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Gravimetric Measurements of Materials Outgassing Applied to Graphite-Epoxy Laminates

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GRAVIMETRIC MEASUREMENTS OF MATERIALS OUTGASSING APPLIED TO GRAPHITE-EPOXY LAMINATES

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PREFACE

The outgassing rates of two Graphite-Epoxy Laminates, American Cyanamide 985B-626 and HST-7B-112, were obtained using a gravimetric method. The rates as a function of time and temperature were derived from the measurements of their mass losses at temperatures varying from 25°C to 150°C and for a time span of up to 400 hours in a vacuum. The data from those measurements were reduced to obtain the outgassing activation energies, the mass losses per unit mass or area, and the corresponding outgassing rates. The rates are expressed in closed form equations and are directly usable for modeling computations. The procedures to obtain these parameters are shown and may be used for the evaluation of other materials. The analysis can be carried out quite rapidly using a computer's algorithm.

The results of the tests and analyses show the following. The activation energies of the two materials are: 4630 cal/mole for the 985B-626 material and 4791 cal/mole for the HST-7B-112 sample #10 Graphite Epoxy.

The outgassing rates of these materials are in the 10E-5 g/cm^{2}hr range and they decay according to a power of time of 0.60 at 25°C, indicating that the outgassing process is mainly a diffusion at that temperature. At higher temperatures, these materials show a time decay approaching a power of one, which may be the result of several outgassing processes occurring simultaneously, and/or the result of the change in heat of desorption as a function of the amount of material on the surface. A large amount, (30 to 40 percent of those rates) consists of water as shown by the mass spectrometer data and by the measured water regained by the material after outgassing at 125°C for 24 hours. Tests also showed that 1 month after that test, a repeated 125°C, 24-hour test produced

a total mass loss comparable to the water regained after the first test. The condensable material accumulated on the 25°C collector disk after the first ASTM-E595 test was 0.01 percent of the total mass and was not measurable after the second test, which occurred a month later.

The normalized mass losses versus time obtained from these tests have been compared to the discrete results obtained from the ASTM-E595 tests. The comparison provides general indications on the effects of temperature and time in relation to the ASTM test values obtained at 125°C for a 24-hour test duration.

INTRODUCTION

Contamination from materials outgassing, and from other molecular or particulate sources, can degrade the performance of an instrument or spacecraft. An understanding of the contaminant source, its transport, deposition, and effects on a surface and on the environment is of importance for the prediction, the mitigation of the contamination, and the expected degradation of the spacecraft's orbital environment. The degradation of an onboard system and of the surrounding environment due to molecular sources can be avoided or reduced by knowing the outgassing properties of the materials in the system. The effects of temperature and time on outgassing rates as well as the chemical nature of the outgassing must be known. A knowledge of these parameters, together with a proper shielding of the critical surfaces, the proper venting and directional dissipation of the outgassing into the environment, and the delayed exposure of outgassing materials to high temperature can all contribute to the avoidance and reduction of contaminant deposits on surfaces—and of the degradation of the surrounding environment.

In this paper, the outgassing of Graphite-Epoxy laminate bars used extensively in structures for space applications is measured using a gravimetric method. The material outgassing activation energy, and the outgassing rates as a function of time, surface area, and temperature are obtained by measuring the weight loss of the material in vacuum at various constant temperatures and as a function of time. These data provide a description of the outgassing rates versus time of the material at those temperatures as well as the energy of activation needed for its removal from a surface on which it may have deposited (if the deposited material was not affected by external sources of radiation.) The time dependence of the outgassing also provides indications of the mechanism of the outgassing, be it surface desorption, diffusion, permeation or a combination of those effects. These indications on the outgassing processes are helpful in finding ways to limit their gaseous releases. The test data obtained in these measurements have been reduced, assuming that the outgassing is produced by a generalized first-order reaction kinetics modified to account for the several products comprising the outgassing. The outgassing results and the present approach are to be compared at a later date to the results obtained from other methods. A comparison with the results obtained from the ASTM-

E595 outgassing method provides additional understanding of those single, discrete results. The ASTM method (Ref. 1) indicates the percentage of weight loss and the percentage deposited on a collector at 25°C when the material under test has been exposed to 125°C in vacuum for 24 hours. The data from that test are easily obtained and extensively used. The screening of materials for space applications is based on those tests. The criteria for acceptance of a material are that it should lose less than 1 percent of its weight and produce less than 0.1 percent volatile condensable material when tested according to the ASTM test. The results using the present methods are expected to be compared at a later time to those obtained from a thermogravimetric analysis (TGA) which utilizes the temperature scanning of the material and the relative weight loss to derive the kinetic properties; i.e., activation energy, pre-exponent for activation, and order of the outgassing reaction process. The TGA method is quite simple and rapid, offering significant saving of time and effort if it can be reliably used and interpreted.

It is also intended at a later date to perform a comparison using simultaneously the present weight loss method and the method employing a Quartz Crystal Microbalance (QCM). The QCM method measures the accretion of outgassing products on a vibrating crystal held at chosen temperatures. That method has the advantage of providing indications for the identification of the accreted products. The temperature of the Quartz Crystal is varied to release or accrete the deposited material and infer from those temperatures the nature of the condensate.

EXPERIMENTAL PROCEDURE

Two types of graphite-epoxy laminates were tested, at 10E-6 torr pressure, and temperatures of 25, 50, 100, 125 and 150°C to measure their weight losses versus time. The measurements were carried out using an Ainsworth Recording Vacuum Balance. The balance, which has a capacity of 100 g, has a sensitivity of 0.1 mg, and the readability on the Bristol strip chart is also 0.1 mg. The specimen weight loss is automatically recorded on the strip charts which also record the temperature. The temperature of the specimen can be varied in increments of 5°C. The vacuum chamber in which the sample is inserted and heated is 3 inches in diameter and is 20 inches long. The initial operation consists of weighing the sample before being inserted in the balance and adjusting the balance accordingly.

The graphite-epoxy samples, provided by the American Cyanamide Co. were: CYCOM HST-7, Batch 112, which will be referred to here as sample #10; and CYCOM 985, Batch 26, referred to as sample #4.

The HST-7 sample #10 is an epoxy-laminated material for use as a structural composite where high toughness and impact properties are required. It is a combination of two layers. A high-modulus resin is used on the graphite fiber. A separate high-strain resin layer is applied to one side of the graphite prepreg to provide a stiffness to the system. Optimum properties are achieved by curing under a pressure of 100 psi at 177°C for 2 hours. The resin content is given as 45.9%; the aereal weight as 146.1 g/m^2 and is specified to have 1% maximum volatile components. The sample used for this test was 1.9985" wide, 0.1230" thick and had an initial mass of 2.888 grams.

The CYCOM-985 sample #4 is a graphite-epoxy composite for applications where a good balance between performance and processing is required. It has a resin content of 33.9%, an aereal graphite weight of 144 g/m^2, and a volatile component of 0.22%. The weave is specified as a 12-1/2 X 12-1/2 plain which will consist of 14 plies. The dimensions of the test sample were 1.9915" long, 0.5000" wide, 0.0920" thick and the initial mass was 2.0631 grams.

The mass loss of the sample was recorded continuously and typed in a Lotus 1-2-3 file. The losses during the initial 2.5 hours of each test run were sampled every 15 minutes. After the initial 2.5 hours of rapid mass changing, the sampling was done every 30 minutes.

