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Standard Reference Materials:

ELECTRICAL RESISTIVITY OF ELECTROLYTIC IRON, SRM 797, AND AUSTENITIC STAINLESS STEEL, SRM 798, FROM 5 TO 280 K

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ELECTRICAL RESISTIVITY OF ELECTROLYTIC IRON, SRM 797, AND AUSTENITIC STAINLESS STEEL, SRM 798, FROM 5 TO 280 K

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Electrical Resistivity of Electrolytic Iron, SRM 797, and Austenitic Stainless Steel, SRM 798, from 5 to 280 K*

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Electrical resistivity data are presented for characterized electrolytic iron, SRM 797, and austenitic stainless steel, SRM 798, at temperatures from 5 to 280 K. Resistivities at ice and liquid helium temperatures were determined for 22 randomly selected iron specimens and the same number of steel specimens. These data indicate that the effect of material variability is about 1% for each of these SRM's.

Key words: Austenitic stainless steel; cryogenics; electrical resistivity; electrolytic iron; Lorenz ratio; standard reference material.

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1. Introduction

The research reported here is part of a larger program to establish several thermal conductivity and electrical resistivity standard reference materials. Several materials are needed to cover wide ranges of conductivity and resistivity. In earlier publications[2,3] of this series, thermal conductivity data were presented for SRM 734 and 735. It has been decided that low temperature electrical resistivity data on these materials would be useful for two reasons. The characterization of the thermal conductivity specimens is improved with electrical resistivity data. Standards of electrical resistivity are useful for calibration and comparisons of the many existing apparatus. Although electrical resistivity measurements are generally considered routine, unsuspected systematic errors may be detected readily through the use of these Standard Reference Materials, SRM's.

The basic characteristics of an SRM are that it be: (a) stable and reproducible under the conditions of use, (b) uniform throughout a single specimen, (c) similar in property value to that of the materials to which it will be applied, (d) readily machined and fabricated to appropriate size and shape, (e) chemically inert with its environment, and (f) useable over a wide temperature range. The iron and steel reference materials satisfy these criteria reasonably well.

2. Apparatus and Data Analysis

A detailed description of this apparatus and the methods of data analysis have been given by Hust, et al.[4] Briefly, the measurement is done by a standard 4 terminal potentiometric method on a 23 cm long cylindrical rod. The diameter of the rod is dependent upon its conductivity with a maximum diameter of about 2.5 cm. The electrical resistivity data were taken simultaneously with the thermal conductivity data on the same specimen with a single apparatus. Data were taken in the presence of temperature gradients along the specimen but the data analysis was performed so as to include the effect of these gradients.

The experimental data are represented by arbitrary functions over the entire temperature 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.

3. Specimen Characterization

Details of specimen characterization have been given by Hust and Sparks[2,3] and will not be repeated here. The results of these measurements showed a material resistivity variability of about 1% for both SRM 797 and 798. Specimens of these SRM's should therefore be used only for standardization to 1%. (See Appendix I for variability data and proper heat treatment of these SRM's.)

4. Results

The electrical resistivity of one specimen of SRM 797 was measured from 6 to 280 K. Three specimens of SRM 798 were measured from 5 to 280 K. These data were functionally represented with the following equation:

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

ρ is electrical resistivity in ohm meters and temperatures, T, are in kelvin based on the IPTS-68 scale above 20 K and NBS P2-20 (1965) scale below 20 K. The parameters, b_i determined by least squares, are presented in table 1. Further details of the fitting procedure are described by Hust, et al.[4] The deviations of the experimental data from this equation are given in figures 1 and 2. Values of ρ, calculated from equation 1, are presented in table 2 and figures 3 and 4.

A detailed error analysis for this system has been presented previously by Hust, et al.[4] Based on this analysis of systematic and random errors, the measurement uncertainty estimate (with 95% confidence) is 0.25% of the resistivity.

