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

Standard Reference Materials:

THERMAL CONDUCTIVITY AND ELECTRICAL RESISTIVITY STANDARD REFERENCE MATERIALS: AUSTENITIC STAINLESS STEEL, SRM's 735 AND 798, FROM 4 TO 1200 K

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Standard Reference Materials:

Thermal Conductivity and Electrical Resistivity Standard Reference Materials: Austenitic Stainless Steel, SRM's 735 and 798, from 4 to 1200 K

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A historical review of the development of selected thermophysical Standard Reference Materials, SRM's, is given and selection criteria for those SRM's are listed. Thermal conductivity and electrical resistivity data for austenitic stainless steel, SRM's 735 and 798, are critically evaluated. Recommended values are presented for the temperature range 4 to 1200 K. Material variability studies, including the effects of heat treatment, have been performed at low temperatures. No irreversible transformations are observed up to temperatures of 1200 K. Based on the results of several types of characterization measurements, effects of material variability are believed to be less than 1% in electrical resistivity and not significantly more in thermal conductivity. The uncertainty of the recommended electrical resistivity data is estimated at 1% at low temperatures and 2% at higher temperatures. The corresponding uncertainty for thermal conductivity is 2% below 100 K, increasing to 3% at 300 K, and 5% at higher temperatures.

Key Words: Austenitic stainless steel, electrical resistivity, high temperature, low temperature, standard reference materials, thermal conductivity.

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 virtually impossible. 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

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* 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 Standards.

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 electrolytic iron (medium-to-high conductivity, 4 to 1000 K). The material reported on here, austenitic stainless steel, is in the low conductivity range.

This paper reviews the historical development of thermal conductivity SRM's. A listing is given of selection criteria for SRM's and a justification is presented for the establishment of both engineering and standards laboratory SRM's. Data are compiled and best values are selected to establish austenitic stainless steel as electrical resistivity and thermal conductivity SRM's 798 and 735, respectively. As discussed later, thermal conductivity and electrical resistivity data have been obtained to certify these SRM's over the range 4 to 1200 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 research 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.

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 which has been done on this material and recommends 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 which 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, 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 12" 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.

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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 [8] 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 [9] 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 60's. This project has been continued by the first author 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 temperatures 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 [10]. 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 [11]. Minges has also summarized some of the results on AFML-AGARD programs [12]. 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 austenitic steel described in this report satisfies these criteria reasonably well.

4. Material Characterization

Austenitic stainless steel was established as a low-temperature (5 to 280 K) thermal conductivity Standard Reference Material (SRM 735) in 1972 by Hust and Sparks [13]. In 1974 this material was established as an electrical resistivity Standard Reference Material (SRM 798) over the same temperature range by Hust [14]. This steel was originally prepared and distributed for study as a high-temperature reference material of thermophysical properties by AGARD, NATO [11]. Several specimens of the AGARD austenitic steel were measured over the low-temperature range at this laboratory in advance of the completion of the AGARD program. Because of its excellent low-temperature behavior it was established as an SRM prior to the completion of the high-temperature AGARD work.

The high-temperature AGARD program has now been completed and the results indicate that this austenitic stainless steel will be a useful SRM of thermal conductivity and electrical resistivity up to temperatures of about 1200 K. The purpose of this special publication is to present an analysis of the AGARD and NBS data, to select best values, and to indicate the uncertainty of these values. The characterization data, described in the following sections, and the accord of the measurements performed at low and high temperatures is sufficient to document the data on this material for use as an SRM of electrical resistivity and thermal conductivity.

4.1 Material Acquisition and Fabrication

Initially, AGARD supplied NBS with four 12" long rods for characterization and measurement. Two of these rods were 6 mm diameter and two were 10 mm diameter. The smaller diameter rods were used for fixed-point electrical resistivity measurements, while the larger two were machined for measurement in the multi-property apparatus (thermal conductivity, electrical resistivity, and thermopower). The larger diameter rods were also used for grain size, hardness, and density measurements. No heat treatment was given these rods after machining. This material, referred to as lot 1, is composed of melts A and B (See table 1).

These preliminary measurements indicated this material to be an excellent low-temperature SRM candidate. Therefore, another lot of this material was purchased from the German manufacturer that produced lot 1. Twelve rods, each 35 mm in diameter and 1 meter long, were obtained, fabricated to appropriate size, and placed in NBS, OSRM stock. This material is referred to as lot 2. Five cm long pieces were cut from the ends of six of these rods for electrical resistivity and other characterization measurements. No heat treatment was given these rods after quartering and machining to 6.25 mm diameter rods. The characterization measurements showed that lot 2 is indistinguishable from lot 1.

The next step was to prepare SRM specimens from the 35 mm diameter rods of lot 2. Because of the high specimen preparation costs and excessive material waste, that would result if the specimens were machined to size, we decided to hot-swage the 35 mm diameter rods to the desired size. This, of course, raises the question whether the material properties remain unchanged after swaging and reannealing. We performed additional characterization measurements on several specimens prepared in this manner by the U.S. Bureau of Mines, Albany, Oregon. The final anneal was the same as that given in table 1. The results of these measurements indicate again that the resulting specimens are indistinguishable from the specimens prepared originally from lots 1 and 2.

4.2 Composition

The aim composition and actual compositions of lot 1 (two melts) were reported by Fitzer in his 1969 progress report [11]. These data along with density values are listed in table 1. The reports by Fitzer also contain data on thermal expansion, specific heat, thermal conductivity, and electrical resisitivity at temperatures from 20 to 900°C. No other characterization data are presented.

Table 1 - Characterization data of austenitic stainless steel, lot 1, as determined by Spyra - AGARD participant No. 7, and reported by Fitzer

Composition in Weight %

Element	Aim Composition	Melt A	Melt B
Ni	20.0 - 20.5	19.90	20.48
Cr	16.0 - 16.5	16.41	15.98
Mn	1.0 - 1.2	1.20	1.22
Si	0.2 - 0.3	0.27	0.28
Nb	8 - 12 x C	0.10	0.09
Mo	< 0.2	< 0.01	< 0.02
C	< 0.02	0.009	0.008
P	< 0.015	0.005	< 0.005
S	< 0.015	0.006	0.004
N	< 0.01	0.009	0.001
Fe	bal	62.1	61.9

Heat treatment - 20 minutes at 1050°C, quenched in water and 5 hours at 900°C, cooled in air.

