

N72-12948

A UNITED STATES
DEPARTMENT OF
COMMERCE
PUBLICATION



NBS SPECIAL PUBLICATION 260-31
CASE FILE
COPY

Standard Reference Materials:

**THERMAL CONDUCTIVITY OF
ELECTROLYTIC IRON,
SRM 734, FROM 4 TO 300 K**

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

NATIONAL BUREAU OF STANDARDS

The National Bureau of Standards¹ was established by an act of Congress 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 consists of the Institute for Basic Standards, the Institute for Materials Research, the Institute for Applied Technology, the Center for Computer Sciences and Technology, and the Office for Information Programs.

THE INSTITUTE FOR BASIC STANDARDS provides the central basis within the United States of a complete and consistent system of physical measurement; coordinates that system with measurement systems of other nations; and furnishes essential services leading to accurate and uniform physical measurements throughout the Nation's scientific community, industry, and commerce. The Institute consists of a Center for Radiation Research, an Office of Measurement Services and the following divisions:

Applied Mathematics—Electricity—Heat—Mechanics—Optical Physics—Linac Radiation²—Nuclear Radiation²—Applied Radiation²—Quantum Electronics³—Electromagnetics³—Time and Frequency³—Laboratory Astrophysics³—Cryogenics³.

THE INSTITUTE FOR MATERIALS RESEARCH conducts materials research leading to improved methods of measurement, standards, and data on the properties of well-characterized materials needed by industry, commerce, educational institutions, and Government; provides advisory and research services to other Government agencies; and develops, produces, and distributes standard reference materials. The Institute consists of the Office of Standard Reference Materials and the following divisions:

Analytical Chemistry—Polymers—Metallurgy—Inorganic Materials—Reactor Radiation—Physical Chemistry.

THE INSTITUTE FOR APPLIED TECHNOLOGY provides technical services to promote the use of available technology and to facilitate technological innovation in industry and Government; cooperates with public and private organizations leading to the development of technological standards (including mandatory safety standards), codes and methods of test; and provides technical advice and services to Government agencies upon request. The Institute also monitors NBS engineering standards activities and provides liaison between NBS and national and international engineering standards bodies. The Institute consists of the following technical divisions and offices:

Engineering Standards Services—Weights and Measures—Flammable Fabrics—Invention and Innovation—Vehicle Systems Research—Product Evaluation Technology—Building Research—Electronic Technology—Technical Analysis—Measurement Engineering.

THE CENTER FOR COMPUTER SCIENCES AND TECHNOLOGY conducts research and provides technical services designed to aid Government agencies in improving cost effectiveness in the conduct of their programs through the selection, acquisition, and effective utilization of automatic data processing equipment; and serves as the principal focus within the executive branch for the development of Federal standards for automatic data processing equipment, techniques, and computer languages. The Center consists of the following offices and divisions:

Information Processing Standards—Computer Information—Computer Services—Systems Development—Information Processing Technology.

THE OFFICE FOR INFORMATION PROGRAMS promotes optimum dissemination and accessibility of scientific information generated within NBS and other agencies of the Federal Government; promotes the development of the National Standard Reference Data System and a system of information analysis centers dealing with the broader aspects of the National Measurement System; provides appropriate services to ensure that the NBS staff has optimum accessibility to the scientific information of the world, and directs the public information activities of the Bureau. The Office consists of the following organizational units:

Office of Standard Reference Data—Office of Technical Information and Publications—Library—Office of Public Information—Office of International Relations.

¹ Headquarters and Laboratories at Gaithersburg, Maryland, unless otherwise noted; mailing address Washington, D.C. 20234.

² Part of the Center for Radiation Research.

³ Located at Boulder, Colorado 80302.

UNITED STATES DEPARTMENT OF COMMERCE • Maurice H. Stans, *Secretary*
NATIONAL BUREAU OF STANDARDS • Lewis M. Branscomb, *Director*

Standard Reference Materials:

**Thermal Conductivity of Electrolytic Iron,
SRM 734, From 4 to 300 K**

J. G. Hust and L. L. Sparks

**Institute for Basic Standards
National Bureau of Standards
Boulder, Colorado 80302**



National Bureau of Standards Special Publication 260-31

Nat. Bur. Stand. (U.S.), Spec. Publ. 260-31, 19 pages, (Nov. 1971)
CODEN: XNBSA

Issued November 1971

For sale by the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402
(Order by SD Catalog No. C13.10:260-31). Price 35 cents.

Page Intentionally Left Blank

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.

Inquiries concerning the technical content of this paper should be directed to the author(s). Other questions concerned with the availability, delivery, price, and so forth will receive prompt attention from:

Office of Standard Reference Materials
National Bureau of Standards
Washington, D.C. 20234

