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DETERMINATION OF THE THERMAL DECOMPOSITION KINETICS OF POLYURETHANE FOAM BY GUGGENHEIM'S METHOD

Leon J. Decker
J. Richard Ward

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The kinetics of the thermal degradation of a rigid polyurethane foam used to reduce gun wear was determined to illustrate how Guggenheim's method could be applied to polymer decomposition. The polyurethane foam decomposed in two distinct steps. The first order activation parameters for each rate coefficient are $k_1 = 2.0\text{x}10^{10}\text{s}^{-1}\text{EXP}(-134\text{kJ/mole/RT})$ and

$$k_2 = 1.2\text{x}10^{10}\text{s}^{-1}\text{EXP}(-154\text{kJ/mole/RT}).$$

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I. INTRODUCTION

The thermal decomposition rate of polyurethanes has long been of interest to polymer chemists in their endeavor to reduce the flammability of these widely used materials. A recent thesis reviews this history.[1]

Thermal decomposition of polyurethanes is also of interest in interior ballistics. Polyurethane mitigates barrel wear when a high-density polyurethane foam is glued to the inside wall of cartridge cases.[2,3] More recently propellants with polyurethane binders have demonstrated reduced sensitivity to accidental or uncontrolled ignition.[4] Wise has suggested the thermal decomposition of the binder governs the decreased vulnerability of the propellant.[5]

A common technique to measure the kinetics of the thermal decomposition is to monitor mass loss vs time at constant temperature ("isothermal"), or mass loss vs temperature at a constant heating rate ("dynamic"). The dynamic technique is experimentally convenient, since activation parameters can be determined in a single experiment. The isothermal technique, by contrast, requires rate coefficient determinations at several temperatures; the time for a reaction to go to completion is much longer than in a dynamic run; and the rate coefficient is sensitive to the value of mass selected at the end of the reaction. For polymers this choice can be arbitrary, since polymers typically decompose by consecutive reactions.

The experimental simplicity of the dynamic method prompted many investigators to apply this technique to polymer decomposition kinetics.[6]

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[1] *M.S. Ramakrishman, "Pyrolysis and Thermal Degradation of Rigid-Urethane Foams," Dept of Chemical Engineering, University of Utah, December 1975.*

[2] *L.A. Dickinson and D.E. McLennan, "Improvement of the Firing Accuracy and Cost Effectiveness of Guns Through the Use of Urethane Foams," J. Cellular Plastics, 1968, p 184.*

[3] *W. Joseph, "Use of Foamed Polyurethane in Decreasing Erosion," Picatinny Arsenal Technical Report No. 2520, June 1958.*

[4] *J.J. Rocchio and R.W. Deas, "Interior Ballistics of Nitramine - Inert Binder Formulations Being Evaluated for Low Vulnerability Propellants," 15th JANNAF Combustion Meeting Vol I, CPIA Publication 297, February 1979.*

[5] *S. Wise, BRL Report in preparation.*

[6] *J.H. Flynn, "The Historical Development of Applied Nonisothermal Kinetics", Thermal Analysis ed, R.F. Schmenker and P.D. Garn Vol 2, p 1111 (1969).*

A number of investigators have questioned the validity of the dynamic method,[7-12] since rate coefficients determined isothermally did not agree with those determined with the dynamic method. MacCallum and Taylor go so far as to question the validity of the equations used in the dynamic method.[8]

In this report, the decomposition of a polyurethane is measured using Guggenheim's[13] method to evaluate the first-order rate coefficients. Guggenheim's technique seems particularly suited, since most polymers decompose by first-order kinetics[14,15] under an inert atmosphere, but the exact mass loss corresponding to the end of the reaction is difficult to discern for consecutive reactions.

II. EXPERIMENTAL

Samples of polyurethane foam were cut from a piece of foam taken from a 105 mm M392A2 APDS round.[2,3] Polymer ingredients are given in Table 1.