The data obtained from the tests are shown in Figures 1 and 2 for the samples #10 and #4, respectively. The mass losses have been normalized to the mass loss of the sample taken after 24 hours at 125°C. This was done to correlate with the mass losses measured using the ASTM-E595 test method and to generalize the tests. The total mass losses and other pertinent data on the sample have been shown in the figures. Figures 3 and 4 show the log to base 10 of the initial mass loss rates, at $t = 0$ versus $1/T$ where T(K) represent the isothermal outgassing temperatures of the samples. These are Arrhenius plots of the outgassing reaction rates shown in terms of log to base 10 rather than log to base e, which are required for the analysis of the data and the derivation of

the activation energy and of the pre-exponent term of the Arrhenius equation. The slopes of the curves indicate the activation energies required for the outgassing. The intersections of the curves with the Y axis showing the outgassing rates represent the pre-exponential term in the Arrhenius expression for the reaction rates. Additional characterizations of the graphite-epoxy material have been provided. The mass spectrum of sample #4, laminate 985B-626, and the results of the ASTM-E595 for both samples are enclosed in the appendix.

Theory

Rate of Mass Loss and Activation Energy

Assuming that the outgassing may be represented by a first-order reaction kinetics within the range of temperatures under consideration (refs. 2 and 3), the rate of mass loss is $dm/dt = -km$, where m(g) is the mass and $k = A \exp -E/RT$ is the Arrhenius expression relating the reaction constant k to the activation energy E, (cal/mole) required for outgassing, the temperature T(K); the frequency factor A(1/s), and R(cal/mole/K) the gas constant. The constant k can also be written for convenience as $k = 1/\tau$, where $\tau = \tau_0 \exp E/RT$ and the mass loss rate is then

$$dm/dt = - m/\tau \quad . \tag{1}$$

The integration of this for $m = m_0$, when $t = o$, indicates that the mass remaining as a function of time is given by

$$m = m_0 \exp - t/\tau \tag{2}$$

and the mass loss comparable to the data reported in Figures 1 and 2 is

$$m_0 - m = m_0 (1 - \exp - t/\tau) \quad . \tag{3}$$

In Figures 1 and 2, the value of $(m_0 - m)$, as in this last expression, has been normalized by the loss (m_f) produced by the material after 24 hours at 125°C. This normalized loss $(m_0 - m)/m_f$ is shown on the plot, and the units along the Y axis are (g/g). The loss can also be expressed in terms of the area in the sample (g/cm^2) when the loss results are expressed in terms of the sample surface areas.

The rate of mass change from the above is representable in any of the following forms:

$$dm/dt = -km = -m/\tau = m_o/\tau \exp - t/\tau = - mA \exp - E/RT \quad . \qquad (4)$$

Taking the natural log of both sides of the above expression after dividing by m, one gets

$$\ln (1/m\ dm/dt) = -\ln A - E/R(1/T) \quad . \qquad (5)$$

The plot of ln 1/mdm/dt versus 1/T indicates the slope of 1/T which is E/R and the value of the frequency factor A which is given by the intersection of the curve with the Y axis.

Figures 3 and 4 show the plot of the log $(1/m_r(m_o - m/t - t_o)$ versus 1/T taken from the data in Figures 1 and 2. The appropriate change from log to base 10 to ln provides the value of E/R.

The equivalent activation energy can also be estimated by comparing the outgassing rates at the same time for two outgassing temperatures, T_1 and T_2, assuming the difference in temperature is small. In fact, from Eq. 4, the rates are related by the Arrhenius relation:

$$|dm/dt|_{T_1} / \left| \frac{dm}{dt} \right|_{T_2} = \exp E/R\ (1/T_2 - 1/T_1) \quad . \qquad (6)$$

This assumes that E remains constant within the range of temperatures being considered and does not change as a function of the mass loss.

The plots in Figures 1 and 2 do not show the exponential time dependence indicated by Eq. 3, nor do they show the time dependence for the rate of outgassing indicated by Eq. 4. This time dependence is, in general, verified in many cases of material outgassing, because the outgassing consists of the release of different concentrations of materials following various processes and the first-order reaction kinetics is not applicable (Refs. 4 and 5). The actual outgassing rates as obtained from Figures 1 and 2 are shown in Figures 5 and 6. These were determined by plotting on log-log coordinates, the rate of change of masses,

$$(m_{n-1} - m_n)/(t_{n+1} - t_n)\ 1/A$$

versus time. The slope of the plotted curve and the intersection with the Y axis show that the outgassing rates are expressible by the function

$$dm/dt\ (1/A) = B/t^n \qquad (7)$$

The constant B is given by the intersection with the Y axis and it includes the effect of the temperature on the outgassing, the activation energy, and the units used for mass and time. Its value includes the Arrhenius factor A exp - E/RT which can be obtained in a process of comparison as shown by Eq. 5. The exponent n indicates how the rate changes with time. By a process of association with the rate change with time for known physical processes, one can obtain an indication of the prevailing process involved in that particular outgassing; i.e., diffusion, surface physical or chemical desorption, permeation, sublimation or a combination of those processes (Ref. 5). In general, the experimental data on outgassing indicates that for fixed-material temperatures and for a considerable length of time, the outgassing rates are expressible by

$$dm/dt = B/t^n \approx (A \exp - E/RT)t^{-n} \qquad (8)$$

where A, T, and E are parameters included in the value of B.

Data Reduction

The normalized mass losses shown in Figures 1 and 2 modified by the appropriate parameters, provide the mass-loss-per-unit mass $(m_0 - m/m_f)m_f/m_0$ and the mass-loss-per-unit area versus time and as a function of the outgassing temperature. The masses of the samples and the surface areas were measured at room temperature with 50% relative humidity. The log of the normalized initial mass loss rates, $\log(m_0 - m/m_f/t - t_0)$ versus the inverse of the absolute temperature of the outgassing (1/T), is shown in Figures 3 and 4. These are the plots of the log to base 10 of Eq. 4. However, the activation energies E and the frequency factor A must be obtained by plotting the data in terms of the natural log. The value E/R (intercept on X) and the A (intercept on Y) shown on the figures, have been changed to the appropriate ln. The results for the activation energy and the frequency factor are 4630.27 (cal/mole) and 37.94 (hr^{-1}), respectively, for sample #4 and 4791.7 cal/

mole and 46.049 (hr^{-1}) for sample #10. Regression analyses on these results show errors of 3.5% for sample #4 and 6.9% for sample #10, for the activation energies; and 2.9 and 4.0% for the frequency factors. The activation energies of sample #4 obtained using Eq. 6, and the rates of mass loss shown in Figure 1 when measured in the interval of time from 20 hours to 30 hours, and for temperature (K) ratios of 323/298, 373/298, 398/298, 398/373, varied from 3404 cal/mole for the ratio of 273/298 to 4994 cal/mole for the 398/373 ratio. The average activation energy was 4070 cal/mole. This average value is 88% of E = 4630 obtained from the initial rates of mass loss.

The outgassing rates per-unit-area 1/A dm/dt, obtained from Figures 1 and 2 plotted as a function of time and for different isothermal outgassing rates are shown in Figures 5 and 6. The data are plotted in log-log coordinates to provide, as indicated by Eq. 8, the constant B and the exponent n for the time. The errors for the n values were less than 5% and for the constant B, about 20%. The equivalent errors for sample #10 were less than 3.4% for the n and about 16% for B. Table 1 shows the values obtained from those plots and the relative equations for outgassing at the various temperatures.

The outgassing rates of the graphite-epoxy material, as found here, are comparable to the results for similar materials indicated in the literature (Ref. 6). The material exhibits considerable outgassing. The test data in the appendix provide some explanation for this behavior. The mass spectrometer fragmentation of the material at 290°C shows the spectrum with major picks at 243, 93, 106, 108, 77 and 65 m/e. These are representative of aromatic epoxy compounds that are amino cured. The mass spectrometer detected no (<1%) outgassing at temperatures less than 200°C. The ASTM tests indicated that the water regained by the two samples at 50% R.H., and 25°C was 0.31% for sample #4 and 0.26% for sample #10. A comparison of these values to those of the total mass losses indicates that a considerable amount of the outgassing, on the order of 30–40%, is water. Also, the condensable amount of the total loss at 25°C is only 0.01%.

