THERMOGRAVIMETRIC DETERMINATION OF KINETIC PARAMETERS FOR THE T-ETC(U)

DEC 80  M R TANT, J B HENDERSON, G R MOORE

UNCLASSIFIED NSWC/TR-80-290
**Title:** Thermo gravimetric determination of kinetic parameters for the thermal degradation of several ablative materials

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**Distribution Statement:** Approved for public release; distribution unlimited.

**Abstract:** The thermal decomposition of several composite ablative materials being considered for shipboard use was studied using thermogravimetry. Thermograms were obtained at six heating rates ranging from 10 to 160°C/min. From these data, the kinetic parameters were determined by a modified version of Friedman's method. Activation energies determined by this method were compared to those obtained by the method of Flynn and Wall.
FOREWORD

This work was performed as part of an investigation to screen and classify candidate ablative materials for use in shipboard applications. Funds were provided by Naval Sea Systems Command (SEA-62R) and by NSWC independent research program Task Area Number ZR000-01-01.

This work was reviewed and approved by J. J. Yagla, Head, Ship Safety Engineering Branch, and J. F. Horton, Head, Systems Safety Division.

Released by:

THOMAS A. CLARE, Head
Combat Systems Department
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INTRODUCTION

The rate of decomposition of an ablative material when exposed to a heat flux may be modeled by the kinetic rate equation. If it is assumed that the material dimensions remain constant, the rate equation predicts the density of the remaining char as a function of time. The ablative characteristics of the material are dependent upon both the rate of decomposition and char density. In order to predict the thermal response of the material, accurate values of the kinetic parameters over the entire range of decomposition are required for use in the rate equation. In the case of an ablative material exposed to a solid rocket motor exhaust, the heat flux may vary widely depending upon the geometry and/or type of motor. Therefore, the effect of the heating rate on the kinetic parameters must be known as well.

We report here the kinetic parameters for the thermal decomposition of several ablative materials being considered for shipboard application. These kinetic parameters were determined by a multiple heating rate thermogravimetric technique developed by Friedman\textsuperscript{1} and later modified by Henderson, Wiebelt, Tant, and Moore\textsuperscript{2} to improve accuracy of the kinetic model over the entire range of decomposition. This modified version of Friedman’s method involves dividing the decomposition into two separate regions. While an average activation energy is determined for the entire decomposition range, separate pre-exponential factors and apparent orders of reaction are determined for the two regions of decomposition. For comparison with the value obtained by Friedman’s method,\textsuperscript{1} the activation energy was also determined by the method of Flynn and Wall.\textsuperscript{3}

THEORY

The kinetic rate equation simply defines the rate of decomposition as the product of a temperature-dependent term and a weight fraction-dependent term, i.e.

\[ \text{rate} = f_1 \text{(temperature)} \cdot f_2 \text{(weight fraction)} \]  

(1)

The temperature-dependent term is generally expressed by Arrhenius’ law

\[ f_1 = A \exp\left(\frac{-E}{RT}\right) \]  

(2)

where $A$ = pre-exponential factor (min$^{-1}$)

$E$ = activation energy (cal/gmol)

$R$ = gas constant (1.987 cal/gmol·K)

$T$ = absolute temperature (K)
The form of the weight fraction-dependent term is, in general, chosen to provide a best fit of experimental data.

**FRIEDMAN'S METHOD**

Friedman's method was chosen for this application because the kinetic parameters are determined from data obtained over a wide range of heating rates. Clearly, parameters obtained over a range of heating rates may be applied with greater confidence at the heating rates to which ablative materials are subjected. Another advantage of this method is that the weight fraction-dependent term is an arbitrary function whose form is determined directly by experiment.

The rate equation proposed by Friedman is

\[-\frac{1}{W_0} \times \frac{dW}{dt} = Af\left(\frac{W}{W_0}\right) \exp\left(-\frac{E}{RT}\right)\]  

where  
- \(W\) = instantaneous weight of material (mg)  
- \(W_0\) = original weight of material (mg)  
- \(\frac{dW}{dt}\) = rate of weight loss (mg/min)  
- \(f\left(\frac{W}{W_0}\right)\) = undefined function of weight

Taking the natural logarithm of both sides of Equation (3) results in

\[\ln\left[-\frac{1}{W_0} \times \frac{dW}{dt}\right] = \ln\left[Af\left(\frac{W}{W_0}\right)\right] - \frac{E}{RT}\]  

