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U.S. DEPARTMENT OF COMMERCE/National Bureau of Standards

Standard Reference Materials:

A Fine-Grained, Isotropic Graphite for Use as NBS Thermophysical Property RM's from 5 to 2500 K he National Bureau of Standards<sup>1</sup> was established by an act of Congress on March 3, 1901. The Bureau's overall goal is to strengthen and advance the nation's science and technology and facilitate their effective application for public benefit. To this end, the Bureau conducts research and provides: (1) a basis for the nation's physical measurement system, (2) scientific and technological services for industry and government, (3) a technical basis for equity in trade, and (4) technical services to promote public safety. The Bureau's technical work is performed by the National Measurement Laboratory, the National Engineering Laboratory, the Institute for Computer Sciences and Technology, and the Center for Materials Science.

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- Polymers
- Metallurgy
- Reactor Radiation

Standard Reference Materials:

# A Fine-Grained, Isotropic Graphite for Use as NBS Thermophysical Property RM's from 5 to 2500 K

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#### PREFACE

Standard Reference Materials (SRM's) as defined by the National Bureau of Standards are "well-characterized materials, produced in quantity, that calibrate a measurement system to assure compatibility of measurement in the Nation." SRM's are widely used as primary standards in many diverse fields of science, industry, and technology, both within the United States and throughout the world. For many of the Nation's scientists and technologists it is of more than passing interest to know the measurements obtained and methods used by the analytical community when analyzing SRM's. An NBS series of papers, of which this publication is a member, called the <u>NBS Special Publi</u>cation - 260 Series is reserved for this purpose.

This 260 Series is dedicated to the dissemination of elemental concentration data for NBS biological, geological, and environmental SRM's. More information will be found in this 260 than is generally found in NBS Certificate of Analysis. This 260 enables the user of these SRM's to assess the validity of data not available in the Certificate of Analysis. We hope that this 260 will provide sufficient additional information so that new applications of these SRM's may be sought and found.

Inquiries concerning the technical content of this compilation should be directed to the authors. Other questions concerned with the availability, delivery, price of specific SRM's should be addressed to:

Office of Standard Reference Materials National Bureau of Standards Gaithersburg, MD 20899

> Stanley D. Rasberry, Chief Office of Standard Reference Materials

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#### A FINE-GRAINED, ISOTROPIC GRAPHITE FOR USE AS NBS THERMOPHYSICAL PROPERTY RM'S FROM 5 to 2500 K\*

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#### ABSTRACT

The Chemical Engineering Science Division (Boulder, Colorado) in conjunction with the Office of Standard Reference Materials (Gaithersburg, Maryland) of the National Bureau of Standards, and the CODATA Task Group on Thermal Transport Properties have investigated graphite as a potential, extended temperature range, Research Material (RM). A large number of isotropic, fine-grained graphite rods in various diameters were obtained for these investigations.

In Phase I, electrical resistivity and density measurements were performed on numerous rods at temperatures from 4 to 300 K. In Phase II, thermal conductivity measurements were performed on thirteen specimens at about 20 °C. These measurements show that transport property variations, both between and within these rods, is relatively large (approximately 10%). However, a correlation between these variables is shown to exist which will allow the calculation of thermal conductivity from simple and inexpensive electrical resistivity and density measurements to within about  $\pm 2\%$ . In Phase III, a large number of specimens were characterized for room temperature electrical resistivity and density. These measurements were in preparation for the worldwide distribution of specimens to participants that agreed to make thermal and electrical property measurements. Phase IV describes the results of the measurements from the various participants. Phase V describes the analysis of these data.

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Key Words: characterization; density; electrical resistivity; graphite; Lorenz ratio; Research Materials; thermal conductivity.

#### 1. INTRODUCTION

Considerable interest has been shown in establishing a fine-grained, isotropic graphite for use as a Research Material (RM) for thermophysical properties. Preliminary work on the Air Force Materials Laboratory-Advisory Group for Aerospace Research and Development, NATO (AFML-AGARD) program showed that graphite is a promising material. It is especially interesting because of its relatively low cost, ease of fabrication, and its wide temperature range.

The AFML-AGARD program (1965 to 1975) resulted in extensive thermophysical property measurements on several materials including graphite. These measurements have been reported in detail by Fitzer [1] and Minges [2]. The graphite portion of that program is summarized below:

#### AFML-AGARD PROGRAM (1965-1975)

#### Material

Fine-grained-isotropic graphite 10 cm dia. x 30 cm long cylinders 5 cm x 10 cm x 15 cm blocks Ave. density =  $1.757 \text{ g/cm}^3$ Max. variations =  $\pm 1.3\%$ Heat treated at 2500 °C

#### Measurements

Property	Temp. Range	Variations	No. of Investigators
Thermal Diffusivity Thermal Conductivity Thermal Expansion Heat Capacity	400-2600 K 300-2500 K 300-2800 K 1900-2800 K	+7% +10% +3%	9 5 10 1
Electrical Resistivity	400-2600 K	<u>+</u> 4% at 1300 K	2

It was concluded as a result of the program that this graphite is very promising, but that further work should be performed. The remaining stock of this AFML material was donated to NBS for further study and/or use. The remaining quantity of material, however, was guite limited. Unfortunately, also, the specimens that

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were distributed to participants of the program were neither individually characterized prior to measurement, nor were they collected after measurement for postcharacterization. Because of this, NBS decided to purchase a supply of the same fine-grained graphite (AXM-5Q1) for further study. To establish a reasonable supply, 400 6.4 mm diameter rods, 150 12.7 mm diameter rods, and 70 25.4 mm diameter rods were purchased. All rods are 30 cm in length. In addition, some AXM-5Q and AXM-9Q material was purchased for research purposes. This material was specified to have a density in the range 1.72-1.75 g/cm<sup>3</sup>.

This fine-grained graphite is produced by molding into blocks or plates. Rods are then cut and machined from the blocks. Petroleum coke is the source of the graphite, and the final graphitization is performed at 2500 °C, or in the case of the 9Q material at 2900 °C. Further preparation details are considered proprietary by the supplier. The supplier indicated that the most homogeneous product will come from relatively thin plates. These rods were machined from 5 cm x 10 cm x 30 cm plates.

#### 2. PHASE I - PRELIMINARY ELECTRICAL RESISTIVITY AND DENSITY CHARACTERIZATION

#### 2.1 Specimen Preparation

To perform electrical resistivity and density measurements, specimens of 6.4 mm diameter and 50 mm length were required. Rods were randomly selected from the 6.4 mm diameter rods and notched with a code indicating the pack and rod from which they came. The code is a three digit number corresponding to the number of notches on each end and on the side near end 1, i.e., 1st digit = notches on end no. 1, 2nd digit = notches on end no. 2, 3rd digit = notches on side no. 1. Rods 50 mm in length were cut from the 25.4 mm diameter rods. These were quartered and machined to 6.4 mm diameter rods. Also some scrap blocks from the AFML-AGARD material were machined to 6.4 mm diameter specimens for intercomparison with the new rod material. A total of 39 specimens were prepared: 21 from

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the 6.4 mm diameter rods, 12 from three 25.4 mm diameter rods, and six from a single slab of AFML material.

#### 2.2 Measurements

All electrical resistivity measurements were performed with four-terminal D.C. potentiometric apparatus. The imprecision of this apparatus is estimated at ±0.5%. (For inhomogeneity determinations the imprecision, not the uncertainty, is paramount). Electrical resistivities were determined at 4 K, 76 K, and 273 K on most of the specimens. Some of the specimens were measured only at 76 K.

Densities were first calculated from air weight-volume measurements. This was done because it was suggested that the usual hydrostatic weighing technique could not be used on graphite. This seemed reasonable because it was assumed that the graphite would absorb water and give incorrect bulk densities. After the initial tests, hydrostatic weighings to check the previous statement were performed. Surprisingly, the hydrostatic weighing method yielded results entirely consistent with the air weight-volume method. No water absorption was observed during the measurements. The densities reported here are, therefore, based on the hydrostatic weighing method which is considered to yield more precise results. All density measurements were performed at ambient temperature, approximately 20 °C. These measurements are estimated precise to +0.1%.

After the NBS-Boulder electrical resistivity and density measurements, recorded in Table 1, a group of seventeen specimens was selected from the 39 specimens. These were sent to Mr. Pears of Southern Research Institute (SoRI) for annealing at 3180 °C (5750 °F). Mr. Pears suggested that the variability in density and electrical resistivity could be considerably reduced after such an anneal. The electrical resistivities and densities were measured at 20 °C both prior to and after the 3180 °C anneal at SoRI. These results are also listed in Table 1. It is noted that the densities of NBS and SoRI agree quite well and the

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Specimen Identification	<sup>Թ</sup> գ µΩ•m	<sup>Թ</sup> 76 µΩ•m	<sup>ρ</sup> 273 μΩ•m	Density* NBS <sub>3</sub> g/cm	Density SoRI (Pre)	P293 SoRI (Pre)	Density SoRI (Post)	<sup>ρ</sup> 293 SoRI (Post)
100 200 AFML Scrap 300 blocks 400 (6.4 mm dia) 500 600	29.42 29.28 29.91 29.35 29.15 28.50	24.02 23.92 24.47 23.91 23.67 23.27	15.23 15.27 15.67 15.21 15.08 14.85	1.755 1.751 1.742 1.754 1.754 1.754 1.765	1.741 1.752 1.757	14.59 14.56 14.03	1.747 1.746 1.757	11.58 11.46 11.16
101 Rod 201 No. 1 301 102 AXM-5Q1 202 Rod POCO 302 No. 2 25.4 mm dia 103 203 303 403	28.59 29.51 28.93 29.60 26.52 26.87 27.06 26.65 29.63 28.90 28.90 29.45	23.28 24.10 23.57 24.12 21.24 21.52 21.69 21.34 24.20 23.58 23.55 24.04	14.81 15.32 15.07 15.29 13.12 13.27 13.49 13.25 15.49 14.99 15.00 15.34	1.721 1.706 1.713 1.707 1.751 1.749 1.746 1.752 1.721 1.735 1.734 1.722	1.720 1.713 1.705	13.96 14.32 14.85	1.717 1.709 1.703	12.74 12.74 13.19
110 Rod end a 210 No. 1 b 310 Rod No. 2 AXM-5Q1 111 Pack 1A 121 Rod 131 No. 1 141	28.23 26.46	23.04 21.47 28.12 22.30 23.35 24.69 23.94	14.74 13.53 18.56	1.732 1.782 1.698 1.768 1.750 1.721 1.732	1.783 1.744 1.722	12.95 14.30 15.30	1.781 1.748 1.720	12.20 13.48 14.27
151 Rod No. 1 211 Rod No. 2 Pack 311 Rod end a 2A 411 No. 3 b	31.94 29.77 32.28 28.22	26.06 24.12 26.66 23.02	16.61 15.20 17.27 14.67	1.727 1.709 1.709 1.775	1.732 1.713 1.778	15.80 16.42 14.09	1.730 1.711 1.775	13.12 13.98 12.19
152 Rod end a 212 No. 1 b 312 Rod No. 2 412 Rod No. 3 Pack 112 Ad 122 Rod 132 No. 1 142	32.00 29.41 30.55 31.40	26.14 23.78 25.13 25.95 25.22 24.65 24.17 23.91	16.68 14.99 16.26 16.80	1.697 1.716 1.747 1.725 1.706 1.717 1.724 1.728	1.700 1.723 1.719 1.727	16.12 14.86 14.34	1.697 1.720 1.717 1.725	13.28 14.15 12.76 12.50
113 Rod No. 1 Pack 4A 104 Rod No. 1 Pack 5A	31.38	23.99 26.22	15.38 17.31	1.727 1.751	1.753	16.49	1.748	12.94

Table 1. Resistivity and Density Data for AXM-5Q1 Graphite Specimens.

\*All NBS densities measured near 20 °C.

effect of the high temperature anneal is small on density. However, the electrical resistivities were affected considerably by the anneal.

#### 2.3 Results and Discussion

To determine the consistency of the NBS measurements and the SoRI measurements, graphs were made of the resistivity as a function of temperature. These plots, Figures 1, 2, and 3 illustrate several important points. First they confirm a measurement imprecision of near  $\pm 0.5\%$ . (They also indicate a comparable accuracy). These plots also indicate that the differences between specimens are temperature independent to within the measurement imprecision from 4 to 300 K. Since the SoRI measurements were performed at 293 K and the NBS measurements were done at 4, 76, and 273 K, direct intercomparison is not possible. However, these plots show excellent correspondence between the two sets of data.

The second set of plots, Figures 4, 5, and 6 illustrate the dependence of electrical resistivity on density at 4, 76, and 273 K, respectively. The lines drawn on these figures show the resistivity-density correlation for groups of specimens coming from single rods or plates. As can be seen, very strong correlation (intra-rod correlation) exists for such groups. It is also clear that the electrical resistivity-density correlation between such groups (inter-rod correlation) is very weak. Obviously electrical resistivity is strongly dependent on density but also on a density independent parameter which varies from rod-to-rod but is fairly constant within each rod. This may be related to the chemical purity, the degree of graphitization, or the void (vacancy) concentration and distribution of each rod. At this time, the source of these inter-rod differences are not clear. The intra-rod correlation corresponds to a four percent change in electrical resistivity per one percent change in density at all temperatures.

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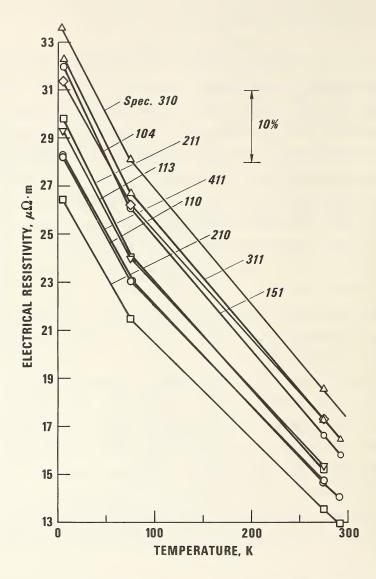


Figure 1. Electrical resistivity as a function of temperature of nine AXM graphite specimens.

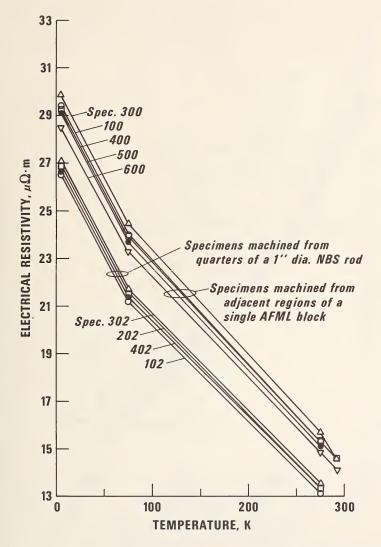


Figure 2. Electrical resistivity as a function of temperature of ten AXM graphite specimens. Four specimens taken from a single 25.4 mm diameter NBS rod and six specimens taken from a single AFML slab to illustrate short range inhomogeneity.