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[7] *J.R. MacCallum and J. Tanner, "A Comparative Study of Some Methods of Assessing Kinetic Parameters from Thermogravimetric Analysis," Euro Polymer J., 6, 907 (1970).*

[8] *J.R. MacCallum and J. Tanner, "Derivation of Rate Equations used in Thermogravimetry," Nature, 225, 1127 (1970).*

[9] *R.A.W. Hill, "Rate Equations in Thermogravimetry," Nature, 227, 703 (1970).*

[10] *R.M. Felder and E.P. Stahel, "Nonisothermal Chemical Kinetics," Nature, 228, (1970).*

[11] *E.L. Simmons and W.W. Wendlandt, "Nonisothermal Rate Equations," Thermochimica Acta, 3, 498 (1972).*

[12] *P.D. Garn, "Nonisothermal Kinetics," J. Thermal Analysis, 6 237 (1974).*

[13] *F. Daniels, Experimental Physical Chemistry, 6th ed. New York, McGraw-Hill Book Co. Inc. 1962, p 140.*

[14] *L.P. Rumao and K.C. Frisch, "Thermal Degradation of Polyurethanes Based on Xylxylene Dusocyamates," J. Polymer Science, A-1, 10, 1499 (1972).*

[15] *E. Dyer and R.J. Hammond, "Thermal Degradation of N-Substituted Polycarbamates," J. Polymer Sci: Part A, 2, 1 (1964).*

TABLE 1. COMPOSITION OF POLYURETHANE FOAM

Resin Prepolymer Ingredients	Parts by Weight
polyethylene glycol 200	10.5
polypropylene glycol 1200	6.5
castor oil	36.5
2,4 toluene diisocyanate	46.5

Catalyst Mixture Ingredients	Parts by Weight*
polypropylene glycol	10.0
glycerine	7.5
polyethylene glycol	3.75
ferric acetylacetonate	0.15
nigrosine black	0.25
dibutyltin dilaurate	0.30

**Remainder of foam is resin.*

Thermogravimetric measurements were made on a DuPont Model 950 thermogravimetric analyzer in a flowing, helium atmosphere (100 ml/min). Procedures for isothermal kinetic runs have been described in an earlier report.[16]

Guggenheim's method may be illustrated with the integrated form of the differential equation for a first-order reaction given below:

$$m_t - m_\infty = (m_o - m_\infty)\, e^{-kt} , \qquad (1)$$

where m_t = mass at time, t,

m_∞ = mass at completion of reaction,

m_o = initial mass,

k = first-order rate coefficient, and

t = time.

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[16] *J.R. Ward, "Kinetics of Talc Dehydroxylation," BRL Memorandum Report No. 2393, June 1974.* (AD #784083)

At time, $t + \Delta t$, equation (1) becomes

$$m_{(t+\Delta t)} = m_\infty + (m_o - m_\infty)\, e^{-k(t+\Delta t)} \ . \tag{2}$$

Subtracting equation (2) from (1) gives

$$m_t - m_{(t+\Delta t)} = (m_o - m_\infty)\, e^{-kt}(1 - e^{-k\Delta t}) \ . \tag{3}$$

Taking logs of both sides of (3) produces

$$\ln(m_t - m_{(t+\Delta t)}) = -kt + \ln((m_o - m_\infty)(1 - e^{-k\Delta t})) \ . \tag{4}$$

A plot of $\ln(m_t - m_{(t+\Delta t)})$ vs t will give a straight line with slope, -k. In the experiments reported here, the readings, R, from the heating curve are used to get the rate coefficient.

III. RESULTS AND DISCUSSION

The polyurethane foam decomposes in two distinct steps with maximum decomposition rates at 558K and 673K. Rate coefficients were determined for each decomposition region by making isothermal TG runs within each region. Appendix A lists the data for the low-temperature region experiments, while Appendix B lists the data for the high-temperature region experiments. In all kinetic runs, the y-axis displacement was 2.0 mg/division. The rate coefficients were determined directly from the y-axis displacements.

Tables 2 and 3 summarize the rate coefficients determined from plots of $\ln(R_t - R_{(t+\Delta t)})$ vs t, where R is the reading from the TG curve.

