

Thermometry – Reliability and Technology

Dean Ripple
Process Sensing Group
NIST, Gaithersburg

IFTPS Annual Meeting, March 2, 2010

- Quick overview & a few concepts
- Different types of thermometers
- Recommendations

Material in this talk compiled from:

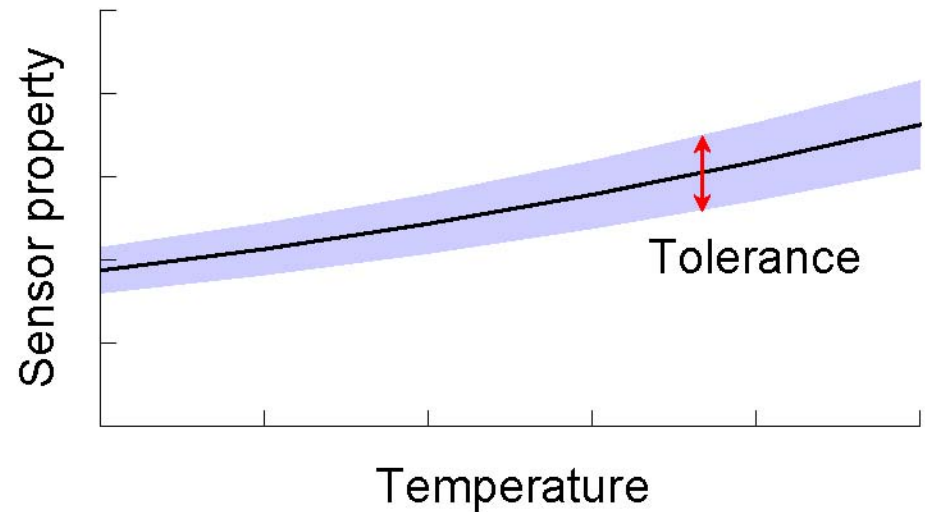
1. D. Ripple, “Thermometry Issues in Destruction Kinetics Measurement,” NISTIR 7602 (2009)
2. D. Ripple, “Cold Chain Storage of Vaccines: A Brief Introduction to Thermometry,” NISTIR 7522 (2008).
3. G. Strouse et al., “A New NIST Automated Calibration System for Industrial-Grade Platinum Resistance Thermometers,” NISTIR 6225 (1998).

Thermometer Types

| | |
|--|-----------------|
| Standard Platinum Resistance Thermometers (SPRTs) (very accurate, but susceptible to shock) −259 °C to 962 °C | \$6000 |
| Industrial Platinum Resistance Thermometers (IPRTs) −196 °C to 850 °C | \$200 to \$2000 |
| Thermistors −50 °C to 100 °C | \$200 to \$2000 |
| Thermocouples −196 °C to 2100 °C | \$100 to \$1000 |
| Liquid-in-Glass Thermometers −150 °C to 400 °C | \$20 to \$400 |
| Digital Thermometers (PRT, thermistor, or thermocouple in disguise) −196 °C to 850 °C | |

Tolerances vs. Calibration Uncertainties

Tolerance band: manufacturer's guarantee that the instrument response will conform to a standard response function to within an error equal to the tolerance.



Calibrated thermometer: may or may not have a response close to the nominal response function for that thermometer type.

Response of individual unit is reported. Expanded uncertainties (95 % confidence limit, or 'coverage factor $k = 2$ ') typically reported for calibration measurement—not including drift & user readout

Individually calibrated thermometers are not interchangeable, unless the readouts or software are adjusted.

Calibration & Traceability of Measurements

A calibration will have traceability to NIST standards if the following conditions are met:

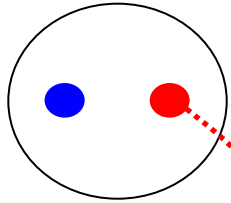
- An unbroken chain of measurements back to NIST standards must be maintained.
- Each step of the chain must have known and documented uncertainties.
- There must be a system to ensure that the thermometers and other equipment used remain accurate between calibrations.

Common misunderstandings:

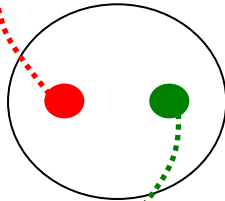
- Traceability only applies to measurements, not devices
- Traceability does not imply a particular level of uncertainty
- There is no limit to how many transfers may take place between the highest-level standards and the final thermometer, as long as the uncertainty is properly calculated.

•See: <http://www.nist.gov/traceability>, especially the Supplemental Materials link

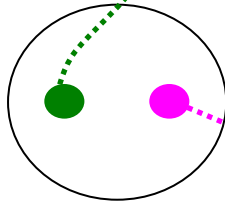
NIST calibrates a platinum resistance thermometer (PRT, shown by the red dot) against NIST standards (blue dot)



