New Generation of Fixed Points

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Abstract

A new generation of fixed points, mainly for calibration of secondary reference thermometers, reference thermocouples and industrial temperature probes, has been developed. Primary fixed points (PFPs), designed for realization of the ITS (International Temperature Scale), are unsuitable for the secondary level and industrial calibration because of their poor productivity, deep immersion-depth requirement and complexity. The new generation of fixed points requires an immersion of only 230 mm, adequate for most secondary reference and industrial temperature probes. They provide high efficiency; a great number of probes can be calibrated in a working day. The realization of the new generation of fixed points and the cell design is described. The comparison data between the new fixed points and PFPs are provided.

Introduction

Fixed points are an important part of the ITS. Up to now, most fixed-point cells and related equipment have been designed for calibration of primary temperature standards such as standard platinum resistance thermometers (SPRTs). Fixed-point cells provide very high accuracy and no reference thermometers are required. These cells and equipment, however, have very poor productivity. Only a few thermometers can be calibrated in a working day. They are also difficult to operate and usually require a deep immersion depth (about 460 mm). All of these reasons make them unsuitable for secondary level and industrial calibration.

Some techniques we developed in recent years(1-4) have made it possible to advance a new generation of fixed points mainly for calibration of secondary-level reference temperature probes and industrial probes. This new generation of fixed points will satisfy the following requirements:

- Efficiency. A large number of temperature probes can be calibrated by a technician in a working day.

- Accuracy. The new fixed points will provide an accuracy level which satisfies all calibrations for secondary level reference platinum resistance thermometers (secondary PRTs), reference thermocouples (type S, R, B and others), other precise thermometers and all industrial temperature probes. The expanded uncertainties (k=2) of these fixed points should be no more than 2 mK to 4 mK below 420°C, 10 mK at 660°C and 30 mK at about 1000°C.
- Stability. Long-term stability and reliability tests indicate that neither recalibrations nor reference thermometers are needed.
- Ease. These cells are exceptionally simple to use, much like working with an ice point reference.
- Cost. SLFPCs will sell for nearly half the price of primary fixed points.
- Length. Probes as short as 230 mm long (9 inches) can be calibrated in the fixed points.

The new generation of fixed points will be widely used to calibrate secondary level temperature probes and industrial probes. Even though the comparison method of calibrating temperature probes may be used principally, one or more fixed points are desirable for the purpose of checking. A temperature comparison calibration system includes one or more reference thermometers, temperature probes to be calibrated, an electrical measuring instrument and an isothermal temperature source, such as a bath or furnace. If doubt about the comparison calibration results exists, it can be very difficult to identify which component of the system caused the problem. In this case, one or two reliable fixed points could be very helpful.

The Secondary Level Fixed Point Cell

The design and manufacturing process for the secondary level fixed point cell (SLFPC) is similar to that for the primary standard fixed point cell(1,2); the difference is that the dimensions of the new SLFPC are a little smaller than those of the primary cell (Figure 1). The total immersion depth in pure metal (from the bottom of the central reentrant well to the surface of the pure metal) decreases from 195 mm for primary cells to 140 mm for SLFPCs. Therefore, a 230 mm long probe (9 inches) can be calibrated in these cells. The mass of pure metal in a new cell is nearly half that in an old cell allowing the cost for the new cell to be cut nearly in half. The purity of metal used is still 99.9999+% as is that used for the primary standard cell. A less pure metal (99.999%) might be considered in the future in order to cut the cost further if the expanded uncertainty can satisfy the requirements for the SLFPC.

High-purity metal was melted into a graphite crucible in high vacuum or in a pure argon atmosphere with total impurities less than 3 ppm. The graphite crucible was subjected to a high-temperature, high-vacuum treatment just before loading the pure metal. The assembled graphite crucible containing the high-purity metal was then encased in a fused silica cell which was connected to a high-vacuum system. The cell was pumped to a proper pressure at a temperature slightly higher than the melting point for about five hours. During this period, the cell was repeatedly purged with high-purity argon to remove any gases absorbed on to the surfaces of the pure metal, fused silica and graphite parts in the cell. The vapor pressure of some metals, such as zinc, is quite high at a temperature near the melting point. If a zinc cell is pumped to high-vacuum at a temperature above the melting point for a long period of time, a great amount of zinc will be pumped out of the cell. As a result,