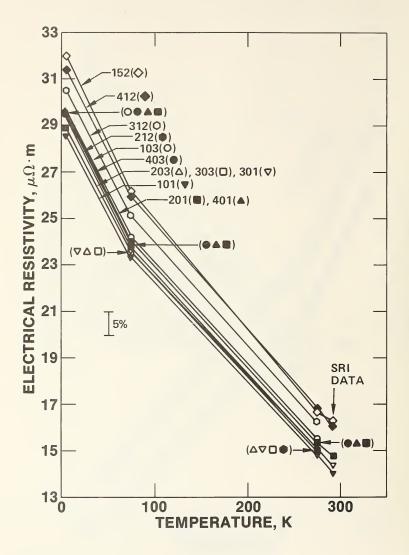
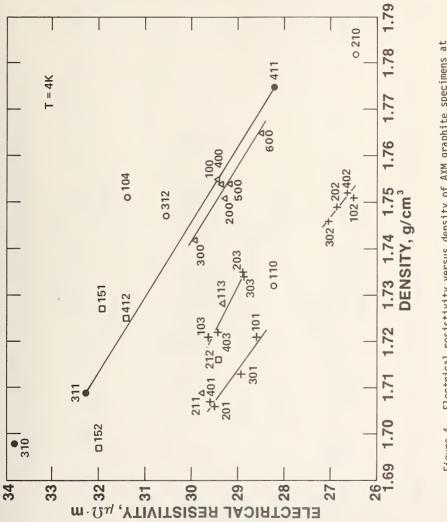
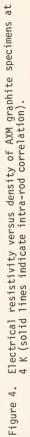
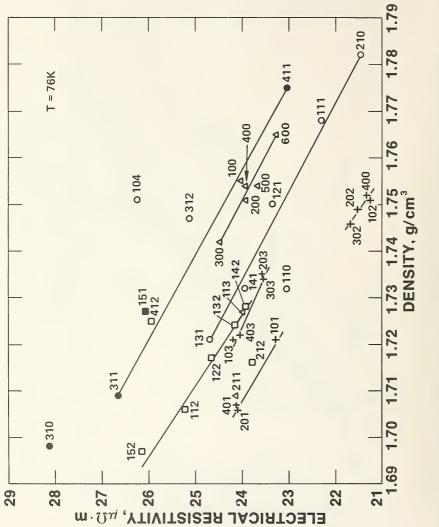
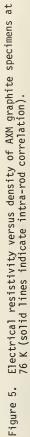


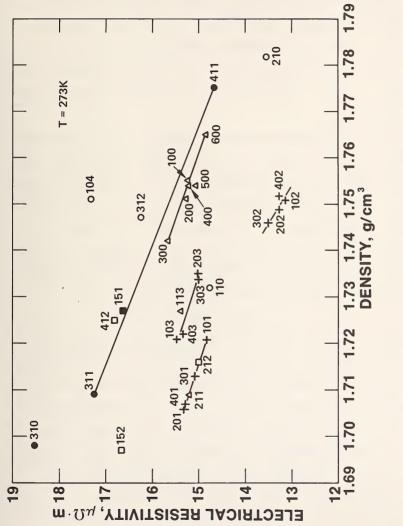
Figure 3. Electrical resistivity as a function of temperature of twelve AXM graphite specimens.











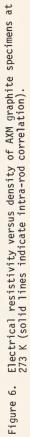


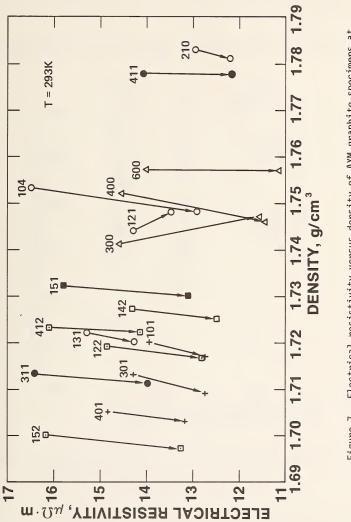
Figure 7 illustrates the resistivity-density data taken at SoRI. The arrows between pairs of data points are directed from the preanneal to the post-anneal data. The average change in density was about 0.2%, and the average change in resistivity about 15%. No significant improvement in homogeneity is observed as a result of the 3180 °C anneal. The intra-rod electrical resistivity-density correlation also remained the same after the high temperature anneal. It is clear that a high temperature anneal is necessary to stabilize the specimens for use above 2500 °C; but, it is also clear that such an anneal does not appreciably improve the homogeneity.

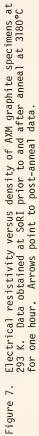
In addition to the above described results on AXM-5Q1, several measurements were performed on one rod of AXM-9Q1 and one rod of AXM-5Q. Each of these rods were cut into six specimens. The densities and electrical resistivities of these specimens are listed in Table 2 and plotted in Figures 8 and 9. These figures show a uniform change in property from one end of the rod to the other. Figure 10 shows the electrical resistivity versus density correlation for these two rods. Again a strong correlation is observed and the value of the slope is in agreement with the results from the AXM-5Q1 measurements.

#### 2.4 Other Graphites

Three rods of AXF-5Q material and two rods of spectroscopic purity graphite, designated as FXI material, were obtained for measurement. The FXI material is higher purity and density than the AXF and AXM material. The average density of each of the five rods and the resistivity as a function of position along these rods at 2.5 cm intervals was measured. These data are illustrated in Figure 11. The results show that the intra-rod variability in electrical resistivity for the AXF material is about 13%, and the inter-rod variability is about the same. These results are comparable to those obtained with the AXM material. The results on the spectroscopic purity rods show the rate of change in resistivity as a

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S	pecimen	<sup>р</sup> 76К	Density	
Iden	tification	μΩ•m	g.cm <sup>-3</sup>	
410 510 610 220 320 420	Pack 6A AXM-9Q1 (Rod 1)	19.64 19.18 18.93 18.95 18.71 19.15	1.724 1.734 1.736 1.737 1.742 1.731	
520 620 330 430 530 630	Pack 7A AXM-5Q (Rod 1)	21.32 21.55 22.01 22.49 23.01 23.15	1.739 1.733 1.721 1.716 1.708 1.696	

Table 2. Resistivity and Density Data for AXM-5Q and 9Q1 Graphite Specimens.

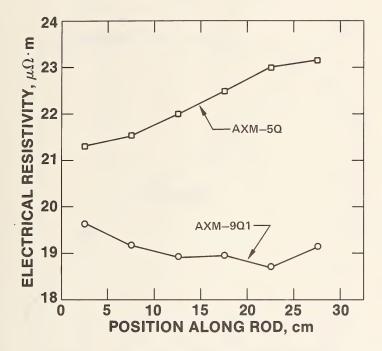


Figure 8. Electrical resistivity versus position along an AXM-5Q and AXM-9Q1 graphite rod at 76 K.

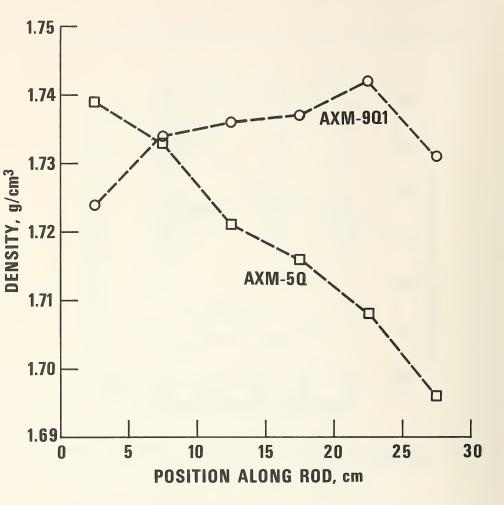
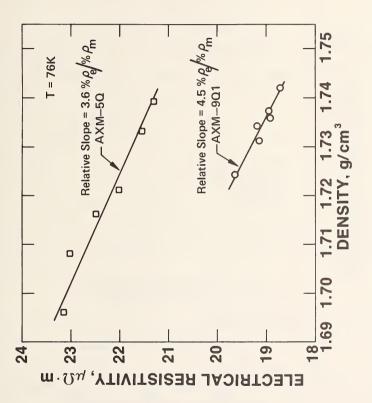


Figure 9. Density versus position along an AXM-5Q and AXM-9Q1 graphite rod at 20°C.





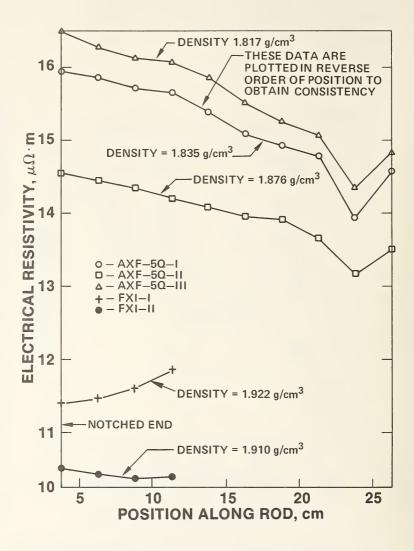


Figure 11. Electrical resistivity versus position along AXF and FXI graphite rods.

function of position (i.e., slope of  $\rho$  vs X) is of comparable magnitude. However, these rods were only 15 cm long compared to 30 cm for the AXF material, and therefore the total change was not as great for the FXI material. The difference between the two FXI rods is near 10% in resistivity. Thus it appears that neither of these materials is better than the AXM material from a homogeneity standpoint.

# 2.5 Conclusions and Recommendations

The inhomogeneity in electrical resistivity of these graphite rods is discouragingly high. It is clear that this lot of graphite cannot be used as an electrical resistivity SRM without characterizing each rod. If the thermal conductivity correlates with electrical resistivity, as expected, the same statements are applicable to thermal conductivity. Thermal expansion and specific heat are not expected to be sensitive to these inhomogeneities and, therefore, this lot of graphite may be acceptable for these properties.

Because of the merits of using graphite for various RM's, it was recommended that further work be done on this lot of graphite to investigate the thermal conductivity-electrical resistivity-density correlations. This work represents Phase II of this investigation.

### 2.6 Summary of Phase I

1. The inhomogeneity in electrical resistivity (and of thermal conductivity by association) of this lot of graphite rods is excessively high for use as SRM's, unless each piece is certified.

 A strong intra-rod correlation exists between electrical resistivity and density. This correlation is practically non-existent over this range of densities for inter-rod specimens.

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3. Annealing the specimens at 3180 °C produces an average decrease of 16% in electrical resistivity and 0.2% in density, but statements (1) and (2) above are still applicable to essentially the same degree for the annealed specimens.

4. The electrical resistivity-density correlation is essentially the same for AXM-5Q1, AXM-5Q, and AXM-9Q graphite.

5. The validity of the above statements is independent of temperature (from 4 to 300 K). Therefore, subsequent homogeneity characterization measurements are conducted at ambient temperature.

6. Further work is recommended to investigate the thermal conductivityelectrical resistivity-density correlation and the feasibility of characterizing each specimen of graphite for SRM use.

3. PHASE II. PRELIMINARY THERMAL CONDUCTIVITY CHARACTERIZATION

#### 3.1 Measurements

A series of ambient temperature thermal conductivity measurements with comparative apparatus was completed. This comparative apparatus is schematically illustrated in Figure 12. Prior to the measurements on graphite, a series of runs on two specimens of electrolytic iron (SRM-1463) was conducted to ascertain the accuracy and precision of this apparatus. These measurements showed that the thermal conductivity results are precise to within  $\pm 1\%$  with no measurable systematic bias.

The thermal conductivity of thirteen graphite specimens was measured covering the entire range of electrical resistivity and density of the previously characterized specimens. Replicate runs were performed to further study the imprecision of the comparative apparatus. These measurements confirm our estimate of imprecision with this comparative apparatus to be  $\pm 1\%$  for thermal conductivity. The measured results on the thirteen graphite specimens are listed in Table 3.

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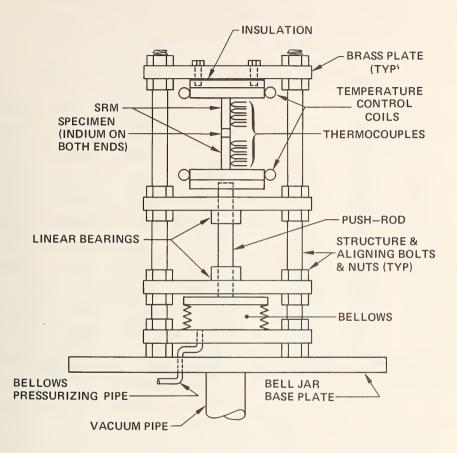


Figure 12. Ambient-temperature, comparative thermal conductivity apparatus for small specimens.

Specimen Identification	T (k)	λ (W•m <sup>-1</sup> •K <sup>-1</sup> )	Electrical <sup>a</sup> Resistivity (μΩ•m)	Density <sup>b</sup> 6.4 mm spec	(g*cm <sup>-3</sup> ) 5 cm spec	Lorenz Ratio (10 <sup>-8</sup> V <sup>2</sup> •K <sup>-2</sup> )
310-0	296.0	72.8	17.5	1.693	1.698	430
н	296.2	72.2	17.5		н	426
	296.7	73.2	17.5			432
310-1	296.8	73.0	17.5	1.689		430
310-2	296.7	73.7	17.5	1.690	н	435
310-3	296.9	73.8	17.5	1.698		435
310-4	296.4	74.4	17.5	1.702	н	439
520	295.6	106.4	12.9	1.731	1.739	464
"	295.8	104.9	12.9			457
н	296.7	105.7	12.9	U II	н	460
400	296.3	121.9	11.3	1.755	1.746	465
	296.4	121.1	11.3			462
410	296.2	120.0	11.6	1.736	1.724	470
	296.6	118.0	11.6			461
102	296.4	112.6	12.6	1.755	1.751	479
	296.8	112.8	12.6	"	"	479
401	296.2	102.5	13.0	1.716	1.703	450
	296.4	102.7	13.0			450
100	297.0	97.0	14.4	1.755	1.755	470
311	296.5	95.1	13.9	1.722	1.711	446
312	296.9	92.5	15.3	1.757	1.747	476
		5230	2000	20,07	/ //	

Table 3. Thermal Conductivity, Electrical Resistivity, Density, and Lorenz Ratio for Thirteen AXM Graphite Specimens.

<sup>a</sup>These measurements were performed on 50 mm long specimens from which the 7 mm specimens were obtained.  $^{\rm b} These$  densities are for T = 293 K and not for the listed temperature.