A linear equation may be fit to \(\ln\left[-\frac{1}{W_0} \times \frac{dW}{dt}\right]\) as a function of \(\frac{1}{T}\) at constant parametric values of \(\frac{W}{W_0}\). These equations will have slopes of \(-\frac{E}{R}\), and each intercept is the value of \(\ln\left[Af\left(\frac{W}{W_0}\right)\right]\) at the parametric value of \(\frac{W}{W_0}\). The weight fraction-dependent term, \(f\left(\frac{W}{W_0}\right)\), is then defined as

\[f\left(\frac{W}{W_0}\right) = \left[\frac{W - W_f}{W_0}\right]^n\]  

where  
- \(n\) = order of reaction  
- \(W_f\) = final weight of charred material (mg)

Finally, multiplying Equation (5) by \(A\) and taking the natural logarithm results in

\[\ln\left[Af\left(\frac{W}{W_0}\right)\right] = n A + n \ln\left[\frac{W - W_f}{W_0}\right]\]
The final weight fraction, \(W_f/W_0\), is determined from the original thermograms. Since \(\ln[\text{Af}(W/W_0)]\) is known for various \(W/W_0\) ratios, Equation (6) can be used to determine values of \(A\) and \(n\). As mentioned previously, Henderson et al.\(^2\) modified this method to improve the correlation of the kinetic model and experimental data. The weight loss curve was separated into two regions, and separate pre-exponential factors and apparent orders of reaction were determined for each region. A single average activation energy was determined for the entire weight loss curve.

FLYNN AND WALL'S METHOD

The method of Flynn and Wall\(^3\) may be used to determine the activation energy utilizing thermograms obtained at several heating rates. The activation energy is given by

\[
E \approx -(R/C) \frac{d(\log \beta)}{dT}
\]

(7)

where \(\beta = \text{heating rate (K/min)}\)

\[
C = C(E/RT)
\]

If \(1/T\) versus \(\log \beta\) is plotted at several weight loss fractions, a series of straight lines with slope \(\Delta(\log \beta)/\Delta(1/T)\) results. The activation energy can then be calculated by Equation (7) using the slope and the appropriate value of \(C\). Since \(C\) is a function of \(E/RT\), the calculation of \(E\) is an iterative process. Values of \(C\) over the range \(7 \leq E/RT \leq 60\) are available from a table constructed by Flynn and Wall.\(^3\) The variation of \(C\) over this range is approximately \(\pm 3\) percent. The simplicity of this method makes its use for the present application quite attractive.

EXPERIMENTAL

MATERIALS

The four ablative materials studied were supplied by Fiberite Corp. and Fiber Materials, Inc. As shown in Table 1, these materials consisted of either a phenol-formaldehyde or acrylonitrile-butadiene resin with specified amounts of glass or fiberglass added as filler.

The materials were converted to powder form by machining and were then filtered through a No. 20 sieve. They were stored overnight in a vacuum dessicator maintained at 35°C to remove traces of water.
Table 1. Composition of Materials Tested

<table>
<thead>
<tr>
<th>Contents</th>
<th>Fiberite Corp. MXBE-350</th>
<th>Fiberite Corp. MXB-360</th>
<th>Fiber Materials, Inc. FR-1</th>
<th>Fiber Materials, Inc. FR-2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Glass powder (SiO₂)</td>
<td>15.5</td>
<td>14.5</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Glass fiber (SiO₂)</td>
<td>-</td>
<td>-</td>
<td>42.0</td>
<td>-</td>
</tr>
<tr>
<td>Fiberglass</td>
<td>41.0</td>
<td>59.0</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Carbon</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>40.0</td>
</tr>
<tr>
<td>Total filler content</td>
<td>56.5</td>
<td>73.5</td>
<td>42.0</td>
<td>40.0</td>
</tr>
<tr>
<td>Pheno-formaldehyde resin</td>
<td>-</td>
<td>26.5</td>
<td>58.0</td>
<td>60.0</td>
</tr>
<tr>
<td>Acrylonitrile-butadiene resin</td>
<td>43.5</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

**APPARATUS AND PROCEDURE**

A Perkin-Elmer TGS-2 Thermogravimetric System was used, with temperature control provided by a Perkin-Elmer System 4 Microprocessor Controller. The sample temperature was measured with a chromel-alumel thermocouple, which was calibrated with a set of five Curie standards in the temperature range of interest at each heating rate used.\(^4\)