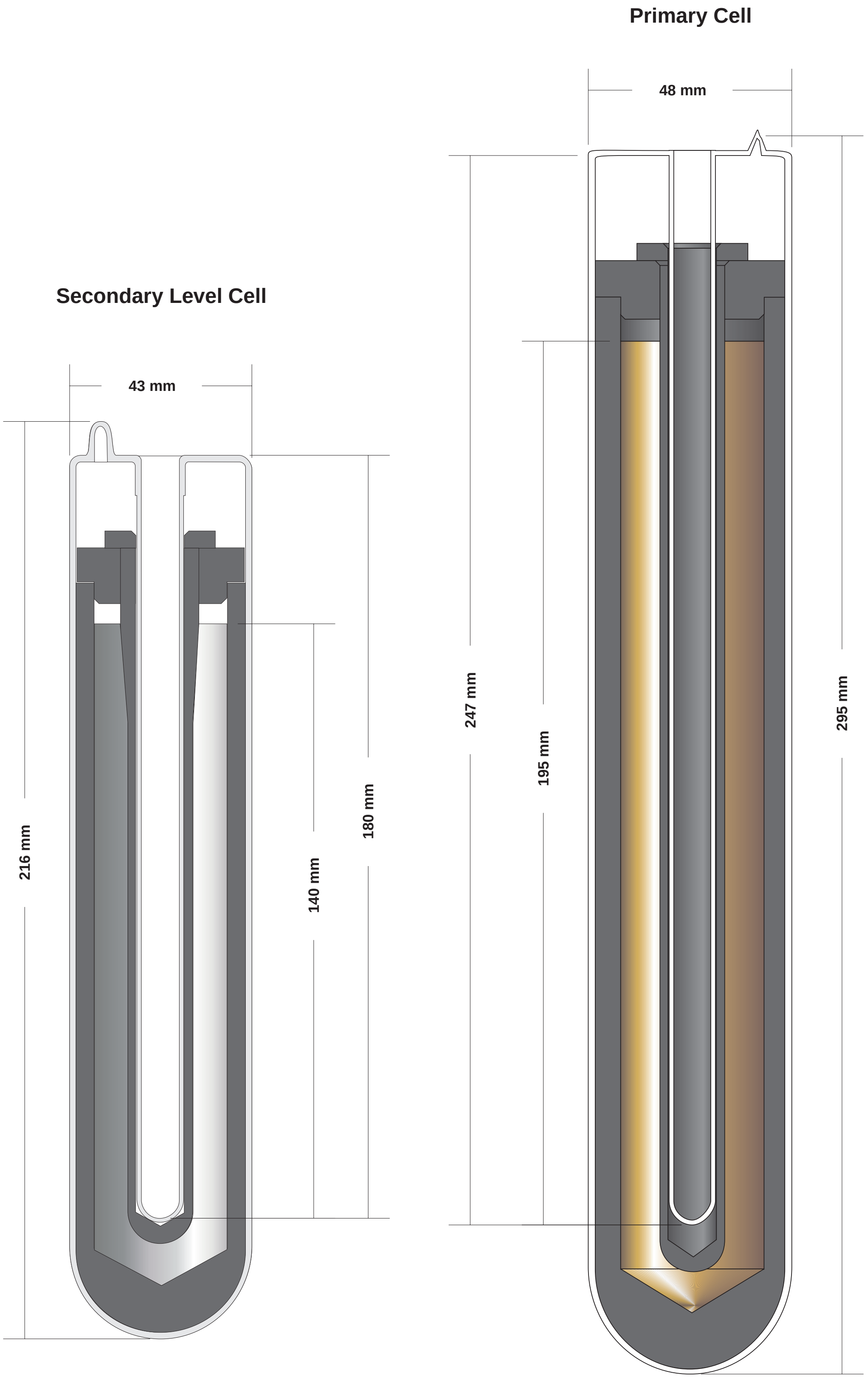

Figure 1. *Secondary level fixed-point cell compared with the primary fixed-point cell*

the pumping procedures must be different for different metals. Finally, the cell was filled with 99.999% pure argon and permanently sealed by using a hydrogen-oxygen torch. The pressure of the argon in the cell at the freezing point was closely adjusted to 101,325 Pa and the practical value of the pressure recorded so that a small temperature correction for pressure difference could be made later.

