Calibrating Thermometers

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A thermometer without a traceable calibration route to recognised National Standards is fairly useless. Yet we all buy mass produced thermometers which are supplied without a calibration and use them. I assume we all hope that the manufacturer has been conscientious and has at least carried out calibration checks on batch samples and has claimed a level of accuracy to the batch. But has the manufacturer used suitable standards for the calibration?

The only way of being confident in a thermometer is to have it calibrated, then we can be sure that the reading it gives is meaningful. You would not rely on the time from your wrist watch being accurate unless you had checked it would you? Yet we run industrial plants with un calibrated thermometers!

Temperature is one of the most commonly measured physical quantities but its basis is not widely understood. Unlike other quantities, such as mass and time, temperature is defined on a theoretical set of conditions whereas other units are based on real physically realisable defined conditions. For instance the perfect kilogram is in Paris and time is based on atomic transitions in a caesium atom.

Temperature is based on the thermodynamics of perfect systems, such as ideal gases and this results in the thermodynamic temperature scale measured in Kelvins (K), which is unattainable. What we do is the next best thing and use imperfect thermodynamic systems to achieve a working temperature scale as near to the theoretical one as we can get. The current working temperature scale is the International Temperature Scale of 1990 (ITS-90) and is measured in degrees Celsius (°C) for temperatures above 0° C and K or °C are used below 0° C (1° C = 1K).

When a thermometer is calibrated it should be to ITS-90 or to an agreed previous temperature scale such as IPTS-68 or even IPTS-48.

ITS-90 and previous scales are Internationally agreed temperature values in ° C assigned to repeatable physical phenomena that occur at a constant temperature (e.g.. freezing point of a pure metal) and an agreed type of standard thermometer to be used over a range of these points as an interpolation instrument. The assigned values to the fixed points are the best practical thermodynamic temperatures that can be obtained experimentally for them using current technology. As technology improves, better measurements can be made and about every 20 years the practical scale is updated. An example of this is the freezing point of Zinc which in 1968 was 419.58° C but in 1990 became 419.527° C. Zinc carries on freezing as it always has but our thermodynamic measurements have improved (we hope) and hence the assigned value of temperature.

The simplest method and the most accurate method of calibrating a thermometer is by using fixed points, FIG 1 but you are limited to a small selection of points over a temperature range and care must be taken in establishing true traceability to recognised National Standards.

FIGURE 1 - SEALED METAL FIXED POINT CELL

Most of the work in temperature measurement is carried out in the range -200° C to 1100° C. The type of fixed point and its currently assigned value under ITS-90 is shown below in Table 1 for the range -200° C to 1100° C.
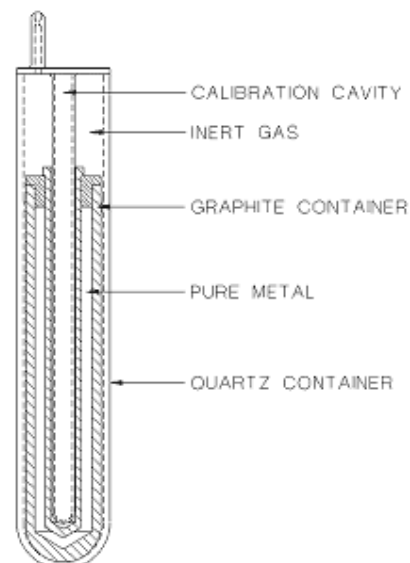

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TABLE 1

FIXED POINTS DEFINING ITS-90 OVER THE RANGE

-200° C TO 1100° C

FIXED POINT	PHYSICAL PROPERTY	TEMPERATURE ° C	INTERPOLATION THERMOMETER
Argon	Triple Point	-189.3442	SPRT
Mercury	Triple Point	-38.8344	SPRT
Water	Triple Point	0.010	SPRT
Gallium	Melt Point	29.7646	SPRT
Indium	Freeze Point	156.5985	SPRT
Tin	Freeze Point	231.928	SPRT
Zinc	Freeze Point	419.527	SPRT
Aluminium	Freeze Point	660.323	SPRT
Silver	Freeze Point	961.78	SPRT
Gold	Freeze Point	1064.18	RT

i. SPRT: Standard Platinum Resistance Thermometer

ii. RT: Radiation Thermometer

iii. Triple Point: Liquid, Solid and Gas are in equilibrium

To realise temperature between these points well defined thermometers have been assigned as shown in Table 1. These are calibrated at the fixed points over a specific range and then used to interpolate between the fixed point values.

