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A COMPARISON OF TWO TRANSIENT METHODS OF MEASURING THERMAL CONDUCTIVITY OF PARTICULATE SAMPLES

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16. ABSTRACT

A comparison is made of two transient methods of measuring thermal conductivity of particulate media in vacuum. The comparison is based on the following criteria: (1) apparatus required, (2) pretest requirements, (3) data reduction procedures, (4) time comparison, (5) heater voltage effects, and (6) comparison of test results. It is concluded that the Differentiated Line Source method, as developed by Merrill, gives results comparable to the established Line Source method and is much easier and faster (by an order of magnitude) to employ.

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LIST OF ABBREVIATIONS AND SYMBOLS

A, B	constants
DLS	Differentiated Line Source method
$\frac{dT}{dt}$	time differential of temperature
E_i	exponential integral
e_{cm}	difference between initial and maximum voltages in DLS system output
I	heater wire current
K	thermal conductivity
LS	Line Source method
l	heater wire length
n	thermoelectric coefficient
q	heater input power per unit length
RC	differentiating circuit time constant
r	distance between wires
S_1, S_2	amplifier ranges
T	temperature
t	time
t_m	time to reach e_{cm}
$t_{\frac{1}{2}}$	time to reach $\frac{1}{2} e_{cm} = 0.37337\ t_m$
V	heater wire voltage
α	thermal diffusivity

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A COMPARISON OF TWO TRANSIENT METHODS OF MEASURING THERMAL CONDUCTIVITY OF PARTICULATE SAMPLES

SUMMARY

In the continuous line source theory as described by Carslaw and Jaegar [1], the temperature at some point in a homogeneous medium is related to the properties of the medium, the distance from the source of heat, and the time after onset of heater power. If the temperature is measured at a known distance from the source as a function of time, the thermal properties of the medium can be determined. This procedure has been used extensively to investigate the thermal properties of particulate media in vacuum [2, 3]. In an effort to extend the utility of the method, Merrill further developed the theory and instrumentation into a more useful form [4]. A comparison of the former method, Line Source method (LS), and the one developed by Merrill, Differentiated Line Source method (DLS), has been made and the results and conclusions are given herein.

INTRODUCTION

Both the LS and DLS methods are transient ones in which, when certain pretest conditions are met, heat is introduced into a sample with a heater wire, and a thermocouple is used as the temperature sensor. In both, the thermocouple output is related to the thermal conductivity of the sample, but in different ways.

For the comparison tests, the samples were placed in the vacuum chamber and were not changed or disturbed between the two series. The differences in procedure lie in the handling of the signal and data outside the sample chamber. All measuring instrumentation is common to both methods, with the exception of the added circuitry of the DLS. Since the sample is the same in both cases, this means, of course, that theoretically the conductivities measured should be identical.

The criteria upon which this comparison is based are the following: (1) apparatus required, (2) pretest conditions, (3) data reduction, (4) time comparison, (5) heater voltage effects, and (6) consistency of results.

APPARATUS REQUIRED

The apparatus required by the LS is shown in Figure 1. The sample is placed in a copper sample holder which is in a vacuum chamber. The sample holder has a network of channels in its walls so temperature control fluids can be circulated to control the initial temperature of the sample. The heater source and detector wires are constantan and iron-constantan and are 0.025 mm in diameter. A test is initiated by supplying voltage to the heater wire from the power supply. The voltage and current are monitored with the meters and are recorded. The rise in thermocouple voltage is detected and recorded on the strip chart recorder. The K-3 potentiometer is used to read initial and final temperatures. The data reduction procedure will be described later.

Figure 2 shows the circuitry for the DLS. It is apparent that the apparatus is the same except for the addition of another amplifier and an RC differentiating circuit which has a 6.8-msec time constant. Instead of printing out thermocouple voltage, the recorder prints the time differential of the voltage increase, which is directly related to the time differential of the temperature rise.

PRETEST CONDITIONS

The theory of both methods is based on certain assumptions or ideal conditions which can be only approximated. Both methods assume an infinitely large, homogeneous and isotropic sample, and heater and thermocouple wires of an infinitely large length to diameter ratio. The validity of the results are determined by how well these conditions are approached. However, for the purposes of this comparison, since the same sample and wires are used in both cases, the errors should be similar. (There may be slight differences since, for example, effects such as axial heat losses in the wires do not act over the same time periods in the two test methods.)

A critical pretest condition that differs between the methods concerns initial sample temperature control. In the LS, the sample temperature must be constant before initiation of a test. If this condition is not satisfied, the experimenter will not be able to measure the true effect of the power input from the heater alone upon the sample's temperature rise, and this will result in an error in the calculated thermal conductivity.

In the DLS, the initial temperature of the sample may change, but if so, it must change at a near linear rate. This is because in the DLS it is the time differential that is recorded, and if the sample temperature changes at a near linear rate, the time differential will be constant (as it would be if the sample temperature were constant).

This relaxation in temperature control requirement has some very important practical results. It eliminates the need for sample temperature control systems. Control systems for temperatures below room temperature and at cryogenic temperatures require significant instrumentation and are a major undertaking in themselves. This is the reason that the data points shown later are taken only at one point below room temperature for the LS. The low temperature tests were obtained by constantly circulating liquid nitrogen through the sample holder and letting the sample come to its minimum temperature about 120°K. This means that the thermal conductivity function must be interpolated over a large range of temperatures, from 120°K to room temperature. By contrast, in the DLS, the sample temperature is lowered with liquid nitrogen to its minimum temperature and several DLS tests are taken. Then the LN_2 flow is shut off and the sample temperature begins to rise slowly to room temperature. Over the short time period of a DLS test (200 seconds), the temperature change is considered to be at a near constant rate and many DLS tests can be run between 120°K and room temperature.