Figures 1 and 2 illustrate plots of ln k vs 1/T. The activation energy, E_a, and the pre-exponential, A, were determined from a linear least-squares fit of the rate coefficients in Tables 2 and 3 to

$$\ln k = \ln A - E_a/RT \ . \tag{5}$$

TABLE 2. RATE COEFFICIENTS FOR LOW-TEMPERATURE REACTION

Run I.D.	mass,mg	T, K	$\Delta t, s, x10^{-3}$	k, s^{-1}, x 10^{3}
3-12/16	25.83	472	36	0.036
1-12/29	22.80	476	36	.031
1-12/28	20.56	477	36	.033
2-12/16	23.29	504	3.6	.26
1-12/16	24.54	504	3.6	.25
2-12/3	17.77	533	0.42	1.6
3-12/3	18.99	533	.42	1.7
1-12/3	25.68	559	.18	5.8
1-12/2	23.79	560	.18	4.4

TABLE 3. RATE COEFFICIENTS FOR HIGH-TEMPERATURE REACTION

Run ID	mass,mg	T, K	Δt,s,	k,s^{-1},x10^3
1-1/14	21.20	593	3,600	0.28
1-1/13	17.91	593	3,600	.29
1-1/12	15.08	593	3,600	.29
1-1/11	20.57	594	3,600	.29
1-11/23	18.23	617	1,800	.91
1-11/24	20.29	618	1,800	1.0
1-11/26	17.50	636	600	2.3
2-11/26	18.00	643	600	3.2
3-11/26	22.76	670	300	12.3
4-11/26	18.31	670	300	11.8