Thermometer shipped



Manufacturer calibrates another PRT (green dot) against the NIST-calibrated PRT



A digital thermometer (pink dot) calibrated using second PRT as reference

Digital thermometer shipped

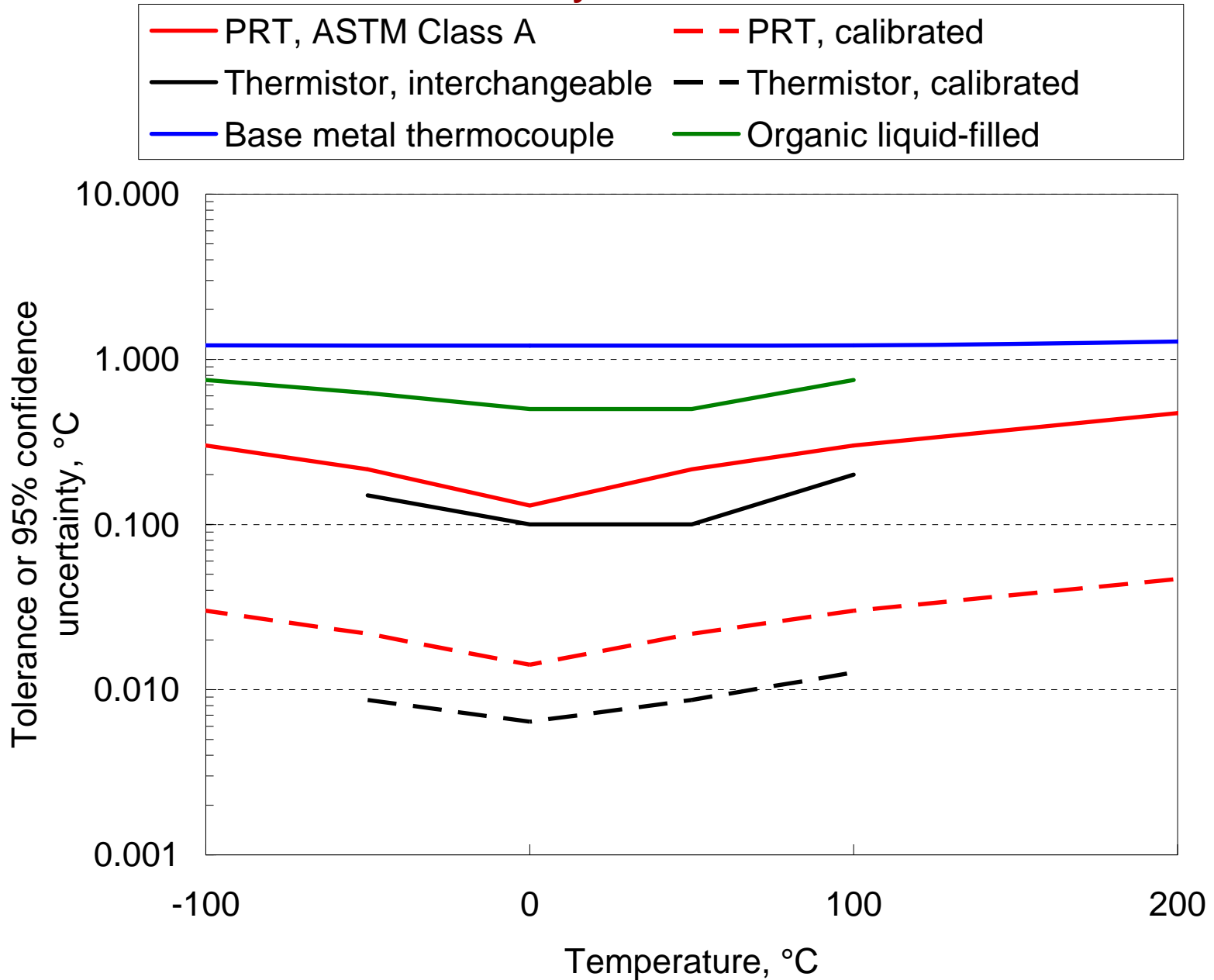


User measures the ice point (pink dots) every 2 months

Thermometer back for recalibration



Approximate Measurement Uncertainties of Complete Systems



Types of Liquid-in-Glass (LiG) Thermometers

Types of LiG thermometers

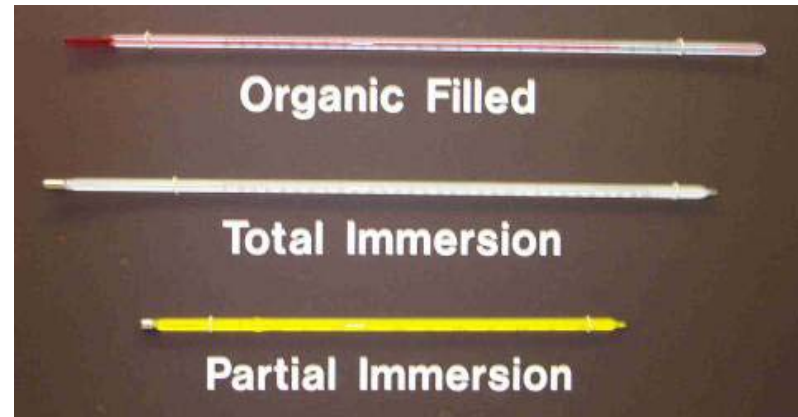
- Total Immersion: immerse thermometer nearly up to top of capillary
- Partial Immersion: immerse a designated, fixed depth (e.g., 76 mm)

Liquids used

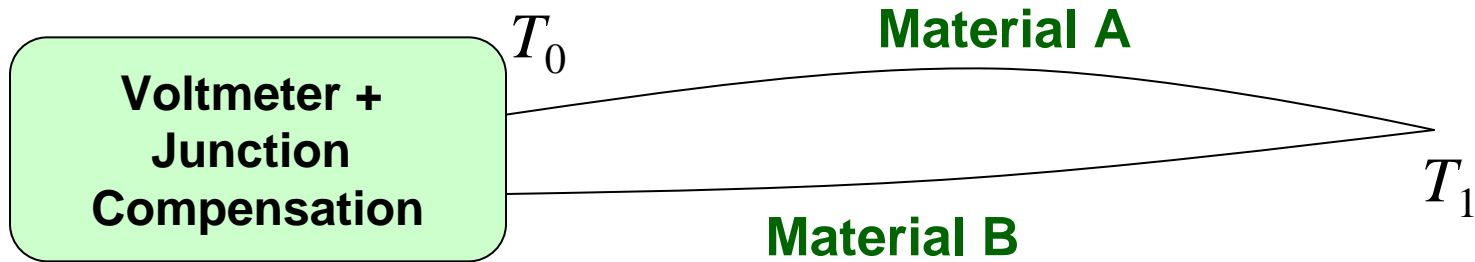
- Mercury (Hg) (sales regulated)
- Organic
- Proprietary (non-toxic)

Organic-liquid thermometers:

- Greater sensitivity to stem temperature: a fundamental limitation for partial immersion thermometers—total immersion preferred
- Best results for use at steady operating temperatures, within range $-100\text{ }^{\circ}\text{C}$ to $+100\text{ }^{\circ}\text{C}$; attainable expanded uncertainties of approximately $0.5\text{ }^{\circ}\text{C}$ to $1\text{ }^{\circ}\text{C}$ for properly used total immersion.
- Beware of separation of liquid column
- **Buyer beware: performance varies greatly above $100\text{ }^{\circ}\text{C}$**



Thermocouples



Standardized combinations of Material A vs. Material B (e.g, Type K).