DATA REDUCTION PROCEDURE

There are several ways to evaluate an LS test, but the "curve matching method" which uses a graphical solution was selected by A. D. Little, Inc. and Air Force Cambridge Research Laboratories as the most reliable method in their extensive thermal measuring programs. The method is fully described in one of their contractor reports [5].

The basic equation for the temperature, T, at a distance, r, from a continuous line source is

$$T = \frac{-q}{4 \pi K} \cdot E_i \left[- \frac{r^2}{4 \alpha t} \right] . \qquad (1)$$

In this expression, α is the thermal diffusivity of the sample, t is the time after onset of heater power, q is the input power per unit length of the heater wire (and is equal to VI/l, the heater wire voltage and current divided by the heater wire length), and K is the thermal conductivity. In the curve matching method one solves for T when $E_i[-r^2/(4\alpha t)] = 1$. This is done by plotting the test data as the logarithm of temperature versus the logarithm of time on a transparent overlay (Fig. 3a). This experimental curve is placed over a graph of $\ln - E_i(-1/x)$ versus $\ln x$ [where $x = (4\alpha t)/r^2$], (Fig. 3b), and is shifted along both axes until it gives the best match with the curve. The value for T is read off the experimental curve where it crosses the $-E_i[-1/x]$ curve at unity. This value of T is called T*. Substituting this into equation (1) and solving for K yields

$$K = \frac{q}{4\pi} \frac{1}{T^*} \quad , \tag{2}$$

and conductivity can be calculated.

The primary cause of error in this method of data reduction is that the curve matching process is a subjective one, subject to differences, depending on the persons doing the data reduction. Consequently, the curves are matched several times, and an average T* is selected.

In the DLS, temperature is differentiated with respect to time. Thus, equation (1) becomes

$$\frac{dT}{dt} = + \frac{q}{4\pi Kt} \exp\left(\frac{-r^2}{4\alpha t}\right) \tag{3}$$

This equation is solved by finding the maximum value of dT/dt and the corresponding time at which the maximum occurs, t_m. Then solving for conductivity,

$$K = \frac{q}{4\pi e \left(\frac{dT}{dt}\right)_m t_m} \quad . \tag{4}$$

The relationship between dT/dt and its maximum value e_{cm} is

$$\frac{dT}{dt} = \frac{S_1 \cdot S_2 \, e_{cm}}{n \cdot RC \cdot 100} \qquad (5)$$

This relationship takes into account n, the thermoelectric coefficient; RC, the time constant of the differentiating circuit; and S_1 and S_2, the ranges of the microvolt amplifiers. Merrill then found that the time $t_{\frac{1}{2}}$, or one-half of the time it takes to reach e_{cm}, could be read from the data chart more precisely than t_m. So he determined the relationship $t_{\frac{1}{2}} = 0.37337\, t_m$. Consequently,

$$K = \frac{0.37337\, V \cdot I \cdot n \cdot RC \cdot 100}{4\pi l \cdot S_1 \cdot S_2 \cdot e \cdot e_{cm} \cdot t_{\frac{1}{2}}} \qquad (6)$$

In a test, V and I are measured; l, S_1, S_2, and RC are known constants; and n, which varies with temperature, is found in standard tables. The only variables are e_{cm} and $t_{\frac{1}{2}}$, where e_{cm} is the difference between the initial and maximum voltages on the chart and $t_{\frac{1}{2}}$ is the time it takes to reach one-half of e_{cm}. Since all the values are known or can be found directly from the recorder, there are no subjective estimates as those in the curve matching method.

TIME COMPARISON

When comparing the two methods, the time consumed by each is of great significance. The times given in the following comparison are approximate because they vary somewhat with conditions such as sample conductivity and temperature. The time comparison of the output curves from the two systems is shown in Figure 4. Part A shows the temperature rise in the LS method. It takes approximately 1 to 1 1/2 hours to run an LS test to get enough points for a good curve match with the $\ln - E_i(-1/x)$ versus $\ln(x)$ curve. It takes another 1 1/2 hours to evaluate the test and calculate the thermal conductivity because the thermocouple voltages must be read from the recorder chart in intervals of 1 to 2 minutes, converted into temperatures, and plotted on log-log graph paper suitable for the curve matching. Since the temperature must be constant before a new test can begin, approximately 5 hours must be allowed between tests. Since the data evaluation may be concurrent with the time period between tests, one may obtain 2 LS data points in approximately 8 hours.

A DLS test takes a maximum of 5 minutes to run and 10 minutes to evaluate since there are only two values which are to be read from the chart, e_{cm} and $t_{\frac{1}{2}}$. The dotted portion of the curve in part B shows the DLS output if the heater were left on. The temperature of the samples is still increasing as in the LS, but at an ever decreasing rate. Therefore, the DLS output would continue to decrease. In practice, the experimenter turns the heater power off to terminate the test once he has determined that the maximum value of dT/dt has been reached. Since the heater is on for only approximately 200 seconds during the test, the temperature rise of the sample is not as high as in the LS, generally one-half to one-fourth. This, coupled with the fact that one does not have to wait for a constant sample temperature greatly reduces the time between tests. Many more data points (7 to 10) may be obtained during an 8-hour period with the DLS.

HEATER WIRE VOLTAGES

In determining thermal conductivity as a function of temperature, it is desirable that the sample temperature rise be as small as possible during a specific test. The practice has been to plot the conductivity at the average temperature of the initial and final temperatures of the test and to assume that the conductivity function of the samples is linear over increments of about 10 degrees. To get small temperature changes and small axial heat losses, the minimal heater wire voltages that are feasible are used. In the LS, good tests have been obtained with 1 to 2 volts. Over the long times of the LS tests, this usually gives temperature rises of 5 to 10 degrees for room temperature tests. Attempts to use small voltages with the DLS produced an interesting result. Significant scatter in the data points resulted when using voltages less than 2.8 volts; so the effects of different heater voltages on the DLS were investigated. These results are shown in Figure 5. The scatter decreased as the heater voltage was increased. The agreement of the data points using 3.5 volts is quite good. However, most of our work was done using 2.8 volts which gives temperature rises of from 1 to 3 degrees over the relatively short times of the DLS.

