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Standard Reference Materials:

THERMAL CONDUCTIVITY AND ELECTRICAL RESISTIVITY STANDARD REFERENCE MATERIALS: ELECTROLYTIC IRON, SRM's 734 AND 797 FROM 4 TO 1000 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 in science, industry, and technology, both within the United States and throughout the world. In many industries traceability of their quality control process to the national measurement system is carried out through the mechanism and use of SRM's. For many of the nation's scientists and technologists it is therefore of more than passing interest to know the details of the measurements made at NBS in arriving at the certified values of the SRM's produced. An NBS series of papers, of which this publication is a member, called the NBS Special Publication - 260 Series is reserved for this purpose.

This 260 Series is dedicated to the dissemination of information on all phases of the preparation, measurement, and certification of NBS-SRM's. In general, much more detail will be found in these papers than is generally allowed, or desirable, in scientific journal articles. This enables the user to assess the validity and accuracy of the measurement processes employed, to judge the statistical analysis, and to learn details of techniques and methods utilized for work entailing the greatest care and accuracy. It is also hoped that these papers will provide sufficient additional information not found on the certificate so that new applications in diverse fields not foreseen at the time the SRM was originally issued will be sought and found.

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Abstract

A historical review of the development of Standard Reference Materials, SRM's, is given and selection criteria of SRM's are listed. Thermal conductivity and electrical resistivity data for electrolytic iron and similar irons are compiled, analyzed, and correlated. Recommended values of thermal conductivity and electrical resistivity for electrolytic iron, SRM's 734 and 797, are presented for the range 4 to 1000 K. These values are based on NBS measurements up to 280 K and on measurements by Oak Ridge National Laboratory on a similar iron above 280 K. The average uncertainty of the thermal conductivity values below ambient is 1.5% and 3% above ambient. The corresponding uncertainties in electrical resistivity are 1% and 2%.

Key Words: Electrical resistivity; electrolytic iron; high temperature; iron; Lorenz ratio; low temperature; standard reference material; thermal conductivity; thermopower.

1. Introduction

Design and development engineers continually demand thermal and electrical property data of technically important materials. Often these data are not in the published literature and immediate measurements must be performed. Since only a handful of laboratories have the proven expertise to make such measurements, usually they are performed by inexperienced personnel using unproven apparatus. The results, as can be seen from the literature, exhibit excessive scatter; 50% differences are commonplace. In such situations, Standard Reference Materials, SRM's, are invaluable to ascertain the accuracy of the engineering measurements. Currently, an inaccuracy of 10% is allowable for most engineering thermal property data, and therefore, SRM's for engineering applications need to be established with an uncertainty no larger than about 5%.

A few research laboratories performing thermal and electrical measurements are obtaining data with uncertainties at the state-of-the-art level, 1% for thermal conductivity and lower for electrical resistivity. SRM's for use at such laboratories must be correspondingly more accurate and may indeed be possible but have not yet been established.

Considerable effort has been directed toward the development of suitable thermophysical SRM's*, over a period of many years, with limited success. This lack of success may be due, in part, to the tacit assumption that SRM data must be accurate to state-of-the-measurement-art to be useful. There are several reasons why the achievement of thermal and electrical property SRM's with certified inaccuracies of less than 1% is extremely difficult. The principal reason is that material variability, generally, causes property variations of greater than 1% even with the most up-to-date production control techniques. The effects of material variability lead to the consideration of three categories of calibration materials and three concomitant certification inaccuracies: (1) A characterized type of material, e.g., copper, gold, iron etc. Based on past experience it appears that inaccuracies of 5-10% can be expected. (2) A characterized specific lot of a given type of material, e.g., austenitic stainless steel, SRM 735, or electrolytic iron, SRM 734. Data uncertainties of one percent appear to be near the lower limit of current production control techniques. (3) Characterized specimens of material. At first glance, it may be thought that the latter SRM's would be invariant; but it is known that the thermal and electrical properties of some specimens change spontaneously with time, aging effects, and are also dependent on their thermal and mechanical histories. These effects are especially significant at low temperatures especially for highly

* The term SRM is used here in a broad sense to denote any material or specimen that is to serve as a calibration standard. The term, as coined by the Office of Standard Reference Materials, generally implies a specific lot of material prepared under strict control and subsequently characterized for chemical composition and homogeneity.

purified materials. Appropriately chosen well-characterized specimens, handled with care to avoid physical and chemical changes, and frequently reexamined to detect changes, presently represent the only means to achieve accuracies in the state-of-the-measurement-art range. This is the basis of round-robin type measurements used by standardizing laboratories for state-of-the-art apparatus intercomparisons (see, for example, Laubitz and McElroy [1]). Category (2) is considered to be the most cost-effective to satisfy engineering needs and, to a lesser extent, the needs of standards laboratories. It is also the philosophical basis of the Office of Standard Reference Materials, National Bureau of Standar

This report is a result of a program to establish several thermal and electrical conductivity metal SRM's with conductivities ranging from pure metals (high conductivity) to structural materials (low conductivity Plans are being formulated to extend this program to insulating materials and dielectric solids as well. The current effort will result in two additional reports: one on tungsten (high conductivity, 4 to 3000 K) and another on austenitic stainless steel (low conductivity, 4 to 1200 K) The material reported on here, electrolytic iron, is in the medium-to-high conductivity range.

This paper reviews the historical development of thermal conductivit SRM's. A listing is given of selection criteria for SRM's and a justification is presented for the establishment of both engineering and standar laboratory SRM's. Data are compiled and best values are selected to esta lish electrolytic iron* as electrical resistivity and thermal conductivity SRM's 797 and 734, respectively. As discussed later, thermal conductivit and electrical resistivity data have been obtained to certify these SRM's over the range 4 to 1000 K to well within engineering accuracy. This material appears to have the qualities of an excellent SRM. An adequate supply of this material exists to insure measurement compatibility among laboratories for about ten years.

The following historical review of SRM efforts is presented to indicate the relatively large amount of research that has been conducted, compared to the few thermophysical SRM's that have been officially established. It is this divergence between expended efforts and concrete results that has prompted us to establish potentially useful SRM's, at what may seem to some as a premature phase of the work. Based on past experience, it appears that if this is not done, a vast amount of researc is lost. Not because the data are lost, but rather, because the stock of material, on which the research was performed, is lost. This consideration also points out the significance of continuity in SRM projects.

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*This electrolytic iron is a specific lot of iron produced for NBS to maximize homogeneity. Throughout this paper it is referred to as NBS electrolytic iron as it is the basis for several SRM's distributed by NBS,OSRM.

2. Historical Review

2.1 Early Efforts

Thermophysical property reference material investigations began, for all practical purposes in the 1930's with the work of R. W. Powell at the National Physical Laboratory (NPL), Teddington, England [2] on iron and Van Dusen and Shelton at NBS [3] on lead. These efforts were successful in that they resulted in frequently used reference materials of thermal conductivity. Powell's work resulted in the establishment of ingot iron* (category 1) as a standard, which is still being used today. Lucks [4] recently reviewed the massive amount of work that has been done on this material and recommended the continued use of ingot iron as a reference material. Van Dusen and Shelton's work resulted in an unofficial lead standard based on a well-characterized lot of pure lead (category 2) distributed by NBS as a freezing point standard.

2.2 Iron

Since the 1930's reference material investigations have been sporadic with notable efforts by researchers from the NBS (National Bureau of Standards, U.S.), NPL (National Physical Laboratory, England), ORNL (Oak Ridge National Laboratory, Tennessee), BMI (Battelle Memorial Institute, Ohio), and AFML (Air Force Materials Laboratory, Ohio). The material that has been the subject of the most extensive investigations is ingot iron. Renewed interest in this material was spurred by the round-robin† experiments initiated by C. F. Lucks of Battelle Memorial Institute during 1959. Twenty-four laboratories requested and received the round-robin material for measurements. Data from eight laboratories were ultimately reported and compiled by Lucks [4]. These data are on specimens obtained from a single lot of ingot iron. The literature, (see Lucks) however, contains data on a total of eleven distinct lots of ingot iron. Lucks [4] has shown that ingot iron is an acceptable reference material at temperatures from about 100 K to 1000 K. In this range, material variability affects thermal conductivity and electrical resistivity by about 5%. At higher temperatures, reported variations increase. At lower temperatures, especially at liquid helium temperatures, variations of 10% have been reported on a single 30 cm long rod by Hust et al [5,6]. Electrolytic iron, SRM 734, was established as a low-temperature standard by Hust and Sparks [7] because it exhibits relatively small low-temperature variability. Based on their high temperature study of ingot iron and a high purity iron, Fulkerson et al [8] also concluded that high purity iron is a more homogeneous and stable SRM.

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* The ingot iron used for this purpose is Armco iron produced by Armco Steel Corporation. The use of trade names of specific products is essential to the proper understanding of the work presented. Their use in no way implies any approval, endorsement, or recommendations by NBS.

† The use of the term "round-robin" is different here from that used earlier where the use of a single specimen was implied; however, this double meaning is allowed to be consistent with the literature on ingot iron.

2.3 NBS, Washington Efforts

D. R. Flynn of NBS, Washington began a study of potential thermal conductivity SRM's during the early 1960's. He examined several ceramics* and alloys†. None of these materials has achieved the status of an SRM. Descriptions of these efforts appear in the unpublished proceedings of the early thermal conductivity conferences. Laubitz and Cotnam [9] reported that Inconel 702 exhibits transformation effects of several percent in thermal conductivity and recommended against its use as a reference material.

