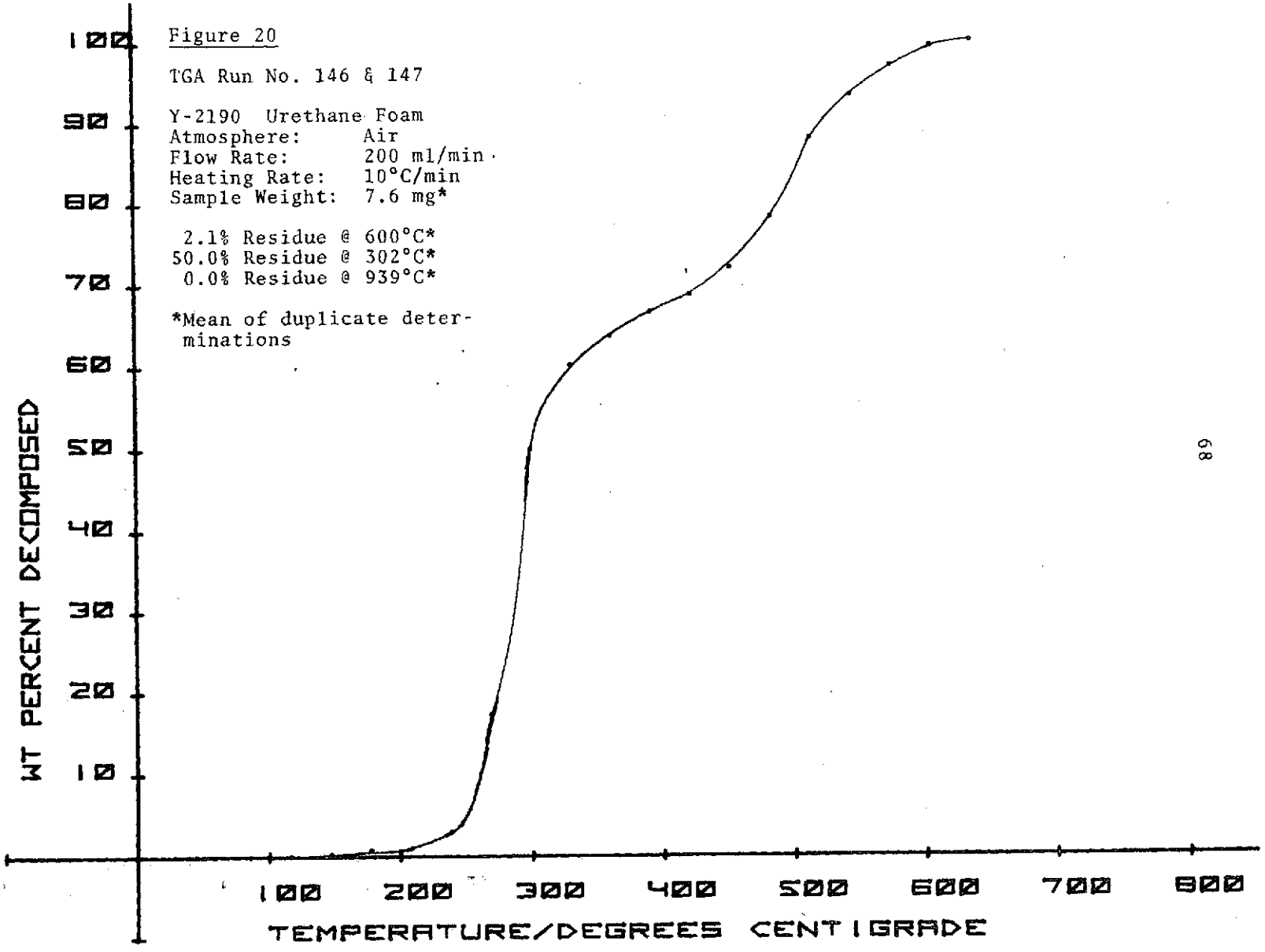


Figure 21:
Carboxyhemoglobin Disappearance from Blood of Living Rats

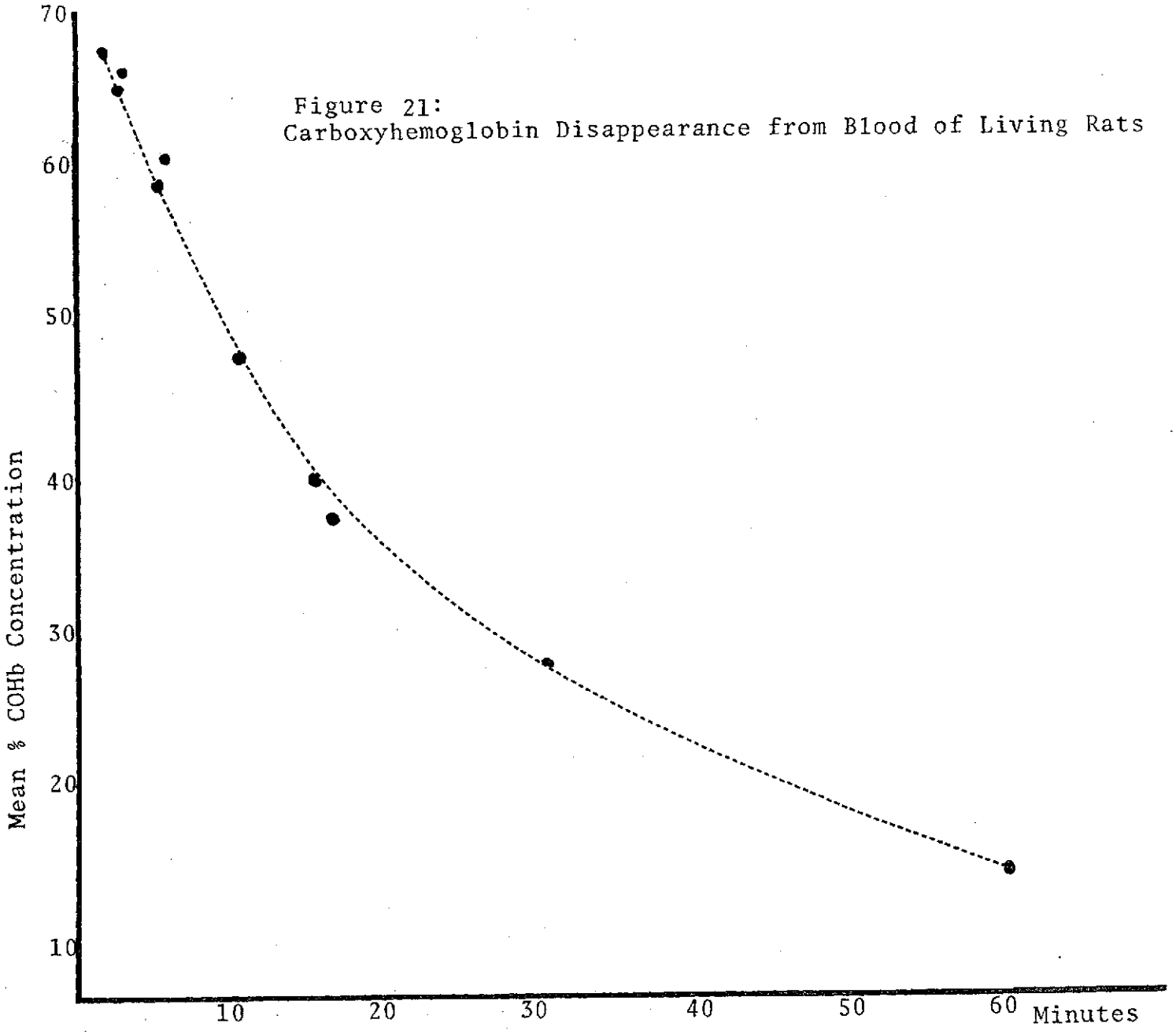
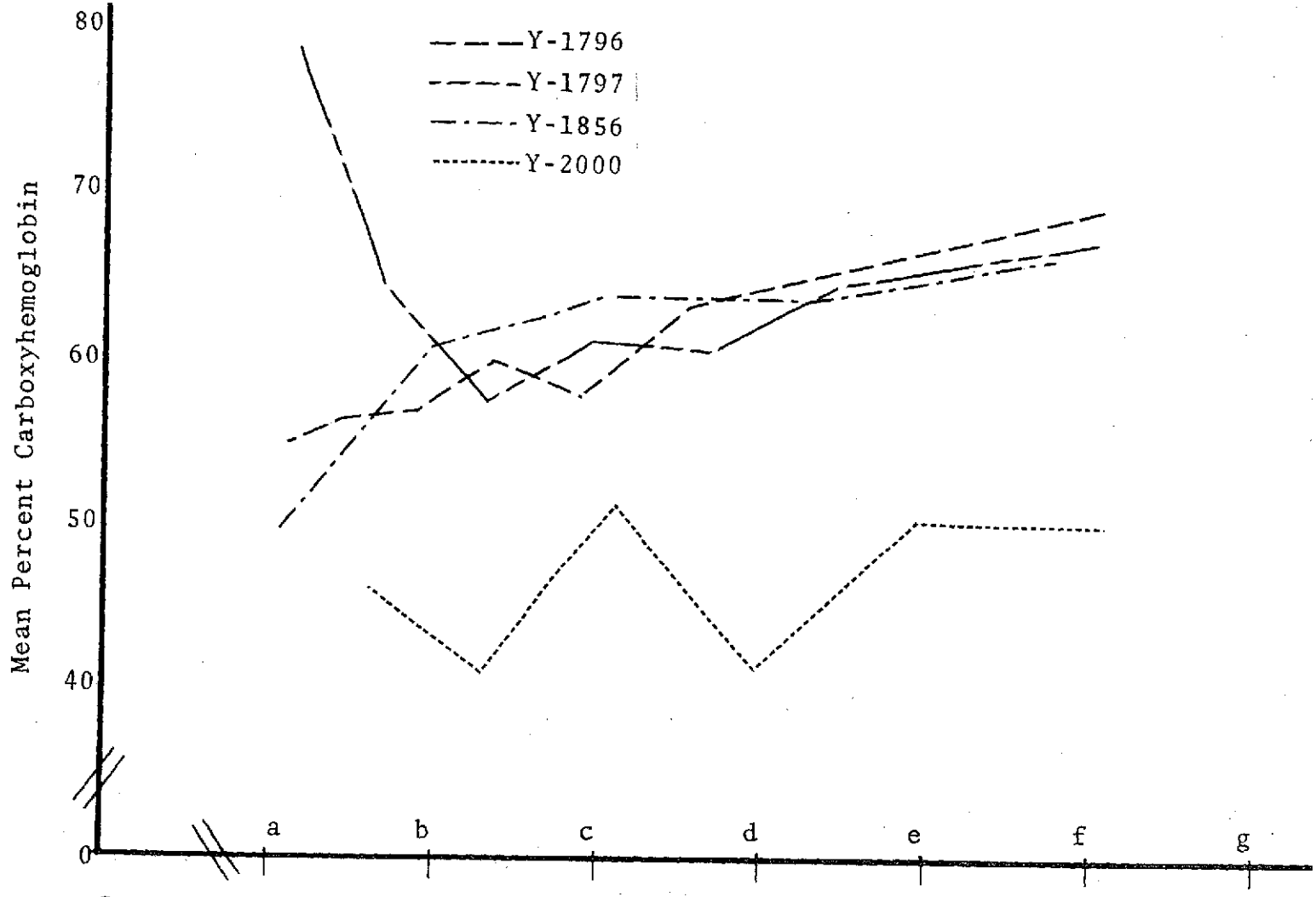


Figure 22: RELATIONSHIP BETWEEN INITIAL SAMPLE WEIGHT AND MEAN PERCENT CARBOXYHEMOGLOBIN OF RATS DYING IN CHAMBER, NASA PROCEDURE



See supplement to Figure for Key, Sample Weight in Grams

FIGURE 22

SAMPLE WEIGHT IN GRAMS

	a	b	c	d	e	f	g
Y-1796	5.0	7.0	9.0	11.0	13.0	15.0	17.0
Y-1797	1.6	1.8	2.0	2.2	2.4	2.6	2.8
Y-1856	2.6	3.0	3.4	3.8	4.2	4.6	
Y-2000	1.0	1.25	1.5	1.75	2.0	2.25	2.5

Figure 23: RELATIONSHIP BETWEEN PERCENT MORTALITIES AND MEAN PERCENT CARBOXYHEMOGLOBIN OF RATS DYING IN CHAMBER, NASA PROCEDURE