The electrical resistivity and thermal expansion of iridium at high temperatures
by James Joseph Halvorson

A thesis submitted to the Graduate Faculty in partial fulfillment of the requirements for the degree of
MASTER OF SCIENCE in Aerospace and Mechanical Engineering
Montana State University

Abstract:
The electrical resistivity and thermal expansion of iridium were measured in the range of 293-2275°K and 891—2221°C respectively.

The electrical resistivity of iridium was obtained by measuring the dc-voltage across the specimen and the voltage drop across a precision resistor which was in series with the wire specimen. The ratio of voltages corresponded to a resistance ratio from which the electrical resistivity was calculated knowing the length and diameter of the specimen. The following equation represents the results: ρt (293-2275 K) = 19.702 x 10^-3t - 2.586 x 10^-6 t^2 + 4.64 x 10^-9 t^3 - 1.08 x 10^-15 t^4 for the electrical resistivity, ρt, expressed in microhm-cm and the temperature t expressed in °K. The results of the present study were compared with earlier studies, and an attempt was made to correlate the experimental data to theory presented by Gruneisen. Mean temperature coefficients of resistivity were calculated.

The linear thermal expansion of iridium was measured in the range of 891-2221°C by taking the experimental data directly by visual means. The following equation was found to fit selected earlier results and the present results oyer the temperature range of 30-2221°C: 100 (Lt-L0)/L0 = 616.7 x 10^_6 t + 151.9 x 10^-9 t^2 - 28.16 x 10^-12 t^3 + 14.63 x 10^-15 t^4 for t expressed in °C. Thermal expansion coefficients and average coefficients of expansion were calculated.

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Date 12/3/71

THE ELECTRICAL RESISTIVITY AND THERMAL EXPANSION OF IRIDIUM AT HIGH TEMPERATURES

by

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A thesis submitted to the Graduate Faculty in partial
fulfillment of the requirements for the degree

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TABLE OF CONTENTS

PART I

THE ELECTRICAL RESISTIVITY OF IRIDIUM AT HIGH TEMPERATURES

PART II

THE THERMAL EXPANSION OF IRIDIUM AT HIGH TEMPERATURES

LIST OF TABLES

Table Page

PART I

THE ELECTRICAL RESISTIVITY OF IRIDIUM AT HIGH TEMPERATURES

PART II

THE THERMAL EXPANSION OF IRIDIUM AT HIGH TEMPERATURES

LIST OF FIGURES

Figure Page

PART I

THE ELECTRICAL RESISTIVITY OF IRIDIUM AT HIGH TEMPERATURES

PART II

THE THERMAL EXPANSION OF IRIDIUM AT HIGH TEMPERATURES

ABSTRACT

The electrical resistivity and thermal expansion of iridium were measured in the range of 293-2275°K and 891-2221°C respectively.

The electrical resistivity of iridium was obtained by measuring the dc-voltage across the specimen and the voltage drop across a precision resistor which was in series with the wire specimen. The ratio of voltages corresponded to a resistance ratio from which the electrical resistivity was calculated knowing the length and diameter of the specimen. The following equation represents the results:

$$\rho_t \ (293\text{-}2275°\text{K}) = 19.702 \times 10^{-3} t - 2.586 \times 10^{-6} t^2 + 4.64 \times 10^{-9} t^3 - 1.08 \times 10^{-15} t^4$$

for the electrical resistivity, ρ_t, expressed in microhm-cm and the temperature $\underline{t}$ expressed in °K. The results of the present study were compared with earlier studies, and an attempt was made to correlate the experimental data to theory presented by Grüneisen. Mean temperature coefficients of resistivity were calculated.

The linear thermal expansion of iridium was measured in the range of 891-2221°C by taking the experimental data directly by visual means. The following equation was found to fit selected earlier results and the present results over the temperature range of 30-2221°C:

$$100 \ \frac{L_t - L_o}{L_o} = 616.7 \times 10^{-6} t + 151.9 \times 10^{-9} t^2 - 28.16 \times 10^{-12} t^3 + 14.63 \times 10^{-15} t^4$$

for $\underline{t}$ expressed in °C. Thermal expansion coefficients and average coefficients of expansion were calculated.

PART I

THE ELECTRICAL RESISTIVITY OF IRIDIUM AT HIGH TEMPERATURES

Introduction

The electrical resistivity of iridium was studied by Powell, Tye, and Woodman[1] in the range of 100-500°K. Other available electrical resistivity data generally refer to temperatures below 500°K. The values given in the "Thermophysical Properties of High Temperature Solid Materials"[2] in graphical form in the range of 75-1593°K are discussed later. (Values were obtained from the work of Powell, *et al.*[3]) A need for additional resistivity data for iridium at higher temperatures prompted making the resistivity measurements presented in the present paper.

As the experimental data reported in the present paper were being obtained, results of a similar study made by Russian workers appeared in the technical literature. The electrical resistivity of iridium was studied by L'voy, Mal'ko and Nemchenko[4] in the range of 80-1900°K. Iridium samples were prepared by a powder metallurgy method (pressed billets were sintered in vacuum at a temperature of about 2500°K). Electrical resistivity data were obtained by the potentiometric method and are compared with the results of the present study.

Specimen Analysis

Commercially pure iridium wire, 0.635 mm (25 mils) in diameter was purchased from Engelhard Industries, Carteret, New Jersey. Results of a spectrographic analysis* of the wire are contained in Table I along with the results of a vacuum fusion analysis** for oxygen, hydrogen, and nitrogen. The total measured impurity content of the metal was seen to be 0.028%. Although analysis did not include all known elements, the iridium content was estimated to exceed 99.95%.

