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Radiance Temperatures (in the Wavelength Range 527 to 1500 nm) of Palladium and Platinum at Their Melting Points by a Pulse-Heating Technique[1]

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The radiance temperatures (at seven wavelengths in the range 527 to 1500 nm) of palladium and platinum at their respective melting points were measured by a pulse-heating technique. The method, based on rapid resistive self-heating of a specimen from room temperature to its melting point in less than 1 s, used two high-speed pyrometers to measure specimen radiance temperatures every 0.5 ms during the heating and melting period. Melting was manifested by a plateau in the radiance temperature-versus-time function for each wavelength. The melting-point radiance temperatures for a given specimen were determined by averaging the measured temperatures along the plateau at each wavelength. The melting-point radiance temperatures for each metal as determined by averaging the results for several specimens at each wavelength are as follows.

Palladium	Platinum
1723 K at 529 nm	1904 K at 527 nm
1696 K at 626 nm	1871 K at 624 nm
1687 K at 657 nm	1859 K at 657 nm
1669 K at 720 nm	1838 K at 717 nm
1643 K at 811 nm	1806 K at 810 nm
1614 K at 908 nm	1773 K at 907 nm
1444 K at 1500 nm	1577 K at 1500 nm

Based on uncertainties arising from pyrometry and specimen conditions, the expanded uncertainty (two-standard deviation level) is about ±7 K for the

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reported values in the range 527 to 900 nm and about ± 8 K for the reported values at 1500 nm.

KEY WORDS: emissivity (normal spectral); high-speed pyrometry; high temperature fixed points; melting; palladium; platinum; pyrometry; radiance temperature.

1. INTRODUCTION

Earlier measurements, performed during the last two decades at the National Institute of Standards and Technology (NIST) in the United States and at the Istituto de Metrologia "G. Colonnetti" (IMGC) in Italy, have indicated that the radiance temperature[5] of a metal at its melting point in the wavelength range 500 to 1000 nm is essentially constant during the initial melting period and is highly reproducible for different specimens of a given metal [1]. These findings suggest that melting-point radiance temperatures of selected metals could be used as easily realizable high-temperature references for secondary calibrations of optical pyrometers and for *in situ* temperature checks on complicated measurement systems. In order to determine the suitability of other metals for these purposes, an effort was made to study as many of the high-melting point pure metals (melting point above 1700 K) as could be done practically. Recent work at NIST extended measurements of melting-point radiance temperatures to metals with lower melting points [2, 3] and to a longer wavelength, 1500 nm [4–6]. With this paper, which presents similar measurements on palladium and platinum, we essentially complete our program of radiance-temperature measurements at the melting point on pure high-temperature metals that could practically be used for secondary temperature references. The few high-melting point metals that have not been studied tend to be very expensive and difficult to obtain in the strip form required by our measurement technique.

The measurement technique, based on rapid resistive self-heating of a strip-shaped specimen by passing a large electrical current pulse through it, heats the specimen from room temperature to its melting point in less than 1 s. As the specimen heats, its radiance temperature at each of seven wavelengths (nominally between 500 and 1500 nm) is measured by two pyrometers every 0.5 ms. Radiances at each of six wavelengths in the nominal

[5] The radiance temperature (sometimes referred to as the brightness temperature) of the specimen surface at a given wavelength is the temperature at which a blackbody at the same wavelength has the same radiance as the surface. The wavelength is the effective wavelength of the measuring pyrometer.

range 500 to 900 nm are measured with a high-speed six-wavelength pyrometer viewing a surface of the specimen. Radiances at two wavelengths, nominally 650 and 1500 nm, are measured by a high-speed two-wavelength pyrometer viewing a surface of the specimen. The melting-point radiance temperatures for a given specimen are determined by averaging the measured radiance temperatures along the melting plateau for each wavelength. All temperatures reported in this paper, except where explicitly noted otherwise, are based on the International Temperature Scale of 1990 (ITS-90) [7].

Details concerning the design and construction of the pulse-heating system [8, 9] and the design, operation, and calibration of the six-wavelength pyrometer [10] are given in the cited publications. The 650-nm channel of the two-wavelength pyrometer is used for calibration of the 1500-nm channel and as a control to compare its results to that of the similar wavelength of the six-wavelength pyrometer. Details concerning the calibration and operation of the two-wavelength pyrometer are given in earlier publications [4, 5].