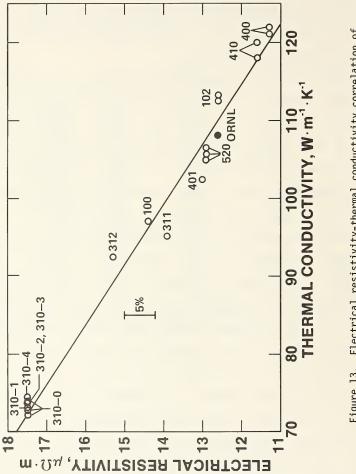
The thermal conductivities have a mean of 96.5 W·m<sup>-1</sup> K<sup>-1</sup> with a range of 72 to 122 or about <u>+25%</u>. The electrical resistivities have a mean value of 15.0  $\mu\Omega$ ·m, and range from 11.3 to 17.5 or about <u>+</u>20%. The mean of the Lorenz ratios,  $\rho\lambda/T$ , is 453 x 10<sup>-8</sup> V<sup>2</sup> K<sup>-2</sup> with a range from 426 to 479 or about <u>+</u>6%. Figure 13 is a plot of electrical resistivity versus thermal conductivity and clearly il-lustrates their interdependence.

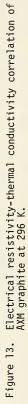
### 3.2 Correlations

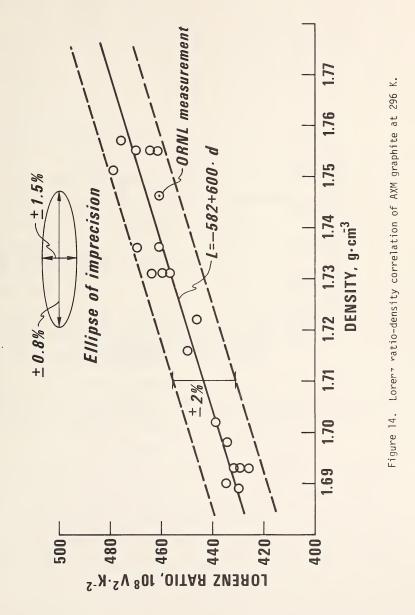
In Phase I, a strong intra-rod correlation of electrical resistivity and density, but hardly any inter-rod correlation, was reported. The above results suggest the same would be true of thermal conductivity and density. A plot was made of these variables which shows this to be the case. The possibility of a Lorenz ratio dependence on electrical resistivity or density was investigated. Figures 14 and 15 are plots of Lorenz ratio versus these variables.

Figure 14 indicates that Lorenz ratio at ambient temperature is a linear function of density. The equation for this relationship is  $L = -582 + 600 \cdot d$ , with d in g/cm<sup>3</sup>. The total range of deviations in L from this line is  $\pm 2\%$ . This is within the combined experimental error of determining L and d. Thus, for these specimens it would be possible to predict thermal conductivity to better than 2% at ambient temperature from measurements of electrical resistivity and density. The densities used here are based on simple volumetric and weight measurements on the 6.4 mm diameter specimens. The agreement of the data with the line in Figure 14 is consistent with the combined imprecision and uncertainty of the various measurements. The imprecision in the measurement of  $\lambda$  is estimated to be  $\pm 1\%$ , and the imprecision in the measurement of  $\rho$  is estimated to be about  $\pm 0.5\%$ . Thus the imprecision in L is about  $\pm 1.5\%$ , since the imprecision in measuring T is negligible. Note that the uncertainty in determining  $\lambda$  and  $\rho$ , caused

-25-







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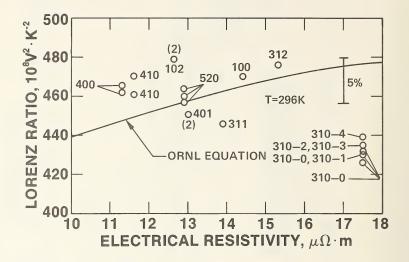


Figure 15. Lorenz ratio-electrical resistivity data of AXM graphite at 296 K.

by the form factor determinations, cancels in the calculation of L. The uncertainty in measuring density on these specimens is estimated to be about <u>+0.8%</u>. The corresponding ellipse of imprecision and error band are shown in Figure 14, and it is noted that none of the data points lie outside the estimated error band. A preliminary result of a single measurement performed at ORNL (Peyton Moore, Private Communication) is included in Figure 14, and is in excellent agreement with the NBS measurements.

The scatter of the data points in Figure 15 is large enough that one cannot be sure of any Lorenz ratio dependence on electrical resistivity. In an ORNL report by McElroy, et al. [3] an equation is given describing the observed relationship between thermal and electrical conductivities for a group of graphite specimens with resistivities ranging from 5 to 100  $\mu$ 0·m. This equation reduces to L = (1.56 x 10<sup>-3</sup> - 2.66 x 10<sup>-9</sup>/ $\rho$ )/T and is shown on Figure 15. The agreement of this line with the average of our data for resistivities from 11 to 18  $\mu$ 0·m is good, but the trend with respect to  $\rho$  is inconsistent with our data. However, McElroy indicated that their equation described their data to within <u>+</u>8% and our data fall within that range.

The Lorenz ratio-density behavior of the AXF and FXI materials was also investigated. Lorenz ratio results for these five specimens deviate by as much as 16% from the line obtained for the AXM material, as shown in Figure 16.

## 3.3 Conclusions and Recommendations

The work described in Phase II shows that it is possible to characterize this lot of graphite, including AXM-5Q1 as received, AXM-5Q1 after anneal at 3180 °C, AXM-5Q and AXM-9Q1, to obtain ambient temperature thermal conductivity values accurate to better than <u>+</u>2% using simple electrical resistivity and density measurements on each specimen. It is now believed that graphite material

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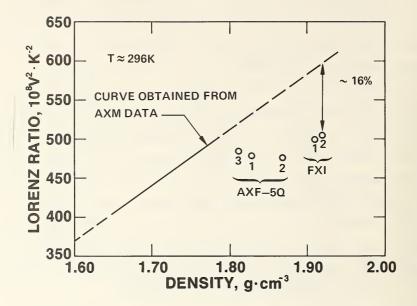


Figure 16. Lorenz ratio-density data of FXI and AXF compared to AXM graphite values.

which is sufficiently homogeneous in transport properties to avoid individual specimen characterization is not available. The inhomogeneities can be reduced through a selection process, but this will necessitate the type of characterization described above.

Further evidence that our inhomogeneity results based on electrical resistivity measurements are not atypical is found in a report by Wilkes [4]. This report is the result of an investigation on the effects of radiation on the transport properties of graphite materials. As part of this research, the electrical resistivity changes along the lengths of numerous rods were determined. Changes on the order of 1%/cm were not uncommon. Such changes are comparable to those observed on the rods reported here.

Although the correlation between L and d reported here for ambient temperature is highly encouraging; considerable research still needs to be performed before we can be certain that the correlation applies to the entire temperature range.

4. PHASE III - COOPERATIVE MEASUREMENTS

In view of the encouraging result found in Phase I and II, that thermal conductivity could be accurately predicted at room temperature from simple electrical resistivity and density measurements, the decision was made to proceed with wide-scale cooperative measurements at temperatures from 4 to 3000 K. This phase of the program was conducted in cooperation with the CODATA Task Group on Thermophysical Properties. Dr. M. Minges of the Air Force Materials Laboratory, Dayton, Ohio was instrumental in organizing this phase. For an overall description of this effort, including other SRM's, see reference 5.

# 4.1 Specimen Characterization

In preparation for the specimen distribution, a large number of rods were characterized for electrical resistivity and density. This step was completed

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with the measurement of 50 rods of each available diameter (6.4, 12.7, and 25.4 mm). The average densities of these rods are presented in Table 4. These rods were notched and assigned numbers as follows: The "one's" digit was notched in the side of end 1, the "ten's" digit was notched on end 1, and the "hundred's" digit was notched on end 2. The one's digit was never "zero", so end 1 is always uniquely defined. The 1500 measured electrical resistivities as a function of position along these specimens are plotted in Figures 17 through 33. Since the variation of resistivity along each rod was of primary interest, no attempt was made to measure all of these rods at exactly the same temperature. The measurements were conducted in a thermally lagged box which slowly varied in temperature with room conditions. The box temperature for each set of measurements is indicated in parentheses on Figures 17 through 33. The temperature in the box, during the measurement of each specimen, remained constant to within 0.05 °C. The most homogeneous of these specimens will be used for transport property SRM's. Corrections to a fixed temperature (say 20 °C) can be made by using the slope indicated in Figures 1, 2, and 3, 0.034  $\mu\Omega^{\circ}m/K$ . In all cases, the correction to 20 °C is less than 1%.

### 4.2 Specimen Distribution and Results

Dr. Minges (AFML) arranged the participation of 18 experimentalists from around the world. Specimens were prepared to the desired approximate sizes and distributed to these participants. When possible, further electrical resistivity and density characterization measurements were performed at NBS on the actual specimens distributed. In some cases, adjacent pieces were characterized. Nearly all of the participants that were able to report results also published their results in the open literature and are listed in the references. Up to the present time, some reported their results only to the committee, and these will be referred to as participant 1, 2, etc., to maintain confidentiality. A summary of the reported results and pre-characterization data is given in Table 5.

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Specimen		Density, g/cm <sup>3</sup>	
Identification	6.4 mm dia	12.7 mm dia	25.4 mm dia
001	1.695	1.718	1.696
002	1.729	1.715	1.728
003	1.733	1.731	1.727
004	1.733	1.722	1.728 1.720
005 006	1.753	1.726 1.718	1.709
011	1.732	1.728	1.704
012	1.737	1.725	1.706
013	1.734	1.753	1.728
014	1.734	1.737	1.718
015	1.736	1.737	1.709
016	1.746	1.717	1.710
021	1.734	1.735	1.722
022	1.731	1.743	1.741
023	1.734	1.718	1.732
024	1.742	1.741	1.736
025 026	1.739 1.744	1.769 1.742	1.709
020	1.725	1.738	1.728
032	1.722	1.728	1.716
033	1.747	1.742	1.706
034	1.728	1.745	1.731
035	1.730	1.737	1.722
036	1.739	1.732	1.710
041	1.717	1.720	1.717
042	1.750	1.743	1.711
043	1.739	1.712	1.706
044	1.741	1.744	1.728
045	1.719	1.744	1.739
046 051	1.743	1.709	1.737
052	1.746	1.726	1.735 1.726
053	1.754	1.725	1.729
054	1.737	1.740	1.717
055	1.749	1.740	1.716
056	1.736	1.725	1.710
061	1.717	1.725	1.735
062	1.748	1.742	1.723
063	1.749	1.731	1.718
064	1.737	1.728	1.732
065	1.762	1.738	1.708
066	1.754	1.723	1.727
101 102	1.726	1.727	1.739 1.731
102	1.709	1.728	1.740
103	1.747	1.742	1.740
104	1.719	1.714	1.740
106	1.720	1.731	1.733
111	1.730	1.728	1.737
112	1.719	1.724	1.745
		• <u> </u>	

Table 4. Densities of 30 cm long AXM-501 graphite rods at 20°C.

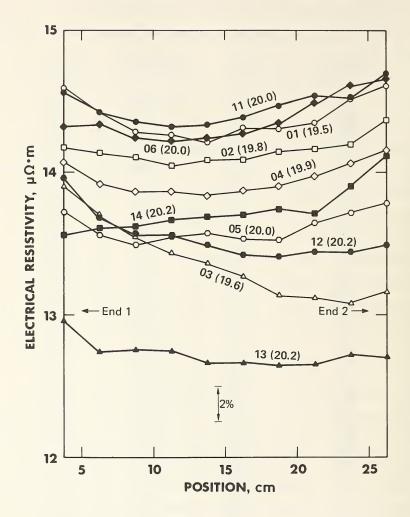


Figure 17. Electrical resistivity versus position of 25.4 mm diameter AXM graphite rods.

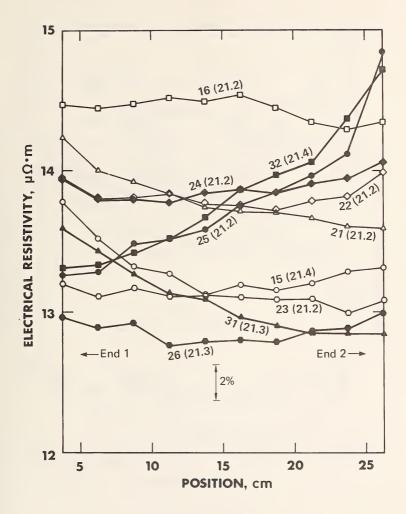


Figure 18. Electrical resistivity versus position of 25.4 mm diameter AXM graphite rods.

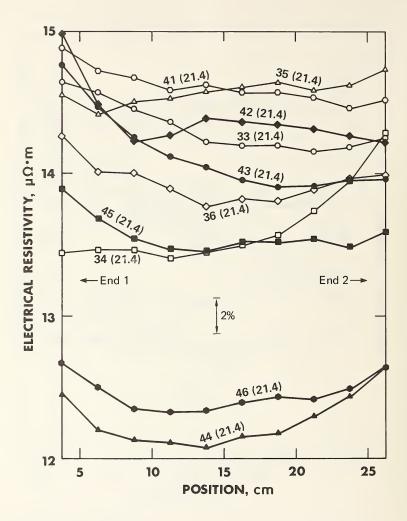


Figure 19. Electrical resistivity versus position of 25.4 mm diameter AXM graphite rods.

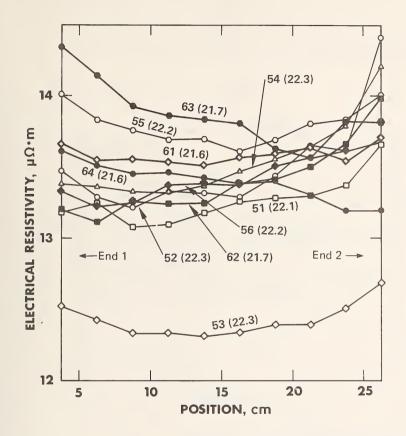


Figure 20. Electrical resistivity versus position of 25.4 mm diameter AXM graphite rods.

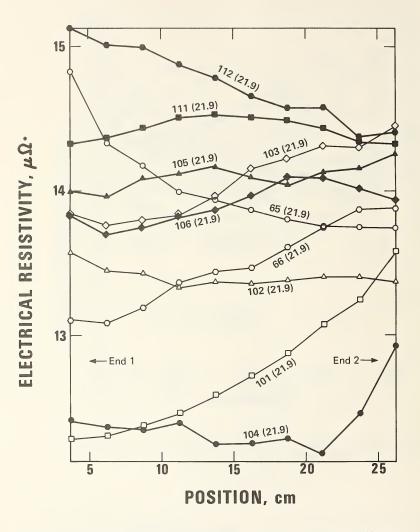


Figure 21. Electrical resistivity versus position of 25.4 mm diameter AXM graphite rods.

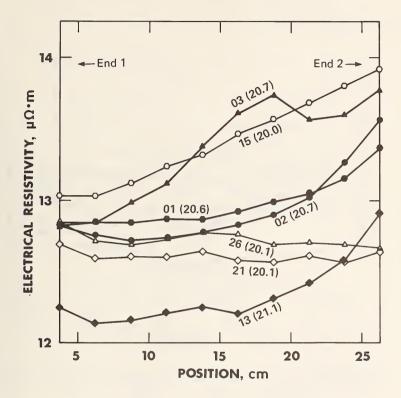


Figure 22. Electrical resistivity versus position of 12.7 mm diameter AXM graphite rods.