Experimental Method

The experimental equipment used in the present study was designed such that the electrical resistivity was obtained by measuring the dc-voltage drop across the specimen and the voltage drop across a precision resistor which was in series with the wire specimen. The ratio of voltages corresponded to a resistance ratio from which the electrical resistivity was calculated knowing the length and diameter of the specimen. Figure 1 is a schematic representation of the experimental equipment. A ceramic plug which contained the iridium wire specimen was made from a castable ZrO_2 cement. The wire specimen was electrically isolated from the ZrO_2 plug by passing the wire through ThO_2 thermocouple tubes, ThO_2 being used because of its high electrical resistivity at high temperatures. The section of the iridium wire specimen for which the resistivity was measured was of uniform temperature

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*Performed at the Matthey Bishop Co., Malvern, Pennsylvania

**Performed at the Magnflux Testing Laboratories, Los Angeles, California

TABLE I ANALYSIS OF THE IRIDIUM WIRE

(Amounts of Impurity Elements Expressed in PPM)

Pt	15	Sb	<10	Fe	87	Sn	< 1
Pd	5	As	<10	Pb	< 1	Ti	6
Rh	54	Bi	< 1	Mg	5	Zn	<10
Ru	25	B	5	Mn	1	Ca	7
Os	< 3	Cd	< 1	Mo	1	O	30
Au	3	Cr	< 1	Ni	3	H	1
Ag	< 1	Co	< 1	Si	7	N	15
Al	9	Cu	2	Te	<10		

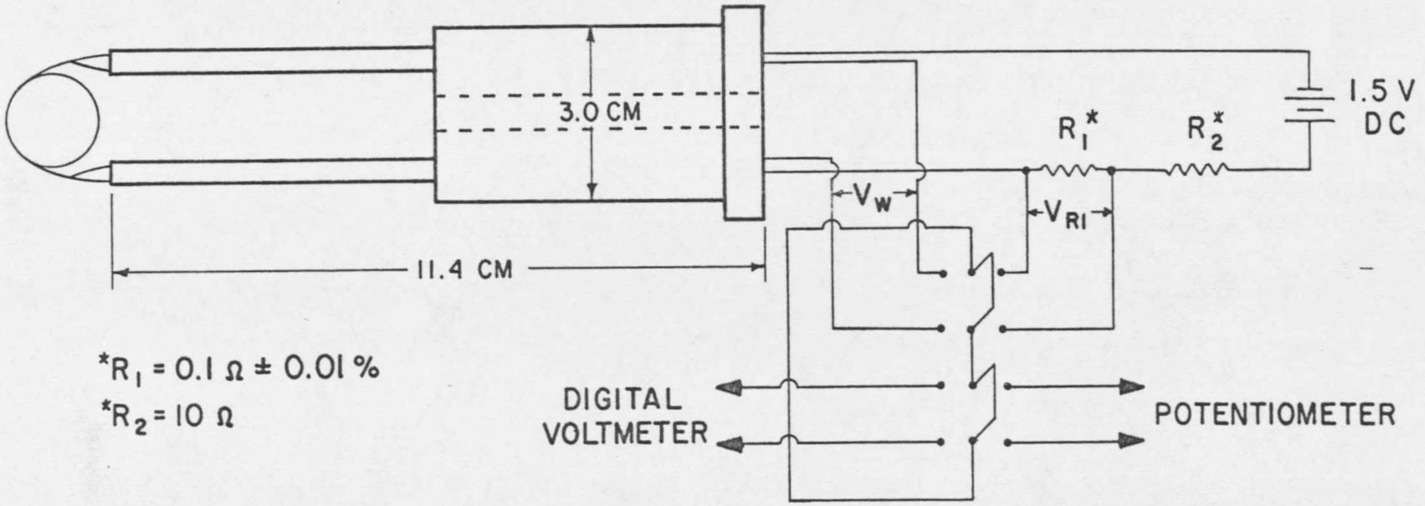

Figure 1. Schematic Representation of the Experimental Equipment

and was located a distance of one centimeter from the end of the ThO_2 thermocouple tubes. A ZrO_2 sight tube was also incorporated in the plug and thus allowed measuring the higher temperatures with a Pyro-Micro-Optical Pyrometer and the lower temperatures by insertion of a chromel-alumel thermocouple. Pyrometer calibration factors were obtained using a Milletron Pyrometer Calibration Standard containing a lamp calibrated by the General Electric Co. (with all measurements traceable to the National Bureau of Standards). The iridium wire specimen was connected in series with a 0.1Ω ± .01% precision resistor. The circuit was completed with a 10Ω resistor and 1.5 dc volt power supply. Both resistors were made from Manganin wire having a temperature coefficient of resistivity of 10 ppm/°C and thus were unaffected by the small changes in room temperature.

The wire specimen was prepared by fusion-welding* two 14-cm iridium lead wires to another piece of the iridium wire having a length of 34 cm. The room-temperature length of the reference section (7.96 cm) of the iridium wire was accurately determined from its weight, diameter, and a published[5] value for the density equal to 22.49 $g\text{-}cm^{-3}$ after the reference section was cut from the system at the conclusion of the series of resistivity measurements. The diameter of the wire was

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*The fusion-welding process is described in Appendix A.

measured optically to ±1μ using a Gaertner Cathetometer, and the weight determined with a precision of ±0.1 mgm. Measurement of the wire diameter before and after the series of resistivity measurements yielded no detectable change. Copper lead wires were fusion-welded to the ends of iridium wires extending from the ends of the ThO_2 tubes; the copper-iridium thermal EMF over the range of 0-100°C was reported[6] to be only 1.1μv/°C. Differences in the temperatures of the four Ir-Cu junctions was minimized by directing a stream of air across the junctions which were in close proximity.

Prior to making the resistivity measurements, the reference section of the specimen was given a recrystallization anneal by heating in argon to approximately 2300°K for 60 minutes in the high-temperature furnace described later.