2. MEASUREMENTS

Measurements of the radiance temperature of palladium at its melting point were performed on 20 specimens in the form of strips cut from 50×50-mm, 99.998% pure (by mass) metal foils. The nominal dimensions of each strip were as follows: length, 50 mm; width, 3 mm; and thickness, 0.25 mm. Fourteen specimens were used with the six-wavelength pyrometer, and six specimens were used with the two-wavelength pyrometer. A typical analysis of the Pd material used to fabricate the foils, as reported by the manufacturer, yielded the following impurities (ppm, by mass): Fe, 2; Ag and Si, 1; Au, Ca, Cu, and Mg, <1; and gases and unreported elements, 12.

Radiance temperature measurements on platinum at its melting point were performed on seven strip specimens cut from strip stock of 99.994% pure (by mass) platinum. The nominal dimensions of each strip were as follows: length, 51 mm; width, 3.2 mm; and thickness, 0.25 mm. A typical analysis of the platinum material, as reported by the manufacturer, yielded the following impurities (ppm, by mass): Rh, 16; B, 11; Fe, 8; Au, 7; Ir and Pd, <5; Si, <2; Ca, 1; and Cu, Ni, and Mn, <1.

The surface of each specimen of palladium and platinum was mechanically abraded with abrasive to remove possible surface contaminants, and all experiments were performed with the specimen in an argon gas environment (approximately 0.15 MPa) to minimize contamination of the specimen surface at high temperatures. The duration of the electrical

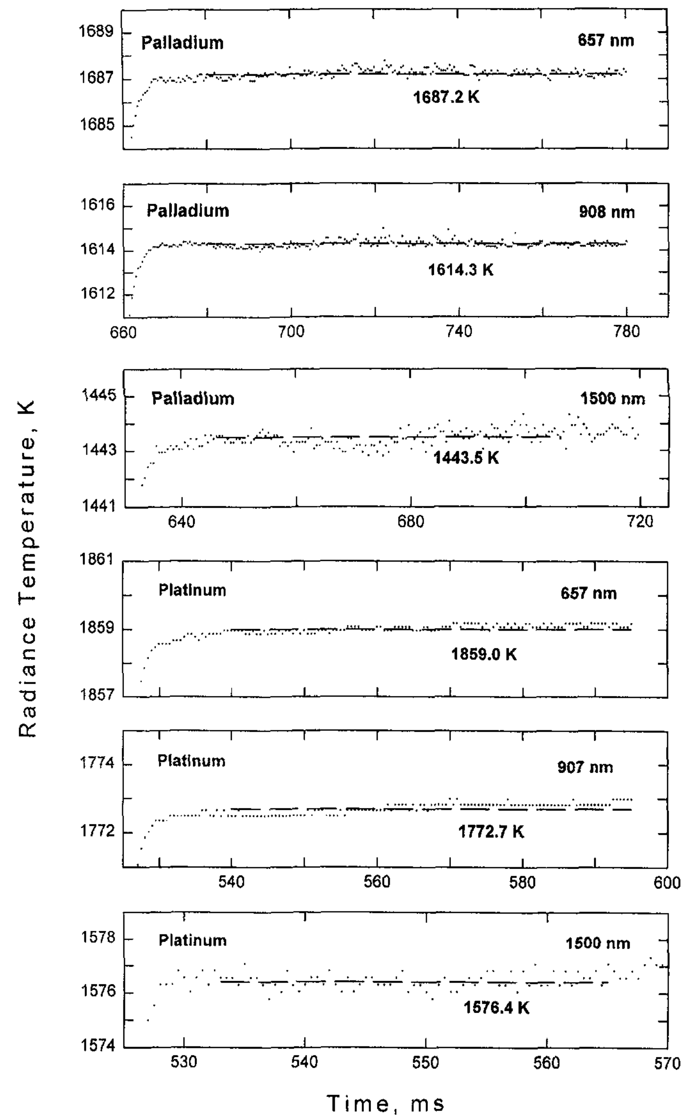

Fig. 1. Variation of the radiance temperatures of palladium and platinum specimens at three representative wavelengths (657, about 900, and 1500 nm) just before and during melting as measured with the pyrometers during typical pulse-heating experiments. The effective wavelengths, shown for each channel, were determined following the definition given by Kostkowski and Lee [11]. Each labeled temperature indicates the average melting-point radiance temperature for that experiment computed from the plateau data indicated by the dashed line.

current pulse, used to heat each specimen from room temperature to its melting temperature, ranged from approximately 150 to 700 ms for palladium and from approximately 525 to 550 ms for platinum.

