



Temperature Metrology



An intermediate course in practical lab skills for comparison calibration of thermistors, RTDs, thermocouples, and other thermometers.

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Temperature Metrology

Isothermal Heat Sources

Introduction

The Purpose of an Isothermal Heat Source

Definitions

An Isothermal Heat Source is a temperature controlled mass which is intended to provide the identical temperature to the Reference Thermometer as it does to the Test Thermometers during comparison calibration. Comparison Calibration exists when traceable temperature knowledge from a reference thermometer is transferred to a test thermometer.

The purpose then of the source is to use its unique properties during the temperature calibration process. These sources come in many forms. The mass may be any matter that can be controlled adequately for the purpose.

Uncertainties

Uncertainties or temperature inaccuracies will be created to some level depending on the sameness of temperature between the reference thermometer and the unit under test (UUT) locations during measurement. These uncertainties are the primary limitations of the calibration transfer. They are caused by variations in:

- Temperature Uniformity: Homogenous temperature
- Temperature Stability: Steadiness of temperature
- Stem Conduction: Errors created by inadequate thermal connection between the heat source and thermometer, and thermal conduction along the thermometer between the source and ambient temperatures.

This discussion will provide some examples of heat sources, their relative thermal properties, temperature ranges, stability and uniformity as well as other considerations for selection and optimization.

Examples of Isothermal Heat Sources

Liquid Baths: Utilize temperature control and stirring of fluids such as water, oils, molten salt, molten metals etc. to maintain a stable and uniform transfer of temperature.

Dry-Well: Utilize metal blocks as a heat transfer medium. These devices may be portable with wide temperature ranges. The temperature controller and control sensor may be calibrated to provide the temperature reference. Changeable inserts permit calibration of different thermometer diameters.

Furnaces: A laboratory instrument utilizing a metal, graphite or some other material as an equalization block. Temperature uniformity may be improved with profiled heating, heat pipes or multiple heating zones. Long immersion depths are typical in laboratory versions.

Fluidized Baths: Utilize a powdered solid material suspended in a flow of air to provide thermal uniformity.

Phase change devices: A Phase Change such as the melting of ice in an ice bath or boiling liquid nitrogen provides a stable and uniform medium. A fixed-point cell may be also used as a constant temperature device using an SPRT as a reference.

Comparison of Performance

Not all heat sources will serve all applications. Your error budget, including all sources of error, will help you determine what the stability and uniformity need to be. The following generalized charts will give you some idea of the relative characteristics of different heat source instruments.

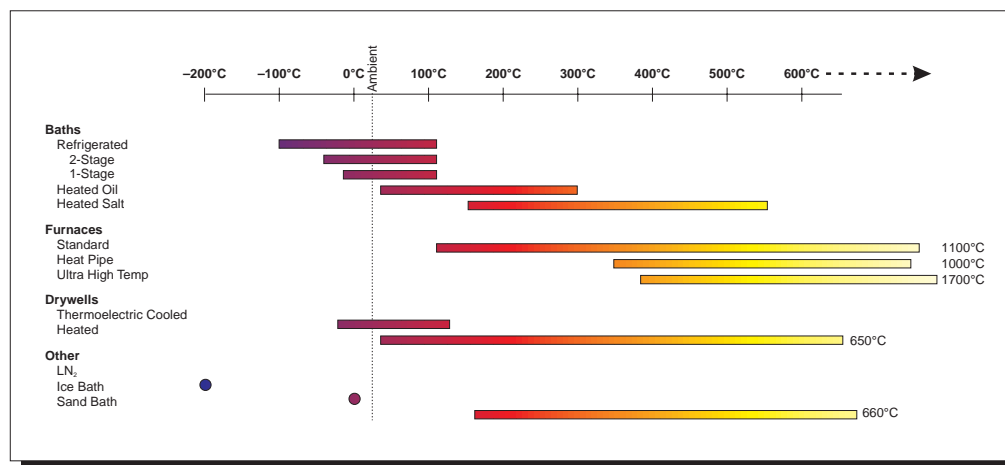


Figure 1. Typical ranges of various heat sources

The temperature range required for a calibration is determined by the use of the UUT. The temperature range of the heat source is limited by design, materials of construction or ancillary material like bath fluids. Refrigerated baths are limited by the breakdown temperatures of

the refrigerant or refrigerant oil contained in the evaporator in the bath. Materials of construction have temperature limits; heater designs have limits, and so on.

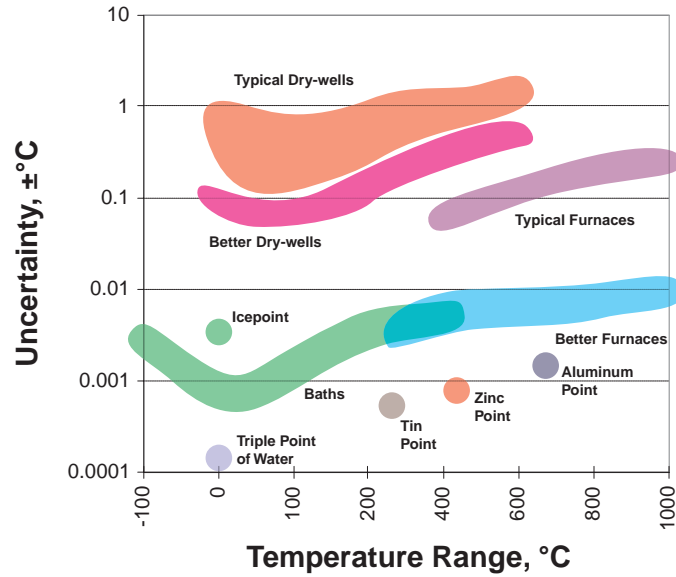


Figure 2. Typical uncertainty of various temperature sources.

Each heat source has limits of uncertainty as well. The chart above compares some typical ranges of uncertainty for different types of heat source instruments. The separation is clear between fixed-point measurements and comparison measurements of the various types. It is also clear that some comparison heat sources have better uncertainty characteristics than others do. While these things are true, there are other considerations that must be made.

The essential performance of any isothermal heat source is to meet the definition above for a particular application.

Discussion of the Basic Elements

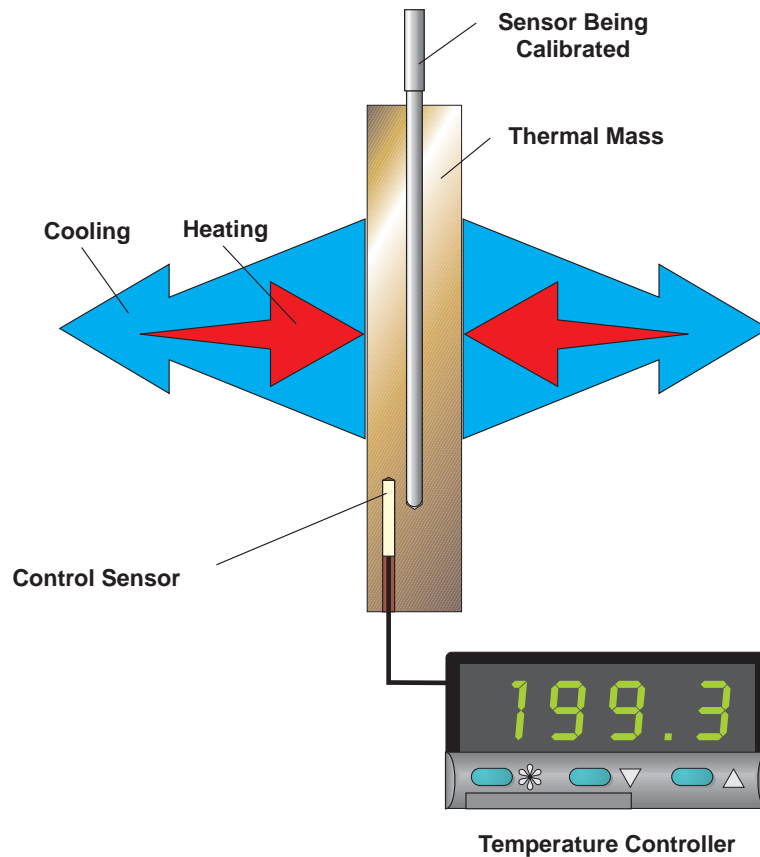


Figure 3. Isothermal Heat Source Basic Elements

The basic heat source will include a “thermal mass” made of some thermally conductive material in which sensors to be calibrated are inserted. It may be a solid mass such as a metal or a fluid such as water or oil. It is also isolated from the environment in such a way that excessive heat gains or losses are minimized and so that gradients are low.

A temperature controller will sense the temperature of the mass by means of a temperature sensor and will control the heating or cooling to it in order to control its temperature.

Fluid media heat sources, such as baths, offer a significant benefit of stirring. The stirring action allows the temperature variations to be physically mixed or homogenized. Because of this, baths have a greater potential for high uniformity than any of the other heat sources. Without stirring, other means must be considered to limit the gradients. Proper location and design of heating and cooling elements, protection from ambient heat losses or gains and so on are required. Heating and cooling must seem to come from the same source and works best if it surrounds the thermal mass rather than come from within it.

Heat Source Selection

The user must ask: *what device of those available meets my needs?* Needs may mean current needs or future needs. Considerations may include whether it is used in the lab or in the field.

It would also consider types and sizes of probes and temperature ranges. And certainly, you must consider the uncertainty levels needed. Available may mean availability in the market place or availability within my organization. Is the source available economically feasible? Other selection considerations may concern the productivity, utility, or practicality of a heat source for your application.

This list includes several important issues besides uncertainty and temperature range:

- Cost (for unit or accessories)
- Speed or throughput
- Cleanliness (media does not contaminate thermometer or area)
- Portability (may be required for some calibrations)
- Safety
- Ability to automate (computer interface)
- Lifetime
- Ease and convenience of use
- Utility or flexibility
- Reliability

Constant Temperature Calibration Baths

Different bath products vary in design details but they generally fall into these 4 basic concepts. Each concept or type has certain advantages and disadvantages and each manufacturer may have succeeded to different levels in attaining optimum results. The types are 1) common utility design, 2) parallel tube design, 3) concentric tube design and 4) heat port design.

Design Types

Utility Bath Design

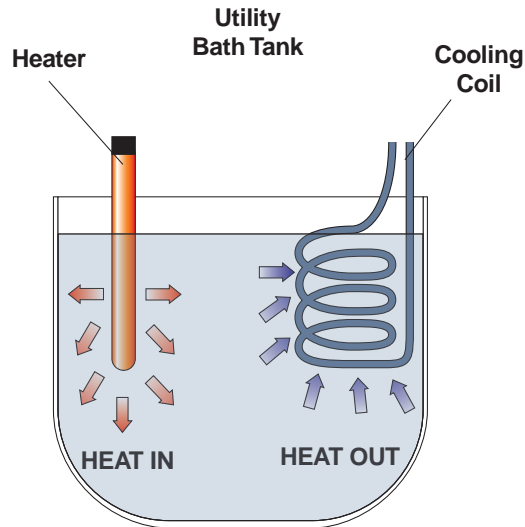


Figure 4. Diagram of utility bath.

Utility baths are available at low cost but fail to provide adequate immersion depth, high enough temperature uniformity or stability. Some may only be 4 to 6 inches deep. Another problem is stirring. Utility baths typically use a pump device for pumping fluid to an external device. These pumps do not typically circulate the fluid well enough inside the bath to homogenize the temperature gradients. Their controllers do not have the resolution for mK stabilities. And, finally, they have serious problems with heating and cooling element design. They typically have separate heating and cooling elements inside the tank. Refer to Figure 4. Separate heating and cooling elements force a temperature gradient in the fluid as heat from the heater flows to the cooling source. At the extreme, several degrees can exist between them. This is a difficult job for their already anemic stirring systems.

Figure 5 illustrates several thermometers in different locations within a water bath. This bath is at 25°C at the center. Each of the other thermometers has two sets of temperature deviations relative to the reference in the center. They represent differences that may be found between two *different* baths. The upper reading is more ideal, varying only fractions of a mK from the setpoint that may be found under ideal conditions for a good design. The lower set illustrates a much larger deviation creating a much larger non-uniformity uncertainty for the calibration process. This illustrates the impact of bath design on calibration performance. Other parame-

ters may effect performance as well, including fluid properties such as viscosity, temperature and others.

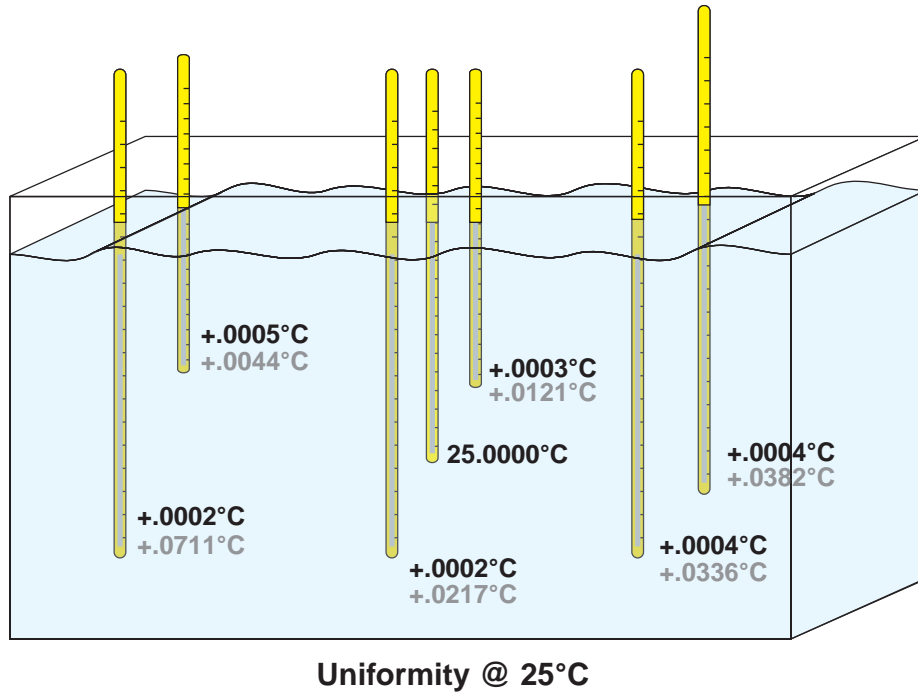


Figure 5. Bath uniformity comparison.

Parallel Tube Design

The parallel tube design overcomes some of the limitations of the utility bath. The heating and cooling elements are separate from the calibration working area and allow the heating and cooling affects to be mixed by an efficient stirrer before they can disturb the probes being calibrated. This concept was developed by scientists at National Laboratories specifically for calibration work. Any temperature gradient in the working area can only be due to the heat losses along its length and the velocity of the fluid as it moves through it. The working space is often

small, however. It can also be susceptible to level changes due to fluid expansion & contraction.

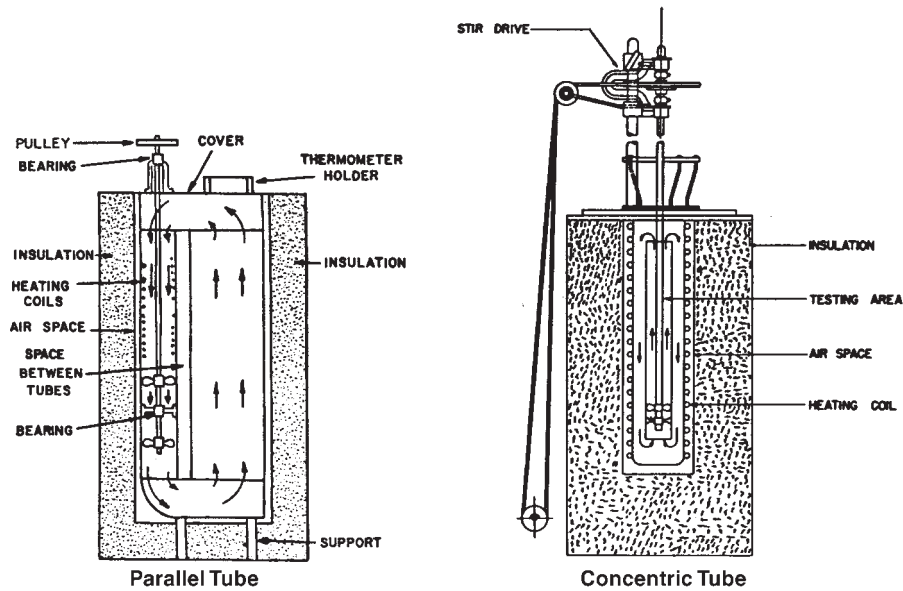


Figure 6. Parallel tube and concentric tube design baths.

Concentric Tube Design

The concentric tube, or tube within a tube concept, features an inner working area containing fluid thermally guarded by fluid from the outer tube. The heating and cooling takes place in the outer area where the fluid is stirred and temperature controlled before it is pumped into the working area. The guarding effect from the fluid in the outer tank helps to temper gradients due to heat losses at extreme temperatures that would otherwise be detrimental to calibration. It can be built to the appropriate depth for better probe immersion. The design overcomes limitations of the parallel tube design regarding stirring problems at low fluid levels and gradients created by high heat losses at extreme temperatures. This concept was also developed by scientists at national laboratories for temperature calibration work. It may be limited by the effectiveness of the design application and temperature controller and other design application issues. It also has limited working space.

Heat Port Design

The heat port design concept features a large unobstructed work space. The heating and cooling elements of a Hart heat port bath are located on the outer wall of the stainless steel tank. Refer to Figure 7 for an illustration of the “heat-port” sandwich. The heating element is next to the tank itself. It is thin and responsive. It covers a large part of the tank surface. Next to the heater is a thin pad of thermal insulating material. It serves to filter the pulsations from the cooling-plate that comes next. The cooling plate covers the entire heater surface. The cooling effect is therefore evenly distributed with the heating, eliminating hot or cold spots. The technique minimizes the job of the stirring system. The actual cooling may be supplied through refrigeration, tap water cooling, or simply heat loss through the walls.

If the cooling is supplied through refrigeration, it is has been thermally stabilized by special refrigeration techniques. The refrigeration system can be adjustable to minimize the amount of power that must be stirred into the fluid.

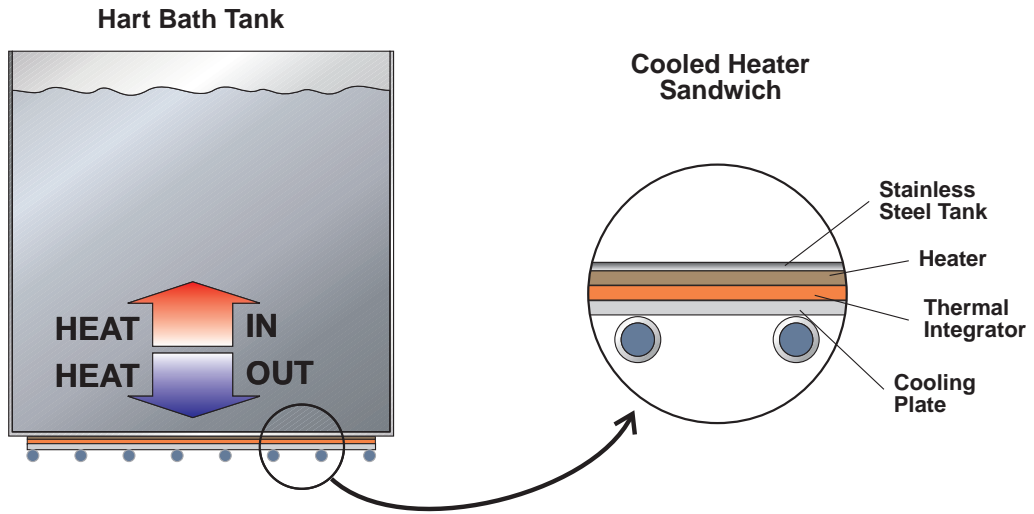


Figure 7. Diagram of Hart cooling plate concept.

Liquid and Glass Requirements

Liquid and glass thermometers require viewing the meniscus of its working fluid. Total immersion thermometers require that all but about 12mm of the liquid column be immersed.

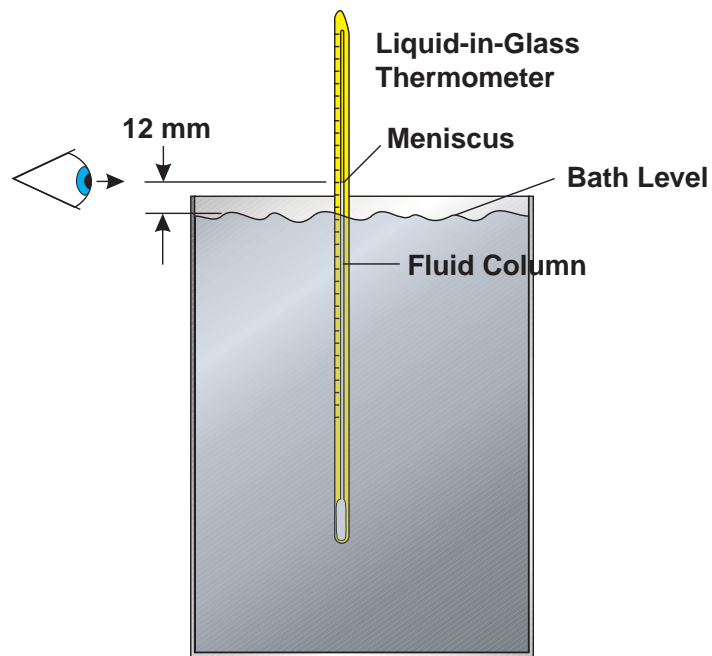


Figure 8. Immersion depth and viewing requirements for a total immersion thermometer

One method is to fill the bath to near the top if the bath has a thin enough lid. Still another method is to have a device that pumps the fluid to a point above the lid so that the meniscus can be viewed conveniently.

Optimizing Performance of Liquid Baths

Consider these issues in order to meet the calibration performance you need.

Immersion depth

A well stirred bath will have minimal thermal gradients. Near the surface, however, there are temperature effects due to heat loss and evaporation. This will reduce temperature stability and uniformity. In addition, stem conduction, which is energy transfer up or down the length of the sensor, is likely to cause measurement errors. The following recommendation will minimize the effect.

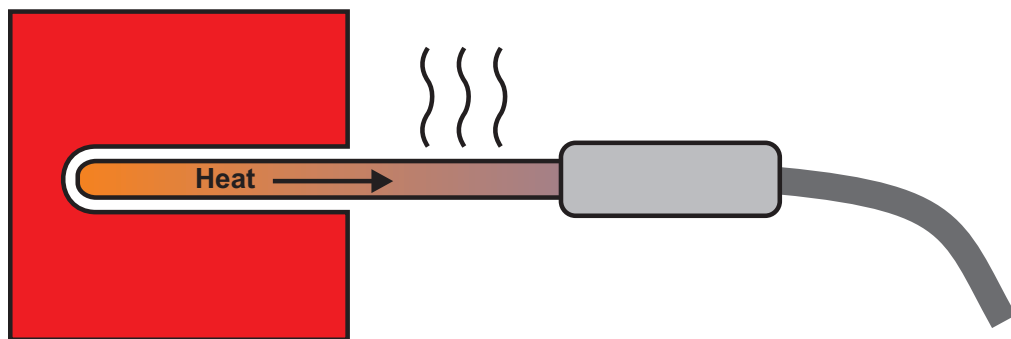


Figure 9. Stem Effect

Rule of Thumb: Immersion Depth = 20 x the diameter + sensor length

Example: The diameter is .25 inches and the sensing element is 1.25 inches long.

Immersion Depth = $20 \times .25 + 1.25 = 6.25$ inches

Immersion Depth Testing

It may become necessary to do testing to determine a minimum critical depth for a particular thermometer. This procedure can help you visualize what depth is required.

A uniform heat source is required for an immersion test. A temperature readout device capable of the resolution required is also needed.

Procedure: Set up the equipment with the heat source at the desired temperature. Insert the thermometer to its maximum safe depth and let it stabilize. Use some type of clamping device to hold it in place. Note the temperature then withdraw thermometer 1 inch. Again wait until it has stabilized and note the temperature. Continue this process until the thermometer sensor reaches the surface (or some other practical limit is reached). Plot the temperature differences between the bottom temperature and the temperature at each depth. The errors will flatten out

through the range of adequate immersion. To eliminate these errors, the minimum depth must be achieved.

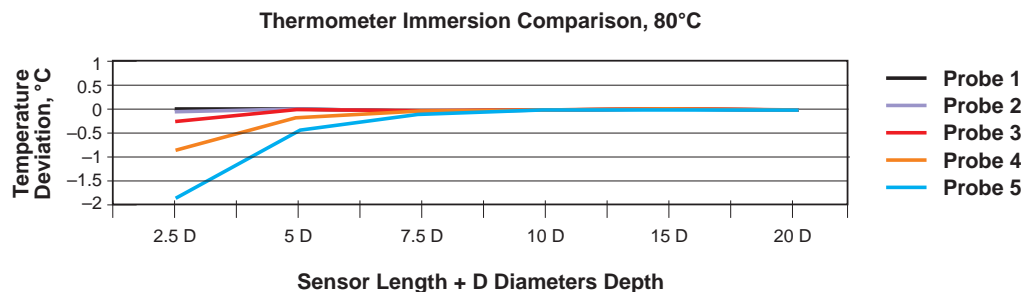


Figure 10. Test Graph Example

You may wish to test a variety of thermometers in this fashion and compare the results. Also compare the results at higher or lower temperatures. You will see that the required immersion depth increases as the heat source and ambient temperatures separate. This temperature difference is the driving force causing the effect. The thermal resistance between the source and the sensor relative to the thermal resistance from the sensor to ambient causes the temperature read to be between the source temperature and ambient temperature under conditions of inadequate immersion.

Fluid Selection

The selection of the right bath fluid is one of the most important optimizing criteria. Consider the following issues when selecting your bath fluid medium.

- Temperature Range: it must fit the range required for calibration.
- Viscosity: it must be low enough to permit adequate stirring and hence adequate stability.
- Conductivity: the higher the conductivity the greater the response and the lower the gradients.
- Specific Heat: a high specific heat reduces temperature disturbances by requiring more energy to change the temperature a given amount.
- Toxicity/Fuming: health effects must be considered; take precautions, use a fume removal system.
- Safety: use fluids below their flash points, always study the Material Safety Data Sheet (MSDS) information.
- Expansion of fluid: allow adequate space for thermal expansion.
- Cost: high temperature fluids tend to be expensive.

- **Water Miscibility:** water will condense or freeze in fluids below the dew point, changing the fluid's characteristics.
- **Sensor Contamination:** some sensors being calibrated may be contaminated by some fluids. Some fluids, such as silicone oils, can be difficult to completely remove.

Viscosity and temperature range are the most critical choices for the best uncertainties. If these cannot be met, the calibration objectives may be unattainable. Often, finding adequate specifications in these areas precludes choices of other criteria such as lowest cost. It is seldom possible to find an ideal fluid for your application. Some compromises will need to be made. This often means more baths with different fluids in them to cover the required range.

Select a viscosity below 10 centistokes if possible. The critical action of stirring can generally be maintained satisfactorily below this point. As the viscosity increases, stirring becomes less effective, creating temperature gradients and greater instability. Viscosities greater than 50 centistokes may reduce stirring effectiveness to a level below that required for even minimum calibration requirements.

Temperature and viscosity are closely tied in determining the acceptable temperature range of a fluid. The temperature range is also limited by other conditions such as evaporation and the boiling point. As the vapor pressure increases with temperature, the cooling of the surface of the fluid increases, creating stability problems. Additionally, the droplets formed on the lid of the bath, which consequently fall back into the fluid, create a disturbance that can be measured as thermal instability of the bath. Boiling fluid is also unstable. Loss of expensive fluid through rapid evaporation is also undesirable.

At lower temperatures (below the dew point of water), water can condense into the fluid. If the fluid is immiscible with water as with oils, the mix of fluid properties may cause problems. This is particularly a problem at temperatures below freezing as the ice creates a thick slush. The slush has poor thermal properties and does not stir well resulting in poor stirring of inhomogeneous materials. The fluid must be heated to evaporate the water out of it. A dry gas purge slowly bled into a nearly closed-off bath can help. A dry-box containing a desiccant placed over the access well can also slow the absorption of water. Fluids such as alcohol can absorb a large amount of water before saturating. Care must be taken for safety, however.

Safety is an important issue with any extreme heat source. Whether or not warnings are in place for burns or fire, care for safety is essential. This is particularly the case with hot oil and salt baths. Toxicity is, of course, an issue as well. Many fluids have ideal thermal properties but are less than ideal with regard to toxicity. Always study the MSDS that must be provided with the fluid and be prepared to act with prudence. Hart Scientific recommends the use of fume removal systems. The type that have tubes to remove the fumes near their exit point from the bath are the most effective. Some hood designs permit the fumes to pass by the user before they are removed. Human contact with fluids or fumes should be minimized.

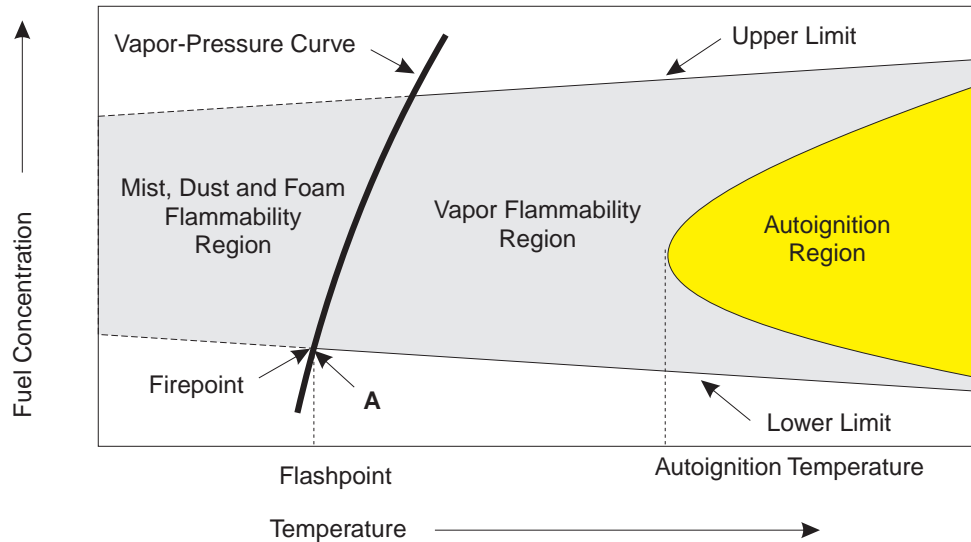
Safety with regard to flammability is important to each fluid user and his organization. Terms such as "firepoint", "flashpoint" and "autoignition temperature" should be understood and considered with care in your application. You should know the flashpoint of the fluid you are using at a minimum.

The flashpoint is the temperature at which a fluid gives off enough vapor to form an ignitable mixture with air near its surface. It may be reported as open cup or closed cup. The closed cup temperature defines a condition much like a bath where the vapor is concentrated within an enclosed volume. It is the most conservative value. Hart does not recommend using a fluid

higher than the flashpoint. When the flashpoint is achieved, there can be a condition of sufficient fumes (fuel) and oxygen to ignite it if there is a source of ignition (spark or flame).

The firepoint is the lowest temperature at which the liquid produces fumes fast enough to support combustion.

The autoignition point is the temperature at which a self-sustained combustion can occur in the absence of an external ignition source. An explosion hazard may exist.



Fine mists approach flammability properties of vapors.
Mists of more than 60 μm are difficult to ignite due to large drop heat and mass transfer limitations.

Figure 11. Flammability Concentration Regions vs. Temperature

Location of Reference and Unit Under Test

Even the very best of baths will have instability and gradients to some level. Good practice suggests that having the reference and UUTs as close together as practical is best. In addition, it is advisable to characterize or profile your equipment so you know what it does under the conditions of calibration.

Test the Bath's Temperature Profile

The profiling can be accomplished with a thermometer inserted into the bath at a central location and then measuring deviations between that temperature and the surrounding area that will contain the UUTs. This characterization should be done with regard to the stability of the bath. Take an average measurement. A dual or multi-thermometer arrangement is ideal, with one thermometer always remaining in the same central location as the reference. The test must be conducted at the same temperature and with the same fluid as the intended calibrations. Testing under a range of conditions will illustrate problems with stirring and temperature difference to ambient.

The resulting uncertainty data can be used to help determine overall uncertainties and make improvements in hardware and procedure.

Thermal Equilibration Blocks

Some improvement in the stability of baths can be made with equilibration blocks. Improvements of 2 to 10 times have been observed depending on the nature of the thermal noise and the block design. The block essentially isolates and filters the temperature changes reducing their magnitude. Disadvantages include slower response time and less flexibility in terms of thermometer & UUT size and quantity. Of course, extra cleaning occasionally is also required.

Bath Summary

Bath Advantages

- Best stability & uniformity
- Best flexibility for size & shape of thermometers
- Best reduction of stem conduction
- Best flexibility for quantity of thermometers

Bath Disadvantages

- Limited temperature ranges
- Slower to change temperature
- Fluids can be messy
- Fluid high cost

Dry-well Calibrators

A dry-well calibrator is a fast, convenient, portable, wide temperature range and often relatively inexpensive heat source. They may also be used as a *temperature reference* when appropriately calibrated. For better accuracy results, they may be used as a *comparator* (comparing a reference sensor with a test sensor). Their higher uncertainties may make them inadequate for secondary calibration but they are quite useful for industrial accuracies varying from a few degrees to tenths of a degree. Selection of a dry-well calibrator can be made after understanding your general calibration requirements. A general understanding of their advantages and limitations will be explained here.