Density at 20°C - melt A = 8.007 g/cm^3

melt B = 8.011 g/cm^3

4.3 Electrical Resistivity Characterization

Electrical resistivity measurements are used extensively to characterize the variability of metals. For pure metals, the correlation of thermal conductivity and electrical resistivity variability is excellent. The theoretical basis for such a correlation in alloys is less rigorous, but empirically we find if electrical resistivity is nearly invariant, thermal conductivity variation will likely be small. The connection between thermal conductivity and electrical resistivity is further discussed in Appendix I.

The ice-point and liquid helium temperature electrical resistivities of ten specimens from lot 1 were measured and are listed in table 2a. Nine specimens from lot 2 in the as-received condition were measured; the results are listed in table 2b. The specimen identifications given in these tables serve only to indicate that these were distinctly different specimens. Six specimens were similarly measured from lot 2 after hot-swaging and annealing. These results are listed in table 2c.

The effect of various heat treatments was also investigated. This includes time-at-temperature as well as cooling rate effects. These data are listed in table 2d.

The results listed in tables 2a-2d indicate a remarkable insensitivity of electrical resistivity on mechanical and thermal treatments for this steel. The mean values of resistivity for each of these sets of data are the same to within the uncertainty of the measurements (0.5%). The range of each set is similarly within the uncertainty of the measurements. Thus all of the specimens are indistinguishable on the basis of electrical resistivity. Based on the earlier discussion, it is likely that thermal conductivity variations for these specimens are not significantly larger than ± 1%.

Table 2a - Electrical resistivity of austenitic stainless steel (lot 1, as-received condition)

Specimen	ρ(273.15 K) (μΩ cm)	ρ(4 K) (μΩ cm)
1 (1/4" dia.)	78.8	59.4
2 (1/4" dia.)	79.2	59.9
1 (1/2" dia.)	78.6	59.5
2 (1/2" dia.)	78.7	59.6
X (1/4" dia.)	78.6	
X (1/4" dia.)	78.7	59.3
1-1 (1/4" dia.)*	78.4	59.0
1-2 (1/4" dia.)*	78.6	59.0
1-3 (1/4" dia.)*	78.6	59.0
1-4 (1/4" dia.)*	78.6	59.1
Mean	78.7	59.3
Std. dev.	0.2 (0.3%)	0.3 (0.5%)
Range	78.4 to 79.2	59.0 to 59.9

* These specimens were measured by three other laboratories with a range of ±0.5%.

Table 2b - Electrical resistivity of austenitic stainless steel (lot 2, as-received condition)

Specimen	ρ(273.15 K) (μΩ cm)	ρ(4 K) (μΩ cm)
R1/Q1	78.5	59.0
R3/Q1	78.6	59.0
R2/Q1	78.6	58.9
R4/Q1	78.5	59.0
R5/Q1	78.7	59.2
R6/Q1	78.6	59.1
R1/Q4	78.6	59.1
R1/Q2	78.6	59.1
R1/Q3	78.5	59.0
Mean	78.6	59.0
Std. dev.	0.1 (0.1%)	0.1 (0.2%)
Range	78.5 to 78.7	58.9 to 59.2

Table 2c - Electrical resistivity of austenitic stainless steel (lot 2, after hot-swaging and annealing)

Specimen	ρ(273.15 K) (μΩ cm)	ρ(4 K) (μΩ cm)
1-0	79.1	59.2
1-1	78.8	59.0
2-0	78.7	58.8
2-1	78.9	59.0
3-0	78.6	58.9
4-0	78.6	59.1
Mean	78.8	59.0
Std. dev.	0.2 (0.3%)	0.1 (0.2%)
Range	78.6 to 79.1	58.8 to 59.2

Table 2d - Electrical resistivity of austenitic stainless steel (lot 2, heat treatment effects)

Specimen	Heat Treatment	ρ(273.15) (μΩ cm)
1-0	600°C in air for 1 hour	78.1
2-0	600°C in air for 1 hour	78.1
4-2	600°C in air for 2 hours	78.6
1-2	600°C in air for 2 hours	78.6
3-2	600°C in air for 4 hours	78.4
4-3	600°C in air for 4 hours	78.3
2-3	600°C in air for 8 hours	78.6
3-3	600°C in air for 8 hours	78.3
4-4	600°C in air for 16 hours	78.7
1-3	600°C in air for 16 hours	78.7
1-4	800°C in vacuum for 16 hours	78.3
4-4	800°C in vacuum for 16 hours	78.3
1-0	1000°C in vacuum for 2 hours	78.3
2-0	1000°C in vacuum for 2 hours	78.4
1-4	1000°C in air for 1 hour, water quench	78.1
4-4	1000°C in air for 1 hour, water quench	78.2
1-0	1000°C in vacuum for 48 hours	78.5
2-0	1000°C in vacuum for 48 hours	78.4
	Mean =	78.4
	Std. dev. =	0.2 (0.3%)
	Range =	78.1 to 78.7

4.4 Other Characterization Data

Other characterization data which support the thesis that thermal conductivity variability is small are as follows: Hardness*, grain size, and density variations, table 2e, are undetectable within the uncertainties of the measurements. Actual thermal conductivity measurements, described in a later section, on three different specimens from both lots 1 and 2 confirm this conclusion.

This steel was also examined for low-temperature austenite-to-martensite transformation. Such a transformation would be undesirable because of its effect on thermal conductivity and electrical resistivity. Magnetization measurements were performed on specimens, which had been cooled to liquid helium temperature for extended periods, to detect the amount of the ferromagnetic martensitic phase. None was observed to below the 0.1% level.

