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Assessment of Uncertainty in the Determination of Kinetic Reaction Parameters for Polymeric Materials

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Polymers and composite materials are widely used in the aerospace industry for bonding, insulating and sealing. In many applications, these materials are exposed to high heating rates, which can cause material degradation. Since material properties can change with degradation, the decomposition kinetics must be investigated over a range of heating regimes[1,2].

This paper will discuss the use of Thermogravimetric Analysis (TGA) [3,4] to obtain data describing the thermal response of a polymeric resin with temperature. This data can then be used to obtain activation energy and pre-exponential factor used in an Arrhenius representation of material ablation. With any type of experimental apparatus, some error is inherent in the process. This investigation will employ the methods presented in Reference 5 to assess the uncertainties associated with the experimental determination of activation energy and pre-exponential factor.

A preliminary investigation of the major sources of error in TGA has been performed [6]. Seven samples of resin were run at 20°C/min. TGA results are shown in Figures 1 and 2. Each sample was held initially at 100°C for 10 minutes to eliminate moisture.

Figure 1 is the most general output from the TGA, a thermogram which plots percent of material remaining versus time or temperature. Each region of slope change (indicated as A, B, and C) on the curve corresponds to an area of rapid weight loss. Usually, these areas represent different reactions taking place in the material as it is heated. By taking the derivative of the curve as shown in Figure 2, the different reactions can be seen more clearly.

The reaction that causes the largest weight loss in the sample was chosen for further examination to assess repeatability. The parameters of peak temperature and Delta Y were evaluated as a means of determining preliminary variations in the data. Peak temperature (figure 3) is the temperature at which the maximum weight loss occurs for a particular reaction. Delta Y (Figure 4) is the percent of weight loss between two temperatures enclosing the primary reaction. Figures 3 and 4 are thermograms of the trials that most closely represent the average values of the parameters peak temperature and Delta Y respectively. Table 1 presents the results of this analysis for all seven samples.

TABLE 1
SC1008 RESIN
SEVEN TRIALS AT 20°C/MIN
ARGON ATMOSPHERE

Sample No.	TGA Wt. (mg)	TGA Wt. After Hold (mg)	% Wt. Drop After Hold	Delta Y (%)	Peak Temp. °C
1	9.178	9.016	1.8	12.8	507.2
2	9.341	9.127	2.3	15.2	485.0
3	9.15	9.007	1.6	12.5	504.2
4	9.071	8.911	1.8	16.3	517.3
5	8.803	8.651	1.7	14.6	511.2
6	9.102	8.941	1.8	13.4	507.0
7	8.951	8.798	1.7	14.4	509.3

The mean of the peak temperature and Delta Y were calculated to be 505.9°C and 14.2% respectively. These values were used to calculate the 95% confidence interval for a Gaussian distribution about the mean for peak temperature and delta Y. This 95% confidence interval is referred to as the precision limit [5], given by

$$P_x = tS_x \qquad (1)$$

where P_x = precision limit
t = distribution corresponding to number of degrees of freedom
S_x = precision index.

Based on this calculation, the peak temperature precision limit is within +/- 24.7°C(5% of mean) of the mean. The delta Y precision limit is within +/- 3.3(23% of mean) of the mean. This analysis does not account for systematic errors.

Anticipated Results

The investigation of this paper will discuss the analysis of a larger data set consisting of 10 trials of polymeric resin SC1008 at 4 different heating rates. A repeatability assessment will be performed for each data set. This data will then be used to determine the resultant kinetic reaction parameters. This will include a detailed evaluation of the propagation of error in the TGA data base through the determination of kinetic reaction parameters.

References

1. Flynn, J. H. and Wall, L. A. "General Treatment of the Thermogravimetry of Polymers", Journal of Research, National Bureau of Standards, August 1966 pp. 487-523.

2. Doyle, C. D. "Kinetic Analysis of Thermogravimetric Data", Journal of Applied Polymer Science, Volume V Issue No. 15, pp. 285-292, 1961.