The higher voltages in the DLS give a faster temperature rise than in the LS, which may be significant for samples having different diffusivities and heat capacities, but with these test samples, it caused little disagreement between the two methods. This effect is one which should be investigated further on different sample materials.

TEST RESULTS

Spherical glass particles of two size distributions, 30 to 38 μm and 590 to 840 μm in diameter, were used as test samples. The results of the two test series are shown in Figures 6 and 7. The smaller particles were measured as a function of temperature over a range of 120°K to 373°K. The low temperature tests were obtained by constantly circulating liquid nitrogen through the sample holder coils. The sample reached a minimal temperature of 120°K and tests were run using both test methods. When the liquid nitrogen flow was shut off, the temperature rose slowly enough to be considered a constant rate over short periods of time. Therefore, DLS tests could be run until the sample reached room temperature. However, since for the LS tests the pretest temperature must be unchanging, and since a cryogenic sample temperature control system is not available, no LS tests were possible in this temperature regime. In Figure 6, Watson's equation for thermal conductivity of a particulate material in vacuum, $K = A + BT^3$ [6], has been curve fitted to the data points. K is the thermal conductivity, T, absolute temperature, and A and B are constants. It is evident that the DLS generally gives slightly lower conductivities than the LS. It is also seen from the curves that the difference between the two methods is somewhat greater at the high temperatures. At 120°K the DLS gives a conductivity value which is 88 percent of the LS value and at 370°K the figure is 82 percent. This may or may not be a real result. It is possible that at the higher temperatures, radiation effects are more prominent in the long-term tests. However, the effect may be apparent only, because of the paucity of data points for the LS at the low temperatures. Overall, the scatter in the DLS is slightly less than that in the LS, but the agreement is good, generally. The estimated error for both methods was ± 10 percent.

The 590 to 840 μm particles were run only at room temperatures (Fig. 7). In this figure, only DLS tests that had at least 2.5 volts applied across the heater wire are used. In this case, the agreement is excellent. All test values are given in Tables 1 through 4.

From these results, it is concluded that the Differentiated Line Source method provides test data that is comparable to the Line Source method and is much easier and faster to employ.

CONCLUSIONS

The comparison of the DLS and the LS is summarized as follows:

1. The DLS requires more instrumentation, an additional amplifier, and a differentiating circuit.

2. The DLS does not require a stable temperature to initiate a test. This eliminates the need for sample temperature control systems.

3. Using the DLS, tests may be taken as the sample temperature is rising slowly from liquid nitrogen temperature to room temperature. This eliminates the necessity of extrapolating the conductivity function over this large range.

4. Using the DLS much less time is required to run a test, usually about 5 minutes compared to about 90 minutes for the LS.

5. The sample temperature rise is less for the DLS, usually about one-half to one-fourth of the temperature rise for the LS.

6. Data reduction time is less for the DLS, on the order of 10 minutes as compared to 90 minutes for the LS. The need for curve matching is eliminated.

7. The advantages of reduced test time and data reduction time, coupled with the fact that the sample temperature rise is usually not as high in the DLS, combine to enable the experimenter to take about 10 DLS tests in the time of 2 LS tests in an 8-hour day. More data points result in better curve fits.

8. Test data from the two methods agree very well. The DLS gives slightly lower conductivity values for the small particles tested. The difference between the two methods seems to be smaller at low temperatures than at high temperatures. The agreement in the large particle tests at room temperature is excellent.

TABLE 1. MEASURED CONDUCTIVITY — DLS TESTS WITH SPHERICAL GLASS PARTICLES 30 to 38 μm IN DIAMETER

Temperature (°K)	Thermal Conductivity (10^{-5} W/cm °K)	Temperature (°K)	Thermal Conductivity (10^{-5} W/cm °K)
122	0.72	245	1.47
124	0.77	248	1.41
128	0.59	251	1.30
130	0.66	251	1.35
147	0.71	254	1.48
159	0.99	257	1.39
162	0.87	257	1.42
176	0.85	258	1.30
181	0.94	260	1.34
187	0.93	264	1.36
195	0.83	264	1.43
196	0.99	268	1.42
200	1.27	268	1.53
202	1.08	271	1.67
206	1.38	271	1.45
206	1.31	273	1.36
210	1.13	275	1.40
212	1.06	278	1.39
220	1.26	281	1.51
221	1.12	284	1.43
229	1.13	284	1.63
230	1.21	287	1.87
235	1.17	292	1.66
243	1.25	292	1.70
243	1.49	293	1.72

TABLE 1. (Concluded)

Temperature (°K)	Thermal Conductivity (10^{-5} W/cm °K)	Temperature (°K)	Thermal Conductivity (10^{-5} W/cm°K)
293	1.85	333	2.04
296	1.67	337	2.28
297	1.79	337	2.31
297	1.99	338	2.22
298	1.92	338	2.35
298	1.93	339	2.35
298	1.84	339	2.34
298	1.64	339	2.28
299	1.91	339	1.98
299	1.90	340	2.14
299	1.67	343	2.15
299	1.96	367	2.70
300	1.96	368	2.67
300	1.80	386	2.52
301	1.71	369	2.67
301	1.63	370	2.39
305	2.02	370	2.55
310	1.98	371	2.61
318	1.85	371	2.24
325	2.16	373	2.65
327	2.10		

TABLE 2. MEASURED CONDUCTIVITY — LS TESTS WITH SPHERICAL GLASS PARTICLES 30 to 38 μm IN DIAMETER

Temperature (°K)	Thermal Conductivity (10^{-5} W/cm °K)	Temperature (°K)	Thermal Conductivity (10^{-5} W/cm °K)
118	0.93	307	1.98
118	0.99	307	2.02
120	1.10	307	2.51
296	1.95	308	2.02
296	2.03	336	2.22
296	2.25	336	2.42
297	1.91	337	2.22
297	1.98	367	3.56
297	2.05	368	3.10
297	2.07	368	3.40
297	2.09	369	2.99
297	2.13	369	3.30
297	2.26	371	2.99
297	2.35	371	3.32
298	2.09	371	3.54
299	2.01	371	3.59
299	2.58		

