Handheld Fixed Points

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Abstract

Hart Scientific has developed a new generation of fixed points for secondary and field applications, which do not require accompanying heating or cooling apparatus to achieve their realization. Three fixed points have been tested, including the melting point of gallium (29.7646°C), the triple point of diphenyl ether (26.864°C), and the melting point of a gallium/8% tin eutectic alloy (20.476°C). The only thermal source needed to realize melting curves lasting a few hours is the warmth of a person's hand.

Introduction

Fixed-point calibrations play an important role in temperature metrology and have obvious merits: the temperature values of fixed-point devices are known and reference thermometers are not required. Up to now, most fixed points have been designed for calibrating primary temperature standards such as standard platinum resistance thermometers (SPRTs). In recent years, new fixed-point apparatus have been developed for *secondary* level thermometer calibrations [1-3]. Unfortunately, the new apparatus remain overly sophisticated for simple applications. Simpler fixed-point devices are attractive for many such applications, including those that require calibration of devices in the field. Hart has been successful developing handheld-style fixed points, which do not require accompanying heating or cooling devices, which produce plateaus lasting several hours, and that have calibration accuracies better than 0.1°C. Preliminary results obtained by Hart during the past three years are encouraging. Some examples are presented in the paper.

Fixed-Point Cells

Three fixed points in the temperature range from 20°C through 30°C have been tried. They are the melting point of gallium (MPG, 29.7646°C), the triple point of diphenyl ether (TPDE, 26.864°C), and the melting point of a gallium/8% tin eutectic alloy (MPGTE, 20.476°C). Four different designs and sizes of sealed cells have been developed for this purpose. Fig. 1 shows their constructions, and their photos are shown in Fig. 2.

The large volume expansion (3.1%) that occurs with pure gallium upon solidification makes necessary a flexible inner container, constructed of Teflon, to contain the high-purity sample. Teflon is permeable to air and moisture, so a sealed outer container is also necessary to protect the gallium from possible contamination. The “Type A” design uses a Pyrex glass shell, and the “Type B” design uses a stainless steel (SS) outer case (Fig. 1). These two designs are also used for the MPGTE cell. The gallium used is 99.99999% pure, and the tin for the MPGTE cell is 99.9999% pure. The gallium and tin samples were melted into the Teflon crucible in a glove box containing a dry, pure argon atmosphere. The Teflon crucible assembly was then inserted in to the Pyrex outer shell or SS outer case. A lid with a re-entrant well was then joined to the shell, or case, using a special epoxy. The cell was connected to a high vacuum system and pumped to a pressure as low as 10^{-5} Pa to ensure a good vacuum seal. The cell was pumped for about 24 hours and, during this period, the cell was repeatedly purged with 99.999% pure argon. Finally, the cell was filled with pure argon and sealed at a pressure close to the standard atmosphere during a melting plateau.

The TPDE is a useful fixed point [4-5] because its temperature is within typical room temperature range. Unfortunately, diphenyl ether is not available in high purity form, and the purity of the diphenyl ether we obtained from Alfa Aesar is about 99%. The minimum purity of diphenyl ether for a repeatable TPDE is on the order of 99.999%, so it was necessary to further purify the diphenyl ether ourselves. With a method similar to Furukawa’s (as described in his paper [6]), the material was pre-purified by fractional distillation. Further purification of the diphenyl ether was achieved by means of repeated fraction freezing of the molten diphenyl ether sample. The diphenyl ether was cooled slowly so about 75% of the molten sample was frozen during a period of 20 hours. The portion of the sample that remained unfrozen was rejected. After melting the frozen sample, the process was repeated. Five fractional freezing cycles might be necessary to purify the sample to the order of 99.999%. The purified diphenyl ether was put into the “Type C” and “Type D” cells. The cells were then connected to a high-vacuum system and pumped. During the pumping period, the sample was frozen completely and then melted. Pure argon was used to purge the cell from time to time during pumping. The freezing-melting cycle was repeated a few times. Finally, the cell was sealed at the lowest possible pressure when the entire sample had been frozen.