A dry-well calibrator basically consists of a mass of metal heated with a heater. The temperature in the block is sensed by a control sensor; a PRT, thermocouple, or thermistor. A temperature controller utilizes the information from the control sensor to control the temperature of the dry-well. Some dry-well designs utilize thermoelectric coolers permitting sub-ambient temperatures with reduced high-end temperatures. Heated-only models typically operate from temperatures just above ambient and higher depending on construction techniques and materials. Some are optimized for speed in heating and cooling, others for sensor volume.

Design Considerations

Some of the design factors that can reduce errors are as follows.

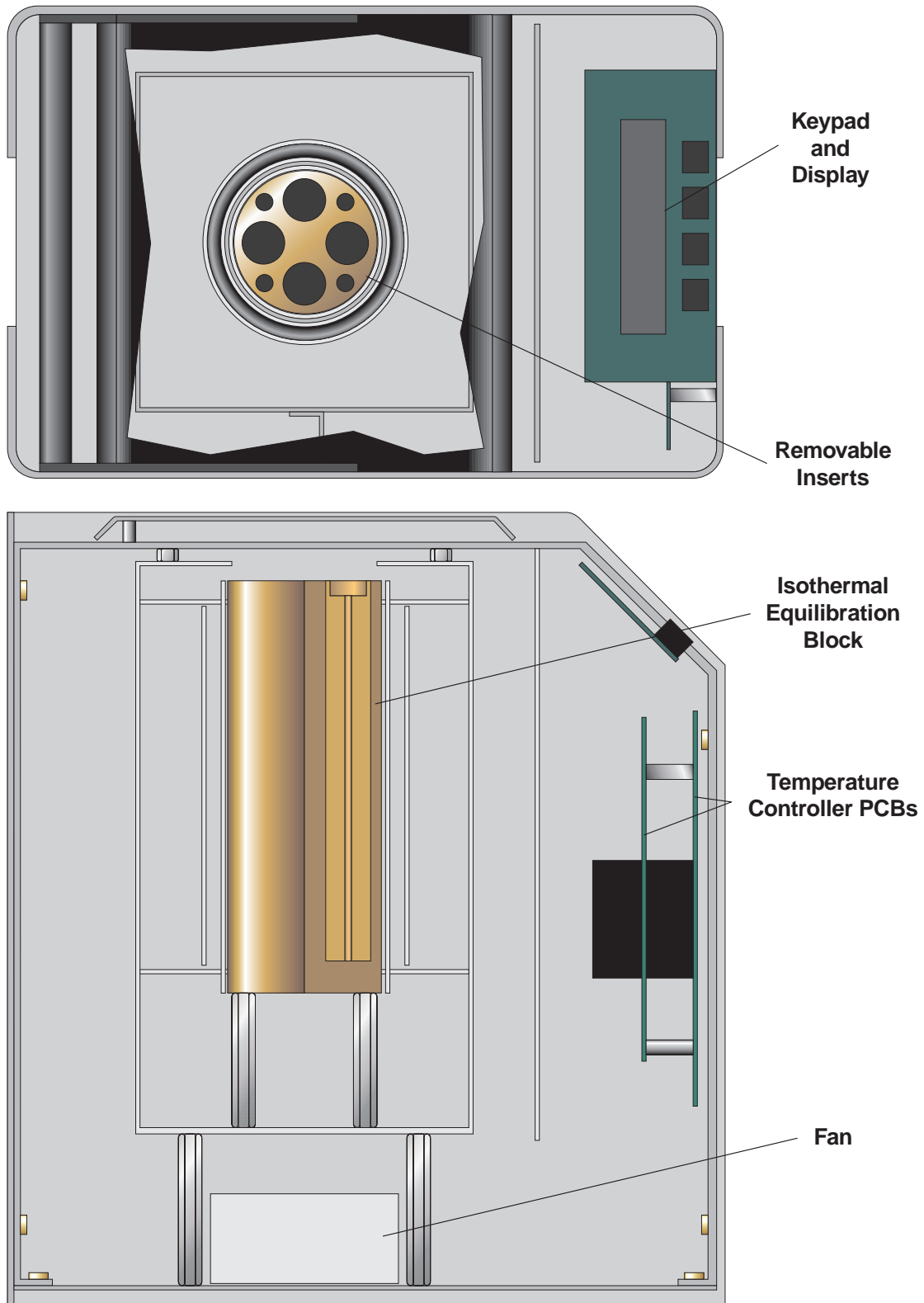


Figure 12. Dry-well Construction

1. Reduction of temperature gradients
2. Allowance of adequate immersion depth
3. High-temperature stability

The metal mass or isothermal block or just block as it may be called, will provide a well or number of wells for the insertion of thermometers of different sizes. This may be done with a variety of fixed size wells or with sleeves or inserts to provide different sizes. A good fit is essential.

Uniformity and Stability

As in the baths, temperature gradients are created by cooling and heating elements or from strong heating and cooling losses or gains. A primary difference is that the thermal mass medium is solid and cannot be stirred like the fluids in the bath. Therefore those gradients that may be in the block cannot be homogenized into a more stable thermal mass. This lack of uniformity creates uncertainties that may be from 10 to 1000 times greater than in a bath depending on makes, models and conditions of use. These gradients may be in any direction in the thermal mass. In multiple well models, the gradient may create temperature differences from well to well. Vertically (along the length of block), the gradient may cause one sensor to read a different average temperature than another due to differences in sensor length or location.

The temperature gradients may be caused by non-uniform heating or cooling. Heating elements that surround the block; i.e. wrapped around it, are superior to elements inserted into it. Inserted elements create hot spots while wrapped heaters tend to create greater uniformity. Heaters can be designed with profiled watt densities to match typical heat losses from the block thus reducing vertical gradients.

Thermal conductivity differences between thermometers and the equilibration block can add to the uncertainties of measurements. Here we are primarily referring to the fit of the thermometer to the equilibration block. The reference and UUTs must see the temperature the same way. Fit has to do with the matching of the diameters of the thermometer and the block, roundness, etc. The clearance between the thermometer and the well is typically from 0.007 to 0.012 inches. There is also a random component of repeatability of measurement from one insertion to another due to differences in thermal contact resistances. All of these things act together to create uncertainties between the temperature of the reference thermometer and the UUTs. The immersion depth of the thermometers helps to minimize these thermal conductivity effects.

Two thermometers that are thermally similar can be compared better than thermometers that differ physically. That is to say, they are the same length and diameter, they have the same sensor length and they fit into the same size well. Under these conditions, many of the affects due to variations in fit, gradient, heat loss etc. will be seen the same way and these errors will cancel out.

Temperature stability of a dry-well is also a factor in making comparison measurements. Just as with the bath, the temperature of a block may oscillate or drift. Many dry-well instruments are only stable to near $\pm 0.1^{\circ}\text{C}$. Higher quality calibrators may have stabilities of $\pm 0.040^{\circ}\text{C}$.

Dry-Well Summary

Dry-well Advantages

- Portable
- No messy fluids
- Wide temperature ranges
- Fastest to change temperature
- Least cost

Dry-well Disadvantages

- Some inflexibility for size & shape of thermometers
- May have some stem conduction problems
- Some inflexibility for quantity of thermometers
- Greater uncertainties (contact resistance, higher gradients & greater instability)

Furnaces

The definition of a furnace as we shall use it here:

A calibration heat source used in the higher temperature spectrum that is used for comparison calibration of laboratory quality thermometers such as SPRTs, noble metal thermocouples, secondary level PRTs, etc.

Heated Zone Design

Of the many aspects of furnace design, one of the most important is that of the heated zone. The heating method must provide the desired temperature point along with safety and long life. As with other heat sources, it must provide a uniform and constant temperature. The heated zone is fairly long in order to accommodate the length of standards level thermometers. Designs are typically cylindrical in order to provide even and identical heating over their length. They utilize thermal equilibration blocks to create a more uniform temperature around the thermometers being compared. Heated zones may be of the following types.

- **Single Zone:** The heater is wound as one uniform element. Insulating and radiation shielding can reduce heat loss from each end.
- **Single Zone Profiled:** The heater is wound with essentially one element but the watt density is increased at the ends in order to compensate for heat losses. Differences at different temperatures cannot be adjusted for.
- **3-Zone:** A main heater provides general heating with additional heaters controlled by separate controllers at each end to minimize heat losses out of the ends. This can

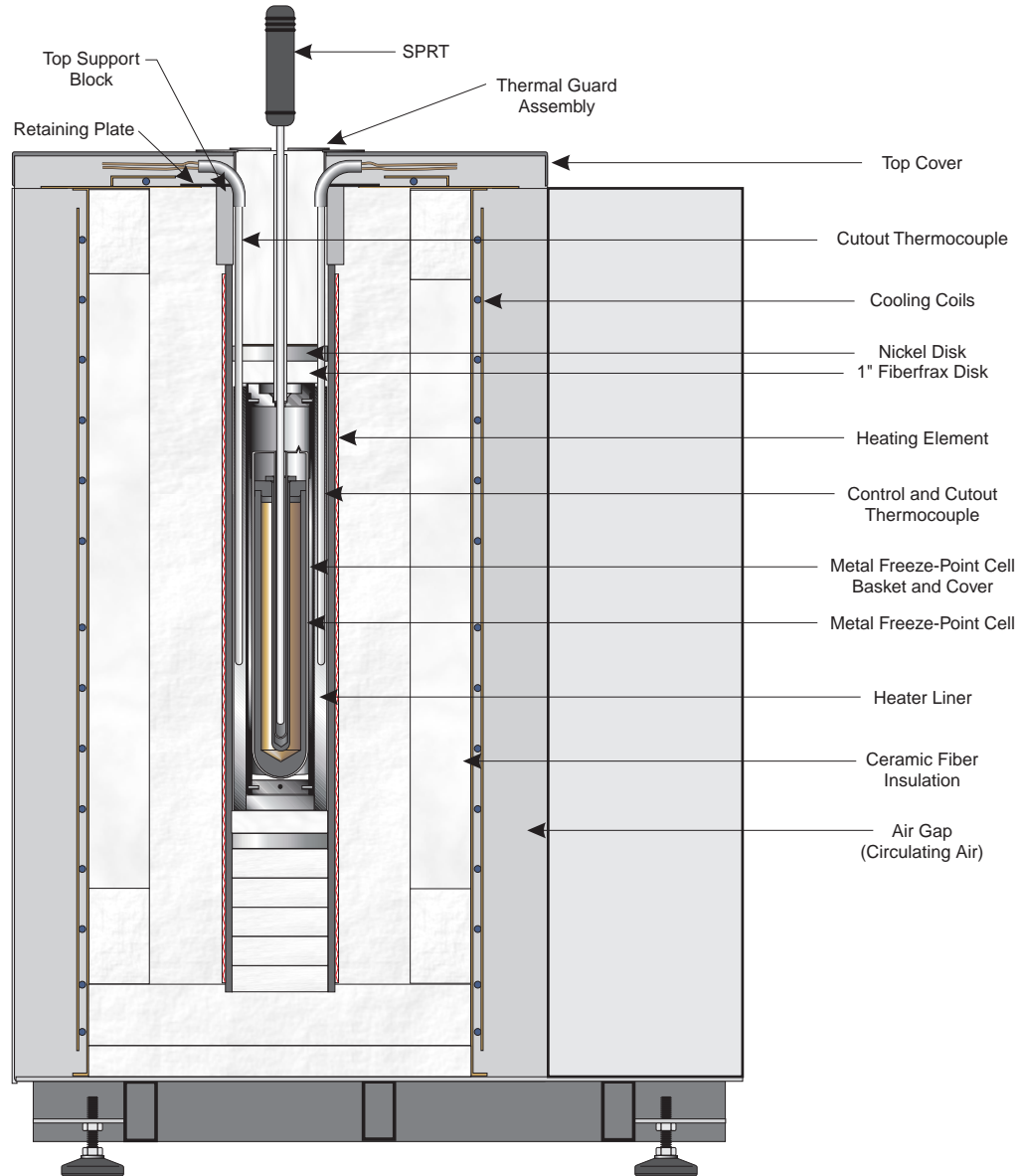


Figure 13. Interior cutaway of a temperature-profiled single-zone furnace.

provide a long, very uniform temperature central zone. Heat loss changes at different temperatures are automatically compensated for.

- Heat Pipe: Typically a single heater heats the heat pipe. The working fluid of the heat pipe will evaporate, efficiently conducting heat along its length and then condense at the other end. The fluid runs through capillary action to the other end by gravity, completing the cycle. The process continues so long as the fluid is being evaporated. This action will provide a uniform temperature over the length of the heat pipe. Working fluids have limited ranges of a few hundred degrees over which they can be used.

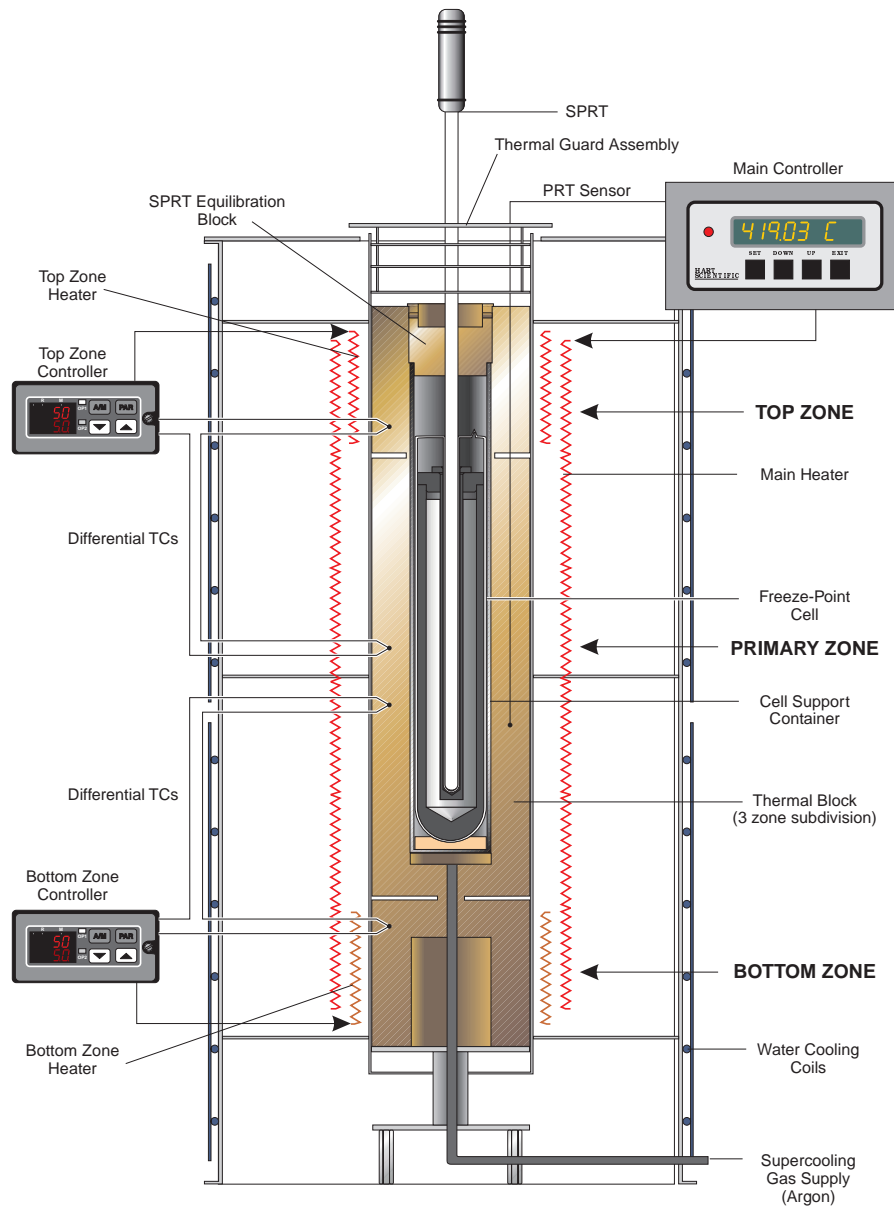


Figure 14. Interior cutaway view of Hart's Model 9114, a three-zone furnace.

The graph shown in Figure 16 on page 21 illustrates data taken from the 4 furnace design types. The superiority of the 3-zone and the heat pipe is clear. The costs are higher for the improved performance, however. Data for the 4 types was not available under identical conditions. The following information explains the differences.

- Single zone furnace: Horizontal design, 700°C, INCONEL equilibration block, TC measurement
- Profiled single zone furnace: Vertical design, 962°C, graphite block, HTPRT measurement

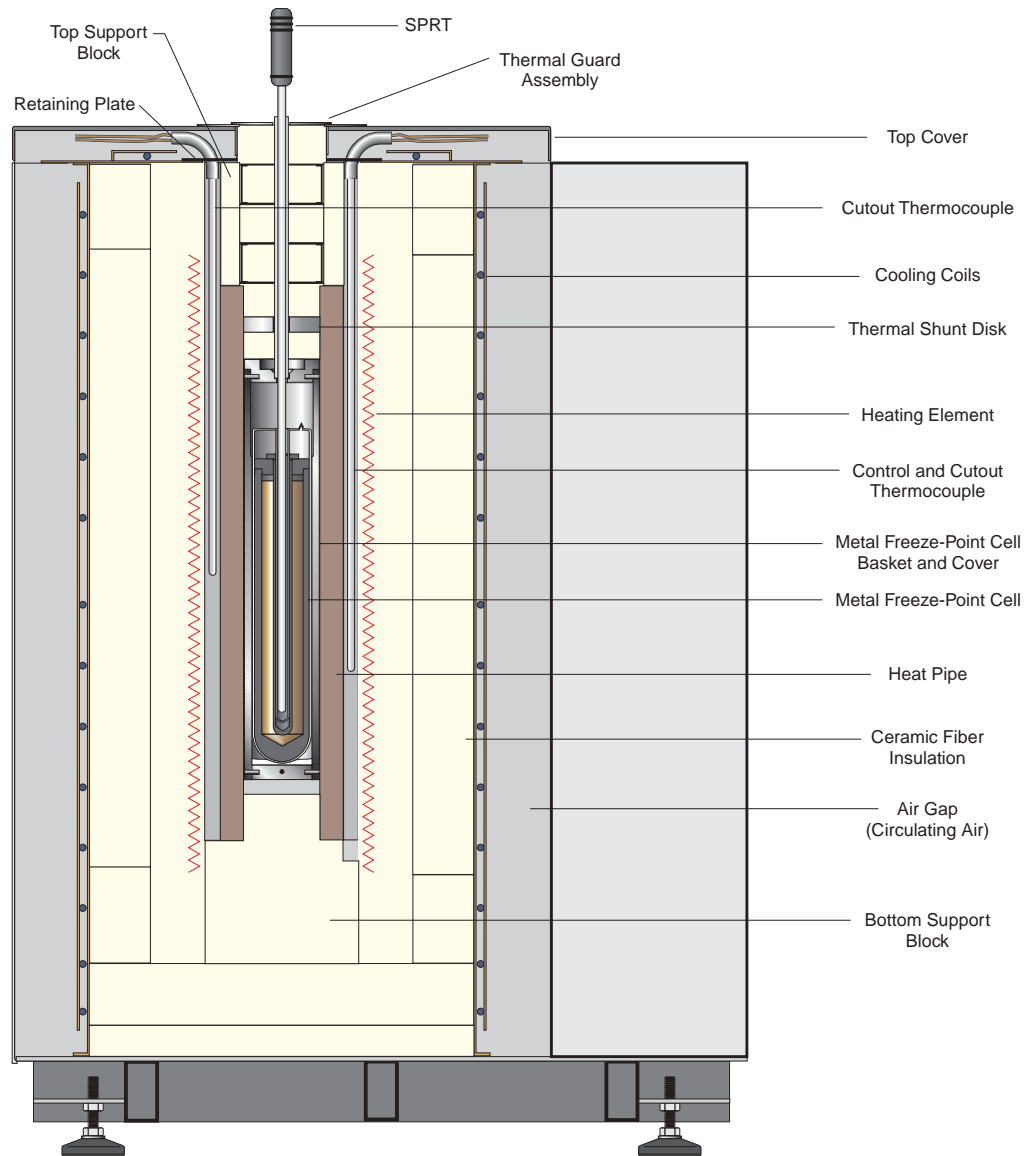


Figure 15. Interior view of Hart's Model 9115 furnace, a sodium heat pipe furnace.

- 3-zone furnace: Vertical design, 662°C, aluminum freeze point cell, SPRT measurement
- Heat Pipe furnace: Vertical design, 900°C, nickel equilibration block with platinum SPRT protection, HTPRT measurement

Equilibration Blocks

The control systems of furnaces may vary depending upon the temperature range. Thermocouple controllers may only provide a stability of $\pm 0.1^\circ\text{C}$ while those using PRTs may provide stabilities of better than $\pm 0.040^\circ\text{C}$. Equilibration blocks are used to increase the stability and

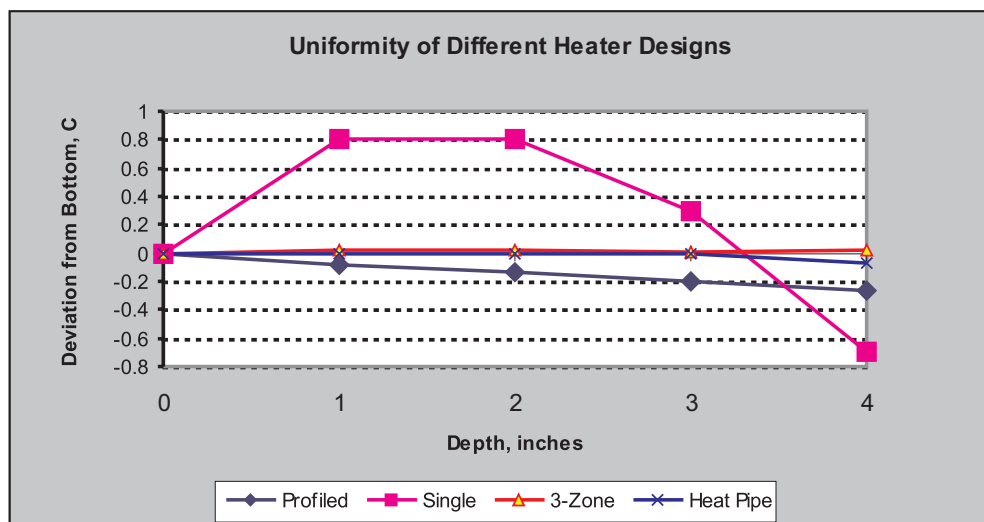


Figure 16. Results of different furnace types.

uniformity of the controlled zone in a calibration furnace. The most highly conductive material that will survive the temperature is typically chosen. Pure oxygen-free copper is common for lower temperatures. Oxidation limits its use so other materials such as aluminum, aluminum-bronze, nickel, and INCONEL may be used. Each of these has increasing oxidation resistance but lowering thermal conductivity. To be effective, the block must be thermally isolated from temperature gradients within the heated zone. Combined uncertainties of stability and uniformity can be ± 1 to 2 mK under ideal conditions.

Ion Contamination

At higher temperatures (above 650°C) metal ions work loose from the equilibration block or other metal components and stream off until they cool or attach themselves to something else. There is a serious danger that the pure platinum thermometer element (either the reference or unit under test) will be contaminated. The platinum element of an SPRT can lose its calibration and be made useless as a precision thermometer in this way. Whether the furnace is used for comparison calibration, preheating, or annealing, the thermometer must be protected from metal ion contamination. This is done by providing some material that is in the path of the metal ions that will prevent penetration of those ions into the thermometer. The quartz glass or alumina surrounding the thermometer element will not adequately protect it. High-density graphite such as is used in fixed-point cells will work, as well as silicon carbide. Another method is to provide a shield made of platinum. The platinum ions driven from it will not contaminate the platinum sensor. It will also stop the ions coming from the equilibration block or other sources.

Furnace Life

Lifetime of the furnace heater elements and heat pipe can be limited, particularly at high temperatures (1000°C and above). Temperature effects such as oxidation corrode the metals increasingly as temperature rises. Heat pipes that have long lifetimes at 900 °C can have relatively short lifetimes at 1000°C. Use of equipment at these temperatures should be limited to that required for the procedure and not be left on unnecessarily.

Immersion Depth

Immersion depth is an important issue with furnaces as it is with baths and other heat sources. Most furnaces are designed with long heated zones to accommodate this need. SPRTs are long as well as many noble metal thermocouples.

Laboratory Furnace Summary

Furnace Advantages

- No messy fluids
- Wide temperature ranges
- Improved uniformity

Furnace Disadvantages

- Some inflexibility for size & shape of thermometers
- Requires long thermometers to reduce stem conduction
- Some inflexibility for quantity of thermometers
- Greater uncertainties than baths but better than dry-wells typically (higher gradients & greater instability)
- Expensive

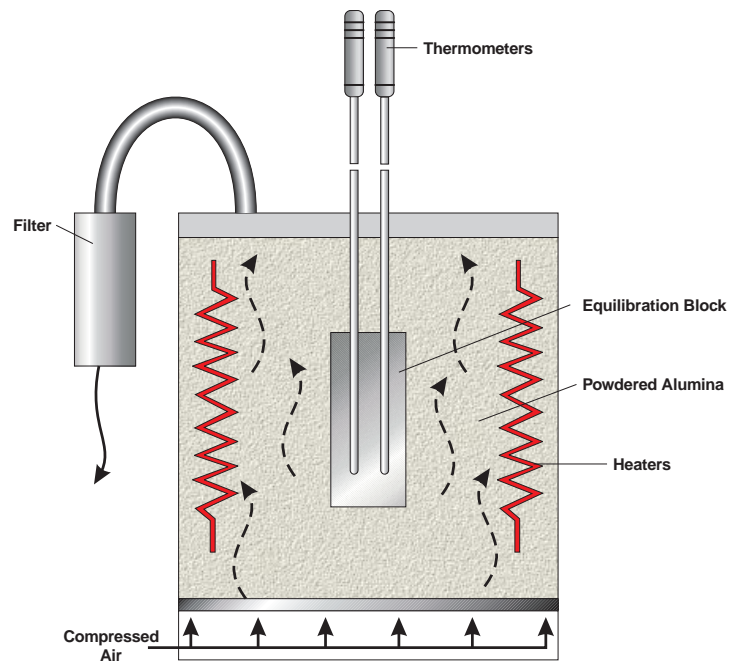


Figure 17. A fluidized bath.

- Slow to stabilize

Fluidized Baths

Fluidized baths consist of a heated tank of a powdered media forced into motion by the action of compressed air. Otherwise, the temperature control process is essentially like that of any bath. The medium is typically a ceramic material such as alumina. The temperature range of these materials is high allowing wide temperature ranges to be achieved without changing the media. Stability and uniformity can only be achieved by the use of an equilibration block. The block can disrupt the flow and cause the powder bed to collapse. Historically, these baths have had the problem of being dirty in that they tend to leak powder into the lab. Some are equipped with filters that remove much of the powder. Additionally, a source of compressed air is required. Stabilities and uniformities vary. Stabilities of a few mK can be achieved by using an equalization block. Salt baths cover most of the fluidized baths range but with improved uncertainties.

Phase change devices

Phase change devices do not directly use temperature controllers and control sensors to maintain a specified temperature. Instead they rely on a change of phase to maintain their temperature. The latent heats involved in changing from liquid to solid or solid to liquid, for example, can provide an extended period of time at a constant and uniform temperature. Examples include:

- Ice Baths



Figure 18. Metal Fixed Point Cells

- Triple Point of Water
- Boiling Liquid Nitrogen
- Dry Ice and Alcohol Bath
- Metal Fixed-points

Metal Fixed-points and the Triple Point of Water

Standard fixed-points such as the triple point of water or metal fixed-points are not normally thought of as heat sources for comparison calibration. However, they provide the most stable sources available. Some labs do not use the intrinsic value of their fixed-point but rather use it as a stable heat source at the fixed-point temperature for comparing thermometers to a NIST traceable SPRT. Extremely low thermal noise can be counted on. Stabilities are sub-mK.

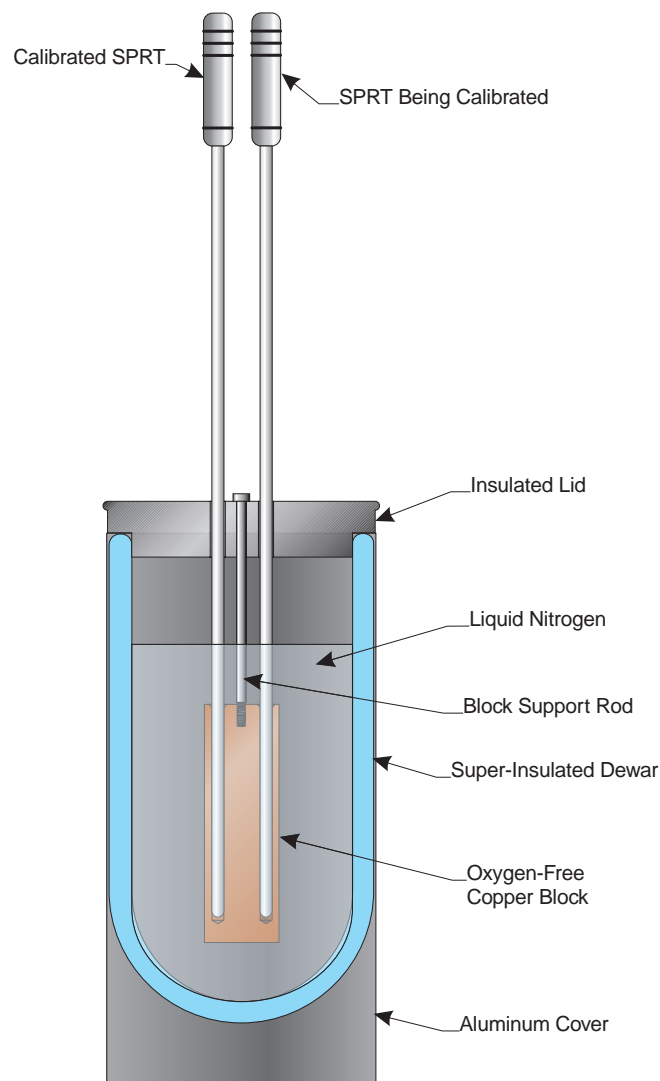


Figure 19. Liquid nitrogen comparator.

Ice Baths

Ice baths depend on the purity of the water and proper preparation and then they become not only a constant temperature bath but also a secondary fixed-point. Under ideal conditions values of 0°C to within $\pm 0.002^\circ\text{C}$ can be reached. Under less than ideal conditions, much greater errors can be found. The proper proportion of ice to water and purity must be maintained. A stirred ice bath might typically achieve 0.005°C .

Liquid Nitrogen Bath

Liquid nitrogen is utilized to realize a temperature near the argon point. Liquid nitrogen boils near -196°C at 1 atmosphere and the argon TP is defined as -189.8344°C .

The extremely low temperature of liquid nitrogen requires using a high-quality covered dewar to insure a usable measurement time. An equilibration block with multiple wells is utilized to compare a traceable reference to the UUTs. The block is usually made from high-purity, oxygen free copper. The block helps to minimize the effects of gradients in the boiling nitrogen. The hydrostatic pressure gradients create thermal gradients within the liquid nitrogen as well. To minimize this effect, the block may be insulated on all but the top surface. In that way, the block “sees” primarily one temperature. The temperature stability is largely dependent on the pressure stability. Sudden building pressure changes or weather changes can effect results.

Isothermal Heat Source Summary

There are a variety of types of heat source instruments that can be used to meet a variety of needs within their design capability. A knowledge of the limitations of each type is critical in making a proper selection for calibration of a particular thermometer. Each type will have its own characteristics. Proper use will depend on a knowledge and application of the thermal properties of that thermal transfer body and the properties of the thermometers involved. It is recommended that first, a careful selection of heat sources be made for the application, second, the instrument be characterized by those using it in order to understand how it will perform under conditions of use, and, third, optimize conditions to get the instrument’s best performance.

Temperature Metrology

Readouts

Part I: RTD and Thermistor Readouts

The fundamental purpose of an RTD or thermistor readout is to measure resistance. However, not every instrument that simply measures resistance is well suited for work with RTDs or thermistors. Many other features ought to be considered when deciding what to use as a readout, such as accuracy. Of course, cost may also be an important issue. Readouts can vary greatly in cost, from a few hundred dollars to tens of thousands of dollars. There is generally a tradeoff between cost and accuracy. When selecting a readout, it is easy enough to determine the maximum allowable cost you can afford. What may not be as easy is determining what accuracy you need in a readout and whether or not you will actually achieve the accuracy you expect.

The following discussion will focus on these issues with the goal of helping you determine what readout is best for your application and how you can achieve the best accuracy possible from your system. First, we will introduce several types of readout devices. Then, we will look at some of the important requirements for RTD and thermistor readouts and consider how various readouts meet these requirements.

Types of Readouts

First, let's look at some of the types of readout instruments that are most often used with RTDs and thermistors. There may be other types we won't discuss, such as an analog ohmmeter, but for modern thermometry requiring accuracies of 0.1°C or better, we recommend digital readouts. They can be grouped into three general categories: digital multimeters, thermometer readouts, and resistance bridges.

Digital Multimeters

Digital Multimeters (DMMs) are general purpose electrical measurement instruments that are capable of measuring voltage, resistance, and usually current, over a wide range of values. They can be and often are used with RTDs and thermistors, though they may not be the best choice for this application. One of their main disadvantages is that they are generally not able to display temperature, only resistance. Another disadvantage is that they offer little or no control of the driving current. Still, they are often used because they are fairly inexpensive—between about \$500 and \$5000—and are readily available. Just a few years ago at Hart we used a Schlumberger Solartron 7081 8-1/2 digit digital multimeter for much of our work with RTDs and SPRTs. DMMs come in a variety of forms and sizes and offer many different features. Accuracies can range from about 1°C to better than 0.005°C.