At the 1963 thermal conductivity conference, Robinson and Flynn [10] presented the results of a survey of thermal conductivity SRM needs. SRM's with a data uncertainty of 3-5% were in greatest demand. The intended use of SRM's, most often stated, was to check and calibrate apparatus. Needs were indicated for SRM's of conductivities from 0.01 W/mK to 500 W/mK at temperatures from 4 to 3300 K.

2.4 NBS, Boulder Efforts

R. L. Powell of NBS, Boulder initiated a low-temperature SRM project during the early 1960's. This project has been continued by the first autl since that time. Materials studied include ingot iron, electrolytic iron, gold, tungsten, graphite, and stainless steel. As a result of these studies, electrolytic iron and stainless steel have been established as low-temperature (4 to 280 K) SRM's of electrical resistivity and thermal conductivity. Current efforts are directed toward the extension of these to higher temperature and to establish graphite and tungsten as SRM's at temperatures up to near 3000 K. It is anticipated that this project will continue until a sufficiently wide range of conductivities and temperature: are included to satisfy existing demands for thermophysical SRM's.

2.5 AFML-AGARD Project

Minges [5th Thermal Conductivity Conference, 1965] reported on the initiation of an AFML sponsored high-temperature reference materials program. This program was divided into two phases. Phase I included the preliminary selection and characterization of materials as potential reference materials. Selection criteria were established, dozens of materials screened, and about 15 were chosen for experimental evaluation. Phase II included further measurements on those materials selected from Phase I studies. Arthur D. Little Corp. contracted with AFML to perform

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* Pyroceram 9606 and Pyrex 7740 (trade names of Corning Glass Works).

† Inconel 702 (trade name of International Nickel Company, Inc.), lead, and 60% platinum - 40% rhodium alloy.

this study. The results were reported in reference [11]. The materials of particular interest in Phase II of this program were aluminum oxide, thorium oxide, tungsten, and graphite.

After partial completion of the AFML program, an international program, principally high-temperature, was initiated under the auspices of the Advisory Group for Aerospace Research and Development, NATO (AGARD). E. Fitzer of Karlsruhe University, Germany, directed this program in close cooperation with the AFML program. The establishment, progress, and results of this program are described in a series of reports by Fitzer [12]. Minges has also summarized some of the results on AFML-AGARD programs [13]. The materials, internationally distributed and measured by numerous laboratories, are: platinum, gold, copper, austenitic steel alloy, tungsten (both sintered and arc-cast), tantalum - 10% tungsten alloy, alumina, and graphite.

3. SRM Selection Criteria

The criteria for screening and selecting potentially useful materials for physical property SRM's are generally well-understood and accepted. These criteria are not met absolutely by any material, but serve as a guide to determine which materials are most suitable. Some of the more significant factors are:

1. The material should be homogeneous* and isotropic throughout a lot. The lot should be large enough to be adequate for at least a decade and renewable with a minimum of effort.

2. Thermophysical properties should not vary with time and should be relatively unaffected by the environment of the measurement apparatus. The material should have chemical stability, thermal shock resistance, low vapor pressure, and insensitivity to stress.

3. The material should be readily available, machinable, be relatively inexpensive, and have sufficient strength to be handled without causing damage.

4. The material should have characteristics similar to the material to be measured.

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* The term homogeneous refers here to the uniformity of the thermophysical property in question. Homogeneity of a thermophysical SRM implies not only chemical homogeneity, as in chemical composition SRM's, but also homogeneity of physical characteristics of the material. The parameters affecting physical property homogeneity are so numerous that detailed characterization of each is prohibitive. Instead, one often reverts to aggregate characterization methods, such as by electrical resistivity as discussed later.

5. The material should be useful over a wide temperature range. The electrolytic iron described in this report satisfies these criteria reasonably well.

4. Material Characterization

The purpose of this work is to establish NBS electrolytic iron as SRM's of thermal conductivity and electrical resistivity at temperatures from 4 to 1000 K. To support our thesis that this lot of material is sufficiently homogeneous and the recommended data are accurate to within the stated uncertainties, we present extensive characterization data. Since the recommended SRM values for NBS electrolytic iron are based, in part, on measurements on other irons, characterization data for ingot iron and ORNL high purity iron [8] are included. The characterization data for NBS electrolytic iron have been presented previously by Hust and Sparks [7,14] and in a supplement to Reference [7]. Since these data are not found in a single source, they are repeated here.

4.1 Electrical Resistivity Characterization

Extensive reliance is placed on electrical resistivity variability as an indicator of thermal conductivity variability for pure metals. The justification for this is presented below.

The electrical resistivity, ρ, and thermal conductivity, λ, of metals are intimately related, especially for pure metals, but also for alloys to a lesser extent. This relationship exists because in a metal most of the heat is transported by the electrons. Some heat is also transported by the lattice vibrations. The total thermal conductivity is the sum of the electronic, λ_e, and the lattice, λ_g, (the German word for lattice is Gitter) components.

$$\lambda = \lambda_e + \lambda_g. \tag{1}$$

In most pure metals λ_g is small compared to λ_e, but in transition metals λ_g may be as large as 20% of λ_e, and in some alloys λ_g is much larger than λ_e. For pure metals and dilute alloys, the relationship between ρ and λ at both high and low temperatures is reasonably well described by the Wiedemann-Franz-Lorenz (WFL) law:

$$\frac{\rho\lambda}{T} = L_o = 2.443 \times 10^{-8}\ V^2K^{-2}, \tag{2}$$

where L_o is the Sommerfeld value of $\rho\lambda/T$ and T is the temperature. At intermediate temperatures, large deviations from the WFL law are observed. For our purposes the ice point is a sufficiently high temperature and liquid helium is a sufficiently low temperature to satisfy the WFL law.

In metals there are two mechanisms that account for most of the scattering of electrons: the interaction of electrons with chemical impurities and physical imperfections, and the interaction of electrons with thermal vibrations of the atoms of the lattice. The former mechanism is usually taken to be independent of temperature while the latter is temperature dependent. If we assume that each of these mechanisms is independent of the other, we may assign a separate resistivity to each. The resistivity arising from impurity and imperfection scattering is usually referred to as the residual resistivity, ρ_o, while the resistivity due to thermal scattering is called the intrinsic resistivity, $\rho_i(T)$. The total resistivity, $\rho(T)$, may be written as the sum of these two terms.

$$\rho(T) = \rho_o + \rho_i(T). \tag{3}$$

This separation of the total resistivity into a constant term (ρ_o) and a temperature dependent term ($\rho_i(T)$) is known as Matthiessen's rule. Although Matthiessen's rule is not strictly valid, it is a sufficiently good approximation for our purposes.

At ambient temperatures the residual resistivity is a negligibly small fraction of the total resistivity; consequently, the total resistivity, $\rho(T)$, is nearly equal to the intrinsic resistivity, $\rho_i(T)$, and therefore a characteristic of the metal itself. As the temperature approaches absolute zero, however, the intrinsic resistivity becomes very small and the total resistivity is essentially the value of ρ_o. The temperature at which $\rho(T)$ becomes constant depends upon the purity of the sample, but for most materials available at the present time, the intrinsic resistivity will be negligible at 4 K (the boiling point of helium).

The residual resistivity, which is caused primarily by impurities and imperfections, provides a good indication of a specimen's purity and freedom from strain. Rather than using the residual resistivity itself for this purpose, a common procedure is to determine a specimen's resistance at the ice-point, R_{273}, and at 4 K, R_4, and calculate the ratio between these two, R_{273}/R_4. This is nearly equal to the ratio of the resistivities at the same temperatures as the geometric form factor nearly cancels in the ratio. The geometric form factors are not quite the same because of thermal expansion, which is seldom over 0.5%. This ratio is called the residual resistivity ratio, RRR, and its magnitude is an indication of the purity and physical perfection of the specimen. Since the specimens measured here were generally in the annealed condition, the RRR value should indicate the effective chemical purity (electrical purity).

As an exercise to show the validity of this statement, we computed the residual resistivity from the measured chemical composition of NBS electrolytic iron. Using the specific resistivities listed by Blatt [15], we obtained a value of 5nΩm assuming that all the impurities are in solution. Since the measured residual resistivity is 4 nΩm, the electrical purity is in good agreement with the chemical purity. Thus, we expect that variations in measured residual resistivity are an excellent indication of chemical inhomogeneities and physical imperfection variations.

Electrical resistivity variations are accompanied by thermal conductivity variations of nearly the same proportion as shown by the WFL law. Therefore, the determination of residual resistivity or residual resistivity ratio variability will directly indicate thermal conductivity variability. The measurement of electrical resistivity is, of course, much easier than the determination of thermal conductivity.

An extensive resistivity variability study was conducted on NBS electrolytic iron prior to its certification as SRM 734 in 1971. The objective was to determine if this material could be heat treated in such manner that the thermal conductivity would be nearly the same (± 1%) for each specimen. This was achieved with a 2-hour, 1000°C anneal in either a vacuum or helium atmosphere. The results of this study were reported as residual resistivity ratios in [14] and are repeated in table 1. The ratio given is resistivity at 273.15 K to resistivity at 4 K.