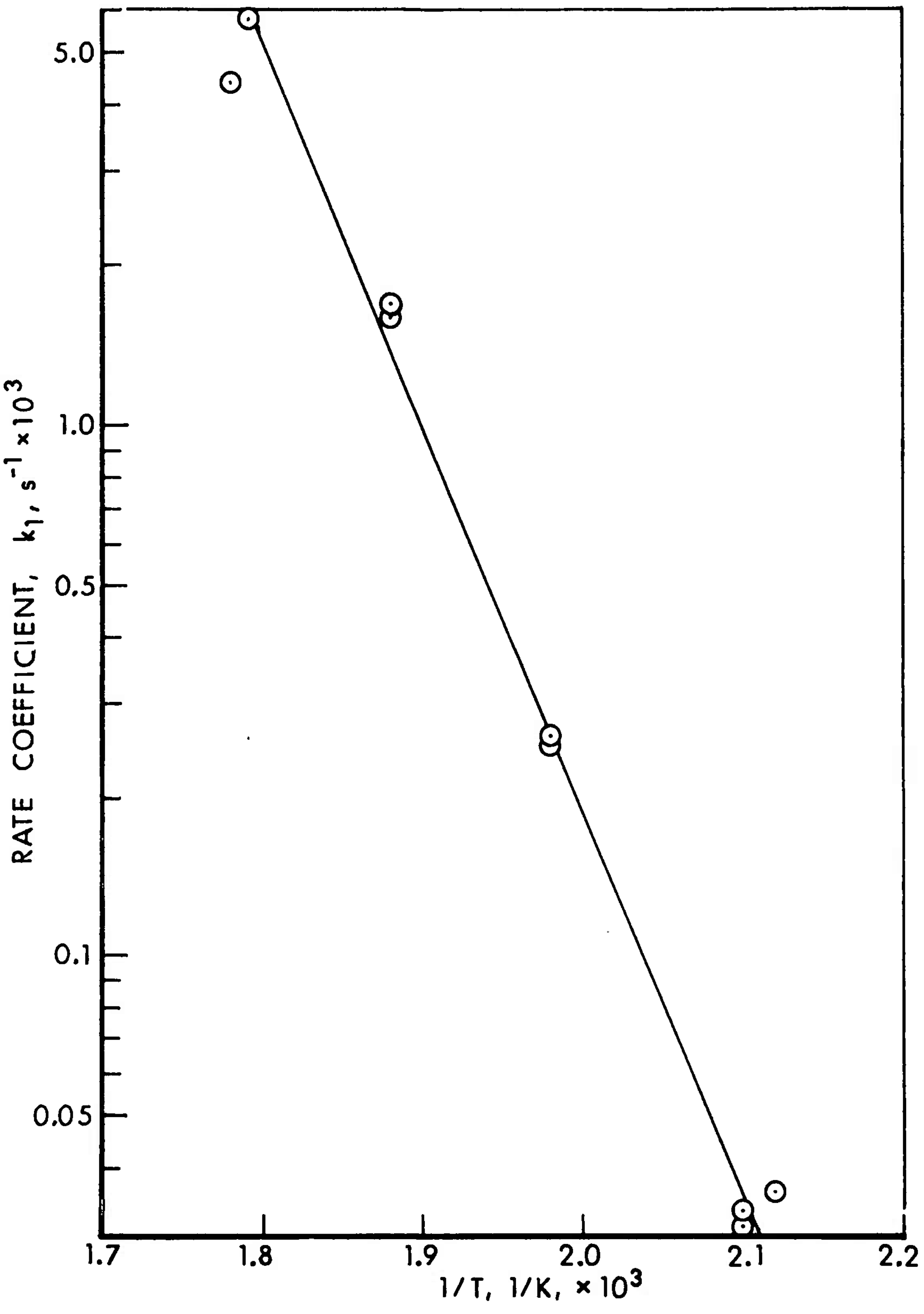

Figure 1. Low-Temperature Rate Coefficient _vs_. Reciprocal Absolute Temperature

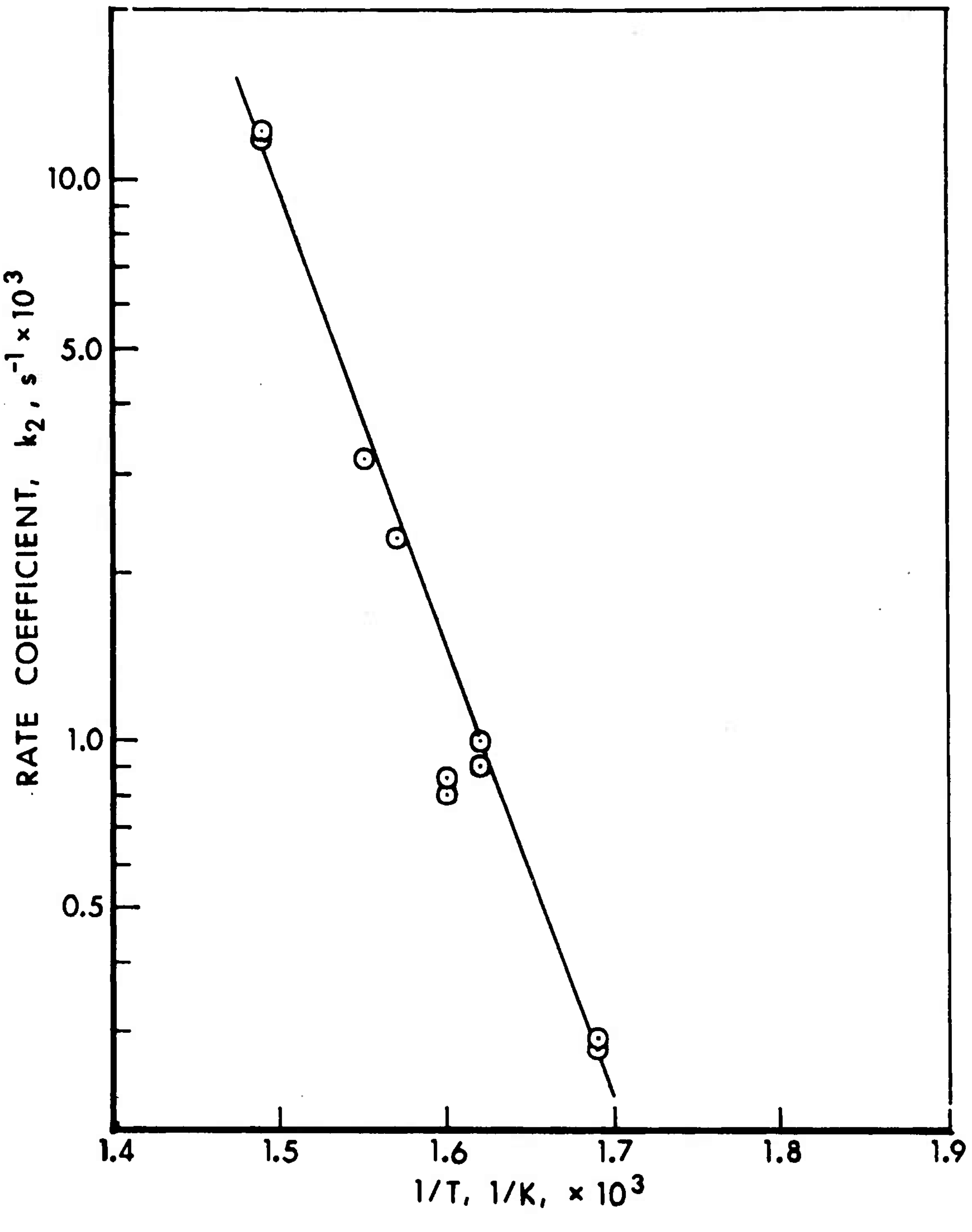

Figure 2. High-Temperature Rate Coefficient *vs.* Reciprocal Absolute Temperature

The results of these calculations are shown below. The error corresponds to the sample standard deviation.

	E_a,kJ/RT/mole	lnA,s^{-1}
k_1	134±5	23.7±1
k_2	154±2	23.2±0.04

IV. CONCLUSION

The kinetics of the thermal degradation of a rigid polyurethane foam used to reduce gun wear was determined to illustrate how Guggenheim's method could be applied to polymer decomposition. The polyurethane foam decomposed in two distinct steps. The first-order activation parameters for each rate coefficient are $k_1 = 2.0 \times 10^{10} s^{-1} EXP(-134kJ/mole/RT)$ and $k_2 = 1.2 \times 10^{10} s^{-1} EXP(-154kJ/mole/RT)$, respectively.

REFERENCES

1. M.S. Ramakrishnan, "Pyrolysis and Thermal Degradation of Rigid-Urethane Foams," Dept of Chemical Engineering, University of Utah, December 1975.

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3. W. Joseph, "Use of Foamed Polyurethane in Decreasing Erosion," Picatinny Arsenal Technical Report No. 2520, June 1958.