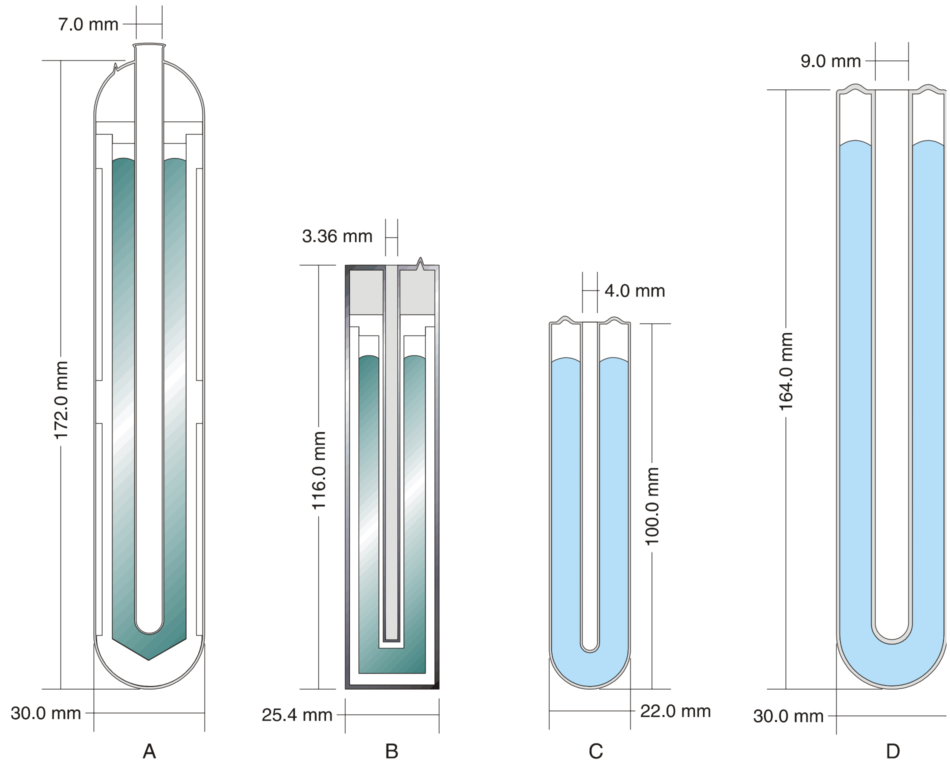

Figure 1: The constructions of four types of sealed cells

Figure 2: Several handheld fixed-point cells

Experimental

Realization of these fixed points is extremely simple. Holding the cell in your hand (or hands) for a few minutes starts the melting. Then the cell is simply placed into a foam package. The plateau will last for a few hours. Typical plateau curves are shown in Fig. 3, Fig. 4, and Fig. 5.

