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EFFECTIVE IGNITION KINETICS FOR LOVA PROPELLANT

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I. BACKGROUND

The most probable mechanism for initiating on-board ammunition stores in armored vehicles attacked by shaped charges or kinetic energy penetrators is thought to be the ignition of propellant by hot spall fragments. Substantial reduction in the sensitivity to hot fragment ignition has been demonstrated for certain nitramine formulations known as LOVA propellants. The degree of sensitivity for a given oxidizer has also been shown to depend strongly on the type of binder used even though the binder is a relatively minor ingredient. Simple correlations have not been found, however, between binder properties and vulnerability characteristics of the corresponding propellant. In fact, observations of ignition by hot fragments under controlled conditions suggest that it involves a dynamic mix of processes such as phase changes, exothermic decomposition, solid phase heat conduction, liquid phase heat conduction and convection, and radiative heat loss. In order to understand the relative importance of each of these processes, a comprehensive model of the conductive ignition event has been developed by Kuo, et al.¹ This model has extensive input data requirements such as mass densities, emissivities, thermophysical properties, and reaction rates for each phase. The present study is an attempt to idealize and quantify the reactions pertinent to hot fragment conductive ignition.

An effort to identify elementary reactions and measure their reaction rates was ruled out as probably infeasible from a technical standpoint and certainly inappropriate to the scope of the modeling effort and the resources available. Since the principal interest in these reactions is as thermal source (or sink) terms in heat transfer equations, Differential Scanning Calorimetry (DSC) was chosen as a suitable technique for measuring reactive heat exchange of decomposing propellant in contact with a metal surface. Operated in ramp mode, this instrument will increase the sample temperature linearly with time at rates up to 100°C/min, measuring the heat absorbed or liberated to maintain the ramp temperature. Although ignition by hot fragments involves much higher instantaneous heating rates, these high rates cannot be sustained for any appreciable time due to conductive and convective loss mechanisms. Observations of hot fragment ignition under controlled conditions indicate it takes some 10 s or more to establish ignition, thus the 100°C/min limitation may not be too restrictive.

II. EXPERIMENTAL DETAILS

The propellant used in this study, Lot #1951, has the same formulation as XM-39 (76% RDX, 12% CAB, 7.6% ATEC, 4% NC, and 0.4% EC) except it has a unimodal distribution of RDX particle sizes (about 5 micron average). The test samples were microtomed to a uniform thickness of about 0.4 mm with mass of about 1 mg ($\pm 5\%$) and placed in covered and crimped pans which were perforated in four places with a straight pin. The pan perforations allow for pressure release while retaining the bulk of any decomposition heat resulting from reactions occurring at or very near the propellant surface. Thus, the technique does not distinguish between energetic reactions in the solid, liquid, or gas phase, the goal being only to measure net "localized" energy release (or absorption). Preliminary tests performed in pans with only two perforations gave total exothermic heats about 10% higher than the four hole configuration. This may be due to retention of heat from gas phase reactions that otherwise would have escaped or, alternatively, heterogeneous catalysis

of condensed phase reactions by gaseous products. Increasing the sample mass by a factor of two similarly increased the exothermic heat by about 10%. Since the larger mass would release more gas during decomposition, the effect may be similar to decreasing the ventilation hole area. No differences were observed for samples of different thickness but same mass, so the sample is probably small enough to achieve adequate thermal equilibration in the instrument.

Experiments were performed in a Dupont Model 910 DSC with Model 1090 controller with the pressure release valve fully open to insure one atmosphere operation. A nitrogen purge flow was maintained through the cell at rates of 10, 50, and 150 cc/min as measured by a rotameter. The rotameter was calibrated using a soap-film bubblemeter and stopwatch. Data was taken for heating rates of 10, 20, 40, and 100°C/min.

The absolute heat flow measured in the DSC is calibrated by adjusting the cell constant, which in turn is measured by using the DSC to determine the heat of fusion of a standard substance. The temperature range pertinent to the propellant thermograms is roughly from 180-300°C. Tin was chosen as the standard in this work as its thermogram spans the range 230-260°C. Thus calibrated, the DSC was used to measure the exotherm for RDX at 8 °C/min with a purge gas flow rate of 200 cc/min. A value of 602 cal/g was obtained which agrees well with the value 613 cal/g reported by P.G. Hall² under the same conditions.