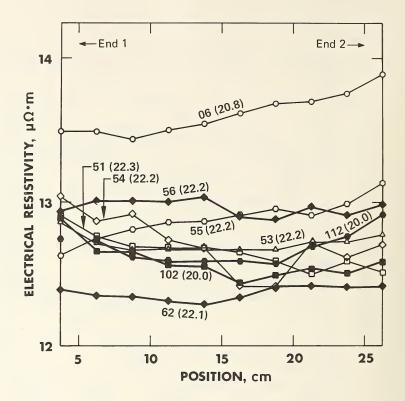


Figure 23. Electrical resistivity versus position of 12.7 mm diameter AXM graphite rods.

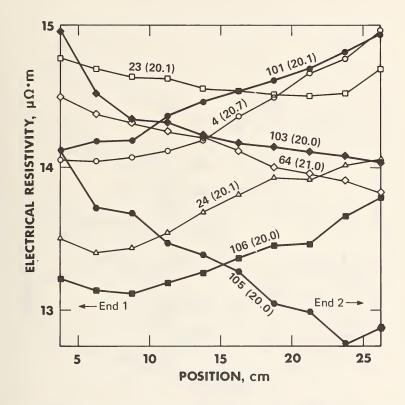


Figure 24. Electrical resistivity versus position of 12.7 mm diameter AXM graphite rods.

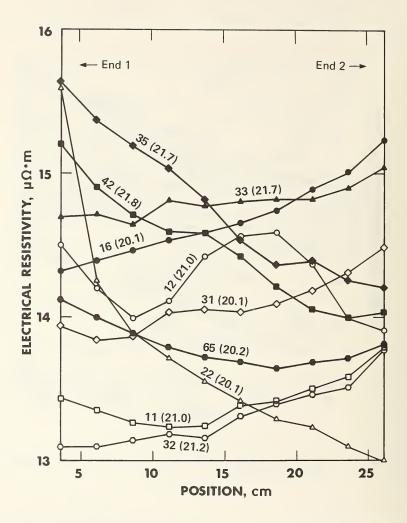


Figure 25. Electrical resistivity versus position of 12.7 mm diameter AXM graphite rods.

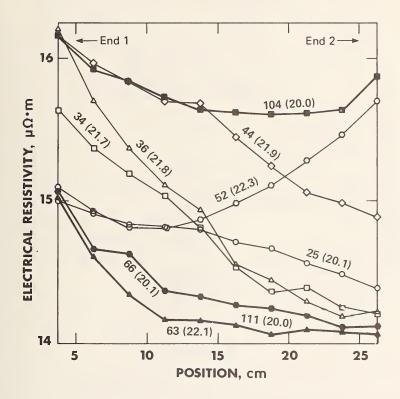


Figure 26. Electrical resistivity versus position of 12.7 mm diameter AXM graphite rods.

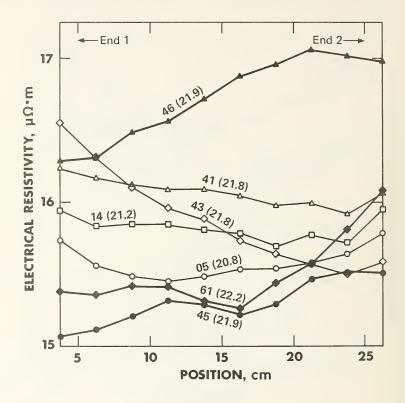


Figure 27. Electrical resistivity versus position of 12.7 mm diameter AXM graphite rods.

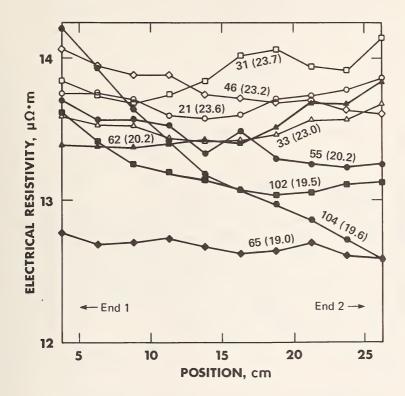


Figure 28. Electrical resistivity versus position of 6.4 mm diameter AXM graphite rods.

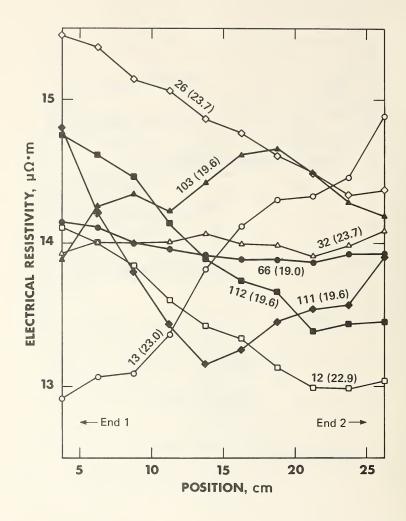


Figure 29. Electrical resistivity versus position of 6.4 mm diameter AXM graphite rods.

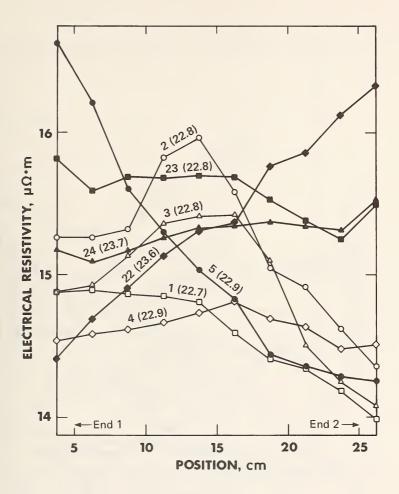


Figure 30. Electrical resistivity versus position of 6.4 mm diameter AXM graphite rods.

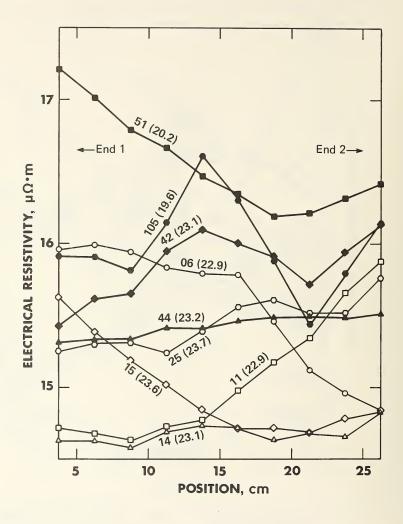


Figure 31. Electrical resistivity versus position of 6.4 mm diameter AXM graphite rods.

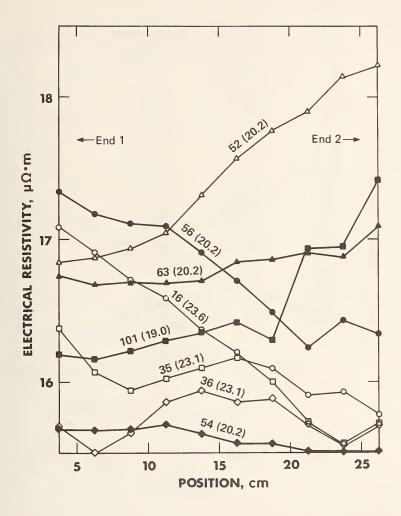


Figure 32. Electrical resistivity versus position of 6.4 mm diameter AXM graphite rods.

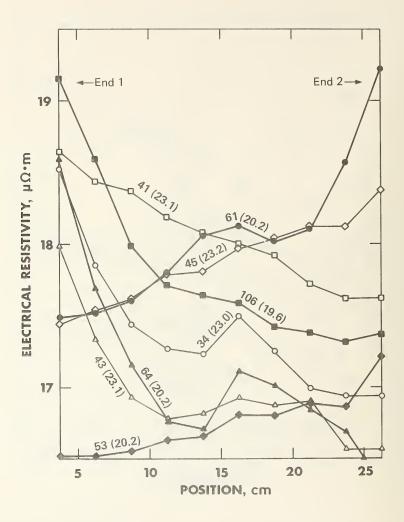


Figure 33. Electrical resistivity versus position of 6.4 mm diameter AXM graphite rods.

Table 5. Summary of reported results on AXM-5Q1 graphite.

			Character	Characterization Data		
Participant (Ref.)	*Properties Measured	Temperature Range (K)	Density (g/cm <sup>3</sup> )	Electrical Resistivity (μΩ•m)		
(Ref.) Brandt (6) Brandt (6) Hust (7) Hust (7)13 specimens Isaacs 1 (8) Maglic (9) Mirkovich (10) Moore (11) Taylor (12) Taylor (12) Taylor (12) Taylor (13) Taylor (13) Participant 1 Participant 3 Participant 4 Participant 4 Participant 5	Measured D D λ,ρ,S λ,ρ,S λ,ρ,C D D Δ,S λ,ρ,C,α,D " C ρ,C λ,ρ D Δ,ρ Δ,ρ Δ Δ Δ Δ Δ Δ Δ Δ Δ Δ Δ Δ Δ	Range (K) 1100-2315 1085-2240 4-300 296 only 83-290 480-1713 273-1073 80-950 400-2400 " 300-2500 530-2933 481-2247 373-873 1350-2900 300-2100 553-2460 593-1343 363-1067	(g/cm <sup>3</sup> ) 1.733 1.723 1.728 1.722 16.9 to 17.6 1.721 1.755 1.724 1.755 1.724 1.770 1.744 1.789 1.698 1.751 1.707 1.757 1.706 1.738 1.666 1.666 1.666 1.696	(μΩ·m) 14.31 14.88 12.58 14.46 11.3 to 17.5 15.49 14.59 15.00 12.60 13.80 13.00 14.40 13.12 15.29  18.81 13.39 15.00 15.00 14.20		
Participant 5 Participant 5 Participant 5	λ λ λ	343-1124 1085-2605 1077-2615	1.708 1.717 1.716	13.50 13.91 14.40		

\*D = thermal diffusivity,  $\lambda$  = thermal conductivity  $\rho$  = electrical resistivity,  $\alpha$  = thermal expansion, C = specific heat S = Seebeck coefficient

The data from the participants are reported in various forms. Some data are in SI units and others are in British units, some are corrected for thermal expansion and others are not, some are closely-spaced direct experimental values and others are more widely-spaced smoothed values. To achieve sets of data for convenient comparison and representation, the following modifications were performed:

- 1. All data were converted to SI units.
- All data known to be uncorrected for thermal expansion were corrected with the following equations:

$$\lambda = \lambda_{obs} / (1 + \Delta L / L)$$

 $\rho = \rho_{obs}(1 + \Delta L/L)$ 

 $D = D_{obs}(1 + \Delta L/L)^2$ 

The values for  $\Delta L/L$  were taken from Touloukian et al. (14). The corrections amount to -0.2% at 0 K, 0% at 293 K, and 2.1% at 2600 K.

3. When very closely-spaced data were reported, a subset was selected that a) covered the entire reported range, b) reflected the scatter in the entire data set, and c) was spaced in temperature for convenient graphical illustration with no loss in the detailed temperature dependence.

All of the data reported and modified in this way are given in Tables 6 through 19.

5. PHASE IV - DATA ANALYSIS AND RECOMMENDED VALUES

# 5.1 Thermal Conductivity

Figures 34 through 37 show the thermal conductivity data from Tables 6 through 19. It is clear that large variations in  $\lambda$  exist throughout the

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TABLE 6. THERMOPHYSICAL PROPERTIES FOR AXM-5Q1 GRAPHITE AS REPORTED BY BRANDT (6).

TEMPERATURE (K)	THERMAL CONDUCTIVITY (W•m-1•K-1)	ELECTRICAL RESISTIVITY (µΩ∙m)	SPECIFIC HEAT (J•g <sup>-1</sup> •K <sup>-1</sup> )	DENSITY (kg·m <sup>-3</sup> )	THERMAL DIFFUSIVITY (µm <sup>2•</sup> s <sup>-1</sup> )	SEEBECK COEFFICIENT (µV•K <sup>-1</sup> )
1110. 1202. 1333. 1450. 1669. 1783. 1910. 2045. 2163. 2275. 2315. 1333.	. 42F, RESISTI		DENSITY = 1733 DENSITY = 1723		$15.82 \\ 14.91 \\ 13.72 \\ 12.91 \\ 12.26 \\ 11.83 \\ 11.05 \\ 10.65 \\ 10.65 \\ 10.65 \\ 10.20 \\ 9.83 \\ 9.59 \\ 9.44 \\ 13.04 \\ 13.04 \\ 13.04 \\ 14.59 \\ 13.55 \\ 12.91 \\ 11.89 \\ 11.52 \\ 10.73 \\ 10.08 \\$	

TABLE 7. THERMOPHYSICAL PROPERTIES FOR AXM-5Q1 GRAPHITE AS REPORTED BY PARTICIPANT 1, SPEC. 62, ROOM TEMPERATURE ELECTRICAL RESISTIVITY = 12.40  $_{\mu} \Omega^* m$ , DENSITY = 1757 kg·m^-3.

TEMPERATURE (K)	THERMAL CONDUCTIVITY (W•m <sup>-1</sup> •K <sup>-1</sup> )	ELECTRICAL RESISTIVITY (μΩ°m)	SPECIFIC HEAT (J•g <sup>-1</sup> •K <sup>-1</sup> )	DENSITY (kg·m <sup>-3</sup> )	THERMAL DIFFUSIVITY (µm <sup>2</sup> •s <sup>-1</sup> )	SEEBECK COEFFICIENT (µV•K-1)
400.0			999.4			
500.0			1207.3			
600.0			1395.4			
700.0			1533.4			
800.0			1651.5			
900.0			1739.7			
1000.			1797.8			
1200.			1824.4			

THERMAL TEMPERATURE CONDUCTIVITY (K) (W•m <sup>-1</sup> •K <sup>-1</sup> )	ELECTRICAL RESISTIVITY (μΩ°m)	SPECIFIC HEAT (J·g <sup>-1</sup> ·K <sup>-1</sup> )	DENSITY (kg•m <sup>-3</sup> )	THERMAL DIFFUSIVITY (µm <sup>2</sup> •s <sup>-1</sup> )	SEEBECK COEFFICIENT (µV•K-1)
SPEC. 310-0					
296.0 .7280E+02	17.50		1693.		
SPEC. 310-1					
296.8 .7300E+02	17.50		1689.		
SPEC. 310-2	17 50		1000		
296.7 .7370E+02 SPEC. 310-3	17.50		1690.		
296.9 .7380E+02	17.50		1698.		
SPEC. 310-4					
296.4 .7440E+02	17.50		1702.		
SPEC. 520					
296.7 .1057E+03	12.90		1731.		
SPEC. 400 296.3 .1219E+03	11 30		1755.		
SPEC. 410	11.50		1/55.		
296.6 .1180E+03	11.60		1736.		
SPEC. 102					
296.4 .1126E+03	12.60		1755.		
SPEC. 401	12 00		1716		
296.2 .1025E+03 SPEC. 100	13.00		1716.		
297.0 .9700E+02	14 40		1755.		
SPEC. 311	14.40		1/ 55.		
296.5 .9510E+02	13.90		1722.		
SPEC. 312					
296.9 .9250E+02	15.30		1757.		