In preparation for the measurements at specimen temperatures in the range of 293-1241°K, the plug containing the wire specimen was placed in a type 54341-A Lindberg Hevi-Duty furnace having a maximum operating temperature of 1283°K. After sealing, the furnace was continuously flushed with argon to prevent oxidation of the specimen. The furnace temperature was controlled with a Variac sliding-contact autotransformer and was monitored with a digital millivoltmeter connected to a Platinel II thermocouple installed in the furnace. Continuous specimen temperature readings were provided by a Digitec dc millivoltmeter connected to a chromel-alumel thermocouple placed next to the iridium wire specimen. Both thermocouples had an ice-bath reference. After the furnace temperature had stabilized, the temperatures indicated by the separate thermocouples were nearly the same. The voltage drops across the reference section of the wire specimen (V_w) and the precision resistor (V_{R1}) were determined using a Honeywell model 2745 potentiometer with a precision of ±0.001 millivolts after preliminary values were obtained using a digital voltmeter (Hewlett Packard Model 3440A having a precision of ±0.01 millivolts). The use of the digital voltmeter (and two double-pole double-throw switches) allowed approximate voltage values to be measured quickly; subsequently the values were measured precisely using the potentiometer, thus allowing a rapid data-collecting procedure. The error due to a very slow drift in the current and voltage provided by the dc power supply was eliminated by alternately and repetitively measuring V_w and V_{R1}. Repeated measurements at the same temperature were reproducable to ±0.005 millivolts.

For the data taken at specimen temperatures in the range of 1585-2275°K, the plug containing the specimen was installed in a ZrO_2 refractory tube which was in turn located inside the tantalum heating element of a high-temperature furnace to which argon was fed. The argon was passed through a purifier containing calcium turnings held at approximately 925°K to remove any traces of oxygen and water vapor present. The purified argon was then introduced into the furnace.

A cast ZrO_2 cylinder and heat-reflector discs were placed in the refractory tube beneath the wire specimen. These discs completed an enclosure for the wire; when heated, this enclosure closely approached being a black-body cavity such that the pyrometer readings were assumed to correspond to true temperature. Temperature measurements were taken with a Pyro-Micro-Optical Pyrometer, which was sighted through the sight tube located in the plug (the pyrometer was calibrated by sighting through this same sight tube onto the calibration lamp mentioned earlier). Voltage measurements for specimen temperature exceeding 1585°K were made using only the digital voltmeter (thus, voltage drops in the range of 13-21 millivolts were measured with a precision of ±0.01 mv). Repeated measurements at the same temperature were reproduceable to ±0.05 mv.

The results of resistivity measurements at 77-273°K and the procedure employed in their determination are contained in Appendix B.

Results and Discussion

The electrical resistivity data obtained in the present study in the range of 293-2275°K are presented in Table II and Figure 2. All of the data tabulated by L'vov, et al[4], are also shown; their tabulated values are in good agreement with those of the present study, with maximum deviation being less than 5.2%. The data points presented graphically by L'vov, et al, at the higher temperatures were noticed to

TABLE II MEASURED AND CALCULATED RESISTIVITY VALUES AND ANALYSIS

Temperature, °K	Measured ρ	Calculated*ρ	Deviation, %
293	5.62	5.66	0.70
294	5.62	5.68	1.04
301	5.73	5.81	1.46
425	8.15	8.23	0.95
428	8.22	8.29	0.81
465	9.14	9.02	-1.33
627	12.33	12.31	-0.14
630	12.39	12.38	-0.11
633	12.44	12.44	-0.01
677	13.22	13.37	1.10
717	14.30	14.22	-0.55
835	16.76	16.82	0.39
836	16.98	16.85	-0.78
838	17.02	16.89	-0.75
905	18.58	18.43	-0.82
996	20.51	20.58	0.34
999	20.79	20.65	-0.66
1093	23.03	22.96	-0.29
1200	25.43	25.70	1.06
1201	25.69	25.72	0.14
1241	26.66	26.78	0.44
1585	36.85	36.40	-1.23
1743	41.17	41.10	-0.18
1789	41.74	42.49	1.79
1875	44.64	45.10	1.04
1975	48.34	48.16	-0.38
2051	51.52	50.48	-2.03
2261	55.02	56.77	3.18
2275	58.48	57.18	-2.22

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*Values calculated from Equation (1)