Various heat treatments were tried during 1970 to stabilize the residual resistivity ratio, RRR, of this iron. After an anneal of 500°C for 1 hour, the ratio increased from 20.11 in the as received condition to 22.54. Raising the temperature to 1000°C for 2 hours produced rods which appeared stable at a ratio of 23.33 ± 0.24. The variation shown is 2s, where s is the estimated standard deviation, and includes material and measurement variability. In order to study the possibility of a change in ratio with age, some of the rods were measured after about 50 days and no significant change was detected. At that time, SRM 734 was established for the range 6 - 280 K with the conclusion that no significant changes would occur with age.

After three years of room temperature aging, a 4% increase in RRR was found. It is also noted that heating to 400°C for 2-1/2 days changed the ratio to 24.94 ± 0.26 when the first measurements were made during 1970. However, in 1973 a similar heat treatment produced a much smaller change (about 1%). This is not understood but the result of the latter measurement allows consideration of extending this SRM to higher temperatures. It is to be noted that the above mentioned room temperature aging effect does not significantly alter the thermal conductivity of SRM 734

TABLE 1. Residual Resistivity Ratios of Various Specimens of NBS Electrolytic Iron.

Specimen	RRR (← 1970)						RRR (1973 →)										
	As received	500°C 1 hr	500°C 8 hr	1000°C 2 hr	400°C 2½ days	Aging 50 days	Aging 3 yrs	1000°C 60 hr	800°C 46 hr	500°C 60 hr	1000°C 2 hr	800°C 2 hr (vac.)	400°C 20 hrs	800°C 4 hr	400°C 46 hrs	800°C 2¾ hrs	1000°C, 2 hr 800°C, 2 hr
C2T	21.97 (a)	23.53	24.12	23.31 (e)	24.84	25.00	25.57	21.88	23.46	23.64	22.45	---	23.65	---	23.88	---	---
A6L	22.16	---	---	23.22 (f)	24.85	24.97	25.85	---	---	---	---	23.59	---	23.40	---	---	---
C5L	21.94	---	---	23.40 (f)	---	23.47	24.46	22.19	23.615	23.81	22.61	---	23.80	---	23.94	---	---
A1L	22.04	---	---	23.59 (c)	---	23.52	---	---	---	---	---	---	---	---	---	---	---
A5T	22.03 (b)	---	---	23.42 (f)	---	23.42	24.90	---	---	---	---	23.64	---	23.58	---	---	---
2A-1-1	19.35	21.96	22.32	23.47 (e)	25.12	25.24	25.69	22.20	23.45	23.76	22.54	---	23.72	---	24.07	---	---
2A-1-2	19.50	---	---	23.31 (c)	---	23.35	---	---	---	---	---	---	---	---	---	---	---
2A-1-3	19.30	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
2A-1-4	19.38	---	---	23.27 (f)	---	---	---	---	---	---	---	---	---	---	---	---	---
2A-3-1	19.77	21.83	22.25	23.20 (e)	24.94	25.01	---	---	---	---	---	---	---	---	---	---	---
2A-3-2	19.92	---	---	23.20 (c)	---	23.25 (d)	---	---	---	---	---	---	---	---	---	---	---
2A-3-3	19.73	---	---	---	---	---	20.29	---	---	---	---	24.52	---	24.48	---	25.12	23.24
2A-3-4	19.93	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
2C-1-1	19.42	21.70	22.00	23.23 (e)	---	23.44	24.48	**22.55**	23.51	23.63	22.51	---	23.60	---	23.94	---	---
2C-1-2	19.12	---	---	23.41 (c)	---	23.40	---	---	---	---	---	---	---	---	---	---	---
2C-1-3	19.46	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
2C-1-4	19.56	---	---	---	---	---	---	---	---	---	---	---	---	---	---	24.75	23.81
2C-3-1	19.34	21.93	21.99	23.27 (e)	---	23.40	24.86	---	---	---	---	23.40	---	23.64	---	---	---
2C-3-2	19.49	---	---	---	---	---	---	---	---	---	---	---	---	---	---	25.16	23.50
2C-3-3	19.57	---	---	---	---	---	---	---	---	---	---	---	---	---	---	24.84	23.56
2C-3-4	19.40	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Average =	20.11	22.19	22.54	23.33	24.94	23.96	24.51	22.20	23.51	23.71	22.53	23.79	23.69	23.78	23.96	24.97	23.53

(a) repeat measurement = 21.91
(b) ratio of A5T thermal conductivity specimen = 21.89
(c) these were heat treated in vacuum, the remaining were heated to 1000°C in a helium atmosphere (1 atm pressure).
(d) repeat measurements = 23.39, 23.31
(e, f) these were done in separate heat treatments to detect reproducibility of heat treatment.

at temperatures above 60 K. At room temperature, the magnitude of the effect on thermal conductivity over the three year period is only about 0.2% while at 60 K it is about 1%. The full effect, 4%, is seen only at 4 K. This statement is based on the observed changes in electrical resistivity at the ice point and at liquid helium temperature. These measurements, reported below as resistivity ratios for convenience, showed the following: The range of residual resistivities measured for all of the specimens and various heat treatments is about 30%. The average residual resistivity ratio for all of these measurements is about 23. Based on Matthiessen's rule, one would expect the ice point resistivity range to be about 1-1/2%. The measured ice point resistivity range is about 3%, which is consistent with the 1-1/2% expected range within the measurement uncertainty of ± 1%. The average intrinsic resistivity of NBS electrolytic iron at the ice point is 87.1 nΩm ± 0.2%.

After performing further anneals to obtain a better understanding of the aging phenomena, it appears clear that our earlier selected anneal procedure, although described insufficiently, was proper in that we obtained the RRR value which is least dependent on heating to temperatures below 800°C and is, therefore, most stable with time. However, we were not aware, at the time, of the importance of the cooling rate of the furnace. At that time, we used a massive furnace which cooled rather slowly (approximate decay time constant of 6 hours). With the smaller furnace (approximate time constant of 3 hours) used in the later measurements, a hold of at least two hours at 800°C was necessary to stabilize this iron. After this heat treatment, heating specimens to intermediate temperatures does not significantly effect the residual resistivity ratio. These measurements show that SRM 734 can be used as a thermal conductivity standard with a variability of about 1% if annealed at 1000°C for 2 hours, cooled to 800°C and held for 2 hours, and furnace cooled to ambient. The effect of more rapid cooling rates below 800°C was not investigated.

4.2 Other Characterization Data

The density of electrolytic iron, determined by air and water weighings (see Bowman and Schoonover [16]), is 7.867 ± 0.005 g/cm^3. Rockwell hardness and grain size are B24 and 0.05 mm, respectively. Grain size was determined by the American Society for Testing and Materials (ASTM) comparative method. The above data were determined with the material in the annealed state. The chemical purity of this electrolytic iron is 99.9+ weight percent Fe. The chemical composition, as certified by NBS, SRM 1265, is given in table 2, along with typical values for ingot iron and the high-purity iron measured at Oak Ridge National Laboratory (ORNL) by Fulkerson et al [8]. The values listed for ingot iron must be considered typical, since the ingot iron investigated was not a single lot of material, but, rather, many lots as produced over a period of many years.

Table 2. Chemical composition of NBS electrolytic iron and ORNL high-purity iron and typical values for ingot iron

Element	Composition (weight percent)		
	NBS electrolytic iron	ORNL high-purity iron	ingot iron
Carbon	0.0067	0.003	0.015
Manganese	0.0057	–	0.028
Phosphorus	0.0025	0.001	0.005
Sulfur	0.0059	0.003	0.025
Silicon	0.0080	<0.01	0.003
Copper	0.0058	<0.001	0.04
Nickel	0.041	<0.01	–
Chromium	0.0072	–	–
Vanadium	0.0006	–	–
Molybdenum	0.005	–	–
Cobalt	0.007	–	–
Titanium	0.0006	–	–
Arsenic	0.0002	–	–
Aluminum	0.0007	<0.001	–
Boron	0.00013	–	–
Lead	0.00002	–	–

– = unknown

For comparative purposes other characterization data are presented. Grain size of ingot iron is about 0.05 mm. Residual resistivity ratio, ρ_{273K}/ρ_{4K}, has ranged for the various lots from about 9 to 14, compared to a mean of 23 for NBS electrolytic iron. Hardness of ingot iron is about Rockwell B40. The high-purity ORNL iron has a reported residual resistivity ratio of 23, the same as for electrolytic iron. The grain size of ORNL high-purity iron is significantly larger than either ingot iron or NBS electrolytic iron. Grain size is undoubtedly dependent on the previous thermal history of each specimen and may not be a significant characterization parameter.

5. Apparatus and Measurements

The intent of this paper is to establish NBS electrolytic iron as SRM's of thermal conductivity and electrical resistivity at temperatures from 4 to 1000 K. The following sections describe the measurements resulting in data pertinent to this study. The low-temperature data originate entirely with NBS, Boulder and the high-temperature data are entirely from the published literature on similar irons.