Table 1. Parameters, b_i, of equation (1)

i	b_i SRM 797	b_i SRM 798
1	$1.52995843 \times 10^{-5}$	$8.51517313 \times 10^{-7}$
2	$-6.57221842 \times 10^{-5}$	$-8.57069352 \times 10^{-7}$
3	$1.28083500 \times 10^{-4}$	$1.18184172 \times 10^{-6}$
4	$-1.50027718 \times 10^{-4}$	$-8.87557838 \times 10^{-7}$
5	$1.17942860 \times 10^{-4}$	$3.97914551 \times 10^{-7}$
6	$-6.57740194 \times 10^{-5}$	$-1.09135708 \times 10^{-7}$
7	$2.67952461 \times 10^{-5}$	$1.78713688 \times 10^{-8}$
8	$-8.08115141 \times 10^{-6}$	$-1.59617291 \times 10^{-9}$
9	$1.80581011 \times 10^{-6}$	$5.97187252 \times 10^{-11}$
10	$-2.95519976 \times 10^{-7}$	
11	$3.44469418 \times 10^{-8}$	
12	$-2.70952664 \times 10^{-9}$	
13	$1.28939040 \times 10^{-10}$	
14	$-2.80388287 \times 10^{-12}$	

Table 2. Calculated values of electrical resistivity for SRM's 797 and 798

Temperature (K)	Resistivity (n Ω m) SRM 797	SRM 798	Temperature (K)	Resistivity (n Ω m) SRM 797	SRM 798
5	--	593	75	9.38	617
6	3.87	593	80	10.56	621
7	3.87	593	85	11.88	625
8	3.85	593	90	13.27	629
9	3.85	593	95	14.76	634
10	3.85	593	100	16.32	638
12	3.87	593	110	19.69	647
14	3.89	593	120	23.30	656
16	3.90	593	130	27.07	665
18	3.90	593	140	31.0	674
20	3.92	593	150	35.0	683
25	3.99	593	160	39.1	692
30	4.10	594	170	43.2	701
35	4.26	595	180	47.5	710
40	4.50	597	190	51.8	718
45	4.84	598	200	56.1	727
50	5.28	601			
55	5.85	604	220	65.2	743
60	6.54	607	240	74.4	760
65	7.37	610	260	84.2	776
70	8.32	613	280	94.3	791