Table 2e - Hardness, grain size, and density of austenitic stainless steel

	Hardness (Rockwell B)	Grain size (mm)	Density (g/cm^3)
Lot # 1	45 ± 2	0.04 ± 0.02	8.003 ± 0.004
Lot # 2 as received	48 ± 2	0.04 ± 0.02	8.009 ± 0.004
Lot # 2 after hot swaging and annealing	46 ± 2	0.07 ± 0.02	8.006 ± 0.004

The absolute thermopower was determined for three specimens of this austenitic stainless steel at temperatures from 4 to 300 K. Two of the specimens were from lot 1 and one from lot 2. One specimen from lot 1 is designated by WH because it was slightly bent during fabrication and may have been work hardened. These data may serve as additional useful characterization of this material. The conversion of the experimental data, based on normal-silver wire, to the absolute scale, was performed using the data of Borelius et al. [15]. The average thermopower of the three specimens are listed in table 3. The deviations among the three data sets are illustrated in figure 1. These measurements were performed with the multi-property apparatus described in the next section.

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* The hardness of lot 2 after swaging and annealing appears larger than the "as-received" lot 2 and lot 1; however, the uncertainty shown is such that this conclusion is not justified.

Although the thermopower differences among the three specimens, illustrated in figure 1, are within the total uncertainty of the measurements, part of the differences shown are attributed to real specimen differences. This is due to the fact that a major part of the uncertainty in these data is due to systematic error which is essentially the same for all specimens. The estimated specimen-to-specimen component of the uncertainty (imprecision) is about 0.02 μV/K above 80 K. Thus, more than half of the deviation shown in figure 1 is attributed to real specimen differences. This difference is equivalent to about 1% in specimen-to-specimen thermopower variation.

Table 3 - Average low-temperature thermopower of three specimens of austenitic stainless steel

Temp (K)	Thermopower (μV/K)	Temp (K)	Thermopower (μV/K)
6	0.05 (0.05)*	60	-0.44 (0.03)
8	0.09 (0.03)	70	-0.59 (0.03)
10	0.12 (0.02)	80	-0.71 (0.03)
12	0.18 (0.01)	90	-0.81 (0.03)
14	0.23 (0.01)	100	-0.89 (0.04)
16	0.27 (0.01)	120	-1.02 (0.04)
18	0.28 (0.02)	140	-1.12 (0.04)
20	0.28 (0.02)	160	-1.21 (0.04)
25	0.22 (0.02)	180	-1.28 (0.04)
30	0.14 (0.03)	200	-1.36 (0.03)
35	0.04 (0.03)	220	-1.44 (0.03)
40	-0.06 (0.03)	240	-1.51 (0.03)
45	-0.16 (0.03)	260	-1.59 (0.03)
50	-0.27 (0.03)	280	-1.66 (0.03)

* Numbers in ( ) are standard deviations.

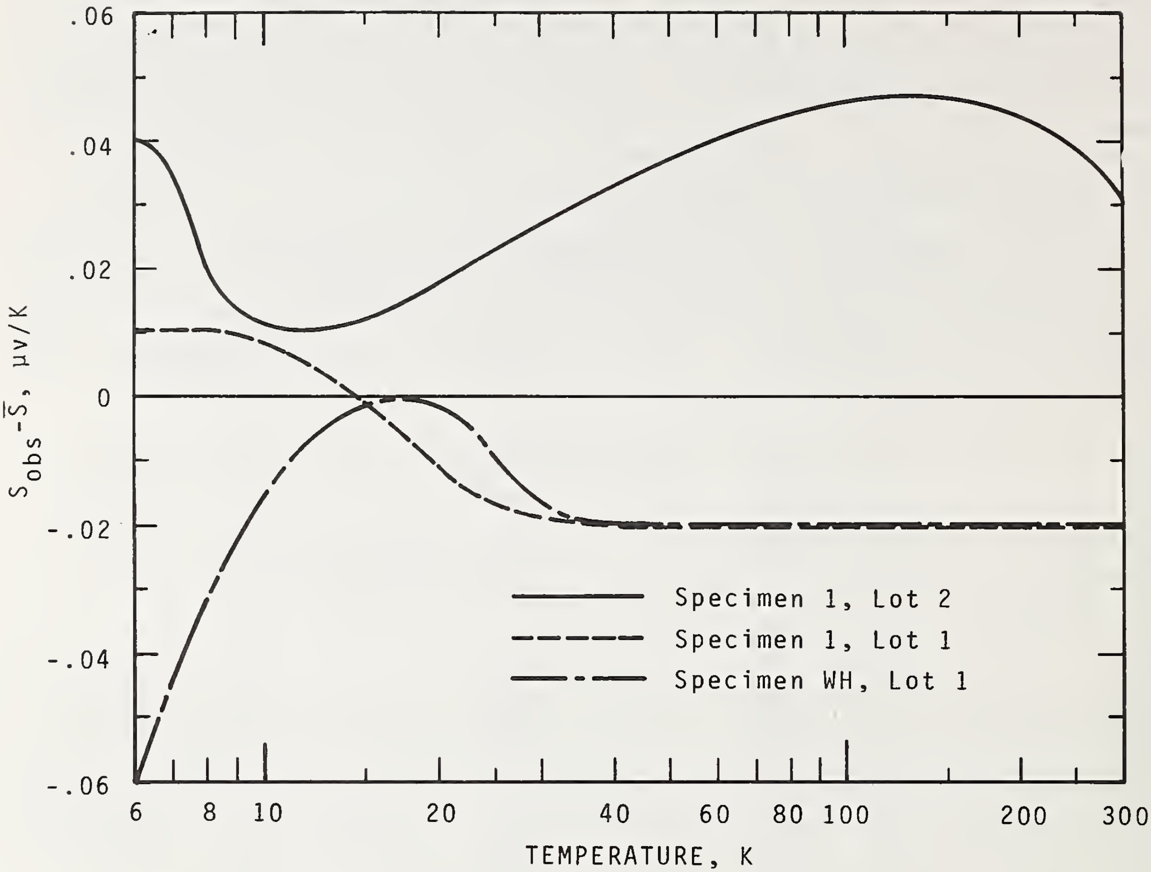

FIGURE 1: Low-temperature thermopower deviations among three specimens of austenitic stainless steel.

