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Calibration of Platinum Resistance Thermometers Using an Intercomparison Scheme

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1.0 Introduction

The International Practical Temperature Scale of 1968 (IPTS 68) is based on a number of defining fixed points, and the standard platinum resistance thermometer (SPRT) is used as an interpolation instrument between these fixed points. From 0 °C to 630.74 °C, these SPRT(s) are calibrated at the National Bureau of Standards using the triple point of water (0.01 °C), the tin-point (231.9681 °C), and the zinc-point (419.58 °C). A thorough discussion of platinum resistance thermometry and IPTS 68 is given in NBS Monograph 126 [1].*

Our current hydrostatic weighing experiment procedures (e.g., see [2]) require that we relate working temperatures (10 - 25 °C) to IPTS 68 with an uncertainty no larger than a few millidegrees Celsius. This requirement is not stringent and is easily obtainable with an SPRT calibrated in the fixed point manner. However, in density measurements made by hydrostatic weighing the long stem SPRT is not practical because of its excessive length and sheath fragility. The commercially available capsule type PRT(s) with flexible leads are most useful in this application, but when sealed for direct immersion in water and other liquids, it can no longer be calibrated over the extreme temperature range of the fixed points.

We have managed this problem by potting the electrical leads exit area of the capsule thermometer in epoxy suitable for direct immersion in our working liquids and the development of an appropriate calibration procedure that relates the thermometer to IPTS 68.

The calibration procedure reported herein we refer to as a wire-to-wire calibration. That is, the platinum resistor of one or more SPRT(s) calibrated by the fixed point method are used as standards to calibrate one or more platinum resistance thermometers (PRT(s)) whose properties of interest are unknown. This comparison at several temperatures results in the calibration of the PRT(s).

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* Numbers in brackets refer to similarly numbered references at the end of this paper.

2.0 Calibration Setup

SPRT(s) calibrated in accordance with IPTS 68 and for use in range of 0 - 630.74 °C are assigned constants alpha (α) and delta (δ). Also determined, but not considered a constant, is the value of resistance at 0 °C called R(0) in absolute ohms. To meet IPTS 68 requirements alpha for any given SPRT must not be less than 0.003925.

To assign IPTS 68 temperatures based on an SPRT, the resistance R(t) at temperature t must also be measured. Likewise, for us to calibrate any PRT by the wire-to-wire method, we must measure R(0) and a sufficient number of selected R(t)s for a meaningful determination of alpha and delta.

The essential elements of our calibration setup are a triple point of water cell, a means of controlling temperature, a bridge for measuring resistance and a switch to expeditiously exchange thermometers in the electrical circuit. A layout of the calibration setup is shown in figure 1.

2.1 Triple Point of Water Cell

One of the defining points of IPTS 68 is the triple point of water (0.01 °C). The care and use of the cell is thoroughly described in literature [1]. It has been used here to measure thermometer resistance at the triple point R(tp) from which R(0) can be calculated. This calibration is discussed in section 3.1.

2.2 Controlled Temperature Bath

The controlled temperature bath is a commercially available unit and requires only minor modification for this calibration procedure. The water bath portion of this unit shares a common chassis with the proportional electronic temperature controller. The bath capacity is about 20 liters with cube-like dimensions.

The range of temperature control is stated to be from -20 to 70 °C, a range far greater than our needs. Our experience shows the bath stability to be about 0.005 °C per hour over a range of 5 - 35 °C. The performance beyond this range is unknown.

Initially the thermostat bath was supplied with a cover plate to enclose the free thermally controlled water surface. However, this left a 10 cm air space above the maximum fill level after filling and for this reason the cover was abandoned.

We supplied a copper cover plate suspended in contact with the free water surface. All of the thermometer leads were clamped to this plate by means of plastic tape to minimize thermal emf's and to provide thermal lagging.

A hole was cut through the plate just large enough to receive a glass tube which contained the thermometers for thermal soaking at various selected temperatures. Contained inside the glass tube is an aluminum cylinder (thermal mass) with six holes, one for each thermometer, in close proximity to each other.