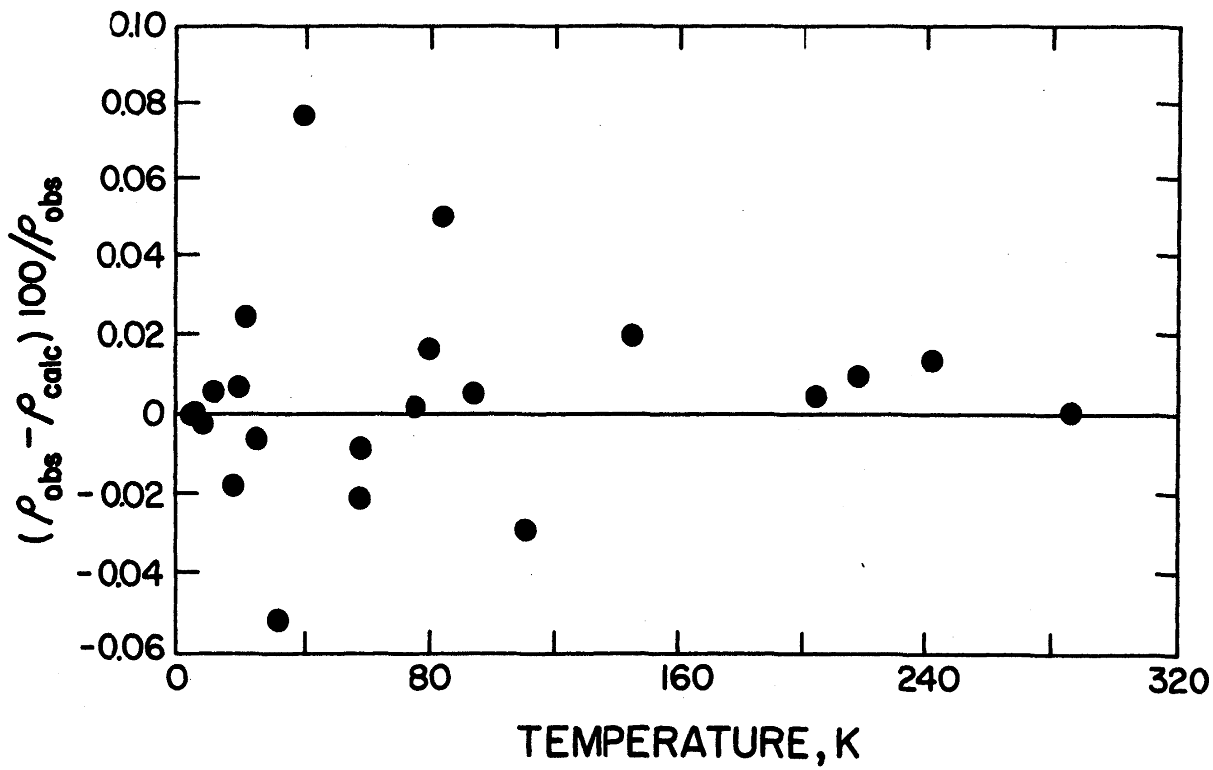

Figure 1. Experimental electrical resistivity deviations from calculated values (equation 1) for SRM 797.