Thermometer Readouts

By “thermometer readout” we mean a digital readout specifically designed to measure temperature using temperature sensors. Because thermometer readouts are designed for this application, they offer many advantages, especially convenience and accuracy. Being able to

immediately display temperatures is a big advantage. There are a large variety of thermometer readouts available. They span a wide range of price and accuracy. Cost can range from \$500 to \$12,000. Accuracies can range from 1°C to better than 0.001°C.

Resistance Bridges

For the ultimate in accuracy, resistance bridges are used. These can achieve accuracies of 0.0001°C or better. They are used with SPRTs in primary temperature calibration labs where extreme accuracy is required. As expected, you will have to pay for this precision. Resistance bridges can cost up to \$60,000. They are also large and heavy which makes them anything but portable. Resistance bridges only measure resistance ratio. You have to use them with a separate standard resistor. Any resistance and temperature calculations must be done externally. A separate computer is used for this function.

Two types of resistance bridges are widely used: AC bridges and DC current comparators. They both use ratio transformers that are adjusted to match the ratio of the two resistors being compared (the sensor and standard resistor). AC bridges apply AC driving current to the resistors whereas DC current comparators use DC current that is periodically reversed to cancel thermoelectric EMFs.

Readout requirements for RTDs

Now let's look at some of the issues related to making measurements with RTDs and see how well different readouts meet our requirements.

Accuracy and resistance range

All readouts have some limit to the accuracy to which they can measure resistance. This limits the accuracy to which you can measure temperature. The temperature error due to resistance measurement error can be easily calculated if you know the sensitivity of the sensor you're measuring in $\Omega/^\circ\text{C}$. The following equation states the relation.

$$U_T = \frac{U_R}{S}$$

In this equation U_T is the temperature error, U_R is the resistance error, and S is the sensitivity of the RTD. This is about $0.4 \Omega/^\circ\text{C}$ for a 100Ω RTD.

As an example, suppose we are measuring a 100Ω RTD with a readout that has an accuracy of 0.01Ω at the resistance we're measuring. The temperature error is calculated as follows:

$$U_T = \frac{0.01\Omega}{0.4\Omega/^\circ\text{C}} = 0.025^\circ\text{C}$$

The following chart (Figure 20) shows the relationship between readout accuracy and temperature accuracy for various types of platinum temperature sensors.

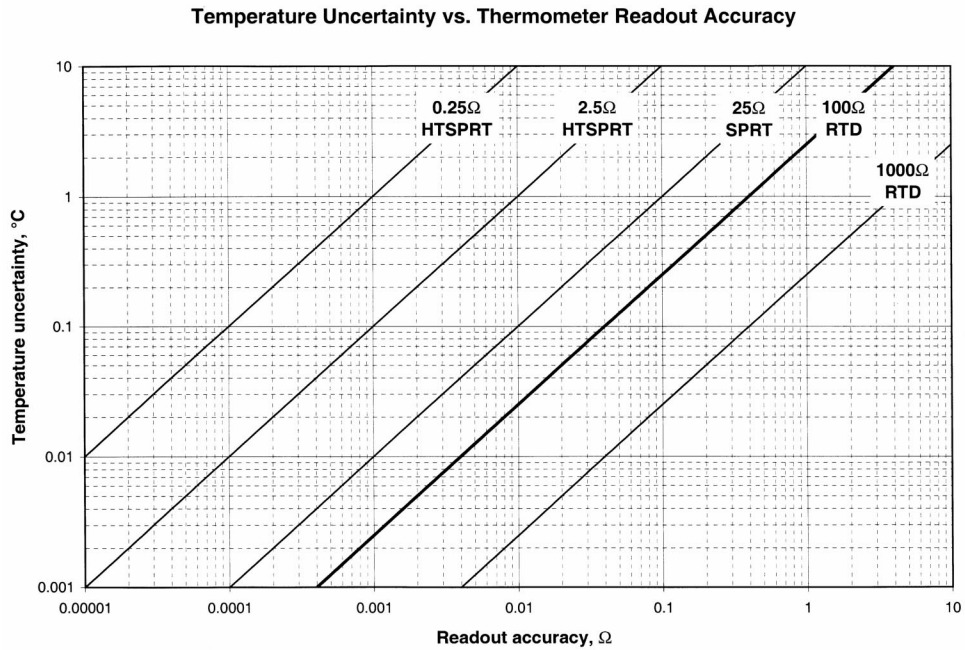


Figure 20. Temperature uncertainty vs. Readout accuracy

The resistance of a typical RTD ranges from about 10Ω to about 400Ω. You must carefully evaluate the accuracy of your readout over this resistance range. Some DMMs may not be very accurate at these low resistances. On the other hand, RTD thermometer readouts are designed for optimum accuracy over this range. Resistance bridges are equally accurate over a wide range of resistances as long as the appropriate standard resistor is used.

The accuracy specifications given with a readout may not show accuracy as a simple resistance error value. Instead, it is often stated as a percent of reading, a percent of full-scale, or a combination of both. For instance, a readout having a full-scale value of 1000Ω may have an accuracy stated as 0.01% of reading plus 0.002% of full-scale. If the resistance of your RTD happened to be, say, 250Ω at the temperature you are measuring, then the accuracy of the readout at this temperature is $0.0001 \times 250\Omega + 0.00002 \times 1000\Omega = 0.045\Omega$. For another example, consider a readout with its accuracy stated simply as 80 ppm. The resistance accuracy at 250Ω is $80 \times 10^{-6} \times 250\Omega = 0.02\Omega$. Some manufacturers may also state different accuracies depending on the calibration interval. Be careful to use the correct specifications based on the time since the readout was last calibrated.

Consider this potential pitfall: many readouts have multiple resistance ranges, each with a different accuracy, and the readout may automatically switch the range while you are making measurements. Many DMMs automatically switch ranges near 100Ω or 200Ω. This is right in the middle of the resistance range of RTDs. The next higher range will likely be ten times greater with an error that is likewise greater. If you are not careful, you may be making measurements with uncertainties that are much greater than you think. Often you will notice when the range switches because the displayed resolution will also change, but don't count on it. A good RTD thermometer readout will maintain excellent accuracy over the full range of resistance you can expect with your RTD. Resistance bridges may have a similar problem in that

the range of ratios they can measure may be limited. Some bridges can only measure ratios less than one. The solution is to select a standard resistor value that is greater than the largest resistance you will measure. Keep in mind, though, that if the standard resistance is too high, the measured resistance ratios will be too low and accuracy will be compromised.

Lead resistance

Consider a sensor with two lead wires as shown in the figure below. The apparent resistance of the sensor is the sum of the resistances of the element and the two lead wires. This will differ from the true resistance of the RTD by the amount of the lead resistance.

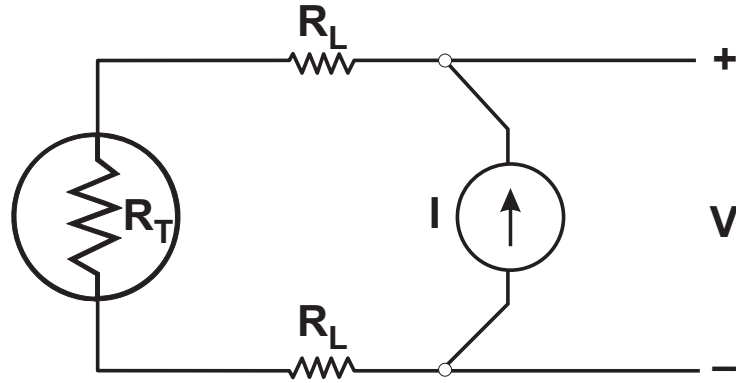


Figure 21. Diagram of a two-wire measurement circuit showing lead resistance error.

The error caused by the lead resistance in a two-wire sensor can be calculated using the following formula:

$$U_L = \frac{r_L}{S}$$

U_L is the temperature uncertainty due to lead resistance, r_L is total lead resistance, and S is the sensitivity of the sensor (again, this is about $0.4 \Omega/^\circ\text{C}$ for 100Ω RTDs).

As an example, consider an RTD at 0°C (100Ω) with 0.2Ω of resistance on each lead wire. The resulting error in the temperature measurement due to the lead resistance is evaluated as follows:

$$U_L = \frac{2 \cdot 0.2\Omega}{0.4\Omega/^\circ\text{C}} = 1.0^\circ\text{C}$$

Lead resistance error can be partially eliminated by calibrating the RTD with the lead resistance included. Still, several factors limit the accuracy you can achieve. First, the added lead resistance changes the resistance-temperature curve so it no longer closely follows the normal characterization equations. Second, the lead resistance will not be very stable. It will change depending on ambient temperature, stress on the lead wires, and quality of connection between the leads and the readout instrument. For these reasons, it is difficult to achieve an accuracy better than about 0.2°C with a two-wire RTD.

To better compensate for lead resistance, many RTDs are constructed with three lead wires. A

special three-wire measurement circuit uses the resistance of one lead wire to cancel the resistance of the others as shown in Figure 22 below.

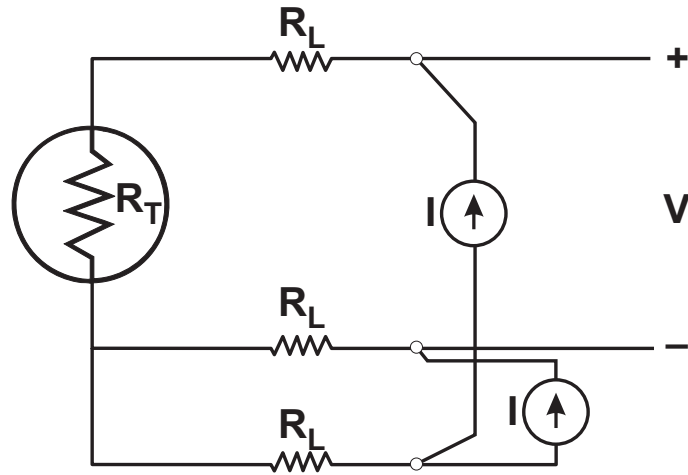


Figure 22. Diagram of a three-wire measurement circuit.

The accuracy of this technique depends on the leads having the same resistance. In reality, the resistance of the leads may differ by as much as 0.01Ω with leads two meters long. This results in temperature errors as much as 0.025°C . With even longer leads, the expected error will be proportionately greater.

Three-wire sensors are intended to be used with specially designed readouts that implement a circuit, such as is shown in the figure above. DMMs, resistance bridges, and many thermometer readouts aren't designed that way. What if you are given the task of calibrating a three-wire sensor but you don't have three-wire readout? It is still possible to determine the true resistance of a three-wire RTD using a conventional two-wire or four-wire readout, though it requires extra steps. It also requires that the readout display resistance instead of temperature. (This may exclude some thermometer readouts.) First, you need to measure the resistance of the RTD. If the readout is a two-wire type leave the extra wire unconnected. If the readout is a four-wire type connect the unpaired wire to two terminals. Next, measure the lead resistance across the two paired wires. Leave the unpaired wire unconnected. Now you must calculate the true resistance using the RTD resistance measurement and lead resistance measurement. If the RTD resistance was measured using a two-wire readout the measured RTD resistance is too high by the resistance of two leads, so subtract the measurement you made of the resistance of the two lead wires. If you are using a four-wire readout the measurement was too high by the resistance of only one lead, so you must subtract only half the measured lead resistance.

To many, this procedure is too complicated and too inconvenient. I certainly agree. It's also quite susceptible to mistakes. There is plenty of opportunity for error in connecting and disconnecting wires, making multiple measurements, writing down the numbers, entering the numbers on a calculator, and performing the math. Furthermore, don't assume that you only need to measure the lead resistance once, and then you can use that value for all subsequent measurements. The lead resistance is very dependent on the temperature you're measuring as well as the ambient temperature. For instance, one three-wire RTD we calibrated had a lead

resistance of 1.435Ω at 0°C . At 200°C it had changed to 1.509Ω . If we didn't repeat the lead resistance measurement at 200°C , we would have seen an error of 0.18°C .

To completely and automatically eliminate any effects of lead resistance, a four-wire or Kelvin circuit is preferred. A diagram of this circuit is shown in Figure 23 below.

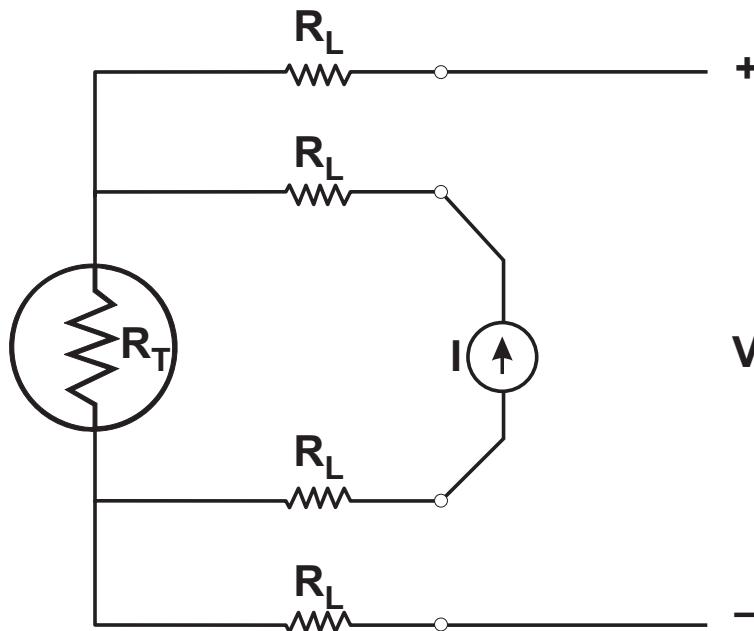


Figure 23. Diagram of a four-wire measurement circuit.

The RTD must have four lead wires—two wires attached to each end of the sensor. Current is driven through two opposite leads and the potential is sensed through the other two wires. As long as the current through the potential leads is negligible, the potential sensed is the same potential at the element with no error at all caused by lead resistance. All high quality RTDs, SPRTs, and thermistors are constructed with four lead wires. The best DMMs, thermometer readouts, and bridges all use four-wire inputs.

Thermoelectric EMF

Thermoelectric EMF (electromotive force) is caused by temperature differences between junctions of dissimilar metals. The result is a small offset voltage, like a tiny battery connected in

series with the wires. RTDs are susceptible to this effect. The most significant thermoelectric EMFs are generated where the platinum element connects to copper lead wires (see Figure 24).

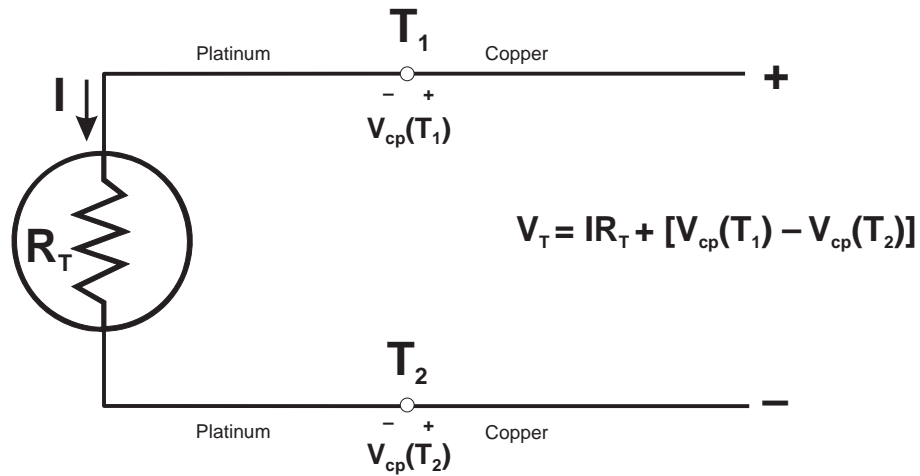


Figure 24. Diagram showing thermoelectric EMF.

A platinum-copper junction has a thermoelectric EMF that changes about 7 mV per °C. If the excitation current is 1 mA, a 7 mV error will appear as a 0.007Ω error. This is equivalent to a temperature error of 0.018°C for a 100Ω RTD. Now this is only the error caused by a 1°C difference between junctions. It can actually be much more than that at high temperatures. We have seen actual thermoelectric EMF errors as much as 0.1°C with some RTDs at 400°C.

In the construction of high-quality RTD probes, precautions are taken to reduce the number of junctions and to make sure opposite junctions are near the same temperature. Even still, a certain amount of thermoelectric EMF will always exist. Preferably, the readout itself should incorporate techniques to cancel thermoelectric EMF. One way this is done is to measure the EMF with the driving current switched off and subtract this from the voltage measured with the current applied. DMMs sometimes use this technique to produce a “true ohms” measurement. One problem with this method is that the magnitude of the current is not constant and self-heating effects can cause errors as we will discuss shortly. A better way to cancel thermoelectric EMF errors is to make two measurements of the resistance, one with the driving current in one direction and the other with the same magnitude of current but in the opposite direction. Averaging the two measurements cancels the error. DC current comparators and some thermometer readouts use this technique. With AC bridges, thermoelectric EMFs are automatically ignored as a result of the AC measurement system, so no extra cancellation techniques are required.

You should be aware of the technique used by your readout. If it uses strictly DC, you should know how much error is caused by thermoelectric EMF. You can measure the EMF using a microvoltmeter or you can swap connections around and observe the differences you see in the measurements. Test for thermoelectric EMF at the highest and lowest temperatures at which the RTD will be operated since those are the temperatures at which you will see the greatest errors.

Electrical current and self-heating

As we all know, the amount of electrical power dissipated in a resistor is the resistance times the square of the current. This fact is important in resistance thermometry because current is necessary to measure the resistance of the sensor. The power that results heats up the sensor, making its temperature higher than the medium you're trying to measure. This is called self-heating. Not only is the self-heating error dependent on resistance and current, but it is also dependent on the medium—specifically, how well the medium dissipates the heat from the sensor. Given the same current and resistance, self-heating will be much greater when you attempt to measure the temperature of an insulating substance such as air than it will be when you measure the temperature of something like stirred water. The following equation can be used to calculate self-heating error.

$$U_H = \frac{i^2 R}{\delta}$$

In this equation U_H is the temperature rise due to self-heating, i is the current, R is the resistance, and δ is the dissipation coefficient.

A requirement for making accurate measurements with resistive sensors is to limit self-heating to a negligible amount or at least in some way cancel the effect of the error. You should not use more current than is recommended for the RTD. With 100 Ω RTDs this is often 1 mA. Since self-heating is dependent on the square of the current, using twice the normal current increases the self-heating error by a factor of four. Using ten times the normal current increases self-heating by a factor of 100. An RTD that has a self-heating effect of 0.01 $^{\circ}\text{C}$ with 1 mA would have an error of 1 $^{\circ}\text{C}$ with 10 mA. This much current not only causes an extreme amount of self-heating error but it might actually cause damage to the sensor.

Conversely, if an RTD calls for 1 mA and you use 0.5 mA instead, the self-heating would be four times less. Wouldn't that be better? Not necessarily. One reason not to have the current too low is because electrical noise becomes more significant. Smaller current produces smaller voltage and thus a lower signal to noise ratio. This reduces the accuracy of the measurement. Assuming the readout is still able to achieve adequate accuracy with less current, isn't less current and less self-heating better? Consider the calibration of the RTD. Suppose 1 mA was used during the calibration and the resulting self-heating effect was 0.004 $^{\circ}\text{C}$. Subsequently, you use it with a readout that drives it with 0.5 mA. The self-heating is then only 0.001 $^{\circ}\text{C}$. The temperature you read will be 0.003 $^{\circ}\text{C}$ lower than it ought to be. In many applications the error that results from using too low of current is negligible. Still, as a general rule, you should use the same current that was used during calibration. However, even this rule may have exceptions.

I ran into a problem once when I was testing a temperature-controlled air chamber. When I measured the temperature with an RTD I measured 0.025 $^{\circ}\text{C}$ higher than with a thermocouple. Comparing the RTD and thermocouple in a water bath, they measured the same temperature. The problem was caused by differences in the dissipation coefficient. The dissipation coefficient of an RTD inside an insulating substance, like air, is much smaller than inside a stirred liquid. In order to accurately measure the temperature of the air with the RTD, I had to reduce the current well below the normal 1 mA.

Ideally, your readout should allow you to set the current to anything you like. From what we've discussed, there are several reasons for this. First, having variable current gives you a way to measure the self-heating error. By changing the current from, say, 1 mA to 1.414 mA and observing the change in the measurement you can determine just how much self-heating there is at 1 mA. Second, different sensors may require different currents. Some RTD probes may work best with 1 mA while others may need 0.5 mA. This depends on the dissipation co-

efficient as determined by the size and construction of the RTD. Finally, you may need to change the current depending on the medium you are measuring since the medium also affects the dissipation coefficient.

Consider these issues when selecting a readout for your RTDs. Does the readout apply the right driving current for your RTD throughout the range of resistances you will measure? DMMs not designed for RTDs very well may not. One particular older DMM operates with a current of 10 mA. This could damage an RTD designed to operate with 0.5 or 1 mA. The readout's manual will usually tell you what level of current it uses. If in doubt, you can measure it. Connect the readout to a 100 Ω resistor and measure the voltage across the resistor with a handheld digital voltmeter. Dividing the voltage by 100 gives you the current. Some readouts may use the right current, but it might not be constant. For instance, our Schlumberger Solartron DMM uses 1 mA but the current is switched off half the time to allow it to cancel thermoelectric EMF. This results in a self-heating effect that is somewhere between that of no current and a constant 1 mA. Also, be aware that the readout may have multiple ranges, each with a different driving current. Will the readout allow you to change the current so you can measure the self-heating error? Will it let you use a lower current for applications where the dissipation coefficient is small? Few, if any, DMMs will allow you to change the current. Many thermometer readouts don't even have this feature. Most bridges and a few good thermometer readouts will allow you to select various values of current.

Temperature calculation

At some point in the use of an RTD the measured resistance must be converted to a temperature value. After all, the whole purpose of RTDs is to measure temperature. You can use a variety of methods to determine temperature from the resistance. One way is to use a resistance-temperature table. This method is not very accurate (unless you use interpolation), is subject to easy mistakes, and is slow. You could use a calculator instead but this is also subject to mistakes and can be very slow and difficult with the more complicated equations. Computers are best suited for the task. Since most readouts already have built-in microprocessors, why not have them perform the calculations for you automatically? Thermometer readouts are programmed to calculate temperature using algorithms such as Callendar-Van Dusen or ITS-90. Most will allow you to enter special coefficients for your probe. Most DMMs are not really intended to measure temperature so they generally lack this capability. Resistance bridges measure resistance ratio only. With these devices you will need to use a separate computer to calculate resistance first, then temperature.

Multiple channels

Some applications require you to work with a large number of multiple sensors. A desirable feature is the ability to connect up and automatically scan multiple sensors. Dedicated automatic switches are called scanners or multiplexers. These work in conjunction with the readout to allow you to measure many sensors in a short amount of time without having to exchange connections. Some readouts may have multiple channels built in. Others may have the capability to connect with and control a separate multiplexer. Some may have no multiplexer capability at all but can be controlled remotely by a computer while the computer also controls a multiplexer.

Speed of measurement

Another consideration when working with a large number of RTDs is speed of measurement. Not only can this improve work efficiency, but it can lead to better results since comparison measurements can be made close together. Bridges can be very slow, requiring one minute or more after switching channels to make a measurement. Most DMMs and readouts can make measurements in 1 to 20 seconds.

Other convenient features of readouts

Following are several other features which can add convenience to a readout.

We mentioned before that resistance bridges measure resistance ratio only and must be used with a separate standard resistor. DMMs and thermometer readouts do not require any external resistors.

In many cases, such as the calibration of RTDs, your work can be made much more efficient by automation. Having a digital interface such as RS-232 or IEEE-488 (GPIB) allows a readout to be controlled by a computer in an automated system.

Some readouts have graphic displays that can show several readings simultaneously either in numeric form or graphic form. This can be helpful in quickly visualizing temperature trends and variations.

Readout Requirements for Thermistors

Many of the same issues discussed for RTD readouts apply equally well to thermistor readouts. However, the wide resistance range and other characteristics of thermistors require some special considerations.

Resistance range and accuracy

Thermistors have a much wider and higher range of resistance compared to RTDs. A readout designed for RTDs may not work at all with thermistors. There are some readouts specially designed to work with both.

Thermistors come in a variety of nominal resistances (usually, its resistance at 25°C) but the most commonly used thermistors have a nominal resistance of about 10 k Ω . The actual resistance will vary from about 300 Ω at 150°C to 1 M Ω at -50°C. This presents a challenge to the readout to accurately measure resistance over such a wide range.

Fortunately, the increased sensitivity of thermistors makes the accuracy of the readout less critical. This is one of the greatest advantages of thermistors. A typical thermistor has a sensitivity of about 400 Ω /°C at 25°C. A readout with an accuracy of 0.01% would give you a temperature accuracy of 0.0025°C. Compare this to what you would get if you used a readout of this accuracy with an RTD: 0.025°C.

When using DMMs with thermistors the DMM is more likely to automatically change range while you're making measurements. If you have to change the range manually, be sure you are using the right range or the accuracy may be poor.

Lead resistance

Lead resistance with a thermistor is much less of a problem than with an RTD for two reasons. First, since the thermistor's resistance is higher, the relative magnitude of the lead resistance is much less. Second, the sensitivity of thermistors is higher so the same resistance error causes less temperature error.

Consider a thermistor at 25°C. Its sensitivity at this point is about 400 Ω /°C. Suppose there is 0.2 Ω of resistance on each lead wire. The resulting error in the temperature measurement due to lead resistance is evaluated as follows:

$$U_L = \frac{2 \cdot 0.2\Omega}{400\Omega/^\circ\text{C}} = 0.001^\circ\text{C}$$

Compare this to the previous example of the RTD where the lead resistance caused an error of 1°C. This may be typical of the error you might see with a two-wire thermistor, but the error can be larger. At higher temperatures where the resistance and sensitivity are much lower, the error can reach 0.01°C or more. For best results, thermistors should also be constructed and operated with four lead wires to completely eliminate lead resistance errors.

Current and Self-heating

The same self-heating issues we discussed relative to RTDs apply to thermistors except thermistors are generally more susceptible to self-heating. There are two reasons for this. First, they have a much higher resistance and, as you remember, self-heating increases with resistance. Second, they often have a much lower dissipation coefficient, which is because the thermistor's sensing element is much smaller. As a result, the driving current must be much smaller with a thermistor. Compared to a 100Ω RTD that operates with 0.5 or 1 mA, a thermistor requires about 10 μA. At the lowest temperatures where the resistance is very high, even 10 μA may cause too much self-heating. 5 μA or even 2 μA may be more appropriate.

Make sure your readout uses the proper current over the range of resistances it will measure from the thermistor. The wide variation of resistance in a thermistor makes this a likely problem, especially with DMMs. Our Schlumberger Solartron DMM uses 10 μA above 10 kΩ but below 10 kΩ it automatically switches to a lower range that uses 1 mA. That's 10,000 times the normal self-heating! Be careful! Thermometer readouts designed for thermistors will more likely operate with the proper current.

Temperature Calculation

Thermistors require special algorithms to calculate temperature from resistance. Equations normally used with RTDs such as ITS-90 or Callendar-Van Dusen do not match up well with the extreme nonlinearity of thermistors. A much better characterization of a thermistor's resistance is given by the Steinhart-Hart equation:

$$R(T[K])[\Omega] = \exp(b_0 + b_1 T^{-1} + b_2 T^{-2} + b_3 T^{-3})$$

The temperature calculation may be done separately after the resistance is measured. As with RTDs, a resistance-temperature table or computer may be used. But again, the quickest and most convenient method is to have the readout do the calculation automatically and display the temperature directly. DMMs and most bridges do not have this capability, especially with thermistors. Only readouts specifically designed to be used with thermistors will be able to display temperature. These will let you enter the special Steinhart-Hart coefficients for your thermistor and automatically apply them in the equation each time a new resistance is measured.

Part II: Thermocouple Readouts

Thermocouples basically require a readout that measures voltage. To be able to achieve reasonable accuracy, though, some special issues must be considered. In this section we will discuss some of the important requirements of readouts for use with thermocouples. We will begin by introducing several types of readout instruments that might be used.

Types of Readouts

Most anything that measures voltage can possibly be used with thermocouples. Practically, though, of the devices best suited for this application, there are three types: DMMs, thermometer readouts, and potentiometers.

DMM

As explained in the section on RTD readouts, DMMs can measure voltage as well as resistance. You might actually use the same DMM to measure RTDs, thermistors, and thermocouples. They often are able to measure voltage even more precisely than they can resistance. When used with thermocouples the accuracies DMMs can achieve range from about 3°C to as good as 0.02°C. Other advantages DMMs may have are low cost and availability. Disadvantages, as we will explain in more detail soon, are lack of capability to display temperature, and no built-in automatic cold-junction compensation.

Thermometer Readout

There are many thermometer readouts available that are designed specifically for thermocouples. For reasonably low cost you can get a convenient, compact thermocouple readout that displays actual temperature. The accuracy it provides may be much better than a DMM of comparable cost. Unlike DMMs, thermocouple readouts offer built-in cold-junction compensation.

Potentiometer

Voltage potentiometers are used for the most precise thermocouple work. They measure voltage with accuracies as good as 0.05 μV (equivalent to about 0.002°C). Because potentiometers are very expensive, as well as bulky and difficult to operate, they are not used very often for general thermocouple measurements.

Thermocouple Readout Requirements

Now we'll look at some of the characteristics and requirements of thermocouples and consider how well various readouts might work for our application.

Voltage range and accuracy

The accuracy to which the readout measures voltage limits the accuracy of your temperature measurements. Again, the formula that relates the two is

$$U_T = \frac{U_R}{S}$$

where U_T is the temperature error, U_R is the resistance error, and S is the sensitivity of the thermocouple. This is about 0.04 mV/°C for a type K thermocouple. The sensitivity can range from about 0.01 to 0.08 mV/°C for other types of thermocouples.

As an example, if a readout has an accuracy of 0.001V the temperature accuracy with a type K thermocouple would be

$$U_T = \frac{(1\text{mV})}{(0.04\text{mV}/^\circ\text{C})} = 25^\circ\text{C}$$

Yes, that's 25°C! You can see that because the sensitivities of thermocouples are so low it takes a pretty accurate readout to give you reasonable temperature accuracy. The average DMM won't cut it. To give you accuracy around 1°C the readout must be accurate to 0.00004V. The voltages measured with thermocouples are low—typically, in the range of 0 to 50 mV. Designing a readout just for this limited range makes it somewhat easier to achieve the necessary accuracy. This is one reason why many thermocouple readouts can give you better accuracy than a DMM for comparable price.

When evaluating temperature accuracy be sure to consider the sensitivity of the thermocouple type you are using. The best place to find this is in the thermocouple tables. Take two voltage values one degree apart near the temperature your measuring and subtract them to figure the sensitivity. Realize that the sensitivity may change significantly depending on temperature.

Thermoelectric EMF

With the low sensitivities of thermocouples, it doesn't take much voltage error to destroy your accuracy. Ironically, the same principle by which thermocouples operate can also cause error. Any additional junctions, including multiplexer switch contacts, between wires of dissimilar metals will produce unwanted EMFs. Unlike the case with RTDs, there is no way to do something like vary the current to cancel spurious EMFs. The readout can't distinguish the undesirable EMF from the desirable EMF of the thermocouple. It's necessary to keep the number of junctions to a minimum. If additional junctions are required you must make sure that the two wires forming the junction are made from identical material.

Cold-junction compensation

No matter what, there are always going to be at least two additional junctions besides the thermocouple itself. You have to connect the thermocouple to the readout somehow. Also, these junctions will have to be between wires of different materials. The wires of the thermocouple are made from certain types of materials and they must contact the copper wires leading to the measurement circuit of the readout. These connections are called the *reference junction*. In short, you are always going to have spurious EMFs. This is a fundamental issue with thermocouples. Some method must be used to cancel the reference-junction EMFs. This is called *reference junction compensation (RJC)* or *cold-junction compensation* or *CJC*.

One way CJC can be accomplished is as follows. First, we measure the exact temperature of the reference junction. Using this temperature we calculate the EMF at the reference junction using the table or formulas that characterize the thermocouple. Then, we subtract the reference-junction EMF from the voltage the readout measures.

Using thermocouples would be very inconvenient if we had to go through this process each time we made a measurement. Fortunately, thermocouple readouts can do this automatically. A sensor is placed near the input connections of the readout. The readout senses the temperature of the junctions, computes the temperature either digitally or using analog circuitry, and subtracts the corresponding voltage from the input voltage. This process, which we call *internal RJC* or *CJC*, is outlined in Figure 33 on page 74.

A limitation to the accuracy of this method is the accuracy with which the readout can determine the temperature of the junctions. Any temperature gradients between the junctions and the sensor directly affect the accuracy. Even though the sensor may be placed close to the junctions, differences in temperature of tenths of a degree are still likely to occur. Also, there is a limit to the accuracy with which the readout can measure the resistance of the sensor. There is also a limit to its ability to predict the resistance-temperature relationship so that it

can accurately compute temperature from resistance. For these reasons, readouts using this method are typically limited in accuracy to no better than about 0.1°C.