Once the thermometers were inserted into the tube and cylinder, a fluorocarbon liquid with good heat dissipation properties was added to insure good thermal contact. The fluorocarbon level was kept slightly below the level of thermostated water surrounding the outside walls of the glass tube. The area above the fluorocarbon was packed with cotton in and around the protruding thermometer stems and lead wires.

The entire area above the copper plate including thermometer stems and lead wires was completely covered with styrofoam pellets for thermal insulation from the surrounding air. In addition, a black photographic cloth was placed above the thermometer stems that protruded to prevent thermal effects from light piping and stray light.

2.3 Resistance Measuring Bridge

The values of R(t) for each of our thermometers at the various temperatures were determined by a Mueller-type bridge (calibrated twice in the last ten years). Detailed instructions for the use of this bridge with SPRT(s) are set forth in reference [1]. This particular bridge is thermostated for stability and the smallest resistance increment is 0.0001 ohms (without interpolation). It should also be noted that the bridge had been very carefully calibrated.

Provisions are made for an external null detector and a wire suspension type galvanometer was used. The galvanometer optical path was increased from 1 to 2 meters by the insertion of an additional lens in the light path. This merely provided more sensitivity making small displacements more visible to the eye.

2.4 Switch

Normally a switch is not used in precise laboratory thermometry. In this experiment, however, it is desirable to switch from one thermometer to another rapidly when taking data. Any switch used in this application will be more convenient with a low and stable contact resistance.

Such a switch was recommended to us by the Electricity Division of NBS. This type of switch is commercially available for use as a high quality instrument switch. The contact resistance is specified to be less than 0.001 ohm and stable to 0.0005 ohm and emfs less than 1 microvolt. There were more switch positions and poles available than used.

We had no problem with switch stability over the very short time span needed for an R(t) measurement, approximately 1 minute. (This does not eliminate thermal soaking the PRT(s) for hours.) The switch was mounted to a heavy brass plate and the leads were clamped to it to minimize thermal emfs. The entire assembly was placed in a glass fiber insulated box to maintain quiescent thermal conditions.

3.0 Wire-to-Wire Calibration

3.1 Calibration Procedure

Two standards, SPRT 1 and 2, and four test thermometers, PRT 3, 4, 5, and 6, are used in this experiment. SPRT 1 has a long history of stability and is used as the primary standard. SPRT 2 was intended to be used as a check standard, i.e. a thermometer the calibrated values of which, derived from this experiment, can be checked against its fixed point calibrations. Unfortunately, we noted a shift in the triple point value of SPRT 2 from its original value. Subsequently, SPRT 2 was recalibrated by our Thermometry Section as it was used and again after annealng.

Resistance for the triple point of water was determined twice for each thermometer, each run consisting of readings in the normal-reverse-normal positions*, using a reversing switch in the battery circuit. The average of the two normal readings is averaged with the reverse reading and the 12 resistance values are given under column OHMS in table 1. The R(0) values are extrapolated from the resistance at triple point by subtracting 0.00101 and are given in the next column.

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* A stable switch and temperature bath make it possible to shorten the NRRN measurement order to NRN not possible in a fast drifting experiment.

Next, the six thermometers were inserted in the constant temperature bath and resistance readings were taken in accordance with a designed schedule for each of 9 temperatures nominally 10, 15, 19, 24, 29, 34, 24, 5 and 15 °C. Each reading, as was in the case of triple point, is the average of the normal-reverse-normal readings. Typical data are given in table 2 for temperature nominally 15 °C.