Figure 1 shows typical radiance temperature data obtained at three representative wavelengths (657, near 900, and 1500 nm) by the pyrometers during pulse-heating experiments on palladium and platinum, respectively, near and at their melting points. Melting of the specimen is manifested by a plateau region in the measured radiance temperature results for each wavelength. The flat region along each plateau (indicated by the dashed line) was used to determine the melting radiance temperature at each wavelength. The radiance temperature along this flat region was essentially constant within a range of less than ± 1 K for all specimens. The effective wavelength for each pyrometer channel was determined at the respective average radiance temperature using the method of Kostkowski and Lee [11].

3. RESULTS

For each specimen, the radiance temperature at the melting point was determined for each wavelength by averaging the measured temperatures along the flat portion of the corresponding plateau (indicated by the dashed lines in Fig.1).

For palladium, the number of temperature measurements along the melting plateau used to obtain an average radiance temperature ranged from 35 to 201, depending upon the heating rate and the behavior of the specimen during melting. The standard deviation of an individual plateau radiance temperature from the average obtained for that specimen was in the range 0.1 to 0.5 K. The trend (or slope) of the radiance temperature along each plateau was determined by fitting a linear function in time to the measured radiance temperatures using the method of least squares. The slope of the linear functions for all experiments on palladium was in the range -17 to 33 $K \cdot s^{-1}$. The temperature difference between the beginning and the end of the plateau, as determined from this slope, was in the range -0.4 to 0.8 K. Heating rates for each specimen were determined by fitting a linear function in time to the radiance temperatures measured during the premelting period. The heating rates for the palladium specimens (slopes of the linear functions approximately 20 K below the melting plateau) ranged from 1280 to 6900 $K \cdot s^{-1}$. An examination of the results on palladium showed no relationship between heating rate and the measured melting-point radiance temperature.

The number of temperature measurements along the melting plateau used to obtain an average radiance temperature for each platinum specimen ranged from 38 to 211. The standard deviation of an individual plateau radiance temperature for a given specimen from the average obtained for that specimen was in the range 0.1 to 0.4 K. The slope of the linear functions representing the melting plateau region of platinum was in the range -7 to 10 $K \cdot s^{-1}$, and the temperature difference between the beginning and the end of the plateau, as determined from this slope, was in the range -0.2 to 0.9 K. Because earlier trial experiments on platinum strips showed that its measured melting temperature was independent of its heating rate, the heating rate for platinum specimens was not varied significantly. The heating rates for platinum, determined as described above for palladium, were in the range 2240 to 3080 $K \cdot s^{-1}$.

Table I presents the final experimental results of the radiance temperature of palladium and platinum at their melting points in the wavelength

Table I. Average Radiance Temperature and Normal Spectral Emissivity, at Seven Wavelengths, of Palladium and Platinum at Their Melting Points

Material and melting temperature (K)[a]	Effective wavelength (nm)[b]	Radiance temp. (K)[c]	SD (K)[d]	Normal spectral emissivity[e]
Palladium (20 specimens),				
$T_m = 1826$ K [12]	529	1722.9	0.3	0.410
	626	1696.1	0.3	0.381
	657	1687.2	0.3	0.373
	720	1669.4	0.3	0.358
	811	1642.5	0.4	0.338
	908	1614.4	0.4	0.321
	1500	1443.5	0.6	0.248
Platinum (7 specimens)				
$T_m = 2041.3$ K [13]	527	1904.4	0.1	0.382
	624	1870.9	0.1	0.357
	657	1859.0	0.1	0.349
	717	1838.4	0.1	0.338
	810	1806.1	0.1	0.322
	907	1772.6	0.2	0.308
	1500	1577.0	0.4	0.249

[a] Melting temperature based on ITS-90.
[b] Determined at the respective radiance temperature using the definition of effective wavelength given by Kostkowski and Lee [11].
[c] Average of the specimen melting-point radiance temperatures at each effective wavelength.
[d] Standard deviation of an individual specimen melting-point radiance temperature from the average of values obtained from all specimens.
[e] Determined by means of Planck's law from the average plateau radiance temperature and the respective melting temperature for each material.

range 527 to 1500 nm. Depending upon wavelength, the standard deviation of an individual average melting temperature from the overall average is in the range from 0.3 to 0.6 K for palladium and 0.1 to 0.4 K for platinum. Also given in Table I are the corresponding values for the normal spectral emissivity of palladium and platinum at their melting points. Emissivity was calculated by means of Planck's law using the present results for radiance temperature and the value 1826 K for the melting point of palladium [12] and 2041.3 K for the melting point of platinum [13].