TABLE 9. THERMOPHYSICAL PROPERTIES FOR AXM-5Q1 GRAPHITE AS REPORTED BY HUST (7).

TEMPERATURE (K)		LECTRICAL ESISTIVITY (µΩ∘m)	SPECIFIC HEAT (J•g <sup>-1</sup> •K <sup>-1</sup> )	DENSITY (kg∘m <sup>-3</sup> )	THERMAL DIFFUSIVITY (µm <sup>2</sup> •s <sup>-1</sup> )	SEEBECK COEFFICIENT (µV•K−1)
6.000 7.000 8.000 9.000 12.00 14.00 14.00 25.00 35.00 40.00 45.00 45.00 45.00 50.00 60.00 70.00 90.00 100.0 120.0 140.0 120.0 140.0 120.0 140.0 220.0 240.0 280.0	<ul> <li>013, RESISTIVIT</li> <li>.6332E-01</li> <li>.9258E-01</li> <li>.1293E+00</li> <li>.1293E+00</li> <li>.284E+00</li> <li>.3687E+00</li> <li>.5521E+00</li> <li>.7845E+01</li> <li>.403E+01</li> <li>.2465E+01</li> <li>.3687E+01</li> <li>.3687E+01</li> <li>.2465E+01</li> <li>.3687E+02</li> <li>.3735E+02</li> <li>.416E+02</li> <li>.5727E+02</li> <li>.6927E+02</li> <li>.995E+02</li> <li>.1050E+03</li> <li>.1052E+00</li> <li>.8887E+00</li> <li>.162E+01</li> <li>.3096E+00</li> <li>.4625E+00</li> <li>.66251E+02</li> <li>.304E+02</li> <li>.4636E+02</li> <li>.7266E+02</li> <li>.7266E+02</li> <li>.7266E+02</li> <li>.7266E+02</li> <li>.7266E+02</li> <li>.7266E+02</li> <li>.7266E+02</li> <li>.8807E+02</li> <li>.8807E+0</li></ul>	26.58 26.54 26.49 26.49 26.43 26.25 26.11 25.97 25.82 25.67 25.27 24.85 24.03 23.61 23.21 22.42 21.68 20.97 20.30 19.67 18.55 17.54 16.51 15.86 15.15 15.86 15.15 14.51 13.93 13.41 12.92				$\begin{array}{c} -3.294\\ -3.870\\ -4.410\\ -4.932\\ -5.418\\ -6.300\\ -7.092\\ -7.758\\ -8.334\\ -8.802\\ -9.954\\ -9.918\\ -9.918\\ -9.918\\ -9.918\\ -9.918\\ -9.918\\ -9.306\\ -8.856\\ -7.812\\ -6.732\\ -5.668\\ -4.716\\ -3.816\\ -2.214\\828\\ -4.716\\ -3.816\\ -2.214\\828\\ -3.006\\ 3.600\\ 4.356\\ -2.214\\828\\ -3.924\\ -4.356\\ -2.952\\ -3.924\\ -4.356\\ -2.952\\ -3.924\\ -4.356\\ -7.0$

TABLE 10. THERMOPHYSICAL PROPERTIES FOR AXM-5Q1 GRAPHITE AS REPORTED BY ISAACS (8), SPEC. 103-2, ROOM TEMPERATURE ELECTRICAL RESISTIVITY = 15.49 μΩ·m, DENSITY = 1721 kg·m<sup>-3</sup>.

TEMPERATURE (K)	THERMAL CONDUCTIVITY (W•m-1.K-1)	ELECTRICAL RESISTIVITY (µΩ*m)	SPECIFIC HEAT (J•g <sup>-1</sup> •K <sup>-1</sup> )	DENSITY (kg•m <sup>-3</sup> )	THERMAL DIFFUSIVITY (µm <sup>2•</sup> s <sup>-1</sup> )	SEEBECK COEFFICIENT (µV•K-1)
82.80			117.			
89.96			156.			
95.38			147.			
111.2			180.			
125.5			223.			
142.7			265.			
160.5			308.			
181.9			423.			
194.3			459.			
215.7			573.			
239.0			659.			
260.8			760.			
279.6			875.			
289.4			955.			

TABLE 11. THERMOPHYSICAL PROPERTIES FOR AXM-5Q1 GRAPHITE AS REPORTED BY MAGLIC (9), SPEC. 4, ROOM TEMPERATURE ELECTRICAL RESISTIVITY = 14.59  $_{\mu\Omega}$ ·m, DENSITY = 1755 kg·m^-3.

TEMPERATURE (K)	THERMAL CONDUCTIVITY (W•m <sup>-1</sup> •K <sup>-1</sup> )	ELECTRICAL RESISTIVITY (μΩ•m)	SPECIFIC HEAT (J•g <sup>-1</sup> •K <sup>-1</sup> )	DENSITY (kg·m <sup>-3</sup> )	THERMAL DIFFUSIVITY (µm <sup>2</sup> •s <sup>-1</sup> )	SEEBECK COEFFICIENT (µV·K-1)
480.0					41.50	
577.0					33.50	
677.0					28.50	
762.0					25.10	
866.0					21.50	
996.0					18.90	
1112.					16.90	
1224.					15.70	
1339.					14.60	
1458.					13.60	
1558.					12.70	
1647.					12.20	
1713.					11.80	

TABLE 12. THERMOPHYSICAL PROPERTIES FOR AXM-5Q1 GRAPHITE AS REPORTED BY MIRKOVICH (10), SPEC. 112B, ROOM TEMPERATURE ELECTRICAL RESISTIVITY = 15.00  $\mu\Omega^*m$ , DENSITY = 1724 kg·m<sup>-3</sup>.

TEMPERATURE (K)	THERMAL CONDUCTIVITY (W•m-1•K-1)	ELECTRICAL RESISTIVITY (µΩ°m)	SPECIFIC HEAT (J•g <sup>-1</sup> •K <sup>-1</sup> )	DENSITY (kg•m <sup>-3</sup> )	THERMAL DIFFUSIVITY (µm <sup>2</sup> •s <sup>-1</sup> )	SEEBECK COEFFICIENT (µV•K <sup>-1</sup> )
273.2					72.50	
323.2					58.10	
373.2					48.70	
473.2					37.60	
573.2					30.30	
673.2					25.70	
773.2					22.40	
873.2					19.70	
973.1					17.60	
1073.					16.10	

TABLE	13.	THERMOPHYSICAL PROPERTIES FOR AXM-5Q1 GRAPHITE AS REPORTED BY MOORE (11),	
		SPEC. 102, ROOM TEMPERATURE ELECTRICAL RESISTIVITY = 12.60 μΩ·m, DENSITY	
		$= 1770 \text{ kg} \cdot \text{m}^{-3}$ .	

TEMPERATURE (K)	THERMAL CONDUCTIVITY (W•m <sup>-1</sup> •K <sup>-1</sup> )	ELECTRICAL RESISTIVITY (µΩ*m)	SPECIFIC HEAT (J•g-1•K-1)	DENSITY (kg•m <sup>-3</sup> )	THERMAL DIFFUSIVITY (µm <sup>2</sup> •s-1)	SEEBECK COEFFICIENT (µV•K-1)
80.00	•2960E+02					-5.094
100.0	.4300E+02					-3.564
120.0	.5610E+02					-2.106
140.0	.6800E+02					774
160.0	•7840E+02					.414
180.0	.8710E+02					1.458
200.0	.9410E+02					2.340
220.0	.9950E+02					3.078
240.0	.1035E+03					3.762
260.0	.1062E+03					4.122
280.0	.1078E+03					4.446
300.0	.1084E+03	12.51				4.644
320.0	.1083E+03					4.716
340.0	.1082E+03					4.698
360.0	.1075E+03					4.572
380.0	.1065E+03	10.00				4.356
400.0	-1053E+03	10.92				4.050
500.0	•9830E+02	10.01				1.782
600.0	-8830E+02	9.36				-1.098
700.0	•8010E+02	8.93				-3.654
800.0 900.0	.7460E+02 .6820E+02	8.66				-5.724
		8.52				-6.372
950.0	.6590E+02					

TABLE 14. THERMOPHYSICAL PROPERTIES FOR AXM-5Q1 GRAPHITE AS REPORTED BY PARTICIPANT 5.

TEMPERATURE (K)	THERMAL CONDUCTIVITY (W·m <sup>-1</sup> ·K <sup>-1</sup> )	ELECTRICAL RESISTIVITY (µΩ°m)	SPECIFIC HEAT (J•g <sup>-1</sup> •K <sup>-1</sup> )	DENSITY (kg·m <sup>-3</sup> )	THERMAL DIFFUSIVITY (µm <sup>2</sup> •s <sup>-1</sup> )	SEEBECK COEFFICIENT (µV•K <sup>-1</sup> )
SPEC 363.2 372.0 538.7 803.7 1067.	<ul> <li>006(CRA-1),</li> <li>.8732E+02</li> <li>.8920E+02</li> <li>.8070E+02</li> <li>.6225E+02</li> <li>.5029E+02</li> </ul>	RESISTIVITY =	14.20, DENSITY	= 1696		
343.2 508.7 807.0 1124.	.9136E+02 .8387E+02 .6614E+02 .5058E+02		13.50, DENSITY			
1085. 1380. 1929. 2606.	.5058E+02 .4222E+02 .3401E+02 .2968E+02		= 13.91, DENSITY			
SPEC 1077. 1115. 1366. 1524. 1877. 2001. 2613. 2615.	<ul> <li>006(RIA-1A)</li> <li>5361E+02</li> <li>4799E+02</li> <li>3920E+02</li> <li>3876E+02</li> <li>3285E+02</li> <li>2853E+02</li> <li>2853E+02</li> <li>3041E+02</li> </ul>	, RESISTIVITY =	= 14.40, DENSITY	' = 1716		

TABLE 15. THERMOPHYSICAL PROPERTIES FOR AXM-5Q1 GRAPHITE AS REPORTED BY PARTICIPANT 2, SPEC. 41B, ROOM TEMPERATURE ELECTRICAL RESISTIVITY = 18.81  $\mu$ G·m, DENSITY = 1706 kg·m<sup>-3</sup>.

TEMPERATURE (K)	THERMAL CONDUCTIVITY (W•m <sup>-1</sup> •K <sup>-1</sup> )	ELECTRICAL RESISTIVITY (µΩ•m)	SPECIFIC HEAT (J•9 <sup>-1</sup> •K <sup>-1</sup> )	DENSITY (kg•m <sup>-3</sup> )	THERMAL DIFFUSIVITY (µm <sup>2</sup> ·s <sup>-1</sup> )	SEEBECK COEFFICIENT (µV•K−1)
1350.		11.23	1933.			
1400.		11.26	1947.			
1500.		11.33	1969.			
1600.		11.43	1990.			
1700.		11.56	2008.			
1800.		11.70	2022.			
1900.		11.86	2039.			
2000.		12.02	2053.			
2100.		12.20	2066.			
2200.		12.36	2076.			
2300.		12.53	2086.			
2400.		12.71	2096.			
2500.		12.89	2104.			
2600.		13.06	2112.			
2700.		13.23	2120.			
2800.		13.40	2122.			
2900.		13.58	2130.			

TABLE 16. THERMOPHYSICAL PROPERTIES FOR AXM-5Q1 GRAPHITE AS REPORTED BY P. RTICIPANT 3, SPEC. 52, ROOM TEMPERATURE ELECTRICAL RESISTIVITY = 13.39 μΩ·m, DENSITY = 1738 kg·m<sup>-3</sup>.

TEMPERATURE (K)	THERMAL CONDUCTIVITY (W•m-1•K-1)	ELECTRICAL RESISTIVITY (µΩ•m)	SPECIFIC HEAT (J•g <sup>-1</sup> •K <sup>-1</sup> )	DENSITY (kg•m <sup>-3</sup> )	THERMAL DIFFUSIVITY (µm <sup>2</sup> •s <sup>-1</sup> )	SEEBECK COEFFICIENT (µV•K-1)
300.0 400.0 500.0 600.0 700.0 800.0 1000. 1100. 1200. 1300. 1300. 1500. 1500. 1500. 1600. 1700. 1800. 2000.	.9240E+02 .8770E+02 .8150E+02 .7180E+02 .710E+02 .6690E+02 .6690E+02 .5390E+02 .5390E+02 .5030E+02	13.62 12.14 10.99 10.25 9.70 9.36 9.10 8.90 8.81 8.83 8.97 9.09 9.26 9.42 9.61 9.80 9.98 9.98 10.16		(-3		
2100.		10.34				

TABLE 17. THERMOPHYSICAL PROPERTIES FOR AXM-5Q1 GRAPHITE AS REPORTED BY TAYLOR (12).