III. ANALYSIS

The DSC thermograms for the LOVA propellant used here typically exhibit an endotherm of about 20 cal/g starting at about 185°C followed by an exotherm of about 300 cal/g which peaks at about 260°C. At the outset of this study the intention was to treat the exotherm as a simple global reaction and the endotherm as a phase change. Ultimately, both were described as separate single reactions, each with its own set of kinetic parameters. Thus, the heart of the analysis is the determination of a set of global kinetics parameters which best represents a thermogram feature (such as the exotherm). The energy released in the exotherm is idealized as resulting from a single hypothetical first-order reaction. It is not essential to assume first order, but it turns out to be close to optimal and simplifies the discussion here. If m is the reactant mass at any given time, then

$$-(dm/dt) = k(T) m(t) , \quad \text{where } k(T) = A \exp(-E/RT). \quad (1)$$

A is the Arrhenius preexponential factor, E the activation energy, and R the universal gas constant. The temperature of the sample in the DSC, operated in ramp mode, is governed by

$$T(t) = T_0 + rt \quad (2)$$

where T_0 is the starting temperature, r the selected heating rate, and t the time elapsed after commencing the ramp. For this relationship Eq. (1) may be integrated to give

$$m = m_0 \exp \left[-\frac{A}{r} \int_{T_0}^T \exp(-E/RT') dT' \right] \quad (3)$$

where m_0 is the initial reactant mass. A linear relationship is assumed between the cumulative heat released in this reaction up to any given time, q , and the mass that has reacted, $(m_0 - m)$ up to that time, i.e., $q = Q (m_0 - m)$, where Q is the mass specific total heat release (in cal/g) for an individual run. Q is determined for a given run by integrating the instantaneous power measurement over the entire temperature interval. Thus, the instantaneous power, $p = (dq/dt)$, measured in the DSC is related to the kinetics by

$$\begin{aligned} p &= Q (-dm/dt) \\ &= Q m A \exp(-E/RT) \end{aligned} \quad (4)$$

where m is given by Eq. (3).

In this work the kinetic parameters were first determined by a nonlinear least squares fit of Eq. (4) to DSC exotherm data. This procedure was applied to each exotherm in order to ascertain if the kinetic parameters varied with heating rate or purge flow rate. The best single set of parameters was obtained by fitting Eq. (4) to a composite of all 36 exotherm data sets.

Having also determined the best single set of parameters for the endotherm by the same procedure, the two reactions were coupled by assuming that the products of the endothermic reaction are the reactants for the exothermic reaction. Thus, LOVA propellant decomposition is viewed as a two-step reaction sequence $A \rightarrow B \rightarrow C$, where A is the unreacted propellant, B is the product of the endothermic reaction, and C is the product of the exothermic reaction. Observation of the decomposition under a temperature-ramped hot-stage microscope suggests that one can associate A with the solid phase, B with a liquid, and C with a gas. One describes this sequence by the coupled rate equations

$$(dm_A/dt) = -k_1 m_A$$

$$(dm_B/dt) = -k_2 m_B + k_1 m_A$$

where k_1 and k_2 are the conversion rates for $A \rightarrow B$ and $B \rightarrow C$, respectively. For a given temperature ramp these equations have the solutions:

$$m_A = m_0 \exp \left\{ - \int_{T_0}^T [k_1(T')/r] dT' \right\} \quad (5)$$

$$m_B = m_0 \exp \left\{ - \int_{T_0}^T [k_2(T')/r] dT' \right\} II \quad (6)$$

where

$$II = \int_{T_0}^T [k_1(T')/r] \exp \left\{ - \int_{T_0}^{T'} [k_1(T'')/r] dT'' \right\} \exp \left\{ \int_{T_0}^{T'} [k_2(T'')/r] dT'' \right\} dT'$$

and the power generated by the sample is given by

$$p = Q_1 (dm_A/dt) - Q_2 (dm_B/dt) \quad (7)$$

Eq. (7) describes the complete thermogram in terms of the two-step reaction kinetics parameters.