Realization of the Secondary Level Fixed Points

The secondary level fixed points used to calibrate secondary level probes and industrial probes should be different from the fixed points used to realize the ITS. The highest possible accuracy is the most important consideration for the primary fixed points. On the other hand, ease of realization and higher efficiency are as important as accuracy for the secondary level fixed points.

Results obtained in the laboratory in recent years show that the melting point is much more attractive than the freezing point for the secondary level fixed point [3,4]. The melting points are easier to realize, require less training and make calibration work more efficient. Because it is so easy to obtain a very long melting plateau (typically longer than twenty-four hours) the melting points can be used in a similar manner to the ice point.

Any time you would like to calibrate a temperature probe, you just place the probe into an SLFPC during its melting plateau. The temperature in the cell is known exactly as is the case for the ice point. So a reference thermometer becomes unnecessary and a single measurement is all you need to complete a calibration. You never have to worry about temperature stability as you do in a comparison calibration.

The secondary level fixed points can be realized by using different temperature sources, such as a bath, a fixed point furnace used for calibrating SPRTs or a small portable furnace (see Table 1).

Table 1. *Temperature sources that may be used with SLFPC*

Temperature Source	Minimum length of probe	Accuracy†
Bath with a depth not less than 305 mm (12 inches)	230 mm (9 inches)	Class 2
Fixed point furnace with normal cell holder	460 mm (18 inches)	Class 1
Fixed point furnace with short cell holder	230 mm (9 inches)	Class 2
Small portable fixed point furnace	230 mm (9 inches)	Class 2

†Accuracy Class 1 is the same as that for primary fixed points(2). Accuracy Class 2 is approximately as follows: an expanded uncertainty (k=2) less than 2 mK below 420°C, less than 5 mK at the freezing point of aluminum, 20 mK at the freezing point of silver and 40 mK at the freezing point of copper.

The SLFPC Used in a Traditional Fixed Point Furnace

The SLFPCs can be used in a traditional fixed point furnace. A special cell holder adapting to the dimensions of an SLFPC, or a liner with a holder for the primary fixed point cell