For each temperature, a sequence of 24 resistance measurements were made in the following time sequence, with every two measurements considered to be within the same time period:

	TIME PERIOD	THERMOMETERS
Set 1	1	3 - 5
	2	1 - 3
	3	1 - 4
	4	2 - 5
	5	2 - 6
	6	4 - 6
Set 2	7	4 - 6
	8	2 - 4
	9	2 - 3
	10	1 - 6
	11	1 - 5
	12	3 - 5

The sequence in the second set is the reverse of that of the first set and with 3 and 5, and 4 and 6 interchanged. The original intention was to use thermometer 1 as standard in set 1 and thermometer 2 as standard in set 2 and compare the calibrated values resulting from the two sets. Since thermometer 2 changed its triple point value since its last fixed point calibration and its stability was open to question, thermometer 1 was the only one used as a standard. Nonetheless, the design has the advantage that any drift in the constant temperature bath over the period of measurements can be detected and compensated for, if necessary. For other designs see for examples NBS Technical Note 844 [4].

3.2 Adjustment for Trend in Constant Temperature Bath

From the 24 measured resistance values for each temperature, estimates of the resistance value for each thermometer were obtained by fitting the following equation to the data, using the method of least squares. The equation takes the form:

$$y_{ij} = R + t_i + \varepsilon_{ij}$$

where y_{ij} : resistance reading of the ith thermometer on the jth occasion (order)

R : mean of all resistance readings

t_i : effect of the ith thermometer, and

ε_{ij} : random error of measurement with mean zero and variance σ^2.

The OMNITAB command FIT [3] was used for the calculation with the constraint $\Sigma t_i = 0$, i.e. the effect of each thermometer is measured from the average of all thermometers. The result using this model was, however, unsatisfactory. A plot of the residuals (observed value - fitted value) against the order in which the measurements were taken showed clearly trends in temperature over the time period required for taking the 24 measurements. This trend was more pronounced in some temperatures than others. A plot of these residuals at nominally 15 °C is shown in figure 2. Standard deviations computed from those fits for the nine bath temperatures varied from .000044 to .00045 ohms.

Next the model was modified to include the time order effect using:

$$y_{ij} = R + t_i + 0_j + \varepsilon_{ij}$$

where the additional term 0 represents the jth order effect, j = 1, 2, ..., 12, with constraint $\Sigma 0 = 0$. The resulting plot of residuals now exhibit the random pattern desired and the plot is shown in the bottom half of figure 2 for comparison. The adjusted resistance values for the six thermometers and the residual standard deviations are given in table 3 for each temperature. All standard deviations computed from these fits were now reduced to about .00005 ohms.

3.3 Calibration Equations for PRT(s) and Tables

Using the adjusted resistance values of thermometer 1, the t_{68} values of the constant temperature baths were calculated from the fixed point calibration. The temperatures in t_{68} scale were changed to t' scale by the relationship (p. 22 of reference [1]):

$$t_{68} = t' + M(t')$$

with the M(t') values interpolated from Appendix D of the same publication. Table 4 gives the t_{68} , M(t'), and t' values for the 9 constant temperature baths. The t' values are needed for the reason that the relationship

$$R(t')/R(0) = 1 + At' + Bt'^2$$

holds for t', but not for t_{68}. Least squares estimates of A and B were computed for each thermometer using the measured R(t') and R(0) values for the more general equation

$$\frac{R(t')}{R(0)} = W(0) + At' + Bt'^2 \qquad (1)$$

Then α and δ can be calculated through the relationship:

$$\alpha = A + 100B$$

$$\delta = -10^{-4}B/(A + 100B)$$

and the use of the thermometer can proceed in the manner as described in section 6.1 of reference [1]. The value of W(0) is very near to unity (difference< .0005 °C) and can be corrected if deemed necessary. We note that α and δ obtained here have the same meaning and can be used in the same manner as those given in reference [1].

Alternately, we found that the equation

$$t' = a + b\left[\frac{R(t)}{R(0)}\right] + c\left[\frac{R(t)}{R(0)}\right]^2 \qquad (2)$$

can be fitted to the data to give adequate t' values given R(t)/R(0). To check this, a table of values of R(t)/R(0) versus value of t for integral values of t at one-degree intervals was generated by the following procedure:

(1) Fit equation (1) using calibration data to obtain A and B. Fit equation (2) using calibration data to obtain a, b, and c.