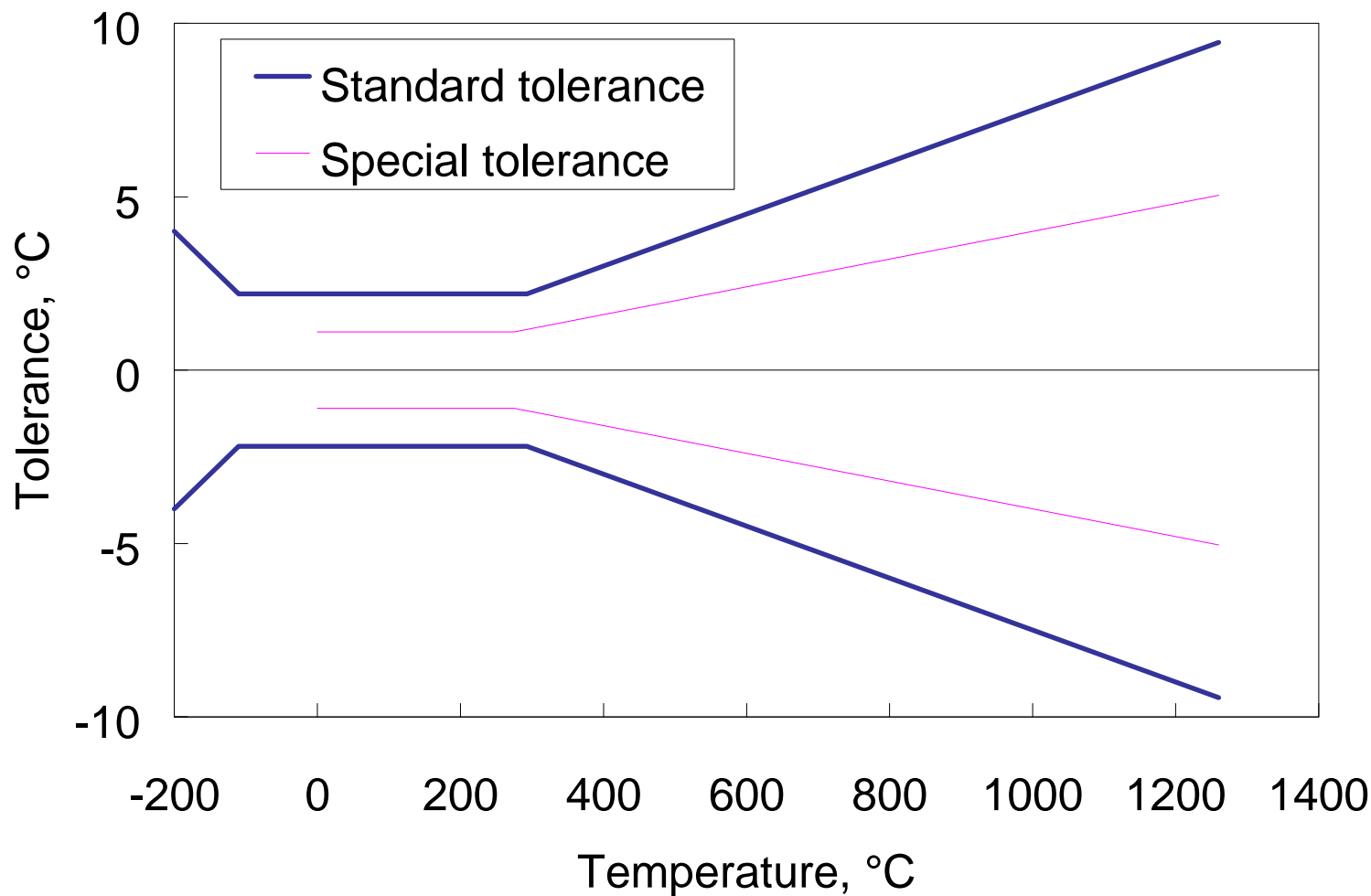
Voltage created by two lengths A & B. The junction itself does not produce a voltage.

When combined with typical readout uncertainty, total uncertainty is fairly large, unless care is taken with calibration.

Good for low-accuracy measurements over a broad temperature range.