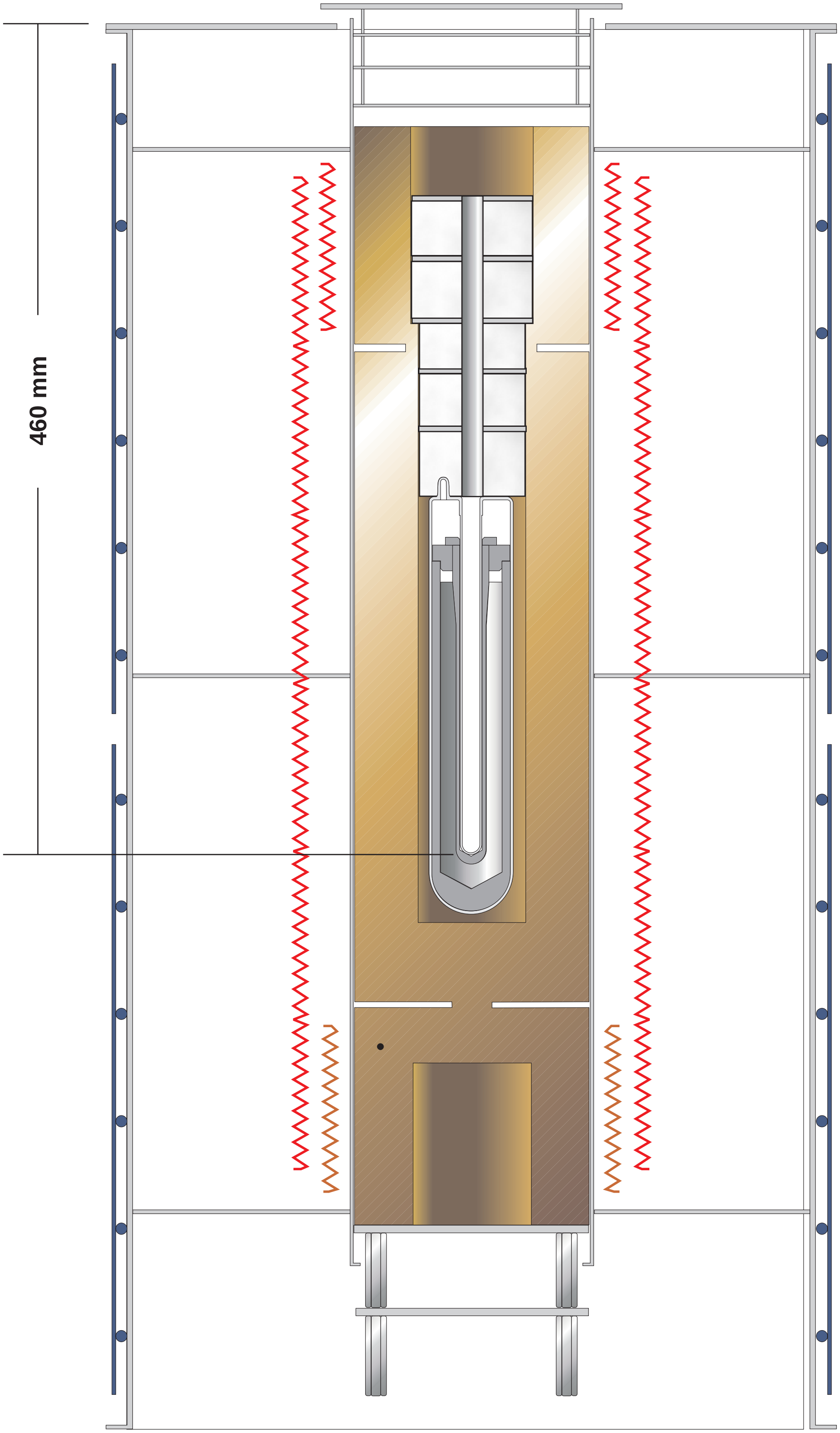

Figure 2. *An SLFPC in a three-zone fixed point furnace*

should be used (Figure 2). Either freezing points or melting points can be realized in this way. The realization procedures can be almost the same as that for the primary fixed points reported in details previously(2,4). Because the same purity (99.9999+ %) of metal is used, the freezing point or melting point should be the same as the primary fixed point version with almost the same uncertainty(2).

The quantity of metal in an SLFPC is only about half that in a primary fixed point cell. The freezing or melting plateau of an SLFPC obtained in this way can still be as long as 24 hours or even longer using an improved technique which we discuss later. A zinc SLFPC and a tin SLFPC were compared with primary fixed point cells and the data are listed in Table 2 and Table 3. The differences among them are within the stability of the SPRT used and the reproducibility of our fixed point measuring system. In other words, no systematic differences between the SLFPC and the primary cell have been detected. Therefore, the SLFPCs can be used to realize the fixed points and to calibrate SPRTs instead of primary fixed point cells. We could call the new cell a small primary fixed point cell. The main purpose of developing the new cell, however, is to calibrate secondary level and industrial temperature probes.

Table 2. *Comparisons among the secondary level zinc cell and primary zinc cells*

S/N of Cell	Type of Cell	W(Zn)	W(Zn) (Compared with Zn08)	t(Zn)	Measured Date
Zn07	Primary	2.56891082	–0.00000172	–0.49 mK	4/25/96
Zn08	Primary	2.56891254	0	0	5/10/96
Zn-s-01	Secondary	2.56891310	0.00000056	0.16 mK	6/28/96

The results were measured by using an SPRT, S/N 5681-5-1016.

Table 3. *Comparisons among the secondary level tin cell and primary tin cells*

S/N of Cell	Type of Cell	W(Sn)	W(Sn) (Compared with Sn09)	t(Sn)	Measured Date
Sn08	Primary	1.89269833	–0.00000034	–0.09 mK	4/18/96
Sn09	Primary	1.89269867	0	0	4/20/96
Sn-s-01	Secondary	1.89269920	0.00000053	0.14 mK	6/20/96

The results were measured by using an SPRT, S/N 5681-5-1027.