In order to reduce temperature gradients in the material and to ensure uniform heating, small weights of a powdered form of the materials were used. Samples weighing 7.5 ± 0.5 mg were heated from 40 to 950°C using heating rates of 10, 20, 40, 80, 100, and 160°C/min. Both the percentage of initial weight and the rate of weight loss were plotted directly as a function of temperature. The samples were maintained in a nitrogen atmosphere throughout the experiment. When the programmed temperature scan reached 950°C, the purge gas was automatically switched to oxygen to thermo-oxidatively degrade the remaining resin. To verify the initial weight fraction of filler, the temperature was held at 950°C until the resin had completely degraded.
RESULTS

In the original thermograms, both the fraction of weight remaining and the derivative of weight loss were plotted as a function of temperature. These data were digitized at 0.01 intervals of the fraction of weight remaining and the experimental temperatures were corrected using the Curie standard temperature calibration. The thermograms were then reproduced from these data. Figure 1 shows the fraction of weight remaining as a function of temperature for each material at each heating rate. Similarly, Figure 2 shows the rate of weight loss as a function of temperature for each material at each heating rate. The digitized data for all four materials are given in the Appendix.

Plots of $\ln(-1/W_0 \times dW/dt)$ versus $1/T$ are shown in Figure 3 for each of the four materials. The slope of each line was determined from a least squares fit of the data, and the corresponding activation energy and intercept $\ln[Af(W/W_0)]$ at each value of weight loss were then determined. These data are shown in Figure 4 for each material. Plots of $\ln[Af(W/W_0)]$ versus $\ln[(W-W_f)/W_0]$ are shown in Figure 5 for each material. Figure 5 clearly illustrates the separation of the decomposition into two regions and the corresponding least squares fit for each region. A separate pre-exponential factor and reaction order was determined for each of these two regions utilizing Equation (6). The average activation energies determined from Figure 4 were used for both regions. Using Flynn and Wall's method and the plots of log $\beta$ versus $1/T$ shown in Figure 6, average activation energies were determined for each of the four materials. A summary of the calculated kinetic parameters for the four materials is given in Table 2.

The kinetic parameters for the thermal decomposition of the four ablative materials were used in Equation (3) to calculate the fraction of weight remaining as a function of temperature at each heating rate. Each set of parameters was applied to that portion of the weight loss curve from which it was determined. The results of these calculations at 10 and 160°C/min are shown along with experimental data in Figure 7 for comparison. The average error, standard deviation of errors, and the 95-percent confidence interval were calculated for the experimental versus calculated points for each material, and these results are presented in Table 3.
Figure 1.a  Weight Loss Thermograms for MXBE-350 at Several Heating Rates

Figure 1.b  Weight Loss Thermograms for MXB-360 at Several Heating Rates
Figure 1.c  Weight Loss Thermograms for FR-1 at Several Heating Rates

Figure 1.d  Weight Loss Thermograms for FR-2 at Several Heating Rates
Figure 2.a  Rate of Weight Loss for MXBE-350 at Several Heating Rates

Figure 2.b  Rate of Weight Loss for MXB-360 at Several Heating Rates
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Figure 2.d  Rate of Weight Loss for FR-2 at Several Heating Rates
Figure 3.a  Plot of Slopes Used to Determine the Activation Energy for MXBE-350

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Figure 5.b Plot to Determine Pre-exponential Factor and Order of Reaction for Two Regions of Weight Loss for MXB-360
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Figure 5.d  Plot to Determine Pre-exponential Factor and Order of Reaction for Two Regions of Weight Loss for FR-2

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Figure 6.a  Plot of Slopes Used to Determine Activation Energy
by Flynn and Wall's Method for MXBE-350

Figure 6.b  Plot of Slopes Used to Determine Activation Energy
by Flynn and Wall's Method for MXB-360
Figure 6.c Plot of Slopes Used to Determine Activation Energy by Flynn and Wall's Method for FR-1

Figure 6.d Plot of Slopes Used to Determine Activation Energy by Flynn and Wall's Method for FR-2
**Table 2. Results of Thermogravimetric Analysis**

