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NBS SPECIAL PUBLICATION 260-31

Standard Reference Materials:

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

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Standard Reference Materials:

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

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

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THERMAL CONDUCTIVITY OF ELECTROLYTIC IRON, SRM 734, from 4 to 300 K*

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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.

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*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 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.

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

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:

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

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_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:

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

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, 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 perfection 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 °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 ± 1% if annealed at 1000 °C for 2 hours.

RESULTS

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

$$\ell n\lambda = \sum_{i=1}^{n} a[\ell nT]^{i+1} \qquad (4)$$

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 ± 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.

[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 nΩm if all of the impurities were in solution. Since the measured residual resistivity is 4 nΩm, 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).

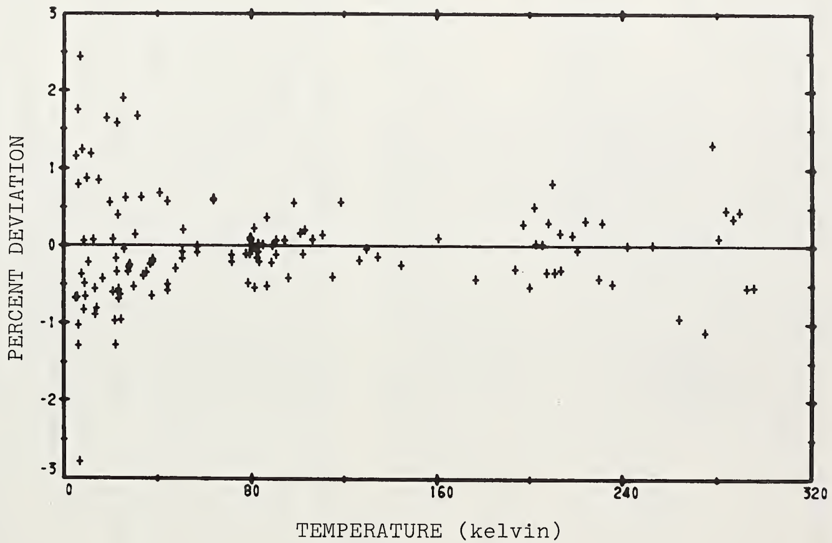

Figure 1. Thermal conductivity deviations for electrolytic iron (SRM 734)

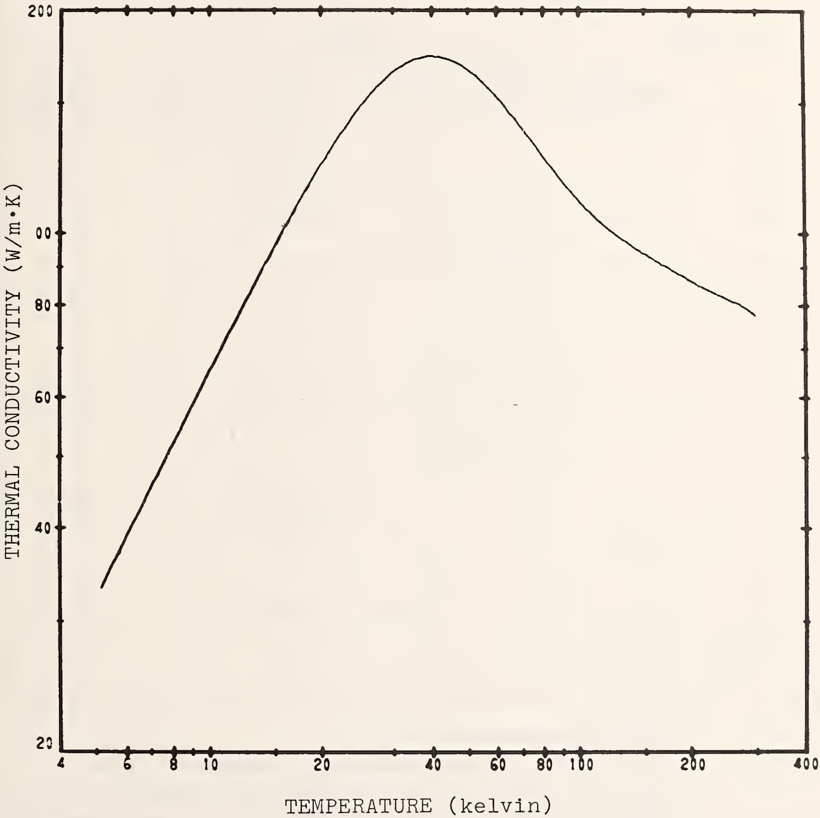

Figure 2. Thermal conductivity of electrolytic iron (SRM 734)

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	$-1.48463068 \times 10^{1}$
2	6.93779265×10^{1}
3	$-1.13470636 \times 10^{2}$
4	1.01420592×10^{2}
5	$-5.68004853 \times 10^{1}$
6	2.10770015×10^{1}
7	$-5.27537674 \times 10^{0}$
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 ($Wm^{-1} K^{-1}$)	Temp (K)	Thermal Conductivity ($Wm^{-1} K^{-1}$)
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

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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
Manganese	.0057
Phosphorus	$.002_5$
Sulfur	.0059
Silicon	$.008_0$
Copper	.0058
Nickel	.041
Chromium	$.007_2$
Vanadium	.0006
Molybdenum	.0050
Cobalt	$.007_0$
Titanium	.0006
Arsenic	(.0002)[b]
Aluminum (Total)	(.0007)
Boron	.00013
Lead	.00002
Iron (by difference)	99.9

[a]This 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.
[b]Values in parenthesis are not certified since they are based on the results from a single laboratory.

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Washington, D. C. 20234
August 6, 1971

J. Paul Cali, Chief
Office of Standard Reference Materials

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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)

Element	Upper Limit	(Estimated value)	Method
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)

Element	Upper Limit	Method
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

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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.

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