Generally speaking, the more high-purity metal in the cell, the larger latent heat of fusion and the longer freezing or melting plateau. The duration of a freezing or a melting plateau also depends on the rate at which heat is transferred to or extracted from the cell. If we can keep the heat transfer rate low, a very long freezing or melting plateau will be obtained. However, at a low heat transfer rate, it is difficult to keep the rate stable during the entire process and uniform around the cell. A stable and uniform heat transfer rate is essential for getting a long, high-quality plateau. By improving the temperature uniformity in the furnace (within about 0.01°C around the cell) and the accuracy of temperature control, we can keep the heat transfer rate not only low, but also stable and uniform around the cell. A 58-hour long melting plateau of tin with an SLFPC was obtained in this way (Figure 3).

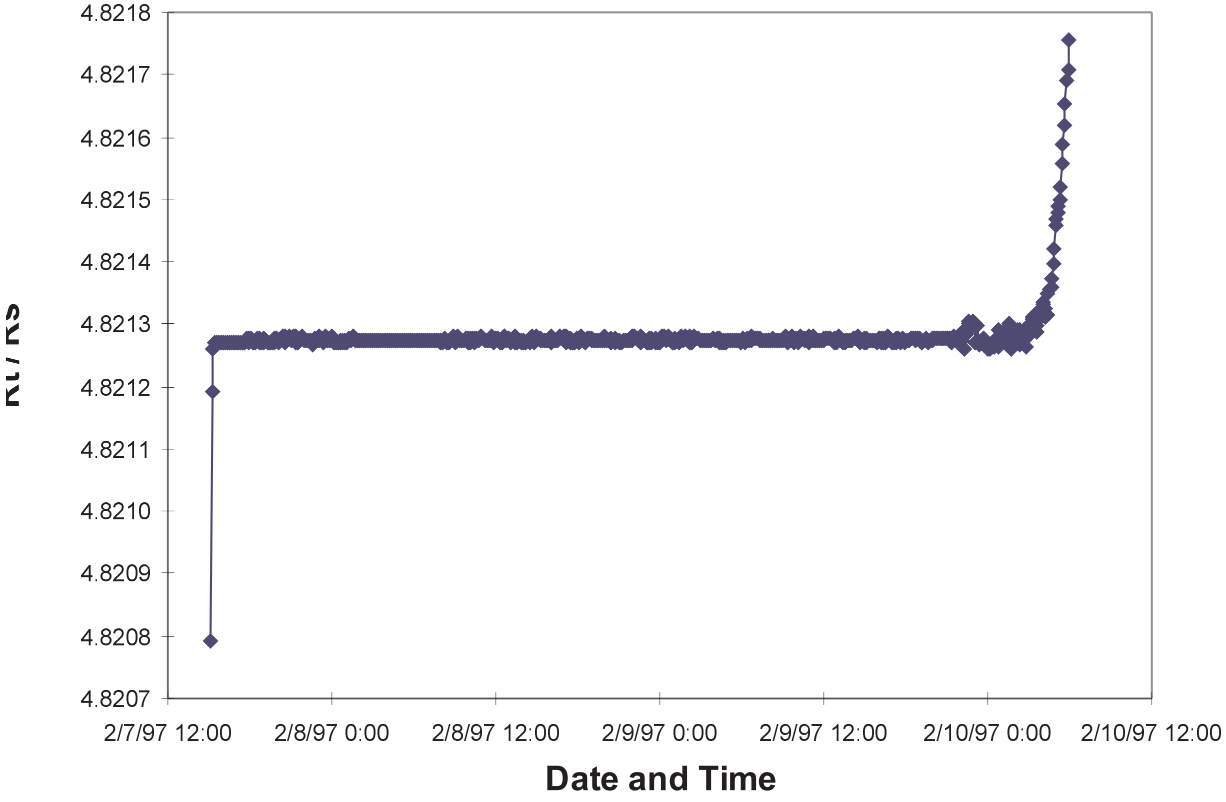

Figure 3. *A typical melting plateau of a tin SLFPC*

The SLFPC Used in a Bath

The melting points (or freezing points) of indium, tin and zinc can be realized in a bath with a depth not less than 304 mm (12 inch), as shown in Figure 4. Immerse the SLFPC with its holder into a bath at a temperature about 1°C below the melting point and wait until equilibrium, or keep the cell at that temperature in the bath overnight. Then raise the bath temperature to about 1.5°C above the melting point and maintain for 10–15 minutes. As soon as the melting starts, set the bath at a stable temperature about 0.2°C above the melting point. A small heater placed in the central well of the cell for four minutes with a power of about 10 watts will melt a thin film of the pure metal around the central well. The inner melting technique described here will improve the melting curve greatly, especially the beginning of the melting curve. Melting curves obtained in this way have lasted for more than 50 hours.