(2) Using t' values corresponding to t_{68} from 0° to 35 °C at 1 degree intervals, compute the corresponding ratio R(t)/R(0) using equation (1).

(3) Using the ratio R(t)/R(0) thus calculated, compute t' values using equation (2) with a, b, c estimated from the data.

The largest bias noted between t' entered and t' computed was of the order of .0002 °C at about 0 °C. Table 5 gives t' (entered), R(t)/R(0) values, and t' (computed). Apparently the discrepancy between the use of equation (2) and the solution of the quadratic equation (1) is tolerable.

4.0 Uncertainty of Calibrated Temperature Values

The following possible sources of errors are discussed separately below, based on data from this experiment and on knowledge of the histories of the two standard platinum resistance thermometers.

(a) Method of Computation. Since the t_{68} and t' temperatures used in the constant temperature baths were based on that of SPRT #1, adjusted for trend, the resulting adequacy of fit of R(t)/R(0) values to equation (1) for SPRT #1 would indicate first, the accuracy of the computation, and secondly, the adequacy of the adjustments. The standard deviation computed from the fit of the nine values to equation (1) was 2.6×10^{-8} in R(t)/R(0), giving assurance that rounding error in computation was negligible and the adjusted values did agree with values obtained from fixed point calibrations. SPRT #1 was annealed and recalibrated by fixed point method after our experiment and no change was detected.

(b) Bias in Fitting t' as a Quadratic of R(t)/R(0). This reverse fitting procedure by equation (2) is an approximation to solving a quadratic equation in t' for given t' over the range 0-35 °C, and as such introduces certain biases in the results. Comparison of t' and t_{68} values confirmed the existence of this bias, although negligible in the use of these PRT(s). For SPRT #1, the maximum bias was +.00023 °C at 0 °C, and changed signs at about 8 °C, 21 °C and 31 °C. These biases were noted on all PRT(s) with the same signs and about the same magnitudes.

(c) Failure of the Check Standard. SPRT #2, which was intended as a check standard, changed its R(0) value considerably between fixed point calibrations before and after our experiment. The R(0) value measured during our experiment was 25.45447, measured after the experiment by our Temperature Section was 25.4541, and after annealing was 25.4536. The standard deviations computed from the residuals of the fit to equations (1) and (2) were also largest among the six PRT(s), and there was also a bias in the calibrated values.

(d) Uncertainty of the Calibrated Capsule PRT(s) #3, #4, #5, and #6. Based on the results obtained for SPRT #1, we are of the opinion that the calibrations of the capsule type thermometers by this procedure are of sufficient precision for our present work. We have computed that the standard deviation of a predicted value of t, given the ratio R(t)/R(0), is less than 0.00035 °C between 5-30 °C, and hence a 3-sigma limit may be taken as 0.001 °C. Below 5 °C and above 30 °C, the standard deviations and 3-sigma limits would be about double this magnitude, or 0.002 °C. The uncertainty in the fixed point calibration of SPRT #1 is taken to be 0.0005 °C, interpolated from figure 17 of reference [1], and based on histories of several such calibrations of this stable SPRT. The uncertainty of the calibration, in the range of 5-30 °C, is therefore 0.0015 °C.

(e) Errors in Using the Calibrated PRT(s). In using these PRT(s), an R(t)/R(0) value is measured. The measurement error in R(t)/R(0) values is in addition to the uncertainty of the calibrated values. In our case, for example, the standard deviation of a measured value of R(t)/R(0) (average of four in the design) is calculated to be 0.000003, equivalent to 0.0007 °C. A 3-sigma limit on this source of error is therefore 0.002 °C.

5.0 Summary

In this experiment we have demonstrated the feasibility of a wire-to-wire calibration procedure using one or more standard platinum resistance thermometers as standard. Within the range of temperature of 0 - 35 °C, the uncertainty of the calibration, apart from the uncertainty of the reference standard, is estimated to be less than 2 millidegrees.