There is a better way to perform RJC. Rather than try to measure the reference-junction temperature we can place it at a certain known temperature. We will then automatically know the temperature and we can easily determine the EMF of the reference-junction. Furthermore, making the temperature constant means we only have to determine the EMF once. Now, let's go a step further. If we hold the temperature of the cold junction at the temperature at which the thermocouple characterization is referenced (0°C) then the contributing EMF of the reference junction will be 0. No EMF calculation and no subtraction will be necessary. This is the basis for external RJC, again, see Figure 33 on page 74.

When using external RJC the cold junction is placed in something like an ice bath that keeps it at 0°C. This method is used to achieve the best accuracy possible with thermocouples. Obviously, though, it has its drawbacks. Requiring an ice bath and having to place the reference junction inside it can be very inconvenient. Anyway, for many applications the 0.1 to 1°C possible with internal RJC may be adequate. If this is true in your case then you won't want to use DMMs or potentiometers. Neither will allow automatic internal RJC. On the other hand, thermometer readouts designed for thermocouples generally have built-in CJC.

What would happen if you didn't do any reference junction compensation? How accurate would your thermocouple be? Generally, the error you would see is about the same as the temperature of the room, in degrees C. If you were measuring 500°C and the room temperature was 25°C the voltage you measured might indicate about 475°C. If this is accurate enough then maybe you don't need RJC. There are some applications, such as pilot flame detectors, where the cold-junction error is not significant. In some cases the error can actually be much less. This is because the sensitivity of some thermocouples is much lower near room temperature than at the normal temperatures they are designed to measure. In fact, type B thermocouples have a sensitivity of nearly 0 at room temperature so no RJC is required.

Temperature calculation

Thermometer readouts offer another significant advantage over DMMs and potentiometers. They can display temperature directly. DMMs can only give you the voltage and potentiometers only show you voltage ratio. Thermometer readouts let you select among various types of thermocouples and perform all the calculations, including RJC, to convert the measured voltage to temperature. Some may even let you enter adjustment factors or calibration parameters to let you improve the accuracy of your thermocouple.

Other thermocouple issues

Finally, there are a few other issues that we should at least mention so problems can be avoided.

Thermocouple wires will have a certain amount of resistance. The resistance increases if the wires are made thinner or longer. Any current passing through the wires will cause a change in the voltage seen by the readout. To avoid errors caused by the wire resistance, the readout should not draw any significant current. Check the input impedance of your readout to make sure it is adequately high. If you can determine how much current there is and if you know the resistance of your thermocouple you can calculate how much voltage error it causes and how much temperature error results from it.

Another cause of current might be ground loops. If the thermocouple is attached to ground near its hot junction and also at the readout, any voltage potential between the two ground points will drive current through the thermocouple wires. This can cause error in the voltage.

To prevent this, the circuit should only be grounded at one point. Many thermocouples are grounded because of the way they are designed or installed. This means that the readout circuits should be isolated from ground.

Error can also be caused by electrical interference. High frequency signals reaching the thermocouple wires can travel down the wires to the readout and interfere with the measurement circuitry causing loss of accuracy. Interference (often referred to as EMI or RFI) can be avoided by shielding the thermocouple wires and connecting the shield to ground. If this is not practical, it may help to connect the thermocouple circuit to ground at some point to provide an easy path for the current to reach ground, thus steering it away from the measurement circuitry. If the readout inputs are floating and the thermocouple is also isolated from ground, you may have to intentionally connect a wire from one of the thermocouple wires to ground.

Calibration Techniques

Common Calibration Techniques

Introduction

All common thermometers are temperature transducers. They exhibit a change in an electrical or physical parameter which is proportional to a change in temperature. To calibrate such a device is to establish and/or define the relationship between temperature and this parameter. For a thermometer to be useful, this relationship must be stable and repeatable. There are many types of instruments which, to a greater or lesser degree, fulfill these requirements. In the following section, we will discuss several of the types of thermometers introduced previously from the point of view of calibration requirements. First, we will review some characteristics of the instrument being discussed and what we must quantify with regard to these characteristics, as well as how these characteristics influence our choice of calibration method. Next, we will detail the instruments, standards, and apparatus necessary for the calibration. Finally, we will describe the actual procedure recommended for calibration of the instrument. The sections are intended to be complete in themselves and as a consequence there will be repetition between sections.

Platinum Resistance Thermometers

Characteristics

A platinum resistance thermometer (PRT) is a thermometer constructed from a high purity platinum element (wire-wound coil or thin film) placed in a tube of metal or glass and sealed with an inert atmosphere and/or mineral insulator. Two, three, or four leads are connected to the element and are used to provide for the measurement of the electrical resistance of the element. PRTs are one of the most commonly used thermometers for temperature measurement in the field and in the laboratory because they have characteristics which make them particularly well suited for applications at all levels of accuracy. In many cases, the reference probe being used in the calibration laboratory is a specially constructed and calibrated PRT called an SPRT (standard platinum resistance thermometer). In this instance, the word “standard” is used to denote a specific construction and behavior, not a position in measurement hierarchy. The specific requirements for the SPRT were discussed previously, so we will not review them here. However, keep in mind that many of the characteristics which make the PRT attractive as a temperature sensor also enable it to be defined in such a specific way as in the case of the SPRT. Some of these characteristics are:

- Electrical parameter is resistance
- Wide temperature Range (-260°C to 1000°C)
- Stable over time
- Stable over temperature

- Well defined mathematically
- Relatively linear
- Shallow slope (low $\Omega/^\circ\text{C}$)
- Relatively easy to measure
- Relatively easy to calibrate
- Commercially available in many configurations

These characteristics benefit the user, the metrologist, or both. All of these characteristics contribute to the popularity of the PRT as a measurement instrument and as a calibration standard and make it suitable for a wide variety of tasks.

The list above shows that PRTs are suitable for use over a wide temperature range. This is true, but actual design and construction will differ in instruments intended for different ranges. No single instrument will be suitable for use over the entire range shown above. In calibration, the electrical resistance is measured at several temperature points and fitted to a mathematical expression. The number of calibration points depends on the range and accuracy desired but, because the temperature response of platinum is relatively linear and very well known, fewer calibration points are required for a given range compared to other sensor types. Also, because of the shallow slope, the readout used for the resistance measurement need not have a large range. PRTs, like any probe, have immersion requirements which vary from configuration to configuration. Often, the required immersion is not stated or specified. Since PRTs are used in so many different applications, we are presented with a large variety of shapes, sizes, and types. Although the basic calibration requirements are the same, these various configurations pose different problems in the laboratory. We must solve these problems satisfactorily to provide a proper calibration. Therefore, we must understand the requirements to an extent that allows us to adapt our process, if necessary, to accommodate a new or unusual configuration.

Instruments, Standards, and Apparatus

Calibration is performed by measurement of the resistance of the unit under test (UUT) while it is exposed to a temperature. Fundamentally, four instruments are required as follows:

- 1) Reference probe
- 2) Readout for the reference
- 3) Readout for the UUT
- 4) Temperature source

Reference Probe

Depending upon the accuracy required, the reference probe will be either an SPRT or a PRT of better quality and calibration than the UUTs. Since this instrument forms the basis for our calibration, its accuracy and stability are of paramount importance.

SPRTs

SPRTs are the most accurate and stable instruments available for this purpose. They are generally available in 0.25, 2.5, 25, and 100 Ω versions with either borosilicate glass (Pyrex), fused

silica glass (quartz), stainless steel, or INCONEL sheath materials. The different resistance values and different sheath materials are intended for different temperature ranges. A typical quartz sheathed 25Ω SPRT will have a temperature range of –200°C to 660°C and with a high quality calibration will have calibration uncertainties from 0.001°C to 0.010°C. Additionally, since these instruments are actually part of the definition of the ITS-90, they are standardized. That is, there are minimum requirements for the purity of the platinum wire and the type of construction used. This results in less confusion as to the suitability of the instrument for a particular application and almost guaranteed good performance if calibrated and used correctly. These instruments are highly stable and accurate, but they are expensive and extremely delicate. They should be reserved for high accuracy applications only.

PRTs

When accuracy requirements are less severe, PRTs can be used successfully. As mentioned, PRTs are available in many configurations, however PRTs which are suitable for use as calibration standards are generally available as 100Ω stainless steel sheathed probes. Historically, they have been limited to a temperature range of –200°C to 420°C but a new type has been introduced which has extended the upper limit to 660°C. Calibration uncertainties range from 0.010°C to 0.025°C. These instruments are not as accurate as SPRTs but they are generally more rugged and easier to work with. Additionally, unlike SPRTs, the design of PRTs is left to the discretion and ingenuity of the manufacturer. Not all designs perform to the level required for use as a reference. Be careful in the selection of a PRT to ensure that the type selected is appropriate for use as a calibration reference over the range of interest and with the required accuracy.

Special Considerations

In addition to accuracy requirements, there are other characteristics which must be considered. For example, PRTs which are not hermetically sealed will allow moisture ingress and will not perform well below 0°C regardless of what is stated in the specifications. Also, not all 25Ω SPRTs are capable of reaching 660°C without damage. Instruments with mica components will begin to deteriorate when exposed to temperatures greater than 500°C. Also, some SPRTs and PRTs have flaws which affect the accuracy of the measurement. For example, there is a specific SPRT design in which the platinum element is so well “insulated” from the outside of the sheath that correct immersion is not possible. This SPRT would cause large measurement errors which would be transparent to the user. Also, consider how the instrument is to be used. If high accuracy measurements are to be performed in a dry-well heat source, a metal sheath SPRT is a better choice than a glass sheath model. These points and others must be understood and taken into account in the selection of a reference probe.

Table 1. Reference Probe Characteristics Summary Table

SPRT	PRT
Capable of very high accuracy	Capable of moderate to high accuracy
Capable of large temperature range	Capable of large temperature range
Extremely stable	Very stable
Available in various resistance values	Resistance values limited
Available in various sheath materials	Configurations limited
Standardized	Not standardized
Relatively expensive to purchase	Relatively inexpensive to purchase
Relatively expensive to calibrate	Relatively inexpensive to calibrate
Extremely delicate	Less delicate

Readout

Readouts were covered in detail previously, so we will not go into individual characteristics or performance here. We will review some of the main considerations. First, when calibrating PRTs against a reference PRT or SPRT, the technical requirements for the readout are the same for the UUTs and the reference. If a switching system is available, one readout can usually be used for both. If the readout is designed for temperature calibration (not just temperature measurement) and has variable settings (current, timing, etc.), then certainly it can be used for both. If the readout is not designed for temperature calibration and/or a switching system is not available, then two or more readouts will probably be required. Before selecting a readout, review the information presented in the readouts section with regard to current settings, timing, multiplexing, etc. Best results will be obtained with readouts designed specifically for thermometer calibration. DMMs and bridges severely limit the flexibility, with no increase or just a negligible increase in accuracy, and usually no cost savings. There are two important points to consider with regard to PRT and SPRT readouts which bear repeating:

- 1) Ensure that the readout has a resistance range appropriate for the reference probe and UUTs for which it is intended. Over the range of -200 to 660°C , a 25Ω SPRT will vary in resistance from approximately 4.6Ω to 84.4Ω , a 100Ω PRT from approximately 18Ω to 338Ω . This will usually require 2 or 3 range changes for typical DMMs (10Ω , 100Ω , and $1\text{k}\Omega$ ranges). Many modern thermometer readouts are designed to cover this span on a single range. Changing ranges can cause discontinuities in the math fit (the equations are intended to fit platinum, not DMM range offsets or gain errors).
- 2) Ensure that the readout is using the proper source current. Too much source current will result in excessive self-heating and incorrect calibration. In some cases, particularly with older DMMs, the source current is so high that damage to the sensor is likely. Additionally, some DMMs use unconventional values of source current such as decades of 2 or 3 rather than 1 (2 mA or 3 mA, not 1 mA). Most certainly, these values of current are not reproduced during calibration of the reference or use of the UUT. Moreover, if the readout is a DMM which requires range changes as mentioned above, the source current will change with the range, meaning different current values for measurements at different temperatures. This will result in inconsistent self-heating and additional calibration errors.

Temperature Source

As with readouts, temperature sources were discussed in depth previously. We will review points that pertain specifically to PRT calibration applications here. For additional information refer to the section on temperature sources. The most common temperature sources for PRT calibration are dry-wells and baths. Dry-wells are used in applications where probe consistency (diameter and length) is present and modest accuracy is desired. When probes of different shapes and sizes must be accommodated, or higher accuracy is required, calibration baths should be utilized. For the lowest temperatures (below -100°C) and the highest temperatures (above 500°C), an LN_2 comparison device and calibration furnace come into play. The two most important considerations are uniformity and stability. Since PRT probes are often massive, immersion depth is also an issue. Insufficient immersion depth will result in calibration errors. Additionally, if the reference probe is a glass sheath SPRT, then some form of protection should be used at higher temperatures to prevent devitrification of the glass sheath and contamination of the platinum sensor. This is a concern particularly with dry-wells and furnaces at temperatures above 400°C and with calibration baths that use liquid salt as the bath fluid. Another matter pertains to the style of UUT to be calibrated. Calibration of short UUTs presents many problems with regard to the temperature source. The probe must be immersed sufficiently without subjecting the transition junction (where the leads join the probe) to extreme temperatures. Often dry-well temperature sources are a better solution in these situa-

tions. Some calibration baths have fluid level adapters which actually raise the fluid up to the top of the bath lid. These adapters can also be used successfully in the calibration of short probes. Whatever type of temperature source is used, the most important consideration is the application itself. Even an excellent instrument may not perform adequately in a specific application if it is not matched to that application. Carefully evaluate the requirements before selecting the temperature source to ensure a good fit.

Calibration Procedure

Introduction

There are two types of calibrations applicable to PRTs—characterization and tolerance testing. The type of calibration to perform is determined by the way in which the UUT is to be used and the accuracy required by the user. Characterization is the type of calibration in which the UUT resistance is determined at several temperature points and the data are fitted to a mathematical expression. Tolerance testing on the other hand is a calibration in which the UUT resistance is compared to defined values at specific temperatures. No data fitting is performed. In the laboratory, we are required to perform both types of calibration depending upon our customer's needs. Often, we are expected to offer advice and support in determining which method is better suited to the user's requirements. We will discuss these points and others in this section.

Characterization

Characterization is the method that is most often used for medium to high accuracy PRT calibration. With this method, the resistance vs. temperature relationship is determined anew with each calibration. Generally, with this type of calibration, new calibration coefficients and a calibration table are provided as a product of the calibration. There are five basic steps to perform as listed below:

- 1) Place the reference probe and the UUTs in the temperature source in close proximity to one another.
- 2) Connect the leads to the readout(s) ensuring proper 2, 3, or 4 wire connection.
- 3) Measure the reference probe and determine the temperature.
- 4) Measure and record the resistance of the UUT(s).
- 5) Fit the data.

Some readouts simplify the technique by combining or eliminating some of the steps. In the following discussion, we will consider an application involving PRT characterization by comparison to an SPRT.

Step 1: Probe Placement

All temperature sources have instabilities and gradients. These translate into calibration errors and/or uncertainties. To minimize the effects, the probes should be placed as close together as practical. In dry-well temperature sources, the probe immersion points are fixed. Baths offer flexibility in probe placement. The probes to be calibrated should be placed in a radial pattern with the reference probe in the center (focus) of the circle. This ensures an equal distance from the reference probe to each of the UUTs. Also, the sensing elements should be on the same horizontal plane. Even though sensing elements are different lengths, having the bottoms of the probes at the same level is sufficient. Sufficient immersion must be achieved so that stem

losses do not occur. Generally, sufficient immersion is achieved when the probes are immersed to a depth equal to 20 times the probe diameter plus the length of the sensing element. For example, consider a 3/16 inch diameter probe with a 1 inch long sensing element. Using the rule of thumb, $20 \times 3/16'' + 1'' = 3 \text{ } 3/4'' + 1'' = 4 \text{ } 3/4''$. In this example, minimum immersion is achieved at 4 3/4 inches. This rule of thumb is generally correct with thin wall probe construction and in situations of good heat transfer. If the probe has thick wall construction and/or poor heat transfer is present (such as in the case of a dry-well with incorrectly sized holes), more immersion is required.

Step 2: Connection to Readout

This step is straightforward. Connections must be tight and in proper 2, 3, or 4 wire configuration. If using 4 wire configuration, ensure that the current and voltage connections are correct. See Figures 25, 26, and 27.

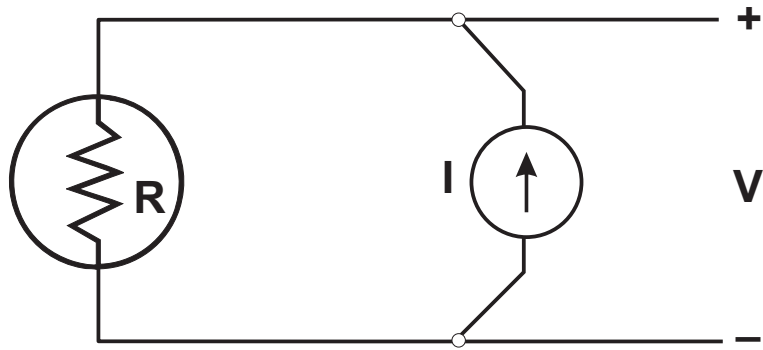


Figure 25. Two-wire Connection

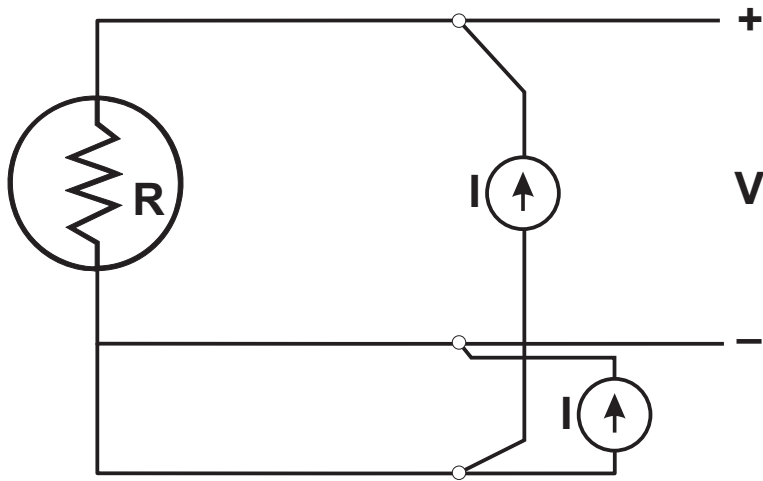


Figure 26. Three-wire Connection

Step 3: Measurement of Reference Probe and Temperature Determination

There are two ways to measure the reference probe and determine the temperature. Both techniques have the same potential accuracy. That is, if done correctly, neither technique is inherently more accurate than the other. The first and best method is used with sophisticated

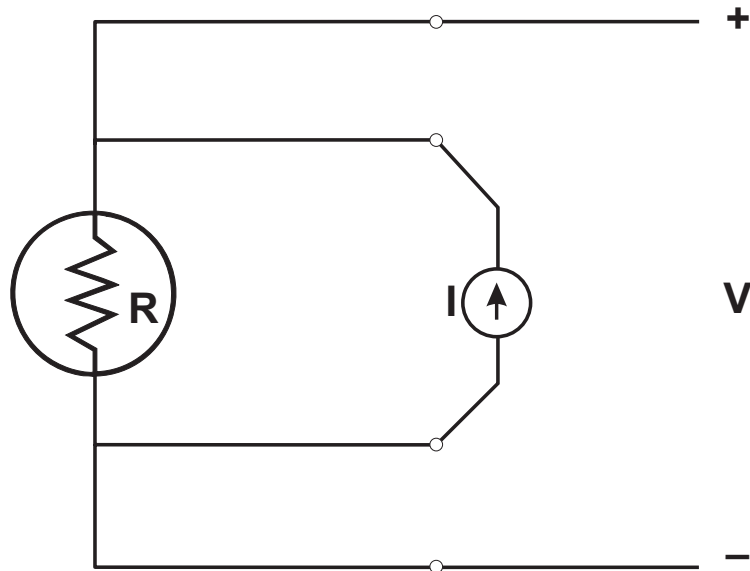


Figure 27. Four-wire Connection

readouts designed for temperature work. The resistance is measured and the temperature calculated from calibration coefficients which were entered into the readout previously. Once these calibration coefficients have been entered, the temperature calculations are accomplished internally and the readout displays in temperature units. The temperature data is available in real time. Some modern readouts also display the data in graphical format, allowing the operator to determine stability at a glance. Both of these features speed up the process and eliminate possible operator error due to incorrect table interpolation. The second method is used when the readout does not provide for proper temperature calculation. (Some readouts, particularly DMMs, have some of the more popular temperature conversions built in. These typically do not allow use of unique calibration coefficients and cannot be used for accurate temperature calibration.) In this case, the resistance is measured and the temperature is determined from either a calibration table or from a computer or calculator program. Since the temperature must be calculated after the resistance is measured, the process is slower and does not provide immediate, real time temperature data. If the temperature source is not stable errors will be introduced due to the time delay. Also, interpolation from a calibration table can lead to large errors if done incorrectly. Use of a calibration table may seem daunting, but with a little practice it can be mastered. See Tables 2 and 3 below.

Table 2. Interpolation from a resistance table

t(°C)	R(t)	dR/dt(t)	t(°C)	R(t)	dR/dt(t)
400	249.8820	0.3514	450	267.3108	0.3456
401	250.2335	0.3513	451	267.6564	0.3455
402	250.5848	0.3512	452	268.0019	0.3454
403	250.9360	0.3511	453	268.3472	0.3452

- 1) Measure the reference probe resistance 249.9071
- 2) Locate where it falls on the table between 249.8820 and 250.2335
- 3) Subtract lower table value from measured value $249.9071 - 249.8820 = 0.0251$
- 4) Divide by $dR/dT(t)$ (slope of curve) $0.0251 / 0.3514 = 0.0714$
- 5) Add fractional temperature to table value $0.0714 + 400 = 400.0714^{\circ}\text{C}$

Table 3. Interpolation from a resistance ratio (W) table

t(°C)	W(t)	dt/dW(t)	t(°C)	W(t)	dt/dW(t)
300	2.1429223	275.2199	350	2.3231801	279.6655
301	2.1465557	275.3075	351	2.3267558	279.7559
302	2.1501880	275.3951	352	2.3303304	279.8464
303	2.1538192	275.4827	353	2.3339037	279.9369

- 1) Measure reference probe resistance 54.75258
- 2) Calculate W (R_t/R_{tpw}) ($R_{tpw} = 25.54964$) $54.75258 / 25.54964 = 2.1429883$
- 3) Locate where it falls on the table between 2.1429223 and 2.1465557
- 4) Subtract lower table value from measured value $2.1429883 - 2.1429223 = 0.000066$
- 5) Multiply by $dt/dW(t)$ (inverse slope of curve) $0.000066 \cdot 275.2199 = 0.0181$
- 6) Add fractional temperature to table value $0.0181 + 300 = 300.0181^{\circ}\text{C}$

Step 4: Measurement of UUTs

Since the UUTs are resistance thermometers similar to the reference probe, they are measured in a similar manner. If several UUTs are undergoing calibration, ensure that when they are connected or switched in, sufficient time is allowed for self-heating to occur before the data is recorded. Also, ensure that the readout is set to the correct range to provide the proper source current and to prevent range changes between the measurements at different temperatures. Typically, the measurements are conducted starting at the highest temperature of calibration and working down. Additionally, it increases the precision of the calibration to use a mean (average) value calculated from multiple measurements at the same temperature. Often, the readout is designed with statistical features to facilitate this practice. It is also a good practice to close the process with an additional measurement of the reference probe. The sequence in which the probes (reference and UUT) are measured is referred to as a measurement scheme. There are many variables to consider when designing a measurement scheme. Some points to consider are:

- Accuracy - the higher the accuracy desired, the more all of the following must be considered.
- Temperature source stability - the more stable the source, the more time exists to conduct the measurements before temperature changes cause unwanted error.
- Number of UUTs - the higher the number, the longer it takes to cycle through all UUTs.

- Number of readouts - will the reference probe and UUTs be measured with the same readout or different readouts?
- Type of readout - a readout designed for temperature calibration often has features which allow flexibility in the measurement scheme.
- UUT characteristics - self-heating time, source current requirements, stability, and overall quality influence the measurement process.

It is not possible for us to anticipate all of the variables and discuss the optimum solutions here. However, in the following examples, we will consider some typical calibration scenarios and suggested measurement schemes.

Example 1: 2 DMM readouts, 1 reference probe and 5 UUTs

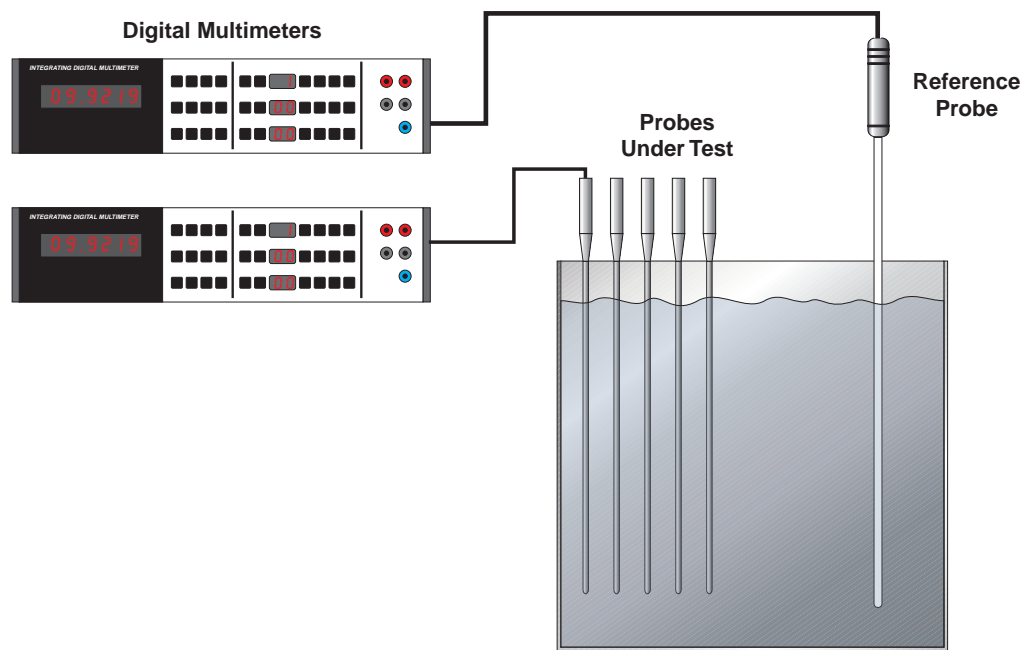


Figure 28. Two-DMM System

The reference probe is connected to one readout and the first UUT is connected to the second readout. This places the probes to be measured under current at all times, thus, eliminating self-heating errors caused by changing current conditions. The UUTs will be connected and measured individually. The scheme is as follows:

REF(1)-UUT (1) - REF(2)-UUT (2) - REF(3)-UUT (3) - REF(4)-UUT (4) - REF(5)-UUT (5)

This provides 5 readings each of the reference and the UUT. Take the average of the readings and use it for the data fit. The reference probe readings are in resistance so the temperature will have to be computed. After completion, repeat the process for the additional UUTs.

Example 2: 1 DMM readouts, 1 reference probe and 5 UUTs

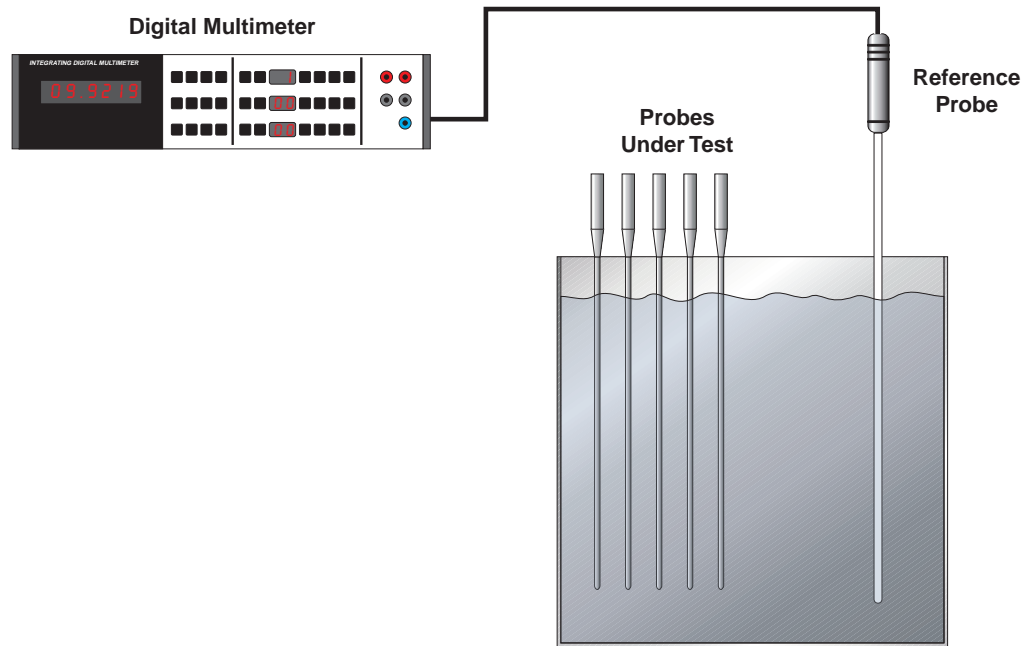


Figure 29. Single-DMM System

This example is similar to the first except that the reference probe and UUT must be measured by the same readout. The same scheme can be followed but more time must be allowed between readings to allow for self-heating. Since more time is involved, it might be beneficial to reduce the number of readings from five to three unless the heat source is extremely stable. Each probe will be connected and measured individually. The scheme is as follows:

wait-REF(1)-wait-UUT (1) - wait-REF(2)-wait-UUT(2) - wait-REF(1)-wait-UUT(3)-done

This provides 3 readings each of the reference and the UUT. Take the average of the readings and use it for the data fit. Again, the reference probe readings are in resistance so the temperature will have to be computed. After completion, repeat the process for the additional UUTs.

Example 3: 1 multi-channel thermometer readout, 1 reference probe and 5 UUTs

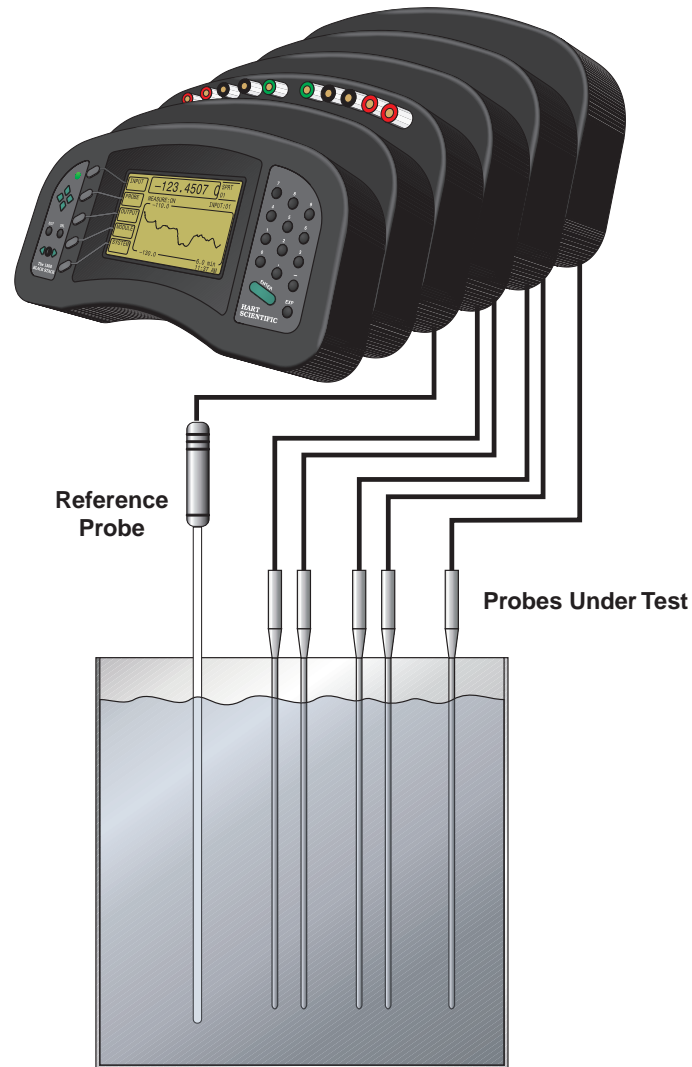


Figure 30. Black Stack System

In this example, all of the probes are connected directly to the thermometer readout. The readout controls the measurement and scans through all probes performing statistics in real time. Current may or may not be supplied at all times depending on the type of thermometer readout. If current is supplied at all times, there will be no self-heating errors. If current is not supplied at all times, ensure that the switching is done rapidly enough to reduce self-heating errors to a negligible level. The scheme is as follows

REF - UUT 1 - UUT 2 - UUT 3 - UUT 4 - UUT 5 - repeat 10 or more times

This provides many readings each of the reference and all of the UUTs. The average can be calculated and displayed directly by the readout. Also, the reference probe readings are in temperature so no further computation is required - the data is ready to fit.

Step 5: Data Fitting

Data fitting is simple in concept but can be complicated in practice. Essentially it is a process of solving a set of simultaneous equations which contain the calibration data to arrive at a set of coefficients unique to the PRT and calibration. There are several commercial software programs available specifically written to accomplish this task. Some are limited in function and do no more than solve the basic temperature functions. Others are more flexible and allow options regarding the number and location of calibration points and provide analysis regarding the precision of the resultant fit. The latter type of program is preferred. For metrologists who wish to tackle the algorithms themselves, a good mathematics application software like Mathcad or Mathematica or a good spreadsheet like Excel is extremely helpful. Of course, programs can be written in any of the modern computer languages (with double precision or better floating point capability) to perform the calculations with equal accuracy.