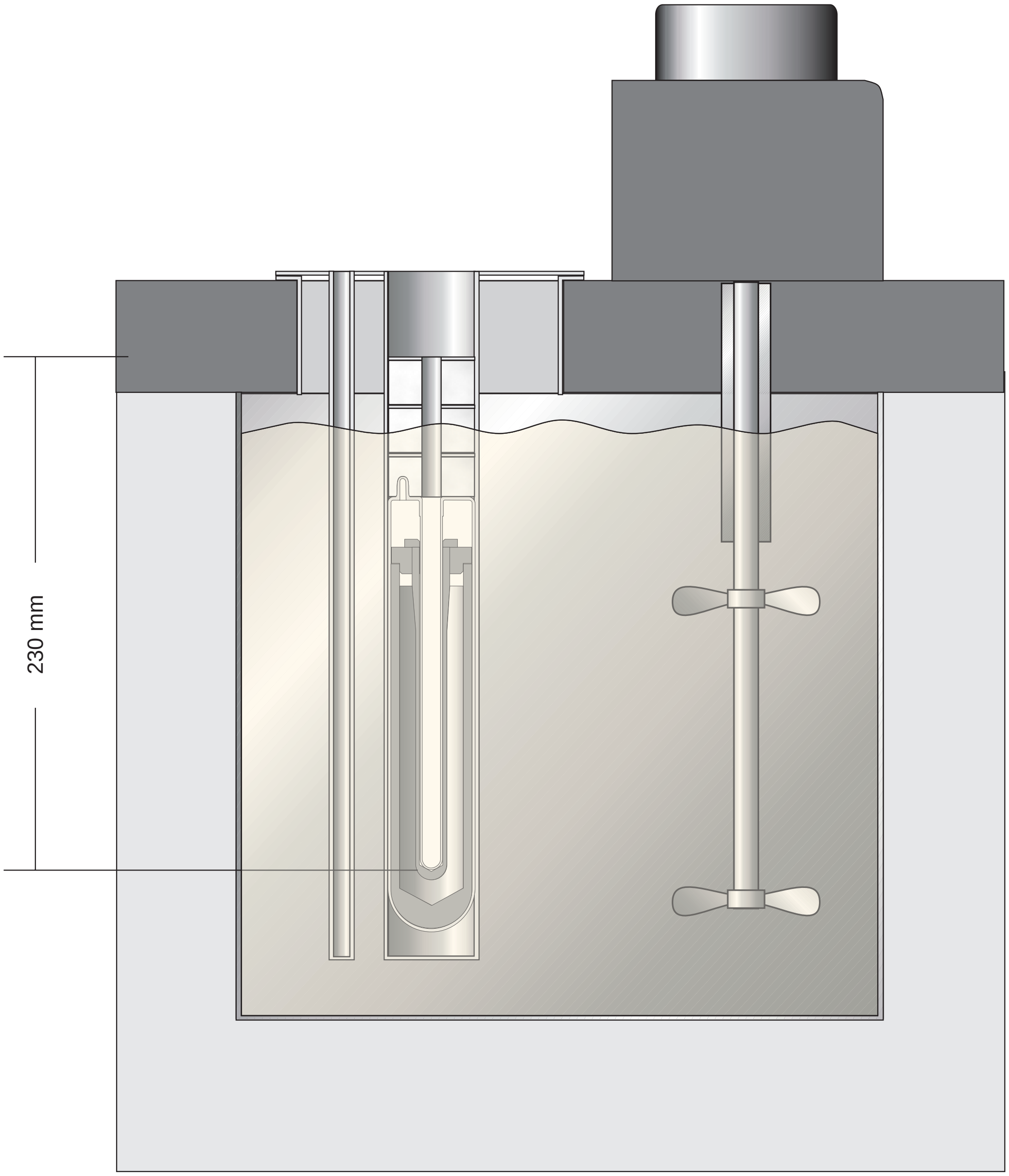

Figure 4. *The SLFPC with its holder in a salt bath*

The temperature at the melting plateau was compared with that of the primary freezing point directly by using five stable SPRTs. In order to minimize the effect of instability of SPRTs on the comparison accuracy, both measurements in the primary freezing point and on the melting plateau of an SLFPC were taken within about an hour. The first measurement was the resistance of an SPRT on the melting plateau of an SLFPC. When the measurement was completed, the SPRT was placed into a primary fixed point cell on a freezing plateau for about twenty minutes to achieve equilibrium. Then resistance of the SPRT was measured again. Finally, the resistance of the SPRT was measured at the triple point of water and the resistance ratio W(Sn) was calculated. The data obtained are listed in Table 4. The temperatures on the melting plateau of a tin SLFPC were found to be 0.00037°C below that in the primary freezing point of tin with a standard deviation of 0.00017°C. The difference was well within 0.001°C. The purity of metal after assembly, resulting in part from the materials and in part from manufacturing procedure, was the same for both kinds of cells. The only difference was immersion depth. The immersion depth into the pure metal was 195 mm (7.7 inches) for the primary cell and 140 mm (5.5 inches) for the SLFPC. The immersion depth into the furnace or the bath was 460 mm (18 inches) for the primary cell and 230 mm (9 inches) for the SLFPC. The measurement difference was mainly caused by thermal conduction along the probe stem in the SLFPC and the bath. The error of about 0.00037°C, however, was so small compared to the allowed expanded uncertainty (0.002°C, k=2) that it can be ignored.