To calibrate a working thermometer at temperatures away from the fixed points the SPRT has to be used and the working thermometer compared against it. This necessitates putting both thermometers into an isothermal volume large enough to ensure that the thermometers are at the same temperature as the volume.

Hence from the defining artifacts of ITS-90 has come the two methods of calibrating thermometers, these are the fixed point method and the comparison method.

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As an example of the use of fixed point cells let us examine the temperature range of an electrical power generating utility in a remote location. The utility would need accurate temperature measurements in the range 0° C to 660° C to maintain efficient generation.

The fixed point cells this utility should have would be those in Table 2 below:

TABLE 2

BEST SELECTION OF FIXED POINT CELLS FOR A POWER UTILITY

SLIM FIXED POINT CELL	PHYSICAL PROPERTY	TEMPERATURE ITS-90 ° C
Water	Triple Point	0.010
Gallium	Melting Point	29.7646
Indium	Freeze Point	156.5985
Tin	Freeze Point	231.928
Zinc	Freeze Point	419.527
Aluminium	Freeze Point	660.323

The cells above would give the greatest versatility in performing calibrations. The utility would only need to send out the minimum of equipment for re-calibration saving in costs and transportation damage. The fixed point cells would not need to be full size cells, as found in Primary Laboratories, the newer, smaller slim cells would suffice at this level of accuracy. Full size cells provide 180mm of actual cell immersion and the slim cells provide 130mm and are less expensive.

Another good example of the use of slim fixed point cells is in calibrating the Kaye Intelligent probe as widely used in the food and drugs industry. The Kaye probe is a resistance thermometer which has the measuring electronics in the handle. The probe gives out engineering units via an RS232 link. Although the electronics are temperature compensated it is best to keep them cool which necessitates a small depth of immersion during calibration. The slim fixed point cells are ideal for this use, as they give high accuracy with a small depth of immersion which is not attainable by other methods. We have developed a system for calibrating these probes which gets the best out of them. We use the slim cells outlined in Table 3 below:

TABLE 3

A SELECTION OF SLIM CELLS FOR CALIBRATING KAYE INTELLIGENT PROBES

SLIM FIXED POINT CELL	PHYSICAL PROPERTY	TEMPERATURE ° C ITS-90

Mercury	Triple Point	-38.8344
Water	Triple Point	0.010
Gallium	Melting Point	29.7646
Tin	Freezing Point	231.928
Zinc	Freezing Point	419.527

To use fixed point cells you either cool them, heat them or hold them at a constant temperature, dependent on the physical property. For example, a Zinc point cell (419.527° C) can be used as a melt point cell or a freeze point cell, the value is the same. To melt it you heat it to 1° C or 2° C above its fixed point temperature and to freeze it you melt the metal and cool it to 1° C or 2° C below its fixed point temperature.

In Primary Laboratories the large cells that operate above 30° C, are operated in the freeze mode. To obtain the flattest plateau quickly the cell is given a cool shock to start the freeze, either using a quartz glass rod at ambient temperature or an air blast placed in the measurement well. Once the freeze has been started, a standard thermometer of known calibration is inserted into the cell to verify the freeze plateau exists and the temperature is correct. Any stable thermometer can be used to verify the plateau is occurring so long as the thermometer's output remains constant, but the use of a standard to check the temperature helps ensure correctness in the method and equipment.

Another check to ensure all is well with the cell, furnace and thermometer is to withdraw the thermometer a few centimeters and check the thermometer's output still remains constant.

For slim cells we have simplified matters by recommending they are used in the melt mode. It becomes a simple matter of taking a frozen cell, putting it in an apparatus, such as our Medusa dry block bath and setting the temperature to 1° C to 2° C above the melt point. A standard thermometer can be used to verify when the melt has started but any stable thermometer should show a steady reading when the cell is melting. This is a much simpler procedure to achieve a plateau than the freeze technique and it has two other advantages; if the thermometers for calibration are slightly cooler than the cell when they are put into it, they will cause some of the melted metal to resolidify and hence prolong the life of this melt. Slim cell plateaus are typically stable to ±0.002° C for over 6 hours dependent on equipment and use.

The more calibrations you do the longer the melt plateau lasts.

This is the opposite case for a cell in the freezing condition, as the cooler thermometer to be tested will shorten the freeze time.

The other advantage is; when in the melt mode the background temperature of the furnace is higher than the cell and this helps reduce stem conduction errors found in thermometers which need larger depths of immersion.