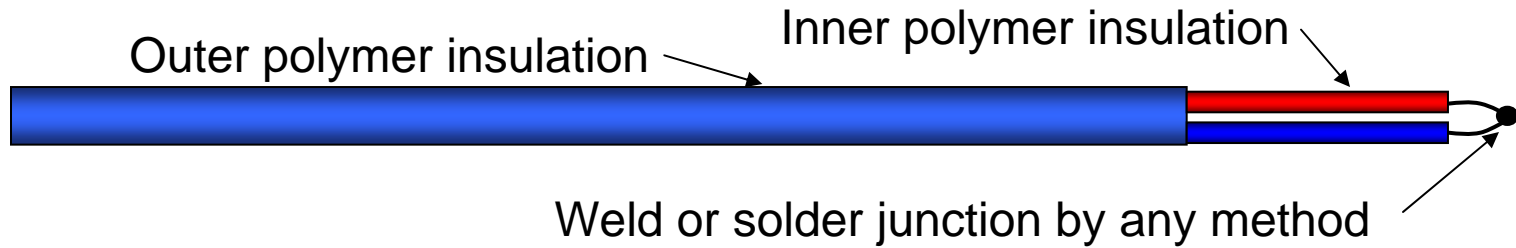


ASTM E230 Tolerances for Type K and Type N Thermocouples



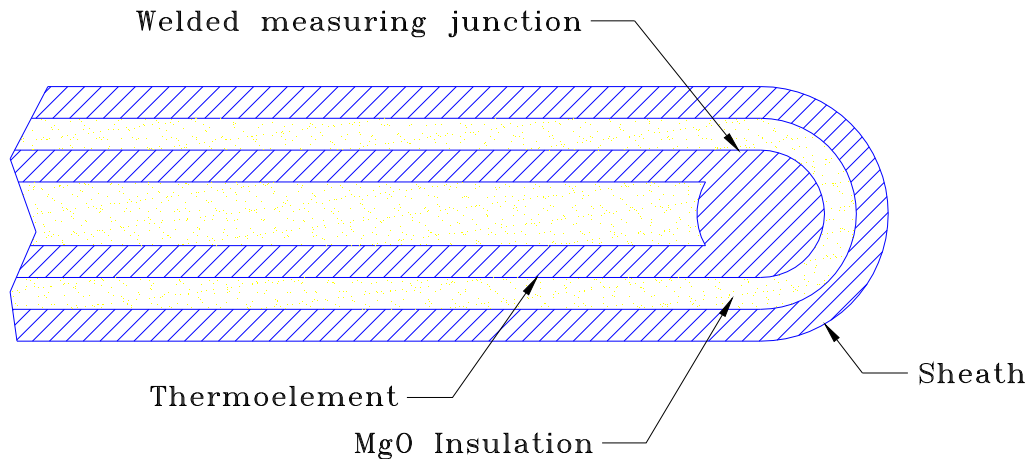
Note that even special tolerances does not meet goal of 0.5 °C (0.9 °F) uncertainty goal for retort applications: need calibration of wire !

Soft-Insulated Thermocouples



- Fluorocarbon insulations suitable to 200 °C (392 °F)
- Note that method for forming junction does not matter!

Mineral-Insulated, Metal-Sheathed (MIMS) Thermocouples



“ungrounded”
junction shown

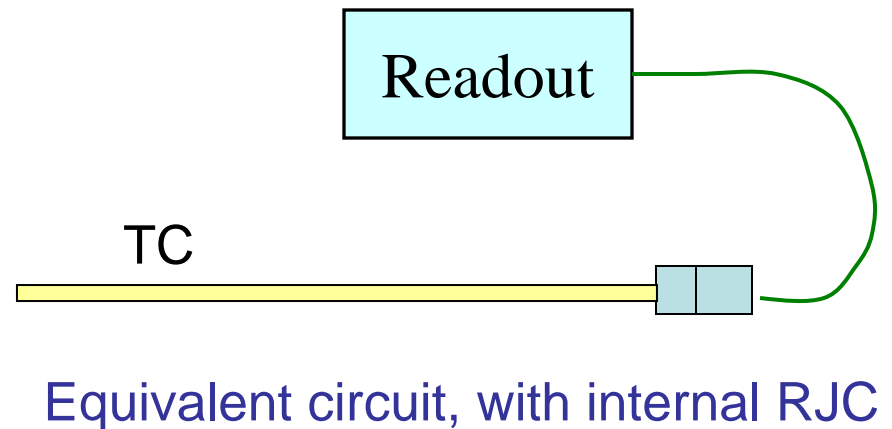
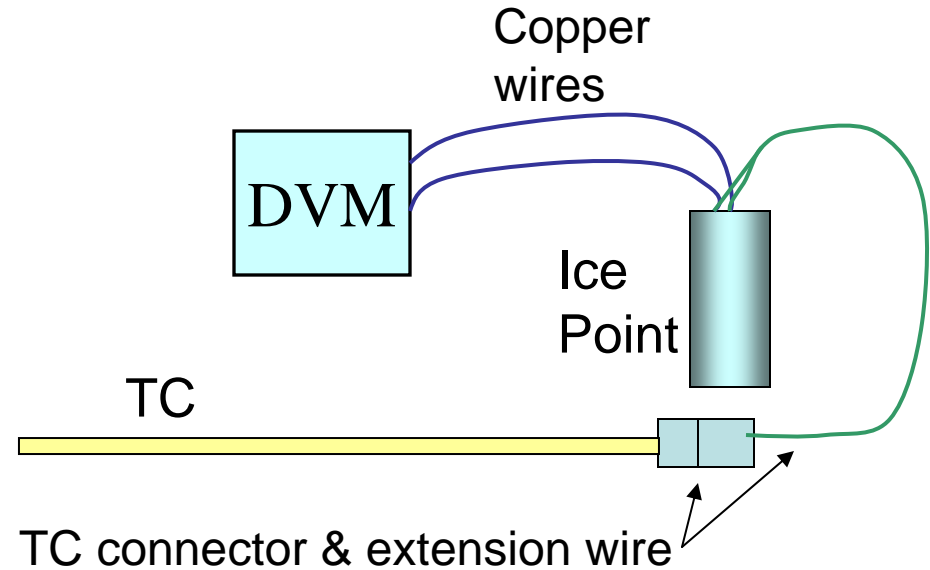
- MIMS thermocouples are available in small diameters (0.25 mm)
- Sheath protects thermoelements & simplifies sterilization

Emf Measurements

Dedicated Readout: equivalent to voltmeter + reference junction compensation + software.