J. Paul Cali, Chief
Office of Standard Reference Materials

OTHER NBS PUBLICATIONS IN THIS SERIES

- NBS Spec. Publ. 260, Catalog of Standard Reference Materials, July 1970. 75 cents.* (Supersedes NBS Misc. Publ. 260, January 1968 and NBS Misc. Publ. 241, March 1962.)
- NBS Misc. Publ. 260-1, Standard Reference Materials: Preparation of NBS White Cast Iron Spectrochemical Standards, June 1964. 30 cents.*
- NBS Misc. Publ. 260-2, Standard Reference Materials: Preparation of NBS Copper-Base Spectrochemical Standards, October 1964. 35 cents.*
- NBS Misc. Publ. 260-3, Standard Reference Materials: Metallographic Characterization of an NBS Spectrometric Low-Alloy Steel Standard, October 1964. 20 cents.*
- NBS Misc. Publ. 260-4, Standard Reference Materials: Sources of Information on Standard Reference Materials, February 1965. 20 cents.*
- NBS Misc. Publ. 260-5, Standard Reference Materials: Accuracy of Solution X-Ray Spectrometric Analysis of Copper-Base Alloys, March 1965. 25 cents.*
- NBS Misc. Publ. 260-6, Standard Reference Materials: Methods for the Chemical Analysis of White Cast Iron Standards, July 1965. 45 cents.*
- NBS Misc. Publ. 260-7, Standard Reference Materials: Methods for the Chemical Analysis of NBS Copper-Base Spectrochemical Standards, October 1965. 60 cents.*
- NBS Misc. Publ. 260-8, Standard Reference Materials: Analysis of Uranium Concentrates at the National Bureau of Standards, December 1965. 60 cents.*
- NBS Misc. Publ. 260-9, Standard Reference Materials. Half Lives of Materials Used in the Preparation of Standard Reference Materials of Nineteen Radioactive Nuclides Issued by the National Bureau of Standards, November 1965. 15 cents.*
- NBS Misc. Publ. 260-10, Standard Reference Materials: Homogeneity Characterization on NBS Spectrometric Standards-II: Cartridge Brass and Low-Alloy Steel, December 1965. 30 cents.*

NBS Misc. Publ. 260-11, Standard Reference Materials:
Viscosity of a Standard Lead-Silica Glass, November
1966. 25 cents.*

NBS Misc. Publ. 260-12, Standard Reference Materials:
Homogeneity Characterization of NBS Spectrometric
Standards III: White Cast Iron and Stainless Steel
Powder Compact, September 1966. 20 cents.*

NBS Misc. Publ. 260-13, Standard Reference Materials:
Mössbauer Spectroscopy Standard for the Chemical Shift
of Iron Compounds, July 1967. 40 cents.*

NBS Misc. Publ. 260-14, Standard Reference Materials:
Determination of Oxygen in Ferrous Materials --
SRM 1090, 1091, and 1092, September 1966. 30 cents.*

NBS Misc. Publ. 260-15, Standard Reference Materials:
Recommended Method of Use of Standard Light-Sensitive
Paper for Calibrating Carbon Arcs Used in Testing
Textiles for Colorfastness to Light, June 1967.
20 cents.*

NBS Spec. Publ. 260-16, Standard Reference Materials:
Homogeneity Characterization of NBS Spectrometric
Standards IV: Preparation and Microprobe Characterization
of W-20% Mo Alloy Fabricated by Powder Metallurgical
Methods, January 1969. 35 cents.*

NBS Spec. Publ. 260-17, Standard Reference Materials:
Boric Acid; Isotopic and Assay Standard Reference
Materials, February 1970. 65 cents.*

NBS Spec. Publ. 260-18, Standard Reference Materials:
Calibration of NBS Secondary Standard Magnetic Tape
(Computer Amplitude Reference) Using the Reference
Tape Amplitude Measurement "Process A", November 1969.
50 cents.*

NBS Spec. Publ. 260-19, Standard Reference Materials:
Analysis of Interlaboratory Measurements on the
Vapor Pressure of Gold (Certification of Standard
Reference Material 745), January 1970. 30 cents.*

NBS Spec. Publ. 260-20, Standard Reference Materials:
Preparation and Analysis of Trace Element Glass
Standards. (In preparation)

NBS Spec. Publ. 260-21, Standard Reference Materials:
Analysis of Interlaboratory Measurements on the Vapor
Pressures of Cadmium and Silver, January 1971. 35 cents.*

- NBS Spec. Publ. 260-22, Standard Reference Materials:
Homogeneity Characterization of Fe-3Si Alloy,
February 1971. 35 cents.*
- NBS Spec. Publ. 260-23, Standard Reference Materials:
Viscosity of a Standard Borosilicate Glass, December 1970.
25 cents.*
- NBS Spec. Publ. 260-24, Standard Reference Materials:
Comparison of Redox Standards. (In preparation)
- NBS Spec. Publ. 260-25, Standard Reference Materials:
A Standard Reference Material Containing Nominally Four
Percent Austenite, February 1971. 30 cents.*
- NBS Spec. Publ. 260-26, Standard Reference Materials:
National Bureau of Standards-U.S. Steel Corporation
Joint Program for Determining Oxygen and Nitrogen in
Steel, February 1971. 50 cents.*
- NBS Spec. Publ. 260-27, Standard Reference Materials:
Uranium Isotopic Standard Reference Materials,
April 1971. \$1.25.*
- NBS Spec. Publ. 260-28, Standard Reference Materials:
Preparation and Evaluation of SRM's 481 and 482 Gold-Silver
and Gold-Copper Alloys for Microanalysis. (In preparation)
- NBS Spec. Publ. 260-29, Standard Reference Materials:
Calibration of NBS Secondary Standard Magnetic Tape
(Computer Amplitude Reference) Using the Reference
Tape Amplitude Measurement "Process A-Model 2",
June 1971. 60 cents.*
- NBS Spec. Publ. 260-30, Standard Reference Materials:
Standard Samples Issued in the USSR (A Translation
from the Russian), June 1971. \$1.00.*

*Send order with remittance to: Superintendent of Documents,
U.S. Government Printing Office, Washington, D.C. 20402.
Remittance from foreign countries should include an
additional one-fourth of the purchase price for postage.