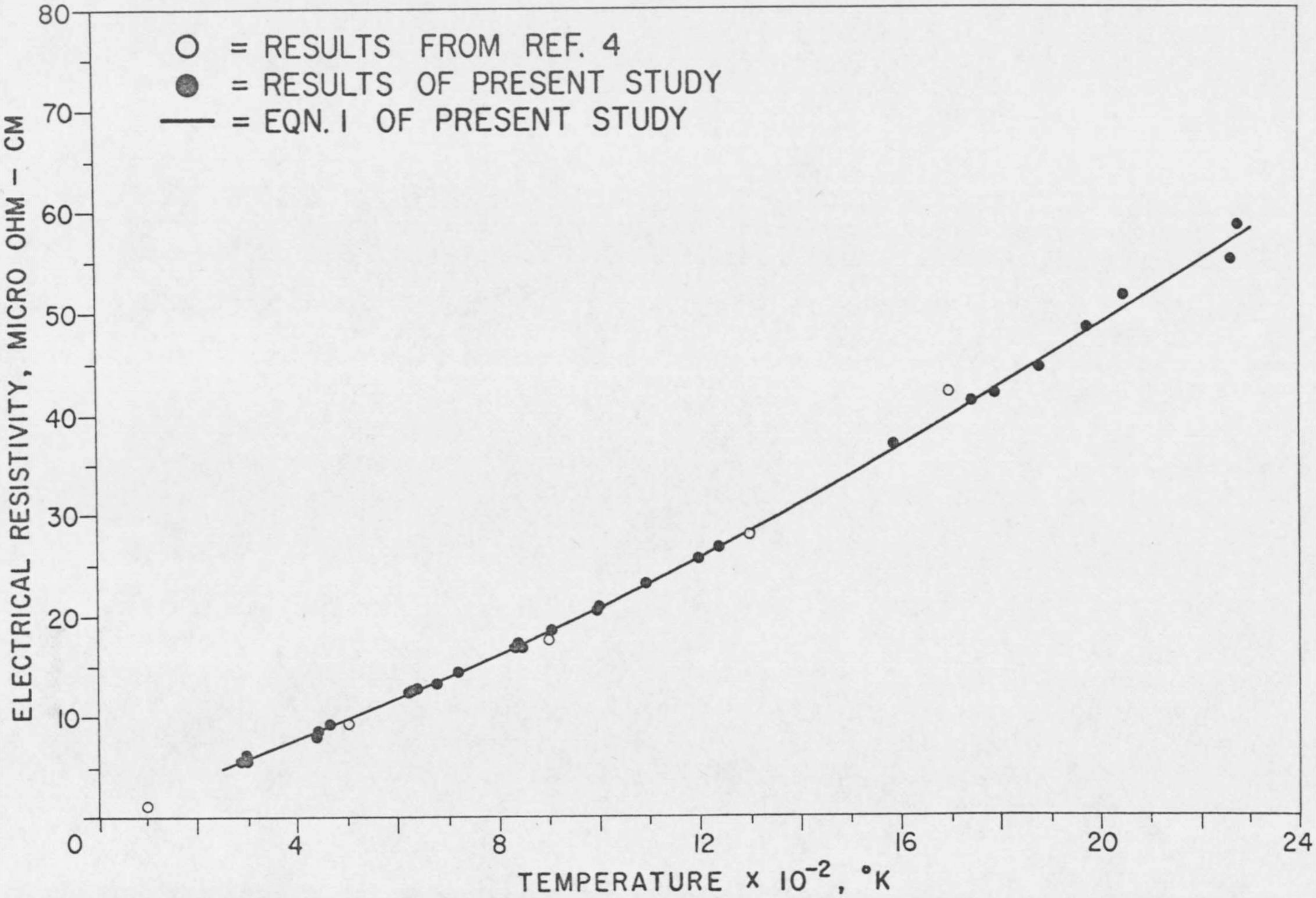

Figure 2. The Temperature Dependence of the Electrical Resistivity of Iridium

be increasing at a greater rate than those of the present study. This effect was probably not due to a difference in purity, since the purity of the iridium was nearly the same in both studies. (L'vov, et al, reported a purity of 99.96% Ir with impurities of Na-0.01, Si-0.01, and the Platinum group metals-0.01%). The difference in the preparation of the specimens could, however, present a possible explanation. The Iridium samples used by L'vov, et al, were prepared from powder by sintering pressed billets in a vacuum at a temperature of about 2500°K. This process left voids which at high temperatures could effectively increase the electrical resistivity. L'vov, et al, point out, though, that an attempt was made to take the effect of residual porosity into account. The wire specimen used in the present study would have considerably fewer and smaller voids following the plastic deformation involved in making the wire.

Values of the resistivity reported in "Thermophysical Properties of High Temperature Solid Materials"[2], if shown on Figure 2, would lie above the values from the present study but below those reported by L'vov, et al. These values were reported in graphical form and therefore an accurate representation of them was somewhat uncertain. Although no purity content was given, impurities of Rh (0.02-0.05), Ru (0.002-0.005), and Pd (0.001%) were reported.

The present data was correlated by the analytic expressions of the form:

$$\rho_t = a + bt + ct^2 + dt^3 + \dots \qquad \text{(A)}$$

where ρ_t is the electrical resistivity expressed in microhm-cm, t is the temperature in °K. The mean temperature coefficient was defined by the following relationship:

$$\alpha_{avg} = 1000(\rho_t - \rho_o) \;/\; \rho_o\,(t - t_o) \qquad \text{(B)}$$

where α_{avg} is the mean temperature coefficient between 273 and t °K and ρ_o is the electrical resistivity at 273°K, which is t_o. The experimental data obtained in the present study were expressed in the analytical form of Equation (A) for various temperature ranges and various orders by means of the least-squares method of curve fitting. The following equations were obtained:

$$\rho_t\ (293\text{-}2275°\text{K}) = 19.702 \times 10^{-3}t - 2.586 \times 10^{-6}t^2 + 4.64 \times 10^{-9}t^3 - 1.08 \times 10^{-12}t^4 \qquad (1)$$

$$\rho_t\ (293\text{-}2275°\text{K}) = 1.105 + 13.962 \times 10^{-3}t + 6.455 \times 10^{-6}t^2 - 0.760 \times 10^{-9}t^3 \qquad (2)$$

$$\rho_t\ (293\text{-}2275°\text{K}) = 0.2883 + 16.887 \times 10^{-3}t + 3.651 \times 10^{-6}t^2 \qquad (3)$$

$$\rho_t\ (293\text{–}1241°\text{K}) = 18.574 \times 10^{-3} t + 1.242 \times 10^{-6} t^2 + 0.881 \times 10^{-9} t^3 \qquad (4)$$

$$\rho_t\ (1200\text{–}2275°\text{K}) = -9.909 + 29.409 \times 10^{-3} t \qquad (5)$$

Error analysis for Equations (1) through (5) yielded the following maximum deviations of the values calculated by means of the respective equations for the experimental data points:

Eqn. (1): Maximum deviation over the range 293–2275°K: 3.18%

Eqn. (2): Maximum deviation over the range 293–2275°K: 3.39%

Eqn. (3): Maximum deviation over the range 293–2275°K: 3.84%

Eqn. (4): Maximum deviation over the range 293–1241°K: 1.60%

Eqn. (5): Maximum deviation over the range 1200–2275°K: 2.84%

Deviation of the individual values calculated by means of Equation (1) are included in Table II.

For maximum accuracy over the entire range of 293–2275°K, using a single expression, Equation (1) should be used. In the same temperature range, only a slight decrease in accuracy (3.39 vs 3.18%) is observed by using Equation (2) and a modest additional decrease in accuracy (3.84 vs 3.39%) by using Equation (3) while calculation of values is simplified because of the lower order of Equations (2) and (3). If the data are broken up into two temperature ranges, Equations (4) and (5) best represent the experimental data with minimum error.

For the convenience of the reader in using the present data, Tables III and IV are included. Table III contains the electrical resistivity values at 100°K intervals. Table IV contains the mean temperature coefficients for the electrical resistivity of iridium at 100°K intervals.

An attempt was made to correlate the experimental data to the theory presented by Grüneisen[7], who defined the parameter σ equal to ρ/T (where ρ is the resistivity at a temperature T), with σ asymptotically approaching a limiting value, $\sigma\infty$, at high temperatures. The following theoretical equation was presented:

$$\frac{\sigma}{\sigma\infty} = \frac{20}{X^4} \int_0^X \frac{\xi^4 \, d\,\xi}{e^{\xi} - 1} - \frac{4X}{e^X - 1}$$

where $X = \Theta/T$, with Θ being the characteristic temperature.