Reference Junction Compensation (RJC)

- Tables assume 0 °C (32 °F) ref. junction temperature
- RJC mimics “missing” section of thermocouple when reference junction temperature is not at 0 °C
- Emf addition by hardware or software
- **RJC errors can easily reach 0.5 °C**



Thermocouple Recommendations

Type & calibration

- Type K is a good match to retort applications. Typical drift of 0.3 °C over one year at 200 °C, for fixed installation
- Do a lot calibration of sensors (first & last of a small lot made from the same batch.)
- For each temperature environment to be measured, a new thermocouple should be made, and it should always be used at the same immersion.
- Check reference junction compensation with an ice bath at retort installation

Care and Feeding

- Protect from mechanical strain and kinks
- Protect from contamination using thermowell or sheath, or use mineral-insulated-metal-sheathed thermocouples.
- Recalibrate in situ, or recalibrate with variable immersion depth

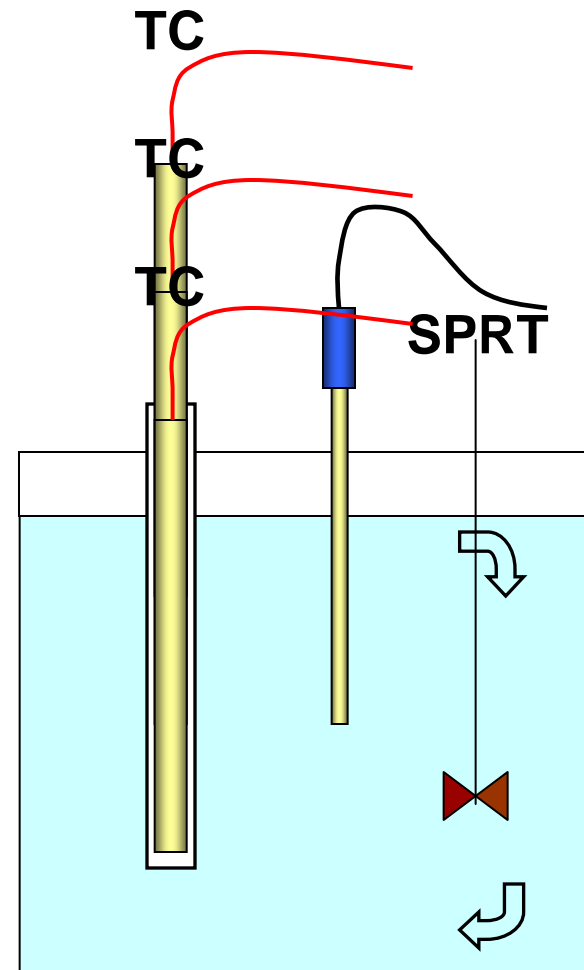
Measurement Assurance of Used Thermocouples

Used thermocouples can give output voltage that depends on temperature profile along length—Recalibration of used thermocouples gives ambiguous results.

Recalibrate thermocouples in separate bath/furnace, but vary immersion depth to bracket immersion depth in use.

If TC agrees with original cal. at all depths, OK for service

If TC is out of tolerance, install a new TC



Recommended by ASTM Manual on the Use of Thermocouples (MNL-12)

Advantages of Thermocouples

- Cheap
- Wide temperature range
- Small (down to 0.25 mm diameter)
- Easy to integrate into automated data systems

Disadvantages of Thermocouples

- Small signals, limited temperature resolution, typically 0.1 °C (0.2 °F)
- Thermocouple wires must extend from the measurement point to the readout. Signal generated wherever wires pass through a thermal gradient.
- At higher temperatures, thermocouples may undergo chemical and physical changes, leading to loss of calibration.
- Recalibration of certain types of thermocouples or in certain applications is very difficult.

Platinum Resistance Thermometers (PRTs)

Resistance element

- Wire wound (most accurate, or for wide temperature range)
- Thick or thin film (rugged)

Resistance increases as a function of temperature

Nominal temperature range of use:

- $-200\text{ }^{\circ}\text{C}$ to $850\text{ }^{\circ}\text{C}$

Nominal resistance at $0\text{ }^{\circ}\text{C}$

- $100\ \Omega$ for wire wound
- often higher for film

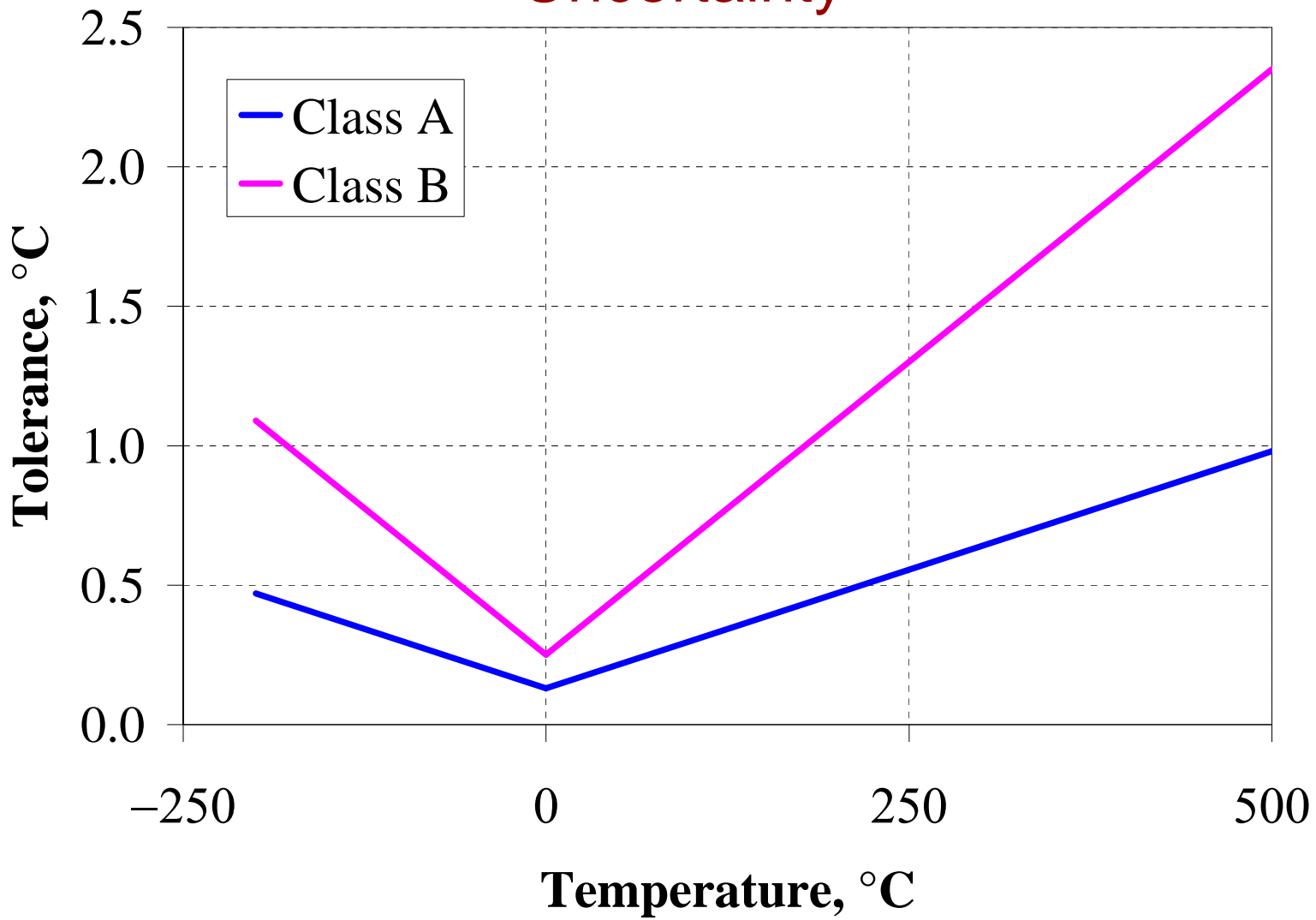
PRTs are most common “Resistance Temperature Detector” (RTD)