Silver leads are used in the internal construction of some resistance thermometers to make them cheaper, but this increases the required depth of immersion for accurate measurements due to the increased thermal conduction of the Silver. Platinum leads are preferred.

The water triple point cell is one of the harder cells to set up. There are basically two methods:

1. Forming an ice mantle on the re-entrant tube by either using liquid Nitrogen or Liquid Nitrogen cooled rods or solid Carbon Dioxide.

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2. The slush method, involves super cooling the water in the cell to about -7° C and then it is shaken once. The physical shock is enough to initiate the formation of ice crystals which travel through the water forming a slush.

The first method is considered the most accurate but relies on having access to a source of low temperature, where as the slush method is less demanding on equipment needed and skill. We make two metal block baths which will conveniently enable the formation of the slush and the short term storage of the cell while it is being used.

Having made the cell it needs to be held at a constant temperature around 0.010° C. The cell is ready for use about 30 minutes after making it. This waiting period allows some of the thermal stresses to relieve themselves. For our very best measurements where we are verifying our own Jarrett-Isotech cells to a UKAS* certificated accuracy of ±0.00015° C, measurements are made over weeks, maintaining the cells in our model 18233 cell storage bath. For maintaining cells over short periods a flask of crushed melting ice is O.K. and as mentioned above two of our metal block baths will make and maintain a cell.

COMPARISON

The comparison method of calibration is the most widely used method of calibration. It involves comparing an inferior thermometer with a superior unit. They could both be the same model of thermometer but the superior unit would have been calibrated to a higher accuracy. The comparison has to take place in something like a liquid bath where a suitably large volume can be made to have the same temperature throughout. This isothermal volume needs to be big enough to contain both thermometers such that they are at the temperature of the bath.

IMMERSION

The immersion depth of a thermometer to achieve the bath's temperature is dependent on; the construction of the thermometer, the temperature difference between the bath and the surrounding atmosphere, the heat transfer capability of the bath and the temperature stability of the bath. A rule of thumb estimation of this depth of immersion is; 10 to 15 times the diameter of the thermometer plus the detector's length. This should be increased for high and low calibration temperatures and where the diameter of the thermometer is significantly smaller than the internal diameter of the pocket it is in.

A simple test to determine if a thermometer is at the temperature of the bath is to withdraw it by 2cm and see if its reading changes. This withdrawal can be continued to determine the minimum immersion depth.

As implied above a thermometer reads its own temperature, you hope this is also the temperature of the surrounding medium. To ensure that it is the correct reading, checks should be made.

*NAMAS National Measurement Accreditation Scheme of the UK. (NAMAS is a very important independent supervising body to verify that calibration laboratories can do what they say they can. This is achieved by monitoring all aspects of the Laboratory and includes regular visits and audit calibrations).

AN EXAMPLE OF A COMPARISON CALIBRATION

As an example of a comparison calibration let us examine two ways of calibrating an industrial platinum resistance thermometer (IPRT) at 150° C. This is best achieved using a stirred oil bath and a standard platinum resistance thermometer (SPRT). For the best results the two thermometers would be mounted deep in the bath ($\geq$ 300mm) and in a Copper or Aluminium equalization block. We next need an instrument which can measure resistance. This can either be a digital multimeter or a purpose built resistance measuring instrument. The accuracy of the calibration system depends on how accurately we are trying to calibrate the IPRT. For this example we will assume it is for the drugs industry and it must be calibrated to ±0.1° C or better. To achieve this our calibration system should be 3 to 10 times more accurate, ie. ±0.03° C to ±0.01° C. We therefore need to be able to establish an isothermal volume, measure its temperature and the resistance of the unknown thermometer to ±0.03° C or better. As it is an IPRT its stability and repeatability will not be good, but our calibration system is good enough to measure

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this performance and assign a value to the thermometer. We can then determine if the IPRT is capable of being a ±0.1° C device. The minimum instrument for measuring the resistance of the thermometers should resolve 3 decimal places and have an accuracy of 0.004% minimum. This allows the resistance of the thermometer to be measured to an equivalent of about ±0.01° C. If the bath and the SPRT also have similar accuracies then we have our basic ±0.03° C system accuracy on first inspection.

To do the measurement we set the bath to as close to 150° C as we can and establish that everything is stable by measuring both thermometers. The simplest calibration method is to take between 5 to 10 readings of each thermometer's resistance in matched data pairs and average these for the calibration values. Use the SPRT's resistance to work out the temperature from its calibration certificate and then present the results as shown below in Table 4.