RESULTS AND CONCLUSIONS

The outgassing rates of two graphite-epoxy laminates, American Cyanamide 985B-626 and HST-7B-

112, were obtained using a gravimetric method. The rates as a function of time and temperature were derived from the measurements of their mass losses at temperatures varying from 25°C to 150°C and for a span time of up to 400 hours in vacuum. The data from those measurements were reduced to obtain the outgassing activation energies, the mass losses per unit mass or area, and the corresponding outgassing rates. The rates are expressed in closed-form equations and are directly usable for modeling computations. The procedures to obtain these parameters have been shown and may be used for the evaluation of other materials. The analysis can be carried out quite rapidly using a computer's algorithm.

The results of the tests and analyses show the following:

- The activation energies of the two materials are: 4630 cal/mole for the 985B-626 material and 4791 cal/mole for the HST-7B-112 sample #10 graphite-epoxy. They show the limited amount of energy needed to affect the outgassing.

- The outgassing rates of these materials are in the 10E-5 g/cm^2 hr range and decay according to a power of time of 0.60 at 25°C. This rate decay indicates that the outgassing process is mainly a diffusion at that temperature. At higher temperatures, these materials show a time decay approaching a power of one. This behavior, theoretically justifiable, occurs when the outgassing is the result of several processes occurring simultaneously, including the change in heat of desorption as a function of the amount of material on the surface.

- The analysis shows that the outgassing does not follow a first-order reaction kinetics but rather, follows a higher order.

- The outgassing rates are relatively high. However, 30-40% of those rates consist of water as shown by the mass spectrometer data and the measured water regained by the material after outgassing at 125°C for 24 hours. Tests have also shown that 1 month after that test, a repeated 125°C, 24-hour test produces a total mass loss comparable to the water regained after the first test.

- The condensable material accumulated on the 25°C collector disk after the first ASTM test was 0.01% of the total mass and not measurable after the second test, a month after.

- The data obtained from these tests can be used for several purposes:

The normalized mass losses versus time can be compared to the discrete results obtained from the ASTM-E595 test which are used extensively for acceptability of materials for space application. The comparison can provide general indications on the effects of temperature and time in relation to the values obtained at a 125°C, 24-hour test.

- The activation energy data allow one to estimate the effect of temperature on outgassing and the effect that the temperature will have on preventing and removing contaminant deposits from a surface, provided that radiation and accumulation of other materials have not changed the nature of the deposit.

- The closed-form expressions for the outgassing rates permit one to estimate, in conjunction with other data, the internal pressure in a system and the density conditions produced in the surrounding environment as a function of time. Certain acceptable conditions of pressure and density can be established in this manner.

- Finally, the gravimetric method for obtaining the needed outgassing data has been shown. The method is relatively simple and requires less manipulation of the data than other methods.

Acknowledgment

The author acknowledges the help of Mr. R. Hunkeler who made weight loss measurements, Ms. J. Kestner who digitized and plotted the data, and Mr. J. Ming for help in the data reduction.

Table 1

Sample #4 Am. CY 985-B - 626 Graphite - Epoxy Laminate

(m_r = 11.2 E-3 g, A = 6.24cm^2, m_o = 2.0631 g)

Temp. (°C)	Slope n	log(dm/dt 1/A)	1/Adm/dt (mg/cm^2 hr)	Outgass. rate g/cm^2/hr
25	-0.6204	-1.36432	4.325 E-2	4.325 E-5 $t^{-.6204}$
50	-0.56169	-1.14980	7.095 E-2	7.095 E-5 $t^{-.561}$
100	-0.72912	-0.69611	2.013 E-1	2.013 E-4 $t^{-.729}$
125	-0.8612	-0.537	2.904 E-1	2.013 E-4 $t^{-.8612}$
150	-.97099	-0.34138	4.560 E-1	4.56 E-4 $t^{-.970}$

Sample #10, Am. CY HST-7B-112 Graphite - Epoxy Laminate

(m_r = 15.4 E-3 g, A = 6.74cm^2, m_o = 2.888 g)

Temp. (°C)	Slope n	log(dm/dt 1/A)	1/Adm/dt (mg/cm^2/hr)	Outgass. rate (g/cm^2/hr)
25	—	—	—	—
50	-0.5986	-1.19956	6.32 E-2	6.32 E-5 $t^{-.598}$
75	-0.6445	-0.89015	1.28 E-1	1.28 E-4 $t^{-.644}$
100	-0.69871	-0.61653	2.42 E-1	2.42 E-4 $t^{-.616}$
125	-0.83342	-0.42813	3.73 E-1	3.73 E-4 $t^{-.833}$
150	-0.96452	-0.30934	4.90 E-1	4.90 E-4 $t^{-.964}$

NOTES:

m_r, the mass loss of the material at 125°C for 24 hours in vacuum.
m_o, the initial mass of the material @ 25°C ambient, 50% R.H.
t, Time (hr).
n, slope of the plot of outgass. rate vs time indicated on plot as X intercept.
log(dm/dt 1/A), indicated on plot as Y intercept.
To express the rate in the usual torr l/s.cm^2 multiply by 22.4 X 760/M3600, where M(g/mole).

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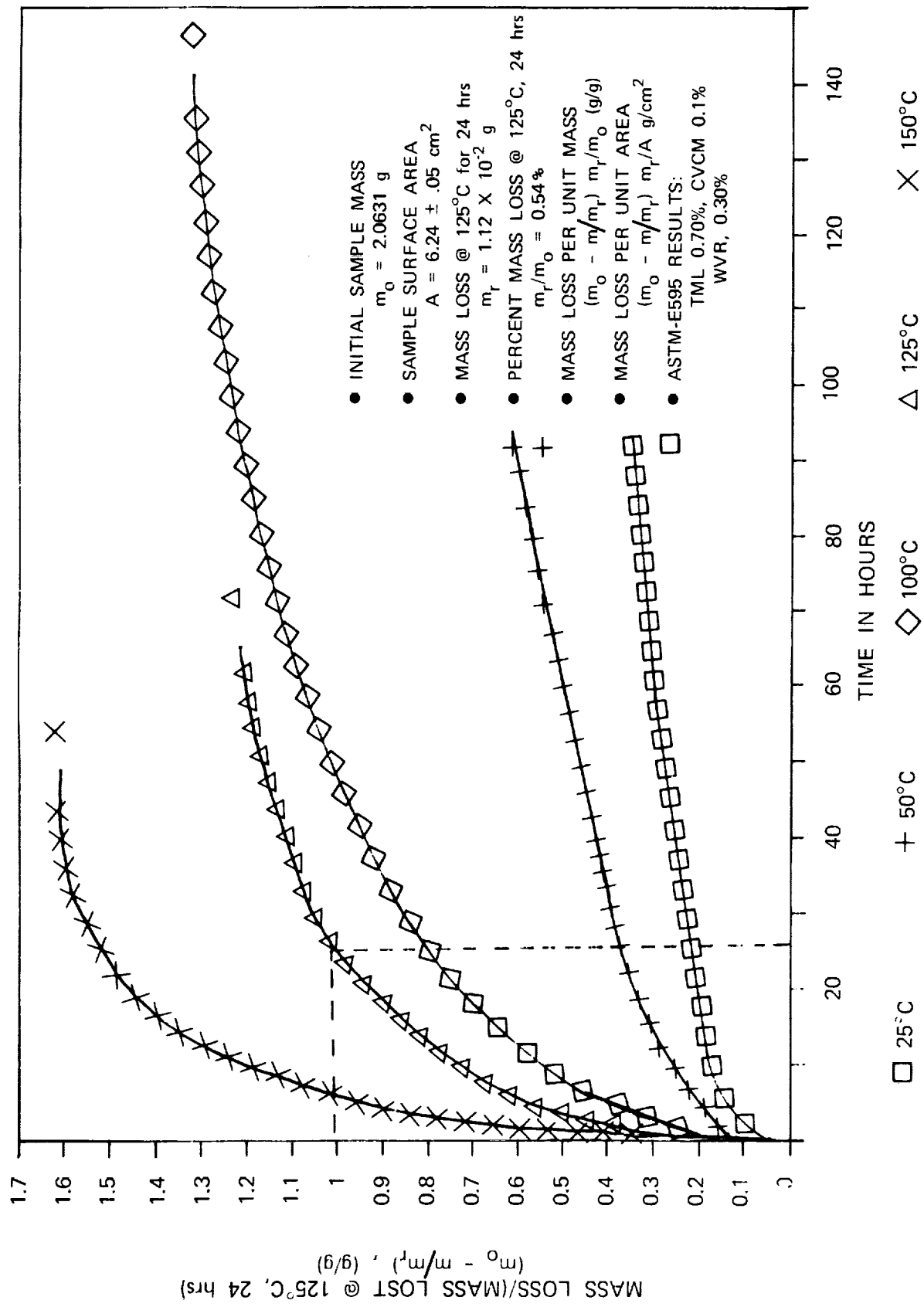