5.1 Low-Temperature (Below Ambient) Measurements

Thermal conductivity, electrical resistivity, and thermopower measurements were performed with a multiproperty apparatus based on the axial one-dimensional heat flow (longitudinal) method. The specimen is 3.6 mm in diameter and 23 cm long with an electric heater at one end and a temperature controlled heat sink at the other. The specimen is surrounded by glass fiber and a temperature controlled shield. Eight thermocouples are mounted at equally spaced points along the length of the specimen to determine temperature gradients in the range 4 to 300 K. A detailed description of this apparatus and an error analysis are presented by Hust et al [6]. The estimated uncertainties (with 95% confidence) are as follows:

Thermal conductivity: 2.5% at 300 K decreasing to 0.7% at 200 K, 0.7% from 200 K to 50 K, and increasing to 1.5% at 4 K.

Electrical resistivity: 0.25%.

One specimen was measured in the low-temperature apparatus over the range 4 to 280 K. The data were smoothed using conventional linear least-squares methods with the following equations:

$$\ln\lambda = \sum_{i=1}^{n} a_i\,[\ln T]^{i+1}$$

$$\rho = \sum_{i=1}^{m} b_i\,[\ln T]^{i-1}$$

where λ = thermal conductivity, ρ = electrical resistivity, and T = temperature, which is based on the IPTS-68 scale above 20 K and on the NBS P2-20 (1965) scale below 20 K. These functions have no theoretical significance, but are chosen from past experience on the basis of their usefulness for smoothing similar data. The optimum number of parameters is selected by utilizing orthogonal fitting analysis to avoid either underfitting or overfitting the data. In the first case, excessive oscillations, or wiggles, may be introduced in the temperature dependence. These equations are used primarily for data analysis and smoothing to within the accuracy of the data. Because of the form of the raw experimental data, the extensive number of data points, and the complexity of the data analysis, the experimental data are not presented here. They are, however, printed in an informal NBS report [17] which may be obtained from the author. No other data sources exist for temperatures below ambient. The previously presented fixed-point electrical resistivity characterization data were obtained using a conventional four-terminal apparatus.

5.2 High-Temperature (Above Ambient) Measurements

No high-temperature measurements have been performed on NBS electrolytic iron, per se. However, measurements have been performed extensively on similar irons at elevated temperatures. In particular, as discussed earlier in this paper, ingot iron has been measured repeatedly since 1932. Ingot iron is somewhat less pure than NBS electrolytic iron, (see table 2). Lucks [4] has recently reviewed the thermal conductivity and electrical resistivity measurements on ingot iron. Although Lucks' paper does not include descriptions of the apparatus used for the past measurements, it can be used as a bibliographic source directing the reader to the original experimental papers. A summarization of these papers must include the statement: ingot iron has been measured using more different types of apparatus at more laboratories than any other material.

Although the ingot iron data are valuable in establishing reference data for NBS electrolytic iron, it is fortuitous that data for an iron almost identical to NBS electrolytic iron has been published by Fulkerson et al [8]. This data set is especially pertinent since it is the result of the most extensive single experimental and analytical work on iron at elevated temperatures and is from workers of proven expertise. This well characterized, high-purity iron is identified as ORNL in the material characterization section. Table 2 shows the composition of NBS electrolytic iron to be between that of ingot iron and the high-purity ORNL iron, but significantly closer to the latter. Confirmation of this is reinforced by the agreement of the measured residual resistivities of these irons, NBS electrolytic iron differs by only 2% from high-purity ORNL iron and 50% from ingot iron.

Thermal conductivity, electrical resistivity, and thermopower measurements were performed by Fulkerson et al [8] at temperatures from near ambient to above 1200 K with a radial heat flow apparatus. The reported most probable errors for these data are about 2% for thermal conductivity and 0.5% for electrical resistivity.

6. Data Analysis (Selection of Best Values)

After the establishment of SRM's 734 (thermal conductivity) and 797 (electrical resistivity) for temperatures below ambient, no new measurements have been reported. Thus no modifications of the previously recommended reference data are necessary. From 6 to 280 K the recommended values of thermal conductivity are those reported by Hust and Sparks [7] and the corresponding values of electrical resistivity are those reported by Hust [14]. These values for SRM's 797 and 734 are listed in table 3.

At temperatures above ambient, the recommended values of electrical resistivity and thermal conductivity for electrolytic iron are based on the values reported for high-purity ORNL iron. Ingot iron data are used to reinforce the validity of this selection and to establish probable error bounds. The following discussion presents the basis of this selection.

Figures 1 and 2 illustrate the thermal conductivity and electrical resistivity for NBS electrolytic iron, ORNL high-purity iron, and ingot iron as reported by the indicated authors. The thermal conductivity of NBS electrolytic iron is measureably greater than that of ingot iron, 12% at 100 K and 5% at 300 K. Figure 2 shows that similar differences, but opposite sign, occur in the electrical resistivities of these irons. It is also observed from figures 1 and 2 that the reported data for NBS electrolytic and ORNL high-purity iron are the same to within the reported uncertainties of the measurements. This is not surprising in view of the previously mentioned similarities of the compositions and other characterization parameters.

Since iron conducts heat primarily by electrons, one would expect the Lorenz ratio, $\rho\lambda/T$, to be a useful tool for correlating the thermal and electrical conductivities of these irons. Figure 3 illustrates the Lorenz ratios above 100 K for these irons. The values plotted are obtained from the total thermal conductivity and, thus, include the lattice component of conductivity. It is noted that the Lorenz ratios of all three of these irons agree to within 2% above ambient temperature. The values for Armco iron are consistently greater than those for the higher purity irons. In spite of the fact that the 2% difference is near experimental uncertainty, it is believed to indicate a real difference. Based on the compositions listed in table 2 one would expect the Lorenz ratio of NBS electrolytic iron to be below that of ingot iron. One would also expect the Lorenz ratio values for ORNL iron to be slightly lower than NBS iron. In view of the combined data uncertainties for these data sets (about 2-3%), the confirmation of the above predictions is remarkable.

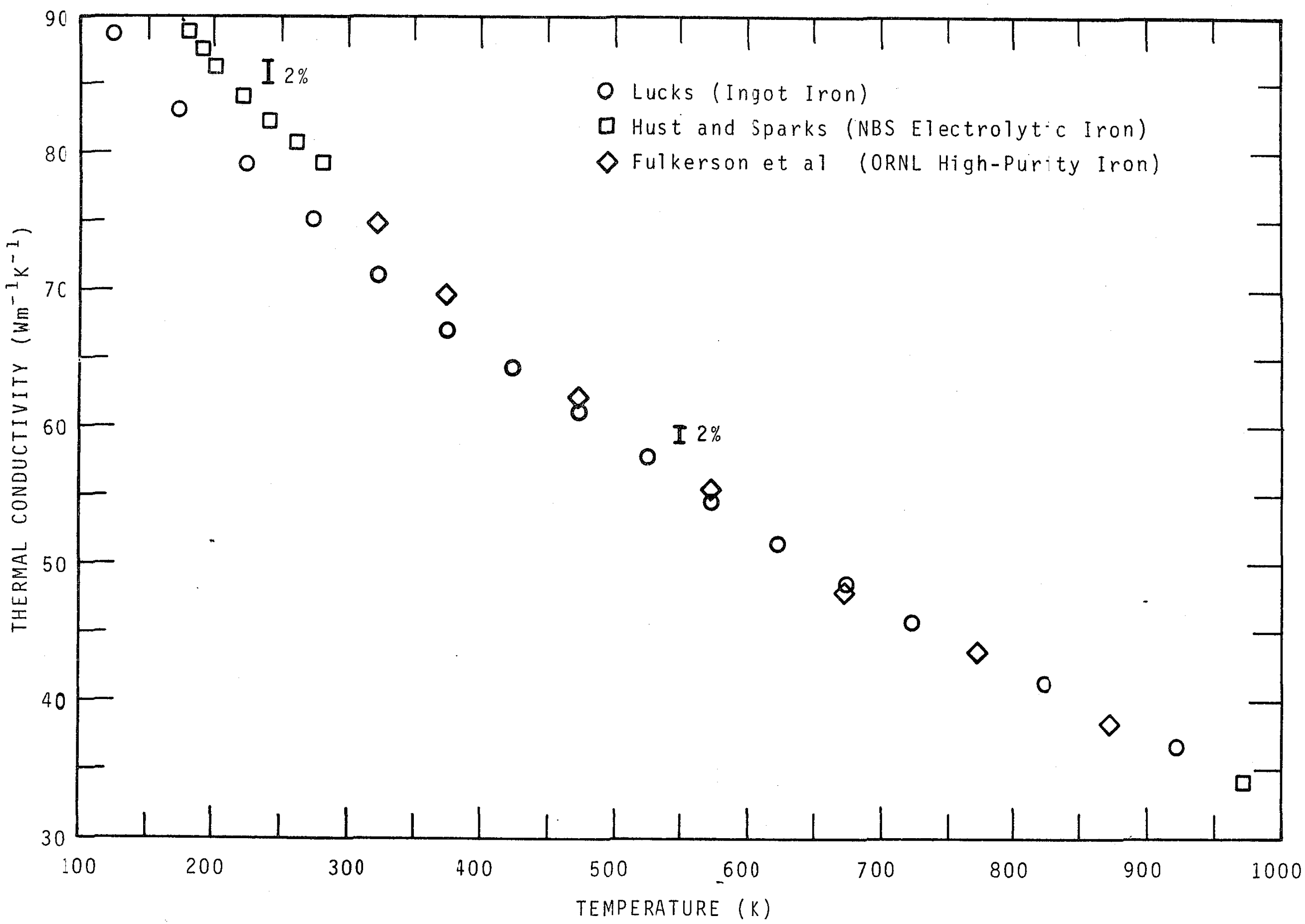


FIGURE 1 - Thermal Conductivity of NBS Electrolytic Iron, ORNL High- Purity Iron, and Ingot Iron above 100 K.