Thus, using Eq. (4) as the fitting function, the kinetics parameters for the exotherm (or endotherm) can be determined by a nonlinear least squares procedure applied to data from a single DSC run or to a composite data set consisting of a number of separate runs. The former strategy reveals if the kinetics are varying with heating rate or purge flow rate, and the latter gives the best single set of kinetics to represent all of the runs. The best kinetics sets for the endotherm and exotherm can then be used in Eq. (7) for comparison of the formal two-step reaction model to the complete DSC thermograms.

IV. RESULTS

Table 1 summarizes the kinetics parameters obtained for each individual exotherm as a function of heating rate and purge flow rate. Three runs were done at each set of conditions. No statistically significant variation of either specific heat release or activation energy with heating rate or gas flow is evident. Analyses of some runs were also done with the reaction order as a fitting parameter. Values ranged from 0.8 to 1.5 and, as the quality of fit was not greatly improved, it was decided that a uniform value of 1.0 would be used so that activation energies could be compared directly for different runs. The average activation energy for all these separate exotherm runs is 45 kcal/mole.

Since the kinetics do not vary significantly with heating or flow rate, it was decided that the best single set of kinetics parameters to describe all of the data sets would be obtained by doing the least squares analysis on a composite data set composed of all the data from the individual runs. To eliminate biases due to different heating rates and sample masses, the fitting function was taken to be Eq. (4) divided by m_0 with Q replaced by the average reaction heat reported in Table 1, and each point weighted by $(1/rQ)$. The results of the fit for both exotherm and endotherm are given in Table 2. During this phase of the work a slight trend in preexponential factor with heating rate was noted for the exotherm. Expressing this dependence as

$$A = a r^b$$

in the fitting function, the parameters a and b were obtained simultaneously with the activation energy. These parameterized kinetics decrease the standard deviation of the fit by almost a factor of 2 at the expense of a slightly more complex kinetics expression requiring the instantaneous local heating rate. The same analysis on the endotherm resulted in a negligible decrease in standard deviation of the fit.

Substituting these kinetics values into Eq. (7) enables one to see how well the two-step kinetics model describes the entire DSC thermogram. Figures 1-4 show the comparison for both the constant and heating-rate-dependent preexponential factor (for the exotherm kinetics). The reproducibility of the DSC thermograms at each heating rate is also evident in these figures. The double peak structure in the endotherms, which disappears at high heating rates, is evidence of the oversimplification of the chemistry afforded by the irreversible two-step idealization. However, the quality of representation,

from the standpoint of its use in a heat transfer model, is considered to be quite good. It is also possible to use Eq. (7) directly as the fitting function in the least squares analysis although the computing time is increased substantially. No significant changes in the kinetics parameters resulted from this extended computation, adding increased confidence to the values given in Table 2.

Table 1. Summary of Effective Kinetics Describing DSC Exotherm for LOVA #1951

GAS FLOW (CC/MIN)	HEATING RATE (DEG C/MIN)															
	10				20				40				100			
	RUN	Q	E	A	RUN	Q	E	A	RUN	Q	E	A	RUN	Q	E	A
10	40	272	42.5	1.72E16	37	350	46.2	6.19E17	34	356	47.5	1.57E18	69	303	44.2	2.70E16
	41	286	43.0	2.59E16	38	326	48.2	3.64E18	35	329	44.5	8.36E16	70	316	44.8	6.63E16
	42	291	42.3	1.26E16	39	284	46.7	8.34E17	36	309	47.2	1.07E18	71	302	45.6	1.38E17
	AVG ±	283 10	42.6 .4		AVG ±	320 33	47.0 1.0		AVG ±	331 24	46.4 1.7		AVG ±	307 8	44.9 0.7	
50	43	285	45.7	4.19E17	46	259	47.7	2.52E18	49	325	48.2	2.86E18	66	283	45.9	1.39E17
	44	243	39.1	6.79E14	47	280	48.6	5.37E18	50	317	46.4	5.25E17	67	286	44.7	4.98E16
	45	282	43.7	5.68E16	48	258	46.8	9.10E17	51	288	46.7	6.45E17	68	281	45.7	1.30E17
	AVG ±	270 23	42.8 3.4		AVG ±	266 12	47.7 .9		AVG ±	310 19	47.1 1.0		AVG ±	283 3	45.4 0.6	
150	52	275	40.3	1.89E15	55	290	50.7	4.09E19	58	293	49.4	7.55E18	63	330	43.9	2.55E16
	53	265	46.5	9.52E17	56	300	48.1	3.40E18	59	322	48.0	2.36E18	64	311	42.7	7.84E15
	54	276	38.6	3.76E14	57	325	43.8	4.64E16	61	294	45.6	2.10E17	65	290	44.4	3.84E16
	AVG ±	272 6	41.8 4.2		AVG ±	305 18	47.5 3.5		AVG ±	303 16	47.7 1.9		AVG ±	310 20	43.7 0.9	