TABLE 3. MEASURED CONDUCTIVITY — DLS TESTS WITH SPHERICAL GLASS PARTICLES 590 to 840 μm IN DIAMETER

Temperature (°K)	Thermal Conductivity (10^{-5} W/cm °K)	Temperature (°K)	Thermal Conductivity (10^{-5} W/cm °K)
297	8.15	298	8.49
297	8.24	298	8.68
297	8.27	298	9.19
297	8.32	298	9.77
297	8.54	298	10.3
297	8.68	299	7.75
297	8.72	299	8.14
298	6.99	299	8.27
298	7.75	299	8.29
298	7.84	299	8.35
298	7.86	299	8.44
298	8.26	300	7.66
298	8.34	300	8.18
298	8.38		

TABLE 4. MEASURED CONDUCTIVITY — LS TESTS WITH SPHERICAL GLASS PARTICLES 590 to 840 μm IN DIAMETER

Temperature (°K)	Thermal Conductivity (10^{-5} W/cm °K)
297	8.32
298	8.62
298	8.69
298	9.33
299	8.15
299	8.34
299	9.39

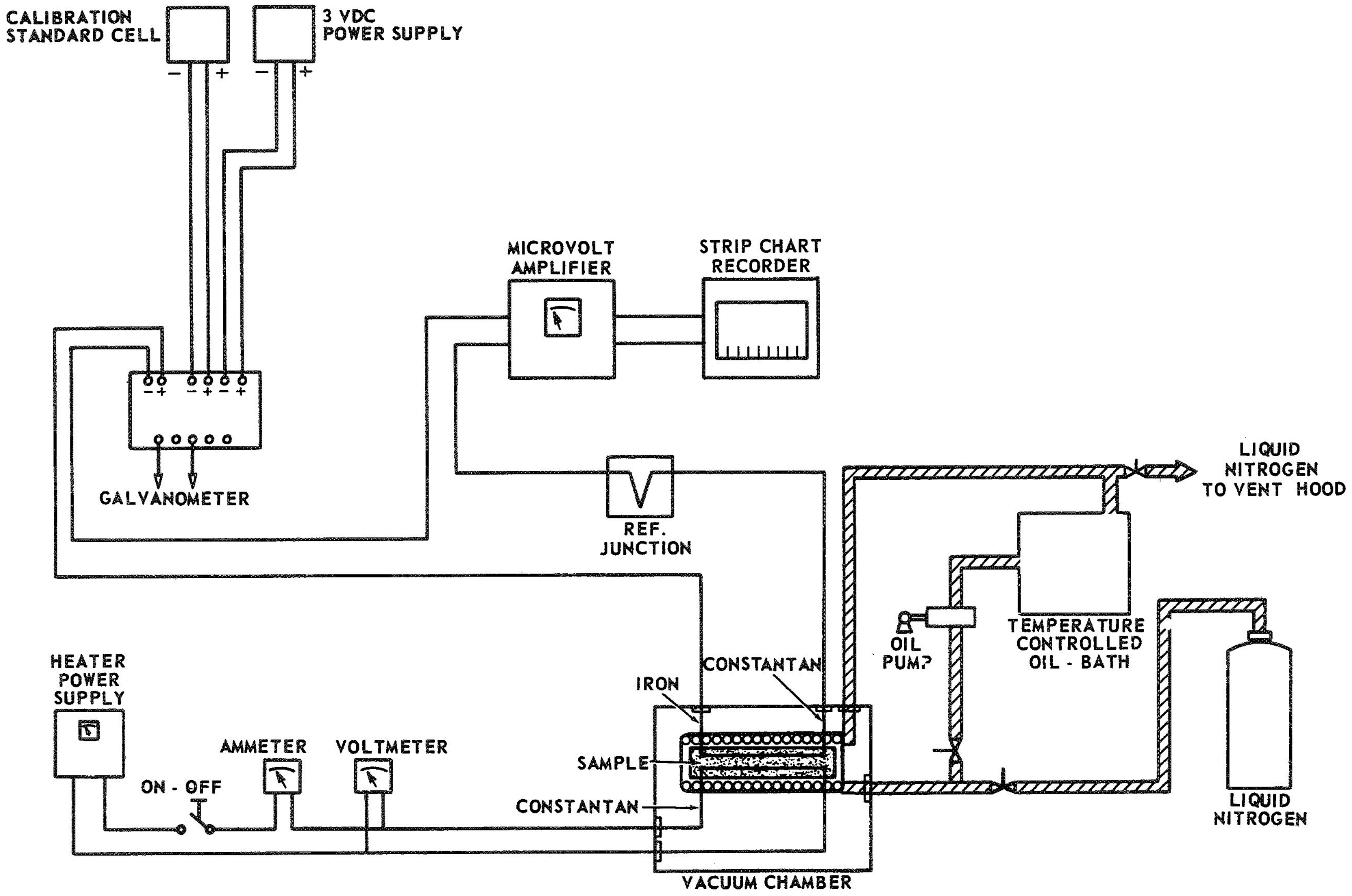


Figure 1. Apparatus required for line source method.