There are several equations which are used for PRT characterization. Among the most common are the International Temperature Scale of 1990 (ITS-90) series, the Callendar-Van Dusen, and third through fifth order polynomials. Obviously, with more than one model available to describe the behavior of a physical system, we must choose which one is best for our situation. The following discussion covers the features and purpose of each of these models and describes the form of the equations. The steps necessary to actually fit the data will be discussed in the section on mathematics later in this manual.

ITS-90: The ITS-90 series of functions were developed through a concerted effort from the international metrology community's leading temperature experts. These functions are intended to describe how the behavior of the SPRT relates, with a very high degree of precision, to the fixed points on which the scale is based. It does this extremely well. For our purposes, we are interested in the equations themselves. It turns out that the ITS-90 series works extremely well with high quality PRTs. The reference function - deviation function structure has many advantages over traditional polynomials. This is the preferred model for high accuracy applications. The ITS-90 temperatures are expressed in Kelvin units.

The equation is of the form:

$$W(T_{90}) = \frac{R(T_{90})}{R_{TPW}} \quad (1)$$

Where: $W(T_{90})$ = resistance ratio at temperature T
 $R(T_{90})$ = measured resistance at temperature T
 R_{TPW} = measured resistance at the triple point of water

The reference function is related to the deviation function with the following expression:

$$\Delta W(T_{90}) = W(T_{90}) - W_r(T_{90}) \quad (2)$$

Where: $\Delta W(T_{90})$ = deviation of calculated W from reference function at temperature T
 $W(T_{90})$ = calculated resistance ratio at temperature T (from equation (1))
 $W_r(T_{90})$ = reference function value at temperature T

Reference function for the range 13.8033 K to 273.16 K (-259.3467°C to 0.01°C):

$$\ln(W_r(T_{90})) = \sum_{i=0}^{12} A_i \cdot \left(\frac{\left(\frac{\ln(T_{90})}{273.16} \right) + 15}{15} \right)^i \quad (3)$$

Where: $W_r(T_{90})$ = reference function value at temperature T
 A_i = reference function coefficients from definition

Deviation function for the subrange 83.8058 K to 273.16 K (−189.3442°C to 0.01°C):

$$\Delta W_4(T_{90}) = a_4 \cdot (W(T_{90}) - 1) + b_4 \cdot (WT_{90}) - 1 \cdot \ln(W(T_{90})) \quad (4)$$

Where: $\Delta W(T_{90})$ = calculated deviation value at temperature T (from equation (2))
 $W(T_{90})$ = calculated resistance ratio at temperature T (from equation (1))
 a_4, b_4 = resulting calibration coefficients

Reference function for the range 273.15 K to 1234.93 K (0.00°C to 961.78°C):

$$W_r(T_{90}) = \sum_{i=0}^9 C_i \left(\frac{(T_{90} - 754.15)}{481} \right)^i \quad (5)$$

Where: $W_r(T_{90})$ = reference function value at temperature T
 C_i = reference function coefficients from definition

Deviation function for the subrange 273.15 K to 692.677 K (0.00°C to 419.527°C):

$$\Delta W_8(T_{90}) = a_8 \cdot (W(T_{90}) - 1) + b_8 \cdot (WT_{90}) - 1^2 \quad (6)$$

Where: $\Delta W(T_{90})$ = calculated deviation value at temperature T (from equation (2))
 $W(T_{90})$ = calculated resistance ratio at temperature T (from equation (1))
 a_8, b_8 = resulting calibration coefficients

The designations 4 and 8 in the deviation functions, equations (4) and (6) were inserted by NIST for identification of specific subranges. The values for the coefficients A_i and C_i in the reference functions, equations (3) and (5) are given in Table 4.

Table 4. ITS-90 Reference Function Coefficients

Coefficient	Value	Coefficient	Value
A ₀	-2.135 347 29	C ₀	2.781 572 54
A ₁	3.183 247 20	C ₁	1.646 509 16
A ₂	-1.801 435 97	C ₂	-0.137 143 90
A ₃	0.717 272 04	C ₃	-0.006 497 67
A ₄	0.503 440 27	C ₄	-0.002 344 44
A ₅	-0.618 993 95	C ₅	0.005 118 68
A ₆	-0.053 323 22	C ₆	0.001 879 82
A ₇	0.280 213 62	C ₇	-0.002 044 72
A ₈	0.107 152 24	C ₈	-0.000 461 22
A ₉	-0.293 028 65	C ₉	0.000 457 24
A ₁₀	0.044 598 72		
A ₁₁	0.118 686 32		
A ₁₂	-0.052 481 34		

Callendar-Van Dusen: The Callendar-Van Dusen equation has a long history. It was the main equation for SPRT and PRT interpolation for many years. It formed the basis for the temperature scales of 1927, 1948, and 1968. This equation is far simpler than the ITS-90 equations but has serious limitations in the precision of fit. As a result, it is not suitable for high accuracy applications but is perfectly suited to modest accuracy applications. Partly due to its history and simplicity, but mostly due to its continued suitability, it continues to be the preferred model for industrial platinum resistance thermometers today.

The general form of the equation is:

$$W(t) = \frac{R(t)}{R_0} \tag{7}$$

Where: W(t) = resistance ratio at temperature t
 R(t) = measured resistance at temperature t
 R₀ = measured resistance 0°C

And...

$$W(t) = 1 + At + Bt^2 + Ct^3 \cdot (t - 100) \tag{8}$$

Where: W(t) = resistance ratio at temperature t (reference 0°C)
 A,B,C = calibration coefficients (C is = 0 for temperatures above 0°C)

NOTE: All temperatures are expressed in °C and the resistance ratio (W) is referenced to 0°C rather than the triple point of water (0.010°C) as with the ITS-90.

Polynomials: Polynomials are frequently used to model physical phenomena from all fields of science. They have limited use with PRTs because of the high order required to achieve a suitable fit. (Recall that the reference functions for the ITS-90 are 9th and 12th order polynomials for the ranges above 0°C and below 0°C.) Additionally, the previous models use resistance ratio as the variable to fit. Most polynomials in use fit the resistance directly. Since resistance is not as stable as the resistance ratio, these models have serious limitations. That having been said, polynomials can be very useful over limited ranges and in applications where accuracy requirements are very modest.

Typical expressions take the form:

$$t = a + bR + cR^2 + dR^3 + eR^4 \quad (9)$$

Where: t = temperature
 a, b, c, d, e = calibration coefficients

Tolerance Testing

PRT calibrations involving tolerance testing are reserved for low accuracy applications. With this type of calibration the UUT resistance is compared to defined values at specific temperatures. The values are defined by one of the common models such as the Callendar-Van Dusen or DIN curve. PRTs calibrated in this way are generally used in industrial style applications where the readout is unable to accept unique coefficients but is preprogrammed with a common PRT curve. The probe must be tested to ascertain its compliance to the curve of interest. There are accuracy classes defined that probes are intended to fit.

The two common accuracy classes are:

$$\text{Class A} \quad \pm 0.15 + (0.002 \cdot t)^\circ\text{C} \quad (10)$$

$$\text{Class B} \quad \pm 0.30 + (0.005 \cdot t)^\circ\text{C} \quad (11)$$

These include errors arising from deviations in R_0 and from errors in slope. Frequently, we will see probes rated at a fraction of Class A. For example, 0.1 ASTM Class A. Fractional accuracy is achievable in sensors alone, but are very difficult to achieve in probes. The calculations are straightforward. See below:

Calculate the accuracy of a 0.1 ASTM Class A probe at 100°C

$$1) \quad = (0.13 + (0.0017 \cdot t)) \cdot 0.1$$

$$2) \quad = (0.13 + (0.0017 \cdot 100)) \cdot 0.1$$

$$3) \quad = (0.13 + 0.17) \cdot 0.1 = \mathbf{0.03}$$

Tolerance testing is straightforward. The measurements are carried out essentially in the same manner as described for characterization. The results are then compared to the defined values and pass or fail is determined. If DMMs are used, then the data is fitted to the standard tables as described previously for interpolation of the reference probe. In this case, both the reference probe and the UUT are interpolated according to their specific tables and the UUT error is the difference between the two results. Alternatively, the calibration temperature can be set precisely to a value from the table and the resistance of the UUT compared to a reference value from that table at that specific temperature. With this method, the calculations are easier to perform because interpolation is performed on only the reference probe, but setup can be

more difficult because the temperature source must be set and maintained at a precise value. The simplest method is available when the readout used for the UUTs has the curve preprogrammed or is programmable. In this case, it can be set to perform the measurement and display directly in temperature according to the curve of interest. The error is then simply the difference in temperature readings based on the UUT and the reference probe.

Thermistors

Characteristics

A thermistor is a temperature sensitive semiconductor. It is available in bead, disc, button, needle, or probed configurations. Two, three, or four leads are connected to the element and are used to provide for the measurement of the electrical resistance of the element. Thermistors have been traditionally used in low accuracy applications or in applications where a very small sensor was required. Lately, however, they are becoming much more popular in medium to high accuracy applications. Thermistors differ from PRTs in that they have a negative temperature coefficient (positive temperature coefficient thermistors are available, but not common) and very high sensitivity. Some thermistor characteristics are:

- Electrical parameter is resistance
- Narrow temperature Range (-50°C to 200°C)
- Stable over time
- Stable over temperature
- Well defined mathematically
- Negative temperature coefficient
- Very high sensitivity
- Exponential curve
- Relatively easy to measure
- Relatively easy to calibrate
- Commercially available in many configurations

Thermistors have a much narrower temperature range than PRTs. They are limited even further by the exponential nature of their curve. The resistance of a typical $10\text{ k}\Omega$ thermistor will vary over a 100°C temperature range from about $28.5\text{ k}\Omega$ at 0°C to about 925Ω at 100°C ($10\text{ k}\Omega$ is the nominal resistance at 25°C). The precision of the exponential fit deteriorates beyond this span. Therefore, like PRTs, no single instrument will be useable over the entire thermistor range. Also, like PRTs, the electrical resistance is measured at several temperature points and fitted to a mathematical expression. The number of calibration points depends on the range and accuracy desired but, because of the non-linearity, usually between 5 and 10 points are required. Also, for the same reason, the readout used for the resistance measurement must have a large range. Thermistors, like any probe, have immersion requirements which vary from configuration to configuration. Often, the required immersion is not stated or specified. Since

thermistors are used in so many different applications, we are presented with a large variety of shapes, sizes, and types. Although the basic calibration requirements are the same, these various configurations pose different problems in the laboratory. We must solve these problems satisfactorily to provide a proper calibration. Therefore, we must understand the requirements to an extent which allows us to adapt our process, if necessary, to accommodate a new or unusual configuration.

Instruments, Standards, and Apparatus

Calibration is performed by measurement of the resistance of the UUT while it is exposed to a temperature. Fundamentally, four instruments are required as follows:

- 1) Reference probe
- 2) Readout for the reference
- 3) Readout for the UUT
- 4) Temperature source

Reference Probe

Depending upon the accuracy required, the reference probe will be either an SPRT, a PRT, or a thermistor of better quality and calibration than the UUTs. Since this instrument forms the basis for our calibration, its accuracy and stability are of paramount importance.

SPRTs

SPRTs are the most accurate and stable instruments available for this purpose. Generally a 25 or 100 Ω glass sheath version is used. A typical glass sheathed 25 Ω SPRT with a high quality calibration will have calibration uncertainties from 0.001 $^{\circ}$ C to 0.005 $^{\circ}$ C over the temperature range of thermistors. Additionally, since these instruments are actually part of the definition of the ITS-90, they are standardized. That is, there are minimum requirements for the purity of the platinum wire and the type of construction used. This results in less confusion as to the suitability of the instrument for a particular application and almost guaranteed good performance if calibrated and used correctly. These instruments are highly stable and accurate, but they are expensive and extremely delicate. They should be reserved for high accuracy applications only.

PRTs

When accuracy requirements are less severe, PRTs can be used successfully. PRTs are available in many configurations, however PRTs which are suitable for use as calibration standards are generally available as 100 Ω stainless steel sheathed probes. These instruments are not as accurate as SPRTs but they are generally more rugged and easier to work with. Additionally, unlike SPRTs, the design of PRTs is left to the discretion and ingenuity of the manufacturer. Not all designs perform to the level required for use as a reference. Be careful in the selection of a PRT to ensure that the type selected is appropriate for use as a calibration reference over the range of interest and with the required accuracy.

Thermistors

Reference grade thermistors are available with uncertainties and stability approaching an SPRT. This grade is suitable as calibration standards. They too are generally more rugged and easier to work with than SPRTs and even PRTs. Also, like PRTs, the design of thermistors is left to the discretion and ingenuity of the manufacturer. Take precautions to ensure that the model selected will perform as required over the temperature range of interest.

Special Considerations

In addition to accuracy requirements, there are other characteristics which must be considered. For example, thermistors which are not hermetically sealed will allow moisture ingress and will not perform well below 0°C regardless of what is stated in the specifications. Also, some of the common potting materials used in thermistor probes will become brittle at cold temperatures, causing damage or destruction of the probe. Additionally, thermistors are available in a wide variety of sheath or coating materials. Ensure that the material is compatible with the bath fluid if the temperature source is a liquid bath. These points and others must be understood and taken into account in the selection of a reference probe.

Table 5. Reference Probe Characteristics Summary Table

SPRT	PRT / Thermistor
Capable of very high accuracy	Capable of moderate to high accuracy
Extremely stable	Very stable
Available in various sheath materials	Wide variety of sheath materials
Standardized	Not standardized
Relatively expensive to purchase	Relatively inexpensive to purchase
Relatively expensive to calibrate	Relatively inexpensive to calibrate
Extremely delicate	Less delicate

Readout

Readouts were covered in detail previously so we will not go into individual characteristics or performance here. We will review some of the main considerations. First, although SPRTs, PRTs and thermistors are all resistance sensors, the technical requirements for a thermistor readout are quite different from those of a readout for SPRTs and PRTs. In most cases, more than one readout is required. Some specialized readouts meet the requirements for both types of sensors and will work with both. If the reference probe is a thermistor of similar nominal resistance as the UUTs, then a single, switching readout can be used. However, if the readout is not designed for temperature calibration and/or a switching system is not available then two or more readouts will probably be required in this situation also. Before selecting a readout, review the information presented in the readouts section with regard to current settings, timing, multiplexing, etc. Best results will be obtained with readouts designed specifically for thermometer calibration. DMMs and bridges severely limit flexibility with no increase or just a negligible increase in accuracy and usually no cost savings. There are two important points to consider with regard to thermistor readouts which bear repeating:

- 1) Ensure that the readout has a resistance range appropriate for the reference probe and UUTs for which it is intended. Over the range of 0 to 1000°C, a 25Ω SPRT will vary in resistance from approximately 25Ω to 35Ω, a 100Ω PRT from approximately 100Ω to 140Ω. This will usually require no range changes for typical DMMs (100Ω range). However, over this same temperature range, a 10 kΩ thermistor will vary in resistance from about 28.5 kΩ to 925Ω. This will require several range changes over the course of calibration. A few specialized thermometer readouts are designed to cover this span on a single range. Changing ranges can cause discontinuities in the math fit (the equations are intended to fit the thermistor, not DMM range offsets or gain errors).
- 2) Ensure that the readout is using the proper source current. Thermistors are even more susceptible to self-heating errors than are SPRTs or PRTs and very low levels of current must be used. The self-heating coefficient of a typical 10 kΩ thermistor probe is 4

mW/°C (4 μ W/0.001°C). Using Ohms Law, that would allow a maximum source current of 12 μ A at 0°C for a self-heating error of 0.001°C! It is quite difficult to achieve adequate sensitivity in a typical DMM with such low levels of source current. Additionally, some DMMs use unconventional values of source current such as decades of 2 or 3 rather than 1 (20 μ A or 30 μ A, not 10 μ A). Most certainly, these values of current are not reproduced during calibration of the reference or use of the UUT. Moreover, if the readout is a DMM which requires range changes as mentioned above, the source current may change with the range, meaning different current values for measurements at different temperatures. This will result in inconsistent self-heating and additional calibration errors.

Temperature Source

As with readouts, temperature sources were discussed in depth previously. We will review points that pertain specifically to thermistor calibration applications here. For additional information refer to the section on temperature sources. The most common temperature sources for thermistor calibration are dry-wells and baths. Dry-wells are used in applications where probe consistency (diameter and length) is present and modest accuracy is desired. When probes of different shapes and sizes must be accommodated, or higher accuracy is required, calibration baths should be utilized. The two most important considerations are uniformity and stability. Because of the limited range of thermistors, highly stable and uniform temperature sources are not difficult to obtain. Another matter pertains to the style of UUT to be calibrated. Calibration of short UUTs presents many problems with regard to the temperature source. The probe must be immersed sufficiently without subjecting the transition junction (where the leads join the probe) to extreme temperatures and in some cases to bath fluid. Often dry-well temperature sources are a better solution in these situations. Another challenge with thermistors is sheath material. Since thermistors are available for very unique applications, ensure that damage to the sheath will not occur if calibrated in your bath. Whatever type of temperature source is used, the most important consideration is the application itself. Even an excellent instrument may not perform adequately in a specific application if it is not matched to that application. Carefully evaluate the requirements before selecting the temperature source to ensure a good fit.

Calibration Procedure

Introduction

As with PRTs, there are two types of calibration applicable to thermistors - characterization and tolerance testing. The type of calibration to perform is determined by the way in which the UUT is to be used and the accuracy required by the user. Characterization is the type of calibration in which the UUT resistance is determined at several temperature points and the data are fitted to a mathematical expression. Tolerance testing on the other hand is a calibration in which the UUT resistance is compared to defined values at specific temperatures. No data fitting is performed. In the laboratory, we are required to perform both types of calibration depending upon our customer's needs. Often, we are expected to offer advice and support in determining which method is better suited to the user's requirements. We will discuss these points and others in this section.

Characterization

Characterization is the method that is most often used for medium to high accuracy thermistor calibration. With this method, the resistance vs. temperature relationship is determined anew with each calibration. Generally, with this type of calibration, new calibration coefficients and

a calibration table are provided as a product of the calibration. There are five basic steps to perform as listed below:

- 1) Place the reference probe and the UUTs in the temperature source in close proximity to one another.
- 2) Connect the leads to the readout(s) ensuring proper 2 or 4 wire connection.
- 3) Measure the reference probe and determine the temperature.
- 4) Measure and record the resistance of the UUT(s).
- 5) Fit the data.

Some readouts simplify the technique by combining or eliminating some of the steps. In the following discussion, we will consider an application involving thermistor characterization by comparison to an SPRT.

Step 1: Probe Placement

All temperature sources have instabilities and gradients. These translate into calibration errors and/or uncertainties. To minimize the effects, the probes should be placed as close together as practicable. In dry-well temperature sources, the probe immersion points are fixed. Baths offer flexibility in probe placement. The probes to be calibrated should be placed in a radial pattern with the reference probe in the center (focus) of the circle. This ensures an equal distance from the reference probe to each of the UUTs. Also, the sensing elements should be on the same horizontal plane. Thermistor elements are very short when compared to SPRT elements (1/8 to 1/4 inch and 1 3/4 inch respectively). It is a good practice to place the tip of the thermistor probe at approximately the center of the SPRT sensor. Sufficient immersion must be achieved so that stem losses do not occur. Generally, sufficient immersion is achieved when the probes are immersed to a depth equal to 20 times the probe diameter plus the length of the sensing element. For example, consider a 3/16 inch diameter probe with a 1/4 inch long sensing element. Using the rule of thumb, $20 \times 3/16'' + 1/4'' = 3 \ 3/4'' + 1/4'' = 4''$. In this example, minimum immersion is achieved at 4 inches. This rule of thumb is generally correct with thin wall probe construction and in situations of good heat transfer. If the probe has thick wall construction and/or poor heat transfer is present (such as in the case of a dry-well with incorrectly sized holes), more immersion is required.

Step 2: Connection to Readout

This step is straightforward. Connections must be tight and in proper 2 or 4 wire configuration. If using 4 wire configuration, ensure that the current and voltage connections are correct. See Figures 25 and 27 on pages 48 and 49.

Step 3: Measurement of Reference Probe and Temperature Determination

There are two ways to measure the reference probe and determine the temperature. Both techniques have the same potential accuracy. That is, if done correctly, neither technique is inherently more accurate than the other. The first and best method is used with sophisticated readouts designed for temperature work. The resistance is measured and the temperature calculated from calibration coefficients which were entered into the readout previously. Once these calibration coefficients have been entered, the temperature calculations are accomplished internally and the readout displays in temperature units. The temperature data is available in real time. Some modern readouts also display the data in graphical format, allowing the operator to determine stability at a glance. Both of these features speed up the process and eliminate possible operator error due to incorrect table interpolation. The second method is used when the readout does not provide for proper temperature calculation. (Some readouts, particularly DMMs, have some of the more popular temperature conversions built in. These

typically do not allow use of unique calibration coefficients and cannot be used for accurate temperature calibration.) In this case, the resistance is measured and the temperature is determined from either a calibration table or from a computer or calculator program. Since the temperature must be calculated after the resistance is measured, the process is slower and does not provide immediate, real time temperature data. If the temperature source is not stable, errors will be introduced due to the time delay. Also, interpolation from a calibration table can lead to large errors if done incorrectly. Use of a calibration table may seem daunting, but with a little practice it can be mastered. See the Tables 6 and 7 below.

Table 6. Interpolation from a resistance table

t(°C)	R(t)	dR/dt(t)	t(°C)	R(t)	dR/dt(t)
0	99.9625	0.3985	50	119.7375	0.3924
1	100.3610	0.3984	51	120.1300	0.3923
2	100.7594	0.3982	52	120.5223	0.3922
3	101.1576	0.3981	53	120.9144	0.3921

- 1) Measure reference probe resistance 119.7422
- 2) Locate where it falls on the table between 119.7375 and 120.1300
- 3) Subtract lower table value from measured value $119.7422 - 119.7375 = 0.0047$
- 4) Divide by dR/dT(t) (slope of curve) $0.0047 / 0.3924 = 0.0120$
- 5) Add fractional temperature to table value $0.0120 + 50 = \mathbf{50.0120^{\circ}\text{C}}$

Table 7. Interpolation from a resistance ratio (W) table

t(°C)	W(t)	dt/dW(t)	t(°C)	W(t)	dt/dW(t)
100	1.39280403	258.5417	150	1.58473995	262.5713
101	1.39667188	258.6214	151	1.58854844	262.6528
102	1.40053853	258.7012	152	1.59235575	262.7344
103	1.40440400	258.7810	153	1.59616188	262.8159

- 1) Measure reference probe resistance 35.59018
- 2) Calculate W (R_t/R_{tpw}) ($R_{tpw} = 25.54964$) $55.59018 / 25.54964 = 1.3929817$
- 3) Locate where it falls on the table between 1.3928040 and 1.3966719
- 4) Subtract lower table value from measured value $1.3929817 - 1.3928040 = 0.0001777$
- 5) Multiply by dt/dW(t) (inverse slope of curve) $0.0001777 \cdot 258.5417 = 0.0459$
- 6) Add fractional temperature to table value $0.0459 + 100 = \mathbf{100.0459^{\circ}\text{C}}$

Step 4: Measurement of UUTs

Since the UUTs are resistance thermometers similar to the reference probe, they are measured in a similar manner. If several UUTs are undergoing calibration, ensure that when they are

connected or switched in, sufficient time is allowed for self-heating to occur before the data is recorded. Also, ensure that the readout is set to the correct range to provide the proper source current and to prevent range changes between the measurements at different temperatures. Typically, the measurements are conducted starting at the lowest temperature of calibration and working up. Additionally, it increases the precision of the calibration to use a mean (average) value calculated from multiple measurements at the same temperature. Often, the readout is designed with statistical features to facilitate this practice. It is also a good practice to close the process with an additional measurement of the reference probe. The sequence in which the probes (reference and UUT) are measured is referred to as a measurement scheme. There are many variables to consider when designing a measurement scheme. Some points to consider are:

- Accuracy - the higher the accuracy desired, the more all of the following must be considered.
- Temperature source stability - the more stable the source, the more time exists to conduct the measurements before temperature changes cause unwanted error.
- Number of UUTs - the higher the number, the longer it takes to cycle through all UUTs.
- Number of readouts - will the reference probe and UUTs be measured with the same readout or different readouts?
- Type of readout - a readout designed for temperature calibration often has features which allow flexibility in the measurement scheme.
- UUT characteristics - self-heating time, source current requirements, stability, and overall quality influence the measurement process.

It is not possible for us to anticipate all of the variables and discuss the optimum solutions here. However, in the following examples, we will consider some typical calibration scenarios and suggested measurement schemes.

Example 1: 2 DMM readouts, 1 reference probe and 5 UUTs

See Figure 28 on page 51.

The reference probe is connected to one readout and the first UUT is connected to the second readout. This places the probes to be measured under current at all times, thus, eliminating self-heating errors caused by changing current conditions. The UUTs will be connected and measured individually. The scheme is as follows:

REF(1)-UUT (1) - REF(2)-UUT (2) - REF(3)-UUT (3) - REF(4)-UUT (4) - REF(5)-UUT (5)

This provides 5 readings each of the reference and the UUT. Take the average of the readings and use it for the data fit. The reference probe readings are in resistance so the temperature will have to be computed. After completion, repeat the process for the additional UUTs.

Example 2: 1 digital thermometer readout, 1 DMM readout, 1 reference probe and 5 UUTs

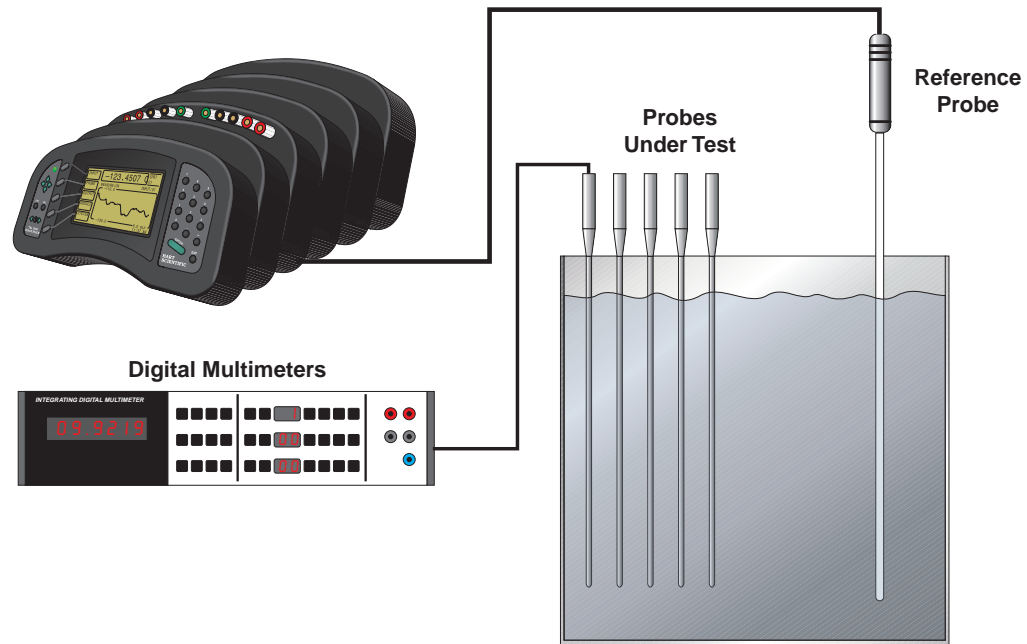


Figure 31. 1 Digital Thermometer & 1 DMM System

This example is similar to the first except that the reference probe readout is a digital thermometer and is displaying in temperature directly. The same scheme is followed. After completion, repeat the process for the additional UUTs.

REF(1)-UUT (1) - REF(2)-UUT (2) - REF(3)-UUT (3) - REF(4)-UUT (4) - REF(5)-UUT (5)

This provides 5 readings each of the reference and the UUT. Take the average of the readings and use it for the data fit. Since the reference probe readout indicates temperature directly, no computations are necessary. After completion, repeat the process for the additional UUTs.

Example 3: 1 multi-channel thermometer readout, 1 reference probe and 5 UUTs

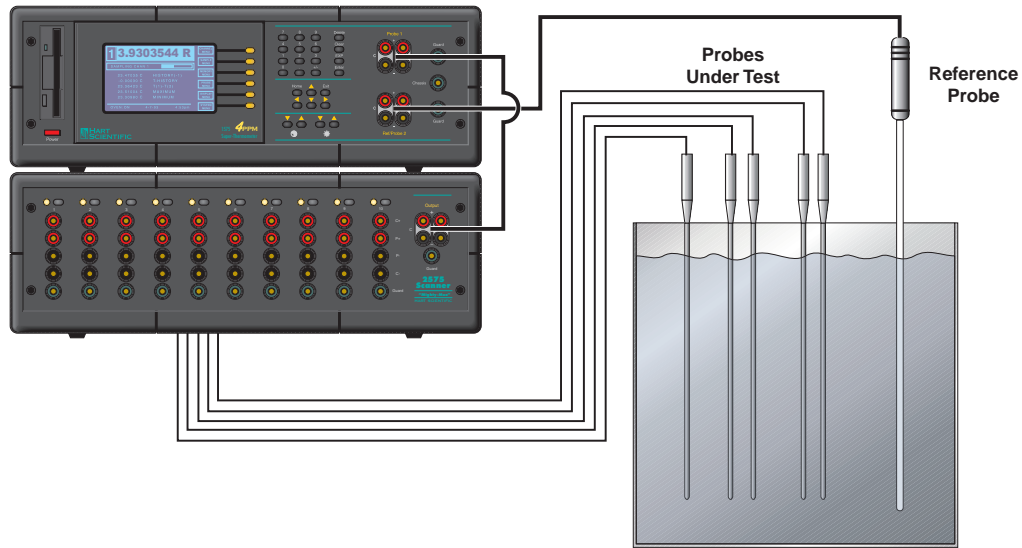


Figure 32. Super-Thermometer System

In this example, all of the probes are connected directly to the thermometer readout. The readout controls the measurement and scans through all probes performing statistics in real time. It also changes current levels to provide the proper measurement current for the type of probe being measured. Current may or may not be supplied at all times depending on the type of thermometer readout. If current is supplied at all times, there will be no self-heating errors. If current is not supplied at all times, ensure that the switching is done rapidly enough to reduce self-heating errors to a negligible level. The scheme is as follows

REF - UUT 1 - UUT 2 - UUT 3 - UUT 4 - UUT 5 - repeat 10 or more times

This provides many readings each of the reference and all of the UUTs. The average can be calculated and displayed directly by the readout. Also, the reference probe readings are in temperature so no further computation is required—the data is ready to fit.

Step 5: Data Fitting

Data fitting is simple in concept but can be complicated in practice. Essentially it is a process of solving a set of simultaneous equations which contain the calibration data to arrive at a set of coefficients unique to the thermistor and calibration. There are several commercial software programs available specifically written to accomplish this task. Some are limited in function and do no more than solve the basic temperature functions. Others are more flexible and allow options regarding the number and location of calibration points and provide analysis regarding the precision of the resultant fit. The latter type of program is preferred. For metrologists who wish to tackle the algorithms themselves, a good mathematics application software like Mathcad or Mathematica or a good spreadsheet like Excel is extremely helpful. Of course, programs can be written in any of the modern computer languages (with double precision or better floating point capability) to perform the calculations with equal accuracy.

There are two common equations (with corresponding inverse functions) which are used for thermistor characterization. They are basically variations of exponential polynomials. Obviously, with more than one model available to describe the behavior of a physical system, we must choose which one is best for our situation. The following discussion covers the features and purpose of each of these models and describes the form of the equations. The steps necessary to actually fit the data will be discussed in the section on mathematics later in this manual.

Third order polynomial: Polynomials are frequently used to model physical phenomena from all fields of science. They have proven to be particularly useful in fitting thermistors and have been used for many years. Since thermistors have an exponential curve, exponential polynomials expressed in terms of resistance and absolute temperature are used. There is no limitation experienced when fitting resistance directly because thermistor resistance is as stable as the resistance ratio. The following polynomials will fit a high quality thermistor with a precision of 0.003 to 0.0001°C over a 100°C temperature span. This is the preferred model for high accuracy applications.

The equation is of the form:

$$T = \frac{1}{A + B(\ln R) + C(\ln R)^2 + D(\ln R)^3} \quad (12)$$

Where: T = absolute temperature in Kelvin
 ln = natural log function
 R = resistance in Ohms
 A,B,C,D = resulting calibration coefficients

And its inverse...

$$R = \exp\left(a + \frac{b}{T} + \frac{c}{T^2} + \frac{d}{T^3}\right) \quad (13)$$

Where: R = resistance in Ohms
 exp = exponent function
 T = absolute temperature in Kelvin
 a,b,c,d = resulting calibration coefficients

Steinhart-Hart: The Steinhart-Hart equation is a version of the third order polynomial shown above. Drs. Steinhart and Hart showed that the squared term can be eliminated from the above functions with no loss in precision when used over a small temperature range (maximum of 35°C span). The work of Steinhart and Hart was confirmed by B.W. Mangum at NIST and R. Koehler at Woods Hole Oceanographic Institute. Additionally, the Steinhart-Hart expression can prove useful over greater ranges when a small loss in precision can be tolerated. The following polynomials will fit a high quality thermistor with a precision of 0.0001 to 0.003°C over a 35°C temperature span and from of 0.003 to 0.010°C over a 100°C span.