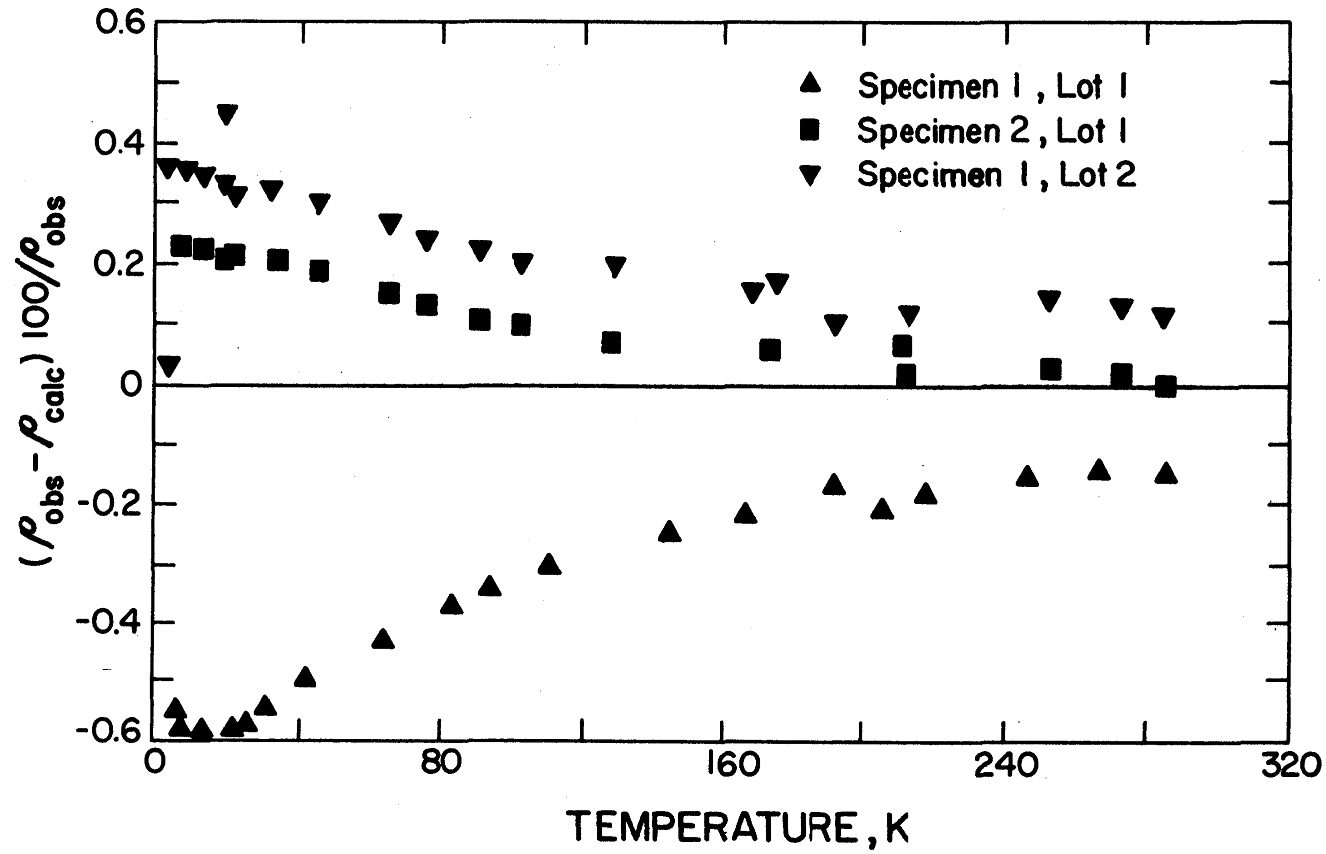

Figure 2. Experimental electrical resistivity deviations from mean values of resistivity for three specimens of SRM 798