* *

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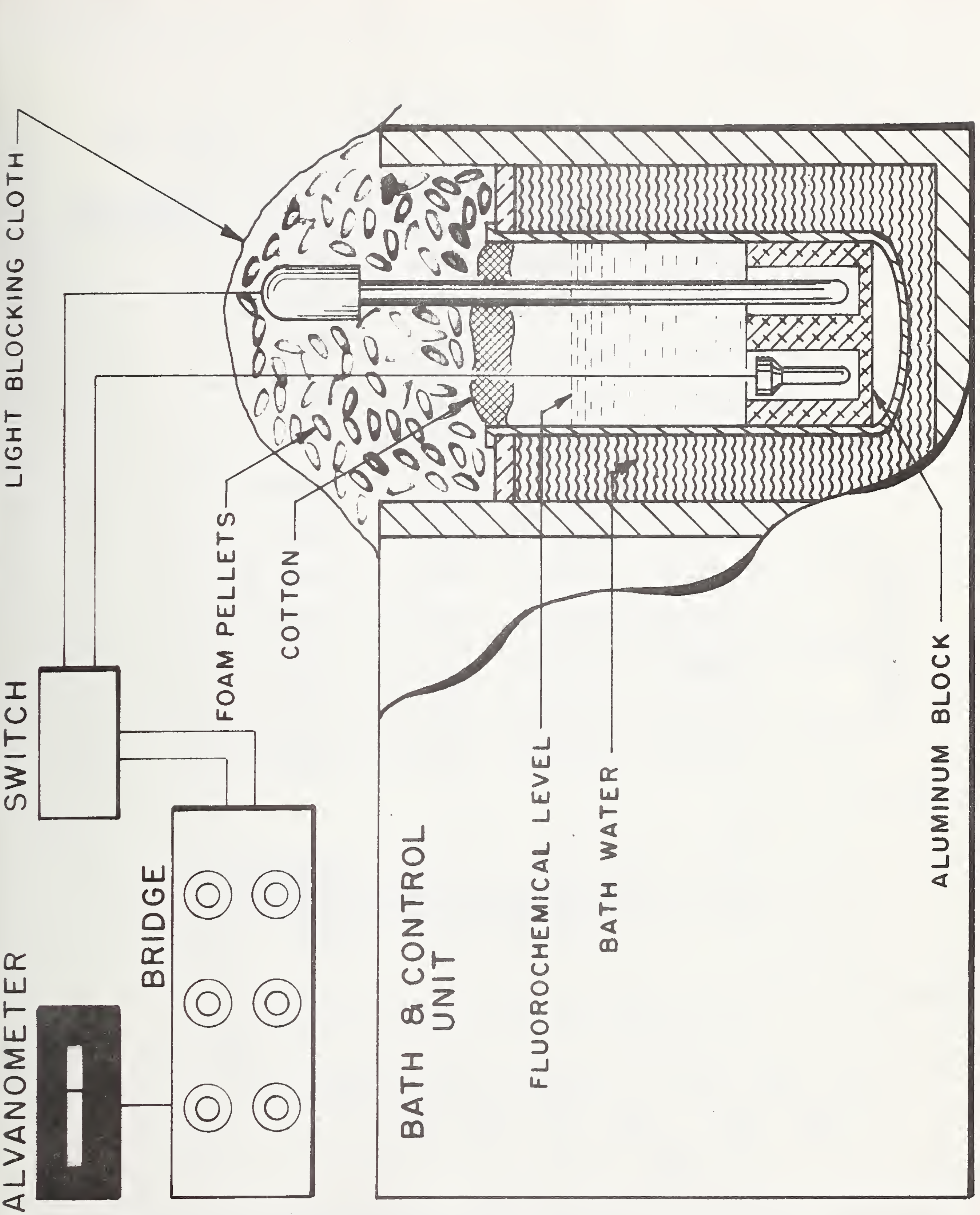

FIGURE 1: SKETCH OF CALIBRATION SETUP

FIGURE 2: Plot of residuals illustrating behavior of bath at 15 °C during 50 minute interval required for the 24 observation series.