TABLE OF CONTENTS

	PAGE
Introduction	2
Apparatus and Data Analysis	3
Specimen Characterization	3
Results	7
Summary	8
Acknowledgements	8

LIST OF FIGURES

	PAGE
<u>Figure No.</u>	
1. Thermal Conductivity Deviations for Electrolytic Iron (SRM 734)	10
2. Thermal Conductivity of Electrolytic Iron (SRM 734)	11

LIST OF TABLES

	PAGE
<u>Table No.</u>	
1. Residual Resistivity Ratio of Electrolytic Iron (SRM 734)	12
2. Parameters for Equation (4)	13
3. Thermal Conductivity of Electrolytic Iron (SRM 734)	14

THERMAL CONDUCTIVITY OF ELECTROLYTIC IRON,

SRM 734, from 4 to 300 K*

J. G. Hust and L. L. Sparks

Institute for Basic Standards
National Bureau of Standards
Boulder, Colorado 80302

Thermal conductivity data are reported for a specimen of electrolytic iron, SRM 734, for temperatures from 4 to 300 K. Variability of this iron was studied by means of electrical residual resistivity ratio measurements on 63 specimens. This study showed that with a two-hour anneal at 1000 °C one can obtain a thermal conductivity Standard Reference Material that has variability of less than 1% in thermal conductivity.

Key words: Cryogenics; electrical resistivity; electrolytic iron; Lorenz ratio; Seebeck effect; thermal conductivity; transport properties.

*This work was carried out at the National Bureau of Standards, Boulder, Colorado under the sponsorship of the NASA-Space Nuclear Propulsion Office, Cleveland, Ohio, and the National Bureau of Standards Office of Standard Reference Materials (NBS-OSRM), Washington, D.C.

INTRODUCTION

This report results from a program to establish several thermal conductivity Standard Reference Materials (SRM's). Measurements are planned for Standard Reference Materials in the high, medium, and low conductivity ranges. The material reported on here, electrolytic iron SRM 734 [1], is in the high-to-medium range of conductivity.

Design and development engineers in the aerospace industry continue to have urgent need for thermal and mechanical property data for new materials. For most materials, especially new or uncommon alloys, measured values of thermal conductivity are not available and predictions cannot be made with adequate confidence. To help satisfy these needs, we have constructed an apparatus for the simultaneous measurement of thermal conductivity, electrical resistivity and thermo-power. Another phase of this program, to establish standard reference data on several Standard Reference Materials, has begun. We intend to measure several specimens of materials that appear to be useful as standards. Standard Reference Material data are useful for intercomparison of existing thermal conductivity apparatus, for debugging new apparatus, and for calibration of comparative apparatus. The apparent large differences among the results of various investigators for a given material (50% is not unheard of) is evidence of the need for intercomparisons, calibrations, and standardization. The availability of Standard Reference Materials will result in more accurate and more permanent transport property data for technically important solids.

The basic characteristics of a thermal conductivity Standard Reference Material are that it be: (a) stable and reproducible under the conditions of use, (b) uniform throughout a single specimen and from specimen-to-specimen, (c) similar in property value to the material that is to be determined in terms of it, (d) readily machined and fabricated in appropriate size and shape, (e) chemically inert to

the materials in the system to which it will be exposed, and (f) usable over a wide range of temperature. Electrolytic iron, SRM 734, does not satisfy (e) and (f) as well as might be desired; however, its availability from the Office of Standard Reference Materials (OSRM) in a large homogeneous lot and the past use of a similar iron as a thermal conductivity standard is considered sufficient justification for this work.

APPARATUS AND DATA ANALYSIS

The apparatus is based on the axial one-dimensional heat flow method. The specimen is a cylindrical rod 3.6 mm in diameter and 23 cm long with an electric heater at one end and a temperature controlled 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.

The experimental data are represented by arbitrary functions over the entire range and smooth tables are generated from these functions. The number of terms used to represent each of the data sets is optimized, through the use of orthonormal functions, so that none of the precision of the data is lost by underfitting, nor are any unnecessary oscillations introduced by overfitting. A detailed description of this apparatus and the methods of data analysis are given by Hust, et al. [2]

SPECIMEN CHARACTERIZATION

Density as measured by air and water weighings (see Bowman, et al. [3]) is $7.867 \pm 0.005 \text{ g/cm}^3$. Rockwell hardness and grain size are B23.5 and 0.0507 mm, respectively. The grain size was determined by the American Society for Testing and Materials (ASTM) comparative method. Each of these values is for the material in the annealed state as described later. The purity of this electrolytic iron is

99.9 + wt. percent. The material is similar in composition to SRM 1265, electrolytic iron, which is certified for its chemical composition. The certificate of analysis for SRM 1265 is shown as Appendix I.