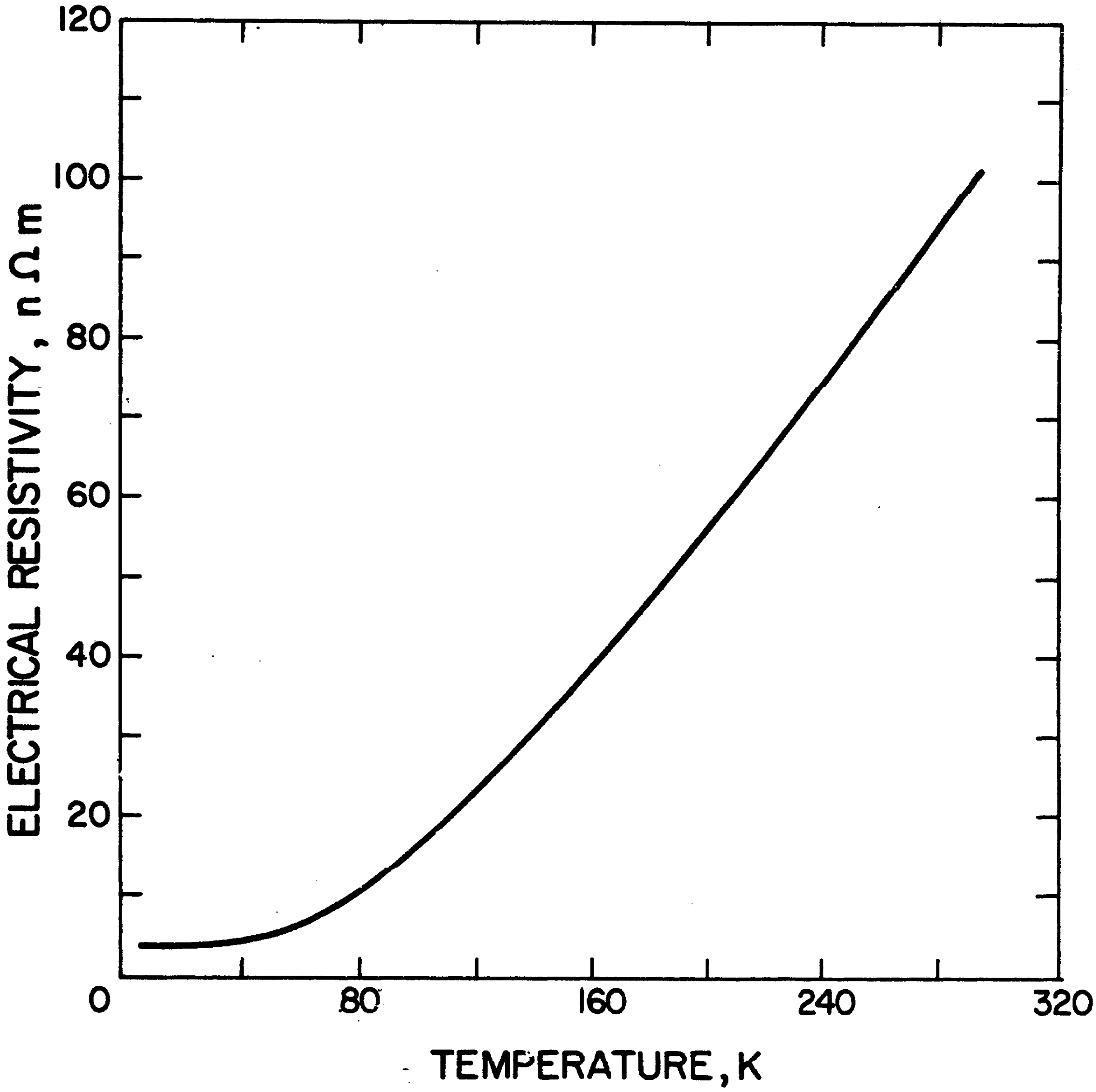

Figure 3. Calculated values of electrical resistivity for SRM 797

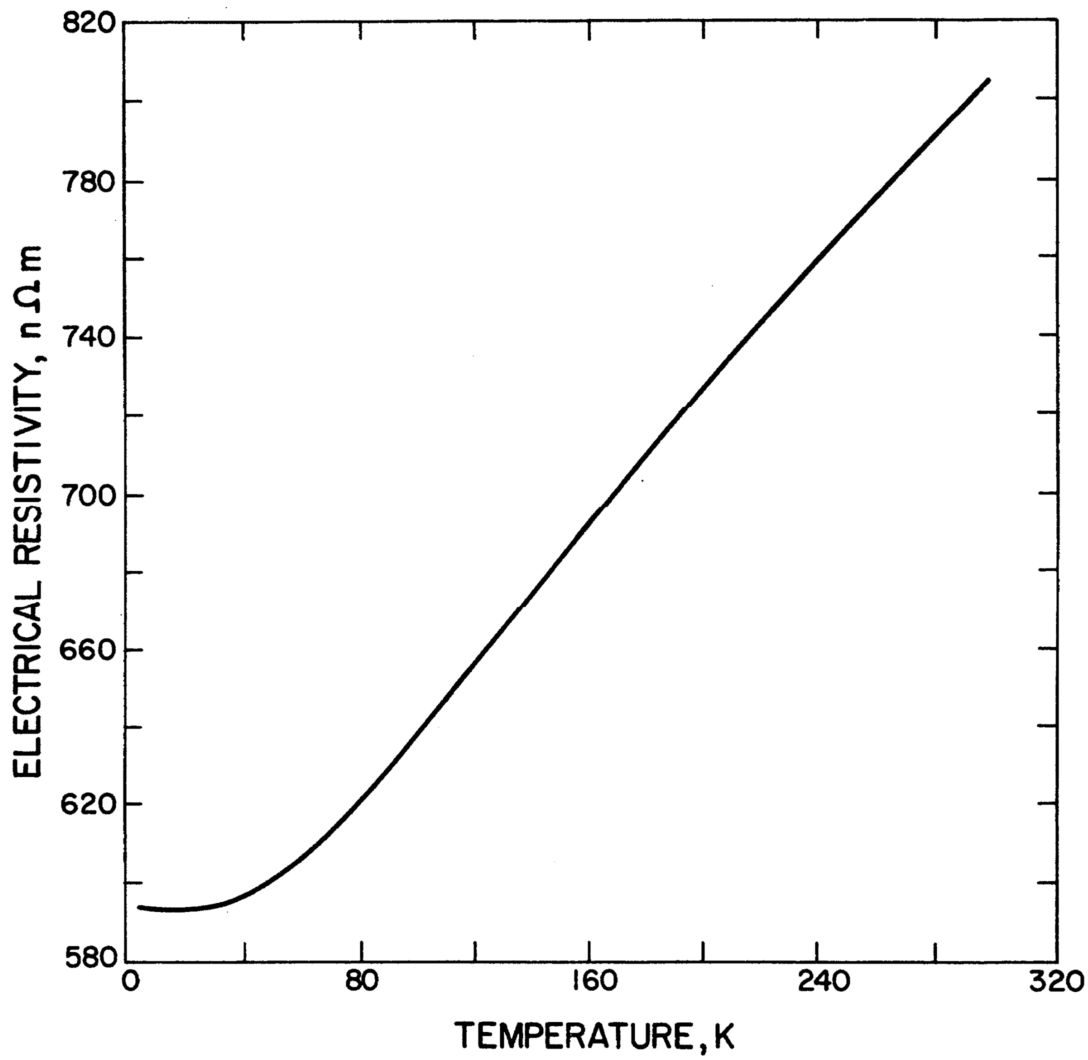

Figure 4. Calculated mean values of electrical resistivity for SRM 798

5. Summary

We have established low temperature electrical resistivity standard reference data for electrolytic iron, SRM 797, and austenitic stainless steel, SRM 798, at temperatures from 5 to 280 K. These data were fitted to an empirical equation which was used to generate tabular values of resistivity at integer temperatures. Material variability affects electrical resistivity by about ± 1% and measurement uncertainty is estimated to be about 0.25%.

6. Footnotes and References

[1] Both SRM's 797 and 798 are available in the form of rods of three different lengths. SRM's 797-1 and 798-1 are rods 5 cm (2 in.) long, and 0.64 cm (1/4 in.) diameter; SRM's 797-2 and 798-2 are rods 10 cm (4 in.) long; and SRM's 797-3 and 798-3 are rods 15 cm (6 in.) long. Longer continuous lengths can be obtained by special order. These SRM's may be ordered from the Office of Standard Reference Materials, National Bureau of Standards, Washington, DC 20234.

[2] Hust, J. G. and Sparks, L. L., Thermal Conductivity of Electrolytic Iron, SRM 734, from 4 to 300 K, Nat. Bur. of Stand. Spec. Pub. 260-31, 19 pp. (Nov. 1971).

[3] Hust, J. G. and Sparks, L. L., Thermal Conductivity of Austenitic Stainless Steel, SRM 735, from 5 to 280 K, Nat. Bur. of Stand. Spec. Publ. 260-35, 15 pp. (Apr. 1972).

[4] Hust, J. G., Powell, R. 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.) No 5, 673-690 (1970).

Appendix I

Electrical Resistivity Variability