The equation is of the form:

$$T = \frac{1}{A + B(\ln R) + D(\ln R)^3} \quad (14)$$

Where: T = absolute temperature in Kelvin
 ln = natural log function
 R = resistance in Ohms
 A,B,D = resulting calibration coefficients

And it's inverse...

$$R = \exp\left(a + \frac{b}{T} + \frac{d}{T^3}\right) \quad (15)$$

Where: T = absolute temperature in Kelvin
 exp = exponent function
 R = resistance in Ohms
 a,b,d = resulting calibration coefficients

Tolerance Testing

Thermistor calibrations involving tolerance testing are reserved for low accuracy applications. With this type of calibration the UUT resistance is compared to defined values at specific temperatures. The values are generally defined by the manufacturer of the thermistor. Thermistors calibrated in this way are generally used when interchangeability is more important than accuracy. Common values for thermistor interchangeability are 0.05, 0.10, and 0.20°C.

Tolerance testing is straightforward. The measurements are carried out essentially in the same manner as described for characterization. The results are then compared to the defined values and pass or fail is determined. If DMMs are used, then the data is fitted to the standard tables as described previously for interpolation of the reference probe. In this case, both the reference probe and the UUT are interpolated according to their specific tables and the UUT error is the difference between the two results. Alternatively, the calibration temperature can be set precisely to a value from the table and the resistance of the UUT compared to a reference value from that table at that specific temperature. With this method, the calculations are easier to perform because interpolation is performed on only the reference probe, but setup can be more difficult because the temperature source must be set and maintained at a precise value. The simplest method is available when the readout used for the UUTs has the curve preprogrammed or is programmable. In this case, it can be set to perform the measurement and display directly in temperature according to the curve of interest. The error is then simply the difference in temperature readings based on the UUT and the reference probe.

Thermocouples

Characteristics

A thermocouple is a transducer comprised of two dissimilar materials joined at a junction. It is, by far, the most widely used of all temperature sensors. Thermocouples are traditionally used in low accuracy applications or in applications where a very small sensor is required. Thermocouples differ from PRTs and thermistors in that rather than having an electrical parameter which is affected by temperature, they actually produce a voltage output which is temperature dependent. Thermocouples can be constructed from almost any material with thermoelectric properties. As a result, there are many different types of thermocouples available. Some thermocouple characteristics are:

- Temperature dependent voltage output

- Wide temperature range (-200°C to 2000°C)
- Well defined mathematically
- Positive temperature coefficient
- Relatively linear output
- Relatively easy to measure
- Somewhat more difficult to calibrate
- Not particularly stable over temperature
- Commercially available in many configurations

Thermocouples have a much wider temperature range than either PRTs or thermistors. Since they are comprised of mixtures of metals, they are limited primarily by degradation of the mixture and resulting change in composition and by oxidation. The mixtures are chosen for specific behavior and specific applications. Therefore, like PRTs and thermistors, no single type will be useable over the entire range. Additionally, because of construction limitations, a particular thermocouple assembly may not be usable over the range suggested by the thermocouple tables for that type of thermocouple. To calibrate a thermocouple, the EMF output is measured at several temperature points and fitted to a mathematical expression. The number of calibration points depends on the range and accuracy desired. Thermocouples have immersion requirements similar to those of PRTs and thermistors. Thermocouples come in basically two forms, bare (insulated) wire, and probes (although probes are available in a variety of shapes, sizes, and types). The basic calibration requirements are the same; these various configurations pose different problems in the laboratory.

Instruments, Standards, and Apparatus

Calibration is performed by measurement of the EMF output of the UUT while one junction is exposed to a temperature and one junction is maintained at a reference (usually 0°C). Five instruments are required as follows:

- 1) Reference probe
- 2) Readout for the reference
- 3) Readout for the UUT
- 4) Temperature source
- 5) Reference junction temperature source

Reference Probe

Depending upon the accuracy required, the reference probe will be either an SPRT, a PRT, or a thermocouple of better quality and calibration than the UUTs. Since this instrument forms the basis for our calibration, its accuracy and stability are of paramount importance.

SPRTs

SPRTs are the most accurate and stable instruments available for this purpose. Generally a 25 or 100Ω glass sheath version is used. A typical glass sheathed 25Ω SPRT with a high quality calibration will have calibration uncertainties from 0.001°C to 0.005°C over the temperature range of thermocouples. Additionally, since these instruments are actually part of the definition of the ITS-90, they are standardized. That is, there are minimum requirements for the purity of the platinum wire and the type of construction used. This results in less confusion as to the suitability of the instrument for a particular application and almost guaranteed good performance if calibrated and used correctly. These instruments are highly stable and accurate, but they are expensive and extremely delicate. They should be reserved for high accuracy applications only.

PRTs

When accuracy requirements are less severe, PRTs can be used successfully. PRTs are available in many configurations, however PRTs which are suitable for use as calibration standards are generally available as 100Ω stainless steel sheathed probes. These instruments are not as accurate as SPRTs but they are generally more rugged and easier to work with. Additionally, unlike SPRTs, the design of PRTs is left to the discretion and ingenuity of the manufacturer. Not all designs perform to the level required for use as a reference. Be careful in the selection of a PRT to ensure that the type selected is appropriate for use as a calibration reference over the range of interest and with the required accuracy.

Thermocouples

Reference grade thermocouples are available with uncertainties and stability approaching a PRT (or even an SPRT) at high temperatures. This grade is suitable as a calibration standard. They are generally almost as delicate as SPRTs and PRTs. Thermocouples which qualify as reference grade generally are standardized in composition but not necessarily construction. Take precautions to ensure that the model selected will perform as required over the temperature range of interest.

Special Considerations

In addition to accuracy requirements, there are other characteristics which must be considered. For example, the reference junction end of the thermocouples must be long enough to allow proper immersion into the reference temperature source (typically an ice bath). Also, some of the common sheath materials used in thermocouple probes have problems of their own at high temperatures. Ensure that the sheath material is compatible with the calibration process for which it is intended. These points and others must be understood and taken into account in the selection of a reference probe.

Table 8. Reference Probe Characteristics Summary Table

SPRT / PRT	Thermocouple
Capable of very high to high accuracy	Capable of moderate accuracy
Extremely stable	Not very stable
Available in various sheath materials	Possible sheath material problem at high temperatures
Standardized	Standardized
Relatively expensive to purchase	Relatively inexpensive to purchase
Relatively expensive to calibrate	Relatively inexpensive to calibrate
Extremely delicate	Less delicate

Readout

Readouts in general and readouts for SPRTs and PRTs in particular were covered in detail previously so we will not go into individual characteristics or performance here. We will review some of the main considerations for thermocouple readouts. First, since thermocouples produce a voltage output, the technical requirements for a thermocouple readout are quite different from those of a readout for SPRTs, PRTs, or thermistors. Unless the reference probe is a thermocouple also, two readouts will definitely be required. Additionally, the thermocouple reference junction must be considered. Most thermocouple readouts have “electronic reference junctions,” often referred to as cold junction compensation. This is an additional circuit which measures the temperature at the thermocouple - readout connection and compensates for the non-zero reference temperature. This type of compensation is very convenient but not usually as accurate as an actual ice point bath. If the reference probe is a thermocouple also, then a single, switching readout can be used. However, if the readout is not designed for temperature calibration and/or a switching system is not available then two or more readouts will probably be required in this situation also. Before selecting a readout, review the information presented in the readouts section with regard to reference junction compensation, multiplexing, etc. Best results will be obtained with readouts designed specifically for thermocouple calibration. DMMs severely limit the flexibility with no increase or just a negligible increase in accuracy and usually no cost savings. There is one important point to consider with regard to thermocouple readouts which bears repeating:

The voltage output from a thermocouple is very low and a small voltage uncertainty equates to a large temperature uncertainty. The voltage measurements must be extremely accurate even for moderate accuracy temperature calibrations. Also, at the low voltage levels that will be measured when calibrating thermocouples, the readout floor error (noise limit or zero offset limit) becomes very significant. Ensure that the readout has a voltage range (usually to 100 mV range) and accuracy appropriate for thermocouple calibrations. Consider an example using a high accuracy 7 1/2 digit DMM to measure a type S thermocouple at 500°C. The example shows the relative contribution from the DMM sources of error.

DMM accuracy on 100 mV range	= (20 ppm of reading + 2 ppm of range)
Type S output at 500°C	= 4.2333 mV
Type S slope at 500°C	= 0.0099 mV/°C

Accuracy calculations:

$$= \frac{(4.2333\text{mV} \cdot 20\text{ppm}) + (100\text{mV} \cdot 2\text{ppm})}{0.0099\text{mV}/^\circ\text{C}}$$
$$= \frac{(0.00008466\text{mV}) + (0.0002\text{mV})}{0.0099\text{mV}/^\circ\text{C}} = 0.0288^\circ\text{C}$$

In this example, the uncertainty resulting from the DMM floor is much larger than the error due to the DMM rated accuracy. This situation is more pronounced at lower temperatures and less pronounced at higher temperatures. This illustrates the importance of readout floor error.

Temperature Source

As with readouts, temperature sources were discussed in depth previously. We will review points that pertain specifically to thermocouple calibration applications here. For additional

information refer to the section on temperature sources. The most common temperature sources for thermocouple calibration are dry-wells and calibration furnaces. However, when higher accuracy is required, calibration baths can be utilized. For the lowest temperatures (below -100°C) an LN_2 comparison device is required. Bare wire thermocouples should never be immersed directly into bath fluid, a protection tube should be used. Thermocouple probes are not usually massive but immersion depth must still be considered. Insufficient immersion depth will result in calibration errors. At elevated temperatures, precautions must be taken to avoid damage to the reference probe. Additionally, if a “real” reference junction is to be used, ensure that the temperature source selected is sufficiently insulated so that the external surfaces do not get so hot as to damage the ice bath or electronic ice bath. Whatever type of temperature source is used, the most important consideration is the application itself. Even an excellent instrument may not perform adequately in a specific application if it is not matched to that application. Carefully evaluate the requirements before selecting the temperature source to ensure a good fit.

Calibration Procedure

Introduction

As with PRTs and thermistors, there are two types of calibration applicable to thermocouples - characterization and tolerance testing. However, with a few exceptions, thermocouples are not sufficiently stable to warrant characterization. Typically, thermocouple probes and/or wire are tested for compliance to American Society for Testing and Materials (ASTM) error ratings. Compliance testing is simply a matter of measuring the EMF output at various temperatures and quantifying the error from the standard tables. Occasionally, we are called upon to characterize a thermocouple probe or wire. In these cases, the difference EMF is corrected by fitting it to a second order polynomial in a manner similar to the ITS-90 reference function—deviation function technique. A higher order polynomial will generally not improve the fit. The following section will discuss the procedures.

Tolerance Testing

In most applications, thermocouples are used without correction or characterization. The user must relay that the thermocouple behaves as the standard model predicts within certain limits. The ASTM has two sets of limits called standard limits of error and special limits of error. The special limits of error are tighter tolerances. To calibrate a thermocouple to ASTM specifications is to determine that it follows the standard model. In some cases, individual probes are calibrated while in other cases, entire rolls of wire require certification. The method is straightforward. No data fitting or complex computations are required. There are six basic steps to perform as listed below:

- 1) Place the reference probe and the UUTs in the temperature source in close proximity to one another.
- 2) Connect the external reference junction if used.
- 3) Connect the leads to the readout(s) ensuring proper 2 wire connection.
- 4) Measure the reference probe and determine the temperature.
- 5) Measure and record the EMF output of the UUT(s).
- 6) Compute the errors.

Some readouts simplify the technique by combining or eliminating some of the steps. In the following discussion, we will consider an application involving thermocouple calibration by comparison to a reference thermocouple.

Step 1: Probe Placement

All temperature sources have instabilities and gradients. These translate into calibration errors and/or uncertainties. To minimize the effects, the probes should be placed as close together as practicable. In dry-well temperature sources and calibration furnaces, the probe immersion points are fixed. Baths offer flexibility in probe placement. The probes to be calibrated should be placed in a radial pattern with the reference probe in the center (focus) of the circle. This ensures an equal distance from the reference probe to each of the UUTs. Also, the sensing elements should be on the same horizontal plane. Thermocouple junctions are usually at the tip of the probe. Sufficient immersion must be achieved so that stem losses do not occur. Generally, sufficient immersion is achieved when the probes are immersed to a depth equal to 20 times the probe diameter plus the length of the sensing element. For example, consider a 1/4 inch diameter probe. Using the rule of thumb, $20 \times 1/4'' = 5''$. In this example, minimum immersion is achieved at 5 inches. This rule of thumb is generally correct with thin wall probe construction and in situations of good heat transfer. If the probe has thick wall construction and/or poor heat transfer is present (such as in the case of a dry-well with incorrectly sized holes), more immersion is required.

Steps 2 and 3: Connection to Reference Junction and Readout

The connection to the readout depends upon whether an internal or external reference junction is used. Internal reference junctions are generally used in high throughput, low to medium accuracy applications. There is less opportunity for operator error and the entire procedure is simpler. The limitation in accuracy is due to the additional uncertainty in the reference junction compensation circuit itself (usually an additional 0.05 to 0.25°C). External reference junctions must be used when accuracy requirements prohibit the use of internal compensation or if the readout is not equipped with internal reference junction compensation (such as a typical DMM). External reference junction is slightly more involved with a single UUT but can become quite complicated when several UUTs are being calibrated and uncertainties must be kept to a minimum.

When using an internal reference (internal to the readout), the connections are straightforward. Simply connect the 2 wire thermocouple either directly or through proper extension wire to the readout, observing polarity. Never use copper connecting wire for the extension wire, errors will result. Ensure that all connections are tight and clean. Loose and/or dirty connections will cause spurious EMFs and measurement errors. Additionally, use of switches and multiplexors will result in errors because these devices are normally constructed of copper. Switches are available which are constructed of thermocouple materials and can be used if a large number of a single type of thermocouple must be calibrated. However, since the switch will still contribute an error which is extremely difficult to quantify, these are not recom-

mended either. If a large quantity of thermocouples must be calibrated, a multi-channel readout or external reference junction technique is recommended.

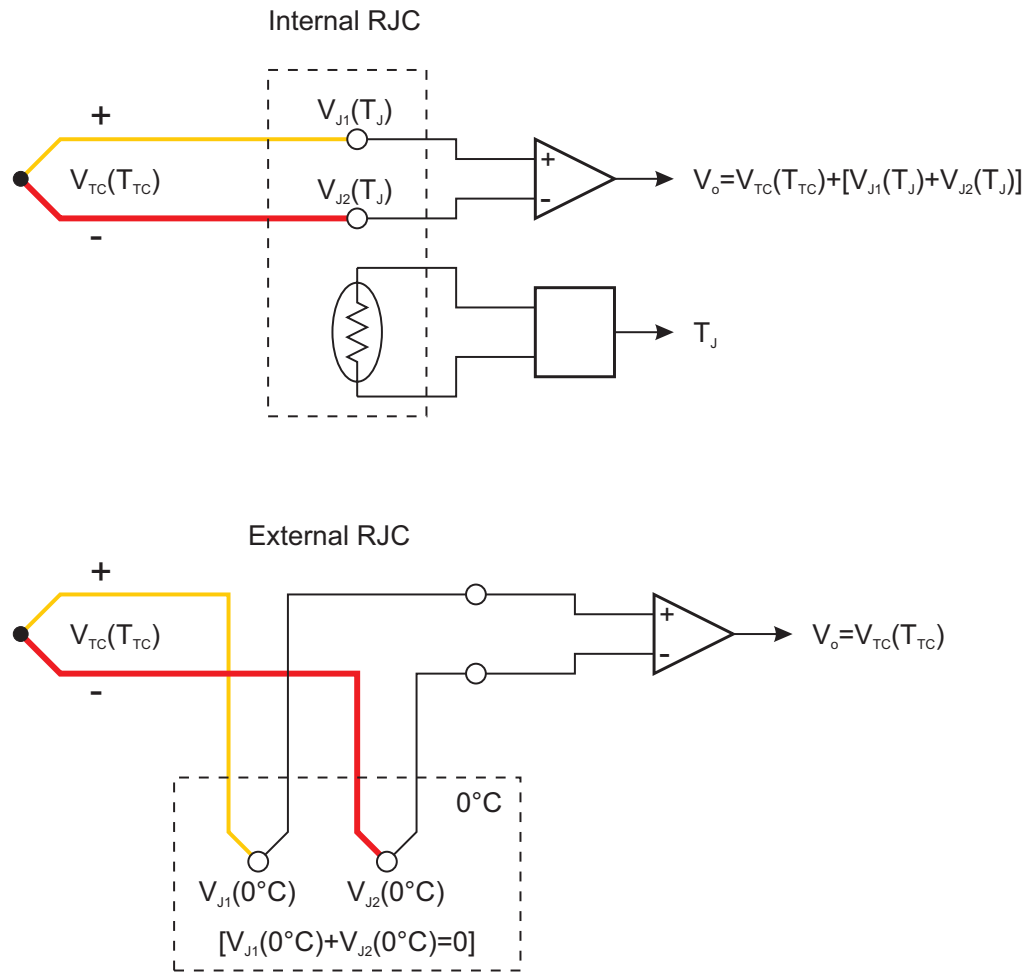


Figure 33. Connections using internal or external reference junction compensation

Connections involving external reference junctions are also straightforward but there is more opportunity for problems. The thermocouple is connected through high quality copper wires to the readout. The thermocouple to copper connections are then immersed into an ice bath to form the reference junction. The connections must be electrically insulated from one another and physically dry. Usually, the wires are welded, soldered, or twisted tightly and protected with heat shrink tubing. The group of wires are inserted into a thin wall metal or glass closed end tube and the tube is inserted into the ice bath. Immersion depth is an issue and depends upon the wire diameter. Usually 6 to 12 inches is sufficient. The copper connecting wires then go either directly to the readout or through a switch to the readout. Each UUT requires an individual reference junction. Some UUTs are terminated in thermocouple connectors and cannot be conveniently connected as just described. In these cases, “reference junction probes” can be constructed out of copper wire and thermocouple wire of the type required. The thermocouple end is terminated with connectors which will mate to the UUT connectors. Of course, these probes must be calibrated if high accuracy is required. Alternatively, internal reference junction compensation can be used. Frequently, readouts equipped with internal refer-

ence junction compensation have thermocouple connectors built in. External reference junctions are capable of the highest accuracy and are almost always used for calibration of noble metal (R and S type) thermocouples. They are generally not necessary for the accuracy requirements for base metal thermocouples.

See Figure 33 on page 74.

Step 4: Measurement of Reference Probe and Temperature Determination

There are two ways to measure the reference probe and determine the temperature. Both techniques have the same potential accuracy. That is, if done correctly, neither technique is inherently more accurate than the other. The first and best method is used with sophisticated readouts designed for temperature work. The resistance is measured and the temperature calculated from calibration coefficients which were entered into the readout previously. Once these calibration coefficients have been entered, the temperature calculations are accomplished internally and the readout displays in temperature units. The temperature data is available in real time. Some modern readouts also display the data in graphical format, allowing the operator to determine stability at a glance. Both of these features speed up the process and eliminate possible operator error due to incorrect table interpolation. The second method is used when the readout does not provide for proper temperature calculation. (Some readouts, particularly DMMs, have some of the more popular temperature conversions built in. These typically do not allow use of unique calibration coefficients and cannot be used for accurate temperature calibration.) In this case, the EMF is measured and the temperature is determined from either a calibration table or from a computer or calculator program. Since the temperature must be calculated after the EMF is measured, the process is slower and does not provide immediate, real time temperature data. If the temperature source is not stable errors will be introduced due to the time delay. Also, interpolation from a calibration table can lead to large errors if done incorrectly. Use of a calibration table may seem daunting, but with a little practice it can be mastered. See Table 9 below.

Table 9. Interpolation from a thermocouple table

°C	0	1	2	3	4	5	6	7	8	9
1000	9.5624	9.5739	9.5854	9.5969	9.6084	9.6199	9.6314	9.6430	9.6545	9.6660
1010	9.6775	9.6891	9.7006	9.7121	9.7237	9.7352	9.7468	9.7583	9.7699	9.7815
1020	9.7930	9.8046	9.8161	9.8277	9.8393	9.8509	9.8625	9.8740	9.8856	9.8972
1030	9.9088	9.9204	9.9320	9.9436	9.9552	9.9668	9.9784	9.9900	10.0017	10.0133

- | | |
|---|--|
| 1) Measure reference probe voltage | 9.5802 |
| 2) Locate where it falls on the table | between 9.5739 and 9.5854 |
| 3) Subtract lower table value from measured value | $9.5802 - 9.5739 = 0.006$ |
| 4) Subtract lower table value from higher value | $9.5854 - 9.5739 = 0.0115$ |
| 5) Divide by difference (slope of curve) | $0.0063 / 0.0115 = 0.548$ |
| 6) Add fractional temperature to table value | $0.548 + 1001 = \mathbf{1001.548^\circ\text{C}}$ |

Step 5: Measurement of UUTs

Since the UUTs are thermocouple thermometers similar to the reference probe, they are measured in a similar manner. If several UUTs are undergoing calibration, ensure that when they are connected or switched in, sufficient time is allowed for the voltage measurement to stabi-

lize before recording the data. For base metal thermocouples, the measurements are conducted starting at the lowest temperature of calibration and working up. For noble metal thermocouples, measurements are conducted in the opposite direction. Additionally, it increases the precision of the calibration to use a mean (average) value calculated from multiple measurements at the same temperature. Often, the readout is designed with statistical features to facilitate this practice. It is also a good practice to close the process with an additional measurement of the reference probe. The sequence in which the probes (reference and UUT) are measured is referred to as a measurement scheme. There are many variables to consider when designing a measurement scheme. Some points to consider are:

- Accuracy - the higher the accuracy desired, the more all of the following must be considered.
- Temperature source stability - the more stable the source, the more time exists to conduct the measurements before temperature changes cause unwanted error.
- Number of UUTs - the higher the number, the longer it takes to cycle through all UUTs.
- Number of readouts - will the reference probe and UUTs be measured with the same readout or different readouts?
- Type of readout - a readout designed for temperature calibration often has features which allow flexibility in the measurement scheme.
- UUT characteristics - self-heating time, source current requirements, stability, and overall quality influence the measurement process.

Step 6: Computation of Errors

The computation of errors is straightforward. The simplest and most common method is available when the readout used for the UUTs has the thermocouple curves built in. In this case, it can be set to perform the measurement and display directly in temperature according to the type of thermocouple being tested. The error is then simply the difference in temperature readings based on the UUT and the reference probe. If DMMs were used to perform the calibration, then the data is fitted to the standard tables as described previously for interpolation of the reference probe. In this case, both the reference probe and the UUT are interpolated according to their specific tables and the UUT error is the difference between the two results. Alternatively, the calibration temperature can be set precisely to a value from the table and the resistance of the UUT compared to a reference value from that table at that specific temperature. With this method, the calculations are easier to perform because interpolation is performed on only the reference probe, but setup can be more difficult because the temperature source must be set and maintained at a precise value. The ASTM specifications are available in many publications and will not be discussed here.

Characterization

Thermocouple characterization is reserved for high accuracy applications involving mostly noble metal thermocouples. Under most circumstances, base metal thermocouples are not stable and will not reproduce the behavior observed during characterization. One exception to this is type T thermocouples for use at cryogenic temperatures. This type of thermocouple has proven itself to be quite stable when used only below zero and can be successfully characterized for use at these temperatures. As mentioned previously, characterization of a thermocouple involves determining the difference between the measured EMF and the standard EMF

and correcting this difference by fitting it to a second order polynomial. This provides the benefits of high order polynomial precision (from the reference function) and the simplicity of lower order fitting. Additionally, work has shown that higher order polynomial “deviation functions” will generally not improve the fit, so a second order polynomial seems to be the correct choice. Fitting the data is simple in concept but can be complicated in practice. Essentially, it is a process of solving a set of simultaneous equations which contain the calibration data to arrive at a set of coefficients unique to the thermocouple and calibration. There are a few commercial software programs available specifically written to accomplish this task. For metrologists who wish to tackle the algorithms themselves, a good mathematics application software like Mathcad or Mathematica or a good spreadsheet like Excel is extremely helpful. Of course, programs can be written in any of the modern computer languages (with double precision or better floating point capability) to perform the calculations with equal accuracy. The steps necessary to fit the data will be discussed in the section on mathematics later in this manual.

Liquid-In-Glass Thermometers

Characteristics

A liquid in glass (LIG) thermometer is a thermometer which relies upon temperature dependent expansion of a liquid to indicate temperature. It is the oldest known type of thermometer, having been in use for over 200 years. Modern style LIGs have been used for decades in many applications ranging from low to very high accuracy. LIGs differ from the types of thermometers discussed thus far in that they are mechanical rather than electrical. LIGs can be constructed using several different thermometric liquids. As a result, there are many different types of LIGs available. Some LIG characteristics are:

- Wide temperature range (-200°C to 500°C)
- Very fragile
- Stable over time
- Easy to measure
- Easy to calibrate
- Cannot be automated
- Commercially available in many configurations

LIGs have a much wider temperature range than one would expect. The range is limited primarily by the type of thermometric liquid used. Table 10 shows the more common thermometric fluid characteristics. Because of construction limitations, a particular LIG thermometer may not be usable over the range suggested by the LIG tables for that type of thermometric liquid. Because these thermometers rely on physical length, resolution vs. range is the limiting factor. For example, a 24 inch mercury LIG with 0.1 inch scale markings in 0.5°C increments could have a maximum range of about 100°C , leaving 4 inches for the expansion chamber and bulb.

$$5^{\circ}\text{C}/\text{inch} \cdot 20 \text{ inches} = 100^{\circ}\text{C}$$

Table 10. Common Thermometric Liquids

Liquid	Typical apparent expansion coefficient ($^{\circ}\text{C}^{-1}$)	Possible Temperature Range
Mercury	0.00016	-35 to 510 $^{\circ}\text{C}$
Ethanol	0.00104	-80 to 60 $^{\circ}\text{C}$
Pentane	0.00145	-200 to 30 $^{\circ}\text{C}$
Toluene	0.00103	-80 to 100 $^{\circ}\text{C}$

LIG thermometers come in three types based upon immersion requirements. Complete immersion, total immersion and partial immersion. Complete immersion thermometers are for applications where the thermometer is completely immersed in the temperature to be measured, such as a freezer or oven. Total immersion thermometers require immersion up to the point of the liquid column, the space above the column should not be immersed. Partial immersion thermometers have an immersion depth which is indicated by an immersion line on the stem. Generally, total immersion thermometers are capable of the highest accuracy.

Instruments, Standards, and Apparatus

Calibration is performed by direct indication on the thermometer scale while the UUT is immersed to the proper depth in a temperature bath. Three instruments are required as follows:

- 1) Reference probe
- 2) Readout for the reference
- 3) Temperature source

Reference Probe

Depending upon the accuracy required, the reference probe will be either an SPRT a PRT, or a LIG of better quality and calibration than the UUTs. Since this instrument forms the basis for our calibration, its accuracy and stability are of paramount importance.

SPRTs

SPRTs are the most accurate and stable instruments available for this purpose. Generally a 25 or 100 Ω glass sheath version is used. A typical glass sheathed 25 Ω SPRT with a high quality calibration will have calibration uncertainties from 0.001 $^{\circ}\text{C}$ to 0.010 $^{\circ}\text{C}$ over the temperature range of LIGs. Additionally, since these instruments are actually part of the definition of the ITS-90, they are standardized. That is, there are minimum requirements for the purity of the platinum wire and the type of construction used. This results in less confusion as to the suitability of the instrument for a particular application and almost guaranteed good performance if calibrated and used correctly. These instruments are highly stable and accurate, but they are expensive and extremely delicate. They should be reserved for high accuracy applications only.

PRTs

When accuracy requirements are less severe, PRTs can be used successfully. PRTs are available in many configurations, however PRTs which are suitable for use as calibration standards are generally available as 100 Ω stainless steel sheathed probes. These instruments are not as accurate as SPRTs but they are generally more rugged and easier to work with. Additionally,

unlike SPRTs, the design of PRTs is left to the discretion and ingenuity of the manufacturer. Not all designs perform to the level required for use as a reference. Be careful in the selection of a PRT to ensure that the type selected is appropriate for use as a calibration reference over the range of interest and with the required accuracy.

LIGs

Reference grade LIGs are available with uncertainties and stability approaching a PRT. Reference quality LIG thermometers are almost exclusively total immersion types. Use of a LIG thermometer as a reference would eliminate the need for a readout.

Special Considerations

In addition to accuracy requirements, there are other characteristics which must be considered. For example, if the UUTs are partial immersion LIGs and the reference is a total immersion LIG they will invariably be immersed to different depths during portions of the calibration. If the calibration bath has vertical gradients, calibration errors will result. Additionally, reference grade LIG thermometers generally have small scale markings. If this is the case, some form of magnification may be required to properly read the scale. These points and others must be understood and taken into account in the selection of a reference probe.

Table II. Reference Probe Characteristics Summary Table

SPRT / PRT	LIG
Capable of very high to high accuracy	Capable of moderate to high accuracy
Extremely stable	Very stable
Available in various sheath materials	
Standardized	Standardized
Relatively expensive to purchase	Relatively inexpensive to purchase
Relatively expensive to calibrate	Relatively inexpensive to calibrate
Extremely delicate	Very fragile

Readout

LIG thermometers do not require readouts. Any readout requirement will apply to the type of reference probe selected. This information was covered in detail previously so we will not repeat that information here.

Temperature Source

As with readouts, temperature sources were discussed in depth previously. We will review points that pertain specifically to LIG calibration applications here. For additional information refer to the section on temperature sources. The most common temperature sources for LIG calibration are ice baths and calibration baths. Because of poor thermal contact and immersion problems, dry-wells are generally not acceptable. For the lowest temperatures (below -100°C) an LN₂ comparison device or cryostat is required. The main considerations are immersion depth and fluid level. LIG thermometers are frequently very long with lengths 12 to 24 inches being quite common. Total immersion types may require immersion of 20 inches or more for the temperatures at the top of the scale. Very deep calibration baths are required. Additionally, since the scale must be observed to take the measurement, the fluid level meniscus must be viewable. The first problem can be corrected by applying what is known as emergent stem corrections. This is a technique to correct for the portion of the stem which should be immersed but is not. It is based upon actual stem temperature, expansion coefficients, and scale

markings. If done correctly, introduced errors are minimal. The second problem can only be solved by either providing a positive meniscus or by providing an unobstructed view of the UUT. Calibration baths have been designed specifically for LIG thermometer calibration which solve these problems. Regular calibration baths can often be adapted to fulfill the unique requirements of LIG calibration.

Calibration Procedure

Introduction

As with PRTs, thermistors, and thermocouples, there are two types of calibration applicable to LIGs - characterization and tolerance testing. However, the difference between the two types of calibration are minimal. Characterization of a LIG is possible because they are generally more stable than accurate. The stability is a result of the materials and construction, whereas the accuracy is dependent upon the scale markings and the uniformity of the capillary. The scale is usually the major source of error. Since the scale location is permanent, these errors must be noted and removed by correction. This is generally performed at cardinal scale points and recorded for future reference. The user can apply these corrections during use to increase the precision of the measurement. This type of correction is reserved for the highest quality LIGs for use in demanding or regulated applications. For the most part, applications requiring high levels of accuracy use one of the electronic thermometers discussed previously. The following section will discuss LIG calibration procedures. There are four basic steps to perform as listed below:

- 1) Place the reference probe and the UUTs in the temperature source in close proximity to one another.
- 2) Measure the reference probe and determine the temperature.
- 3) Measure and record the UUT indication(s).
- 4) Correct for emergent stem if applicable.

Some readouts simplify the technique by combining or eliminating some of the steps. In the following discussion, we will consider an application involving LIG calibration by comparison to an SPRT with a direct indicating readout.

Step 1: Thermometer Placement

All temperature sources have instabilities and gradients. These translate into calibration errors and/or uncertainties. To minimize the effects, the thermometers should be placed as close together as practical. The UUTs should be placed in a radial pattern with the reference probe in the center (focus) of the circle. This ensures an equal distance from the reference probe to each of the UUTs. A device which holds the UUTs in a vertical position will reduce parallax errors. Additionally, it is very helpful if the UUT holder can be rotated. This will allow each UUT to be rotated into direct line of sight and provide for a more convenient calibration. The UUTs should be immersed to the proper level as indicated by the immersion line (partial immersion LIG) or to within a few mm of the scale marking corresponding to the temperature of calibration (total immersion LIG). The sensing element of the SPRT should be at the approximate depth of the UUT bulbs.

Steps 2: Measurement of the Reference Probe and Determination of Temperature

In this example, the readout is a direct indicating type. Simply note or record the indication.

Step 3: Measurement of the UUT

LIG indications should be read to a fraction of 1/4, 1/5, or 1/10 of a minor scale division. A magnifier or telemicroscope is very helpful. Ensure that the eye is at the same level as the meniscus to avoid parallax errors. Also, when interpolating between scale markings, use the center of the markings, not the edges. Refer to Figure 34 below.