The resistivity data obtained in the present study were divided by the respective temperatures, and the resultant σ values were plotted versus temperature. However, the plotted values did not asymptotically approach a limiting value, therefore no correlation with his theory was possible. When L'vov's, et al, resultant σ values were plotted versus temperature, there was even less tendency for the σ values to converge upon a limiting value. Lack of correlation to the theory presented by Grüneisen might be associated with a progressively changing value for the characteristic temperature, as was suggested by L'vov, et al.

TABLE III ELECTRICAL RESISTIVITY VALUES FOR IRIDIUM

Temperature, °K	ρ,* microhm-cm
293	5.66
300	5.79
400	7.74
500	9.72
600	11.75
700	13.86
800	16.04
900	18.31
1000	20.68
1100	23.14
1200	25.70
1300	28.35
1400	31.10
1500	33.93
1600	36.84
1700	39.81
1800	42.82
1900	45.87
2000	48.92
2100	51.96
2200	54.97
2300	57.91

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*Values calculated from Equation (1)

TABLE IV MEAN TEMPERATURE COEFFICIENTS OF ELECTRICAL RESISTIVITY OF IRIDIUM

Temperature Range, °K	α_{avg}* °K^{-1}
273 - 300	4.044
273 - 400	3.801
273 - 500	3.798
273 - 600	3.826
273 - 700	3.876
273 - 800	3.933
273 - 900	3.999
273 - 1000	4.074
273 - 1100	4.151
273 - 1200	4.232
273 - 1300	4.316
273 - 1400	4.399
273 - 1500	4.482
273 - 1600	4.565
273 - 1700	4.644
273 - 1800	4.717
273 - 1900	4.786
273 - 2000	4.848
273 - 2100	4.901
273 - 2200	4.946
273 - 2300	4.980

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*α_{avg} was calculated using Equation (B) with $\rho_o = 5.22$ calculated from Equation (1) at 273°K (an experimental value of 5.20 was obtained at 273°K)

Summary

The electrical resistivity of iridium was measured in the range of 293–2275°K. The following equation represents the results:

$$\rho_t = 19.703 \times 10^{-3} t - 2.586 \times 10^{-6} t^2 + 4.64 \times 10^{-9} t^3 - 1.08 \times 10^{-12} t^4$$

for the electrical resistivity, ρ_t, expressed in microhm-cm and t expressed in °K. Mean temperature coefficients of resistivity were calculated.

PART II

THE THERMAL EXPANSION OF IRIDIUM AT HIGH TEMPERATURES

Introduction

The thermal expansion of iridium was studied recently by Singh[1] in the range of 30-865°C using the X-ray diffraction technique. The following equations represented his results:

$$a_t = 3.8383 + 23.52 \times 10^{-6} t + 4.92 \times 10^{-9} t^2 + 0.89 \times 10^{-12} t^3$$

$$\alpha_t = 6.13 \times 10^{-6} + 2.56 \times 10^{-9} t + 0.70 \times 10^{-12} t^2$$

where a_t is the lattice parameter in Å at t°C and α_t is the coefficient of thermal expansion. Earlier studies[2] made by Holborn and Valentiner (1907) in the range 0 to 1000°C are discussed later. Other available data[3-5] on the expansivity of iridium either refer to low temperatures or average values over large temperature ranges. A need for expansivity data for iridium at temperatures ranging to 2200°C prompted making the expansivity measurements reported in the present paper.

Specimen Analysis

Commercially pure iridium wire, 0.635 mm (25 mils) in diameter, was purchased from Engelhard Industires, Carteret, New Jersey. Results of a spectrographic analysis* of the wire are contained in Table I along with the results of a vacuum fusion analysis** for oxygen,

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*Performed at the Matthey Bishop Co., Malvern, Pennsylvania

**Performed at the Magnflux Testing Laboratories, Los Angeles, California

TABLE I ANALYSIS OF THE IRIDIUM WIRE

(Amounts of Impurity Elements Expressed in PPM)

Pt	15	Sb	<10	Fe	87	Sn	< 1
Pd	5	As	<10	Pb	< 1	Ti	6
Rh	54	Bi	< 1	Mg	5	Zn	<10
Ru	25	B	5	Mn	1	Ca	7
Os	< 3	Cd	< 1	Mo	1	O	30
Au	3	Cr	< 1	Ni	3	H	1
Ag	< 1	Co	< 1	Si	7	N	15
Al	9	Cu	2	Te	<10		

hydrogen, and nitrogen. The total measured impurity content of the metal was seen to be 0.028%. Although the analysis did not include all known elements, the iridium content was estimated to exceed 99.95%.

Experimental Method

The experimental equipment used in the present study was of a simple design which allowed taking the experimental data directly by visual means. On one side of a chamber, a 12.7-cm diameter pyrex window was provided. Through this window expansivity measurements were made with a Gaertner cathetometer and telemicroscope with a precision of ± 1 micron. The window was checked quantitatively for optical quality at various locations by measuring a lined grid. No change in the measured length of a selected line segment was detected.

On the opposite side of the chamber was located a view port containing a pyrex window of optical quality. Through this window temperature measurements were taken with a 8630 series Leeds and Northrup optical pyrometer provided with a 3.8-cm focal-length lens, which provided a magnified image of the wire specimen. The temperature range was extended below that obtainable with this pyrometer by use of a Pyro-micro optical pyrometer.

The electrical current used to self-resistance heat the wire specimen was supplied by a 20:1 step-down transformer in series with a constant voltage power supply, the current being controlled by two Variac sliding-contact autotransformers in sequence. The electrical current was passed into the chamber by means of water-cooled copper-alloy terminals which were electrically isolated from the chamber by means of vacuum-tight teflon gaskets. One end of the wire specimen was rigidly clamped to one terminal while the other end of the wire was passed through a polished ruby watch bearing into a mercury pool located in the other terminal. This arrangement allowed unrestrained thermal expansion of the specimen to occur in the absence of stress and buckling deflection.