<table>
<thead>
<tr>
<th>Material</th>
<th>( W_f/W_0 )</th>
<th>Range of ( W/W_0 )</th>
<th>( E_{av} ) (kcal/gm-mole)</th>
<th>( A(\text{min}^{-1}) )</th>
<th>( n )</th>
<th>( W/W_0 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fiberite</td>
<td>0.660</td>
<td>0.99 - 0.73</td>
<td>52.11</td>
<td>4.07 ( \times 10^{46} )</td>
<td>55.40</td>
<td>( &gt; 0.94 )</td>
</tr>
<tr>
<td>MXBE-350</td>
<td></td>
<td></td>
<td></td>
<td>7.77 ( \times 10^{17} )</td>
<td>3.81</td>
<td>( &lt; 0.94 )</td>
</tr>
<tr>
<td>Fiberite</td>
<td>0.840</td>
<td>0.98 - 0.87</td>
<td>56.87</td>
<td>2.74 ( \times 10^{38} )</td>
<td>22.10</td>
<td>( &gt; 0.93 )</td>
</tr>
<tr>
<td>MXB-360</td>
<td></td>
<td></td>
<td></td>
<td>1.15 ( \times 10^{23} )</td>
<td>7.60</td>
<td>( &lt; 0.93 )</td>
</tr>
<tr>
<td>Fiber materials</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>FR-1</td>
<td>0.650</td>
<td>0.99 - 0.67</td>
<td>94.62</td>
<td>3.05 ( \times 10^{94} )</td>
<td>92.43</td>
<td>( &gt; 0.84 )</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>5.65 ( \times 10^{35} )</td>
<td>11.88</td>
<td>( &lt; 0.84 )</td>
</tr>
<tr>
<td>Fiber materials</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>FR-2</td>
<td>0.675</td>
<td>0.99 - 0.83</td>
<td>49.05</td>
<td>1.71 ( \times 10^{55} )</td>
<td>59.50</td>
<td>( &gt; 0.89 )</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>2.00 ( \times 10^{28} )</td>
<td>19.10</td>
<td>( &lt; 0.89 )</td>
</tr>
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</table>

**Table 3. Statistical Analysis of Errors in Computed Versus Experimental \( W/W_0 \)**

<table>
<thead>
<tr>
<th>Material</th>
<th>Average Error (percent)</th>
<th>Standard Deviation (percent)</th>
<th>95-Percent Confidence Interval</th>
<th>Number of Data Points</th>
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<tr>
<td>MXBE-350</td>
<td>1.36</td>
<td>1.41</td>
<td>1.17 - 1.55</td>
<td>162</td>
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<tr>
<td>MXB-360</td>
<td>0.18</td>
<td>0.48</td>
<td>0.08 - 0.29</td>
<td>72</td>
</tr>
<tr>
<td>FR-1</td>
<td>0.50</td>
<td>1.22</td>
<td>0.38 - 0.72</td>
<td>198</td>
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<tr>
<td>FR-2</td>
<td>1.07</td>
<td>2.30</td>
<td>0.74 - 1.41</td>
<td>90</td>
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Figure 7.a Comparison of Calculated and Experimental Weight Loss Curves at 10 to 160°C/min for MXBE-350

Figure 7.b Comparison of Calculated and Experimental Weight Loss Curves at 10 to 160°C/min for MXB-360
Figure 7.c Comparison of Calculated and Experimental Weight Loss Curves at 10 to 160°C/min for FR-1

Figure 7.d Comparison of Calculated and Experimental Weight Loss Curves at 10 to 160°C/min for FR-2
DISCUSSION

As can be seen from the calculated versus experimental weight loss curves presented in Figure 7, the model predicts the weight loss with reasonable accuracy. The error in predicting the weight loss curves for MXBE-350, FR-1, and FR-2 could likely be reduced by separating the reaction into more than two regions, and determining a set of kinetic parameters for each region. Improving the fit of the lines to the data in Figure 5 leads to kinetic parameters that better predict the weight loss curve. The poor correlation of the calculated versus experimental weight loss curves at high temperatures for FR-2 in Figure 7d is the result of not utilizing experimentally deduced kinetic parameters in this region. Application of Friedman's method in this region led to negative activation energies, a result that is clearly not realistic in view of the physical meaning of activation energy. Therefore, the kinetic parameters determined from experimental data at lower temperatures and involving positive activation energies were used in the high-temperature region.