Figure 1. Normalized Mass Loss vs. Time
A.M. CY. 985B-626 Sample 4.

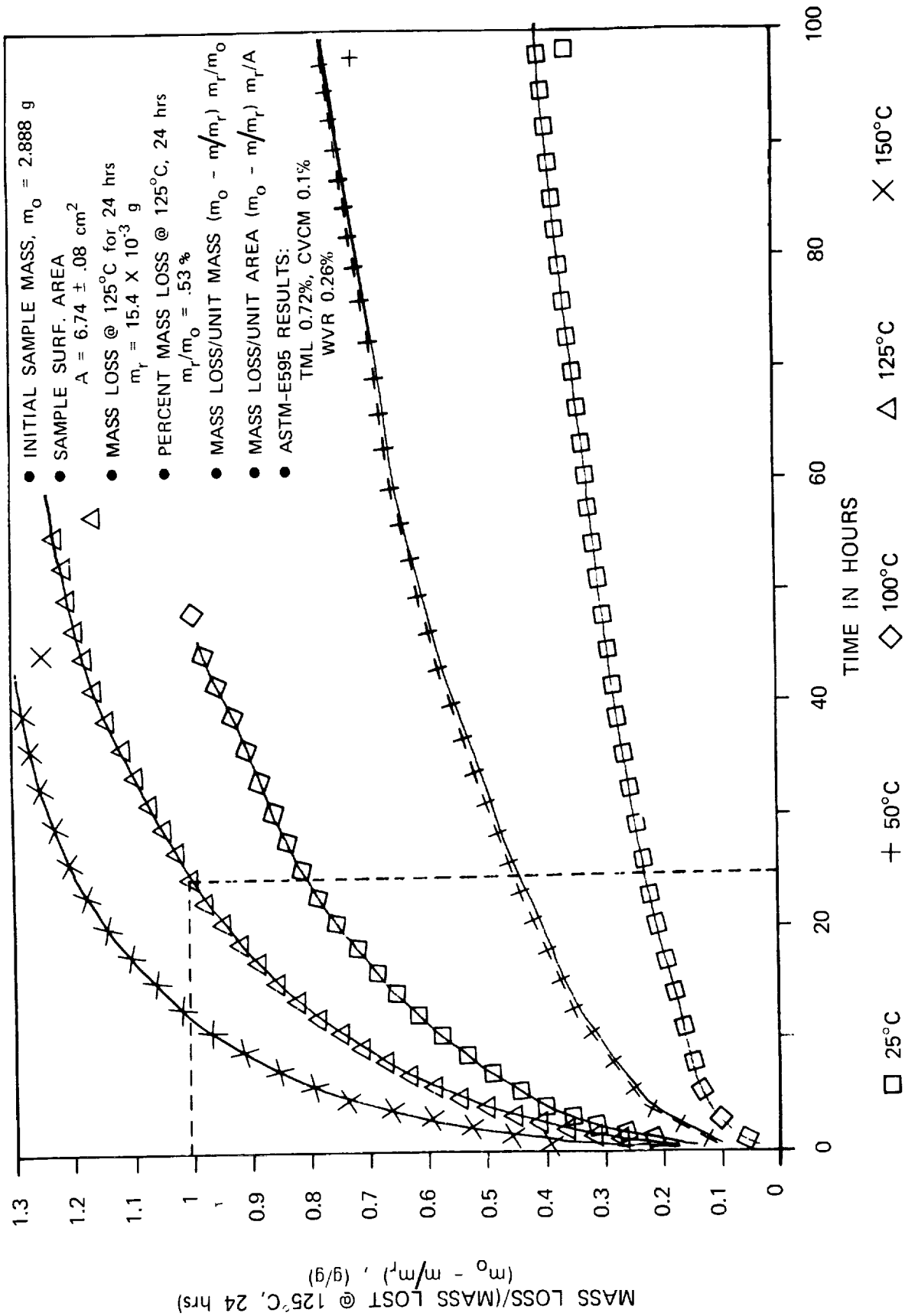
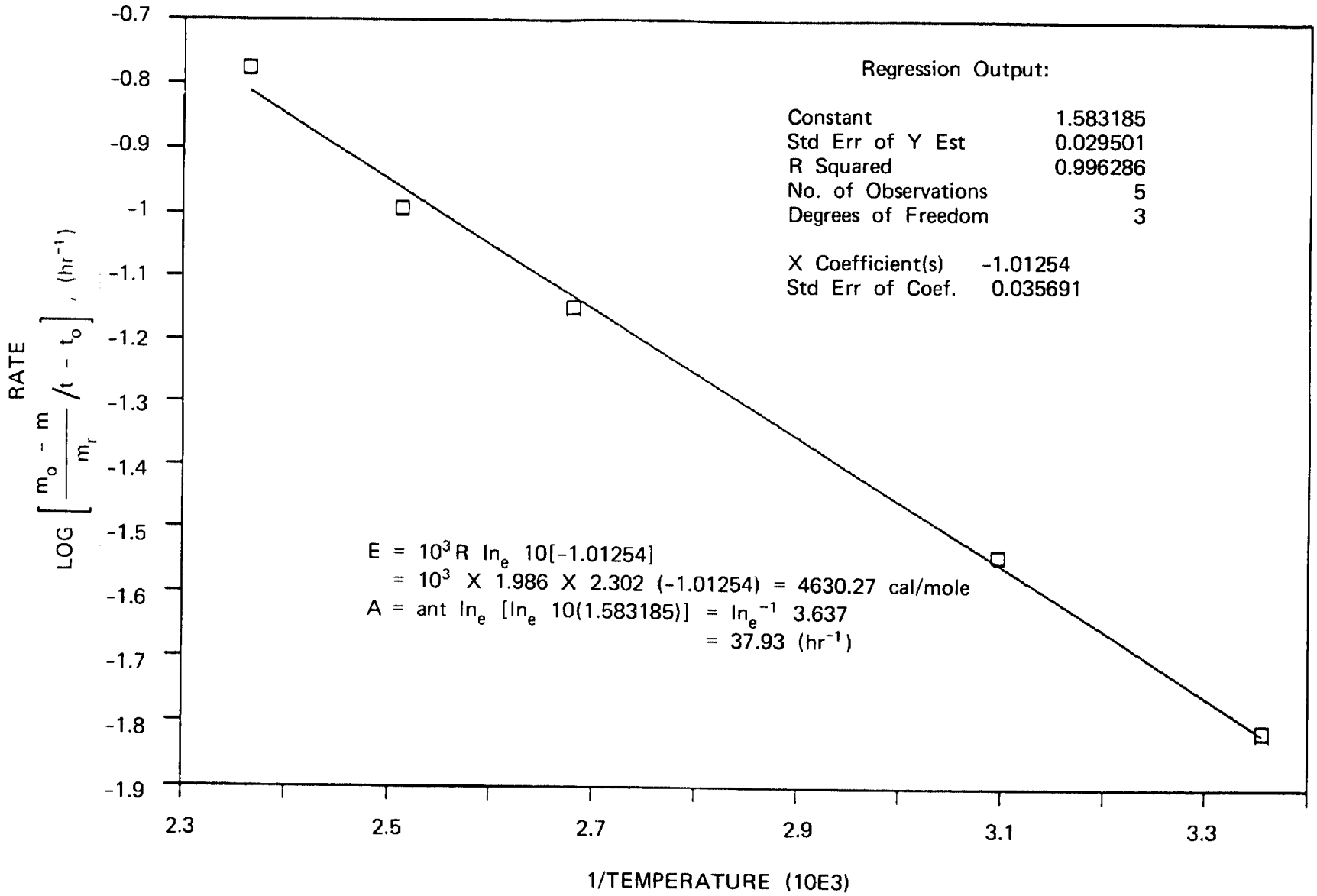