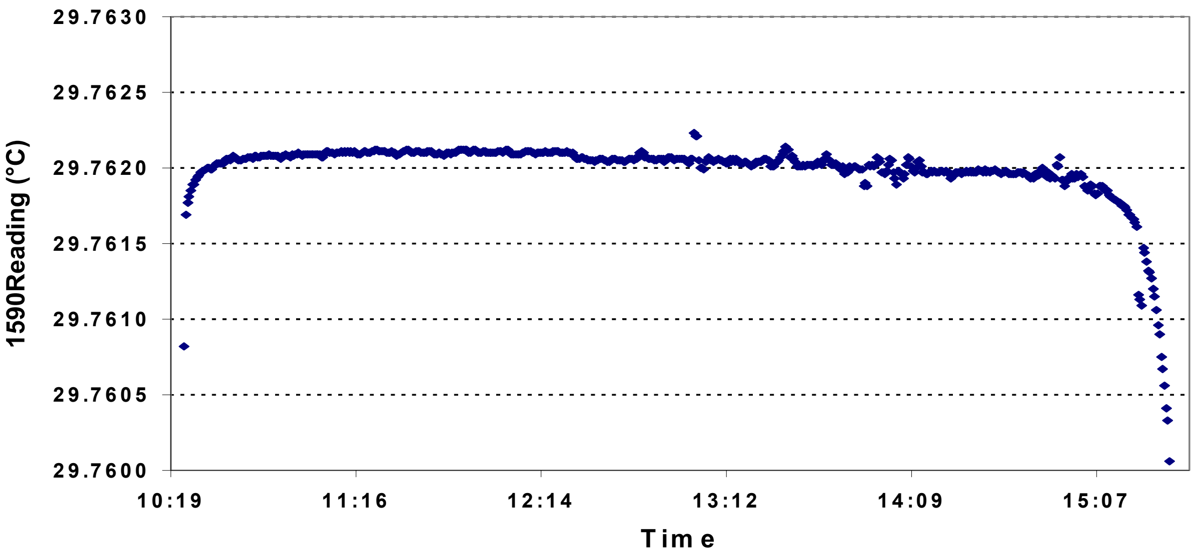

Fig. 3: A typical plateau curve for a handheld gallium cell

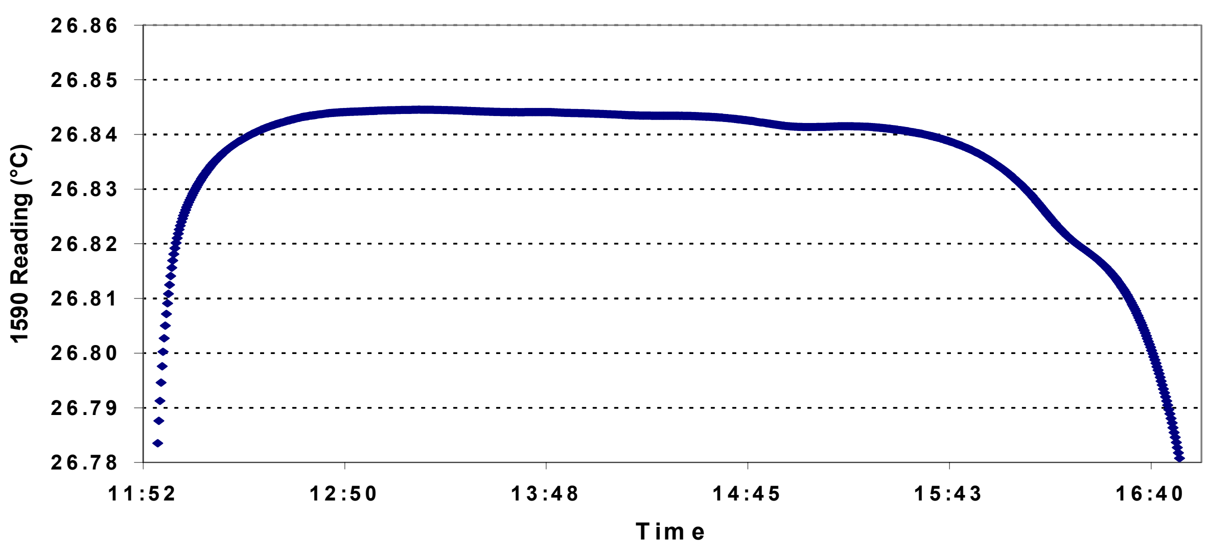

Fig. 4: A typical plateau curve for a handheld diphenyl ether cell

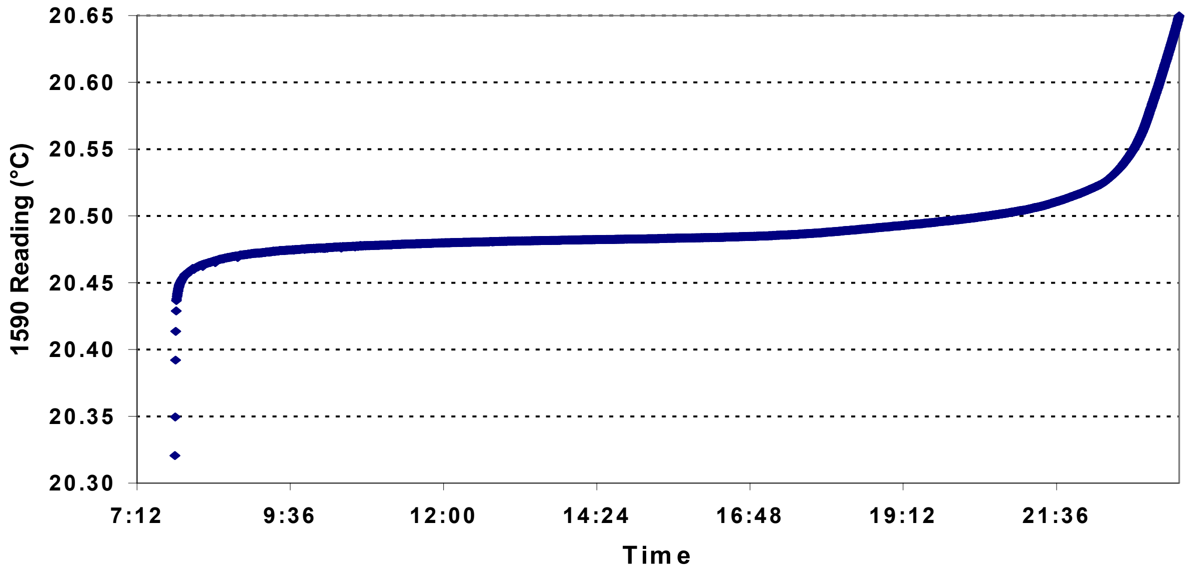

Fig. 5: A typical melting curve for a gallium-tin eutectic alloy cell

The MPGTE is usually below room temperature, so it is unnecessary to use "hand heat" to start a melting curve. The MPGSE cell is kept in a refrigerator overnight to thoroughly freeze the alloy in the cell. The cell is then taken out of the refrigerator and placed into a foam package. The melting curve starts within about thirty minutes.

The reproducibility of the TPDE using a small Pyrex cell (Fig. 1, Type C) was investigated in detail. It was found that a minimum holding period was required to obtain a reproducible equilibrium temperature. The minimum holding period changes with the surrounding temperature and also depends on the person. The lower the surrounding temperature, the longer the minimum holding period. If the holding period is too short, the equilibrium temperature may be lower. Different types and different sizes of cells might also require different minimum holding periods. The minimum holding period for each situation must be determined by testing. At Hart, the minimum holding period for the small TPDE cell was found to be about six minutes for an