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

$$\lambda = \lambda_e + \lambda_g \quad (1)$$

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 . 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:

$$\frac{\rho\lambda}{T} = L_0 = 2.443 \times 10^{-8} \text{V}^2\text{K}^{-2} \quad (2)$$

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 the thermal vibrations of the ions of the lattice. The former mechanism is 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_0 , 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:

$$\rho(T) = \rho_0 + \rho_i(T) \quad (3)$$

This separation of the total resistivity into a constant term (ρ_0) 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_0 . 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, the usual procedure is to determine a specimen's resistance at the ice-point, R_{273} , and at 4 K, R_4 , and to 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 per-

fection of a specimen.[4] Thus the variability in RRR for various specimens in a given lot of material is an indication of the variability in chemical impurity concentration and physical imperfection concentration in the lot. Such variability also affects the thermal conductivity as indicated by the WFL law. Therefore, a determination of RRR variability will directly indicate thermal conductivity variability. The determination of RRR is considerably easier than the determination of λ .

An extensive resistivity variability study was conducted on this electrolytic iron, the object being to determine if it could be heat treated in such a manner that the thermal conductivity variability would be acceptably small. This was achieved with a 2-hour, 1000 °C anneal in either a vacuum or helium atmosphere. The results of this study are shown as residual resistivity ratios in table 1. The ratio given is resistivity at 273.15 K to resistivity at 4 K. Specimens labeled C2T, A6L, C5L, A1L, and A5T were obtained from the 6.35 mm diameter rods; the remaining specimens were machined from 31.8 mm diameter rods. Based on the 63 residual resistivity ratio measurements made on these specimens in various stages of heat treatment, the following is concluded: The specimens machined from the large diameter rods are significantly different in residual resistivity ratio from the unmachined specimens in the as received condition. The ratio of the unmachined rods is 22.01 ± 0.20 while the ratio of the machined rods is 19.52 ± 0.44

Various heat treatments were tried to remove the differences in ratio of the two sets of rods. After heat treating at 500 °C for 1 hour, the ratios increased but were still different (ratio of unmachined rods = 23.53 ± 0.20 ; ratio of machined rods = 22.14 ± 0.34). Raising the temperature to 1000 °C for 2 hours produced rods which are indistinguishable, (ratio of unmachined rods, 23.39 ± 0.28 ; ratio of

machined rods, 23.29 ± 0.20 ; ratio of all rods, 23.33 ± 0.24). The variation shown is $2s$, where s is the estimated standard deviation and includes material and measurement variability. To study the possibility of a change in these ratios with age, one set of rods was measured after about 50 days from the $1000\text{ }^\circ\text{C}$ treatment; no significant change was detected (23.40 ± 0.20).

These measurements show that electrolytic iron SRM 734 can be used as a thermal conductivity standard below room temperature with an estimated material variability of about $\pm 1\%$ if annealed at $1000\text{ }^\circ\text{C}$ for 2 hours.

RESULTS

The thermal conductivity of specimen A5T was measured. The experimental data were functionally represented with the following equation:

$$\ln\lambda = \sum_{i=1}^n a_i [\ln T]^{i+1} \quad (4)$$

where λ = thermal conductivity and T = temperature. Temperatures are based on the IPTS-68 scale above 20 K and the NBS P2-20(1965) scale below 20 K. The parameters, a_i , determined by least squares, are presented in table 2. Further details of this procedure are described by Hust, et al. [2] The deviations of the experimental data from these equations are given in figure 1. Calculated values of λ are presented in table 3 and in figure 2.

A detailed error analysis for this system has been presented previously by Hust, et al. [2] Based on this analysis of systematic and random errors, the uncertainty estimates (with 95% confidence) are as follows:

2.5% at 300 K, decreasing as T^4 to 0.70% at 200 K, 0.70% from 200 K to 50 K, increasing inversely with temperature to 1.5% at 4 K.

SUMMARY

We have established low temperature thermal conductivity standard reference data for electrolytic iron SRM 734. Thermal conductivity measurements have been made on this iron from 6 to 300 K. These data were fitted to an empirical equation that was used to generate tabular values. Material variability is estimated to be less than $\pm 1\%$ in thermal conductivity, and measurement uncertainty is less than 2.5%.

ACKNOWLEDGEMENTS

We wish to thank R. E. Michaelis of NBS, OSRM, for supplying these specimens along with helpful discussions. This measurement program has been carried out under the helpful guidance of R. L. Powell.

FOOTNOTES AND REFERENCES

- [1] This SRM is available in the form of rods of two different diameters and may be ordered from the Office of Standard Reference Materials, National Bureau of Standards, Washington, D.C. 20234. SRM 734-S is a rod 6.4 mm (1/4 in.) in diameter and 305 mm (12 in.) in length. SRM 734-L1 is a rod 31.8 mm (1 1/4 in.) in diameter and 152 mm (6 in.) in length; SRM 734-L2 is the same diameter but 305 (12 in.) in length. Longer continuous lengths can be obtained by special order to the OSRM.
- [2] Hust, J. G., Powell, Robert L., and Weitzel, D. H., Thermal Conductivity Standard Reference Materials from 4 to 300 K: I. Armco Iron: Including Apparatus Description and Error Analysis, J. Res. Nat. Bur. Stand., (U.S.), 74A(Phys. and Chem.) 673-690(1970).
- [3] Bowman, H. A., and Schoonover, R. M., Procedure for High Precision Density Determinations by Hydrostatic Weighing, J. Res. Nat. Bur. Stand., (U.S.), 71C(Engr. and Instr.) 179-198(1967).
- [4] Since the specimens were in the annealed condition the RRR value should indicate the effective chemical purity (electrical purity) of the specimen. Using the specific resistivities listed by Blatt [5] and the measured chemical composition of this iron we obtain a residual resistivity of 5 n Ω m if all of the impurities were in solution. Since the measured residual resistivity is 4 n Ω m, the electrical purity is essentially the same as the chemical purity, 99.9%.
- [5] Blatt, F. J., Physics of Electronic Conduction in Solids, p. 199 (McGraw-Hill Book Co., Inc., New York, N. Y., 1968).