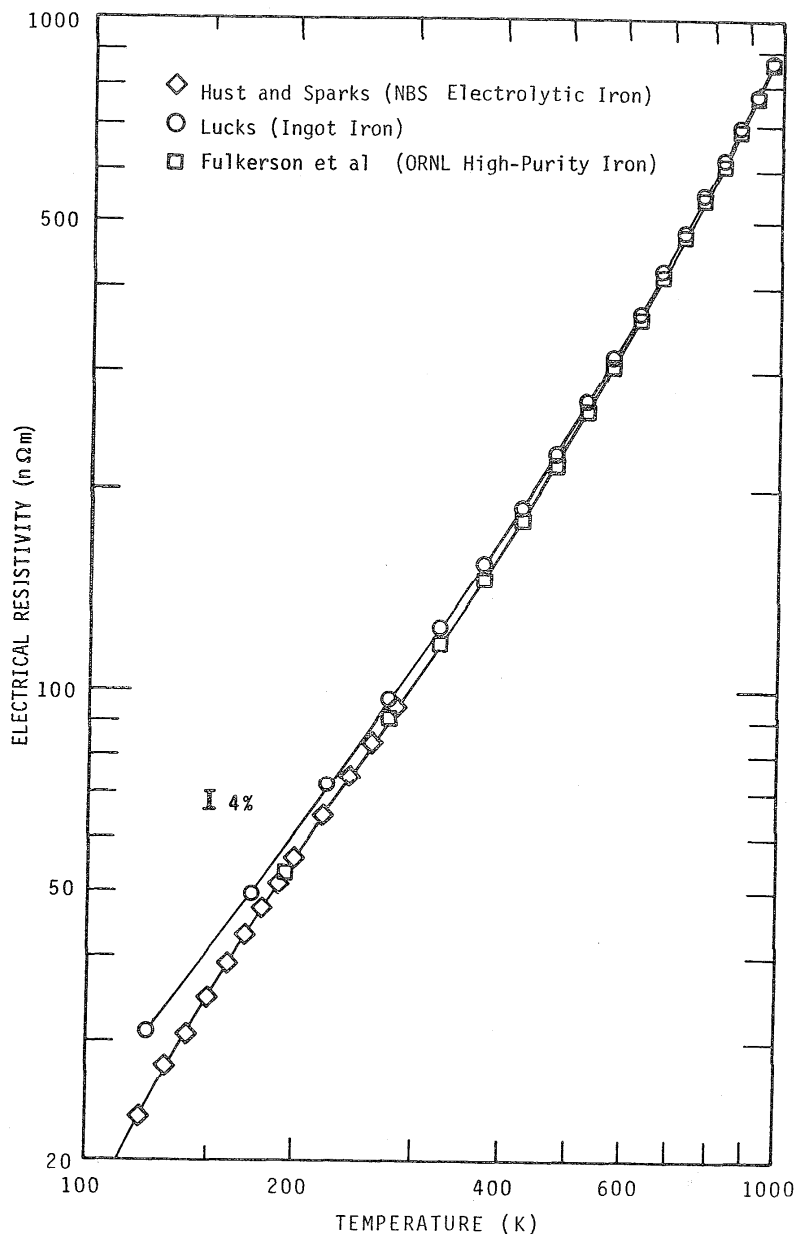
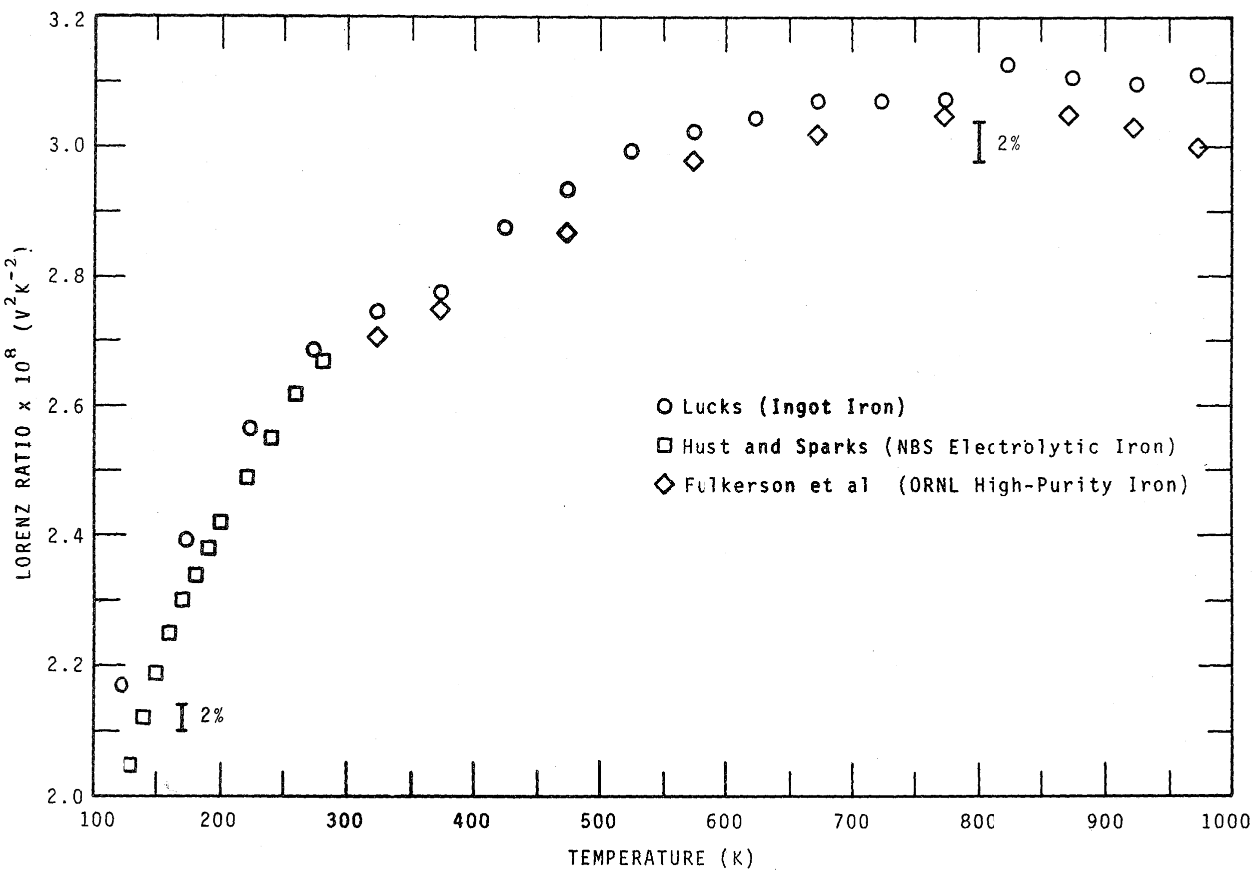


FIGURE 2 - Electrical Resistivity of NBS Electrolytic Iron, ORNL High-Purity Iron, and Ingot Iron above 100 K.



FIGJRE 3 - Lorenz Ratio of NBS Electrolytic Iron, ORNL High-Purity Iron, and Ingot Iron above 100 K.

Additional evidence that these data sets are compatible to within the stated uncertainties is illustrated in figures 4 and 5. Figure 4 is a plot of the intrinsic electrical resistivities, ρ_i, as obtained from Matthiessen's rule, $\rho = \rho_o + \rho_i$. The value of residual resistivity, ρ_o, used for ingot iron is 10 nΩm, as estimated from the literature cited by Lucks. The data above ambient were represented with $\rho_i = aT^n + bT$ and figure 5 illustrates the differences among the three data sets using this function as the baseline. The differences are consistent with the uncertainties of the data. Fulkerson et al [8] have reported increasing absolute resistivity differences between ingot iron and ORNL high-purity iron of about 1% from ambient to 1000 K, i.e., non-Matthiessen's rule behavior. This may be caused by an increase of impurities in solution for ingot iron as temperature increases, as discussed in the same paper [8]. In any event the effect on the difference between intrinsic resistivity as obtained for ORNL high-purity iron and NBS electrolytic iron should be below 1%.

The thermopower of NBS electrolytic iron was reported by Hust and Sparks [17] at temperatures up to 280 K. Fulkerson et al [8] reported thermopower of the ORNL high-purity iron and ingot iron above 273 K. The results, illustrated in figure 6, are in good agreement and again the values for NBS electrolytic iron are between those for the other two irons. Thermopower data by Hust et al [6] on ingot iron below 300 K are in excellent agreement with those published by Fulkerson et al [8].