Notes:

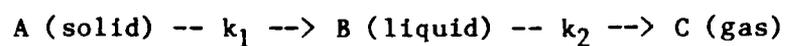
Q in cal/g
E in kcal/mole
A in s⁻¹

GLOBAL AVERAGES: Q = 297 ± 26
E = 45.4 ± 2.7
A = 1.89E17 (antilog of the average of ln A)
ln A = 39.78 ± 2.66

V. CONCLUSIONS

A quantitative description for the reactive heat release of nitramine propellant in contact with a hot metallic surface at atmospheric pressure has been developed for use in a theoretical model of conductive ignition. The description consists of a two-step sequence of global reactions in which the products of an endothermic reaction are the reactants of an exothermic reaction. Kinetic parameters are determined by a nonlinear least squares analysis of an extensive set of DSC measurements conducted over a wide range of heating rates and purge flow rates. The exotherm is best described using a heating-rate-dependent preexponential factor in the Arrhenius expression. Since the local heating rate may not be conveniently available in the conductive ignition model, the best constant preexponential factor is also given.

Table 2. Reaction Scheme and Kinetics Parameters



$$k_i = A_i \exp(-E_i/RT)$$

$$A_i = \text{constant or } A_i \text{ (s}^{-1}\text{)} = a_i r^{b_i} \text{ (r in deg C/s)}$$

REACTION i	Q _i (cal/g)	A _i (s ⁻¹)	a _i	b _i	E _i (kcal/mole)
1	-21.1 ±5.6	1.31E31 ±0.57E31	--	--	69.4 ±0.4
2	297 ±26	1.98E14 ±0.80E14	--	--	38.2 ±0.4
2	297 ±26	---	7.79E17 ±2.89E17	-0.516 ±0.014	47.2 ±0.4