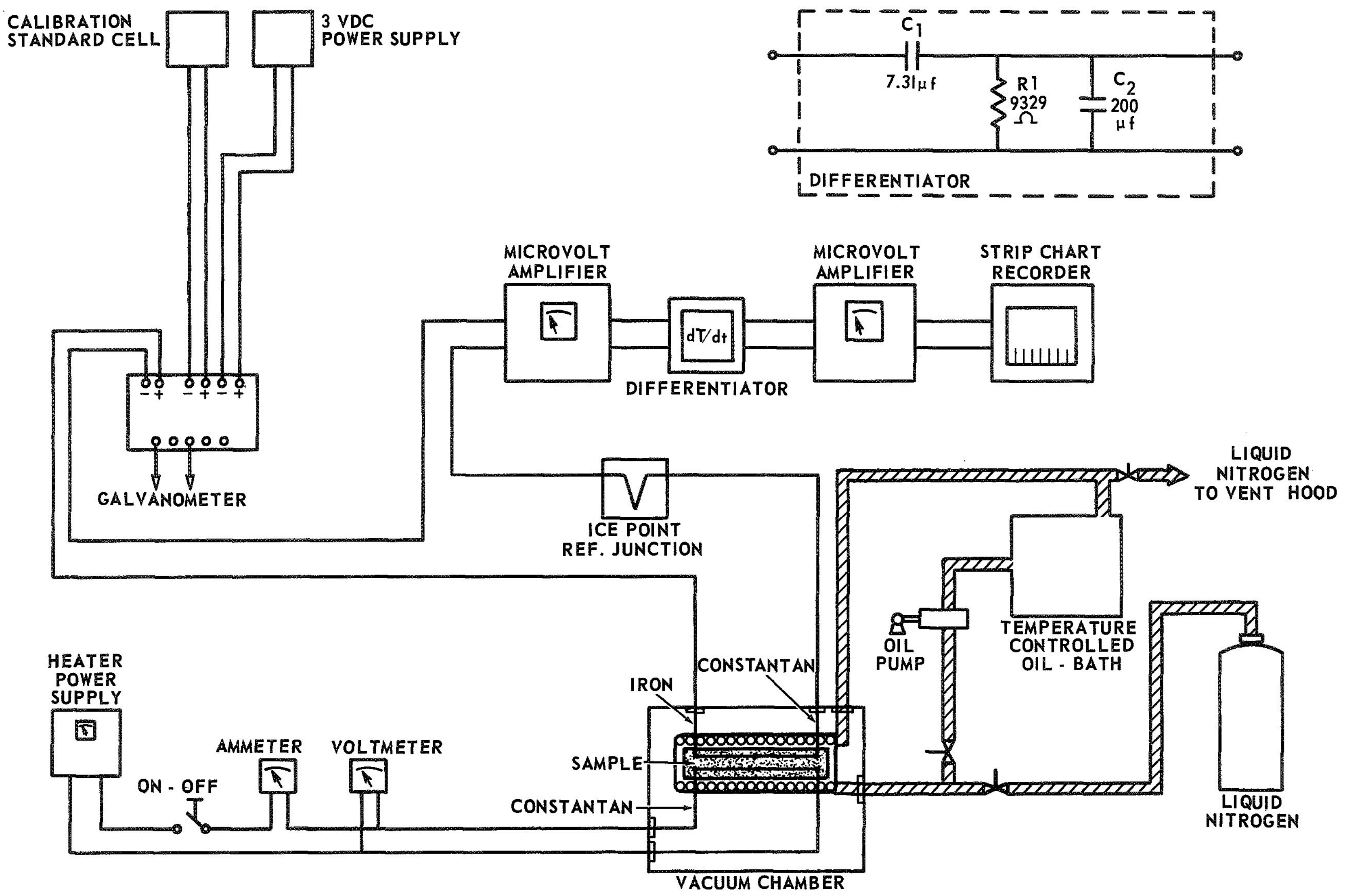
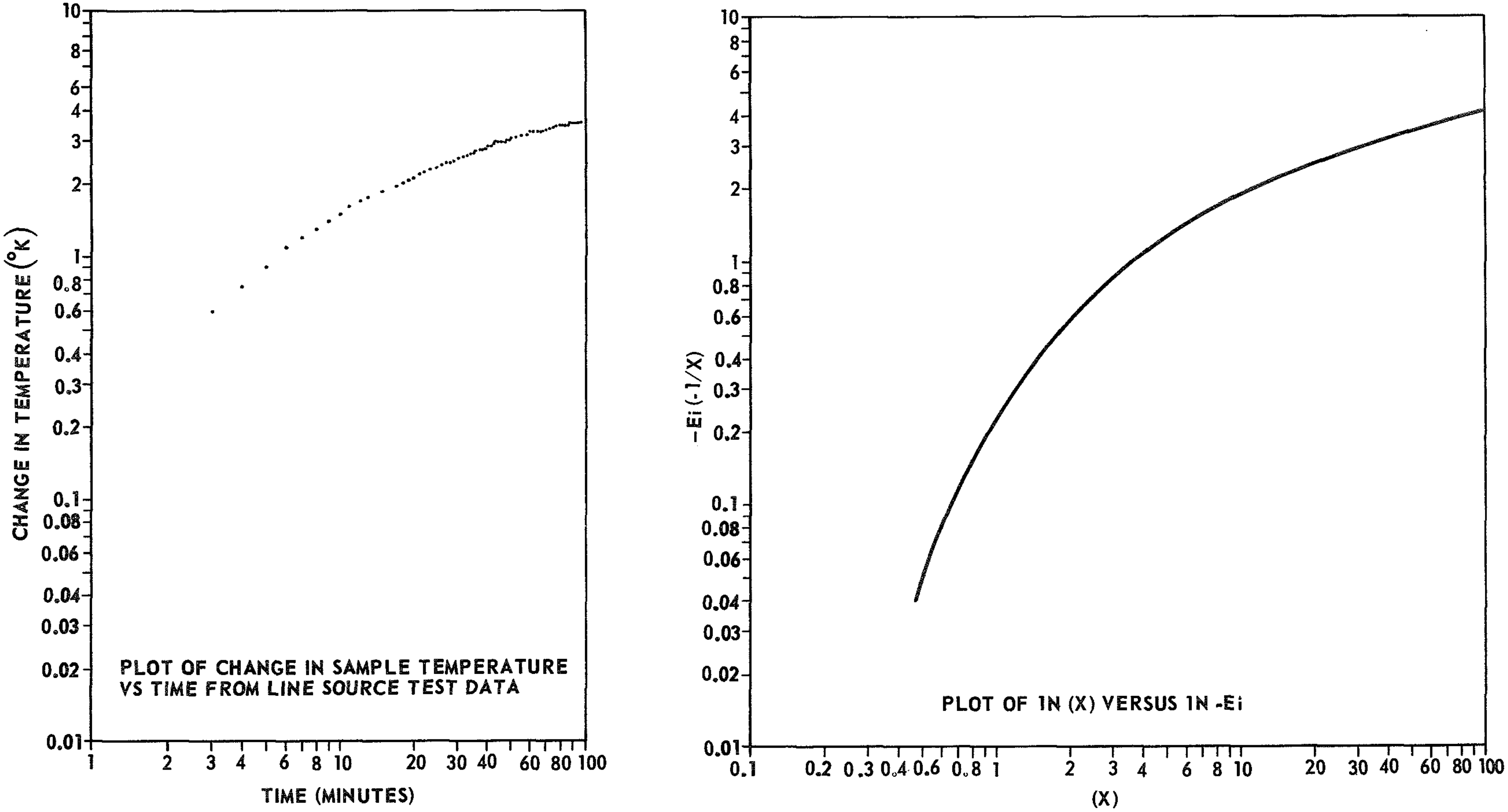