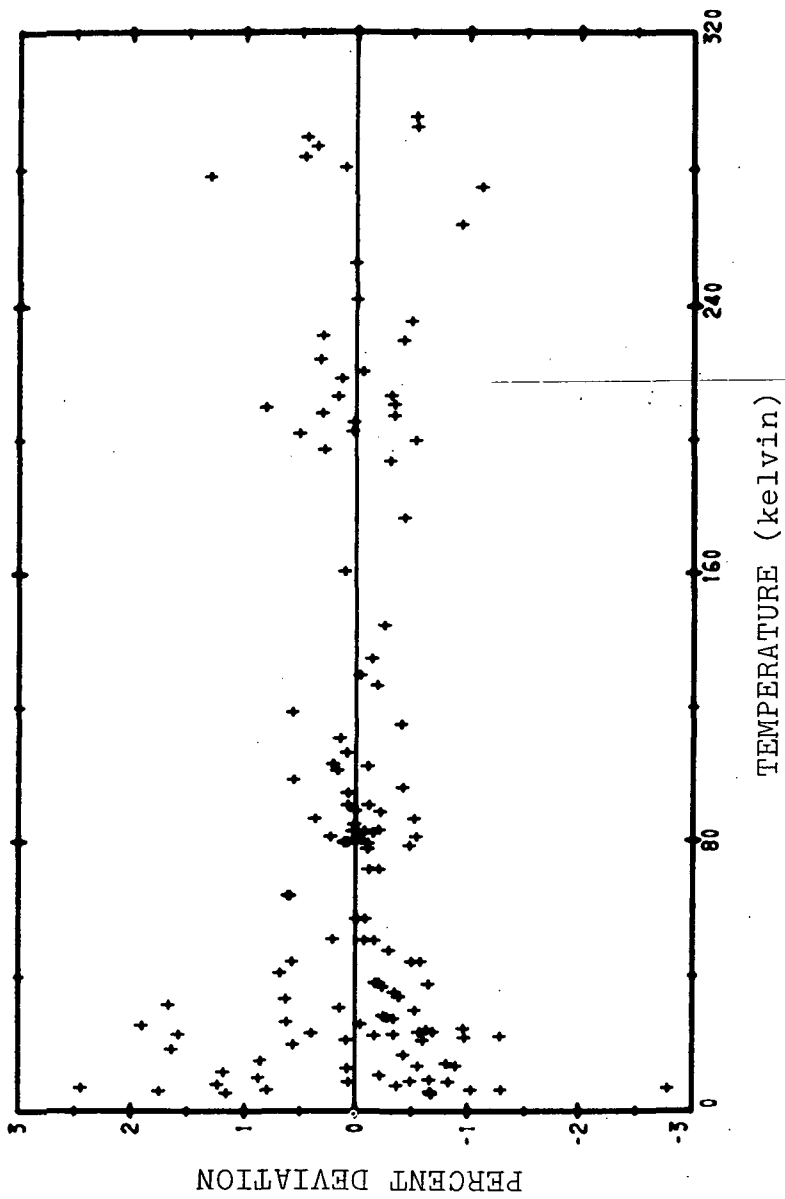


Figure 1. Thermal conductivity deviations for electrolytic iron (SRM 734)