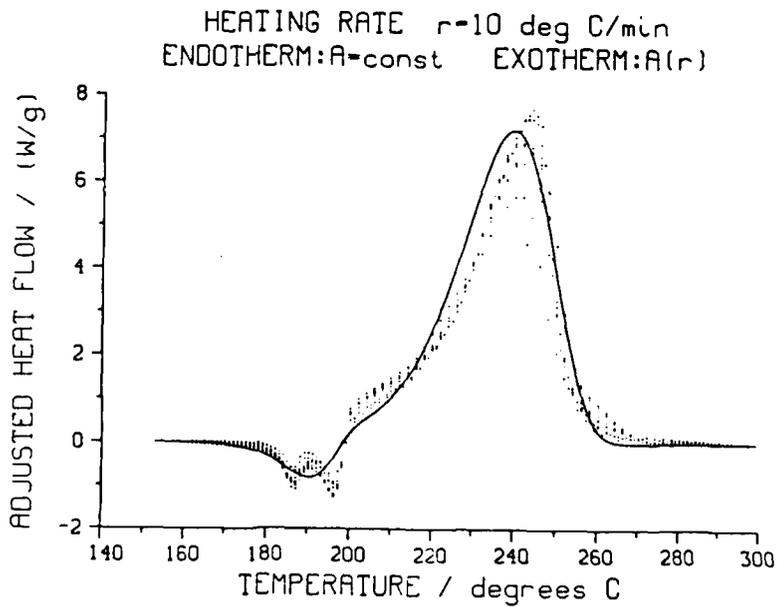
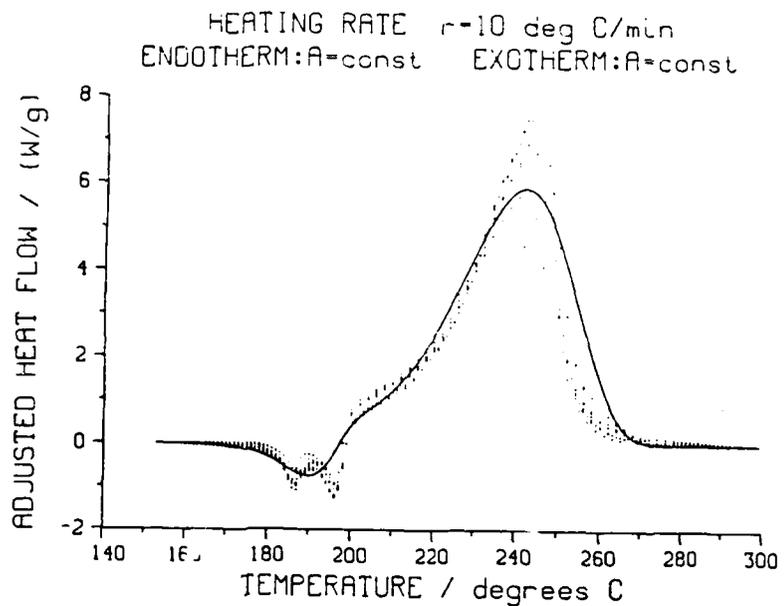


Figure 1. Comparison of Composite DSC Thermograms (Points) to Both Forms of the Two-Reaction Model (Lines) at 10°C/min.
 1a: Constant exotherm A factor. 1b: Heating rate dependent A factor.

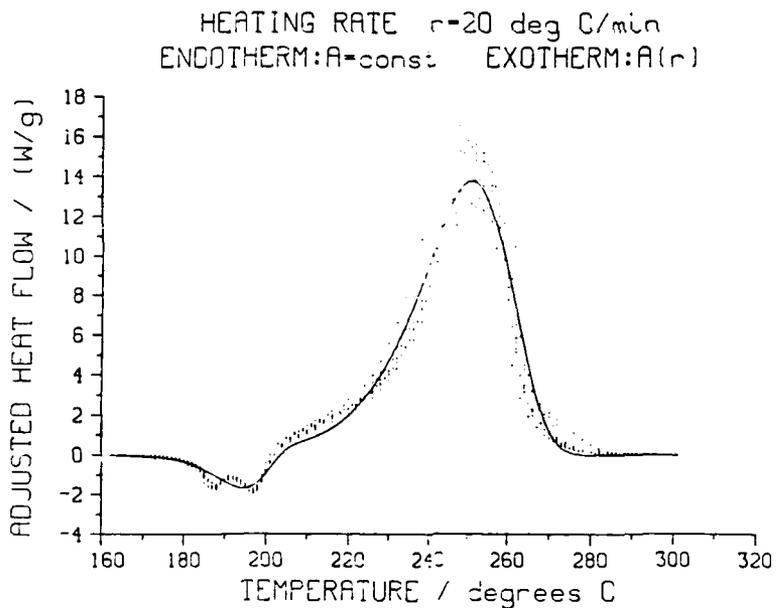
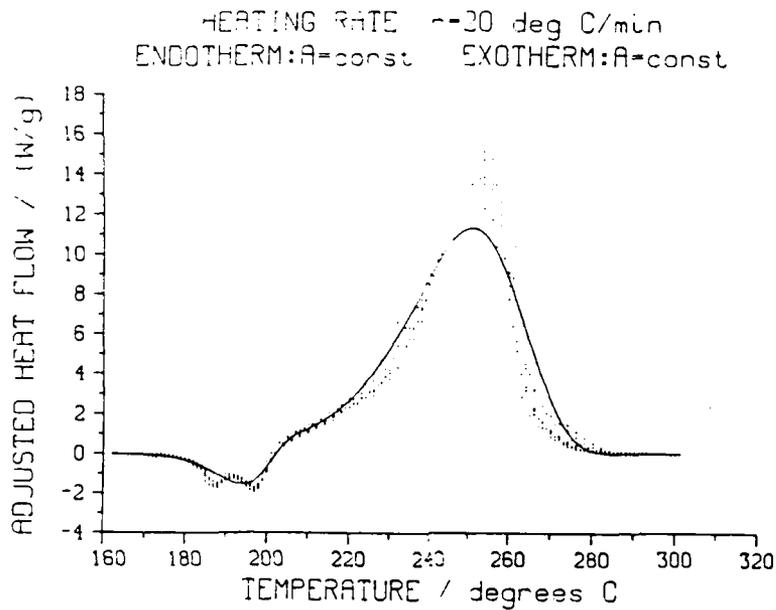