Since the intrinsic electrical resistivities are in good agreement as shown in figures 4 and 5, we recommend the high-temperature ρ_i values as reference data for NBS electrolytic iron. Smoothed high temperature intrinsic resistivities were calculated from the equation

$$\rho_i = aT^n + bT.$$

The parameters $a = 6.512 \times 10^{-5}$, $n = 2.3438$, and $b = 0.1965$ were obtained by a least squares fit to the high temperature intrinsic resistivities. The ice point data on NBS electrolytic iron was used to constrain the function at 273.15 K. The deviations of this equation from the data of Fulkerson et al [8] are illustrated in figure 5. Also included in this plot are the data of Hust [14] above 200 K. Note that this equation joins with the low-temperature data of Hust [14] at 273 K but below 273 K the equation diverges rapidly from the three sets of data. The reader is therefore cautioned not to extrapolate this equation below 273 K. Total resistivities are then obtained by adding the residual resistivity of NBS electrolytic iron, 3.85 nΩm.

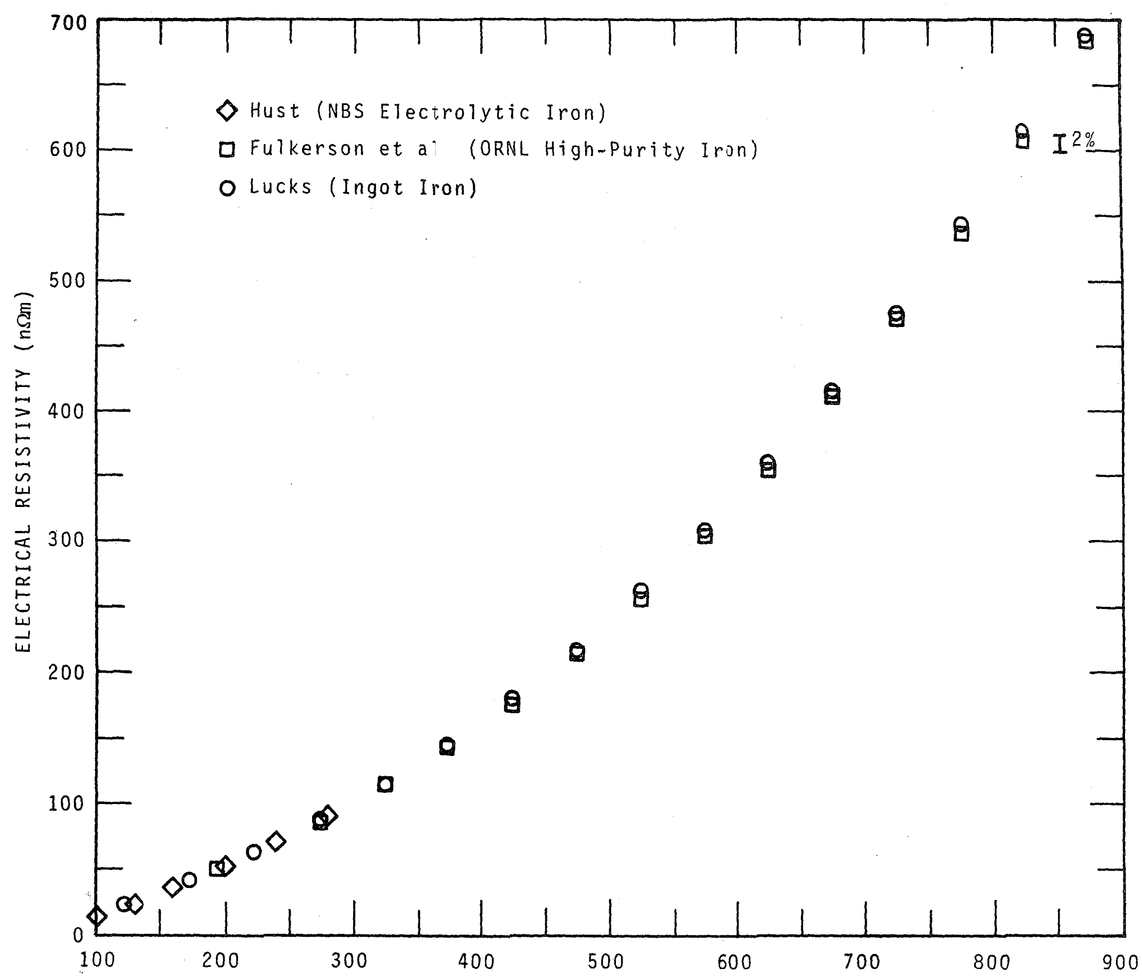


FIGURE 4 -Intrinsic Electrical Resistivity of NBS Electrolytic Iron, ORNL High-Purity Iron, and Ingot Iron above 100 K.

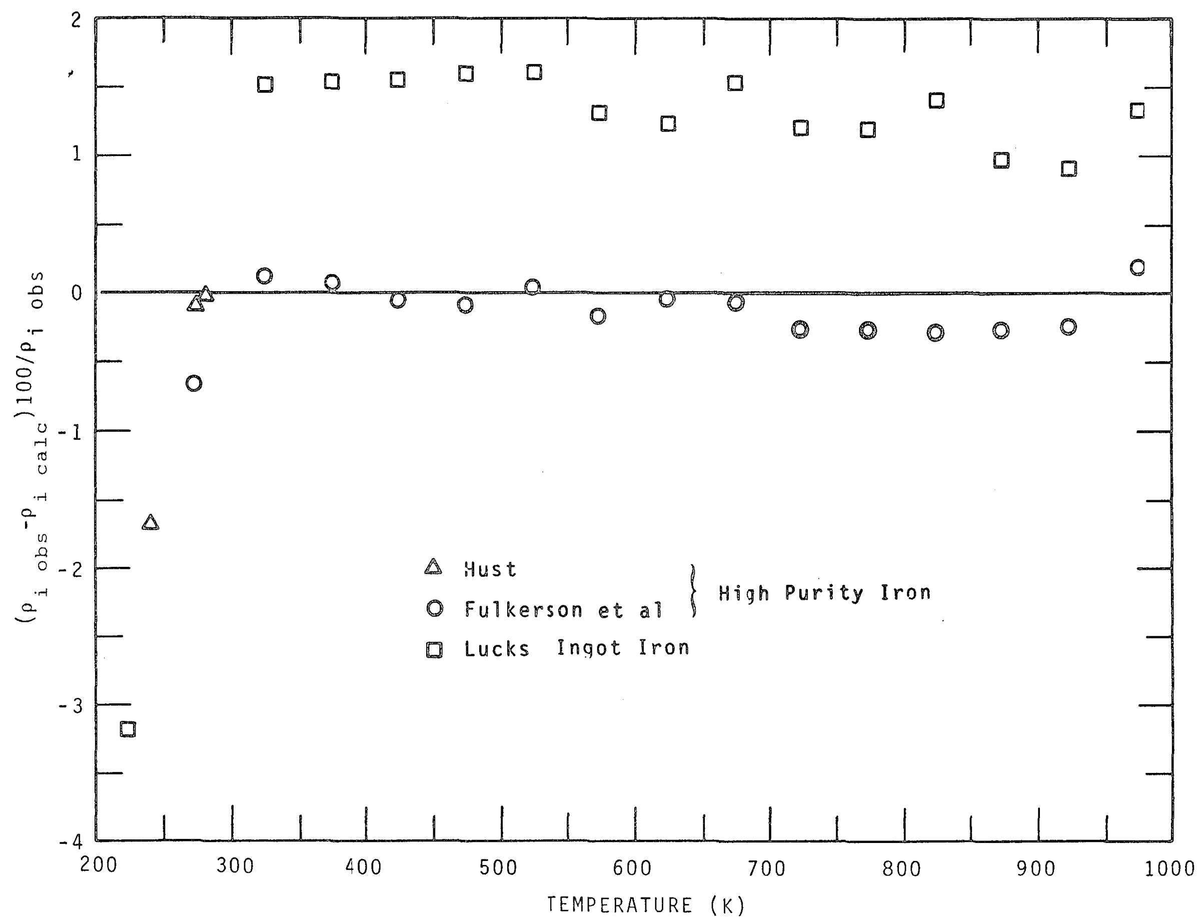


FIGURE 5 - Deviations of Intrinsic Electrical Resistivity Data for Iron from $\rho_i = aT^n + bT$.