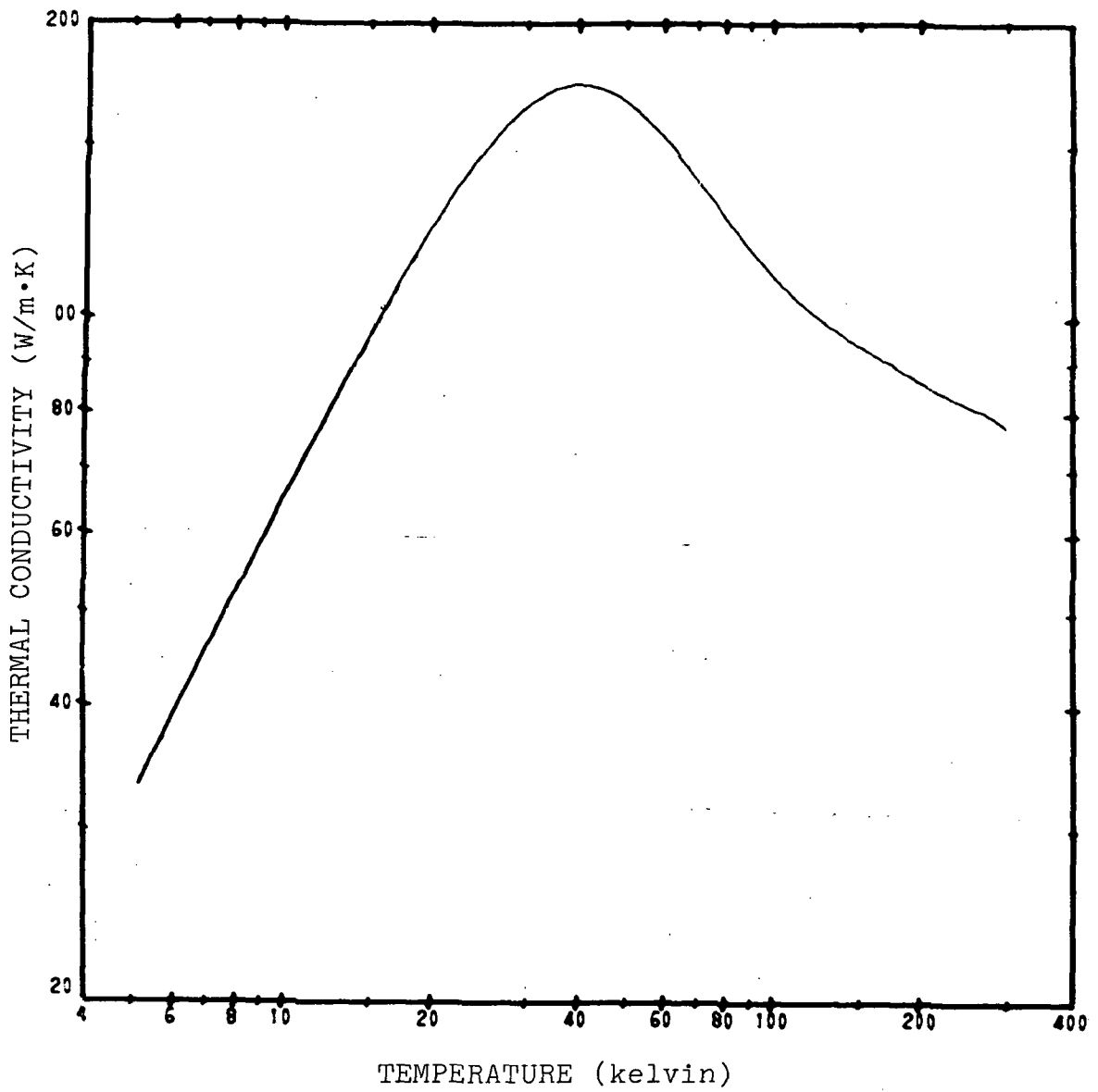


Figure 2. Thermal conductivity of electrolytic iron (SRM 734)

Table 1. Residual resistivity ratio (ρ_{273K} / ρ_{4K}) of electrolytic iron SRM 734

Specimen	Ratio					
	As received	500°C 1 hr	500°C 8 hr	1000°C 2 hr	400°C 2½ days	Aging 50 days
C2T	21.97(a)	23.53	24.12	23.31(e)	24.84	25.00
A6L	22.16	-----	-----	23.22(f)	24.85	24.97
C5L	21.94	-----	-----	23.40(f)	-----	23.47
A1L	22.04	-----	-----	23.59(c)	-----	23.52
A5T	22.03(b)	-----	-----	23.42(f)	-----	23.42
2A-1-1	19.35	21.96	22.32	23.47(e)	25.12	25.24
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	-----	-----	-----	-----	-----
2A-3-4	19.93	-----	-----	-----	-----	-----
2C-1-1	19.42	21.70	22.00	23.23(e)	-----	23.44
2C-1-2	19.12	-----	-----	23.41(c)	-----	23.40
2C-1-3	19.46	-----	-----	-----	-----	-----
2C-1-4	19.56	-----	-----	-----	-----	-----
2C-3-1	19.34	21.93	21.99	23.27(e)	-----	23.40
2C-3-2	19.49	-----	-----	-----	-----	-----
2C-3-3	19.57	-----	-----	-----	-----	-----
2C-3-4	19.40	-----	-----	-----	-----	-----

- (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

Table 2. Parameters for equation (4)

i	a_i
1	-1.48463068×10^1
2	6.93779265×10^1
3	-1.13470636×10^2
4	1.01420592×10^2
5	-5.68004853×10^1
6	2.10770015×10^1
7	-5.27537674×10^0
8	$8.81839451 \times 10^{-1}$
9	$-9.43950407 \times 10^{-2}$
10	$5.85191930 \times 10^{-3}$
11	$-1.59785857 \times 10^{-4}$

Table 3. Thermal conductivity of electrolytic iron (SRM 734)

Temp (K)	Thermal Conductivity ($\text{Wm}^{-1} \text{K}^{-1}$)	Temp (K)	Thermal Conductivity ($\text{Wm}^{-1} \text{K}^{-1}$)
6	38.8	75	132
7	45.3	80	127
8	51.8	85	122
9	58.2	90	117
10	64.7	95	114
12	77.4	100	110
14	89.7	110	105
16	101	120	101
18	113	130	98.3
20	123	140	95.8
25	146	150	93.8
30	162	160	92.0
35	171	170	90.3
40	173	180	88.9
45	171	190	87.5
50	167	200	86.2
55	160	220	84.0
60	153	240	82.3
65	145	260	80.8
70	139	280	79.3

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 1265

Electrolytic Iron

This standard is in the form of disks 32 mm (1 1/4 in) in diameter and 19 mm (3/4 in) thick, generally for use in optical emission and x-ray spectrometric analysis.^a

Element	Percent, by weight
Carbon	0.0067
Manganese0057
Phosphorus002 ₅
Sulfur0059
Silicon008 ₀
Copper0058
Nickel041
Chromium007 ₂
Vanadium0006
Molybdenum0050
Cobalt007 ₀
Titanium0006
Arsenic	(.0002) ^b
Aluminum (Total)	(.0007)
Boron00013
Lead00002
Iron (by difference)	99.9

^aThis material also is available in the form of chips, SRM 365, for use in chemical methods of analysis; rods, SRM 1099, 6.4 mm (1/4 in) in diameter and 102 mm (4 in) long for the determination of gases in metals by vacuum fusion and neutron activation methods of analyses; and rods, SRM 665, 3.2 mm (1/8 in) in diameter and 51 mm (2 in) long for application in microchemical methods of analysis such as electron probe microanalysis, spark source mass spectrometric analysis, and laser probe analysis.