5. Apparatus and Measurements

5.1 Low-Temperature (Below Ambient)

Thermal conductivity and electrical resistivity measurements were performed with a multiproperty apparatus based on the axial one-dimensional heat flow (longitudinal) method. This apparatus also yielded the previously presented thermopower characterization data. The specimen is 11.3 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%

Thermopower: 0.2 μV/K at 4 K, decreasing to 0.03 μV/K at 80 K and above.

Three specimens were measured in the low-temperature apparatus, two from lot # 1 and one from lot # 2, over the range 4 to 280 K. The data for each specimen were smoothed using conventional linear least-squares methods with the following equations:

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

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

$$S = \sum_{i=1}^{\ell} c_i\ [\ell n\ T']^{i}/T'\ ;\ T' = \frac{T}{10} + 1$$

where λ = thermal conductivity, ρ = electrical resistivity, and S = absolute thermopower. Temperatures are 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 informal NBS reports [16,17] which may be obtained from the author. No other data sources exist for temperatures below ambient.

5.2 High-Temperature (Above Ambient)

High-temperature thermal and electrical measurements were performed by several participants of the AGARD, NATO project. These data are tabulated by Fitzer [11]. Participants 5 and 7 used a comparative (longitudinal heat flow) method to determine thermal conductivity above 400 K. Participant 34 used an absolute (longitudinal heat flow) method as did participant 7 below 400 K.

Thermal diffusivity values, which can be readily converted to thermal conductivity data, were also measured on the AGARD, NATO program. These data, also reported by Fitzer [11], were obtained as follows: participants 15 and 27 used a modulated electron beam technique, participants 22 and 24 used the angstrom technique, and participants 23, 30, and 5 used a laser flash method. To convert thermal diffusivity to thermal conductivity one needs specific heat and density data. Participant 7 measured specific heat with an adiabatic calorimeter over the range 300 to 1100 K. These are the only specific heat values available for this austenitic steel alloy. Densities were calculated from ambient density and linear thermal expansion data as compiled by Fitzer.

In the next section, we describe the selection of "best values" of thermal conductivity and electrical resistivity. This is done by the method of weighted averaging; where the weight is assigned according to the inverse square of the uncertainty. Thus it is imperative to obtain reasonable estimates of the true uncertainty of each data set. It is difficult, at best, to assess the true uncertainty of one's own data; and even more difficult to assess the validity of anothers' data. First, one must consider the published claims of the authors, second, one can make data and theoretical intercomparisons. In making data intercomparisons (from several sources) one runs the risk of confusing differences caused by apparatus measurement errors and true specimen differences. Based on the previously described characterization data, we believe that specimen-to-specimen property variations caused by material inhomogeneities are not significantly larger than ± 1%. Since the differences in reported experimental thermal conductivity and thermal diffusivity values are appreciably greater than 1%, they are assumed to be caused primarily by measurement errors. Note: above about 1200 K an irreversible transformation has been reported and may be a partial cause of larger reported variations. Our data show no such effects below 1200 K and, thus, no further consideration is given to data above 1200 K. If this transformation does in fact exist, one of the previously described desireable qualities of an SRM is lacking and the use of this material should be limited to below this temperature.

The uncertainty of the NBS thermal conductivity data near ambient is taken as 2.5%, as previously reported by Hust and Sparks [16,17]. The thermal conductivity data reported by the two AGARD participants 5 and 7 agree within 1-2% near ambient temperatures and within 4-5% at temperatures near 900 K. The agreement near ambient appears fortuitus considering the combined minimum uncertainties of their apparatus, the degree of data scatter at only slightly higher temperatures, potential material variability of their ingot iron standards (comparative methods were used above 400 K), and the disagreement of participant 5 from the data of other participants on different materials on the AGARD, NATO project (see Fitzer [11]). In view of this and other data presented by Fitzer [11], the uncertainty for the high temperature thermal conductivity measurements will be estimated as ± 5%.

The reported thermal diffusivity data of seven participants for a total of ten specimens exhibit a scatter of about ± 40% at ambient temperature. If one data source is omitted (participant 5) the range is reduced to ± 10%. At temperatures near 1200 K the range is about ± 15%. The uncertainty in the mean of this entire data set as converted to thermal conductivity values is estimated as ± 5%.

6. Data Analysis (Selection of best values)

No new thermal conductivity or electrical resistivity low-temperature data are reported. Therefore, the electrical resistivity data previously presented by Hust [14] and the thermal conductivity data presented by Hust and Sparks [13] are selected for the low temperature range. At temperatures near ambient, joining of the low-temperature and high-temperature data sets necessitates slight changes in the previously recommended values, however. These changes are consistent with the previously estimated uncertainty for each data set.

At temperatures above ambient, recommended values are based solely on the data obtained from the AGARD, NATO project. Again, joining the high-temperature and low-temperature data sets near ambient will alter this somewhat, but not beyond the uncertainty of the data. Although, other data exist in the literature for similar steels, characterization of these steels is insufficient to warrant intercomparisons at the ± 5% level.

6.1 Electrical Resistivity

Preliminary data analysis indicated that the electrical resistivity data above 200 K varied as a constant power of temperature. Therefore, the parameters of

$$\rho = aT^{b} + c \tag{1}$$

were fitted to the high-temperature data using an iterative non-linear least-squares method. Because of the numerous measurements performed on many specimens at 273.15 K, equation (1) was forced to yield the average ice point value from table 2 (78.6 μΩ cm). The values of the constants obtained are:

$$a = 154.1$$
$$b = 0.0997$$
$$c = -191.0$$

with ρ in μΩ cm and T in Kelvin. The maximum deviations of the experimental data from this equation are near 1%, consistent with the combined material variability and measurement uncertainties. A systematic trend of ± 0.5% occurs in the high temperature deviations but this is believed consistent with systematic measurement uncertainties. The deviations of the data from equation 1 are shown in figure 2. Equation 1 connects with the low-temperature data of Hust [14] at 273 K. Note that below 273 K equation 1 does not have the correct form to represent the low temperature data of Hust [14]. Above 273 K, equation 1 was used to obtain values of electrical resistivity for SRM 798. Table 4 lists these values above 273 K and the existing low-temperature data from Hust [14]. The deviations of the low-temperature data from the recommended values are illustrated in figure 3.