REFERENCES


## Weight Loss and Rate of Weight Loss Data for MXBE-350

<table>
<thead>
<tr>
<th>( w_0 )</th>
<th>Heating Rate 160°C/min</th>
<th>Heating Rate 100°C/min</th>
<th>Heating Rate 80°C/min</th>
<th>Heating Rate 40°C/min</th>
<th>Heating Rate 20°C/min</th>
<th>Heating Rate 10°C/min</th>
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<tr>
<td>( w_0 = 7.8020 )</td>
<td>( w_0 = 7.8451 )</td>
<td>( w_0 = 7.9097 )</td>
<td>( w_0 = 7.8589 )</td>
<td>( w_0 = 7.9766 )</td>
<td>( w_0 = 7.8000 )</td>
<td></td>
</tr>
<tr>
<td>( T(°C) )</td>
<td>(-1/w_0)(dw/dt) ( (1/min) )</td>
<td>( T(°C) )</td>
<td>(-1/w_0)(dw/dt) ( (1/min) )</td>
<td>( T(°C) )</td>
<td>(-1/w_0)(dw/dt) ( (1/min) )</td>
<td>( T(°C) )</td>
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<tr>
<td>0.99</td>
<td>255.9</td>
<td>0.0172</td>
<td>242.6</td>
<td>0.0121</td>
<td>234.0</td>
<td>0.0054</td>
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<tr>
<td>0.98</td>
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<td>0.0245</td>
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<td>0.97</td>
<td>354.4</td>
<td>0.0565</td>
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<td>0.0388</td>
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<td>0.0728</td>
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<tr>
<td>0.95</td>
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<td>0.94</td>
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<td>0.0875</td>
<td>379.1</td>
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<td>374.1</td>
<td>0.0436</td>
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<td>0.93</td>
<td>407.7</td>
<td>0.1015</td>
<td>393.2</td>
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<td>388.1</td>
<td>0.0559</td>
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<tr>
<td>0.92</td>
<td>419.2</td>
<td>0.1318</td>
<td>404.2</td>
<td>0.0909</td>
<td>401.2</td>
<td>0.0742</td>
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<td>0.91</td>
<td>428.7</td>
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<td>0.1120</td>
<td>408.2</td>
<td>0.0951</td>
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<td>0.90</td>
<td>432.9</td>
<td>0.1965</td>
<td>417.3</td>
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<td>0.1085</td>
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<td>0.89</td>
<td>439.2</td>
<td>0.2152</td>
<td>425.3</td>
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<td>0.88</td>
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<td>431.3</td>
<td>0.1706</td>
<td>427.3</td>
<td>0.1379</td>
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Weight Loss and Rate of Weight Loss Data for MXBE-350 (Continued)

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<tr>
<th>w0^w</th>
<th>Heating Rate 160°C/min</th>
<th>Heating Rate 100°C/min</th>
<th>Heating Rate 80°C/min</th>
<th>Heating Rate 40°C/min</th>
<th>Heating Rate 20°C/min</th>
<th>Heating Rate 10°C/min</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>T(°C)</td>
<td>(-1/w0)dw/dt (l/min)</td>
<td>T(°C)</td>
<td>(-1/w0)dw/dt (l/min)</td>
<td>T(°C)</td>
<td>(-1/w0)dw/dt (l/min)</td>
</tr>
<tr>
<td>0.87</td>
<td>450.8</td>
<td>0.2775</td>
<td>435.5</td>
<td>0.1807</td>
<td>434.3</td>
<td>0.1440</td>
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<td>0.86</td>
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<td>0.2947</td>
<td>440.4</td>
<td>0.1917</td>
<td>438.3</td>
<td>0.1508</td>
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<td>0.3111</td>
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<td>0.1980</td>
<td>444.4</td>
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Weight Loss and Rate of Weight Loss Data for MXBE-350 (Continued)

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Weight Loss and Rate of Weight Loss Data for MXB-360
Weight Loss and Rate of Weight Loss Data for FR-1

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<td>(-1/w₀)dw/dt (l/min)</td>
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## Weight Loss and Rate of Weight Loss Data for FR-1 (Continued)

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<th>Heating Rate 80°C/min ( w₀ = 7.6211 )</th>
<th>Heating Rate 40°C/min ( w₀ = 7.7786 )</th>
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<td>( T(°C) ) ((-1/w₀)(dw/dt) ) (1/min)</td>
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## Weight Loss and Rate of Weight Loss Data for FR-1 (Continued)

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