^bValues in parenthesis are not certified since they are based on the results from a single laboratory.

CERTIFICATION: The value listed for a certified element is the best estimate of the true value based on the results of the cooperative analytical program. The value listed is not expected to deviate from the true value by more than ± 1 in the last significant figure reported; for a subscript figure, the deviation is not expected to be more than ± 5 . Based on the results of homogeneity testing, maximum variations within and among samples are estimated to be less than the accuracy figures given above.

Washington, D. C. 20234
 August 6, 1971

J. Paul Cali, Chief
 Office of Standard Reference Materials

(over)

PLANNING, PREPARATION, TESTING, ANALYSIS: This standard is one of five replacements for the original eight 1100 series iron and steel SRM's. Material from the same melt is available in a variety of forms to serve in checking methods of analysis and in calibrating instrumental techniques.

The material for this standard was vacuum melted and cast at the Carpenter Technology Corporation, Reading, Pennsylvania, under a contract with the National Bureau of Standards. The contract was made possible by a grant from the American Iron and Steel Institute.

The ingots were processed by Carpenter Technology Corporation to provide material of the highest possible homogeneity. Following acceptance of the composition based on NBS analyses, selected portions of the ingot material were extensively tested for homogeneity at NBS by J. R. Baldwin, D. M. Bouchette, S. D. Rasberry, and J. L. Weber, Jr. Only that material meeting a critical evaluation was processed to the final sizes.

Chemical analyses for certification were made on composite samples representative of the accepted lot of material.

Cooperative analyses for certification were performed in the Research Laboratories of Armco Steel Corporation by R. L. LeRoy and J. F. Woodruff.

Analyses were performed in the Analytical Chemistry Division of the National Bureau of Standards by the following: R. Alvarez, J. R. Baldwin, E. Belkas, B. S. Carpenter, M. M. Darr, E. R. Deardorff, E. L. Garner, T. E. Gills, L. A. Machlan, E. J. Maienthal, L. J. Moore, C. W. Mueller, T. J. Murphy, P. J. Paulsen, K. M. Sappenfield, B. A. Thompson, and S. A. Wicks.

The overall direction and coordination of the technical measurements at NBS leading to certification were performed under the direction of O. Menis, B. F. Scribner, J. I. Shultz, and J. L. Weber, Jr.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. E. Michaelis.

ADDITIONAL INFORMATION ON THE COMPOSITION: Certification is made only for the elements indicated. The five replacements, however, contain a graded series for 40 elements and information on the elements not initially certified may be of importance in the use of the material. Although these are not certified, upper limit values are presented in the following table for the remaining elements. (Some may be certified at a later date.)

Elements Detected (ppm by weight)

<u>Element</u>	<u>Upper Limit</u>	<u>(Estimated value)</u>	<u>Method</u>
W	< 1	(0.4)	Neutron activation
Sn	< 5	(2)	Spark source mass spectrometry
Nb	< 0.5	(<0.1)	Spark source mass spectrometry
Ag	< 0.2	(0.02)	Spark source mass spectrometry
Zn	< 3	(2)	Spark source mass spectrometry
N	<20	(~11)	Distillation-photometric
Ge	<50	(~14)	Spark source mass spectrometry
O	<70	(63)	Vacuum fusion
H	< 5	(1)	Vacuum fusion

Elements Sought But Not Detected (ppm by weight)

<u>Element</u>	<u>Upper Limit</u>	<u>Method</u>
Ta	<0.5	Neutron activation
Zr	<0.1	Spark source mass spectrometry
Sb	<0.5	Neutron activation
Bi	<0.1	Spark source mass spectrometry
Ca	<0.1	Atomic absorption
Mg	<0.2	Atomic absorption
Se	<0.1	Spark source mass spectrometry
Te	<0.1	Spark source mass spectrometry
Ce	<0.05	Spark source mass spectrometry
La	<0.05	Spark source mass spectrometry
Pr	<0.05	Spark source mass spectrometry
Au	<0.02	Neutron activation
Hf	<0.2	Spark source mass spectrometry
Nd	<0.05	Spark source mass spectrometry