Table 4 - Recommended values of electrical resistivity for SRM 798, austenitic stainless steel

Temp (K)	Electrical Resistivity (μΩ cm)	Temp (K)	Electrical Resistivity (μΩ cm)
5	59.3	250	76.8
10	59.3	300	81.1
15	59.3	350	85.3
20	59.3	400	89.0
25	59.3	450	92.3
30	59.4	500	95.3
40	59.7	600	100.6
50	60.1	700	105.1
60	60.7	800	109.1
70	61.3	900	112.6
80	62.1	1000	115.8
100	63.8	1100	118.8
120	65.6	1200	121.4
140	67.4		
160	69.2		
180	71.0		
200	72.7		

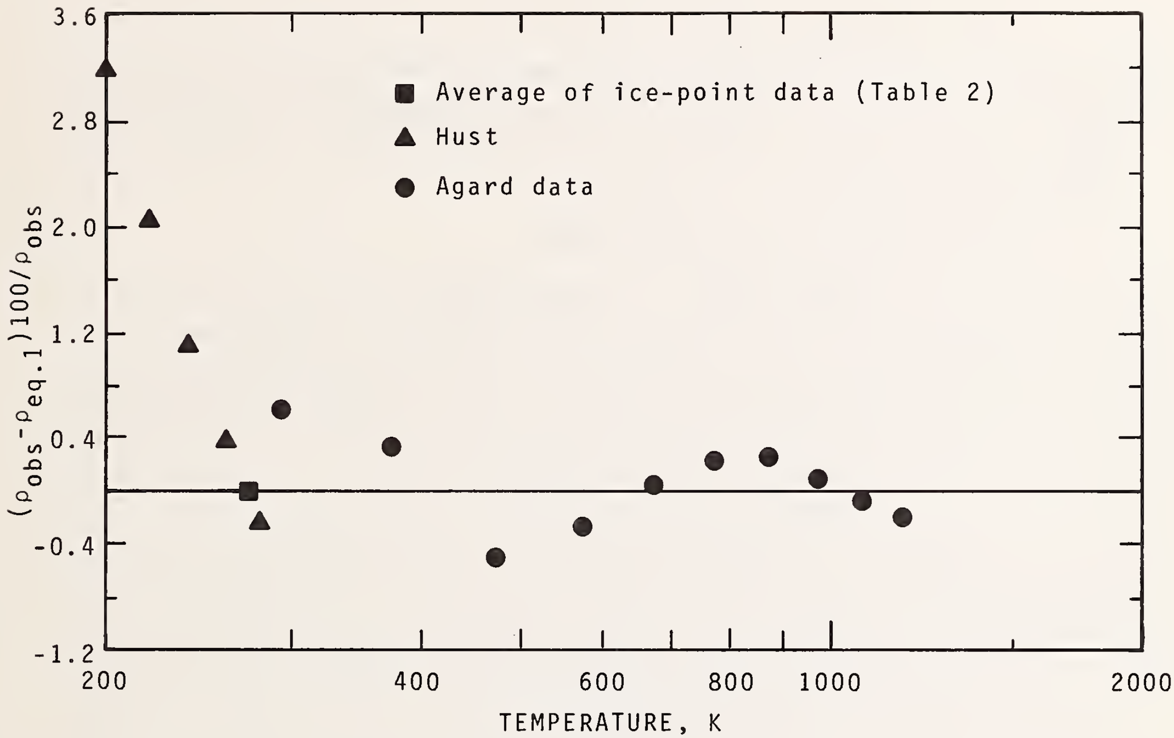

FIGURE 2: Deviations of electrical resistivity data for SRM 798 from equation 1 (parameters fitted to data above 273K)

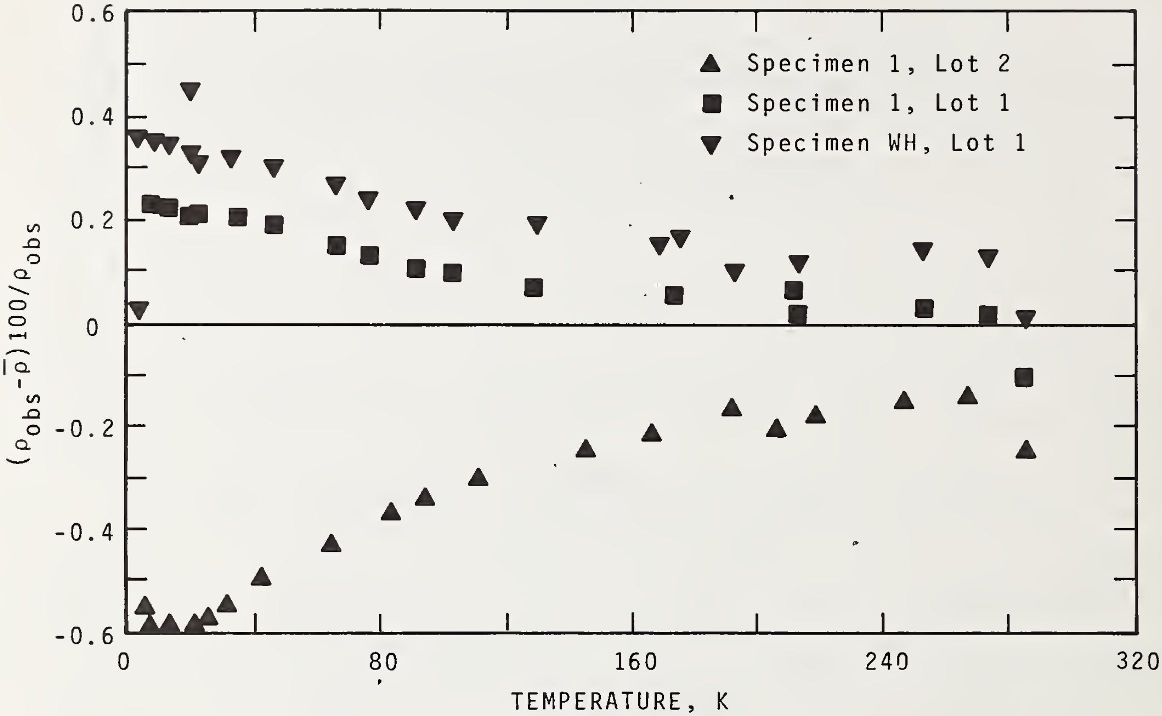

FIGURE 3: Deviations of low-temperature electrical resistivity data for SRM 798 from recommended values.