typical man at a room temperature of about 23°C. When the cell was held in the hand for six minutes, the temperature in the re-entrant well reached the equilibrium temperature in about 30 minutes and the temperature was stable (within ± 0.002°C) for about two hours. A Hart Scientific Model 1590 Super-Thermometer and a thermistor (S/N: 604011, Model: 5610BT) were used for the measurements. The average of all data within ± 0.002°C in a plateau curve was used as the mean value for the plateau. Seven independent realizations were performed. Table 1 lists the data obtained. The standard deviation from one realization to another was 0.0011°C.

Table 1: Reproducibility of the TPDE realized by using a handheld Type C cell

No.	Mean Equilibrium Temperature (°C)
1	26.8485
2	26.8489
3	26.8493
4	26.8504
5	26.8482
6	26.8501
7	26.8511
Average	26.84950
Standard Deviation	0.0011

A handheld gallium cell (s/n #73001; Type B in Fig. 1) was compared against a traditional gallium cell (s/n #Ga03027). The traditional cell was maintained in a bath at a temperature approximately 0.05°C above the gallium melting point. The handheld cell was operated as described above. (The same thermistor and Super-Thermometer used to determine the reproducibility of the TPDE were used for this comparison.) The average difference in the equilibrium temperatures between the two cells was found to be well within 0.001°C (Fig. 6).