U.S. DEPT. OF COMM. BIBLIOGRAPHIC DATA SHEET	1. PUBLICATION OR REPORT NO. Spec. Publ. 260-31	2. Gov't Accession No.	3. Recipient's Accession No.
4. TITLE AND SUBTITLE Thermal Conductivity of Electrolytic Iron, SRM 734, from 4 to 300 K		5. Publication Date November 1971	
7. AUTHOR(S) J. G. Hust and L. L. Sparks		6. Performing Organization Code	
9. PERFORMING ORGANIZATION NAME AND ADDRESS NATIONAL BUREAU OF STANDARDS DEPARTMENT OF COMMERCE WASHINGTON, D.C. 20234		8. Performing Organization	
12. Sponsoring Organization Name and Address Same		10. Project/Task/Work Unit No. 3020585	
15. SUPPLEMENTARY NOTES		11. Contract/Grant No.	
12. Sponsoring Organization Name and Address		13. Type of Report & Period Covered Final	
16. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here.) Thermal conductivity data are reported for a specimen of electrolytic iron, SRM 734, for temperatures from 4 to 300 K. Variability of this iron was studied by means of electrical residual resistivity ratio measurements on 63 specimens. This study showed that with a two-hour anneal at 1000 °C one can obtain a thermal conductivity Standard Reference Material that has variability of less than 1% in thermal conductivity.		14. Sponsoring Agency Code	
17. KEY WORDS (Alphabetical order, separated by semicolons)Cryogenics; electrical resistivity; electrolytic iron; Lorenz ratio; Seebeck effect; thermal conductivity; transport properties			
18. AVAILABILITY STATEMENT <input checked="" type="checkbox"/> UNLIMITED.		19. SECURITY CLASS (THIS REPORT) UNCLASSIFIED	21. NO. OF PAGES 19
FOR OFFICIAL DISTRIBUTION. DO NOT RELEASE TO NTIS.		20. SECURITY CLASS (THIS PAGE) UNCLASSIFIED	22. Price 35¢

NBS TECHNICAL PUBLICATIONS

PERIODICALS

JOURNAL OF RESEARCH reports National Bureau of Standards research and development in physics, mathematics, chemistry, and engineering. Comprehensive scientific papers give complete details of the work, including laboratory data, experimental procedures, and theoretical and mathematical analyses. Illustrated with photographs, drawings, and charts.

Published in three sections, available separately:

• Physics and Chemistry

Papers of interest primarily to scientists working in these fields. This section covers a broad range of physical and chemical research, with major emphasis on standards of physical measurement, fundamental constants, and properties of matter. Issued six times a year. Annual subscription: Domestic, \$9.50; \$2.25 additional for foreign mailing.

• Mathematical Sciences

Studies and compilations designed mainly for the mathematician and theoretical physicist. Topics in mathematical statistics, theory of experiment design, numerical analysis, theoretical physics and chemistry, logical design and programming of computers and computer systems. Short numerical tables. Issued quarterly. Annual subscription: Domestic, \$5.00; \$1.25 additional for foreign mailing.

• Engineering and Instrumentation

Reporting results of interest chiefly to the engineer and the applied scientist. This section includes many of the new developments in instrumentation resulting from the Bureau's work in physical measurement, data processing, and development of test methods. It will also cover some of the work in acoustics, applied mechanics, building research, and cryogenic engineering. Issued quarterly. Annual subscription: Domestic, \$5.00; \$1.25 additional for foreign mailing.

TECHNICAL NEWS BULLETIN

The best single source of information concerning the Bureau's research, developmental, cooperative, and publication activities, this monthly publication is designed for the industry-oriented individual whose daily work involves intimate contact with science and technology—for *engineers, chemists, physicists, research managers, product-development managers, and company executives*. Annual subscription: Domestic, \$3.00; \$1.00 additional for foreign mailing.

Order NBS publications from:

Superintendent of Documents
Government Printing Office
Washington, D.C. 20402

NONPERIODICALS

Applied Mathematics Series. Mathematical tables, manuals, and studies.

Building Science Series. Research results, test methods, and performance criteria of building materials, components, systems, and structures.

Handbooks. Recommended codes of engineering and industrial practice (including safety codes) developed in cooperation with interested industries, professional organizations, and regulatory bodies.

Special Publications. Proceedings of NBS conferences, bibliographies, annual reports, wall charts, pamphlets, etc.

Monographs. Major contributions to the technical literature on various subjects related to the Bureau's scientific and technical activities.

National Standard Reference Data Series. NSRDS provides quantitative data on the physical and chemical properties of materials, compiled from the world's literature and critically evaluated.

Product Standards. Provide requirements for sizes, types, quality, and methods for testing various industrial products. These standards are developed cooperatively with interested Government and industry groups and provide the basis for common understanding of product characteristics for both buyers and sellers. Their use is voluntary.

Technical Notes. This series consists of communications and reports (covering both other agency and NBS-sponsored work) of limited or transitory interest.

Federal Information Processing Standards Publications. This series is the official publication within the Federal Government for information on standards adopted and promulgated under the Public Law 89-306, and Bureau of the Budget Circular A-86 entitled, Standardization of Data Elements and Codes in Data Systems.

Consumer Information Series. Practical information, based on NBS research and experience, covering areas of interest to the consumer. Easily understandable language and illustrations provide useful background knowledge for shopping in today's technological marketplace.

NBS Special Publication 305, Supplement 1, Publications of the NBS, 1968-1969. When ordering, include Catalog No. C13.10:305. Price \$4.50; \$1.25 additional for foreign mailing.

UNITED STATES
GOVERNMENT PRINTING OFFICE
PUBLIC DOCUMENTS DEPARTMENT
WASHINGTON, D.C. 20402

OFFICIAL BUSINESS

PENALTY FOR PRIVATE USE, \$300

POSTAGE AND FEES PAID
U.S. GOVERNMENT PRINTING OFFICE