4. ESTIMATE OF UNCERTAINTIES

The major sources of uncertainty are (i) the calibration and operation of the pyrometer and (ii) the physical/chemical conditions and melting behavior of each specimen. A detailed analysis of the magnitudes of the uncertainties from all sources is given in an earlier publication [14]. Specific items in the error analysis (particularly those related to temperature range) were recomputed whenever the present conditions differed from those in the earlier publication. The expanded uncertainty (two-standard deviation level) in the reported values for melting-point radiance temperatures is estimated to be about ± 7 K in the wavelength range 527 to 908 nm and about ± 8 K at 1500 nm.

5. DISCUSSION

Only a few papers were found in the literature that report radiance temperatures of palladium and platinum at their melting points. The values for radiance temperature at the melting point as reported in the literature and the present results are listed in Table II and are plotted as functions of wavelength in Fig. 2. Since the results reported in the literature were obtained at different wavelengths than the present results, when comparisons are made below, the present results are interpolated to determine values at the same wavelengths as those reported in the literature. The results of Treverton and Margrave [15] for palladium at 650 nm are 3 K higher than the present results and the results of Bonnell et al. [16] for palladium at 645 nm are 6 K lower. Both results are within the combined uncertainty of the experiments. For platinum, the results of Hampson and Walker [18] at 660 nm are 16 K lower than the present results.

Krishnan et al. [17] measured directly the normal spectral emissivity of both palladium and platinum at several wavelengths in the liquid state as a function of temperature using laser polarimetry. Although much of their data for palladium and platinum are at temperatures too far above

Table II. Radiance Temperatures and Normal Spectral Emissivities at Wavelengths (λ) in the Range 527 to 1500 nm of Palladium and Platinum at Their Melting Points as Reported in the Literature

Element	Investigators	Ref. No.	Year	Purity (mass%)	λ (nm)	Radiance temperature (K) As reported	Radiance temperature (K) On ITS-90	Normal spectral Emissivity As reported	Normal spectral Emissivity Adjusted for a common MP[a]
Palladium	Treverton and Margrave	15	1971	99.95	650	1689	1691 ± 2	0.374	0.38
	Bonnell et al.	16	1972	99.95	645	1684	1684 ± 2	0.354	0.357
	Krishnan et al.	17	1990	99.9	514.5		1718[b]		0.381[c]
					633		1694[b]		0.38[c]
	Present work			99.998	529		1723 ± 7		0.410
					626		1696 ± 7		0.381
					657		1687 ± 7		0.373
					720		1669 ± 7		0.358
					822		1643 ± 7		0.338
					908		1614 ± 7		0.321
					1500		1444 ± 8		0.248
Platinum	Hampson and Walker	18	1961	99.7	660	1840	1842	0.308	0.315
	Krishnan et al.	17	1990	99.9	633		1878[d]		0.38[c]
					1064		1726[d]		0.298[c]
	Present work			99.994	527		1904 ± 7		0.382
					624		1871 ± 7		0.357
					657		1859 ± 7		0.349
					717		1838 ± 7		0.338
					810		1806 ± 7		0.322
					907		1773 ± 7		0.308
					1500		1577 ± 8		0.249

[a] Adjusted values for emissivity are based on the reported melting-point radiance temperatures (on ITS-90) and the respective melting temperature of 1826 K (on ITS-90) for palladium [12] and 2041.3 K for platinum [13].

[b] Based on the emissivities measured by the investigators and a melting temperature of 1826 K (on ITS-90) for palladium [12].

[c] Emissivity of the liquid measured directly by laser polarimetry.

[d] Based on the emissivities measured by the investigators and a melting temperature of 2041.3 K (on ITS-90) for platinum [13].