The wire specimen was prepared by fusion-welding two short pieces of the iridium wire (at a separation of 2.1 cm from each other) to a piece of the wire having a length of 11.4 cms. Cutting of the attached wires produced two optical reference points which extended approximately 0.6 mm from the surface of the specimen (the magnitude of the cooling effect produced by the reference points was measured; at the highest specimen temperatures, the temperature was constant over the 2.1 cm reference section to within ± 7°C).

After the specimen was mounted in the chamber, the latter was evacuated and back-filled with argon twice. The chamber was filled with argon at a pressure of <u>ca</u> 50 torr in excess of atmospheric pressure at all times when the specimen was heated. Prior to making the expansion measurements the specimen was given a recrystallization anneal by heating to 2200°C for 40 minutes. While making the expansion determinations, the specimen was held at each temperature level until equilibrium occured. Sequential measurements of the length of the heated reference section agreed within 1-2 microns.

A separate experiment was conducted to determine the relationship between the observed temperature and the corresponding true wire temperatures. Each of the legs of an Ir-Ir 40 Rh thermocouple (calibrated by Engelhard Industries with traceability to the National Bureau of Standards) was separately fusion welded to a second specimen of the iridium wire such that a separation of approximately 2 mm existed between the points of attachment of the two legs. The pyrometer was sighted on the specimen at the point of attachment of the Ir 40 Rh wire while the specimen was self-resistance heated in argon. The thermocouple EMF (read with a Honeywell 2745 potentiometer, ± 1 μ volt) was translated to a temperature taken to be the true wire temperature at the point of attachment of the Ir 40 Rh thermocouple leg. The true wire temperatures thus obtained were estimated to be accurate within ± 20°C. The extent of the drift of the thermocouple output due to diffusion during the brief periods at the higher temperatures was measured by repeating the series of measurements and was found to correspond to a temperature error of ca 10°C. Correction for the drift was then made.

Calibration factors for the pyrometers and loss factors for the window due to reflection and absorption were obtained using a Milletron Pyrometer Calibration Standard containing a lamp calibrated by the General Electric Co. (with all measurements traceable to the NBS). Use of the calibration factors and loss factors allowed observed temperatures to be converted to true brightness temperatures (which would be appreciably lower than true wire temperature because of the low emittance of iridium). Resultant emittance values provided confirmation of the accuracy of the true-wire-temperature determinations by comparison of the emittance values with those determined elsewhere. Kuriakose and Margrave[6], using a blackbody method, obtained values of 0.30 ± 0.04 for the emittance (0.65 μ) of iridium wire heated in argon to temperatures in the range of 1180-1760°C. A value of 0.29 ± 0.01 was obtained in the present study in the same temperature range at virtually the same wave length (0.655 μ in the present study) and thus suggests confirmation of the accuracy of the true-wire-temperature determinations.

Results and Discussion

The expansion data obtained in the present study in the range of 891-2221°C are presented in Table II and Figure 1. The data range

TABLE II MEASURED AND CALCULATED EXPANSION VALUES AND ANALYSIS[1]

Temperature °C	Measured* % Expansion	Calculated** % Expansion	% Deviation
30	.0182	.0186	2.41
133	.0808	.0847	4.77
235	.1563	.1530	-2.11
338	.2162	.2249	4.03
440	.3022	.2989	-1.08
552	.3856	.3833	- .59
655	.4637	.4639	.05
767	.5497	.5548	.92
865	.6409	.6371	- .59
891	.670	.659	-1.58
1077	.834	.825	-1.09
1177	.919	.918	- .06
1192	.923	.933	1.06
1229	.951	.968	1.84
1236	.984	.975	- .88
1394	1.106	1.134	2.52
1460	1.190	1.203	1.10
1477	1.256	1.221	-2.77
1615	1.382	1.373	- .64
1718	1.500	1.493	- .50
1798	1.575	1.589	.90
1905	1.739	1.724	- .86
2025	1.875	1.884	.47
2061	1.940	1.934	- .32
2113	1.992	2.007	.77
2221	2.174	2.166	- .35

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*Measured values in the range 30-865°C are Singh's values[1]