Figure 2. Normalized Mass Loss vs. Time
HST-7B-112 Sample 10.



Y INTERCEPT: 1.583185 X COEFFICIENT: -1.01254

Figure 3. Log of Normalized Initial Mass Loss vs. 1/T
985B-626 Sample 4.

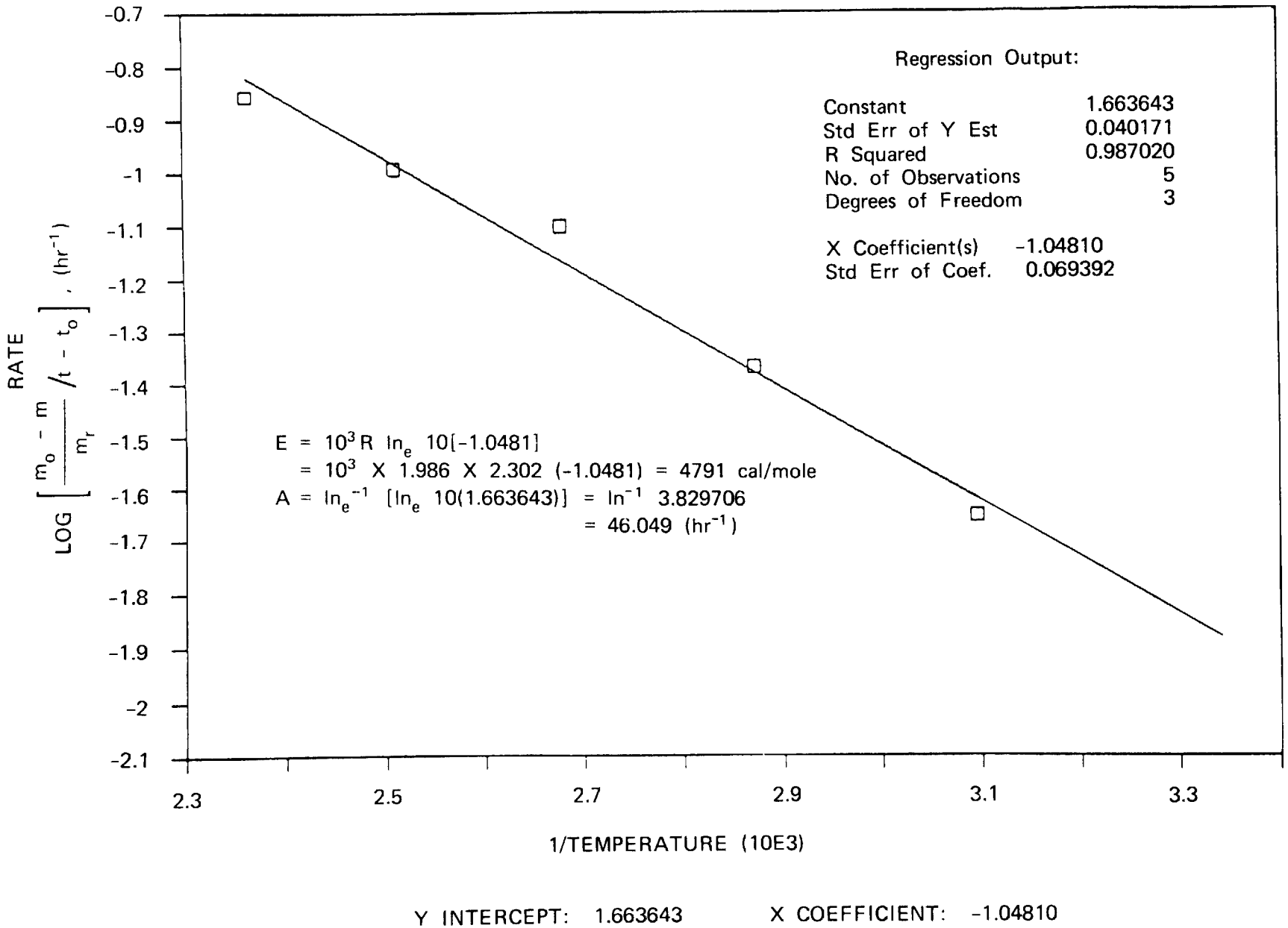


Figure 4. Log of Normalized Initial Mass Loss vs. 1/T
HST-7B-112 Sample 10.