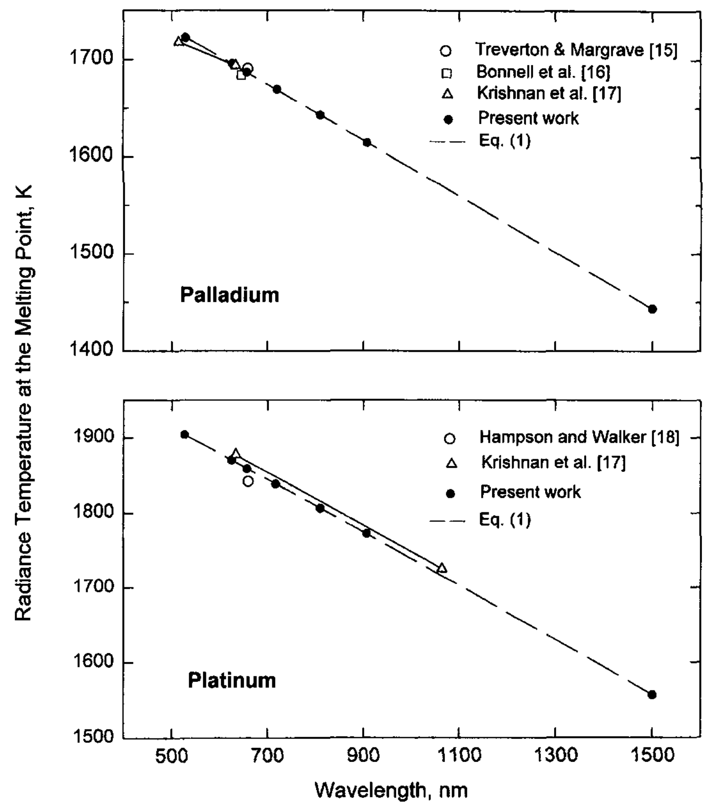

Fig. 2. Comparison (on ITS-90) of literature values and present results for the radiance temperatures of palladium and platinum at their melting points as a function of wavelength. The dashed line represents the quadratic function, Eq. (1), fitted to the radiance temperature-versus-wavelength data obtained in the present work.

the melting points to be useful for comparisons, the values for normal spectral emissivity reported in Table II were selected for those wavelengths where their measured data were taken at temperatures close to the melting point of the material. Values for the radiance temperatures at the melting point were computed from their emissivity results using the melting temperature of each material. For palladium, the resulting radiance temperature at 514.5 nm determined by Krishnan et al. is 9 K lower than the present value and is equal to the present value at 633 nm. Their results for platinum are 10 K higher than the present values at both reported wavelengths.

Also listed in Table II are values from the literature and the present values for the normal spectral emissivity of palladium and platinum at their melting points. The emissivity values obtained by the investigators were either computed using Planck's law and reported values for the melting temperatures of palladium and platinum or measured directly by laser polarimetry. To provide a common basis for comparison, the values for normal spectral emissivity were adjusted to a common melting temperature (ITS-90) of 1826 K for palladium [12] and 2041.3 K for platinum [13]. The results for normal spectral emissivity are plotted as functions of wavelength in Fig. 3.