Figure 2. Comparison of Composite DSC Thermograms (Points) to Both Forms of the Two-Reaction Model (Lines) at 20°C/min.
 1a: Constant exotherm A factor. 1b: Heating rate dependent A factor.

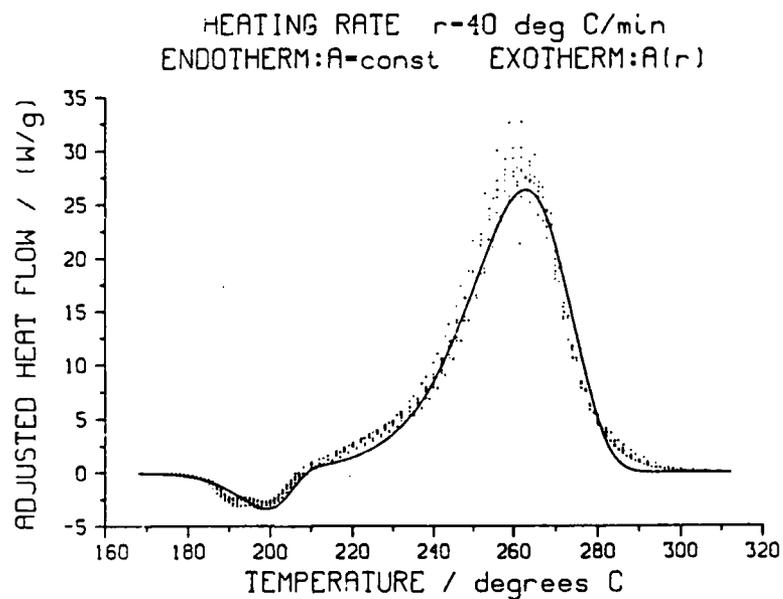
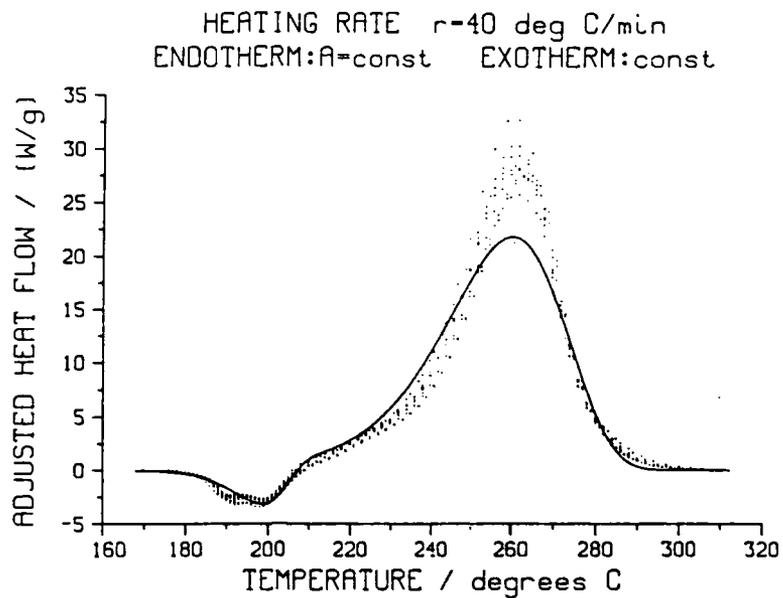


Figure 3. Comparison of Composite DSC Thermograms (Points) to Both Forms of the Two-Reaction Model (Lines) at $40^{\circ}\text{C}/\text{min}$.
 1a: Constant exotherm A factor. 1b: Heating rate dependent A factor.