The uncertainty of the electrical resistivity data for SRM 798, including material variability, is estimated as 1% at low temperatures and 2% at high temperatures.

6.2 Thermal Conductivity

Preliminary analysis of the thermal conductivity data for this austenitic steel indicated that above 200 K the data varied as a constant power of temperature. This prompted us to fit the equation

$$\lambda = aT^{b} \tag{2}$$

by non-linear least-squares methods to the data above 200 K. The data were weighted according to the inverse square of the previously specified uncertainties. The resultant values of the constants are

$$a = 1.22$$
$$b = 0.432,$$

the maximum deviations of the thermal conductivity and converted thermal diffusivity data are 8%, slightly larger, but not unreasonably so, than the estimated uncertainties of these data, 5%. The deviations of the experimental data from those calculated with equation 2 are presented in figure 4. The values calculated from equation 2 agreed with the NBS low-temperature data at 230 K. Thus, the recommended values below 230 K are those given by Hust [13] and above 230 K those obtained from equation 2. These recommended values are tabulated in table 5. The deviations of the NBS low-temperature data from the previously recommended values are shown in figures 5a and 5b. The currently recommended values between 230 K and 280 K differ slightly from the previously recommended values in order to achieve a smooth transition at ambient temperature. The maximum difference is 1% at 280 K, decreasing to zero at 230 K. The estimated uncertainty of the recommended thermal conductivity data, including material variability, is 2% up to 100 K, increasing to 3% at 300 K, and 5% at higher temperatures.

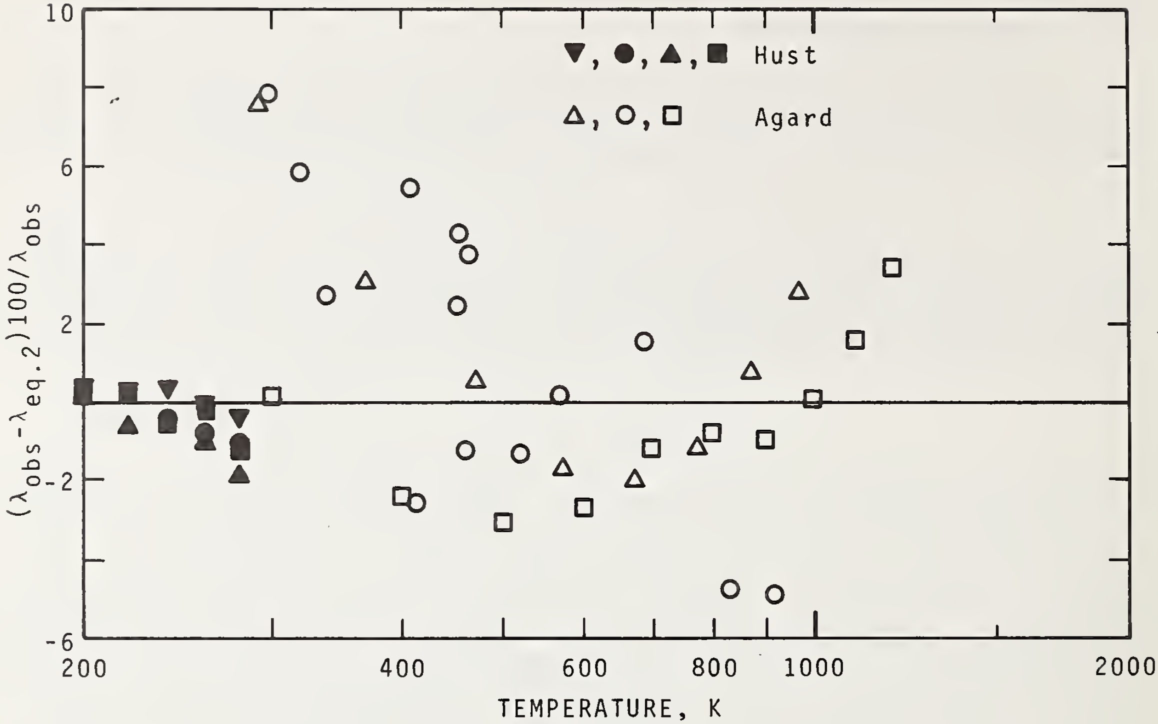

FIGURE 4: Deviations of Thermal Conductivity Data for SRM 735 above 200K from equation 2.

Table 5 - Recommended values of thermal conductivity for SRM 735, austenitic stainless steel

Temp (K)	Thermal Conductivity (W/m K)	Temp (K)	Thermal Conductivity (W/m K)
5	0.466	100	9.25
6	0.565	110	9.65
7	0.676	120	9.99
8	0.796	130	10.3
9	0.921	140	10.6
10	1.05	150	10.9
12	1.32	160	11.1
14	1.58	170	11.4
16	1.86	180	11.6
18	2.13	190	11.9
20	2.40	200	12.1
25	3.07	250	13.2
30	3.72	300	14.3
35	4.34	350	15.3
40	4.92	400	16.2
45	5.47	450	17.1
50	5.98	500	17.9
55	6.45	600	19.3
60	6.88	700	20.6
65	7.28	800	21.9
70	7.64	900	23.0
75	7.97	1000	24.1
80	8.27	1100	25.1
85	8.55	1200	26.1
90	8.80		
95	9.04		