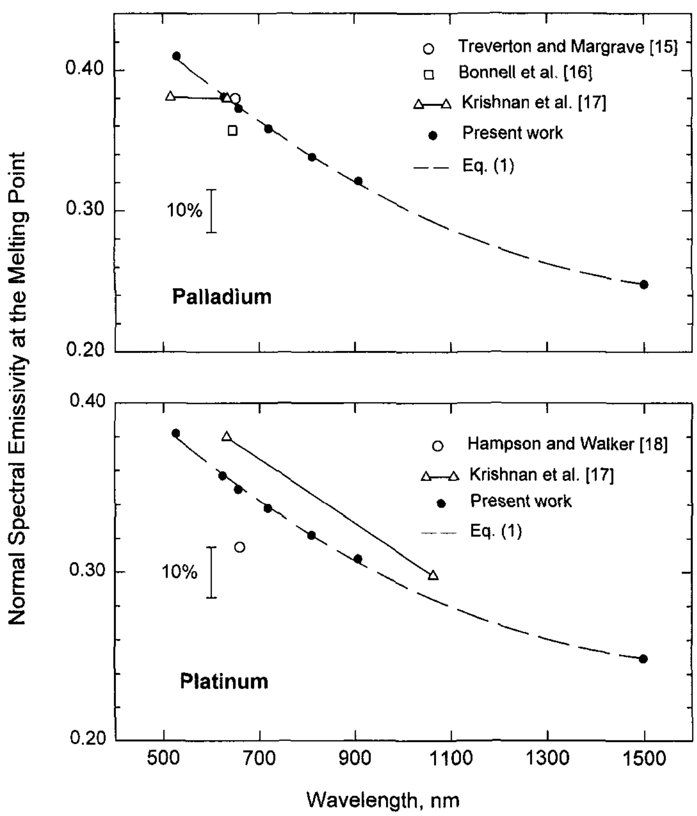

Fig. 3. Variation of the normal spectral emissivity of palladium and platinum at their melting points as a function of wavelength as reported by different investigators. The plotted data correspond to emissivity values adjusted for a common melting temperature: 1826 K for palladium [12] and 2041.3 K [13] for platinum. The dashed line on each graph represents the quadratic function, Eq. (1), fitted to the normal spectral emissivity-versus-wavelength data obtained in the present work.

Table III. Coefficients of Eq. (1) for Radiance Temperature (T_r) and Normal Spectral Emissivity ($\varepsilon_{n,\lambda}$) at the Melting Points for Palladium and Platinum in the Wavelength Range 527 to 1500 nm

	Palladium		Platinum	
Coeff.	T_r (K)	$\varepsilon_{n,\lambda}$	T_r (K)	$\varepsilon_{n,\lambda}$
a	1873.7	0.5899	2079.5	0.5304
b	−0.28237	-4.067×10^{-4}	−0.32445	-3.394×10^{-4}
c	-2.9732×10^{-6}	1.192×10^{-7}	-1.5886×10^{-5}	1.013×10^{-7}
SD[a]	0.53	0.002	0.28	0.002

[a] Standard deviation of an individual point from the curve represented by Eq. (1).

The present results for normal spectral emissivity at the melting point decrease with increasing wavelength for both palladium and platinum. For palladium, the emissivity decrease is about 40% over the entire wavelength range of the experiment, 530 to 1500 nm, and for platinum the decrease is about 30%. This decrease in the normal spectral emissivity at the melting point as a function of increasing wavelength is consistent with the results reported by Cezairliyan et al. [19] in a literature review of the wavelength dependence of the emissivity of selected high-temperature metals at their melting points.

The dashed curves in Figs. 2 and 3 represent quadratic functions fitted to the present results using the least-squares method. The functions are of the form

$$y = a + b\lambda + c\lambda^2 \tag{1}$$

where y is either radiance temperature, T_r, in K, or normal spectral emissivity, $\varepsilon_{n,\lambda}$, and λ is wavelength in nm. The values for these coefficients are given in Table III.

6. CONCLUSIONS

The present work on palladium and platinum has shown that pulse-heating experiments yield highly reproducible results (within ± 1 K) for the melting-point radiance temperatures at seven wavelengths in the range 527 to 1500 nm. These results are similar to those found for many other metals [1] and contribute additional data to suggest that the radiance temperature of selected metals at their melting points can be used as easily realizable temperature standards for high-temperature optical pyrometry.

The melting behavior of these materials, especially platinum, with their very flat plateau regions (illustrated in Fig. 1) and their highly reproducible results, identify them as suitable candidates for the establishment of secondary temperature references based on melting-point radiance temperatures.

As mentioned in Section 1, this work on palladium and platinum essentially completes our program to measure the radiance temperatures at the melting point of high-temperature transition metals (melting points above 1700 K) in the wavelength range 500 to 1500 nm. Over the last two decades, we have performed radiance temperature measurements on 15 metals with melting points in the temperature range 1729 K (Ni) to 3693 K (W). Only chromium remains as a possible material to be studied. It has not been studied because its high vapor pressure at the melting point necessitates the use of a pressure vessel. Other metals not studied, in particular, the platinum group metals, ruthenium, osmium, and rhodium, are very rare, and, due to cost, are not likely candidates for secondary temperature references, while the metal manganese has a melting temperature that is probably too low for optical pyrometry. Future work in this area will concentrate on confirming the results of past work and selecting the materials and proper procedures for using them as reference values for secondary calibrations in optical pyrometry.

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