Table 4. *Direct comparison between a tin SLFPC and primary tin cell*

S/N of SPRT	R(Sn)/ohm		W(Sn)			
	SLFPC	Primary cell	SLFPC	Primary cell	W	t/°C
1075	47.5376336	47.5376909	1.89271183	1.89271411	–0.00000228	–0.00061
1077	47.9503688	47.9503862	1.89270484	1.89270552	–0.00000068	–0.00018
1079	47.6738319	47.6738676	1.89270315	1.89270457	–0.00000142	–0.00038
1080	48.2253802	48.2254222	1.89269958	1.89270123	-0.00000165	-0.00044
1081	48.2128290	48.2128529	1.89271011	1.89271105	-0.00000094	-0.00025

The SLFPC with a Shorter Cell Holder Used in a Traditional Fixed Point Furnace

As we mentioned earlier in the paper, an SLFPC can be used with a traditional fixed point furnace, but only very long probes (longer than 460 mm) can be calibrated in this way. A specially designed shorter cell holder allows probes as short as 230 mm (9 inches) to be calibrated in the SLFPC (Figure 5). Many secondary PRT probes were calibrated at the aluminum point in an SLFPC with a short cell holder in a three-zone furnace in the laboratory.

Final Remarks

1. The new secondary level fixed point provides a very long melting plateau (usually longer than 50 hours). Therefore, many temperature probes can be calibrated on a single plateau. This makes calibration work more efficient. If there are a few fixed points at your lab (for example, freezing points of tin, zinc and aluminum for the range from 0°C through 661°C), you can calibrate a probe from one fixed point to another successively. In this way, a great amount of temperature probes can be calibrated over the whole range in a working day.

2. The secondary level fixed point is easy to achieve and requires less training.

3. The SLFPC provides reasonably high calibration accuracy at a low cost and uses very simple methods. The expanded uncertainty (k=2) is well within 0.002°C below 420°C and within 0.005°C at 660°C.