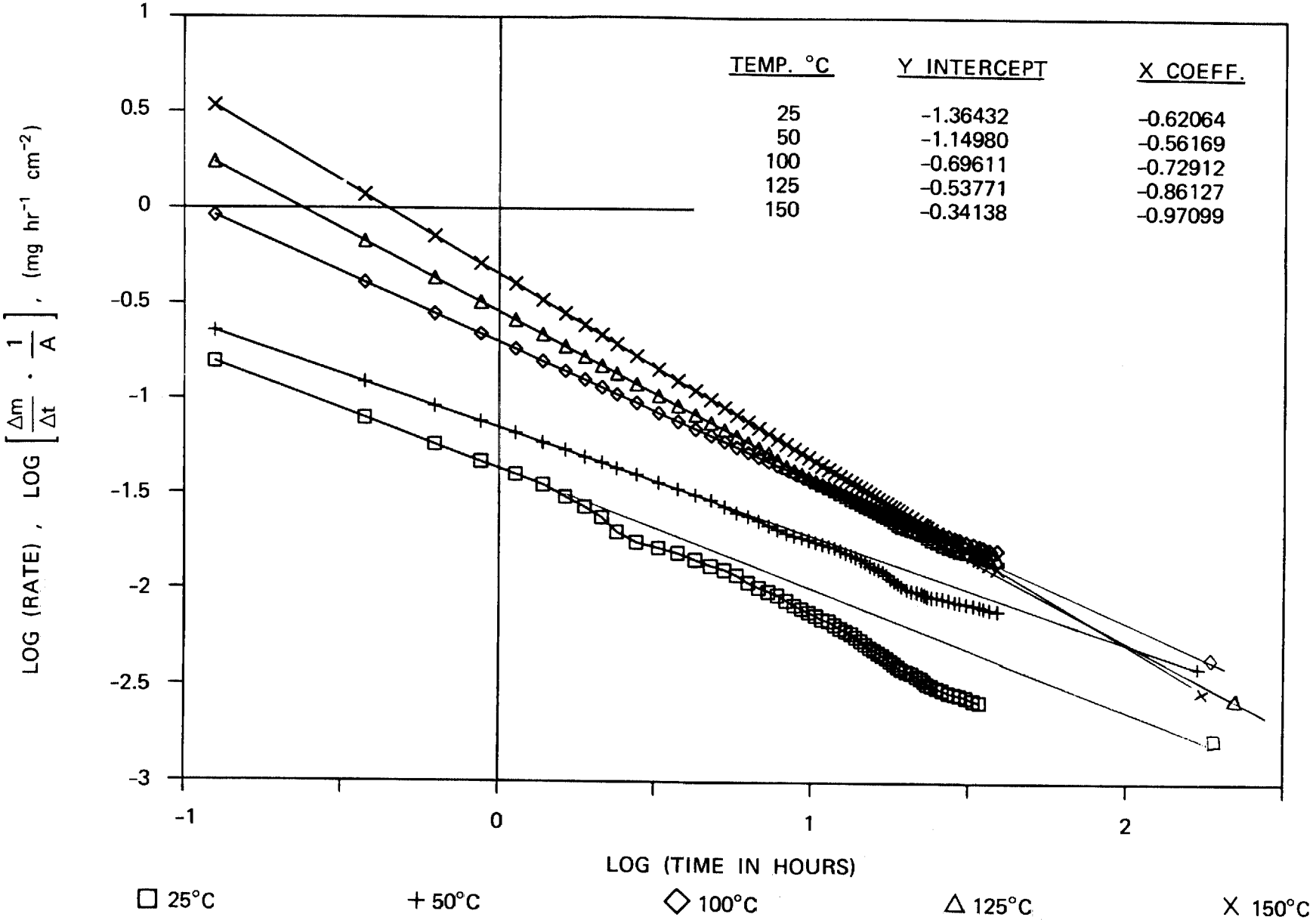


Figure 5. Outgassing Rates
985B-626 Sample 4.

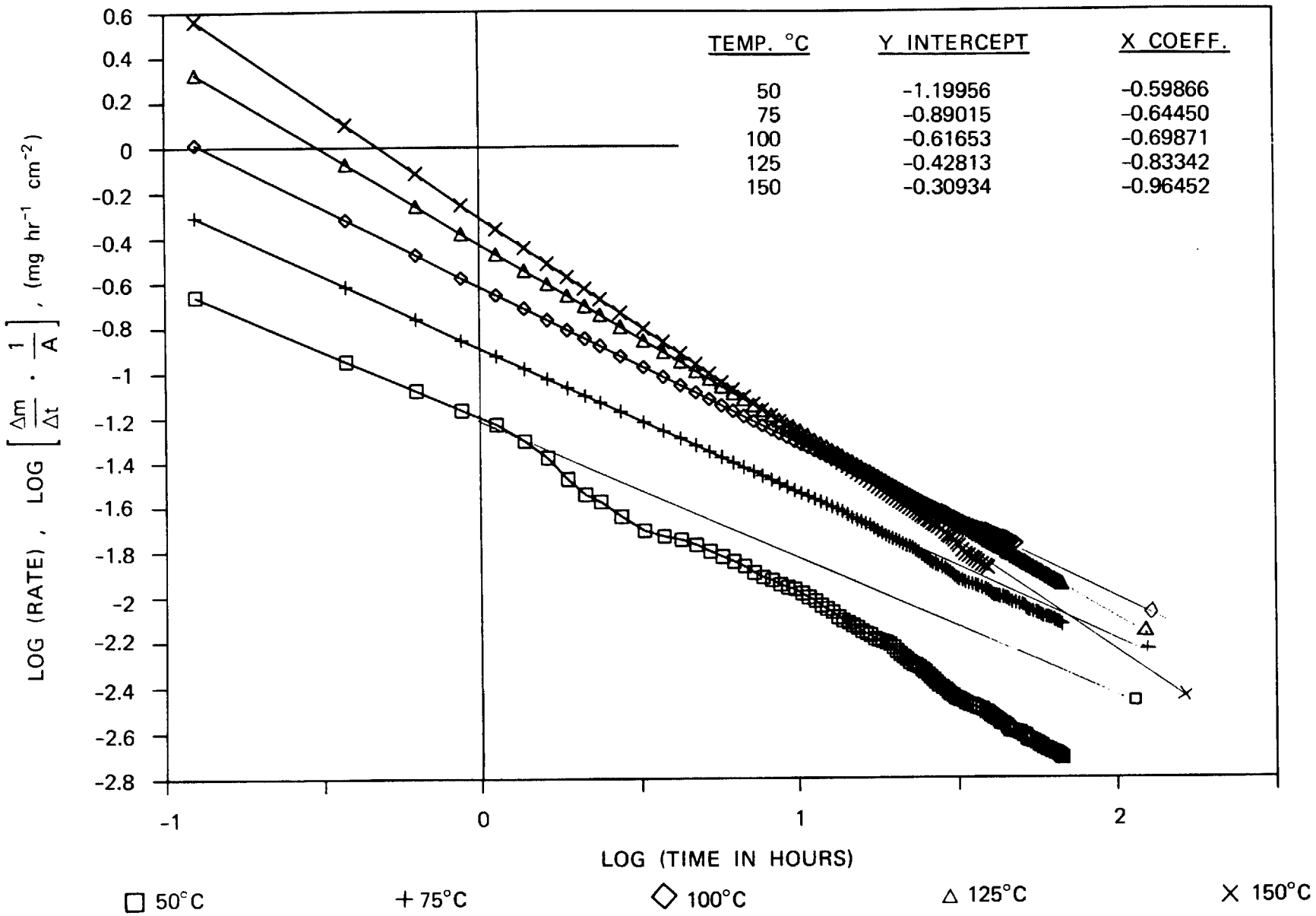


Figure 6. Outgassing Rates
HST-7B-112 Sample 10.

MICRO VCM TEST REQUEST AND DATA SHEET
(Requestor please read, then furnish all information above asterisks.)

Requested by Scialdone Phone Date 2-5-88
Material Identification (product name, number, description, classification, etc)
Laminate epoxy/graphite 985B-626
Manufacturer of Material (Name, City and State Address) (ACC-10)
Material application (general usage, i.e. adhesive, coating, potting, etc.) Laminate
J. O. Number 313-845-17-03-01 COBE mat'l Request Infra-Red Spectra? YES X NO

Indicate here all processing required by GSFC Materials personnel. Include the recipe, cure(s), and special instructions to be performed.

If material is to be tested in the "as received" condition, without further processing required, indicate in full, past history, recipe, cure(s), post cure(s), nomenclature of components, etc.