Figure 2. Apparatus required for differentiated line source method.



(a)

(b)

Figure 3. Curves for curve matching method.

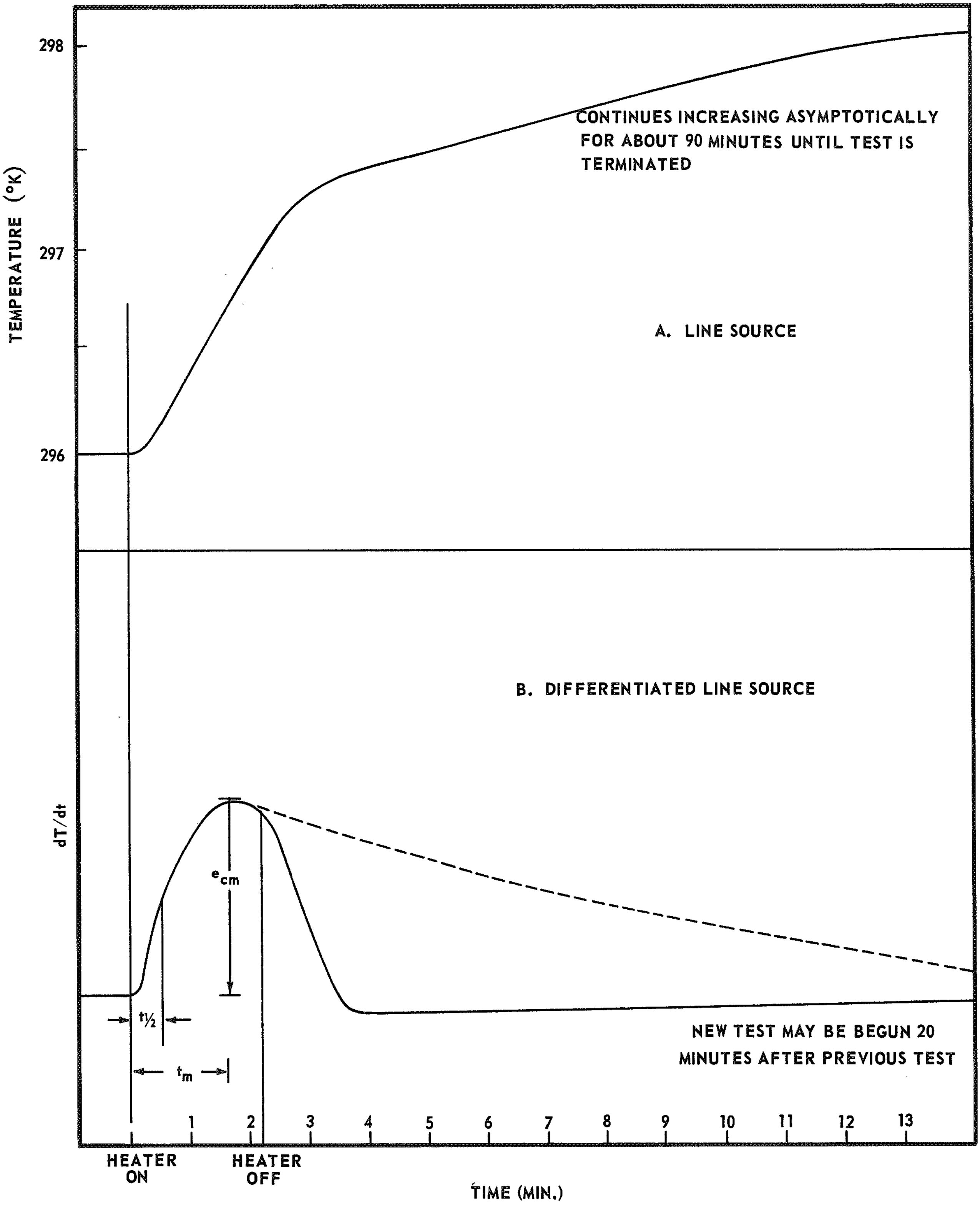


Figure 4. Output curves from LS and DLS systems.