An extensive resistivity variability study was conducted on SRM-797 the object being to determine if it could be heat treated in such manner that the electrical resistivity would be the same for each specimen. This was achieved with a 2-hour, 1000°C anneal in either a vacuum or helium atmosphere. The results of this study are shown as residual resistivity ratios below. The ratio given is resistivity at 273.15K to resistivity at 4K. Specimens labled C2T, A6L, C5L, A1L, and A5T were obtained from 1/4" diameter rods; the remaining specimens were machined from 1-1/4" rods. Based on the 63 residual residual resistivity ratio measurements made on these specimens in various stages of heat treatment, the following is concluded: The large 1-1/4" diameter specimens are significantly different in residual resistivity ratio from the smaller 1/4" diameter specimens in the as received condition. The ratio of the small rods is 22.01 ± 0.20 while the ratio of the larger rods is 19.52 ± 0.44.

Various heat treatments were tried to remove the differences in ratio of the two sets of rods. After 500°C for 1 hour the ratios increased but were still different (small rods = 23.53 ± 0.20; large rods = 22.14 ± 0.34). Raising the temperature to 1000°C for 2 hours produced rods which are indistinguishable, (small rods = 23.39 ± 0.28; large rods = 23.39 ± 0.20; all rods = 23.33 ± 0.24). The variation shown is 2s, where s is the estimated standard deviation, and includes material and measurement variability. In order to study the possibility of a change in these ratios with age, some of the rods were measured after about 50 days and again after 3 years; no significant change was detected after 50 days but in 3 years a 4% increase in RRR occurred. Heating to 400°C for 2-1/2 days changed the ratio to 24.94 ± 0.26 with no difference between the large and small rods when the first measurements were made during 1970. However, in 1973 a similar heat treatment produced a much smaller change. This is not understood.