Stainless-steel sheath containing sensor

Sensor or element (wire-wound shown)

ASTM E1137 “Off the Shelf” Tolerance and Uncertainty



Calibrate individual units, or buy special manufacturer's tolerances for lower uncertainty

PRT Degradation & Drift

1. Intrinsic drift from thermal exposure. Little public data!

Moiseeva et al., 2002, heat-meter sensors, 365 cycles to 170 °C:

Thin-film PRTs stability: ≈ 0.2 °C worst case; 30% < 0.03 °C

Wire-wound PRTs stability: ≈ 0.2 °C worst case; 60% < 0.03 °C

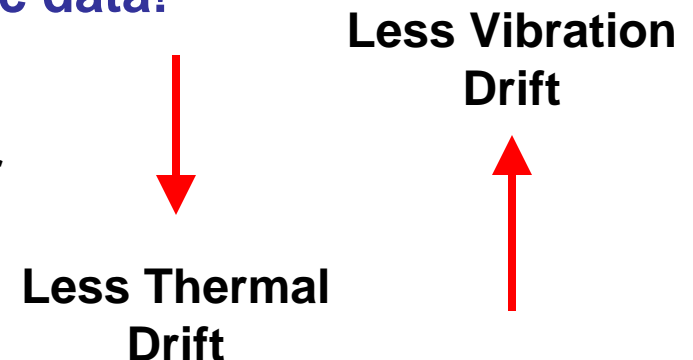
Hashemian & Peterson, 1992: ≈ 0.2 °C stability at 300 °C + 35 % failure or high drift(!) over 18 months of thermal cycling + humidity exposure

2. Drift of PRT with vibration. Little public data!

Thin-film PRTs

Wire-wound “fully supported” with powder

Wire-wound “partially supported”



IPRT Wiring Configurations

2-Wire: $R_{\text{meas}} = V/i = i(R_L + R_L + R_S)/i = 2R_L + R_S$

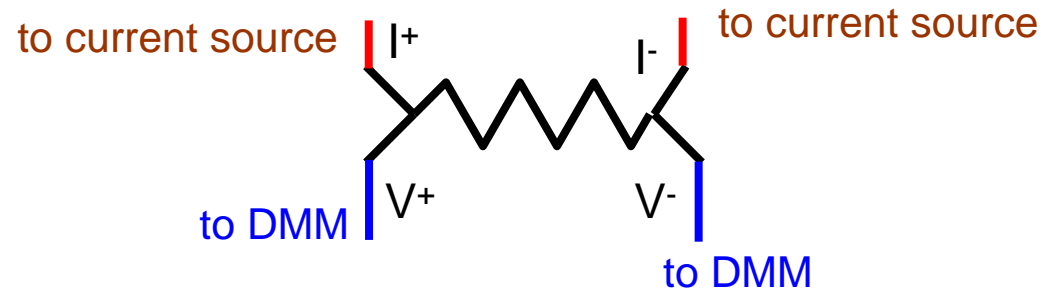
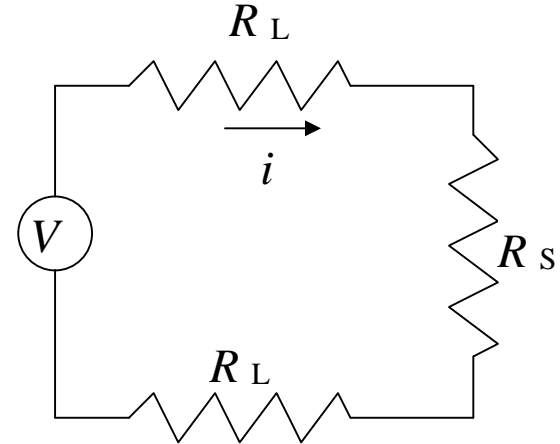
possible error of $(2 R_L/R_S)(1/4 \times 10^{-3})^\circ\text{C}$

calibration will change if R_L changes

3-Wire: partial compensation of R_L

4-Wire: No current passes through voltage-sensing leads. Consequently, there is no extra voltage drop along these leads, and measured voltage is R_S .