3. Dodd, J. W. and Tonge, K. H., "Thermal Methods", John Wiley and Sons 1987.

4. Perkin Elmer Corporation, "TGA 7 Thermogravimetric Analyzer Operator's Manual", November 1991.

5. Coleman, H. W. and Steele, W. G., " Experimentation and Uncertainty Analysis for Engineers", John Wiley and Sons, 1989.

6. Darby, S. P. and Landrum, D. B., "Experimental Uncertainty In Determining Kinetic Reaction Parameters for Polymeric Materials", 6th AIAA/ASME Joint Thermophysics and Heat Transfer Conference, June 1994.

Figure 1

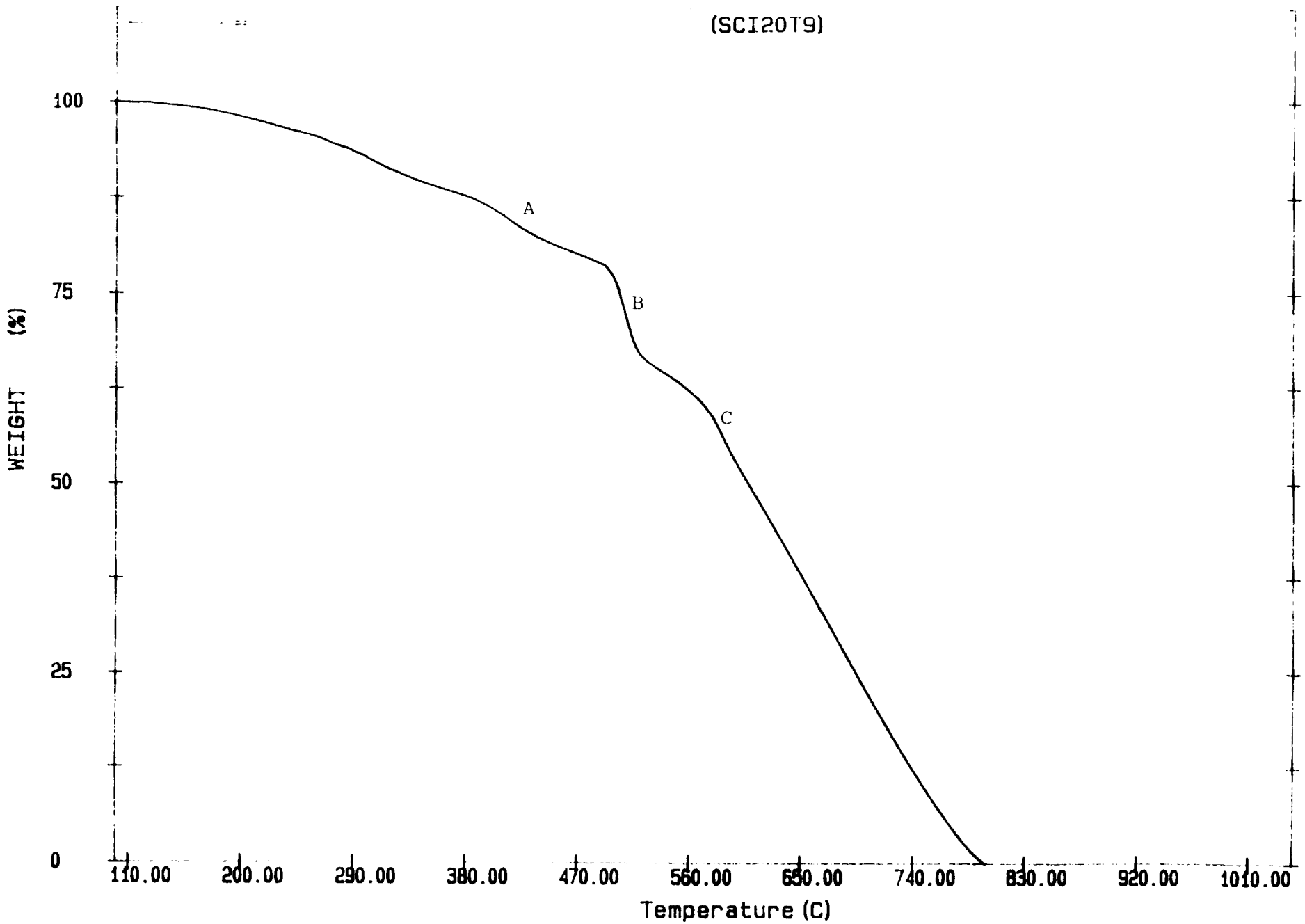

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Figure 2