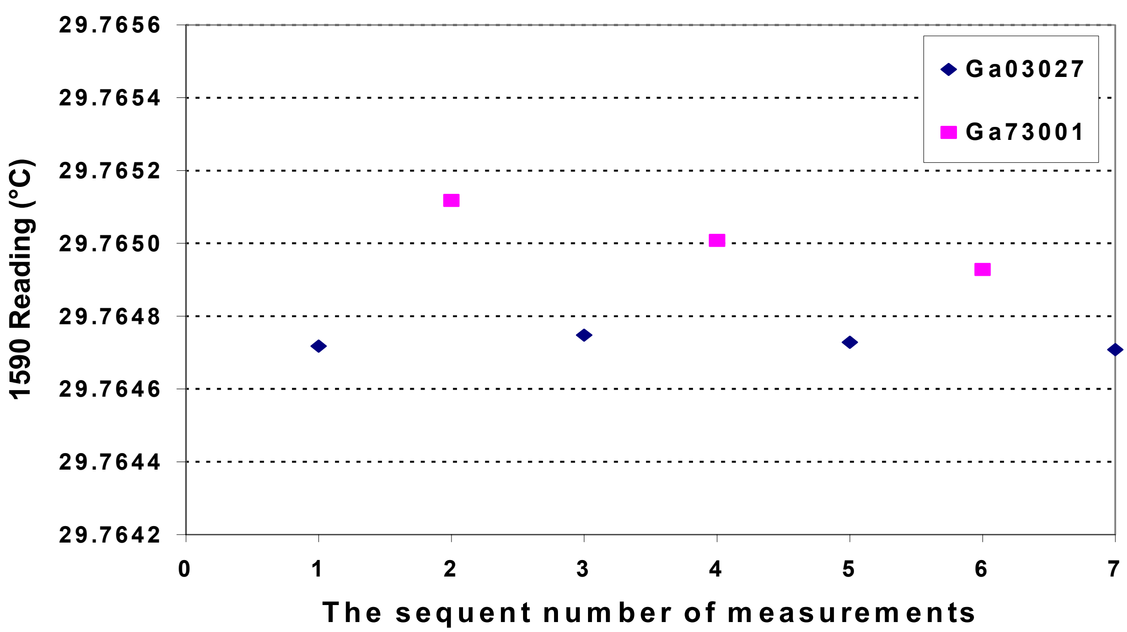

Fig. 6: A direct comparison between a handheld gallium cell and a reference gallium cell

Discussion

These handheld fixed points are special purpose cells that can be used to realize fixed point calibrations in the field or lab without heating or cooling devices. The realizations of these fixed points are extremely easy and simple. With no heat source other than your hand, you can perform fixed-point temperature calibrations anywhere in less than an hour with an uncertainty of about 0.01 °C. It is the lowest cost fixed-point temperature calibration method we have found.

Present data suggests the gallium and diphenyl ether cells can be used across a broad ambient temperature range and are very reproducible. The melting point of the gallium/tin eutectic alloy, however, needs further investigation. In outdoor applications with colder atmospheric temperatures, the eutectic cell may require slightly different techniques, including the introduction of heat sources other than the hand. Experiments have been performed on a preliminary basis using chemical processes, which produce heat but require no power source. More experiments are needed to reach absolutely repeatable and predictable results.

Commercial versions of these cells are only a few months away. These truly portable, easy-to-use cells open up the possibilities of many types of validation and calibration applications at fixed point accuracy levels within these temperature ranges, that were not possible previously. In addition to the obvious benefits of ease of use and portability, these cells are very inexpensive, both in terms of purchase price for the cell and the extremely low cost of supporting apparatus.

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References

1. X. Li and M. Hirst, New Generation of Fixed Points, Proceedings of 1997 NCSL Workshop and Symposium, vol. 2, pp. 567-577, 1997.
2. X. Li and M. Hirst, Fixed Points for Secondary Level Calibration, Proceedings of TEMPMEKO 99, The 7th International Symposium on Temperature and Thermal Measurements in Industry and Science, vol. 1, pp. 74-79, 1999.
3. X. Li, et al, Mini Metal-Cased fixed-Point Cells, Proceedings of 8th International Symposium on Temperature and Thermal Measurements in Industry and Science, vol. 2, pp. 777-782, 2001.
4. J. D. Cox, F. M. Vaughan, The Triple Point of Phenoxybenzene, Metrologia, **16**, pp. 105-109, 1980.
5. J. D. Cox, F. M. Vaughan, Temperature Fixed Points: Evaluation of Four types of Triple-point Cell, TMCSI, **5**, pp. 267-280, 1982.
6. G. T. Furukawa, D. C. Ginnings, R. E. McCoskey and R. A. Nelson, Calorimetric Properties of Diphenyl ether From 0° to 570°K, J Res Nat Bur Stand, 46, pp. 195-206, 1951.