After performing further anneals to obtain a better understanding of the aging phenomena, it appears clear that our earlier selected anneal procedure was proper in that we obtained the RRR value corresponding to the stabilizing condition of this iron. However, we were not aware at the time of the importance of the cooling rate of the furnace. At that time we used a rather massive furnace which cooled rather slowly. With the smaller furnace used in these later measurements, a hold of at least two hours at 800°C is necessary to stabilize this iron. After this heat treatment, heating the specimens to intermediate temperatures does not significantly affect the residual resistivity ratio. These measurements show that SRM 797 can be used as an electrical resistivity standard below room temperature with a variability of about 1% if annealed first at 1000°C for 2 hours and then 800°C for 2 hours.

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Residual resistivity ratio (ρ_{273K}/ρ_{4K}) of SRM-734

Specimen	Ratio: As received	500°C 1 hr	500°C 8 hr	1000°C 2 hr	400°C 2½ days	1970 Aging 50 days	1973 Aging 3 yrs	1000°C 60 hr	800°C 46 hr	500°C 60 hr	1000°C 2 hr	800°C ½ hr (vac.)	400°C 20 hrs	800°C 4 hr	400°C 46 hrs	800°C 2¾ hrs	1000°C, 2 hr 800°C, 2 hr
C2T	21.97 (a)	23.53	24.12	23.31 (e)	24.84	25.00	25.57	21.88	23.46	23.64	22.45	---	23.65	---	23.88	---	---
A6L	22.16	---	---	23.22 (f)	24.85	24.97	25.85	---	---	---	---	23.59	---	23.40	---	---	---
C5L	21.94	---	---	23.40 (f)	---	23.47	24.46	22.19	23.615	23.81	22.61	---	23.80	---	23.94	---	---
A1L	22.04	---	---	23.59 (c)	---	23.52	---	---	---	---	---	---	---	---	---	---	---
A5T	22.03 (b)	---	---	23.42 (f)	---	23.42	24.90	---	---	---	---	23.64	---	23.58	---	---	---
2A-1-1	19.35	21.96	22.32	23.47 (e)	25.12	25.24	25.69	22.20	23.45	23.76	22.54	---	23.72	---	24.07	---	---
2A-1-2	19.50	---	---	23.31 (c)	---	23.35	---	---	---	---	---	---	---	---	---	---	---
2A-1-3	19.30	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
2A-1-4	19.38	---	---	23.27 (f)	---	---	---	---	---	---	---	---	---	---	---	---	---
2A-3-1	19.77	21.83	22.25	23.20 (e)	24.94	25.01	---	---	---	---	---	---	---	---	---	---	---
2A-3-2	19.92	---	---	23.20 (c)	---	23.25 (d)	---	---	---	---	---	---	---	---	---	---	---
2A-3-3	19.73	---	---	---	---	---	20.29	---	---	---	---	24.52	---	24.48	---	25.12	23.24
2A-3-4	19.93	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
2C-1-1	19.42	21.70	22.00	23.23 (e)	---	23.44	24.48	72.55	23.51	23.63	22.51	---	23.60	---	23.94	---	---
2C-1-2	19.12	---	---	23.41 (c)	---	23.40	---	---	---	---	---	---	---	---	---	---	---
2C-1-3	19.46	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
2C-1-4	19.56	---	---	---	---	---	---	---	---	---	---	---	---	---	---	24.75	23.81
2C-3-1	19.34	21.93	21.99	23.27 (e)	---	23.40	24.86	---	---	---	---	23.40	---	23.64	---	---	---
2C-3-2	19.49	---	---	---	---	---	---	---	---	---	---	---	---	---	---	25.16	23.50
2C-3-3	19.57	---	---	---	---	---	---	---	---	---	---	---	---	---	---	24.84	23.56
2C-3-4	19.40	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Average =	20.11	22.19	22.54	23.33	24.94	23.96	24.51	22.20	23.51	23.71	22.53	23.79	23.69	23.78	23.96	24.97	23.53

(a) repeat measurement = 21.91

(b) ratio of A5T thermal conductivity specimen = 21.89

(c) these were heat treated in vacuum, the remaining were heated to 1000°C in a helium atmosphere (1 atm pressure).

(d) repeat measurements = 23.39, 23.31

(e, f) these were done in separate heat treatments to detect reproducibility of heat treatment.