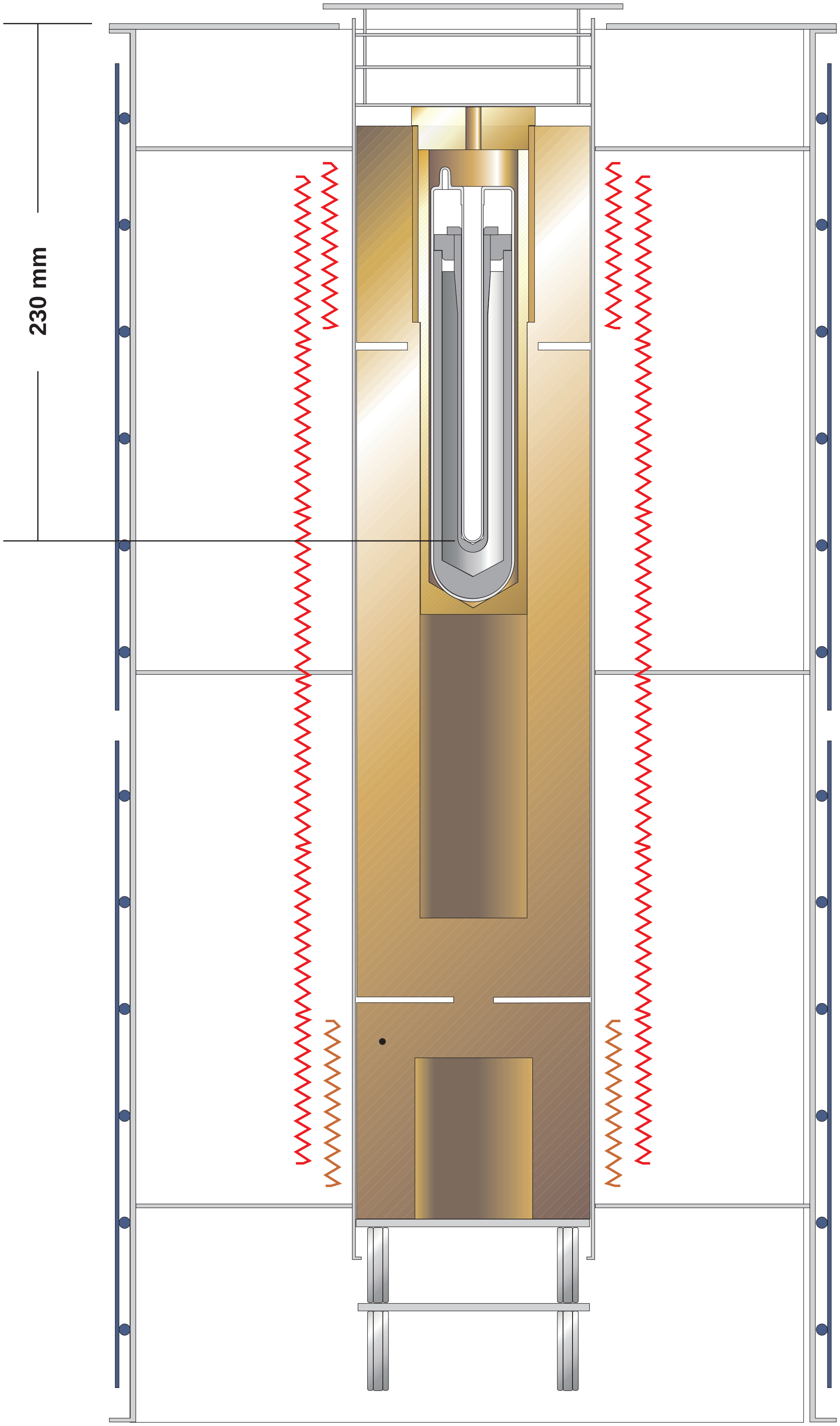

Figure 5. *An arrangement for calibrating a 9-inch probe in an SLFPC with a traditional fixed point furnace*

4. The SLFPC can be used to realize the ITS-90 and to calibrate SPRTs in a traditional fixed point furnace instead of the primary fixed point cell.

5. We plan to cut costs further by using 99.999% purity metal and by simplifying the manufacturing procedure. The expanded uncertainty will rise a little, but will be good enough to calibrate secondary level temperature probes.

Acknowledgments

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