- compensates for changes in R_L
- preferred method



2-wire for non-demanding applications ($\pm 5^\circ\text{C}$, $\pm 9^\circ\text{F}$)

3-wire for $\pm 1^\circ\text{C}$, $\pm 2^\circ\text{F}$ measurements, or $\pm 5^\circ\text{C}$, $\pm 9^\circ\text{F}$ over long cables

4-wire for all high-accuracy measurements

Drift Mechanisms in PRTs

- **Water ingress into sensor through moisture seal**
- **Moisture causing electrical short between lead wires in head, just outside of sheath seal. (Beware of effects of steam cleaning.)**
- **Mechanical shock**
- **Strain due to thermal cycling**
- **Chemical contamination**

User should minimize shock and exposure to moisture

Special care/validation/testing needed if thermometer head is exposed to steam

Measurement Assurance for PRTs or Thermistors

- **Single-point verification of a PRT has value as a system check, but is generally not sufficient to identify all problems**
- **Two-point validation (ice point and maximum usage temperature) may be used to determine if a probe is still within tolerance.**
- **If probe is out of tolerance, replace or re-calibrate probe with a full set of points (3 points evenly spaced above 0 °C suffices for retort applications).**
- **Verification points can be conducted in the lab or on-site.**
- **PRT Performance widely variable, depending on**
 - Particular unit
 - Thermal history
 - Mechanical shock

Which Industrial PRT (IPRT) Should I Use?

Probes vs. Bare Element: metal-sheathed probes recommended. Note that moisture seal at top of probe is NOT resistant to autoclaves/steam sterilization.

Film IPRTs: good time response, small size, shock resistant; not as good as wire-wound over large (>200 °C) temperature spans. Drift after 365 cycles to 160 °C ≈ 0.2 °C.

Wire-wound IPRTs with constrained coils: low accuracy, but shock resistant. Drift after 365 cycles to 160 °C ≈ 0.1 °C.

Wire-wound IPRTs with slightly constrained coils: best accuracy (approaching ± 0.01 °C over 400 °C span), sensitive to shock. Drift better than constrained coils, but highly variable among units.

Resistor configuration

- 3-wire configuration in an industrial environment may have difficulty achieving 0.5 °C uncertainty. 4-wire preferred
- 4-wire for all high-accuracy measurements

Considerations in Selecting IPRTs

Advantages

- Wide temperature range
- R vs. T is well characterized
- Available in different shapes and sized to meet most application requirements
- Can be used with a digital temperature read-out device

Disadvantages

- Some sensitivity to vibration
- 2- and 3- wire devices have imperfect lead-wire compensation
- Non-hermetically sealed IPRTs will deteriorate in environments with excessive moisture
- Performance of IPRTs can be quite variable from unit to unit

Thermistors (Thermal Resistor)

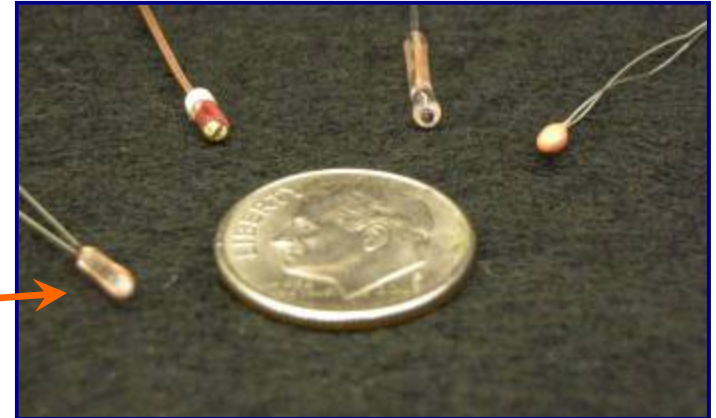
Sensor formed from special ceramic semiconductors

Temperature Range: $-50\text{ }^{\circ}\text{C}$ to $100\text{ }^{\circ}\text{C}$

Standard Sensor Forms:

bead $300\ \Omega$ to $100\ \text{M}\Omega$

probe bead in glass rod



NTC: Negative Temperature Coefficient - The vast majority of commercial thermistors used as thermometers are in the NTC category.

Commonly packaged in stainless-steel sheaths

Good choice for applications near room temperature

Use only glass-coated variety: 1-year stability $<0.01\text{ }^{\circ}\text{C}$ up to $90\text{ }^{\circ}\text{C}$; much higher drift at higher temperatures

What is a Digital Thermometer?