Sample #4 delaminates some on chopping & sawing

* *

CVCM test specimen preparation and description Sawed and cut into small irregularly shaped pieces

	19 16946		20 16947
Bar position-CVCM Test Number.....			
Init mass, holder and specimen, gm.....	.285228	.	.282538
Mass of holder, gm.....	.033365	.	.033830
Init specimen mass @ 50 %RH, gm.....	.251863	.	.248708
Mass after 50 %RH re-soak, gm.....	.284229	.	.281590
Final mass, holder & specimen, gm.....	.283468	.	.280820
Total mass loss, specimen, gm.....	.001760	.	.001718
Per-cent TML, specimen.....	0.70 %	. %	0.69 %
Average value TML	→	0.70 %	
Total mass, water vapor regain, gm.....	.000761	.	.000770
Per-cent water vapor regain.....	0.30 %	. %	0.31 %
Average value WVR @ 50 %RH	→	0.31 %	
Final mass, collector, gm.....	1.781255	.	1.790205
Initial mass, collector, gm.....	1.781235	.	1.790185
Collected mass - CVCM, gm.....	.000020	.	.000020
Per-cent CVCM.....	0.01 %	. %	0.01 %
Average value CVCM	→	0.01 %	

Remarks: CVCM (unweighable) on separator plate to ____ in diameter. CVCM appearance as follows: XX is not detectable on collector plates: _____ thin: _____ heavy: _____ color: _____ smooth: _____ smoky: _____ splotchy: ____ partially opaque: ____ opaque: ____ matte: _____ transparent: _____ interference fringes: ____ foggy: _____ colored liquid: ____ distorts eye reflection: ____ clear liquid: _____ liquid runs in excess: Deposit covers _____ % of collector discs:

Specimen appearance after test: no visible change

Test started FEB 9 1988 Test completed: FEB 10 1988 Period 24 Hrs.

Pressure 6.7 x 10-5 Pascal Specimen Temp. 125 °C Collector Temp. 25 °C

VCM REV 9 31 AUG 87 Analyst W.A. Campbell, Jr.

A-1. Sample 4, 985B-626 (#16946).

MICRO VCM TEST REQUEST AND DATA SHEET

(Requestor please read, then furnish all information above asterisks.)

Requested by Scialdone Phone _____ Date _____

Material Identification (product name, number, description, classification, etc)

Laminate epoxy/graphite 985B-626

Manufacturer of Material (Name, City and State Address) _____ (Acc -

Material application (general usage, i.e. adhesive, coating, potting, etc.) laminate

J. O. Number 313-473-19-06-01 Space Stn Request Infra-Red Spectra? YES ____ NO X

Indicate here all processing required by GSFC Materials personnel. Include the recipe, cure(s), and special instructions to be performed.

Rerun of GSC 16946 Duplicates
run Feb 9
positions 19 & 20

If material is to be tested in the "as received" condition, without further processing required, indicate in full, past history, recipe, cure(s), post cure(s), nomenclature of components, etc.

has seen 24hr @ 125°C @ 10^{-6} + ambient 30 days

* *

CVCM test specimen preparation and description small irregularly shaped pieces placed in boats

Bar position-CVCM Test Number.....	13 17004		14 17005
Init mass, holder and specimen, gm.....	.284703	.	.282230
Mass of holder, gm.....	.033973	.	.034383
Init specimen mass @ 30 %RH, gm.....	.250730	.	.247847
Mass after 36 %RH re-soak, gm.....	.284350	.	.281900
Final mass, holder & specimen, gm.....	.283894	.	.281455
Total mass loss, specimen, gm.....	.000809	.	.000775
Per-cent TML, specimen.....	0.32 %	. %	0.31 %
Average value TML	→	0.32 %	
Total mass, water vapor regain, gm.....	.000456	.	.000445
Per-cent water vapor regain.....	0.18 %	. %	0.18 %
Average value WVR @ 36 %RH	→	0.18 %	
Final mass, collector, gm.....	1.763980	.	1.767572
Initial mass, collector, gm.....	1.763985	.	1.767570
Collected mass - CVCM, gm.....	-.000005	.	.000002
Per-cent CVCM.....	0.00 %	. %	0.00 %
Average value CVCM	→	0.00 %	

Remarks: CVCM (unweighable) on separator plate to ____ as diameter. CVCM appearance as follows: X is not detectable on collector plates: ____ thin: ____ heavy: ____ color: ____ smooth: ____ smoky: ____ splotchy: ____ partially opaque: ____ opaque: ____ matte: ____ transparent: ____ interference fringes: ____ foggy: ____ colored liquid: ____ distorts eye reflection: ____ clear liquid: ____ liquid runs in excess: Deposit covers ____ % of collector discs:

Specimen appearance after test: no visible change

Test started MAR 8 1988 Test completed: MAR 9 1988 Period 24 Hrs.

Pressure 6.7×10^{-5} Pascal Specimen Temp. 125 °C Collector Temp. 25 °C

VCM REV 9 31 AUG 87 Analyst W.A. Campbell, Jr.

A-2. Retest of Sample 4, 985B-626 (#17004).

MICRO VCM TEST REQUEST AND DATA SHEET
(Requestor please read, then furnish all information above asterisks.)

Requested by Scialdone Phone _____ Date 2-5-88
Material Identification (product name, number, description, classification, etc)
laminate epoxy/graphite HST-7B-112-
Manufacturer of Material (Name, City and State Address) _____ (Acc-10)
Material application (general usage, i.e. adhesive, coating, potting, etc.) _____
J. O. Number 313-895-17-03-01 COR= Mat-1 Request Infra-Red Spectra? YES X NO _____

Indicate here all processing required by GSFC Materials personnel. Include the recipe, cure(s), and special instructions to be performed.

If material is to be tested in the "as received" condition, without further processing required, indicate in full, past history, recipe, cure(s), post cure(s), nomenclature of components, etc.
Sample #10 delaminates some on chopping & sawing

* *
CVCM test specimen preparation and description sawed and cut into small irregularly shaped pieces.

	21 16948		22 16949
Bar position-CVCM Test Number.....			
Init mass, holder and specimen, gm.....	.284270	.	.284953
Mass of holder, gm.....	.033385	.	.033205
Init specimen mass @ 50 %RH, gm.....	.250835	.	.251748
Mass after 50 %RH re-soak, gm.....	.283035	.	.283840
Final mass, holder & specimen, gm.....	.282450	.	.283135
Total mass loss, specimen, gm.....	.001770	.	.001818
Per-cent TML, specimen.....	0.71 %	. %	0.72 %
Average value TML		0.72 %	
Total mass, water vapor regain, gm.....	.000585	.	.000705
Per-cent water vapor regain.....	0.23 %	. %	0.28 %
Average value WVR @ 50 %RH		0.26 %	
Final mass, collector, gm.....	1.793053	.	1.799413
Initial mass, collector, gm.....	1.793035	.	1.799400
Collected mass - CVCM, gm.....	.000018	.	.000013
Per-cent CVCM.....	0.01 %	. %	0.01 %
Average value CVCM		0.01 %	

Remarks: CVCM (unweighable) on separator plate to ____ dia. CVCM appearance as follows: ____ is not detectable on collector plates: extremely thin: _____ heavy: blue color: _____ smooth: _____ smoky: _____ splotchy: ____ partially opaque: ____ opaque: ____ matte: _____ transparent: _____ interference fringes: ____ foggy: _____ colored liquid: ____ distorts eye reflection: ____ clear liquid: _____ liquid runs in excess: Deposit covers 0-10 % of collector discs: barely detectable

Specimen appearance after test: no visible change

Test started FEB 9 1988 Test completed: FEB 10 1988 Period 24 Hrs.

Pressure 6.7×10^{-5} Pascal Specimen Temp. 125 °C Collector Temp. 25 °C

VCM REV 9 31 AUG 87 Analyst W.A. Campbell, Jr.

A-3. Sample 10, HST-7B-112 (#16948).

MICRO VCM TEST REQUEST AND DATA SHEET

ORIGINAL PAGE IS OF POOR QUALITY

(Requestor please read, then furnish all information above asterisks.)