$\begin{array}{llllllllllllllllllllllllllllllllllll$	SPECIFIC HEAT (J•g-1•K-1)	DENSITY (kg•m <sup>-3</sup> )	THERMAL DIFFUSIVITY (µm2•s-1)	SEEBECK COEFFICIENT (µV•K-1)
SPEC. 3A-1, RESISTIVITY = 13.80,           400.0         .9720E+02         12.13           500.0         .8920E+02         10.66           600.0         .8280E+02         10.36           700.0         .7730E+02         9.88           800.0         .250E+02         9.50	DENSITY = 1744 957. 1168. 1382. 1520. 1636.	1737. 1734. 1713. 1727. 1722.	58.50 44.00 34.60 29.40 25.70	
900.0         .6730E+02         9.34           1000.         .6280E+02         9.30           1100.         .5860E+02         9.31           1200.         .5470E+02         9.39           1300.         .5120E+02         9.48           1400.         .4820E+02         9.64	1726. 1797. 1859. 1905. 1942. 1975.	1718. 1714. 1709. 1705. 1700. 1696.	22.70 20.40 18.30 16.80 15.50 14.40	
1500.         .4560E+02         9.80           1600.         .4360E+02         9.96           1700.         .4210E+02         10.15           1800.         .4080E+02         10.35           1900.         .3980E+02         10.57	2002. 2028. 2050. 2070. 2087.	1692. 1687. 1682. 1677. 1673.	13.50 12.70 12.20 11.80 11.40	
20003880E+02 10.81 21003800E+02 11.04 22003740E+02 11.27 23003700E+02 11.46 24003650E+02 11.68 SPEC. 3A-2, RESISTIVITY = 13.00,	2100. 2111. 2127. 2140. 2155. DENSITY = 1789	1668. 1663. 1658. 1653. 1647.	11.10 10.80 10.60 10.50 10.30	
400.0         .1022E+03         11.28           500.0         .9530E+02         10.24           600.0         .8870E+02         9.54           700.0         .8230E+02         9.66           800.0         .7680E+02         8.76		1781. 1777. 1774. 1770. 1766.	60.00 45.90 36.20 30.60 26.60 23.60	
1000.         .6680E+02         8.54           1100.         .6250E+02         8.52           1200.         .5890E+02         8.57           1300.         .5530E+02         8.67           1400.         .5210E+02         8.78		1761. 1757. 1752. 1748. 1743. 1739.	21.20 19.20 17.70 16.30 15.20	
1500.         .4930E+02         8.94           1600.         .4710E+02         9.10           1700.         .4530E+02         9.26           1800.         .4360E+02         9.45           1900.         .4230E+02         9.65           2000.         .4140E+02         9.85		1734. 1729. 1725. 1720. 1715. 1710.	14.20 13.40 12.80 12.20 11.80 11.50	
21004060E+02 10.06 22004000E+02 10.29 23003940E+02 10.53 24003900E+02 10.77 SPEC. 001, RESISTIVITY = 14.40, D	ENSITY = 1698	1705. 1699. 1694. 1689.	11.30 11.10 10.90 10.70	
300.0         .1005E+03         14.30           400.0         .9350E+02         12.59           500.0         .8650E+02         11.48           600.0         .8040E+02         10.75           700.0         .7470E+02         10.31           800.0         .6980E+02         9.96	702.	1698. 1695. 1691. 1688. 1684. 1680.	84.30 57.60 43.70 34.50 29.20 25.40	
900.0         .6470E+02         9.76           1000.         .6040E+02         9.70           1100.         .5600E+02         9.71           1200.         .5220E+02         9.87           1300.         .4860E+02         10.13		1676. 1672. 1668. 1664. 1660.	22.40 20.10 18.10 16.50 15.10	
1400.         .4570E+02         10.34           1500.         .4330E+02         10.55           1600.         .4110E+02         10.76           1700.         .3960E+02         10.98           1800.         .3820E+02         11.20           1900.         .3700E+02         11.41		1655. 1650. 1646. 1641. 1636. 1631.	14.00 13.10 12.30 11.80 11.30 10.90	
2000.         .3610E+02         11.65           2100.         .3540E+02         11.85           2200.         .3640E+02         12.10           2300.         .3430E+02         12.44           2400.         .3430E+02         12.64           2500.         .3350E+02         12.64	2168.	1626. 1621. 1616. 1611. 1606. 1601.	10.60 10.30 10.10 9.90 9.80 9.70	

TEMPERATURE (K)	THERMAL CONDUCTIVITY (W*m <sup>-1</sup> *K <sup>-1</sup> )	ELECTRICAL RESISTIVITY (µΩ∘m)	SPECIFIC HEAT (J•g <sup>-1</sup> •K <sup>-1</sup> )	DENSITY (kg•m <sup>-3</sup> )	THERMAL DIFFUSIVITY (µm <sup>2</sup> •s <sup>-1</sup> )	SEEBECK COEFFICIENT (µV•K-1)
530.0 615.0 733.0 863.0 989.0 1113. 1315. 1423. 1575. 1717. 1890. 1982. 2155. 2318. 2410. 2503. 2515. 2705. 2810. 2933.	856E+02 7730E+02 7730E+02 6620E+02 5740E+02 55740E+02 4860E+02 4860E+02 3840E+02 3840E+02 3840E+02 3756E+02 3450E+02 3100E+02 2920E+02 2920E+02 2920E+02 2920E+02		DENSITY = 1751 DENSITY = 1707		37.40 31.30 25.90 22.30 19.50 17.90 15.40 14.40 13.20 12.50 11.10 11.20 10.70 9.82 9.87 8.80 9.35 8.30 8.31 8.62 41.30 33.50 16.50 14.70 13.60 12.80 12.80 12.80 12.80 12.80 12.80 12.80 12.80 12.80 12.80 12.90 11.10 11.90 11.10 10.70	

TABLE 19. THERMOPHYSICAL PROPERTIES FOR AXM-501 GRAPHITE AS REPORTED BY PARTICIPANT 4, SPEC. 111-END 1, ROOM TEMPERATURE ELECTRICAL RESISTIVITY = 15.00  $\mu \Omega^*m$ , DENSITY = 1666 kg·m^-3.

TEMPERATURE (K)	THERMAL CONDUCTIVITY (W•m <sup>-1</sup> •K <sup>-1</sup> )	ELECTRICAL RESISTIVITY (µΩ°m)	SPECIFIC HEAT (J•g <sup>-1</sup> •K <sup>-1</sup> )	DENSITY (kg·m <sup>-3</sup> )	THERMAL DIFFUSIVITY (µm <sup>2</sup> •s <sup>-1</sup> )	SEEBECK COEFFICIENT (µV•K <sup>-1</sup> )
553.2					29.60	
732.2 868.2					22.60 20.30	
1049.					16.70	
1225.					14.50	
1421.					12.80	
1598.					12.09	
1824. 2020.					10.60 10.90	
2220.					8.90	
2460.					8,10	
593.2					27.10	
868.2					19.20	
1005.					17.30	
1188. 1343.					15.70 13.60	
1040.					13.00	

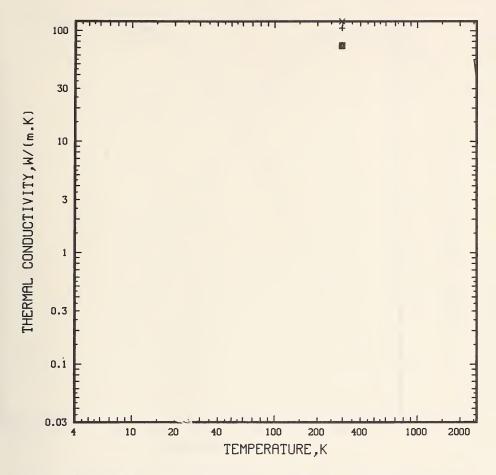


Figure 34. Thermal conductivity data from Hust (7) on seven AXM-5Q1 graphite specimens at 296 K.

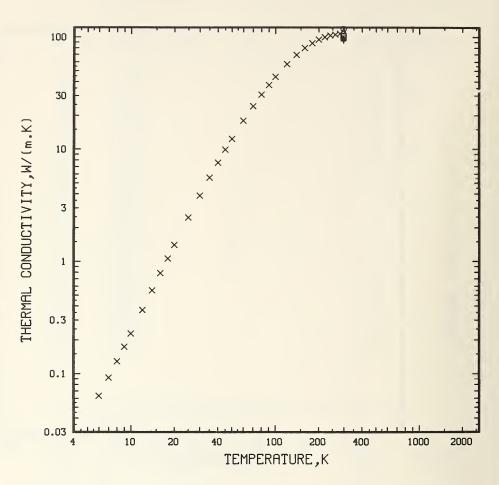


Figure 35. Thermal conductivity data from Hust (7) on seven AXM-5Q1 graphite specimens, six at 296 K and one specimen from 6 to 300 K.

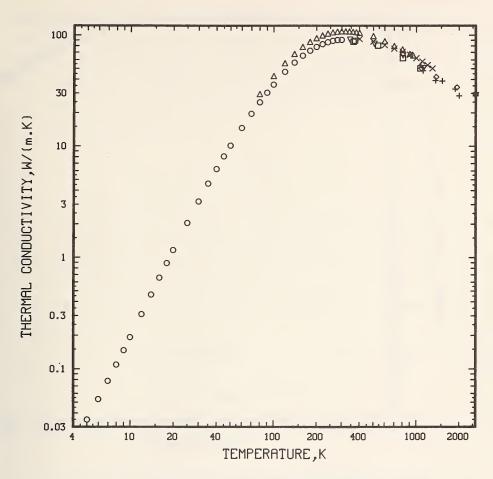


Figure 36. Thermal conductivity data for seven AXM-5Q1 graphite specimens from the following participants at temperatures from 5 to 2600 K:

O = HUST(7) $\diamond = PARI$  $\Delta = MOORE(11)$ + = PARI $\Box = PARTICIPANT 5$  $\times = PARI$  $\nabla = PARTICIPANT 5$ 

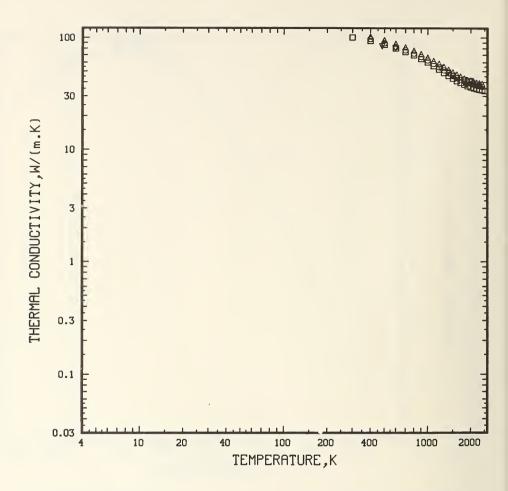


Figure 37. Thermal conductivity data for four AXM-501 graphite specimens from the following participants at temperatures from 300 to 2600 K:

O = TAYLOR(12) $\triangle = TAYLOR(12)$  $\Box = TAYLOR(12)$  $\nabla = TAYLOR(13)$ 

temperature range as was the case at room temperature in Phase II. Numerous equations were tried to represent the general temperature dependence of these thermal conductivity data. Because of the relatively large differences between data sets, especially at high temperatures, an equation with a large number of coefficients was undesirable. The base equation finally selected to represent the general behavior of these data is

$$\lambda_{\rm b} = G_1 T^{\rm G2} / (1 + G_3 T^{\rm G4})^{\rm G5}$$
(5.1.1)

This equation was modified in two ways to obtain the best representation of the data. First, the equation was modified according to the correlation between L and  $\rho$  described in Phase II which is equivalent to the multiplier M<sub>1</sub>. Second, it was modified by a multiplier function, M<sub>2</sub> to remove most of the remaining oscillatory systematic deviations. The final representation of the thermal conductivity data is

$$\lambda = \lambda_b M_1 M_2 \tag{5.1.2}$$

where

$$M_1 = (-18.51 + 0.01908 d_0) \times 10^{-6} / \rho_0$$
 (5.1.3)

 $M_2 = 1 + 0.0012 \ln(T/5.4) \ln(T/15) \ln(T/58) \ln(T/180) \ln(T/1000) \ln(T/1700) (5.1.4)$ 

 $T = temperature in K, \lambda is in W \cdot m^{-1} \cdot K, d_0 is room temperature density in Kg/m<sup>3</sup> and \rho_0 is electrical resistivity in \Omega \cdot m, and the parameters are$ 

 $G_1 = 0.000537$   $G_2 = 2.589$   $G_3 = 0.000202$   $G_4 = 1.678$   $G_5 = 2.02$ 

Figures 38 through 41 show the deviations of the observed thermal conductivity data from eq. (5.1.2). For convenience of comparison, Figures 42-44 contain all of the data on each plot. Figure 42 is a composite of Figures 38-41. Figure 43 shows the deviations of all the data from eq. (5.1.2) with  $M_1 = 1$ 

-65-

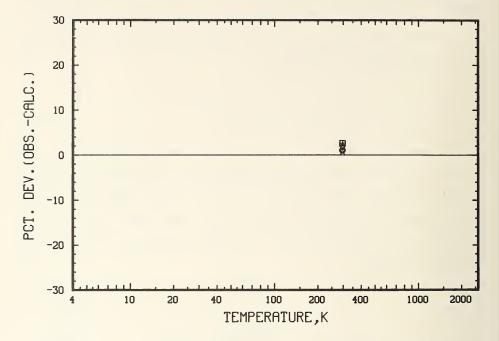


Figure 38. Thermal conductivity deviations for seven AXM-5Q1 graphite specimens as reported by Hust (7) from eq. (5.1.2) at 296 K.

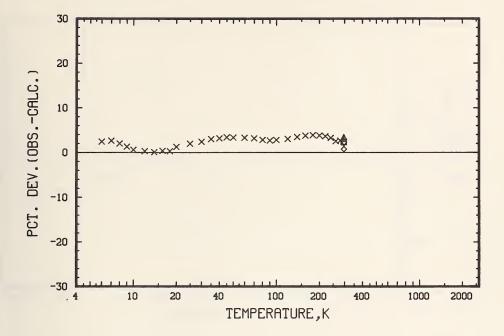
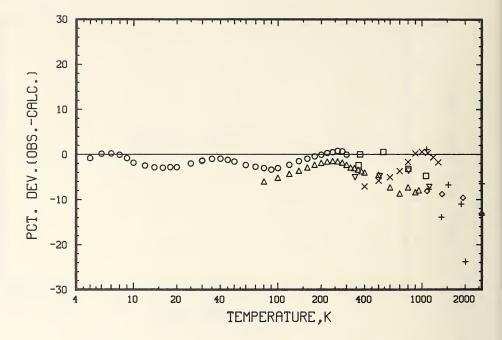
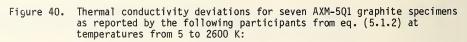


Figure 39. Thermal conductivity deviations for seven AXM-501 graphite specimens as reported by Hust (7) from eq. (5.1.2), six at 296 K and one at temperatures from 6 to 300 K.





O - HUST (7)		٥-	PARTICIPANT	5
$\Delta = MOORE(11)$		+ =	PARTICIPANT	5
PARTICIPANT	5	$\times$ -	PARTICIPANT	3
▽ - PARTICIPANT	5			

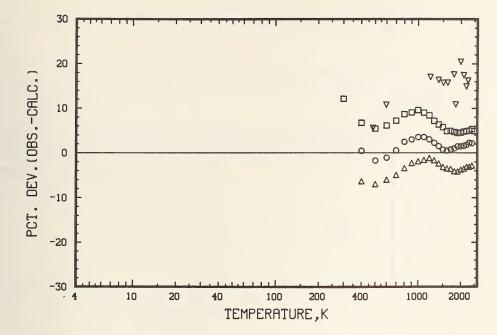


Figure 41. Thermal conductivity deviations for four AXM-5Q1 graphite specimens as reported by the following participants from eq. (5.1.2) at temperatures from 300 to 2600 K:

O = TAYLOR(12) $\triangle = TAYLOR(12)$  $\Box = TAYLOR(12)$  $\nabla = TAYLOR(13)$ 

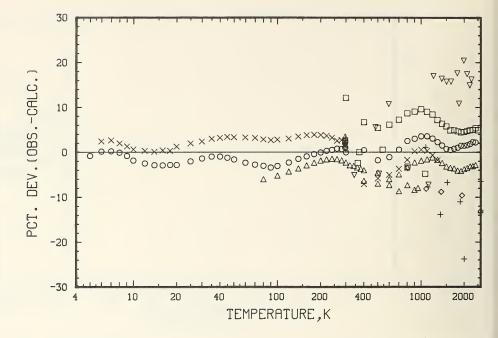


Figure 42. Thermal conductivity deviations of all AXM-5Q1 graphite data from eq. (5.1.2). Composite of Figures 38-41.

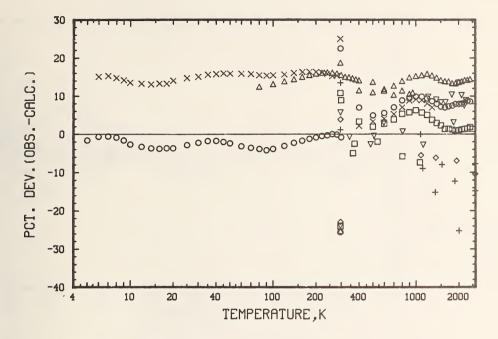


Figure 43. Thermal conductivity deviations of all AXM=5Q1 graphite data from eq. (5.1.2) with  $M_1 = 1$ .