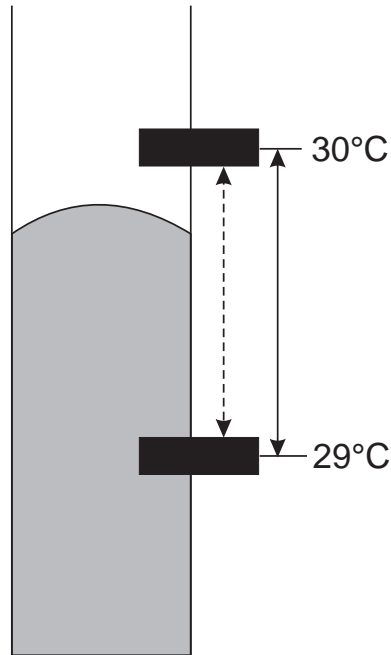


Figure 34. LIG scale interpolation

Step 4: Emergent Stem Correction

Emergent stem corrections are required when proper immersion cannot be achieved because either the bath lacks sufficient depth or proper immersion would prevent viewing of the UUT scale. These corrections are based upon the difference between the temperature that the stem should have been (the calibration temperature) and the actual temperature of the emergent stem, the emergent length (in scale divisions), and the expansion coefficients of the thermometric liquid and glass stem. The emergent stem temperature must be measured. Usually, this is accomplished with a special LIG thermometer called a faden thermometer. This is a LIG thermometer with a very long bulb designed to measure the average temperature of the emergent stem. The thermometer is placed in close proximity to the UUT with the bulb extending the length of the emergent stem. Faden thermometers are available with various bulb lengths. If a faden thermometer is not available, normal thermometers can be used. Two will be required, one is placed at the bottom and one is placed at the top of the emergent stem. The mean temperature is used for the calculation.

The calculations are as follows:

$$\text{Stem correction} = k \cdot n(t_1 - t) \quad (15)$$

Where: k = differential expansion coefficient for thermometric liquid in the particular type of glass of which the thermometer is made.
 n = length of emergent stem (in degrees)

t_1 = temperature of the bath
 t = mean temperature of the emergent stem

For example, suppose the observed reading in a 85°C bath was 84.76°C and the thermometer was immersed to the 20°C mark on the scale so that 65°C of the column projected into the air, and the measured temperature of the stem was 38°C, then

$$\text{Stem correction} = 0.00016 \cdot 65(85 - 38) = 0.49$$

$$\text{Corrected temperature} = 84.76 + 0.49 = 85.25$$

Table 12. Values of *k* for Mercury in Glass Thermometers

For Celsius Thermometers			For Fahrenheit Thermometers		
mean temp (t_1+t)/2	<i>k</i> for "normal" glass	<i>k</i> for "borosilicate" glass	mean temp (t_1+t)/2	<i>k</i> for "normal" glass	<i>k</i> for "borosilicate" glass
0°C	0.000158	0.000164	0 °F	0.000088	0.000091
100°C	0.000158	0.000164	200 °F	0.000088	0.000091
150°C	0.000158	0.000165	300 °F	0.000088	0.000092
200°C	0.000159	0.000167	400 °F	0.000089	0.000093
250°C	0.000161	0.000170	500 °F	0.000090	0.000095
300°C	0.000164	0.000174	600 °F	0.000092	0.000097
350°C		0.000178	700 °F		0.000100
400°C		0.000183	800 °F		0.000103
450°C		0.000188			

Regulatory Requirements

Regulatory bodies place requirements on thermometer performance and testing methods which often increase the complexity of calibration. The goal of these bodies is to standardize calibration techniques and improve the quality of the final result. The American Society for Testing and Materials (ASTM) exists to aid in this effort. For decades, it has created, reviewed, modified, and tested countless standards regarding all forms of industrial supplies and methods. Among the body of standards are comprehensive guidelines for liquid in glass, platinum resistance, and thermocouple thermometer performance and testing. Rather than duplicate this work, most US industries and regulatory bodies specify compliance to ASTM standards. Some of these industries are: automotive industry, aerospace industry, defense industry, pharmaceutical industry, public utilities, food producers, and countless others. Regulatory and standardization bodies include the Society of Automotive Engineers (SAE), Military Standards (Mil-Std), Nuclear Regulatory Commission (NRC), and the Food and Drug Administration (FDA), among others. In some cases, duplicate standards are written (such as SAE standards on thermocouple testing), but these are generally based on ASTM procedures.

The ASTM standards specify the construction methods and minimum performance standards for thermometers used for specific tests and detail the calibration and testing requirements for these thermometers. Additionally, some of these standards provide requirements for proper use of the covered thermometers. If the industry that we or our customers serve answers to one of these regulatory bodies, our calibrations should comply with the applicable standards. In some situations, the instruments that we are calibrating are not being used for regulated purposes and compliance is not required. Unless knowledge of this situation exists, it would be unwise to presume that it is true. These standards are specific technical instructions and procedures, they exist because they are needed. They are not bureaucratic requirements that serve no real purpose. In some cases, our customers do not even know that these standards exist or that they must comply with them. It is our obligation to provide guidance and instructions to aid our customers in these matters. Their success is our success.

Currently, the requirements for PRTs are undergoing review, but specific requirements exist for LIGs and thermocouple wire and probes.

Liquid-In-Glass Requirements

The ASTM specifies liquid in glass (specifically mercury in glass) thermometers for many tests. These are known as ASTM thermometers and are numbered according to the test for which they are intended. These thermometers have specific construction, performance, and calibration requirements. These requirements include stability, accuracy, and scale precision parameters which may exceed what we would consider as normal expectations for these types of thermometers. In order for these thermometers to provide correct results, they must meet the specifications. If our job requires that we perform calibration of ASTM thermometers, we should familiarize ourselves with the requirements, and follow the procedures specified by the ASTM. The ASTM standards which apply to liquid in glass thermometers are:

- E1 Specification for ASTM Thermometers
- E77 Test Method for Inspection and Verification of Liquid-In-Glass Thermometers

For additional information regarding ASTM requirements, please refer to these documents.

Thermocouple Requirements

The ASTM is widely known for its coverage of thermocouples and thermocouple materials. The familiar limits of error specifications for thermocouple performance are ASTM specifications (taken from standard E-230). ASTM thermocouple standards form the basis of thermocouple requirements for most US and many foreign based industries. If our job requires that we perform any thermocouple calibration, particularly if we must certify entire rolls of thermocouple wire, we should familiarize ourselves with the requirements, and follow the procedures specified by the ASTM. The ASTM standards which apply to thermocouple thermometers and materials are:

- E207 Method of Thermal EMF Test of Single Thermoelement Materials by comparison with a Reference Thermoelement of Similar EMF-Temperature Properties
- E220 Method for Calibration of Thermocouples by Comparison Techniques
- E230 Specifications for Temperature Electromotive Force (EMF) Tables for Standardized Thermocouples

- E235 Specification for Thermocouples, Sheathed, Type K, for Nuclear or Other High Reliability Applications
- E452 Test Method for Calibration of Refractory Metal Thermocouples Using an Optical Pyrometer
- E574 Specification for Duplex, Base-Metal Thermocouple Wire with Glass Fiber or Silica Fiber Insulation
- E585 Specification for Sheathed Base-Metal Thermocouple Materials
- E608 Specification for Metal-Sheathed Base-Metal Thermocouples
- E696 Specification for Tungsten-Rhenium Alloy Thermocouple Wire
- E839 Test Methods for Sheathed Thermocouples and Sheathed Thermocouple Material
- E988 Temperature-Electromotive Force (EMF) Tables for Tungsten-Rhenium Thermocouples
- E1129 Specification for Thermocouple Connectors
- E1159 Specification for Thermocouple Materials, Platinum-Rhodium Alloys, and Platinum
- E1223 Specification for Type N Thermocouple Wire
- E1350 Test Methods for Testing Sheathed Thermocouple Prior to, During, and After Installation

For additional information regarding ASTM requirements, please refer to these documents.

Calibration Techniques

Optimizing Your Calibrations

Introduction

Thus far, we have discussed, among other topics, components of uncertainty and calibration procedures. The question is: How do these relate to our UUT and how do these items fit together to ensure an acceptable calibration? The following section deals with these topics, outlining different approaches to take. Understanding these concepts is key to arriving at a sound calibration of which we are sure and that we can justify to others.

Test Accuracy Ratio (TAR)

Description

A test accuracy ratio (also known as a test uncertainty ratio) is a method of expressing the uncertainty of a measurement in a way which relates the calibration uncertainty to the rated accuracy of the UUT. This method of expression is the preferred method for most applications and has been specifically stated in most of the recent quality assurance (QA) programs that apply to metrology (including the current ANSI/NCSL Z540-1). However, this method is controversial in primary metrology circles and is generally not accepted by measurement scientists. They argue that it is no substitute for proper, thorough uncertainty analysis and that it is simply a way to cover up blunders. This argument may have merit but accuracy ratios are here to stay and are widely relied upon outside of these top echelons of metrology. Measurement scientists do not trust, nor do they depend upon instrument specifications. It is, therefore, not surprising that a method of evaluation which is based, in part, upon instrument specifications is objectionable to them. For the most part, they investigate the actual performance of an instrument and use this data in the ensuing uncertainty analysis. This is the purest form of determining the quality of a measurement but is not practical in many situations. It was because of the complicated, time consuming nature of uncertainty analysis, and the need for a realistic approach to measurement quality that the test accuracy ratio was conceived.

Background

One might wonder where the 4 in the 4:1 standard came from, and how it relates to the statistical concepts in true uncertainty analysis. In the late 1950s and early 1960s, the US military (specifically, the US Air Force), began a comprehensive calibration program. The scientists and statisticians involved in the development set guidelines pertaining to calibration accuracy. At the highest measurement levels, they specified true uncertainty analysis, however, they did not feel that these techniques were warranted for simply verifying whether or not an instrument met its performance criteria. They pioneered the *TAR* concept for these circumstances. Originally, 10:1 was selected to model a statistical probability slightly exceeding a 3 sigma limit (99.7% confidence interval). In the early days of industrial metrology (to be differentiated from scientific metrology) it was not difficult to achieve a 10:1 TAR. Over the years, measuring and test equipment has improved in accuracy. Accuracy of instruments that are in common use in the field today far exceed the measurement standards of yesterday. The meas-

urement standards have improved also, but not at the same rate. As a result, the requirements have been relaxed. The 4:1 TAR is the accepted standard today and more closely models a 2 sigma limit (97% confidence interval). For most applications, this is considered a reasonable compromise.

For the past two decades, conventional QA programs have required a 4:1 or better TAR. Some of these programs are: the programs within the US military, Mil-Std 45662 & Mil-Std 45662A (for military contractors), US Code 10 CFR 50-21 (for the nuclear power industry), International Standards Organization (ISO) Guide 25, and the American National Standards Institute / National Conference of Standards Laboratories (ANSI / NCSL) Z540 (for all calibration laboratories). The ANSI/NCSL Z540 is the current program and was written specifically to satisfy the ISO requirements. This program has been adopted by many US industries and has superseded almost all existing programs. Some sectors of the community have not yet accepted this standard, but in time, they probably will. The statement from the Z540 addressing the TAR (paragraph 10.2b) states:

“The laboratory shall ensure that the calibration uncertainties are sufficiently small so that the adequacy of the measurement is not affected. Well defined and documented measurement assurance techniques or uncertainty analyses may be used to verify the adequacy of the measurement process. If such techniques or analyses are not used, then the collective uncertainty of the measurement standards shall not exceed 25% of the acceptable tolerance (e.g., manufacturer’s specifications) for each characteristic of the measuring and test equipment being calibrated or verified.”

This paragraph suggests that proper uncertainty analysis is recommended, but application of a 4:1 TAR is acceptable. In reality, most choose the TAR route when possible because uncertainty analysis is complicated, time consuming, and simply is not practical in many cases. In its simplest form, the test accuracy ratio is the ratio between the rated accuracy of the UUT and the collective uncertainty of the measurements standards. For example, if the UUT has a rated accuracy of 0.050°C at 100°C, and the collective uncertainty of the measurements standards was 0.010°C at 100°C, the TAR would be 5:1. That is, the uncertainty of the measurement standards is 5 times better than the accuracy of the UUT. In this example, the 4:1 TAR requirement has been satisfied. There are several approved techniques for combining the uncertainty of the standards to arrive at a calibration uncertainty. These will be discussed in the section on measurement uncertainty.

Application

When an instrument is calibrated, errors in the measurement standard are transferred to the UUT. This reduces the measurement accuracy of an instrument in real terms. For example, with a 4:1 TAR, measurement errors can actually approach 125% of the UUTs specification (100% from the UUT to the measurement standard and 25% from the measurement standard to reality). Errors would approach 120% and 110% for TARs of 5:1 and 10:1 respectively. Our actual accuracy is not what it seems. Since 4:1 is the accepted ratio, any technique that models the 125% absolute accuracy should be acceptable. Additionally, the real accuracy is reduced

with each transfer. Table 13 below shows the propagation for several TAR values using simple summation (worst case) results.

Table 13. Propagation for several TAR values.

TRANSFERS	ACCURACY RATIOS					
	1:1	2:1	3:1	4:1	5:1	10:1
1	2.0	1.5	1.33	1.25	1.2	0.1
2	3.0	1.75	1.44	1.31	1.24	1.11
3	4.0	1.88	1.48	1.33	1.25	1.111
5	5.0	1.97	1.5	1.33	1.25	1.1111
Terminal	8	2.0	1.5	1.33	1.25	1.1118

In some cases, even a 4:1 ratio cannot be achieved. Must we accept the reduced confidence interval, or can we apply the concept above to improve the situation? We can reduce the calibration tolerance for the UUT to compensate for the <4:1 condition. Consider the following expression:

$$U_t = U_x - (U_s - 0.25 \cdot U_x) \quad (1)$$

or

$$U_t = 125 \cdot U_x - U_s$$

Where: U_t = new test tolerance
 U_x = UUT specified accuracy
 U_s = calibration uncertainty

For example, if the UUT was rated at 0.055°C and the calibration uncertainty was 0.020°C, the TAR would be 2.75:1. We could accept the reduced confidence interval or recalculate the test tolerances as follows:

$$U_t = 125 \cdot 0.055^\circ \text{C} - 0.020^\circ \text{C}$$

$$U_t = 0.48^\circ \text{C}$$

Reducing the calibration tolerance from 0.055°C to 0.048°C would model the confidence interval of a straight 4:1 TAR. Three precautions to consider: First, if the UUT belongs to a customer outside of your organization, or if you do not have authority to modify calibration tolerances, receive proper approval before using this technique. This technique is approved by the NCSL but is considered a variation and a waiver from the customer may be necessary. Second, if the UUT is marginal in performance, reducing the calibration tolerances may lead to an increase in out of tolerance conditions which are not due to the UUT itself. Consider the above example, if the UUT is at +0.050°C. It would be in tolerance with a 4:1 TAR but out of tolerance with the reduced calibration tolerance. Best results are obtained when this method is only used with instruments which are expected to pass with the reduced tolerances. Finally, use this technique only when it is very difficult, very expensive, or simply impossible to

achieve a straight 4:1 TAR. Do not use this technique if it is merely inconvenient to achieve a 4:1 TAR. For example, there is no reason why a 4:1 TAR cannot be achieved in the example given above. The measurement uncertainty would have to be 0.0135°C or less. This may be challenging, but is certainly achievable without difficulty.

Error Budgeting

Description

An error budget is an analysis tool used to help determine how the uncertainties of a measurement fulfill the TAR before the measurement is even undertaken. The name is somewhat of a misnomer, it should be called an uncertainty budget, but the name has been around for years, so that is how we refer to it. As the name implies, this tool is a budget like any other budget. In a conventional budget, we start out with a sum of money and have portions allocated to individual expenses. The total of the allocated expenses must not exceed the sum of money available. If it does, we go over budget and either have to reduce costs elsewhere or get more money. We are constantly hearing of cost overruns pertaining to government projects. Usually, the contractor who has the project has some form of built in protection in the contract and can rely on receiving more money from the government if he exceeds the budget. In industry, our budgets are more concrete. The money runs out when the money runs out. An error budget should be viewed in this manner.

For example, consider a laboratory improvement project. We made a good case to management and were allocated 75K to improve the facilities and purchase some additional capital equipment. We already have a tentative budget (or we would not have known the level of funding required), so spending the money will not be a problem. However, very seldom do we get exactly what we ask for and we must trim back our plans to spend what we have and get the most benefit. Our original budget looked like this:

ITEM	COST
New Environmental Controls	25,000
New Furniture and Workbenches	5,000
Pentium MMX Computer with Laser Printer	3,800
1 High Precision DMM	8,000
1 Precision Digital Oscilloscope	6,000
1 Universal Calibrator	9,500
1 Precision Frequency Counter	4,000
2 SPRTs with Fixed Point Calibration	9,500
2 Triple Point of Water Cells	1,990
1 TPW Cell Maintenance Bath	9,100
TOTAL	81,890

We are \$6,190 over budget, not bad. We have three options to pull in the project. (1) Remove some of the items, (2) Try to negotiate lower prices for some of the items, and (3) Go back to management and get more money. We have the potential to solve our problem with any one, or a combination of these three approaches. How we eventually reduce the total to acceptable limits depends upon our priorities and the degree of difficulty to apply each option. Error budgeting is the same exercise only with instrument accuracy requirements and measurement uncertainty instead of money.

Application

When we evaluate our calibration requirements, a straightforward budget approach is very effective. We look at the instrument specifications, the desired TAR, the resulting maximum allowable measurement uncertainty, and each individual component of that uncertainty and create our budget. Consider the following example.

UUT is a digital thermometer rated at 0.05°C		
Desired TAR = 4:1		
Maximum allowable measurement uncertainty = (0.05°C)/4	=	0.0125°C
SPRT calibration accuracy	=	0.005°C
Reference readout accuracy	=	0.005°C
Bath Stability	=	0.003°C
Bath Uniformity	=	0.003°C
TOTAL UNCERTAINTY (LINEAR ADDITION)	=	0.016°C

We are over budget. We need to apply one of the three approaches outlined above to bring us within budget. (1) Can we eliminate any of the components from the total measurement uncertainty? Since this a straightforward comparison calibration, all listed components are present and must be considered. There is no method that we can substitute that will totally remove the effects of any of these components. Unfortunately, this is not an option. (2) Can we reduce the effects from any of the individual components to bring our total down? Yes, at a cost. We could have our SPRT calibrated more precisely (by fixed point rather than comparison) at a higher cost. We could purchase a better readout or bath with improved specifications to reduce the contribution from these sources. Perhaps we could use an equilibration block to improve the stability and uniformity at a cost of reduced flexibility in our process. Since we are so close to meeting our requirements, one or two of the above would bring us in. We would have to look at the relative costs to make our decision as to how to proceed. (3) Can we increase our allowable total measurement uncertainty? Possibly, depending upon laboratory policy and customer requirements. The first option is to recalculate the TAR based upon the situation and determine if this is acceptable. With our numbers, this would calculate as follows, $0.05^\circ\text{C} / 0.016^\circ\text{C} = 3.125$. TAR of 3.125. The second option is to reduce the calibration tolerance to model 4:1 TAR as shown in the application section above. With our numbers, this would calculate as follows.

$$U_t = 125 \cdot 0.050^\circ\text{C} - 0.016^\circ\text{C}$$

$$U_t = 0.046^\circ\text{C}$$

If the UUT is likely to pass with this reduced calibration tolerance, and this approach is within our company policy and acceptable to our customer, this could also be an option.

Our example was straightforward. This exercise becomes more complicated as the measurement process becomes more complex, and some calibration methods are not represented by linear addition as shown in our example. However, this method will work in these situations as well and it is a very effective tool for approaching a calibration to ensure good results.

Uncertainty Analysis

Measurement Error

All measurements have error. In the context of measurement, error is the difference between the measured value and the true value. Shown algebraically below:

$$\text{measurement error} = \text{measured value} - \text{true value}$$

Since all measurements have error, the true value is unknown and unknowable. Therefore, the measurement error can only be estimated. Traditionally, measurement professionals have divided error into two types: random and systematic. Random errors are errors that arise from variations in the process from one measurement to another. The causes are continuously fluctuating. These errors are present as a “scatter” around the mean value of the measurement. This scatter may reflect changes in the process or changes in the quantity being measured. Systematic errors are generally considered as those resulting from instrumentation calibrations or other physical phenomena which remain constant during the measurement process. These errors are present as a bias in the measurement, and, if known, can be corrected for.

Uncertainty

Since a measured quantity can never be known without error, to be of any value, the reported quantity must include an estimate of the measurement error present. This estimate is called the uncertainty of the measurement. The uncertainty is the band around the measured value within which the true value is expected to lie. The objective of a measurement is to determine a value for the quantity being measured. The uncertainties are an expression of the quality of this determination. The degree to which that quantity has been determined is reflected in the statement of uncertainty. The smaller the uncertainty, the better the measurement. The magnitude of the uncertainty and the precision of its estimate play a vital role in the overall quality of a measurement. The importance to a laboratory of proper uncertainty analysis cannot be over-emphasized.

International standardization bodies have recently modified the method by which we analyze and state measurement uncertainties. Previously, uncertainties were classified into random and systematic. These categories suggest that we *know where the uncertainties originate*. In actuality, we do not know where they originate, we only know how they appear to manifest themselves. The current technique does not attempt to classify the uncertainties based on where they originate, only on *how they are evaluated*. Thus, the issue of where the uncertainty is thought to have originated may suggest a course of analysis, the method of analysis itself is what will determine its classification. The two classifications are: Type A, those evaluated by statistical methods, and Type B, those evaluated by all other means. An uncertainty arising from a measurement instrument can be evaluated statistically as a type-A, or its accuracy after calibration can be propagated and evaluated as a type-B, whichever seems more appropriate under the circumstances. Either method, if done properly, is correct. After the uncertainties are evaluated, they are combined, expanded (by a coverage factor, similar to the “sigma” number), and reported. The remainder of this section will address these issues.

Statistical Uncertainties - Type A Evaluation

In resistance thermometry, the uncertainties generally evaluated statistically are: 1) repeated measurements of the check standard(s), 2) switch or multiplexer noise, and, 3) the precision of the mathematical expression used to relate resistance to temperature. Other uncertainty sources can be evaluated statistically, such as readout linearity, self-heating effects, and effects due to the calibration medium.

Check Standards

A check standard provides a means of characterizing the behavior of a measurement process by way of repeated measurements of the same artifact, combination of artifacts, or instrument over a substantial period of time and over fluctuating environmental conditions. The instrument chosen as a check standard should be similar in type, design, and quality to the instruments that are normally calibrated in the process being analyzed. For example, in the comparison processes, SPRTs, PRTs, and thermistors are used as check standards in each of the respective processes. In the fixed point process, high quality SPRTs are used as check standards. For the results to accurately represent the UUTs, the check standard should be measured as if it were a UUT. It should not be treated differently. Also, the check standard should be included in each calibration run.

After the measurement, the data is converted to the unit to best represent the process and plotted on a control chart. The unit to plot depends on the process. For example, in the PRT comparison process, the resistance of the check standard is plotted. In the fixed point process, the resistance ratio (W) and the resistance at the triple point of water (R_{TPW}) of the check standard are plotted. The measurement data are plotted against the vertical axis and the measurement sequence along the horizontal axis. After a sufficient number of data are collected, the sample standard deviation can be calculated. The check standard standard deviation is one

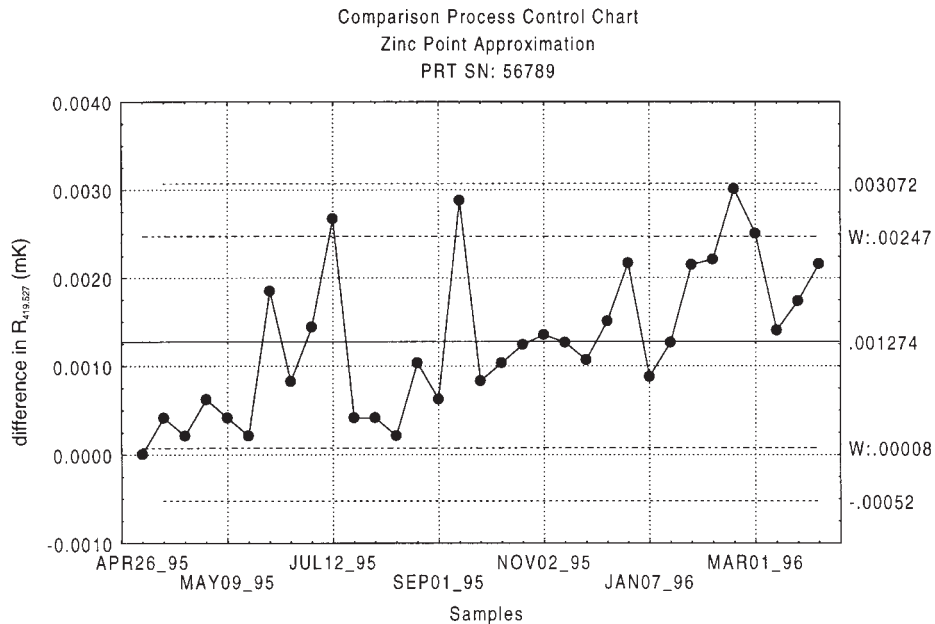


Figure 35. PRT Zinc Comparison Point Process Control Chart.

component of uncertainty evaluated statistically. This is denoted as U_{a1} . It is important to understand that for the standard deviation calculated from the check standard data to represent the uncertainties of typical UUTs, the check standard has to model the behavior of the UUTs. If our check standard was more stable than the UUTs normally calibrated in the process, the standard deviation calculated from our data would *underestimate* the variability in the process *as it relates to the UUTs*. In this context, the UUTs are an integral part of the process.

The control chart is used primarily as a graphical representation of the process. The process parameters calculated from the data describe the process. The control chart, on the other hand, will illustrate drift, random behavior (a positive attribute), structure (a negative attribute), and outliers. Each of these features has significance in the process. Examples of control charts for both the comparison and fixed point process are shown in Figure 35.

At first, the process parameters are considered preliminary. Each successive data point will cause a change in the calculated process parameters. Unless a major change occurs, do not recalculate the uncertainty based on the more recent U_{a1} . Allow a sufficient period of time to pass for the process to mature. During this period of time, pay very close attention to the control chart. (This will be discussed in more detail later.) After a year or so, sufficient data should have accumulated to allow the process parameters to be updated.

Multiplexer Noise

Every switching system has noise associated with it. This noise is easily evaluated statistically in one of two ways. First, the data can be captured as an inherent component of the check standard data. If the check standard is moved from one switch position to another in a somewhat random manner from calibration run to calibration run, differences in the switch positions will be included with the check standard data. The second method is required if the check standard cannot be moved from position to position or if the switch is “hardwired” to standard resistors or specific calibration bath positions (thus being associated with specific temperatures). In either of these cases, the switch must be tested by itself. The switch manufacturer can suggest a technique which is appropriate for the switch in question. In some cases, the switch might have characteristics that suggest that it should be tested individually. Again, the manufacturer can offer guidance in this matter. If the switch is tested individually, the result would be denoted as U_{a2} . Since the noise is related to components that age and deteriorate, the test should be repeated at regular intervals.

Function Analysis

The mathematical relation that is used to describe the behavior of the UUT does not model the behavior perfectly. There will be some error. Usually, the scientific body or individual researcher who provided (or suggested) the model will also provide an analysis of the fit. The description of the analysis as well as the result is generally available. Under almost all circumstances, this evaluation was carried out using statistical techniques. If the analysis is consistent with our use of the model, the results will represent the errors in our fitting as well. If our use of the model differs in some way, we may have to perform the evaluation ourselves, using our process. For example, the ITS-90 functions were established to fit SPRTs. Many researchers have studied the performance of these equations as they relate to real SPRT behavior. We would be fairly safe in assuming that the results represent the behavior of our SPRT as well. If, however, we use these functions to model the behavior of secondary-PRTs, the analysis would not represent our use. Under these circumstances, we would have to perform an evaluation ourselves. An example of such an analysis is provided in reference 3. We would denote this component as U_{a3} .

Other Type A Analyses

Statistical techniques are generally the preferred method by which to analyze uncertainties. This is true because the techniques are straightforward and very little guesswork is required. For this reason, any uncertainty component that can be evaluated statistically usually is. The following are some examples. If used, these would be denoted as U_{an} .

Readout Linearity

Most of us have our readout calibrated and use the manufacturer's specification for accuracy or linearity as the component of uncertainty arising from this source. This would be a type B evaluation. However, readout linearity can be evaluated statistically if a process is established for it. NIST uses this method for the bridge in the NIST SPRT laboratory. This is a satisfactory method of analysis for instrument uncertainty if performed correctly. Most commercial laboratories do not choose to analyze instrument uncertainty in this manner.

Errors From Self-Heating

PRTs self-heat as a result of the current passing through them. If a measurement process induces a self-heating situation different than that experienced in actual use, an error will result. It may be possible to evaluate this error statistically. For example, in our laboratory we use the direct comparison technique with a readout and multiplexer. The current is always flowing through the standard SPRT, but it is only flowing through the UUT during the time when the UUT is switched in the circuit. We can consider this a "duty cycle" with a period of $1/n$, where n represents the number of UUTs. In a four UUT situation, the duty cycle would be 0.25 or 25%. The current is passing through each UUT 25% of the time. To analyze this statistically, we might perform a series of experiments where we increase the number of UUTs in steps from 1 (duty cycle of 100%) to the maximum for our multiplexer (10, duty cycle of 10%). We would then determine the differences statistically and include this in our uncertainty analysis. This should be repeated at various temperatures and in the various baths, cryostats, and furnaces we use. Included in this evaluation are any components resulting from spurious EMFs and bath fluctuations.

Errors From The Calibration Medium

If the calibration medium has gradients, it can be evaluated statistically with the check standard or by itself. For example, in a cryostat with an equilibration block the check standard can be placed in different reentrant wells for each run. Any gradients would be included in the check standard data. In a calibration bath, it might be more appropriate and convenient to test the calibration zone with an SPRT or PRT and determine the gradients explicitly. This data would be evaluated statistically and included with the other type A results.

Non-Statistical Uncertainties - Type B Evaluation

In resistance thermometry, the uncertainties generally evaluated by methods other than statistical methods are: 1) standard thermometer (SPRT) accuracy, 2) readout accuracy, 3) calibration medium uniformity and stability (if not evaluated statistically), and fixed point realization (if a fixed point process is used). Some of the uncertainty sources evaluated statistically can be evaluated non statistically as well, if the situation warrants it. The uncertainties evaluated in this manner are converted to a "standard deviation'-like quantity" to make combination of all uncertainties easier. Reference 1 discusses the foundation for the conversion in detail. Under most circumstances, this quantity is assumed to be represented by a rectangular distribution. That is to say, the probability that the value will be at any position between the limits is 100%. The value is calculated by the expression below, where U_b is the result and a is the limit given by the instrument specification or the uncertainty from the Report of Calibration.

$$U_b = \frac{a}{\sqrt{3}}$$

SPRT Accuracy

The Report of Calibration for the SPRT should state the uncertainty of the calibration at each defining fixed point or each approximation point. The propagation of errors of the ITS-90 functions have been published in a number of sources and copies are easily obtained. (Propagation will be discussed later.) From this data, the uncertainty resulting from the SPRT at any temperature within its calibrated range can be easily determined. Most calibration laboratories use a coverage factor of 2 when stating the total “Expanded Uncertainty.” The value determined above must be reduced before inclusion in our total uncertainty (we will expand our total later).

Readout Accuracy

The readout accuracy is specified by the manufacturer. If we subject our readout to periodic calibration (as we should), it is appropriate to use the manufacturer’s specifications in our evaluation. It is important that we understand the specification and do not leave any component out of our calculations. Many national laboratories and some independent or commercial laboratories choose to determine the “real” accuracy of the instrument under process conditions. This is an excellent method and should be considered. However, most commercial laboratories, due to quality control requirements, are required to use the manufacturer’s specifications. Although this is not the purest method, it is widely used and accepted.

Calibration Medium Uniformity

Oftentimes, the calibration bath, furnace, or cryostat is specified by the manufacturer for stability and uniformity. These specifications can be included in the same manner as the readout. However, in many cases, it is advantageous to analyze the calibration medium statistically and include it in the type A evaluation.

Total Uncertainty

Each component of the Type A and Type B uncertainties are combined through either uncertainty propagation techniques or statistical techniques.

In propagation of uncertainty techniques, the effect that an uncertainty in one variable has on the result is determined while holding the other variable(s) constant. Each variable in the mathematical model is thus evaluated. For example, the value of a resistor can be calculated by measuring both the voltage drop across the resistor and the current through the resistor. The uncertainty in the voltage measurement will *propagate* to the result. Similarly, the *uncertainty* in the current measurement will *propagate* to the result. The propagation relation is determined by the relationship each variable has with the result. Partial derivatives can be used to perform this calculation. This method is described in detail in reference 5.

The statistical techniques are much more straightforward. The most common method of combination is the “root-sum-of squares” (square root of the sum of the squares) or RSS method. In this method, each component which has been estimated as a standard deviation is squared and summed with the others. This method assumes independence between variables. Covariances (lack of independence between variables) must be considered and accounted for when using this method. For example, consider the resistance measurement mentioned above. If the same DMM was used to measure both the voltage drop and the current flow, there would clearly be a linkage between the measurements. A covariance would exist. Similarly, if two

DMMs were used, but both were calibrated by the same standard calibrator, a covariance would exist. The former would result in a stronger covariance than the latter. In situations where covariances exist, they must be determined and included. If this is not possible, the *non-independent* components should be summed *linearly*.

The U_a and U_b , once summed, are referred to as the “Standard Uncertainty.” These components must be combined into a final “total” uncertainty. This is designated as the “Combined Standard Uncertainty.” This quantity is taken to represent the estimated standard deviation of the result. It is obtained by combining the individual standard uncertainties (U_a and U_b) using the usual method for combining standard deviations, the RSS method. The combined standard uncertainty is usually *expanded* by the multiplication of a coverage factor (k) to increase the level of confidence. In most national and commercial laboratories, the value of 2 has been chosen for k . Once this has been done, the result is the “Expanded Uncertainty,” and is the uncertainty reported for our measurement. Two examples of uncertainty evaluation are included in Appendix A.