Requested by Sciaidone Phone _____ Date _____

Material Identification (product name, number, description, classification, etc)

laminate epoxy/graphite HST-7B-112

Manufacturer of Material (Name, City and State Address) _____ (ACC -

Material application (general usage, i.e. adhesive, coating, potting, etc.) laminate

J. O. Number 313-473-19-06-01 Space Stn Request Infra-Red Spectra? YES ____ NO X

Indicate here all processing required by GSFC Materials personnel. Include the recipe, cure(s), and special instructions to be performed.

Reveal asc 16 Feb 88 Duplicates positions 21 & 22

If material is to be tested in the "as received" condition, without further processing required, indicate in full, past history, recipe, cure(s), post cure(s), nomenclature of components, etc.

has seen 24 hr @ 125°C @ 10^{-6} + ambient 30 days

* *

CVCM test specimen preparation and description small irregularly shaped pieces placed in boats

Bar position-CVCM Test Number	15 17006		16 17007
Init mass, holder and specimen, gm	.285586		.284416
Mass of holder, gm	.034817		.034600
Init specimen mass @ 30 %RH, gm	.250769		.249816
Mass after 36 %RH re-soak, gm	.285220		.284013
Final mass, holder & specimen, gm	.284787		.283677
Total mass loss, specimen, gm	.000799		.000739
Per-cent TML, specimen	0.32 %	%	0.30 %
Average value TML		0.31 %	
Total mass, water vapor regain, gm	.000433		.000336
Per-cent water vapor regain	0.17 %	%	0.13 %
Average value WVR @ 36 %RH		0.15 %	
Final mass, collector, gm	1.770410		1.772093
Initial mass, collector, gm	1.770409		1.772090
Collected mass - CVCM, gm	.000001		.000003
Per-cent CVCM	0.00 %	%	0.00 %
Average value CVCM		0.00 %	

Remarks: CVCM (unweighable) on separator plate to ___ dia. CVCM appearance as follows: X is not detectable on collector plates: ___ thin: ___ heavy: ___ color: ___ smooth: ___ smoky: ___ splotchy: ___ partially opaque: ___ opaque: ___ matte: ___ transparent: ___ interference fringes: ___ foggy: ___ colored liquid: ___ distorts eye reflection: ___ clear liquid: ___ liquid runs in excess: Deposit covers ___ % of collector discs:

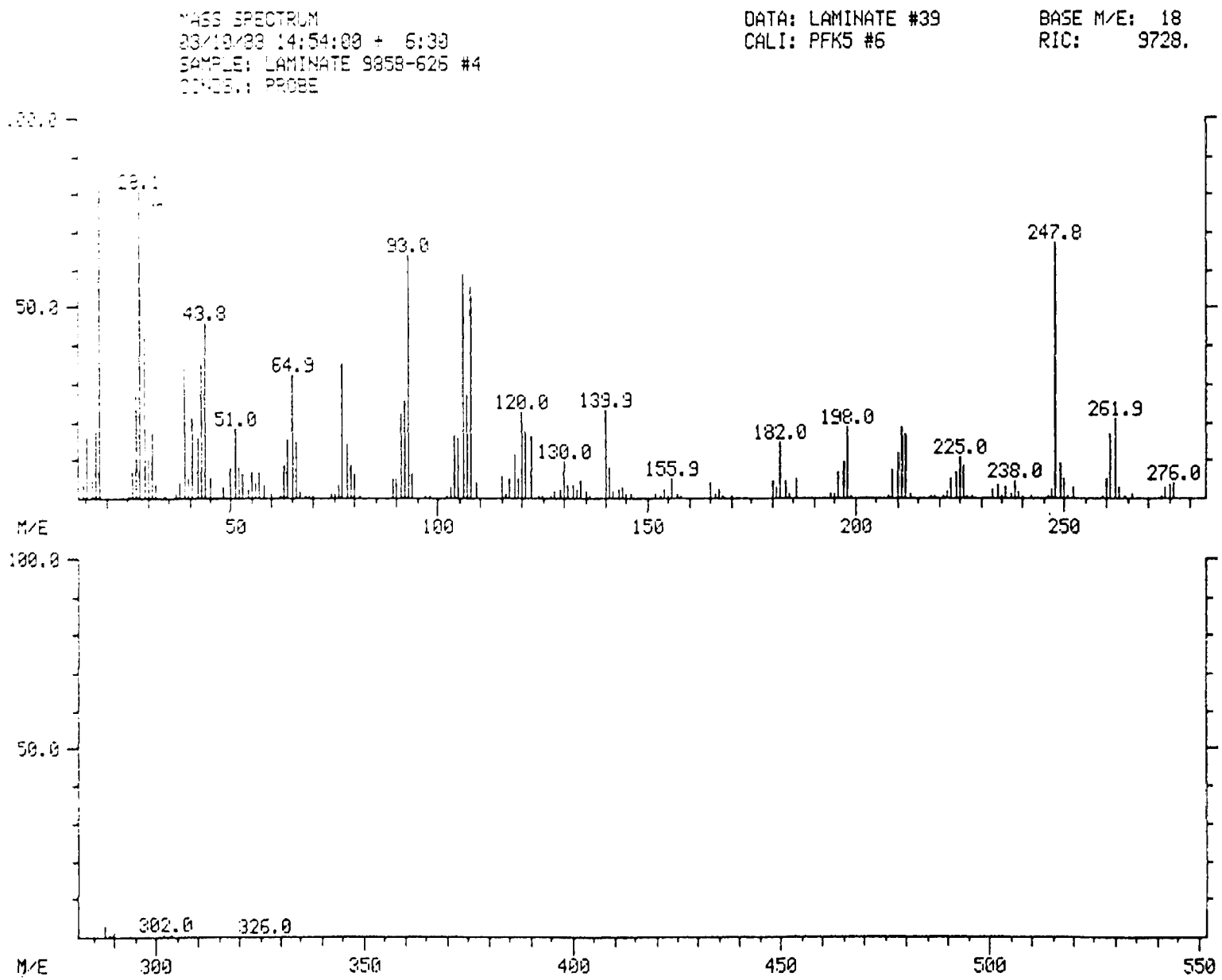
Specimen appearance after test: no visible change

Test started MAR 8 1988 Test completed: MAR 9 1988 Period 24 Hrs.

Pressure 6.7 x 10^{-5} Pascal Specimen Temp. 125 °C Collector Temp. 25 °C

VCM REV 9 31 AUG 87 Analyst W.A. Campbell, Jr.

A-4. Retest of Sample 10, HST-7B-112 (#17006).



A-5.

NASA
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Report Documentation Page

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15. Supplementary Notes

16. Abstract The outgassing rates of two Graphite-Epoxy laminates, American Cyanamide 985B-626 and HST-7B-112, were obtained using a gravimetric method. The rates as a function of time and temperature were derived from the measurements of their mass losses at temperatures varying from 25°C to 150°C and for a time span of up to 400 hours in a vacuum. The data from those measurements were reduced to obtain the outgassing activation energies, the mass losses per unit mass or area, and the corresponding outgassing rates. The rates are expressed in closed-form equations and are directly usable for modeling computations. The procedures to obtain these parameters are shown and may be used for the evaluation of other materials. The results of the tests show that the activation energies of the two materials are: 4630 cal/mole for the 985B-626 material and 4791 cal/mole for the HST-7B-112 sample #10 Graphite Epoxy. The outgassing rates of these materials are in the 10E-5 g/cm^{2}hr range and they decay according to a power of time of 0.60 at 25°C, indicating that the outgassing process is mainly a diffusion at that temperature. The normalized mass losses versus time obtained from these tests have been compared to the discrete results obtained from the ASTM-E595 teses. The comparison provides general indications on the effects of temperature and time in relation to the ASTM test values obtained at 125°C for a 24-hour test duration.

17. Key Words (Suggested by Author(s))
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