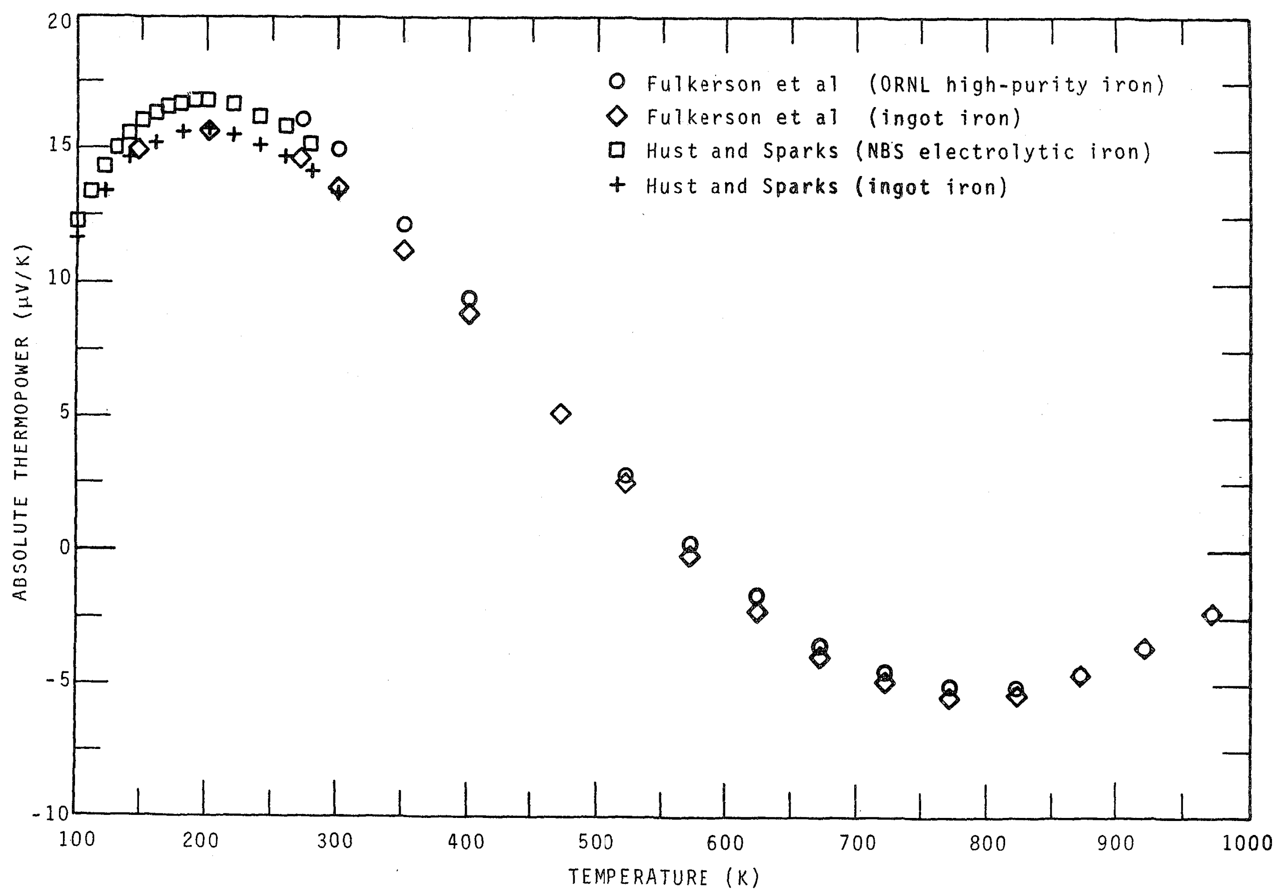


FIGURE 6 - Thermopower of NBS Electrolytic Iron, ORNL High-Purity Iron, and Ingot Iron above 100 K.

These high-temperature data combined with the data of Hust [14] are the recommended values for SRM 797 and are listed in table 3 and plotted in figure 7. The uncertainty of these values of electrical resistivity is estimated as 1% below 280 K and 2% above. The electrical resistivity values listed in table 3 are based on ambient temperature specimen dimensions, i.e., they are not corrected for thermal expansion. This is believed to be the most convenient form for the user. To obtain true resistivity one would increase the resistivity in table 3 linearly with temperature above ambient. The increase at 1000 K is about 1%.

Thermal conductivity values for NBS electrolytic iron at temperatures above ambient are obtained directly from the ORNL high purity iron data. Consideration was given to correcting these data to account for the slight residual resistivity difference (2%) between the ORNL high-purity and NBS electrolytic irons. This correction, however, is less than 0.2% above 280 K, which is negligible compared to the uncertainty of the recommended values (3%). The recommended thermal conductivities are listed in table 3 and plotted in figure 8. Lorenz ratios as calculated from the recommended values of electrical resistivity and thermal conductivity are listed in table 3 and plotted in figure 9.

7. Discussion

The principal factors determining the validity of SRM data are measurement uncertainty and material variability. Measurement uncertainty is a highly speculative quantity, as evidenced by the fact that most experimentalists present optimistically low uncertainties for their own work. The best way to obtain realistic uncertainties is through round-robin type measurements using apparatus as basically different as possible. Such programs are expensive and, therefore, not often performed. It is essential for standardizing laboratories to be involved in such programs for this forms the basis of essentially all other measurements. SRM's resulting from measurements by these standards laboratories make it possible for all other laboratories to perform measurements on a common basis.

Material variability is determined by the degree of control exercised during material production, and the sensitivity of property values to physical and chemical variations in the material. As pointed out earlier, however, transport properties at low temperatures are strongly dependent on the detailed nature of the microscopic material structure. Because of this, it is necessary to make measurements to determine the property variability of a lot of material produced even under the best of conditions. The only truly foolproof method of determining material variability effects is to measure the property of interest on a random sampling of specimens from the entire lot of material. For a thermal conductivity SRM, this is costly and one must resort to less expensive characterization measurements and careful production record keeping to insure maximum benefit from a minimum number of measurements.

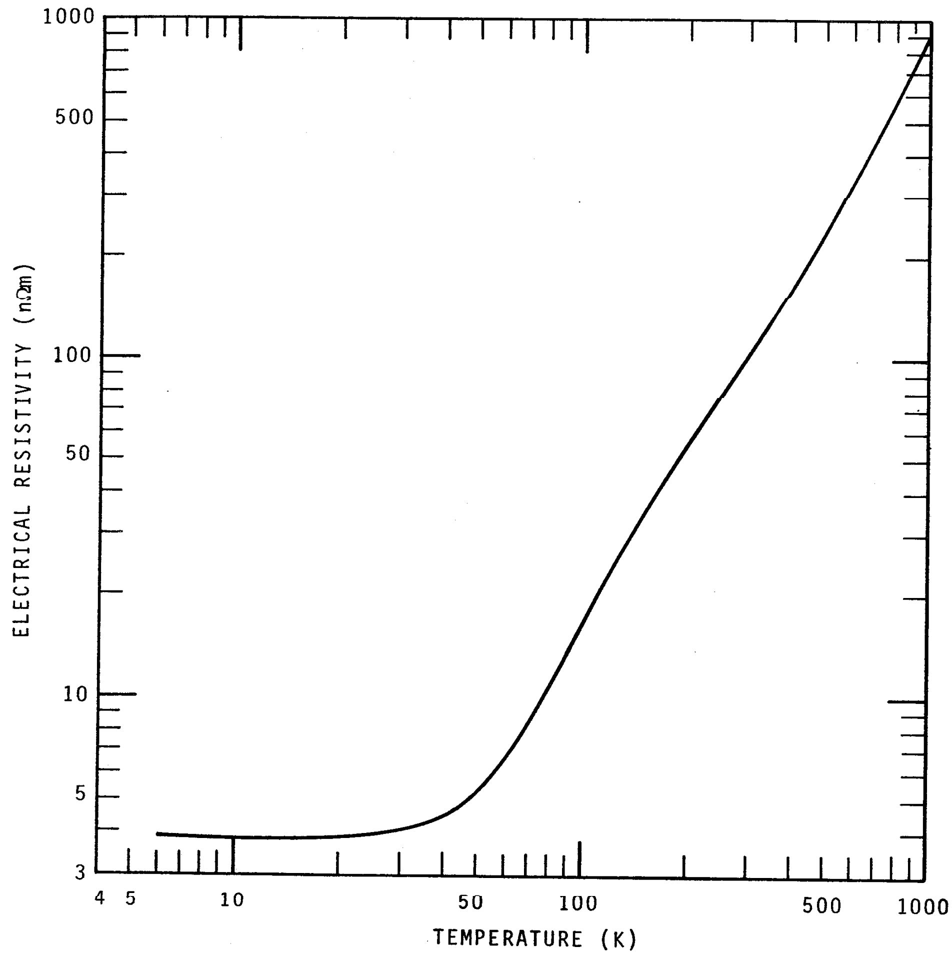


FIGURE 7 - Recommended Electrical Resistivity Values for NBS Electrolytic Iron. (SRM 797).

Table 3. Electrical resistivity, SRM 797, Thermal conductivity, SRM 734, and Lorenz ratio values for NBS electrolytic iron

Temp (K)	λ ($Wm^{-1}K^{-1}$)	ρ ($n\Omega m$)	L ($V^2K^{-2}x10^8$)
6	38.8	3.87	2.50
7	45.3	3.87	2.50
8	51.8	3.85	2.49
9	58.2	3.85	2.49
10	64.7	3.85	2.49
12	77.4	3.87	2.50
14	89.7	3.89	2.49
16	101	3.90	2.47
18	113	3.90	2.43
20	123	3.92	2.42
25	146	3.99	2.33
30	162	4.10	2.21
35	171	4.26	2.08
40	173	4.50	1.95
45	171	4.84	1.84
50	167	5.28	1.76
55	160	5.85	1.70
60	153	6.54	1.67
65	145	7.37	1.65
70	139	8.32	1.65
75	132	9.38	1.66
80	127	10.56	1.67
85	122	11.88	1.70
90	117	13.27	1.73
95	114	14.76	1.77
100	110	16.32	1.80
110	105	19.69	1.88
120	101	23.30	1.97
130	98.3	27.07	2.05
140	95.8	31.0	2.12
150	93.8	35.0	2.19
160	92.0	39.1	2.25
170	90.3	43.2	2.30
180	88.9	47.5	2.34
190	87.5	51.8	2.38
200	86.2	56.1	2.42

Table 3. Electrical resistivity, SRM 797, Thermal conductivity, SRM 734, and Lorenz ratio values for NBS electrolytic iron (continued)