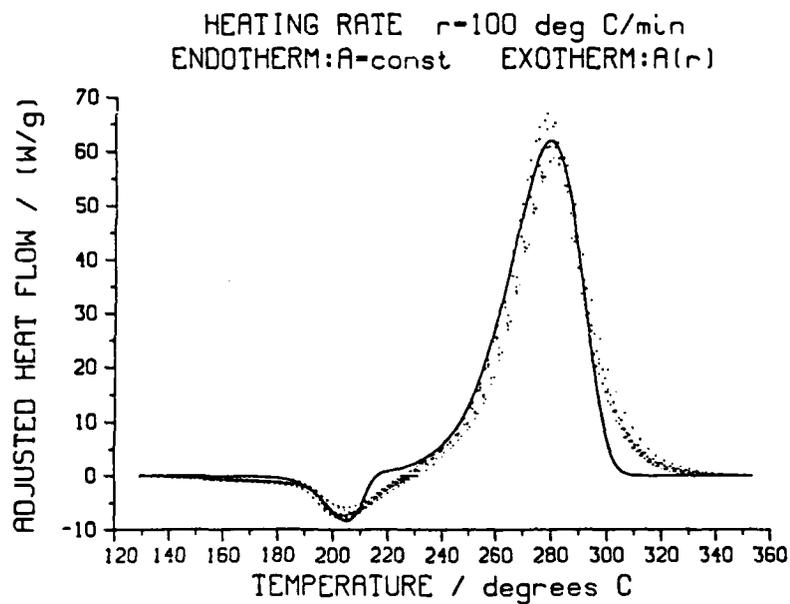
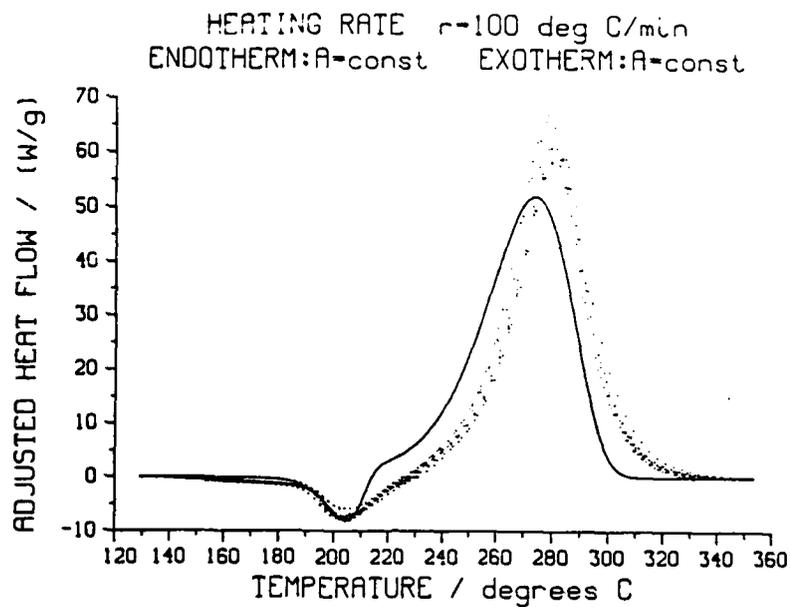


Figure 4. Comparison of Composite DSC Thermograms (Points) to Both Forms of the Two-Reaction Model (Lines) at $100^{\circ}\text{C}/\text{min}$.

1a: Constant exotherm A factor. 1b: Heating rate dependent A factor.

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