**Values calculated from 4th order equation (Eqn. 3)

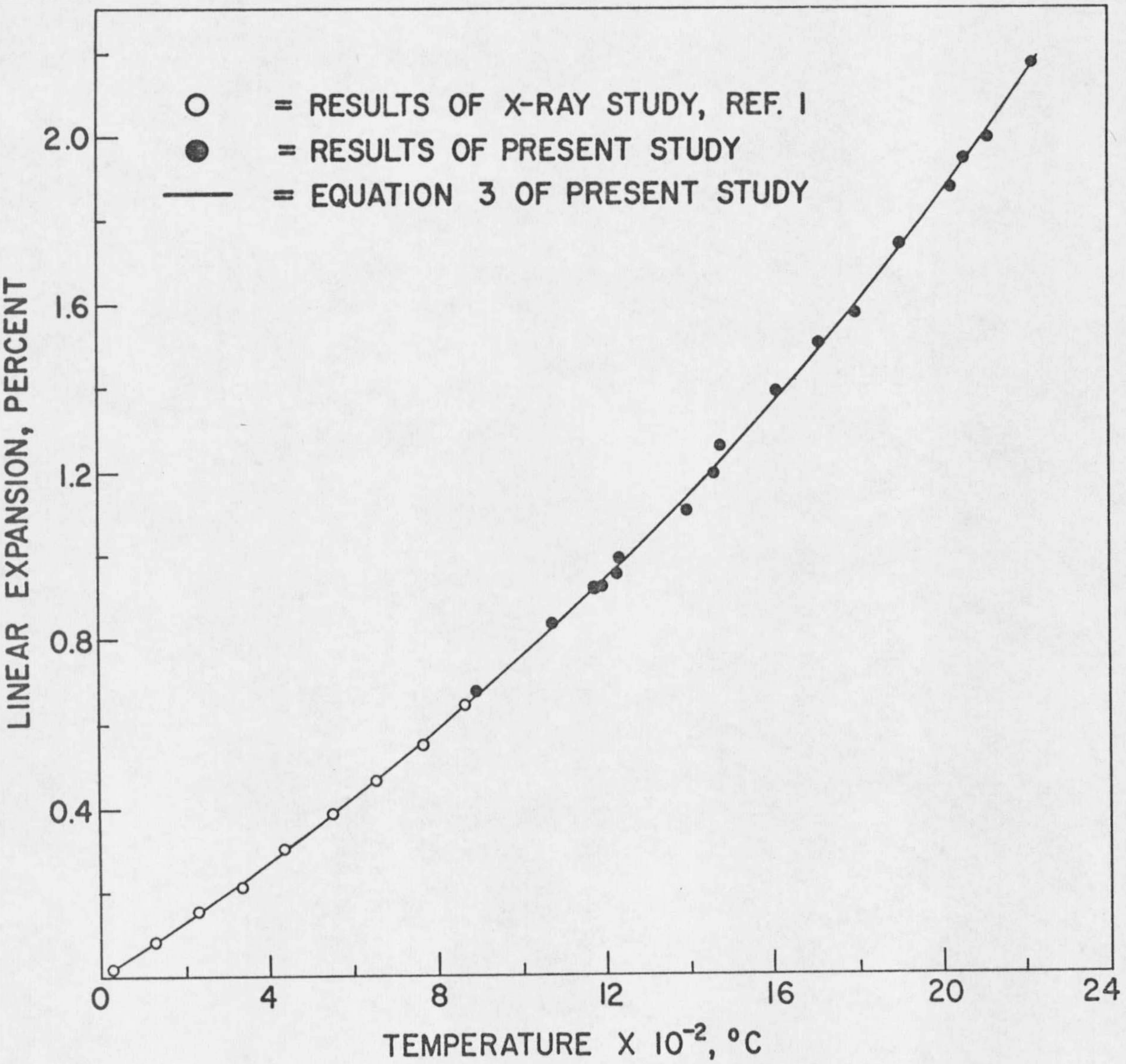

Figure 1. The Temperature Dependence of the Thermal Expansion of Iridium

was extended to include values down to 30°C by using values reported by Singh.[1] Values reported by Holborn and Valentiner[2], if shown on Figure 1, would lie above the values presented. These points were not included because the iridium used was of undetermined purity and the wire was under tension during the expansion measurements. The elongation due to the tensile stress would have been superimposed on the thermal expansion and would have increased in magnitude with increasing temperature. Values reported by Krikorian[5] were also not included because no experimental procedure or purity information was indicated in his report, which was stated to be of a preliminary nature. Singh used "Specpure" iridium powder in his X-ray diffraction study; this material was assumed to be spectroscopically pure and as such would be of higher purity than the wire material used in the present study (the wire used was of the highest purity available for iridium in wire form).

The data of Singh combined with the present data were correlated by analytical expressions of the following form:

$$100\left(\frac{L_t - L_o}{L_o}\right) = at + bt^2 + ct^3 + \ldots \qquad \text{(A)}$$

where L_t = reference length at t°C and L_o = reference length at 0°C; $100\,(L_t - L_o)/L_o$ is referred to as "percent expansion". The thermal expansion coefficient and average coefficient of expansion were defined by the following relationships, respectively:

$$\alpha_t = \frac{1}{L_o}\left(\frac{dL}{dt}\right) \qquad \text{(B)}$$

$$\alpha_{avg} = \left(\frac{\Delta L}{L_o \Delta t}\right) \qquad \text{(C)}$$

The experimental data were expressed in the analytical form of equations (A) and (B) for various orders by means of the least-squares method of curve fitting. The curve fitting included Singh's data. The following equations were obtained:

$$\text{Percent Expansion} = 575.1 \times 10^{-6}t + 175.3 \times 10^{-9}t^2 \qquad (1)$$

$$\text{Percent Expansion} = 647.3 \times 10^{-6}t + 71.9 \times 10^{-9}t^2 + 33.7 \times 10^{-12}t^3 \qquad (2)$$

$$\text{Percent Expansion} = 616.7 \times 10^{-6}t + 151.9 \times 10^{-9}t^2 - 28.16 \times 10^{-12}t^3 + 14.63 \times 10^{-15}t^4 \qquad (3)$$

Differentiation of Equations (2) and (3) after division by 100 yielded the following expressions for the thermal expansion coefficients:

$$\alpha_t = 6.473 \times 10^{-6} + 1.438 \times 10^{-9} + 1.011 \times 10^{-12}t^2 \qquad (4)$$

$$\alpha_t = 6.167 \times 10^{-6} + 3.038 \times 10^{-9}t - 0.8448 \times 10^{-12}t^2 + 0.5852 \times 10^{-15}t^3 \qquad (5)$$

Error analysis for Equations (1) through (3) yielded the following maximum percent deviations of the values calculated by means of the respective equations from the experimental data points:

Equation (1): Maximum Percent Deviation, range 30-2221°C = 7.34

Maximum Percent Deviation, range 655-2221°C = 3.29

Equation (2): Maximum Percent Deviation, range 30-2221°C = 8.22

Maximum Percent Deviation, range 440-2221°C = 2.75

Equation (3): Maximum Percent Deviation, range 30-2221°C = 4.77

Maximum Percent Deviation, range 440-2221°C = 2.77

Deviation of the values calculated by means of Equation (3) from each of the experimental data points is included in Table II.

For maximum accuracy over the entire range of 30-2221°C Equation (3) should be used. In the range 440-2221°C an almost negligible gain in accuracy is obtainable by using Equation (2) while calculation of values is simplified because of the lower order of Equation (2).

For the convenience of the reader in using the present data, Tables III and IV are included. Table III contains the percent expansion and thermal expansion coefficients of iridium at 100° intervals. Also included in Table III are values calculated by means of Singh's equations. Table IV contains the average coefficients of expansion of iridium at 100° intervals.