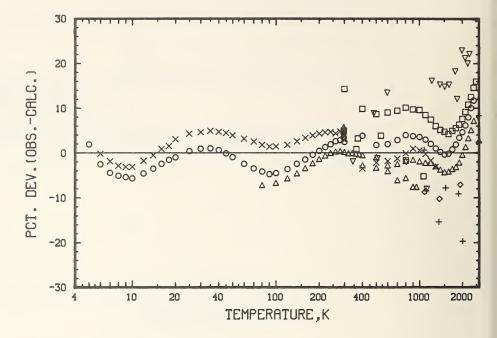


Figure 44. Thermal conductivity deviations of all AXM-501 graphite data from eq. (5.1.2) with  $M_2$  = 1.

(i.e., no corrections for room temperature differences in density and electrical resistivity). Figure 44 shows the deviations of all data from eq. (5.1.2) with  $M_2 = 1$  (i.e., no corrections for the residual oscillations). It is clear from these plots that the corrections for the room temperature characteristic differences are essential. However, relatively large scatter still exists at the higher temperatures. This scatter between data sets may be caused by a) experimental error and b) if the L vs d correlation found at room temperature is also a function of temperature. The data of Hust (7) on two specimens measured from 5 to 300 K suggests that the correlation is quite good to the lowest temperature measured. The data of Taylor (12) on three specimens, and the data of participant 5 on four specimens suggest that the correlation is not as good at higher temperatures.

Based on this research, it is concluded that this graphite can be a useful thermal conductivity standard with the following limitations:

- a) Only those rods should be selected that show the smallest electrical resistivity vs position dependence.
- b) The actual specimen used should be characterized for room temperature electrical resistivity and density values.
- c) Specimens with room temperature electrical resistivities outside the range 13.0 to 15.0  $\mu\Omega^{\bullet}m$  and densities outside the range 1.70 to 1.75 g/cm^3 should be excluded.
- d) The uncertainty of eq. (5.1.2) at high temperatures is as high as 10%.

Table 20 contains recommended values of  $\lambda$  for temperatures from 5 to 2600 K as given by eq. (5.1.2) with  $\rho_0 = 14.5 \ \mu\Omega \cdot m$  and  $d_0 = 1.73 \ g/cm^3$ . It is noted that the correction for  $\rho_0$  and  $d_0$  is zero at that point, i.e.,  $M_1 = 1$ .

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Table 20. Thermophysical properties of AXM-5Ql graphite as calculated from the equations indicated in this report, for room temperature electrical resistivity = 14.5  $\mu\Omega^*m$  and density = 1730 kg/m<sup>3</sup>.

TEMPERATURE (K)	THERMAL CONDUCTIVITY (W∘m-1∘K-1)	ELECTRICAL RESISTIVITY (µΩ∙m)	SPECIFIC HEAT (J•kg-1•K-1)	THERMAL DIFFUSIVITY (µm <sup>2</sup> •s <sup>-1</sup> )	THERMAL EXPANSION (%)
5 6 7 8 9	.0354 .0537 .0783 .1099 .1494	28.78 28.73 28.67 28.62 28.55			198 197 196 196 195
10 15 20 25 30 35 40 45	.1971 .573 1.201 2.095 3.255 4.675 6.33 8.23	28.48 28.11 27.71 26.85 26.43 26.00 25.59			194 191 188 184 181 178 174 171
50 55 60 70 75 80 85 90 95	10.32 12.59 15.03 17.59 20.25 23.00 25.81 28.65 31.51 34.37	25.19 24.79 24.41 23.68 23.33 22.98 22.65 22.33 22.02			168 164 161 158 154 151 148 144 141 137
100 120 140 160 180	37.21 48.16 57.9 66.4 73.3	21.72 20.60 19.60 18.71 17.92			134 120 107 093 079
200 220 240 260 280 300	78.9 83.2 86.4 88.8 90.4 91.3	17.20 16.55 15.97 15.44 14.96 14.52			065 051 037 023 009 .005
400 500 600 700 800 900	90.2 84.6 78.0 71.7 65.9 60.9	12.83 11.74 11.03 10.58 10.30 10.15	995 1208 1381 1518 1629 1718	52.52 40.67 32.90 27.54 23.67 20.78	.078 .152 .228 .306 .386 .468
1000 1100 1200 1400 1500 1600 1700 1800 1900	56.5 52.7 49.48 46.68 44.28 42.22 40.46 38.95 37.67 36.58	10.10 10.12 10.19 10.29 10.43 10.58 10.76 10.94 11.14 11.34	1790 1849 1898 1939 1973 2002 2027 2048 2066 2082	18.55 16.80 15.39 14.26 13.32 12.56 11.92 11.39 10.95 10.58	.551 .636 .723 .811 .901 .993 1.08 1.18 1.27 1.37
2000 2100 2200 2300 2400 2500	35.66 34.89 34.25 33.72 33.29 32.96	11.55 11.75 11.96 12.17 12.38 12.59	1096 2108 2118 2128 2136 2143	10.28 10.03 9.82 9.66 9.53 9.43	1.47 1.57 1.67 1.78 1.88 1.99

The uncertainty of the  $\lambda$  values in Table 20 is estimated to be  $\pm 2\%$  at temperatures below 300 K. At higher temperatures, the uncertainty increases to  $\pm 10\%$  at 2600 K.

## 5.2 Electrical Resistivity

Figures 45 through 47 show the electrical resistivity data from Tables 6 through 19. Based upon the abnormal behavior of the electrical resistivity data from Table 15, i.e., the results reported by participant 2, (see Figure 47) this data set was excluded from the following comparisons. It is noted that this specimen had an abnormally high room temperature electrical resistivity and is the basis for the previous restriction on the range of valid thermal conductivities. Again, an equation with relatively few coefficients was desired to represent the general temperature behavior of the data. The equation selected is

$$\rho = G_1 - G_2 Exp(-(n(T/G_3)/G_4)^2)$$
(5.2.1)

This equation was modified to account for the room temperature differences, and the resulting final equations is

$$\rho = G_1 - G_2 E_{xp}(-(\ell n(T/G_3)/G_4)^2) + \rho_0 - 14.5$$
(5.2.2)

where  $\rho$  = electrical resistivity in  $\mu \Omega \cdot m$ , at temperature, T, in K and  $\rho_0$ = the room temperature resistivity of the specimen in  $\mu \Omega \cdot m$ . The least squares values of the coefficients are G<sub>1</sub> = 28.9, G<sub>2</sub> = 18.8, G<sub>3</sub> = 1023, and G<sub>4</sub> = 2.37. Although some systematic differences are apparent in the following deviation plots, no further modification seemed justified because of the larger scatter between data sets.

-75-

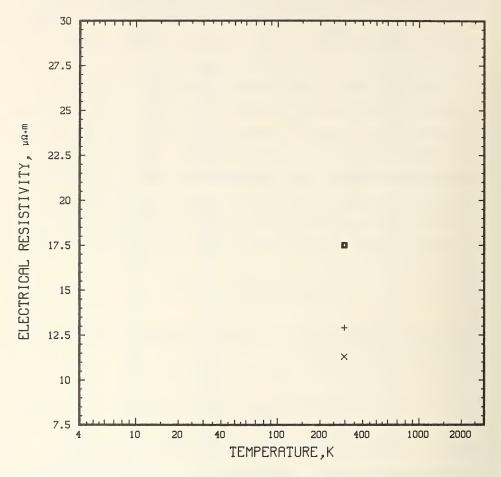


Figure 45. Electrical resistivity data from Hust (7) on seven AXM-501 graphite specimens at 296 K.

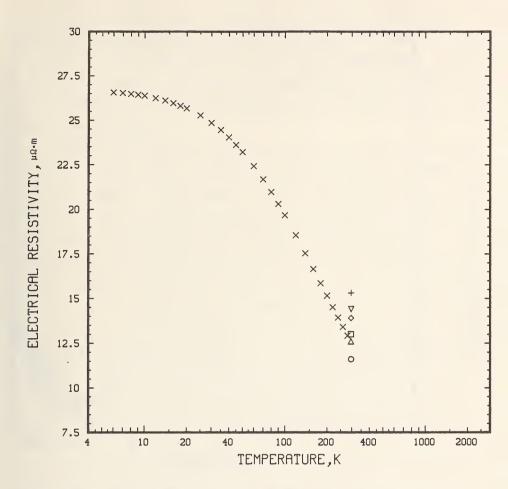


Figure 46. Electrical resistivity data from Hust (7) on seven AXM-5Q1 graphite specimens, six at 296 K and one specimen from 6 to 300 K.

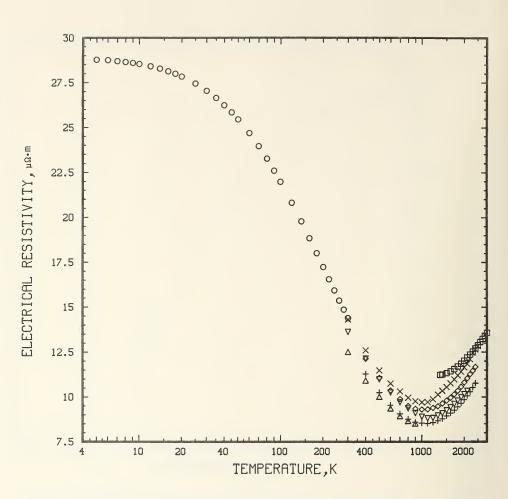


Figure 47. Electrical resistivity data for seven AXM-5Q1 graphite specimens from the following participants from 5 to 2900 K:

- O = HUST(7)  $\Delta = MOORE(11)$   $\Box = PARTICIPANT 2$   $\nabla = PARTICIPANT 3$
- ◇ = TAYLOR(12)
  + = TAYLOR(12)
  × = TAYLOR(12)

Figures 48 through 50 show the deviations of the experimental data from eq. (5.2.2). For convenience of comparison, these data are all shown on a single graph in Figure 51. Figure 52 shows the deviations of the data from eq. (5.2.1) which is uncorrected for room temperature differences. It is clear that most of the differences at high and low temperatures are accounted for simply by the room temperature differences.

A comment about the data of participant 2 (the data set excluded earlier) is in order. It appears that for an unknown reason the value of  $\rho_0$  is incorrect for this specimen. The actual specimen used was in the form of a thin hollow cylinder. The rod from which it was machined was measured at NBS and by the participant. These  $\rho_0$  values agreed to within 2%. The participant also measured the  $\rho_0$  of the hollow cylinder and obtained a value only slightly higher. However, the deviations of the data from participant 2 from eq. (5.2.2) are near -30% at all temperatures. This difference can be nearly eliminated if a value of 15.2  $\mu_0$ ·m is used for  $\rho_0$  instead of 18.81  $\mu_0$ ·m. Using a value of 15.2  $\mu_0$ ·m yields deviations of 1.5% at 1350 K, and -4.0% at 2900 K, varying smoothly and nearly linearly at temperatures between these extremes.

Based on this research, it is concluded that this graphite can be a useful electrical resistivity standard with the same limitations given in the previous section.

Table 20 contains recommended values of electrical resistivity for temperatures from 5 to 2500 K as given by eq. (5.2.2) with  $\rho_0 = 14.5 \ \mu \Omega^{\circ}m$ .

## 5.3 Specific Heat

Figure 53 shows the specific heat data from Tables 6 through 19.

The high temperature data of participants 1 and 2 and Taylor (12) are in reasonable agreement. However, the low temperature data of Isaacs is not only

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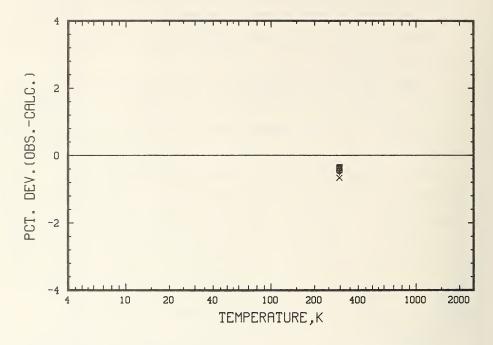
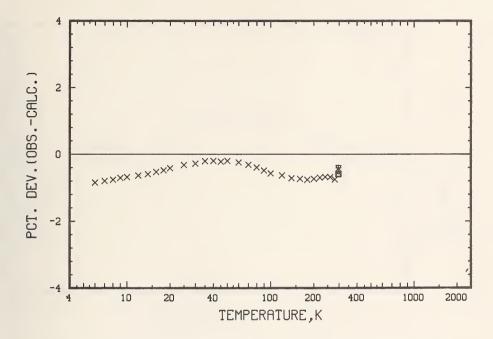


Figure 48. Electrical resistivity deviations for seven AXM-5Q1 graphite specimens as reported by Hust (7) from eq. (5.2.2) at 296 K.



Tyure 49. Electrical resistivity deviations for seven AXM-5Q1 graphite specimens as reported by Hust (7) from eq. (5.2.2), six at 296 K and one at temperatures from 6 to 300 K.

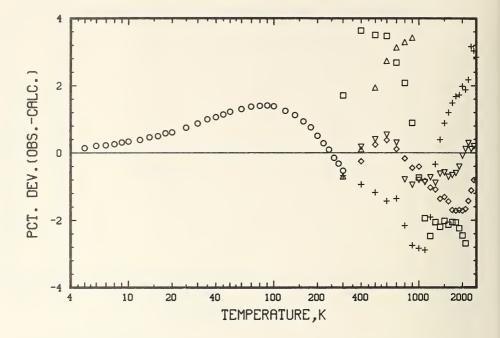


Figure 50. Electrical resistivity deviations for six AXM-501 graphite specimens as reported by the following participants from eq. (5.2.2) at temperatures from 5 to 2500 K.

O - HUST(7)	$\nabla$ = TRYLOR(12)
凶 - MOORE(11)	$\diamond$ = TRYLOR(12)
- PARTICIPANT 3	+ - TAYLOR(12)

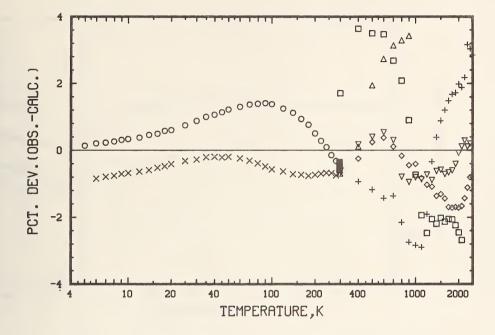


Figure 51. Electrical resistivity deviations of all (excluding participant 2) AXM-5Q1 graphite data from eq. (5.2.2). Composite of Figures 48-50.