SPC

Statistical process control when applied to metrology, is a technique used to determine if the calibration process is behaving predictably. That is, that the process is behaving as our statistical parameters say it is. Each check standard measurement is subjected to a statistical test for control. The outcome of that test is used as a mechanism for accepting or rejecting the results of the measurement process. During the initial stages of the process, when it is being characterized and is not yet “in control” the statistical process parameters are being established. The control chart can be used as a visual aid in assessing the progress. It will provide a visual indication that all is well or that something is wrong long before the numbers themselves appear to reflect a pattern. Once the process is mature, the control chart is used as a visual indication of the “health” of the process. Some process phenomena are very difficult to detect without the aid of the control chart. Structure (sine-wave-like curve) and frequent outliers are some examples. There are several good computer applications which can be of use in maintaining the control chart and calculating the check statistic mentioned earlier in this paragraph. It is important to be disciplined in the taking of data and in maintaining the control chart. More detail about this topic can be obtained from reference 2.

Calibration Techniques

Mathematics

Introduction

The main equations used for fitting PRTs and thermistors were introduced in the section on common calibration techniques. Here we will show the steps necessary to actually fit the data to these equations. As mentioned previously, data fitting is an exercise in simultaneously solving equations which contain the calibration data. There are several approaches to this exercise. The two most common methods are the least squares method and iteration. The least squares technique is a statistical curve fitting technique which is used to find the best curve for a set of data. It is very effective and quite accurate. Iteration is a technique used in software applications and computer programs where guess values (or seed values) are substituted for the unknowns and the software computes solutions while adjusting the unknowns until convergence occurs. Since complete convergence seldom occurs, the computer program must be told when the answer is good enough by being given tolerances. It will stop computing when coefficients are found which satisfy the tolerances. Both of these techniques are effective and will provide approximate, and usually very accurate, solutions. The third technique uses matrix methods to simultaneously solve the equations. This technique is especially effective and always provides the best solution possible with the given set of data. Iterative and least squares methods are described in detail in many publications so we will not illustrate them here. The following examples detail the step by step procedures using matrix methods. Examples are provided for all of the equations described thus far.

ITS-90

The ITS-90 series of functions was created for SPRTs, not for “regular” PRTs. However, it works very well for these instruments if a couple of precautions are taken. First, fit the data using more data points than the equation requires. This provides an overdetermined solution and a means for verification of a good fit through residual analysis. Second, pay close attention to the magnitude of the residuals, they will differ from probe to probe and from calibration to calibration. Unlike fitting an SPRT to the ITS-90, fitting a PRT to the ITS-90 is an inexact procedure and good results are not guaranteed.

The key to a good fit with secondary-PRTs is a sufficient number of data points. In this case the data points provide necessary information to provide an acceptable fit. It turns out that, depending on the PRT design, one or two extra data points between the ITS-90 fixed-point values is sufficient. More data will not improve the fit of a “bad” PRT to the point of being acceptable. Nor will it provide necessary information to significantly improve the fit of a “good” PRT. Whether one point or two is necessary depends basically on how closely the PRT “behaves” like an SPRT. Residual analysis will reveal this characteristic. Once we have determined what is necessary for a given PRT design, we can stick with it as long as the design is not changed. If the design has been changed without our knowledge, the residuals will increase, telling us that investigation may be prudent.

Solving for ITS-90 coefficients is straightforward and not difficult once understood. The high order polynomial reference functions are easily handled by modern computers with double precision math applications. The deviation functions are simply second or third order polynomials which are solved as a series of simultaneous solutions to sets of equations. Follow the example below.

1) The following data was obtained for the UUT.

Temperature	UUT Resistance
0.010°C	99.96653
156.599°C	160.89476
231.928°C	189.16982
300.000°C	214.15407
419.527°C	256.72668

2) Calculate the UUT resistance ratio (W) and deviation (ΔW) at the temperatures.

Temperature (K)	Measured Resistance	UUT R _{TPW}	UUT W _{T90}	W _{refT90}	UUT W _{T90}
429.749	160.89476		1.6094863	1.6098037	-3.174 E-04
595.078	189.16982	+ 99.96653 =	1.8923317	- 1.8927977 =	-4.660 E-04
573.150	214.15407		2.1422579	2.1428403	-5.824 E-04
692.677	256.72668		2.5681264	2.5689173	-7.909 E-04

3) Simultaneously solve the set of equations for the coefficients *a* and *b* using matrix methods.

$$\begin{aligned}
 \Delta W_{T_1} &= [a(W_{T_1} - 1) + b(W_{T_1} - 1)^2] \\
 \Delta W_{T_2} &= [a(W_{T_2} - 1) + b(W_{T_2} - 1)^2] \\
 \Delta W_{T_3} &= [a(W_{T_3} - 1) + b(W_{T_3} - 1)^2] \\
 \Delta W_{T_4} &= [a(W_{T_4} - 1) + b(W_{T_4} - 1)^2]
 \end{aligned}$$

$$\begin{aligned}
 \text{Matrix}_1 &= \begin{bmatrix} \Delta W_{T_1} \\ \Delta W_{T_2} \\ \Delta W_{T_3} \\ \Delta W_{T_4} \end{bmatrix} & \text{Matrix}_2 &= \begin{bmatrix} (W_{T_1} - 1)(W_{T_1} - 1)^2 \\ (W_{T_2} - 1)(W_{T_2} - 1)^2 \\ (W_{T_3} - 1)(W_{T_3} - 1)^2 \\ (W_{T_4} - 1)(W_{T_4} - 1)^2 \end{bmatrix}
 \end{aligned}$$

For a square matrix (i.e., two unknowns in two equations) the solution would be arrived at with the following operation:

$$\text{Solution} = \text{Matrix}_2^{-1} \cdot \text{Matrix}_1$$

Since our matrix is overdetermined (two unknowns in four equations), the following expression is used:

$$\text{Solution} = (\text{Matrix}_2^T \cdot \text{Matrix}_2)^{-1} \text{Matrix}_2^T \cdot \text{Matrix}_1$$

C

$$\text{Solution} = (-5.358167 \text{ 1E} - 04 \quad 2.030704 \text{ 9E} - 5)$$

The coefficients are: a = -5.3581671 E-04, and b = 2.0307049 E-05

4) Finally, calculate the residuals:

- a) Calculate the temperature at each W value with the ITS-90 *inverse functions* using the newly determined coefficients.
- b) Subtract the reference temperatures from the calculated temperatures. The result is the residuals. This is an indication of the precision of the fit and the quality of the data points. The results from our example are shown below.

Calculated Temperature (K)	Actual Temperature (K)	Residuals (K)
429.7494	429.7490	0.0004
595.0770	595.0780	-0.0010
573.1509	573.1500	0.0009
692.6768	692.6770	-0.0002

Callendar-Van Dusen

The Callendar-Van Dusen equation remains one of the most commonly used equations for PRT calibrations. It does not provide as precise a fit as the ITS-90 but is considered acceptable for medium to low accuracy applications. If overdetermined solutions are computed, as described in the preceding section on the ITS-90, insight into the precision of the fit can be obtained. The steps are as follows.

- 1) The following data was obtained for the UUT.

Temperature	UUT Resistance
0.000°C	99.96261
156.599°C	160.89476
231.928°C	189.16982
300.000°C	214.15407
419.527°C	256.72668

- 2) Calculate the UUT resistance ratio (W) at the temperatures.

Temperature (°C)	Measured Resistance	UUT R ₀	UUT W
156.599°C	160.89476		1.6094771
231.928°C	189.16982	+ 99.96261	= = 1.8924258
419.527°C	256.72668		2.5682011
300.000°C	214.15407		2.1424007

3) Simultaneously solve the set of equations for the coefficients A and B using matrix methods.

Notes: (1) $C = 0$ for temperature above 0°C.

(2) The 1 was moved to the left side of the equation for the matrix solution.

$$\begin{aligned}
 &W_{t_1} = 1 + A(t_1) + B(t_1)^2 + C(t_1)^3 \cdot (t_1 - 100) \\
 \mathbf{A} \quad &W_{t_2} = 1 + A(t_2) + B(t_2)^2 + C(t_2)^3 \cdot (t_2 - 100) \\
 &W_{t_3} = 1 + A(t_3) + B(t_3)^2 + C(t_3)^3 \cdot (t_3 - 100) \\
 &W_{t_4} = 1 + A(t_4) + B(t_4)^2 + C(t_4)^3 \cdot (t_4 - 100)
 \end{aligned}$$

$$\mathbf{B} \quad \text{Matrix}_1 = \begin{bmatrix} W_{t_1} - 1 \\ W_{t_2} - 1 \\ W_{t_3} - 1 \\ W_{t_4} - 1 \end{bmatrix} \quad \text{Matrix}_2 = \begin{bmatrix} t_1 & (t_1)^2 \\ t_2 & (t_2)^2 \\ t_3 & (t_3)^2 \\ t_4 & (t_4)^2 \end{bmatrix}$$

For a square matrix (i.e., two unknowns in two equations) the solution would be arrived at with the following operation:

$$\mathbf{C} \quad \text{Solution} = \text{Matrix}_2^{-1} \cdot \text{Matrix}_1$$

Since our matrix is overdetermined (two unknowns in four equations), the following expression is used:

$$\begin{aligned}
 \mathbf{C} \quad \text{Solution} &= (\text{Matrix}_2^T \cdot \text{Matrix}_2)^{-1} \cdot \text{Matrix}_2^T \cdot \text{Matrix}_1 \\
 \text{Solution} &= (3.9836461 \text{ E} - 03 \quad -5.8547918 \text{ E} - 07)
 \end{aligned}$$

The coefficients are: $A = 3.9836461 \text{ E} - 03$, $B = -5.8547918 \text{ E} - 07$, and $C = 0$

4) Finally, calculate the residuals:

- a) Calculate the resistance at each W value with the Callendar-Van Dusen equation using the newly determined coefficients.
- b) Subtract the measured resistance values from the calculated resistance values and divide the results by the nominal slope of the curve ($0.3925 \text{ } \Omega / ^\circ\text{C}$). The result is the residuals. This is an indication of the precision of the fit and the quality of the data points. The results from our example are shown below.

Calculated Resistance ()	Actual Resistance ()	Residuals ()	Residuals (°C)
160.88752	160.89476	-0.00724	-0.0184
189.17181	189.16982	0.00199	0.0051
214.15996	214.15407	0.00589	0.0150
256.72407	256.72668	-0.00261	-0.0067

Polynomials

Two examples of polynomial solutions are shown below. The first is for a PRT and the second is for a thermistor. Note how the natural log function is incorporated in the thermistor example. The procedure is the same for both solutions.

Example 1: PRT

- 1) The following data was obtained for the UUT. Note that a 0°C point is not necessary for polynomial fitting, any desired range can be fitted.

25.0072	110.324
36.9963	115.068
50.0057	120.198
89.9991	135.846
124.9950	149.378

- 2) Simultaneously solve the set of equations for the coefficients A , B , and C using matrix methods.

Note: The temperatures are expressed in Kelvin

$$\begin{aligned}
 T_1 &= A + B \cdot (R_1) + C \cdot (R_1)^2 \\
 T_2 &= A + B \cdot (R_2) + C \cdot (R_2)^2 \\
 \mathbf{A} \quad T_3 &= A + B \cdot (R_3) + C \cdot (R_3)^2 \\
 T_4 &= A + B \cdot (R_4) + C \cdot (R_4)^2 \\
 T_5 &= A + B \cdot (R_5) + C \cdot (R_5)^2
 \end{aligned}$$

$$\mathbf{B} \quad \text{Matrix}_1 = \begin{bmatrix} T_1 \\ T_2 \\ T_3 \\ T_4 \\ T_5 \end{bmatrix} \quad \text{Matrix}_2 = \begin{bmatrix} 1 (R_1) (R_1)^2 \\ 1 (R_2) (R_2)^2 \\ 1 (R_3) (R_3)^2 \\ 1 (R_4) (R_4)^2 \\ 1 (R_5) (R_5)^2 \end{bmatrix}$$

For a square matrix (i.e., two unknowns in two equations) the solution would be arrived at with the following operation:

$$\mathbf{C} \quad \text{Solution} = \text{Matrix}_2^{-1} \cdot \text{Matrix}_1$$

Since our matrix is overdetermined (four unknowns in five equations), the following expression is used:

$$\mathbf{C} \quad \text{Solution} = (\text{Matrix}_2^T \cdot \text{Matrix}_2)^{-1} \cdot \text{Matrix}_2^T \cdot \text{Matrix}_1$$

$$\text{Solution} = (32.221308 \quad 2.3000810 \quad 0.00100119)$$

The coefficients are: A = 32.221308, B = 2.3000810, C = 0.00100119

3) Finally, calculate the residuals:

- a) Calculate temperature values at the measured resistance values using the newly determined coefficients.
- b) Subtract the measured temperature values from the calculated temperature. The result is the residuals. This is an indication of the precision of the fit and the quality of the data points. The results from our example are shown below.

25.0113	25.0072	0.0041
36.9934	36.9963	-0.0029
50.0012	50.0057	-0.0045
90.0042	89.9991	0.0051
124.9931	124.9950	-0.0019

Example 2: Thermistor

- 1) The following data was obtained for the UUT.

0.0068	29678.58
24.9849	10047.65
50.0243	3904.599
74.9874	1708.684
99.9929	822.5296

- 2) Simultaneously solve the set of equations for the coefficients A, B, C, and D using matrix methods.

Note: The temperatures are expressed in Kelvin

$$\begin{aligned}
 \frac{1}{T_1} &= A + B \cdot \ln(R_1) + C \cdot \ln(R_1)^2 + D \cdot \ln(R_1)^3 \\
 \frac{1}{T_2} &= A + B \cdot \ln(R_2) + C \cdot \ln(R_2)^2 + D \cdot \ln(R_2)^3 \\
 \text{A} \quad \frac{1}{T_3} &= A + B \cdot \ln(R_3) + C \cdot \ln(R_3)^2 + D \cdot \ln(R_3)^3 \\
 \frac{1}{T_4} &= A + B \cdot \ln(R_4) + C \cdot \ln(R_4)^2 + D \cdot \ln(R_4)^3 \\
 \frac{1}{T_5} &= A + B \cdot \ln(R_5) + C \cdot \ln(R_5)^2 + D \cdot \ln(R_5)^3
 \end{aligned}$$

$$\text{B} \quad \text{Matrix}_1 = \begin{bmatrix} (T_1)^{-1} \\ (T_2)^{-1} \\ (T_3)^{-1} \\ (T_4)^{-1} \\ (T_5)^{-1} \end{bmatrix} \quad \text{Matrix}_2 = \begin{bmatrix} 1 & \ln(R_1) & \ln(R_1)^2 & \ln(R_1)^3 \\ 1 & \ln(R_2) & \ln(R_2)^2 & \ln(R_2)^3 \\ 1 & \ln(R_3) & \ln(R_3)^2 & \ln(R_3)^3 \\ 1 & \ln(R_4) & \ln(R_4)^2 & \ln(R_4)^3 \\ 1 & \ln(R_5) & \ln(R_5)^2 & \ln(R_5)^3 \end{bmatrix}$$

For a square matrix (i.e., two unknowns in two equations) the solution would be arrived at with the following operation:

$$\text{C} \quad \text{Solution} = \text{Matrix}_2^{-1} \cdot \text{Matrix}_1$$

Since our matrix is overdetermined (four unknowns in five equations), the following expression is used:

$$\begin{aligned}
 \text{C} \quad \text{Solution} &= (\text{Matrix}_2^T \cdot \text{Matrix}_2)^{-1} \cdot \text{Matrix}_2^T \cdot \text{Matrix}_1 \\
 \text{Solution} &= \begin{pmatrix} 1.0218088213\text{E}-3 & 2.3920666017\text{E}-4 \\ 2.4399776706\text{E}-7 & 1.3718081204\text{E}-7 \end{pmatrix}
 \end{aligned}$$

The coefficients are: A = 1.0218088 E-03, B = 2.3920666 E-04, C = 2.4399777 E-07, D = 1.3718081 E-07

3) Finally, calculate the residuals:

- a) Calculate temperature values at the measured resistance values using the newly determined coefficients.
- b) Subtract the measured temperature values from the calculated temperature. The result is the residuals. This is an indication of the precision of the fit and the quality of the data points. The results from our example are shown below.

0.0068	0.0068	0.0000
24.9847	24.9849	-0.0002
50.0246	50.0243	0.0003
74.9871	74.9874	-0.0003
99.9930	99.9929	0.0001

Steinhart-Hart

The Steinhart-Hart equation is a variation of the polynomial previously presented. The example shown below is defined by three unknowns in three equations.

- 1) The following data was obtained for the UUT.

0.0101	29400.46
15.0008	14884.61
29.9989	7965.180

- 2) Simultaneously solve the set of equations for the coefficients A , B , and C using matrix methods.

Note: The temperatures are expressed in Kelvin

$$\mathbf{A} \quad \begin{aligned} \frac{1}{T_1} &= A + B \cdot \ln(R_1) + C \cdot \ln(R_1)^3 \\ \frac{1}{T_2} &= A + B \cdot \ln(R_2) + C \cdot \ln(R_2)^3 \\ \frac{1}{T_3} &= A + B \cdot \ln(R_3) + C \cdot \ln(R_3)^3 \end{aligned}$$

$$\mathbf{B} \quad \text{Matrix}_1 = \begin{bmatrix} (T_1)^{-1} \\ (T_2)^{-1} \\ (T_3)^{-1} \end{bmatrix} \quad \text{Matrix}_2 = \begin{bmatrix} 1 \ln(R_1) \ln(R_1)^2 \\ 1 \ln(R_2) \ln(R_2)^2 \\ 1 \ln(R_3) \ln(R_3)^2 \end{bmatrix}$$

Since our matrix is a square matrix (i.e., three unknowns in three equations) the solution is arrived at using the following operation:

$$\mathbf{C} \quad \text{Solution} = \text{Matrix}_2^{-1} \cdot \text{Matrix}_1$$

The coefficients are: $A = 1.0528049 \text{ E-}03$, $B = 2.3891663 \text{ E-}04$, $C = 1.3762461 \text{ E-}07$

- 3) Since the solution was exact, there is no data with which to calculate the residuals.

Calibration Techniques

Maintaining Your Standards

Introduction

Calibration standards used at the highest echelons of measurement in most fields of metrology require maintenance to ensure continuing proper performance and accuracy. This is not maintenance in a traditional sense akin to tuning up a car, but consists mostly of behavior observation, performance verification, and tracking. The reference instruments used in primary temperature calibration are no different. However, unlike most metrological disciplines, in temperature calibration, this necessity for maintenance reaches down to the secondary level as well. There are genuine reasons for this: First, many of the instruments and techniques used in secondary temperature calibration are derivatives of those used in primary temperature calibration and the maintenance requirements are similar. Second, standards used in temperature calibration drift with time and drift with normal use. This drift will impact the accuracy of the instrument and should be observed. Third, most reference probes are fragile; any rough handling, cold quenching, vibration, or other seemingly innocuous treatment can cause large shifts in characterization and will result in calibration errors. Fourth, if any of these problems exist, they will cause calibration errors which are often transparent to the operator. When the result of a calibration is a set of coefficients, not a simple comparison of indications, errors that occur during calibration likely will not be apparent until the calibration is complete, and then only if specifically checked. Finally, many of the instruments calibrated in a secondary laboratory are calibrated by characterization with accompanying uncertainties, not simple tolerance verification. This places responsibility on the laboratory to ensure that the stated uncertainties are valid. In the case of temperature calibration, this requires that the standards used are known to be performing properly and within requirements. These points will be covered in the following section.

Reference Probes

Most platinum reference probes and some thermistors are specified for stability, rather than accuracy. The level of stability is dependent upon both use and time. In order to ensure continued performance as required, these probes should be regularly monitored. Fortunately, this is not difficult, time consuming, nor expensive. The best way to observe the behavior of a reference probe is to periodically measure the resistance at the triple point of water (R_{tpw}) and plot the results on a control chart. The R_{tpw} will vary some naturally, but large changes will indicate that problems exist. If and when problems do arise, we can resolve them before we proceed. In this way, calibration errors caused by the reference probe can be prevented. The characterization of a platinum probe is affected mainly by three phenomena: stress and strain, cold quenching, and oxidation. Thermistor probes, on the other hand, are both physically and chemically stable and not affected by these phenomena. However they are not immune to problems caused by shock, moisture ingress and outright damage. Additionally, both types of probes, as resistors, exhibit natural aging effects which cause the resistance to drift with time. We will discuss each of these phenomena individually.

Stress and Strain

Stress and strain is caused by normal use. As the platinum element expands and contracts with changes in temperature, or vibrates slightly in calibration baths, tiny deformations occur in and on the platinum wire. These deformations tend to increase the resistance and the effects are cumulative. Over a period of time, the resistance at the triple point of water (R_{tpw}) can increase anywhere from a 0.001 °C to 0.050 °C or more, depending upon the probe and the magnitude and frequency of the stresses and strains. Related to this is shock. This is a one-time event which can cause a very large change in resistance. Dropping the probe and tapping it hard are two common causes of shock. Most changes produced by stress and strain are reversible by annealing (we will discuss annealing later).

Cold Quenching

Cold quenching is caused by rapidly cooling a hot probe. When hot, the crystal structure of the platinum element is expanded. If the probe is cooled quickly, the structure contracts too rapidly for the molecules to reorder themselves properly. This results in incorrect crystal geometry and in what is known as crystal lattice vacancies. Locations in the crystal structure which should contain platinum molecules are vacant. This also tends to cause an increase in resistance but the effects are generally reversible by annealing.

Oxidation

Oxidation is the chemical reaction of the platinum with the oxygen in the probe space. Some oxidation is good and tends to protect the platinum. Too much oxidation will cause an increase in resistance and measurement errors. There are two phases of oxidation with which we are concerned: two dimensional and three dimensional. Two dimensional oxidation is surface oxidation. It builds at temperatures between 200°C and 400 to 450°C. Continuous or frequent use of a reference probe in this temperature range will almost certainly result in formation of a two dimensional oxidation layer. Three dimensional oxidation on the other hand is in the platinum itself, below the surface. It generally forms at temperatures above 400°C and has similar effects as two dimensional oxidation, but usually to a larger degree. Both types of oxidation can be removed, or “burned off” by annealing. Therefore, oxidation effects are also reversible.

Shock, Moisture Ingress, and Damage

Thermistors are generally very stable and not affected by the phenomena described above. However, thermistors are not indestructible. There are three common causes of thermistor problems: physical shock, moisture ingress, and outright damage. Physical shock usually results in a broken thermistor element and an open circuit. Calibration errors generally do not result because the thermistor cannot be used at all. Moisture ingress results when the hermetic seal is compromised and moisture leaks into the probe. This usually results in a resistance error caused by shunting and a decrease in effective resistance. The effect can be subtle or pronounced. Damage to a thermistor probe results when the temperature range is exceeded. Either the thermistor element or the potting compound, or both are damaged. The result is unpredictable. None of these effects are reversible. Generally, when a thermistor probe appears unstable, it requires replacement.

Drift

Drift is a natural occurrence with resistors and can either increase or decrease the resistance of the probe. Generally, it is a very slow process and does not adversely affect probe performance. Changes caused by drift are not reversible.

Annealing

Annealing is a heat treating process which is very effective in removing the problems discussed above. Normally, SPRTs are annealed before calibration begins to place them in a stable, baseline condition in preparation for calibration. When changes occur during use, annealing can usually return the SPRT to that condition, thus preserving the calibration. The annealing procedure for SPRTs differs depending upon the range of the instrument and the type of corrections being attempted. Low temperature annealing is done at 475°C to 480°C for 4 hours. High temperature annealing is done at 670 °C for about 1 hour with a slow ramp down to 480 °C (about 3 hours). When the probe is at 480 °C , it can be removed to ambient. Particularly stubborn problems can sometimes be removed by repeated annealing or long annealing at the above temperatures. Caution should be exercised to ensure that the maximum temperature is not exceeded. Refer to the graph below for an example.

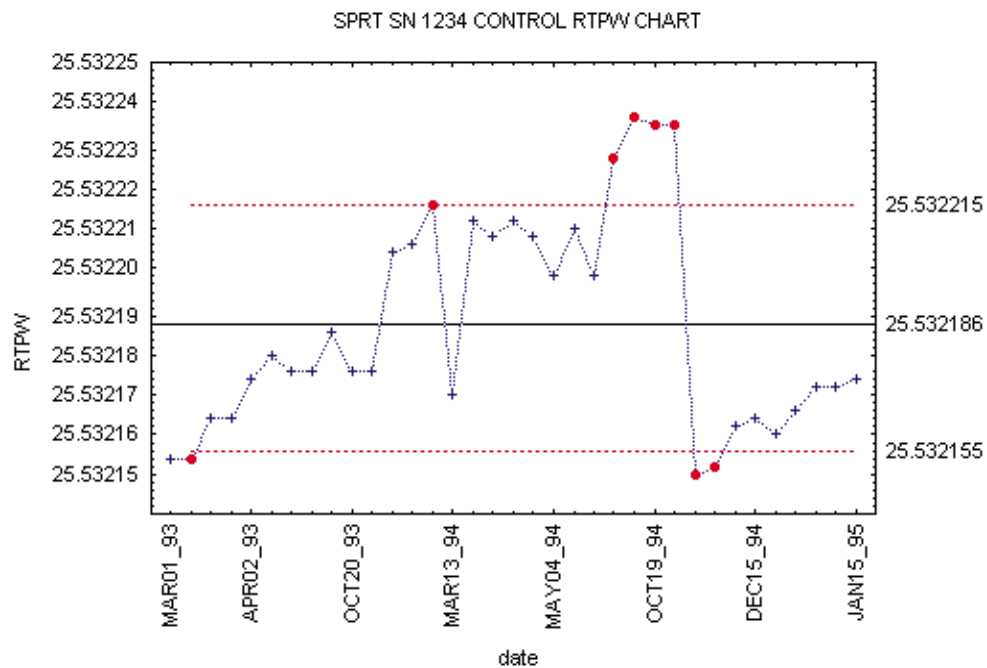


Figure 36. R_{tpw} Control chart for SPRT

Readouts

Most readouts require periodic calibration. In some cases, this will prove sufficient. However, it is wise to subject the readout to interim checks to ensure that it is performing within specifications. The Z540 standard suggests this course. Some of these instruments have self check or self calibration routines while others have zero and unity checks. Many have no self check capability at all. A few of these instruments have internal standard resistors as well which drift (although the drift rate is built into the specifications). There are a couple options available pertaining to readout maintenance which we will describe.

First, for instruments which have legitimate self check capability, routinely perform self checks and log the results. If malfunctions arise which affect measurement performance, they

will surface with the self check. Some instruments have self check capability for the digital portions only. These are of limited value; additional verification is usually required for the analog portions. Some instruments that have legitimate self check routines do not provide a written record. However, these instruments generally halt if an error is found or otherwise indicate when a specific test fails. In these cases, logging the date and successful run status should be sufficient. If an error is indicated, it can be corrected before proceeding. A few of these instruments can download the self check results to a computer or dumb terminal. If this is available, it is wise to keep the records in a file.

Second, for instrument with zero and unity features, it is wise to run these checks frequently, perhaps once a day or before the instrument is used in a specific day. A record of the results should be kept in a logbook or file. This may seem excessive, however, some of the malfunctions with these types of instruments will result in measurement data which seems correct. The zero or unity function will usually catch these types of malfunctions.

Finally, there are instruments which have no capability for self check, or which have internal components subject to drift. These instruments should be checked periodically to ensure continued accuracy. For example, a readout instrument which has internal standard resistors should be checked against external resistors periodically. Any failure discovered can be remedied before calibrations are impacted. If these steps are not taken, failures discovered during routine calibration will have impacted an unknown set of calibrations that had been performed with that instrument.

Temperature Sources

Proper performance of temperature sources is vital to the quality of our calibrations. Instabilities and gradients directly affect calibration accuracy. It is wise to periodically verify that the stability and uniformity remains within limits. This is particularly important if your laboratory provides calibrations where uncertainties are quoted. If temperature source performance deteriorates, then the uncertainties may be incorrect. Fixed point cells have unique maintenance requirements (except for the triple point of water). It is recommended that a set of complete freezing or melting plateaus are recorded at least once a year, starting when the cells are first put into operation. If the shape (slope) of the plateaus change, there may be a problem to be

investigated. If available, it is advisable to include one measurement with an SPRT to verify that the correct temperature is achieved. An example of a complete tin freeze is shown below.

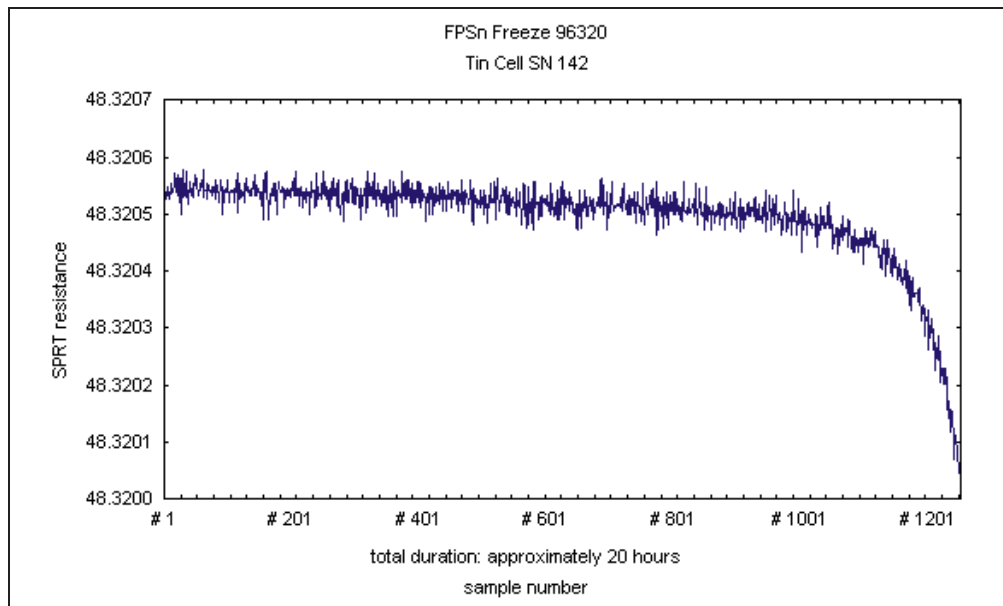


Figure 37. FPSn Cell Plateau

Auxiliary Standards and Instruments

Standard resistors, current sources, decade boxes, and any other auxiliary instruments that are used in critical measurements should be subjected to periodic verification checks. Records of performance should be kept on file.

SPC

SPC was introduced in the section on measurement uncertainty. This tool can be used along with the checks discussed in this section to monitor process performance. Between these two techniques, any variations in the process will be visible as they arise and can be isolated. If rigorous attention is paid to the instruments and the process, calibrations will not be compromised.

Calibration Techniques

Compliance Issues

Introduction

This section will deal with issues pertaining to QA program compliance. The ANSI/NCSL Z540 is the current industry standard. Since this is the accepted program, and the most comprehensive program in use today, we will use this as our model. The purpose of programs such as the Z540 is to provide procedural guidelines for a laboratory to follow which will ensure acceptance within the community. Oftentimes, the purpose is forgotten and the programs become complicated, bureaucratic, self serving entities. The goal of the Z540 committee was to create a program which will satisfy the vague ISO requirements without placing undue restrictive stipulations upon the laboratory. They succeeded. The standard is concise, well written, and thorough, without being overly restrictive or complicated. As a consequence of the varied community this document is intended to serve, compromises were inevitable. There are some features which will create difficulties and others which certain readers will feel are too liberal.

This section will discuss many of the aspects of the Z540, and hopefully dispel some of the myths. Our main text is the document itself, which is contained in the appendix. We will also go over requirements for calibration reports and tables, and examples will be presented of each.

ANSI/NCSL Z540

Structure and Contents

The document covers requirements pertaining to the calibration laboratory operation and to the measuring and test equipment (M&TE) used in the laboratory and calibrated by the laboratory. It is divided into three parts, each dealing with general requirements and specific requirements pertaining to these subjects. Like most documents of this nature, it has an accompanying document to help guide in the interpretation of the main document.

The following list delineates the contents of the document, we will be referring to the actual document for the following discussion.

Calibration Laboratories and Measuring and Test Equipment - General Requirements

- 1 Scope
- 2 References
- 3 Definitions

Part I General Requirements for the Competence of Calibration Laboratories

- 4 Organization and Quality
- 5 Quality system, audit and review

- 6 Personnel
- 7 Accommodation and environment
- 8 Equipment and reference materials
- 9 Measurement Traceability and calibration
- 10 Calibration Methods
- 11 Handling of calibration items
- 12 Records
- 13 Certificates and reports
- 14 Sub-contraction of calibration
- 15 Outside support services and supplies
- 16 Complaints

Part II Quality Assurance Requirements for Measuring and Test Equipment (M&TE)

- 17 General requirements
- 18 Detailed requirements

Examples

Here we will go over three different Reports of Calibration. The first is for an instrument which has accuracy specifications and is calibrated using a TAR. The second and third are for instruments which do not have actual specifications and were calibrated with stated uncertainties. There is a difference between the last two which requires that the difference be included on the report. Finally, we will show a typical interpolation table to demonstrate table requirements.

Example 1: Hart Scientific Model 1575 “Super-Thermometer” Report of Calibration.

Example 2: Typical SPRT Report of Calibration

Example 3: Typical Reference Thermistor Report of Calibration

Example 4: Interpolation Table.