TABLE III PERCENT EXPANSION AND THERMAL EXPANSION COEFFICIENTS OF IRIDIUM

Temperature (°C)	% Expansion*	% Expansion** (Singh)	$\alpha_t \times 10^{6*}$ (°C^{-1})	$\alpha_t \times 10^{6**}$ (Singh)
25	.0155	.0154	6.24	6.19
100	.0632	.0626	6.46	6.39
200	.129	.128	6.75	6.67
300	.198	.196	7.02	6.96
400	.270	.267	7.28	7.27
500	.344	.341	7.55	7.59
600	.421	.419	7.81	7.92
700	.500	.500	8.08	8.27
800	.582	.584	8.36	8.63
900	.667		8.64	
1000	.755		8.95	
1100	.846		9.27	
1200	.940		9.61	
1300	1.038		9.97	
1400	1.140		10.37	
1500	1.246		10.80	
1600	1.356		11.26	
1700	1.471		11.77	
1800	1.592		12.31	
1900	1.718		12.90	
2000	1.850		13.55	
2100	1.989		14.24	
2200	2.135		14.99	
2225	2.173		15.19	

*Values calculated using Equations (3) and (5)

**Calculated using Singh's Equations

TABLE IV AVERAGE COEFFICIENTS* OF EXPANSION OF IRIDIUM

Temperature Range, °C	$\alpha_{avg} \times 10^6$, °C^{-1}	Temperature Range, °C	$\alpha_{avg} \times 10^6$, °C^{-1}
25-100	6.35	25-1300	8.02
25-200	6.50	25-1400	8.18
25-300	6.64	25-1500	8.34
25-400	6.77	25-1600	8.51
25-500	6.91	25-1700	8.69
25-600	7.04	25-1800	8.88
25-700	7.18	25-1900	9.08
25-800	7.31	25-2000	9.29
25-900	7.45	25-2100	9.51
25-1000	7.59	25-2200	9.74
25-1100	7.73	25-2225	9.80
25-1200	7.87		

*$\alpha_{avg} = \dfrac{\Delta L}{L_o \Delta t}$

Summary

The linear thermal expansion of iridium was measured in the range of 891–2221°C. The following equation was found to fit selected earlier results and the present results over the temperature range of 30–2221°C:

$$100\,\frac{L_t - L_o}{L_o} = 616.7 \times 10^{-6}t + 151.9 \times 10^{-9}t^2 - 28.16 \times 10^{-12}t^3 + 14.63 \times 10^{-15}t^4$$

for *t* expressed in °C. Thermal expansion coefficients and average coefficients of expansion were calculated.

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THE ELECTRICAL RESISTIVITY OF IRIDIUM AT HIGH TEMPERATURES

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PART II

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APPENDICES

APPENDIX A

A capacitor discharge welder which consisted of two parallel 16,500 μ F capacitors in series with a 10 ohm, 8.5 amp reostat was used for the fusion-welding process. The reostat provided partial control over the discharge time. The welding was done in air and consisted of two iridium-iridium welds and four copper-iridium welds. The separate wires, which were to be welded together, were placed in the circuit of the capacitor discharge welder and positioned such that a small air gap existed where the welded junction was to be made. When the air gap was closed, the capacitors would discharge causing an electric arc to pass between the wires at the junction sight. Through trial and error, selected voltage and resistance settings were established for each of the different type welds. The voltage and ohm values indicated below represent those settings which at the time produced the best results.

The iridium-iridium welds were done at a 65 volt setting and a 1 ohm reostat setting. At these high values, a hot arc was formed. If the speed to contact was fast, a solid and rigid joint could be established. Using this procedure, a solid joint was made during one of the welds, but during the second weld, the time to contact was apparently too slow, and the arc melted through the main wire. After repeated attempts, another solid joint was formed.

The copper-iridium welds were done at a 70 volt setting and a 4 ohm reostat setting. Since copper has a much lower melting point than that of the iridium (1083 vs 2443°C)*, the copper would melt back during the welding attempt. To form the junction, the copper wire was forced onto the iridium wire during the discharge to cause the copper to solidify around the iridium wire.

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*Melting point temperatures obtained from Handbook of Chemistry and Physics, The Chemical Rubber Co., 52nd Edition, 1971-72, P. D 142

APPENDIX B

The electrical resistivity data for iridium in the range of 77-273°K were measured by placing the plug which contained the wire specimen into an insulated Thermos bottle containing a low temperature liquid—LN_2, LO_2, solid CO_2 and acetone, or ice water. The LO_2 was obtained by passing gaseous oxygen into a flask surrounded by LN_2. A chromel-alumel thermocouple, referenced to an ice bath, was placed through the sight tube into the low temperature liquid. The electrical resistivity was obtained by measuring the dc-voltage drop across the specimen (V_W) and the voltage drop across a precision resistor (V_{R1}) as before. The voltages were determined using a Honeywell model 2745 potentiometer with a precision of ±0.001 millivolts. Repeated measurements at the same temperature indicated that the values were reproducable to ±0.003 millivolts.

The following table contains the electrical resistivity values obtained from the experimental measurements:

ELECTRICAL RESISTIVITY VALUES FOR IRIDIUM AT LOW TEMPERATURES

Low Temperature Liquid	Temp* °K	ρ_t microhm-cm
Liquid nitrogen	77	.85
Liquid oxygen	88	1.13
Solid carbon dioxide-acetone	183	3.38
Ice water	273	5.20

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*Temperatures for LN_2 and LO_2 were determined using their respective boiling points corrected for atmospheric pressure.

These data points were not included in the data analysis and curve fitting because the chromel-alumel thermocouple was apparently ineffective in determining the temperature of LN_2 and LO_2. (Temperatures measured with the thermocouple for LN_2 and LO_2 were as much as 16°K lower than those reported in the aforementioned table. No reasonable explanation for this temperature difference was found.)

Data analysis and curve fitting of the data points in the range of 77-999°K indicate that the values sighted are not unreasonable. The following analytical expression was found which best fit the data points in the range of 77-999°K:

$$\rho_t = -.725 + 21.143\ 10^{-3} t$$

where ρ_t is the electrical resistivity in microhm-cm, and the temperature $\underline{t}$ in °K. The maximum deviation for this equation was 6.36% and occured at the lowest temperatures.