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9. R.A.W. Hill, "Rate Equations in Thermogravimetry," Nature, 227, 703 (1970).

10. R.M. Felder and E.P. Stahel, "Nonisothermal Chemical Kinetics," Nature, 228, (1970).

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16. J.R. Ward, "Kinetics of Talc Dehydroxylation," BRL Memorandum Report No. 2393, June 1974. (AD #784083)

APPENDIX A

Readings from Isothermal TG Curve for Low-Temperature Decomposition (1 div = 2 mg)

Run ID	3-12/16
mass, mg	25.83
T, K	472
Δt, min	600

R_t	$R_{(t+\Delta t)}$	t, min
7.42	5.24	50
7.16	5.16	100
6.92	5.09	150
6.66	5.02	200
6.41	4.97	250
6.21	4.91	300
6.01	4.86	350
5.84	4.80	400

Run ID	1-12/29
mass, mg	22.80
T, K	476
Δt, min	600

R_t	$R_{(t+\Delta t)}$	t, min
7.84	5.89	10
7.80	5.87	20
7.73	5.84	35
7.68	5.81	50
7.45	5.74	100
7.25	5.68	150
7.05	5.61	200
6.85	5.55	250
6.67	5.49	300
6.50	5.44	350
6.36	5.39	400
6.22	5.34	450
6.10	5.30	500

Run ID	1-12/28
mass, mg	20.56
T, K	477
Δt, min	600

R_t	$R_{(t+\Delta t)}$	t, min
7.73	5.89	10
7.69	5.88	20
7.62	5.84	35
7.56	5.81	50
7.34	5.74	100
7.15	5.68	150
6.97	5.62	200
6.77	5.58	250
6.60	5.52	300
6.44	5.48	350
6.31	5.43	400
6.19	5.38	450
6.08	5.36	500