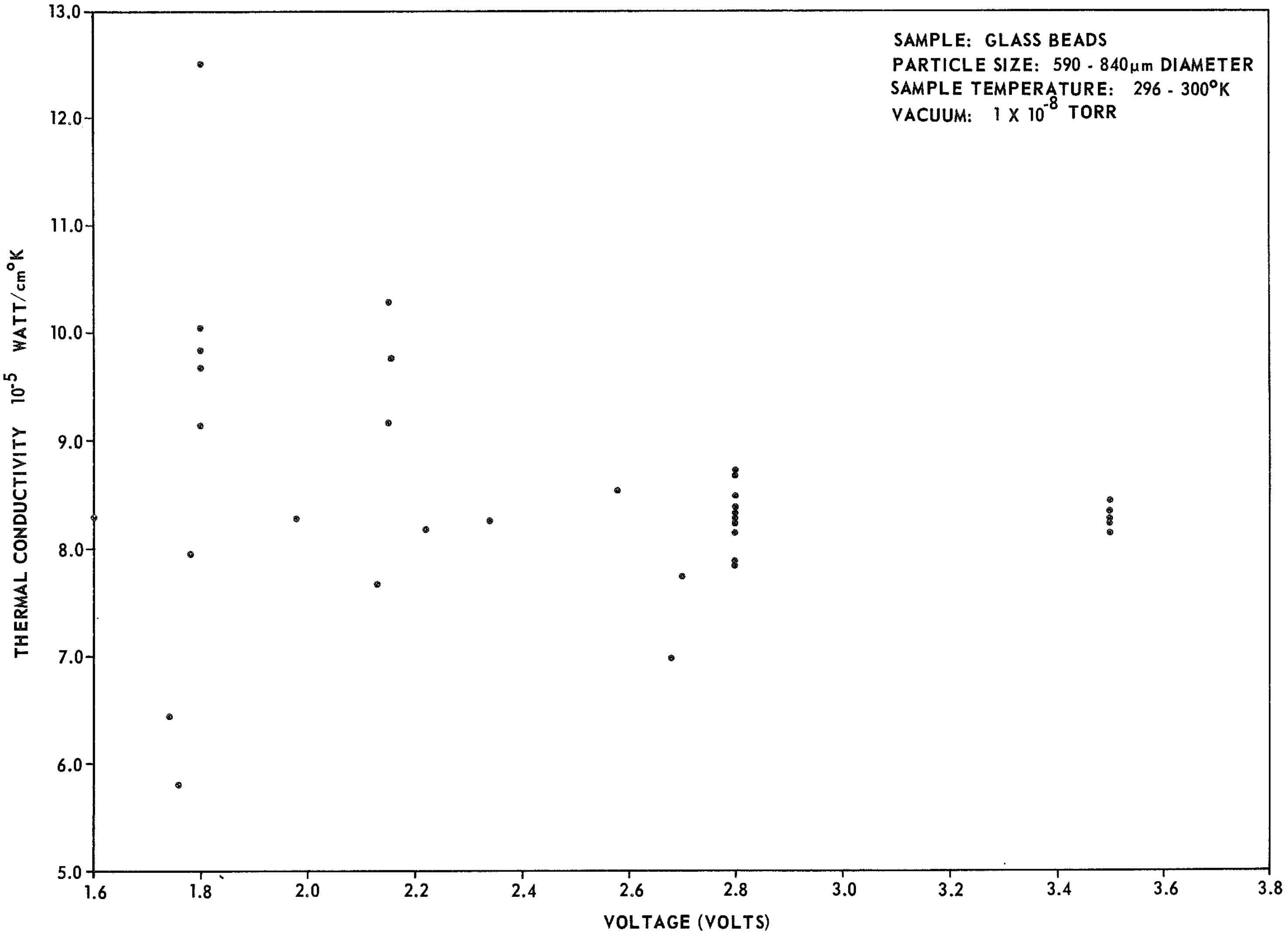


Figure 5. The effect of heater wire voltage on thermal conductivity measurements in the DLS.