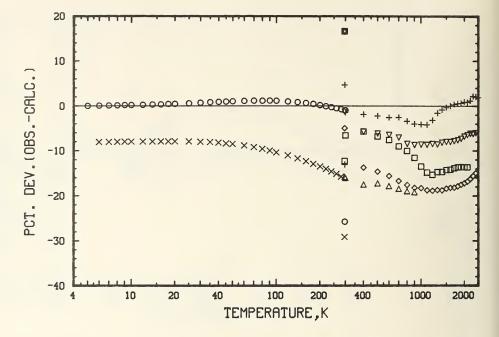


Figure 52. Electrical resistivity deviations of all AXM-5Q1 graphite data from eq. (5.2.2) without the correction for room temperature differences.

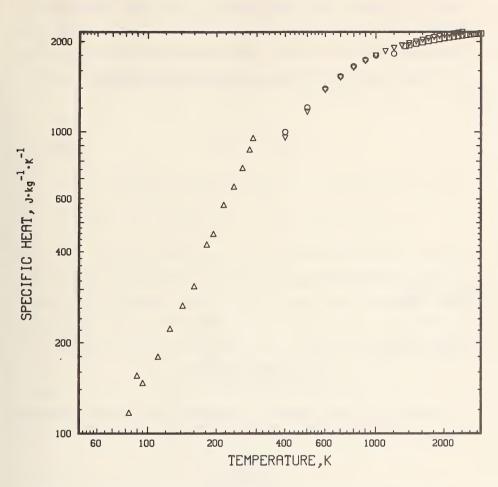


Figure 53. Specific heat data for four AXM-5Q1 graphite specimens from the following participants at temperatures from 80 to 2900 K:

 $\bigcirc = \mathsf{PARTICIPANT} \ 1$  $\triangle = \mathsf{ISAACS}(8)$  $\square = \mathsf{PARTICIPANT} \ 2$  $\nabla = \mathsf{TAYLOR}(12)$ 

abnormal for expected specific heat behavior, but also clearly discordant with the other three data sets. With this in mind and the recognition that the lowest temperature values may be in error, Isaacs data were given a low weight in fitting an equation to the entire data base. The equation selected for this purpose is

$$C = (G_1 T^{-G_2} + G_3 T^{G_4})^{-1}$$
 (5.3.1)

where C is in  $J \cdot kg^{-1} \cdot K^{-1}$ , T is in K and the parameters obtained are

$$G_1 = 11.07$$
  $G_2 = 1.644$   $G_3 = 0.0003688$   $G_4 = 0.02191$ 

The deviations of the data from eq. (5.3.1) are shown in Figure 54.

Because of the limited specific heat data and the discordance between data sets, recommended values are not given at this time. Research on specific heat of graphite is continuing. Table 20 lists values of specific heat as calculated from eq. (5.3.1)

## 5.4 Thermal Diffusivity

Figures 55 and 56 show the thermal diffusivity data from Tables 6 through 19. Equation (5.4.1), the definition of diffusivity, was selected to represent the temperature dependence of these data.

$$D = \lambda/Cd \tag{5.4.1}$$

where  $\lambda$  is given by eq. (5.2.1), C is given by eq. (5.3.1) and d = d<sub>0</sub>/(1 +  $\Delta$ L/L)<sup>3</sup>.

Figures 57 and 58 show the deviation of the experimental data from eq. (5.4.1). Figures 59 and 60 show the deviations without the corrections for room temperature differences.

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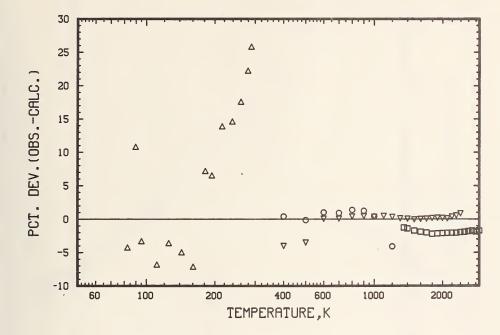


Figure 54. Specific heat deviations for four AXM-5Q1 graphite specimens as reported by the following paticipants from eq. (5.3.1) at temperatures from 80 to 2900 K:

O = PARTICIPANT 1  $\Delta = ISAACS(8)$   $\Box = PARTICIPANT 2$  $\nabla = TAYLOR(12)$ 

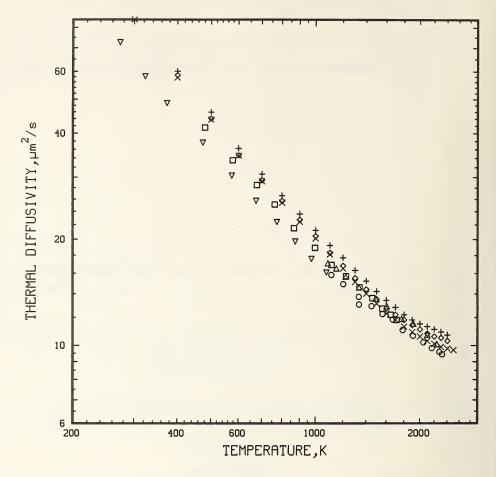


Figure 55. Thermal diffusivity data for seven AXM-5Q1 graphite specimens from the following participants from 280 to 2600 K:

O - BRANDT (6)	A = TAYLOR(12)
$\Delta = \text{BRANDT}(6)$	+ - TAYLOR(12)
- MAGLIC(9)	X - TAYLOR(12)
$\nabla = MIRKOVICH(10)$	

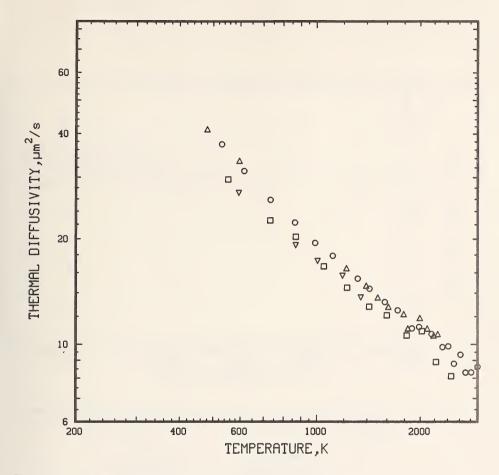


Figure 56. Thermal diffusivity data for four AXM-5Q1 graphite specimens from the following participants from 480 to 2900 K:

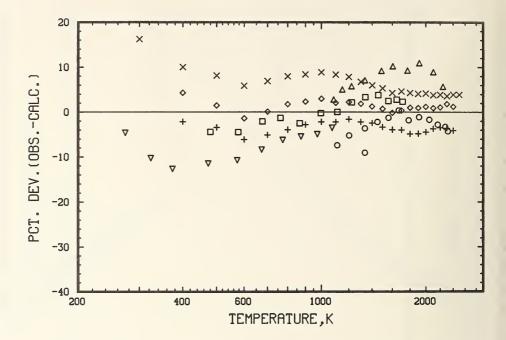


Figure 57. Thermal diffusivity deviations for seven AXM-5Q1 graphite specimens as reported by the following participants from eq. (5.4.1) at temperatures from 280 to 2600 K:

O - BRANDT(6)	TAYLOR(12)
△ = BRANDT(6)	+ = TAYLOR(12)
🗆 - MAGLIC(9)	X = TAYLOR(12)
$\nabla = MIRKOVICH(10)$	

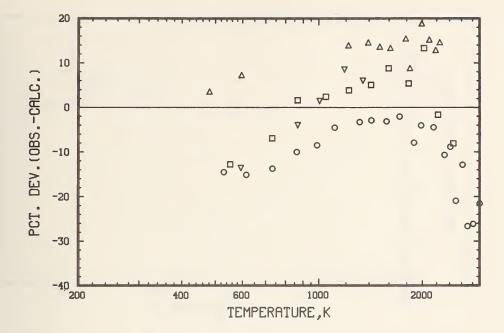


Figure 58. Thermal diffusivity deviations for four AXM-591 graphite specimens as from the following participants from eq. (5.4.1) at temperatures from 480 to 2900 K:

O = TAYLOR(13) $\triangle = TAYLOR(13)$  $\Box = PARTICIPANT 4$  $\nabla = PARTICIPANT 4$ 

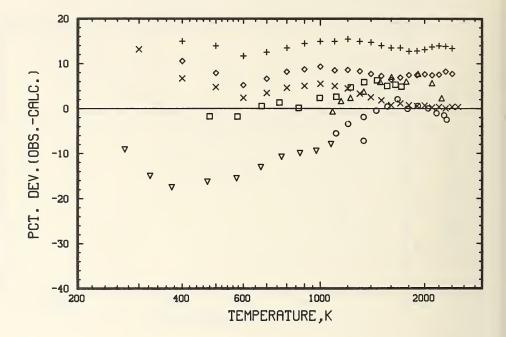


Figure 59. Thermal diffusivity deviations for seven AXM-501 graphite specimens as from the following participants from eq. (5.4.1) (without corrections for room temperature differences) at temperatures from 280 to 2600 K:

O - BRANDT(6)	$\diamond = \text{TAYLOR}(12)$
$\Delta = BRANDT(6)$	+ - TAYLOR(12)
- MAGLIC(9)	× - TAYLOR(12)
∇ - MIRKOVICH(10)	

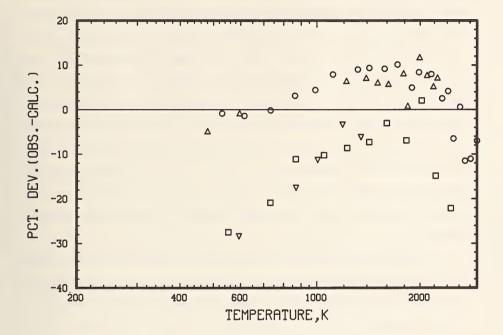


Figure 60. Thermal diffusivity deviations for four AXM-501 graphite specimens as from the following participants from eq. (5.4.1) (without corrections for room temperature differences) at temperatures from 480 to 2900 K:

O = TAYLOR(13) $\Delta = TAYLOR(13)$  $\Box = PARTICIPANT 4$  $\nabla = PARTICIPANT 4$ 

These deviation plots indicate, first, that an independent equation for diffusivity is not necessary at this time and, second, that the deviations between the participants are about that expected from previously reported experimental uncertainties of such measurements. In addition, the difference in the spread of data is not reduced appreciably through the application of the room temperature correlation. The reason for this is not clear but the following should be noted: a) thermal diffusivity data from other studies are typically spread by about  $\pm 10\%$ and b) thermal diffusivity specimens are frequently very small, making accurate electrical resistivity characterization difficult. The latter point should not contribute significantly however, because the bulk specimens were characterized and highly localized inhomogeneities should not be this large. It is concluded therefore that most of this spread is caused by experimental variability. The excellent agreement of eq. (5.4.1) with the mean of all the data is very encouraging.

A recommendation for use of this graphite as a thermal diffusivity standard will be given further consideration. Table 20 lists values of thermal diffusivity as calculated from eq. (5.4.1) with  $\rho_0$  = 14.5  $\mu\Omega$ \*m and d<sub>0</sub> = 1730 Kg/m<sup>3</sup> from 400 to 2500 K.

### 5.5 Thermal Expansion

No thermal expansion data per se were reported in this study. However, the density data reported by Taylor (12) were actually obtained from thermal expansion measurements. These data were analyzed by comparing them to

$$d = d_0 / (1 + \Delta L / L)^3$$
 (5.5.1)

where  $\Delta L/L$  values were computed from the equation

 $\Delta L/L = -0.201 + 6.595 \times 10^{-4}T + 9.593 \times 10^{-8}T^2 - 3.427 \times 10^{-12}T^3$ (5.5.2)

Eq. (5.5.2) was obtained from Touloukian, et al. (14). The density deviations obtained are illustrated in Figure 61. It is noted that excellent agreement is obtained. The fact that the deviations for each specimen are nearly parallel to the zero line indicate that the thermal expansion data of Taylor (12) are in good agreement with eq. (5.5.2). Values of thermal expansion as calculated from eq. (5.5.2) are listed in Table 20.

### 5.6 Seebeck Coefficient

Figure 62 shows the Seebeck coefficient data from Tables 6 through 19. No representation of these data was undertaken. Until an interest is expressed in this property as an SRM, no further work is planned.

### 6. ACKNOWLEDGMENTS

I acknowledge the assistance, suggestions, and encouragement of several associates involved in this program. In particular, I thank Greg Ruff, Susan Fiske, Kevin Kayse, and Bruce Howrey of this laboratory for the numerous measurements and data analysis they have performed. My appreciation is given to Peyton Moore and his associates at ORNL for advice and assistance on the construction of the ambient-temperature comparative thermal conductivity apparatus. The continued support, encouragement, and suggestions of M. Minges (AFML) and R. E. Michaelis and R. K. Kirby (NBS, OSRM) is also acknowledged. The interest and cooperation of Wayne Fagen of POCO Graphite, Inc. has helped to assure the success of this program. The efforts of each of the cooperative participants of the measurement program are acknowledged. Without their participation this work could not have been completed. My appreciation is also expressed to Carole Montgomery, who typed the manuscript and assisted in the layout of the figures and tables.

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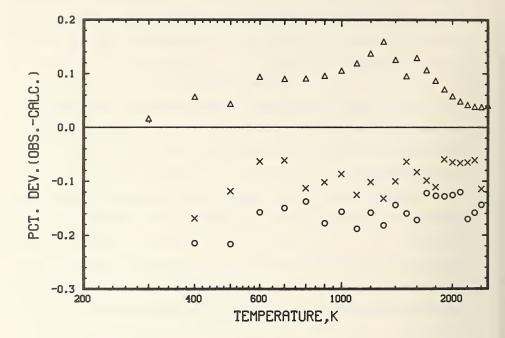


Figure 61. Density deviations for three AXM-5Q1 specimens as reported by Taylor (12) from eq. (5.5.1) for temperatures from 300 to 2500 K.

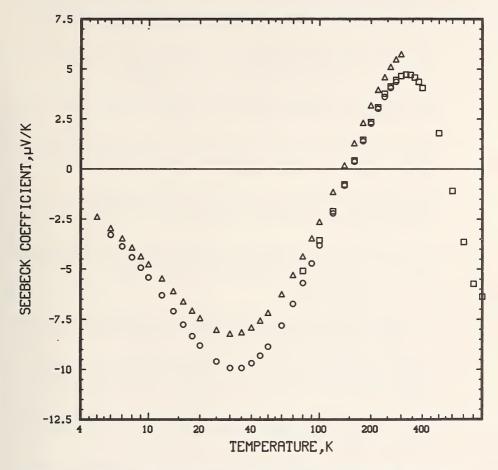


Figure 62. Seebeck coefficient data for three AXM-5Q1 graphite specimens. Two data sets from Hust (7) at temperatures from 5 to 300 K and one set from Moore (11) at temperatures from 80 to 800 K.

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 Y. S. Touloukian, R. K. Kirby, R. E. Taylor, and T. Y. R. Lee, Thermophysical Properties of Matter, Vol. 13, Thermal Expansion, Nonmetallic Solids, p. 75, Plenum Press, New York (1977).

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