Run ID	2-12/16
mass, mg	23.29
T, K	504
Δt, min	60

R_t	$R_{(t+\Delta t)}$	t, min
7.41	5.49	5
7.19	5.39	10
6.98	5.29	15
6.77	5.22	20
6.57	5.14	25
6.39	5.08	30
6.23	5.02	35
6.07	4.97	40
5.93	4.91	45
5.80	4.86	50
5.68	4.81	55
5.59	4.77	60
5.49	4.73	65
5.39	4.69	70

Run ID	1-12/16
mass, mg	24.54
T, K	504
Δt, min	60

R_t	$R_{(t+\Delta t)}$	t, min
7.29	5.19	5
7.05	5.07	10
6.82	4.98	15
6.61	4.90	20
6.39	4.82	25
6.20	4.75	30
6.01	4.68	35
5.85	4.61	40
5.69	4.54	50
5.55	4.48	55
5.41	4.42	60
5.29	4.38	65
5.19	4.33	70

Run ID	2-12/3
mass, mg	17.77
T, K	533
Δt, min	7

R_t	$R_{(t+\Delta t)}$	t, min
8.00	6.77	1.25
7.85	6.55	2.0
7.65	6.42	3.0
7.42	6.30	4.0
7.23	6.20	5.0
7.05	6.09	6.0
6.88	5.99	7.0
6.71	5.91	8.0
6.55	5.84	9.0
6.42	5.77	10.0
6.30	5.71	11.0
6.20	5.66	12.0
6.09	5.61	13.0
5.99	5.56	14.0
5.91	5.51	15.0
5.84	5.47	16.0

Run ID	3-12/3
mass, mg	18.99
T, K	533
Δt, min	7

R_t	$R_{(t+\Delta t)}$	t, min
6.20	7.59	1.0
6.11	7.47	1.5
6.01	7.36	2.0
5.85	7.16	3.0
5.71	6.97	4.0
5.57	6.76	5.0
5.48	6.56	6.0
5.37	6.38	7.0
5.29	6.20	8.0
5.20	6.01	9.0
5.13	5.85	10.0
4.94	5.48	13.0
4.84	5.29	15.0

Run ID	1-12/3
mass, mg	25.68
T, K	559
Δt, min	3

R_t	$R_{(t+\Delta t)}$	t, min
6.39	4.20	2
5.28	3.89	3
4.61	3.66	4
4.20	3.51	5
3.89	3.37	6
3.66	3.24	7

Run ID	1-12/2
mass, mg	23.79
T, K	560
Δt, min	3

R_t	$R_{(t+\Delta t)}$	t, min
7.46	5.10	1.5
7.07	4.86	2.0
6.14	4.45	3.0
5.39	4.15	4.0
4.86	3.90	5.0
4.45	3.70	6.0
4.15	3.55	7.0
3.90	3.41	8.0
3.55	3.30	10.0

APPENDIX B

Readings from Isothermal TG Curve for High-Temperature Decomposition (1 div = 2 mg)

Run ID	1-1/14
mass, mg	21.20
T, K	593
Δt, min	60

R_t	$R_{(t+\Delta t)}$	t, min
3.61	5.07	10
3.50	4.69	20
3.39	4.39	30
3.29	4.15	40
3.20	3.94	50

Run ID	1-1/13
mass, mg	17.91
T, K	593
Δt, min	60

R_t	$R_{(t+\Delta t)}$	t, min
3.65	4.90	10
3.54	4.57	20
3.46	4.31	30
3.37	4.11	40
3.30	3.94	50

Run ID	1-1/12
mass, mg	15.08
T, K	593
Δt, min	60

R_t	$R_{(t+\Delta t)}$	t, min
3.87	2.78	10
3.59	2.70	20
3.38	2.62	30
3.18	2.54	40
3.01	2.48	50