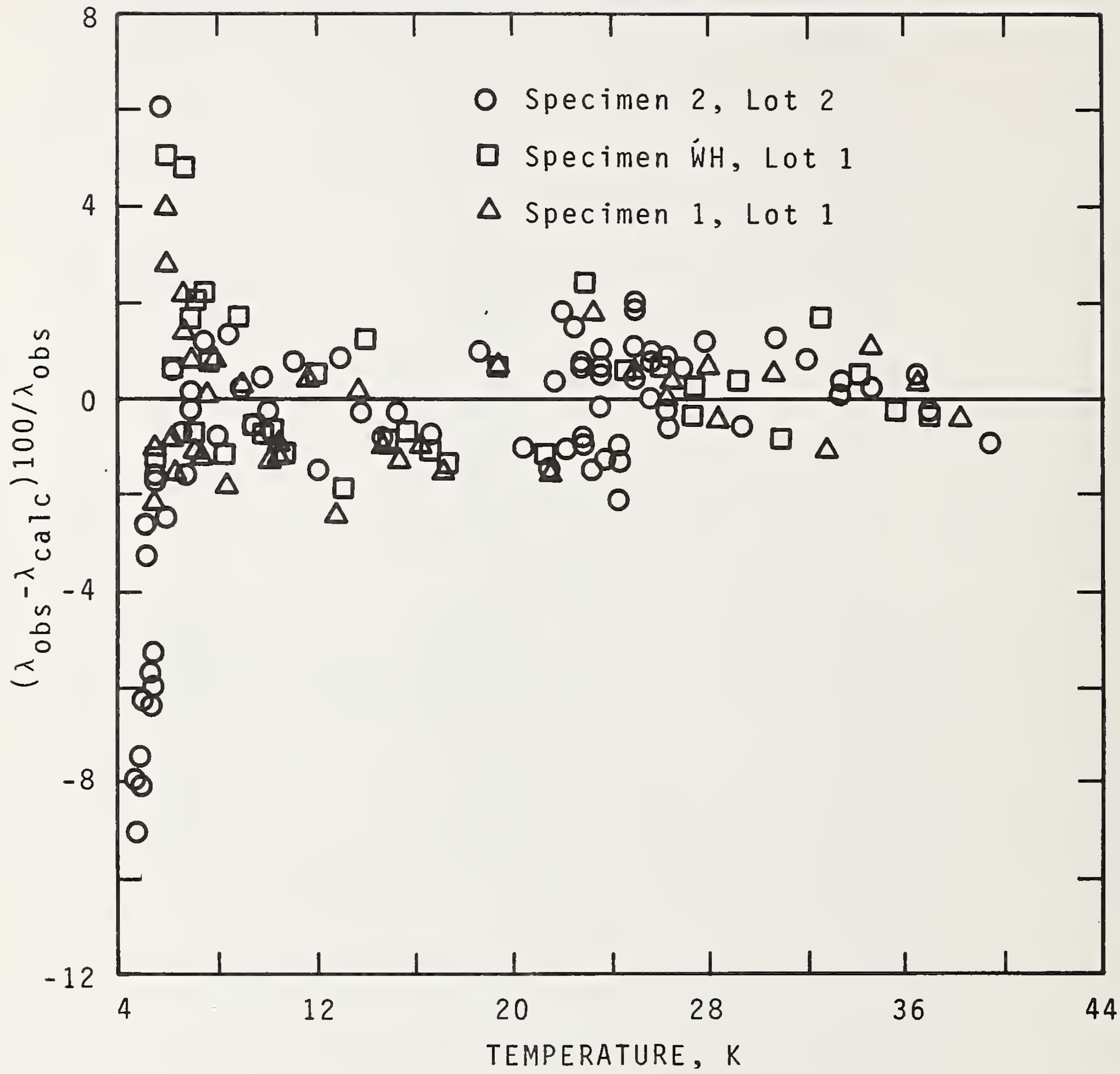

FIGURE 5a: Deviations of Thermal Conductivity Data for SRM 735 from recommended values at temperatures from 5 to 40K.