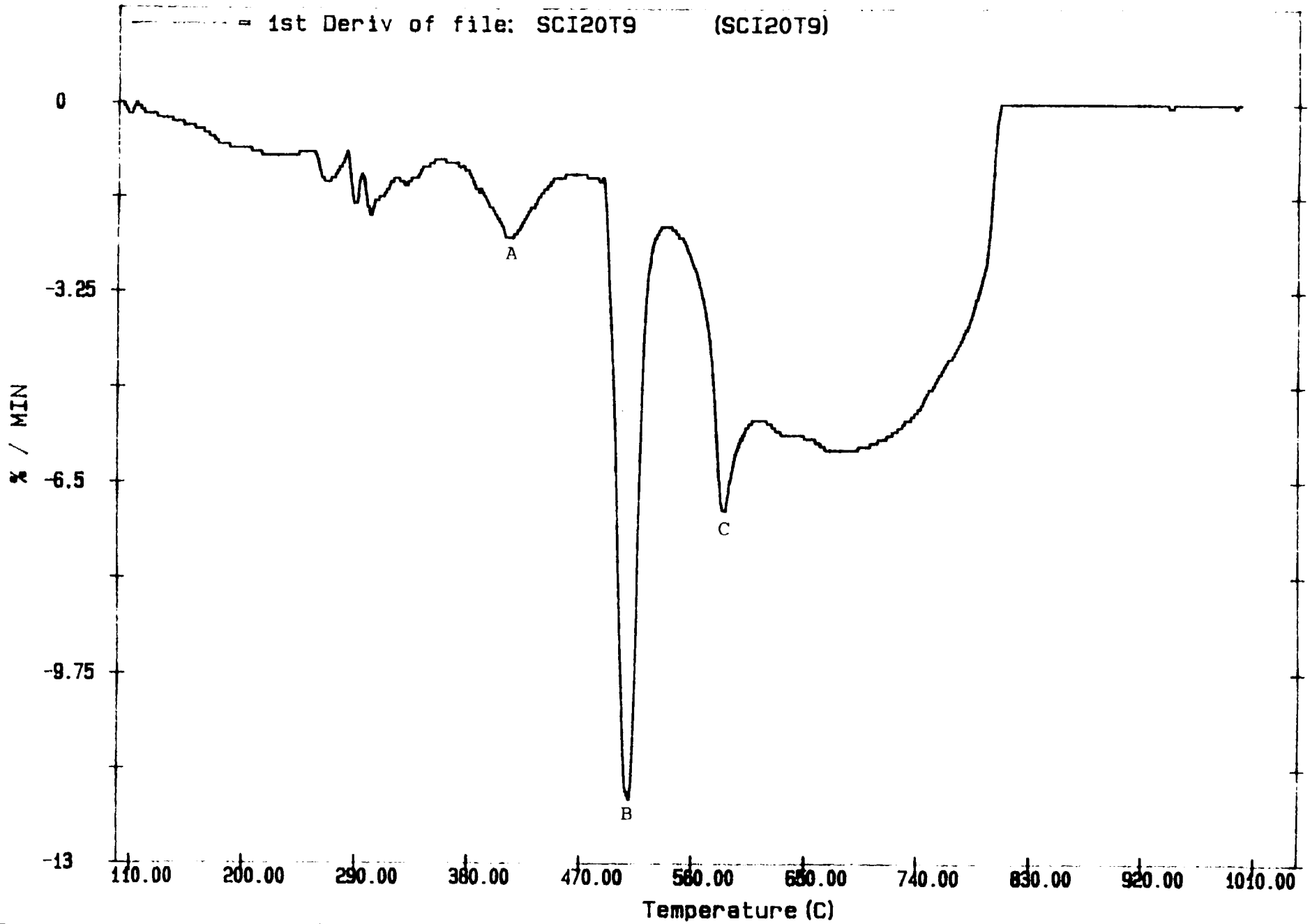

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Figure 3

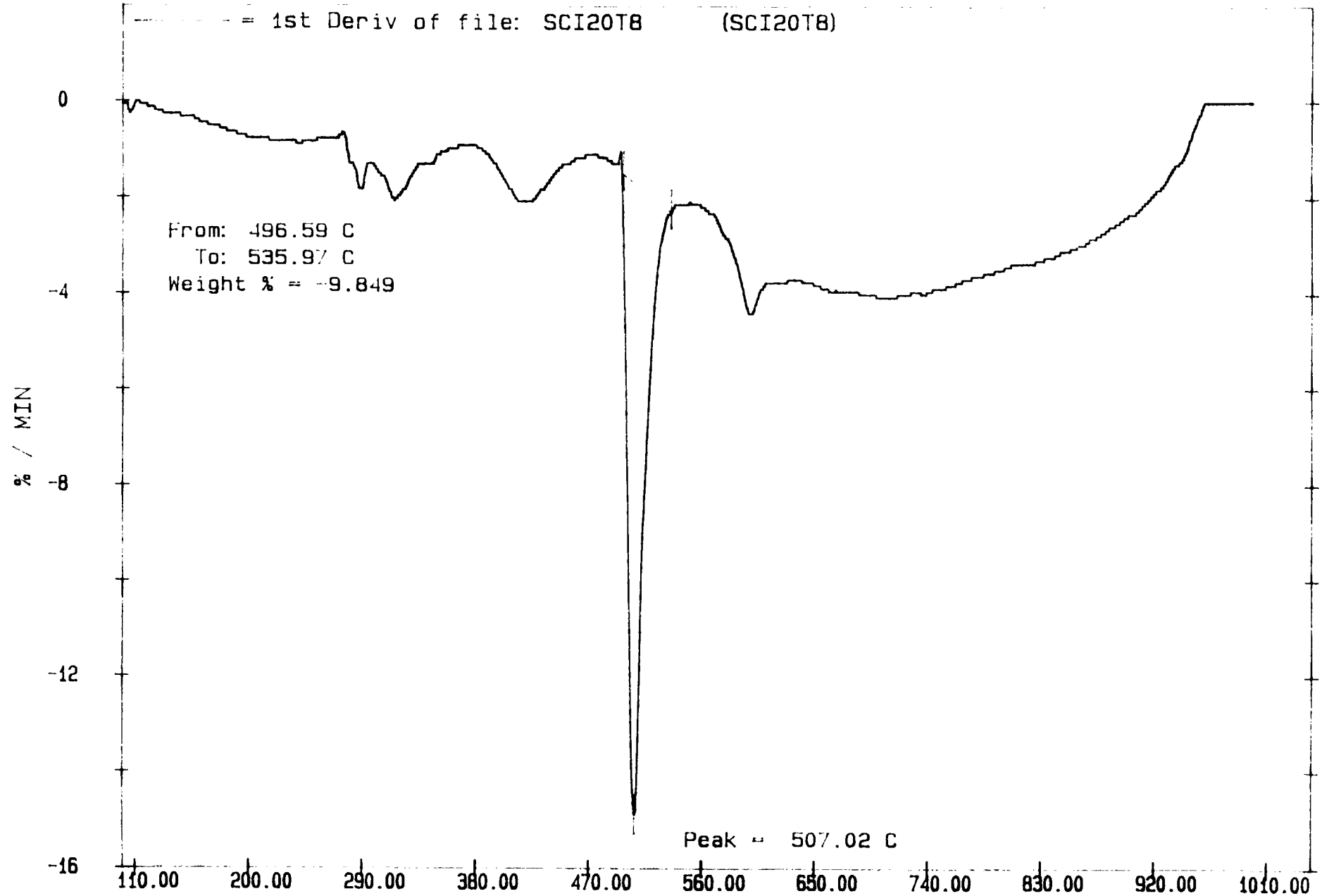

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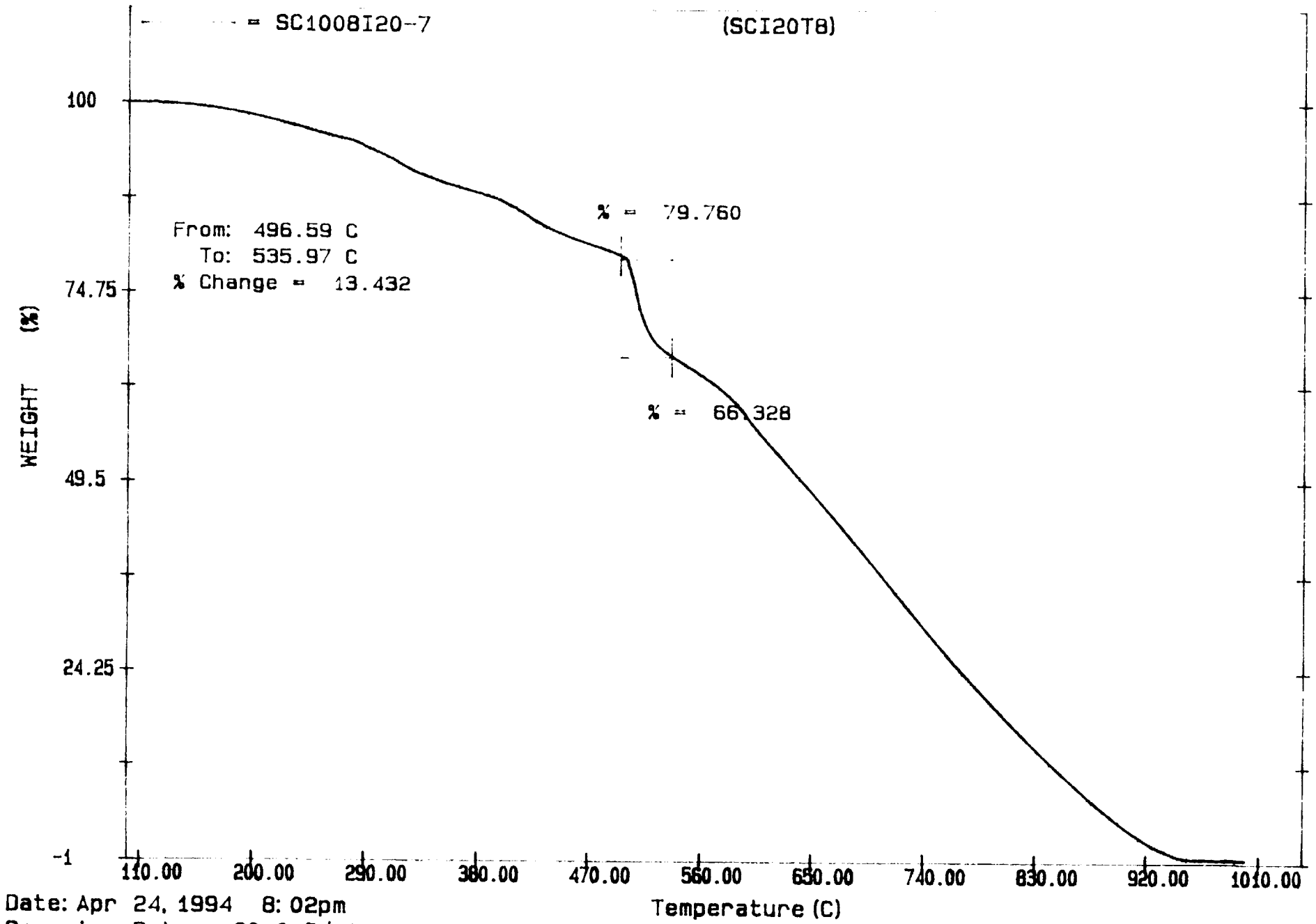
Figure 4


SC1008I20-7
(SCI20T8)
100
74.75
49.5
24.25
-1
WEIGHT (%)
From: 496.59 C
To: 535.97 C
% Change = 13.432
% = 79.760
% = 66.328
110.00
200.00
290.00
380.00
470.00
560.00
650.00
740.00
830.00
920.00
1010.00
Temperature (C)
Date: Apr 24, 1994 8:02pm
Scanning Rate: 20.0 C/min
Sample Wt: 8.941 mg Path: C:\PE\
File: SCI20T8 S.DARBY
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