Data shown before and after adjustment for trend.

	Resistance Measurements				
Thermometer	Normal	Reverse	Normal	R(tp)	R(0)
1	25.47277	25.47077	25.47277	25.47177	25.47076
1	25.47277	25.47077	25.47277	25.47177	
2	25.45538	25.45558	25.45538	25.45548	25.45447
2	25.45538	25.45558	25.45538	25.45548	
3	25.55339	25.60209	25.55339	25.57774	25.57676
3	25.55339	25.60209	25.55349	25.57777	
4	25.59119	25.57608	25.59119	25.58363	25.58260
4	25.59109	25.57608	25.59109	25.58358	
5	25.62159	25.60890	25.62149	25.61522	25.61424
5	25.62139	25.60910	25.62149	25.61527	
6	25.54780	25.55529	25.54760	25.55150	25.55048
6	25.54770	25.55529	25.54760	25.55147	

TABLE 1: Resistance measurements at triple point of water and 0 °C for the six thermometers.

	Resistance Measurements			
Thermometer	Normal	Reverse	Normal	R(15)
3	27.11835	27.16194	27.11845	27.14017
5	27.17874	27.17874	27.17884	27.17876
1	27.02593	27.03093	27.02613	27.02848
3	27.11885	27.16234	27.11905	27.14064
1	27.02634	27.03123	27.02644	27.02881
4	27.14364	27.14964	27.14374	27.14667
2	27.01143	27.01113	27.01153	27.01130
5	27.17974	27.17964	27.17984	27.17971
2	27.01153	27.01153	27.01173	27.01158
6	27.11094	27.11524	27.11104	27.11311
4	27.14404	27.14994	27.14434	27.14707
6	27.11094	27.11534	27.11104	27.11317
4	27.14414	27.15004	27.14434	27.14714
6	27.11104	27.11534	27.11114	27.11322
2	27.01163	27.01153	27.01183	27.01163
4	27.14414	27.15004	27.14424	27.14711
2	27.01183	27.01163	27.01203	27.01178
3	27.11994	27.16334	27.11994	27.14164
1	27.02724	27.03203	27.02724	27.02964
6	27.11114	27.11554	27.11114	27.11334
1	27.02714	27.03193	27.02714	27.02954
5	27.18014	27.18004	27.18014	27.18009
3	27.11984	27.16324	27.11984	27.14154
5	27.18014	27.18004	27.18014	27.18009

TABLE 2: Resistance measurements at temperature nominally 15 °C for the six thermometers.

Nominal Temperature of Series °C	Adjusted R(t)						Residual Standard Deviation
	Thermometer #1	Thermometer #2	Thermometer #3	Thermometer #4	Thermometer #5	Thermometer #6	
10	26.535733	26.518510	26.645948	26.651595	26.684005	26.618263	4.4×10^{-5}
15	27.029173	27.011420	27.141273	27.146919	27.179810	27.112960	5.4
19	27.464060	27.445855	27.577759	27.583499	27.616521	27.548921	4.0
24	27.922975	27.904653	28.038506	28.044212	28.077617	28.009115	3.6
29	28.440909	28.422237	28.558515	28.564335	28.598047	28.528521	6.3
34	28.914267	28.895221	29.033716	29.039457	29.073532	29.003111	2.5
24	27.922547	27.904157	28.038041	28.043672	28.077254	28.008642	7.7
5	26.074660	26.057650	26.182965	26.188640	26.220705	26.155810	4.7
15	27.029803	27.012183	27.141961	27.147534	27.180449	27.113586	4.0
R(o)	25.47076	25.45447	25.57676	25.58260	25.61424	25.55048	—

Table 3. Adjusted resistance values and residual standard deviations for the intercomparison of six thermometers at nine bath temperatures.