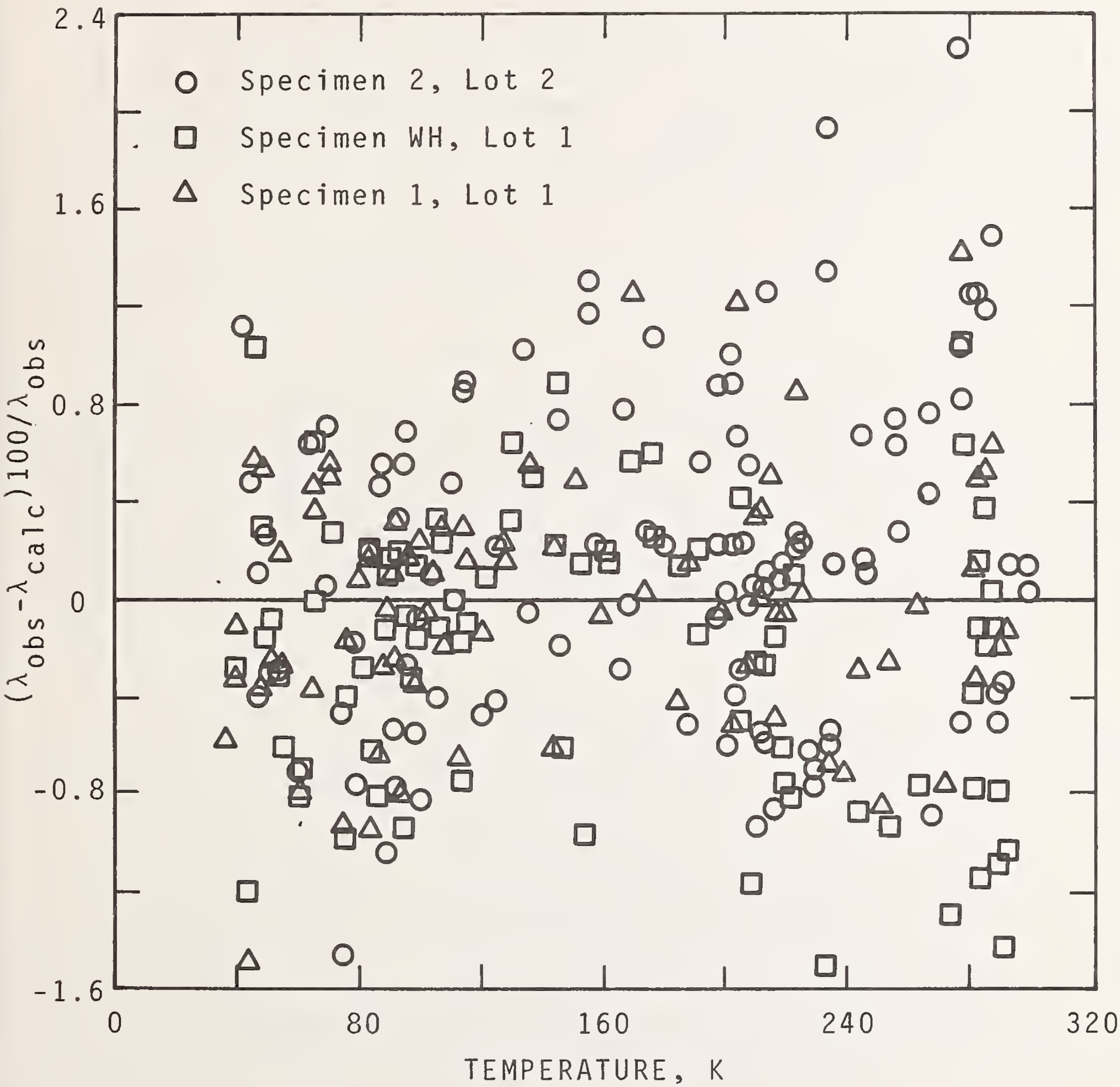

FIGURE 5b: Deviations of Thermal Conductivity Data for SRM 735 from recommended values at temperatures from 40 to 280K.

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 by standards laboratories 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.

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 austenitic steel are not significantly larger than 1%. The results of thermal conductivity, electrical resistivity, and thermopower measurements on three specimens of this steel over the temperature range 5 to 280 K are also presented. These results are in accord with maximum material variability effects of near 1%. The correlation of the deviations of these properties for these three specimens may shed additional light on the true specimen differences. As pointed out earlier, although the measured differences between specimens are in all cases nearly as large as the experimental uncertainties specimen differences somewhat below the measurement uncertainty can be detected. This is so because the imprecision of the measurements is about one-half the total uncertainty.

It is observed from figures 1, 3, and 5 that the specimen from lot 2 has a slightly more positive (smaller in absolute magnitude) thermopower, lower electrical resistivity, and an almost imperceptibly larger thermal conductivity than the other two specimens. In the absence of concomitant opposing effects these changes are consistent with the indicated larger grain size of the specimen from lot 2. A larger grain size results in less grain boundary scattering increasing the electrical and thermal conductivity. It is more difficult to predict the effect on thermopower, but, recent unpublished work on thermocouple materials [18] suggests that the absolute value of thermopower decreases with increasing grain size. This is in agreement with the observed change.

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. This is especially true at temperatures above ambient where the estimated uncertainty is ± 5% in thermal conductivity. 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 first author.

8. Summary

The electrical resistivity and thermal conductivity data has been compiled and analyzed for an austenitic stainless steel. Recommended values are tabulated for temperatures from 5 to 1200 K and form the basis of SRM's 798 (electrical resistivity) and 735 (thermal conductivity). Electrical resistivity values are estimated uncertain by 1% below ambient and 2% above ambient. Thermal conductivity uncertainties are 2% below 100 K, increasing to 3% at ambient temperature, and 5% above ambient. Material variability effects, included in the above uncertainties, are estimated to be near 1%.

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 735-S	0.64 cm diameter, 30 cm long
SRM 735-M1	1.25 cm diameter, 15 cm long
SRM 735-M2	1.25 cm diameter, 30 cm long
SRM 735-L1	3.4 cm diameter, 5 cm long
SRM 735-L2	3.4 cm diameter, 10 cm long
SRM 798-1	0.64 cm diameter, 5 cm long
SRM 798-2	0.64 cm diameter, 10 cm long
SRM 798-3	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 multiproperty apparatus. E. Fitzer was very cooperative in the procurement of the stock of material from the German manufacturer. He also supplied the AGARD program results and the original specimens for measurement. We wish to thank the personnel of the U.S. Bureau of Mines, Albany, Oregon, especially Steve O'Hare, for fabricating the final rod sizes for these SRM's. Last, but not least, we wish to thank R. E. Michaelis of NBS, OSRM for supporting this work and many helpful discussions.

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Appendix I. Discussion of electrical resistivity and thermal conductivity variability

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 complex alloys such as steel, equation (2) does not hold, but it has been observed that the value of the Lorenz function, $\rho\lambda/T$, is reasonably independent of material within a given class of alloys such as austenitic stainless steel (Fe-18Cr-8Ni alloys). This indicates a close but unexplained relationship between ρ and λ even though λ_e is small compared to λ_g.

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. For steels, the residual resistivity is a significant part of the total resistivity, even at room temperature; thus, values of either room temperature resistivity or residual resistivity can be used as indicators of material variability. This differs from pure metals in that ρ_o is much smaller than ρ(293 K) for pure metals; therefore, ρ(293 K) is not an indicator of purity. The variability in resistivity for various specimens in a given lot of material is an indication of the variability in chemical composition and physical imperfection concentration in the lot. These material variations also cause thermal conductivity variations as indicated by the Lorenz function for a given class of alloys. Therefore, a determination of resistivity variability will usually approximate the thermal conductivity variability in alloys. The determination of electrical resistivity is considerably easier than the determination of λ.

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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.)*

A historical review of the development of selected thermophysical Standard Reference Materials, SRM's, is given and selection criteria for those SRM's are listed. Thermal conductivity and electrical resistivity data for austenitic stainless steel, SRM's 735 and 798, are critically evaluated. Recommended values are presented for the temperature range 4 to 1200 K. Material variability studies, including the effects of heat treatment, have been performed at low temperatures. No irreversible transformations are observed up to temperatures of 1200 K. Based on the results of several types of characterization measurements, effects of material variability are believed to be less than 1% in electrical resistivity and not significantly more in thermal conductivity. The uncertainty of the recommended electrical resistivity data is estimated at 1% at low temperatures and 2% at higher temperatures. The corresponding uncertainty for thermal conductivity is 2% below 100 K, increasing to 3% at 300 K, and 5% at higher temperatures.

17. KEY WORDS *(six to twelve entries; alphabetical order; capitalize only the first letter of the first key word unless a proper name; separated by semicolons)*

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