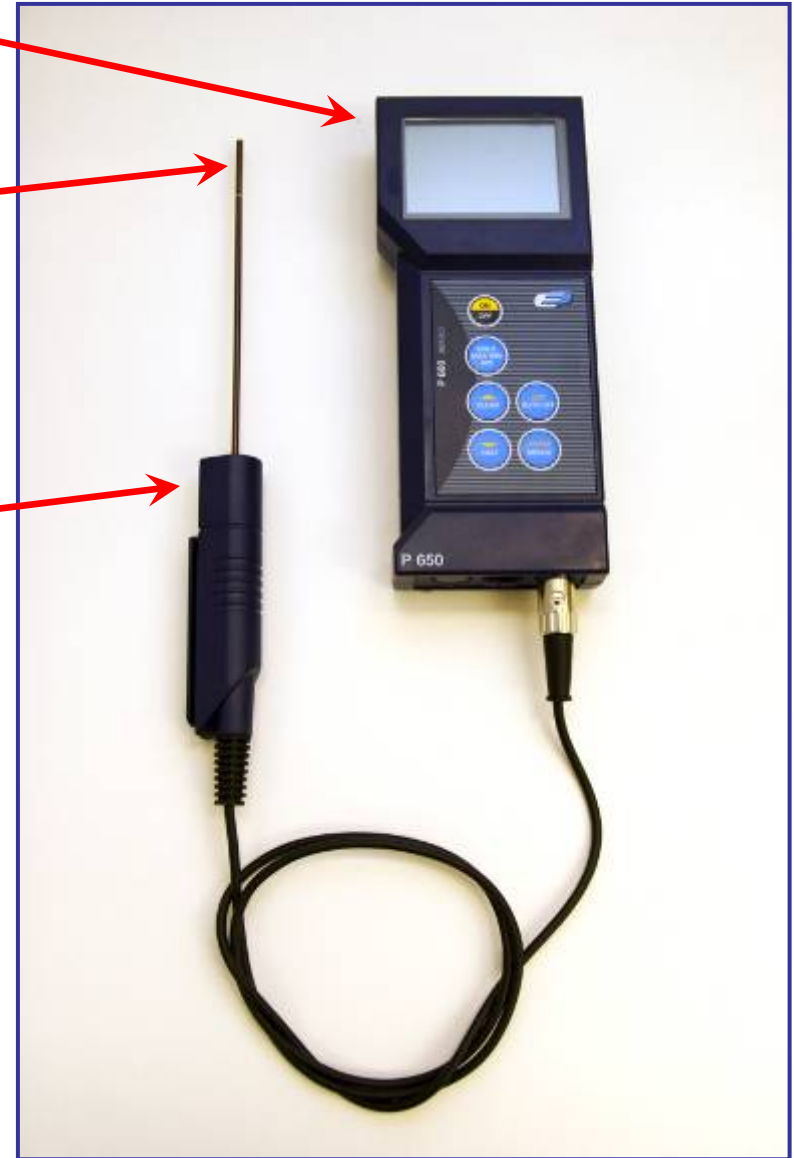
Readout

Sensor inside

Probe

An electronic measurement box that converts either resistance or emf of a thermometer to temperature

ASTM specifications for thermocouples, PRTs, thermistors pertain to sensor or probe only, not complete thermometer



Digital Thermometers

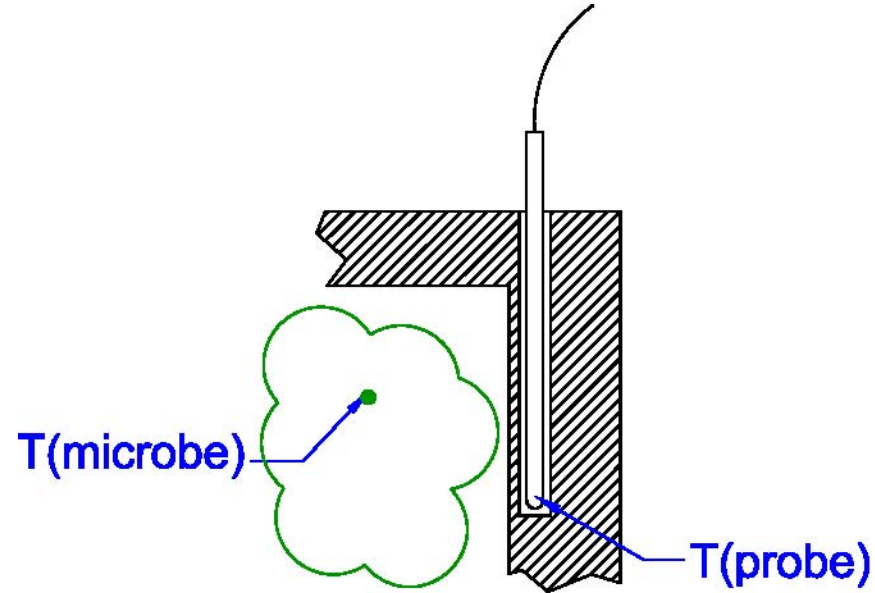
- Device displays temperature directly by using the calibration coefficients of the thermometer
- Uncertainty: 0.001 °C to 1 °C; Resolution: 0.0001 °C to 1 °C
- Careful use requires careful reading—look at manufacturer's specifications & know the probe type and limitations
- “Digital Thermometer” only means that you can watch the numbers on a display—no guarantee of better uncertainty
- Calibration can be as a system or as readout + separate probe

System calibration is cheaper and simpler

Probe and readout calibration allows easier repair if one element fails, allows identification of source of drift

Rules of Thumb for Installation Effects

Probe location: temperature differences between probe location and microbes of interest as a function of time (e.g., thermal modeling, study of wall vs. product temperature)



Avoid large air gaps around probe: still air is a very poor conductor (500x worse than stainless steel)—can lead to large errors for measurement of temperature transients. Keep gaps small, or use grease.

Design measurement geometry so at least 15 cm (6") of probe is at temperature of interest. (Use fine diameter probes if this is not possible.)

Ensuring Good Measurements

1. **Avoid shock to the sensor and readout.** With metal-sheathed thermometers, damage to the sensor will not be visually apparent.
2. **For thermocouples, avoid kinks in the thermocouple wires,** especially in regions where the temperature is changing from one point to another. For thermocouples used above 150 °C (302 °F), use a separate thermocouple for each apparatus, at a fixed depth.
3. **For resistance thermometers, calibrate and use with same lead configuration & excitation current**
4. **Do not switch probes** unless probes are interchangeable or coefficients are updated.
5. **Be absolutely certain that the readout is set to the proper thermometer type** (e.g., avoid type K thermocouple read as a type J.)
6. **Do not exceed the recommended temperature limits.**
7. **Check the performance of the instrument regularly,** following manufacturer's recommendations or past device history.

Recommendations for Retort Applications

PRTs Are the 1st Choice

- PRTs are a good general choice. Use either wire-wound with powder-supported coils or thin-film elements.
- 4-wire sensors in metal-sheathed probes
- Protect moisture seal

Thermocouples

- Type K thermocouples have a role in environments where PRTs suffer vibration or end-seal degradation.
- Verify reference junction compensation on site.
- Type K thermocouples also useful as an independent cross-check on PRT readings.

Learning More

Good general guides:

Traceable Temperatures by J. Nicholas and D. R. White (Wiley, 2001)

Handbook of Temperature Measurement (Vols 1-3), ed. Robin E. Bentley (Springer, 1998).

Additional reference for thermocouples:

ASTM Manual on the Use of Thermocouples in Temperature Measurement, MNL-12 (ASTM, West Conshohocken, PA, 1993).

Modern uncertainty analysis:

<http://physics.nist.gov/cuu/Uncertainty/index.html>

Methods to set calibration intervals, see:

“Guidelines for the determination of calibration intervals of measuring instruments,” ILAC-G24, (International Laboratory Accreditation Cooperation, 2007).