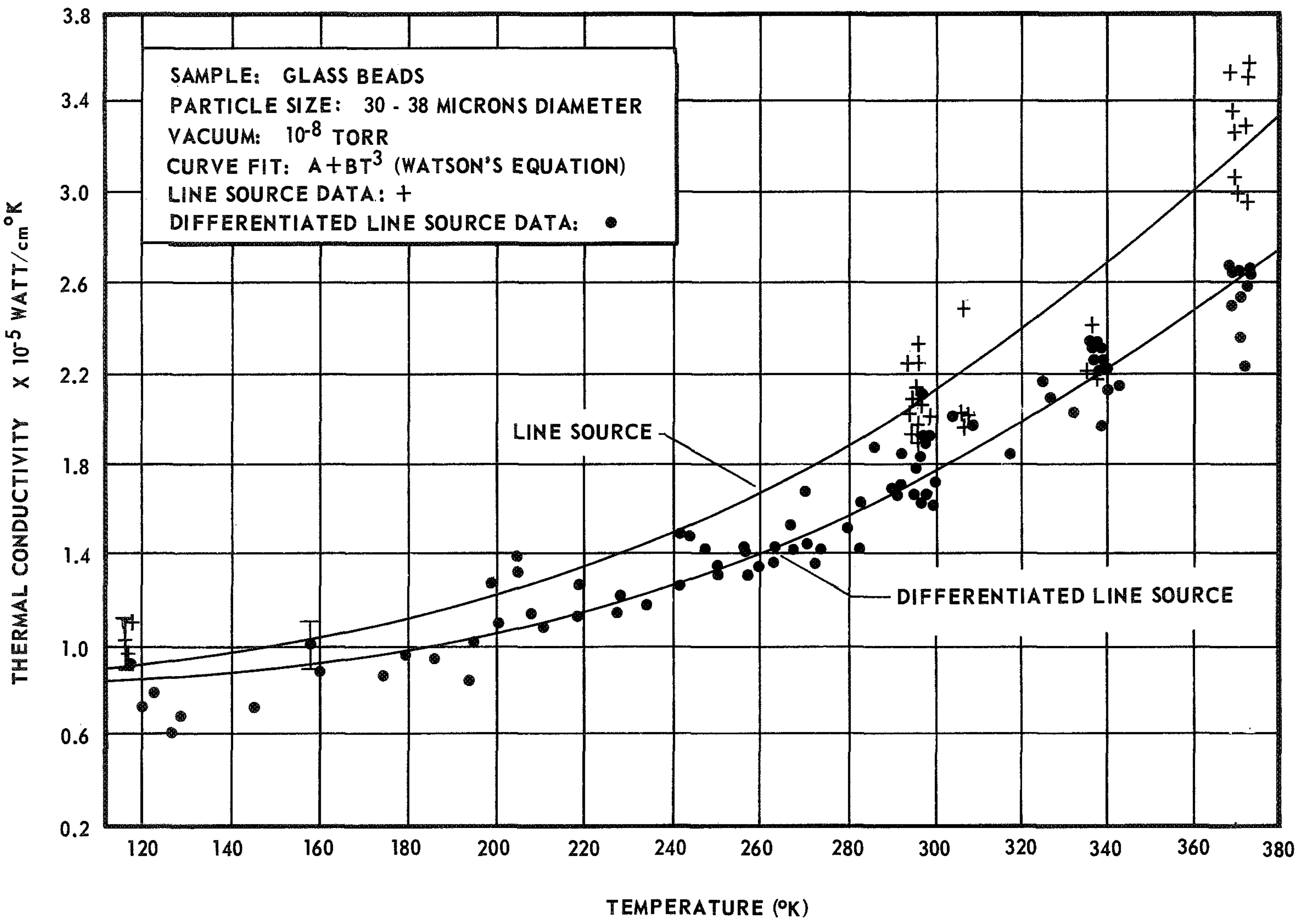
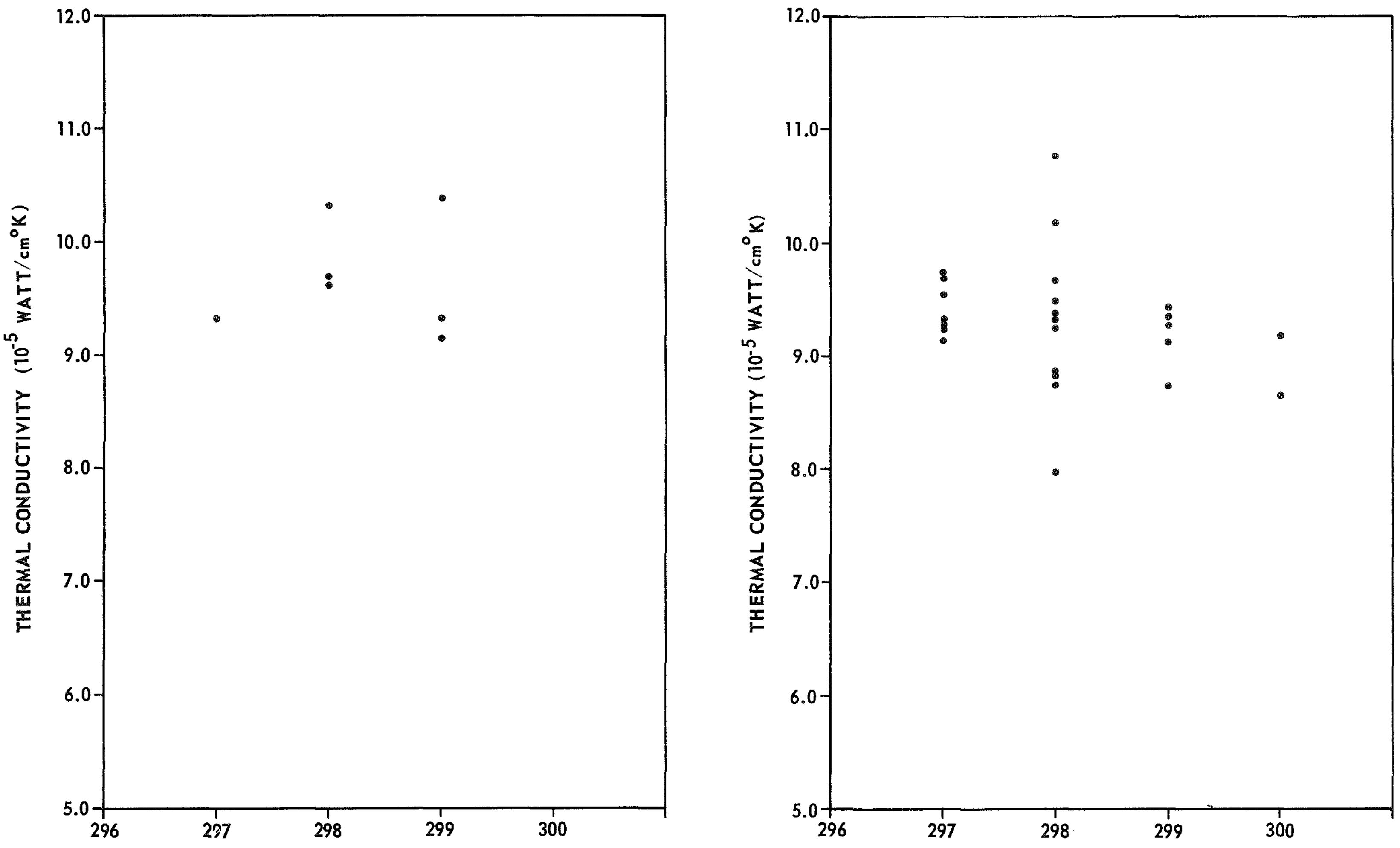


Figure 6. Thermal conductivity of a particulate sample (under vacuum) versus temperature.



(a) Line Source average conductivity 8.65×10^{-5} W/cm °K.

(b) Differentiated Line Source average conductivity 8.36×10^{-5} W/cm °K.

Figure 7. Results of tests on glass beads 590 to 840 μm diameter.

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APPROVAL

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By Ronald W. Scott and James A Fountain

The information in this report has been reviewed for security classification. Review of any information concerning Department of Defense or Atomic Energy Commission programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

This document has also been reviewed and approved for technical accuracy.

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