Temp (K)	λ ($Wm^{-1}K^{-1}$)	ρ ($n\Omega m$)	L ($V^2K^{-2}x10^8$)
220	84.0	65.2	2.49
240	82.3	74.4	2.55
260	80.8	84.2	2.62
280	79.3	94.3	2.67
300	77.1	104	2.68
350	72.0	132	2.72
400	67.5	164	2.77
450	63.9	200	2.84
500	60.3	240	2.89
550	57.0	284	2.94
600	53.7	333	2.98
650	50.2	387	2.99
700	47.2	445	3.00
750	44.5	508	3.01
800	42.1	576	3.03
850	39.5	649	3.01
900	37.2	728	3.00
950	34.8	811	2.97
1000	32.5	901	2.92

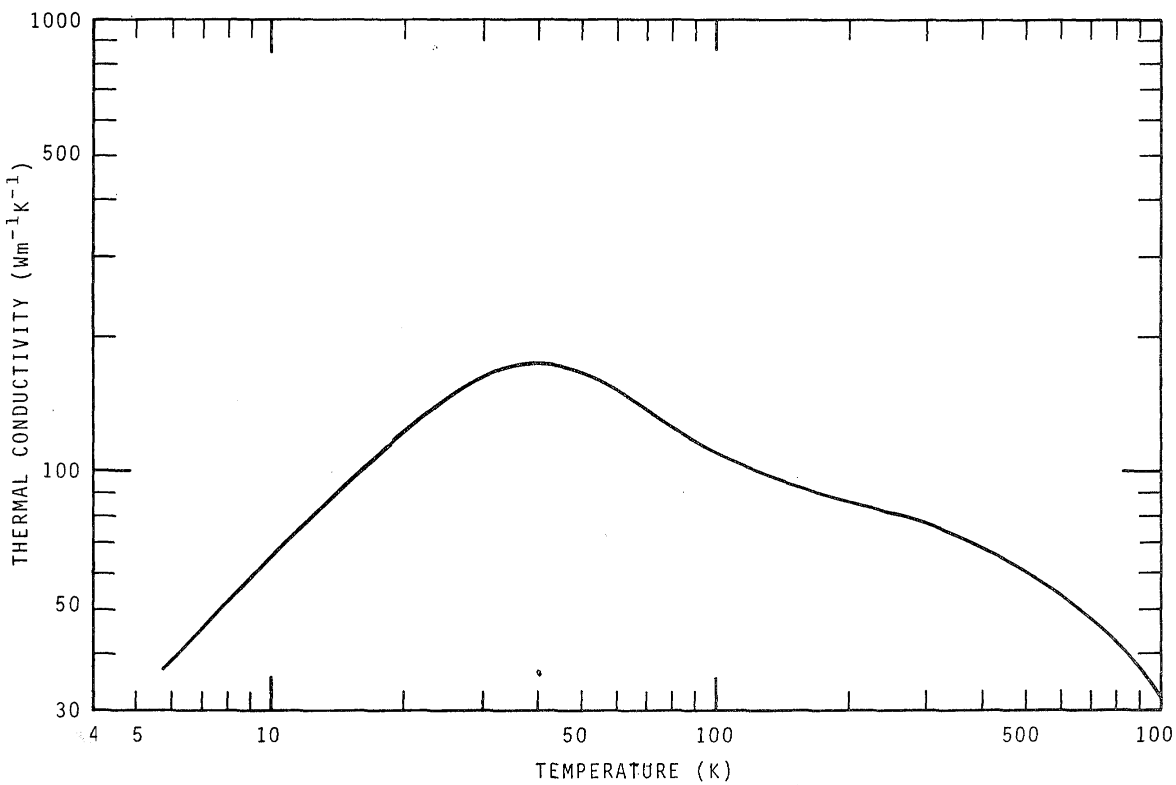


FIGURE 8 - Recommended Thermal Conductivity Values for NBS Electrolytic Iron.
(SRM 734)

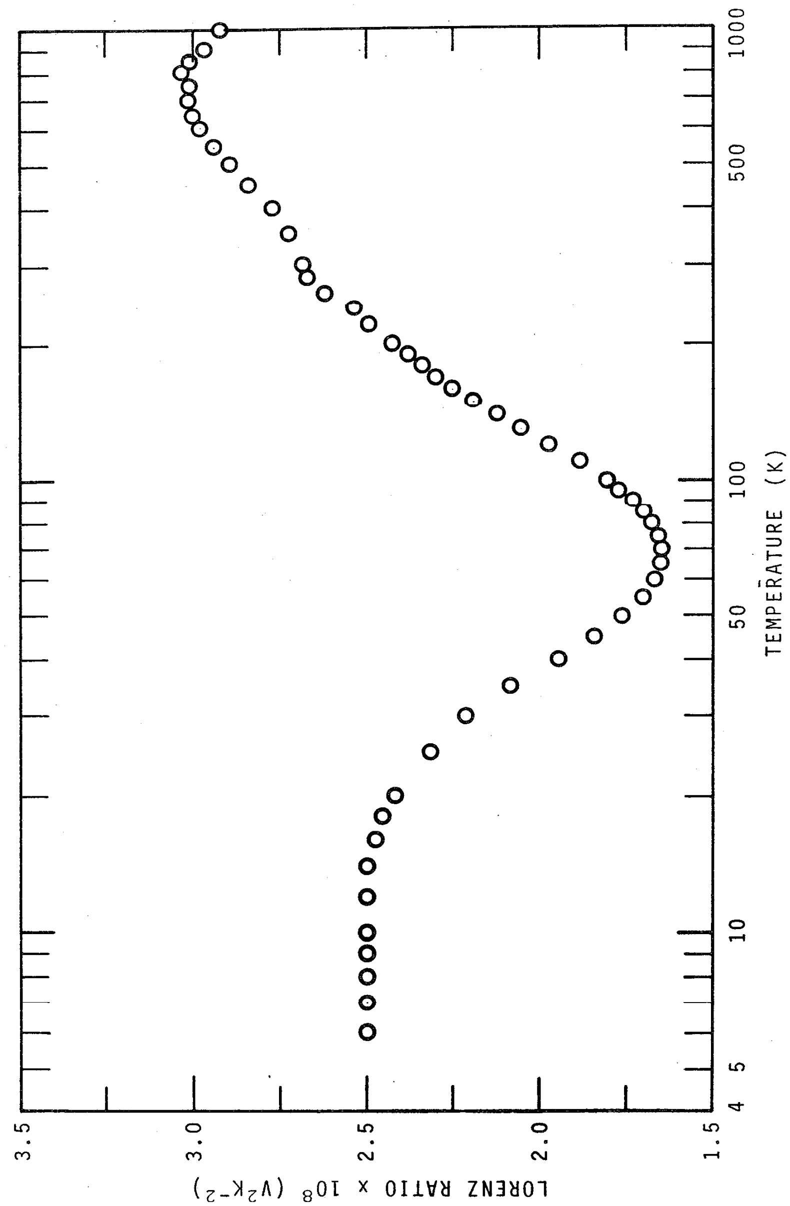


FIGURE 9 - Lorenz Ratio Values for NBS Electrolytic Iron.

Fixed-point electrical resistivity, density, grain size, and hardness data have been compared earlier in the text. These comparisons suggest that the effects of material variability in this electrolytic iron are not larger than 1% in thermal conductivity and electrical resistivity.

Although the SRM's described in this paper are considered quite adequate for engineering use, improvement in the accuracy and credibility of the values presented would be improved with additional measurements. Through its use as an SRM this material will be measured by other laboratories. These data will be compiled and when sufficient reduction in uncertainty is achievable, the recommended values will be updated. Anyone measuring this material with an absolute method is urged to make the data available to the author.

8. Summary

Recommended values of thermal conductivity (SRM 734) and electrical resistivity (SRM 797) for NBS electrolytic iron at temperatures from 4 to 1000 K have been presented. The values up to 280 K are based on direct measurements by Hust and Sparks [17]. Above 280 K the values are based on measurements reported by Fulkerson et al [8] on a similar iron and are confirmed by correlations with data for ingot iron. Material variability of NBS electrolytic iron affects the above values by no more than about ± 1%. Maximum uncertainties are estimated as follows:

	Thermal Conductivity	Electrical Resistivity
Below 280 K	2.5%	1%
Above 280 K	3 %	2%

These SRM's are available in the form of rods from the Office of Standard Reference Materials, National Bureau of Standards, Washington, D.C. 20234. Available sizes are as follows:

SRM 734-S	(0.64 cm diameter, 30 cm long)
SRM 734-L1	(3.17 cm diameter, 15 cm long)
SRM 734-L2	(3.17 cm diameter, 30 cm long)
SRM 797-1	(0.64 cm diameter, 5 cm long)
SRM 797-2	(0.64 cm diameter, 10 cm long)
SRM 797-3	(0.64 cm diameter, 15 cm long)

Longer continuous lengths can be obtained by special order.

9. Acknowledgements

This work has been in progress for several years and over this period of time many people have assisted in various ways. R. L. Powell was instrumental in suggesting the importance of the work and in formulating the preliminary program. Larry L. Sparks assisted in the measurement of thermal conductivity with the miltiproperty apparatus. Last, but not least, we wish to thank R. E. Michaelis of NBS, OSRM for supporting this work and for many helpful discussions.

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