Run Id	1-1/11
mass, mg	20.57
T, K	594
Δt, min	60

R_t	$R_{(t+\Delta t)}$	t, min
5.28	3.74	10
4.89	3.61	20
4.57	3.50	30
4.31	3.40	40
4.10	3.33	50
3.90	3.26	60

Run ID	1-11/23
mass, mg	18.23
T, K	617
Δt, min	30

R_t	$R_{(t+\Delta t)}$	t, min
6.54	2.97	1.0
6.02	2.97	1.2
5.70	2.96	1.4
5.53	2.96	1.6
5.41	2.95	1.8
5.32	2.95	2.0
5.18	2.94	2.5
5.08	2.92	3.0
4.91	2.90	4.0
4.77	2.88	5.0
4.60	2.85	6.0
4.48	2.83	7.0
4.35	2.81	8.0
4.24	2.79	9.0
4.16	2.78	10.0
3.72	2.70	15.0
3.41	2.61	20.0

Run ID	1-11/24
mass, mg	20.29
T, K	618
Δt, min	30

R_t	$R_{(t+\Delta t)}$	t, min
7.20	2.93	1.1
6.90	2.93	1.2
6.65	2.92	1.3
6.45	2.92	1.4
6.17	2.92	1.6
5.96	2.91	1.8
5.83	2.90	2.0
5.46	2.87	3.0
5.26	2.84	4.0
5.05	2.82	5.0
4.89	2.79	6.0
4.60	2.74	8.0
4.36	2.70	10.0
4.09	2.63	12.5
3.81	2.60	15.0
3.49	2.56	20
3.20	2.49	25
2.98	2.42	30
3.82	2.39	35

Run ID	1-1/17
mass, mg	19.80
T, K	623
Δt, min	30

R_t	$R_{(t+\Delta t)}$	t, min
4.67	2.68	5.0
4.34	2.62	7.5
4.06	2.56	10.0
3.81	2.50	12.5
3.60	2.46	15.0
3.43	2.40	17.5
3.27	2.36	20.0
3.13	2.32	22.5
3.01	2.29	25.0
2.82	2.22	30.0

Run ID	1-1/18
mass, mg	15.32
T, K	624
Δt, min	30

R_t	$R_{(t+\Delta t)}$	t, min
3.68	2.11	5
3.18	2.02	10
2.82	1.95	15
2.57	1.88	20
2.37	1.80	25

Run ID	1-11/26
mass, mg	17.50
T, K	636
Δt, min	10

R_t	$R_{(t+\Delta t)}$	t, min
4.23	2.22	2.0
3.57	2.09	4.0
3.07	1.98	6.0
2.71	1.90	8.0
2.43	1.83	10.0
2.02	1.71	15.0
1.83	1.65	20.0
1.71	1.60	25.0
1.65	1.58	30.0

Run ID 2-11/26
mass, mg 18.00
T, K 643
Δt, min 10

R_t	$R_{(t+\Delta t)}$	t, min
5.04	2.37	1.2
4.78	2.33	1.6
4.58	2.30	2.0
4.35	2.27	2.5
4.15	2.23	3.0
3.77	2.18	4.0
3.46	2.13	5.0
3.20	2.10	6.0
2.97	2.07	7.0
2.78	2.04	8.0
2.62	2.02	9.0
2.50	2.00	10.0
2.39	1.99	11.0

Run ID	3-11/26
mass, mg	22.76
T, K	670
Δt, min	5

R_t	t, min	$R_{(t+\Delta t)}$
4.12	1.0	0.56
3.72	1.2	.55
3.37	1.4	.54
3.02	1.6	.53
2.70	1.8	.52
2.41	2.0	.51
1.39	3.0	.47
0.86	4.0	.42
.64	5.0	.37

Run ID	4-11/26
mass, mg	18.31
T, K	670
Δt, min	5

R_t	$R_{(t+\Delta t)}$	Δt, min
4.40	1.52	1.0
4.09	1.51	1.2
3.76	1.50	1.4
3.47	1.49	1.6
3.21	1.48	1.8
2.97	1.48	2.0
2.77	1.47	2.2
2.59	1.46	2.4
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