R(t)/R(0)	t_{68}	M(t')	t'
1.0418116	10.50307	0.004054	10.50712
1.0611844	15.38107	0.005531	15.38657
1.0782584	19.68631	0.006568	19.69288
1.0962757	24.23561	0.007485	24.24310
1.1166101	29.37759	0.008277	29.38587
1.1351945	34.08415	0.008780	34.09294
1.0962589	24.23137	0.007484	24.23886
1.0237095	5.95161	0.002459	5.95407
1.0612091	15.38730	0.005505	15.39281

Table 4. Bath temperatures in t_{68} and t' as determined from adjusted values of R(t)/R(0) for thermometer #1.

t_{68}	t' (entered)	R(t)/R(0) (computed using Eq. 1)	t' (computed using Eq. 2)
0	·0000000	·9999966	.0002217
1	1·0004440	1·0039813	1.0006204
2	2·0008750	1·0079647	2.0010117
3	3·0012940	1·0119470	3.0013989
4	4·0017010	1·0159281	4.0017782
5	5·0020950	1·0199079	5.0021448
6	6·0024779	1·0238866	6.0025088
7	7·0028490	1·0278640	7.0028619
8	8·0032079	1·0318402	8.0032074
9	9·0035549	1·0358152	9.0035417
10	10·0038910	1·0397890	10.0038723
11	11·0042149	1·0437616	11.0041907
12	12·0045279	1·0477329	12.0045007
13	13·0048299	1·0517031	13.0047983
14	14·0051210	1·0556720	14.0050929
15	15·0054009	1·0596398	15.0053766
16	16·0056698	1·0636063	16.0056467
17	17·0059278	1·0675716	17.0059090
18	18·0061760	1·0715357	18.0061638
19	19·0064130	1·0754986	19.0064044
20	20·0066388	1·0794604	20.0066385
21	21·0068560	1·0834209	21.0068638
22	22·0070620	1·0873801	22.0070722
23	23·0072579	1·0913382	23.0072732
24	24·0074439	1·0952951	24.0074649
25	25·0076199	1·0992508	25.0076468
26	26·0077870	1·1032053	26.0078149
27	27·0079439	1·1071586	27.0079682
28	28·0080910	1·1111106	28.0081193
29	29·0082288	1·1150615	29.0082517
30	30·0083568	1·1190112	30.0083728
31	31·0084758	1·1229596	31.0084827
32	32·0085859	1·1269069	32.0085845
33	33·0086865	1·1308530	33.0086713
34	34·0037795	1·1347979	34.0087452
35	35·0088630	1·1387415	35.0088115

Table 5. Comparison of t'(entered) and t'(computed) for thermometer #5 using:

(1) $R(t)/R(0) = W(0) + A\ t' + B\ t'^2$, and then

(2) $t' = a + b[R(t)/R(0)] + c[R(t)/R(0)]^2$,

where W(0), A, B, and a, b, and c are calculated from calibration data.

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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.)*

In this report we describe a procedure for the calibration of capsule type platinum resistance thermometers, PRT(s), using one or more standard platinum resistance thermometers, SPRT(s), in the temperature range of 0 - 35 °C. These PRT(s) were designed to be used in density work through hydrostatic weighing where SPRT(s) cannot be used because of space and other limitations, but the procedure is thought to be generally useful in other applications as well.

The schedule of intercomparisons was designed to eliminate possible trends in temperature variations of the constant temperature bath setup. Results of calibration can be expressed either in terms of the two constants alpha (α) and delta (δ), or in a table relating $R(t)/R(0)$ to t_{68} within the range of calibrations.

The uncertainty of the values of t_{68} calibrated by this procedure is believed to be within 2 millidegrees Celsius, not including the uncertainty of the SPRT that is used as standard.

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)*

Adjustment for trend; calibration; capsule type thermometers; intercomparison; IPTS-68; platinum resistance thermometers; wire-to-wire calibration.

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