TABLE 4

SIMPLE COMPARISON RESULTS

TEMPERATURE ° C	OUTPUT OF THERMOMETER UNDER TEST OHMS	UNCERTAINTY OF THERMOMETER UNDER TEST ±° C
150.032	157.319	0.1

The second method of calibration relies less on adjusting the bath temperature to be so close to 150° C. The baths temperature is brought to within ±0.1° C of 150° C and the resistance of both the thermometers measured as above then becomes:

$$_{150}R_x = (R_x \div R_{SPRT}) \text{ x } _{150}R_{SPRT} \text{ ohms}$$

Where $_{150}R_x$ is the resistance of the thermometer under test at ideally 150.000° C

R_x is the resistance of the thermometer under test measured in the bath at about 150° C

R_{SPRT} is the resistance of the SPRT measured in the bath at about 150° C

$_{150}R_{SPRT}$ is the resistance of the SPRT at 150.000° C derived from its calibration certificate.

This method relies on the ratio R_x / R_{SPRT} being reasonably constant and this is normally the case for stable temperatures within ±0.1° C of the required temperature value. Consider if the thermometers were exactly the same then this ratio would always be 1 across the whole temperature range. Another advantage of this method is that it relies less on the accuracy of the meter especially if the ratio is 1. The meter just has to be stable over the measurement period and have sufficient resolution.

The results could be presented as shown below in Table 5

TABLE 5

RATIO METHOD COMPARISON RESULTS

TEMPERATURE ° C	OUTPUT OF THE THERMOMETER UNDER TEST, OHMS	UNCERTAINTY OF THE THERMOMETER UNDER TEST ±° C
150	157.307	0.1

With modern true 2 channel digital thermometers, such as our TTI 2, the above type of result can very easily be obtained. The TTI 2 can be programmed with the calibration of the SPRT so it can indicate the actual temperature of the bath. It will then work out the ratio R_x /R_{SPRT} since the IPRT will be connected to the other channel.

To improve the accuracy of resistance measurements made with DMM's especially if they do not have an accuracy of ±0.004%, a standard resistor can be placed in the measuring sequence. The resistor should be of a similar value to the thermometers, e.g.. 100 ohms and have an accuracy of $\leq$ ±0.004%.

Then as long as the DMM can resolve 3 decimal places and has a good short term stability accurate results can be achieved, as shown below:

$R_x = (R_{DX} \div R_{DS}) \times R_S$

Where R_x is the resistance of the unknown resistor being calibrated.

R_{DX} is the resistance of the unknown resistor measured on the DMM

R_{DS} is the resistance of the standard resistor measured on the DMM

R_S is the true value of the standard resistor from its certificate.

For the most accurate resistance measurement a 4 wire configuration should be used along with a switched dc or an ac resistance measurement system.

The 4 wire configuration helps to eliminate errors in the connecting wires found in 2 wire systems. The use of switched dc. (ie. measuring the resistance with the dc current in one direction and then repeating the measurement with the current in the other direction and taking the average of the two readings) or ac resistance measuring systems help eliminate thermo electric errors in the connecting wires.

TRACEABLE CALIBRATIONS

Finally, in this article we would like to discuss 'traceable calibrations'. In calibration a traceable measurement is one where the start and end of a calibration chain can be followed. The IPRT we calibrated in the example above would be one end of the calibration chain and a National Standard would be the other. In between, on average, would be one or two comparison calibrations of standards. However, it could be much more and still comply with the concept of a traceable calibration. The longer the chain the less accurate will be the result at the end.

There is no time period associated with traceability.

Hence a long traceable chain will probably have many years between its start and finish. Consider an SPRT calibrated at NIST which has a 2 year recalibration period and this SPRT is used to calibrate a working standard every 2 years. That working standard can be 4 years away from the National Standard and still be doing work!

Having a traceable route does not guarantee good measurements. That is down to the staff, equipment, environment and procedures.

In the UK, we have an independent body (NAMAS) which monitors the work of its Accredited Laboratories to ensure the final calibration is sensibly traceable to recognised National Standards.

The term 'Recognised National Standards means that other country's Primary Standards can be used as long as an agreement of co-operation exists between them.

Hopefully with this article we have made some interesting points and passed on some practical advice. It is the practical techniques that interest us the most and in future articles we will be concentrating on these.

Finally, Isotech's sales catalogue is also a good guide to currently available equipment in temperature calibration.