FLUKE®
Calibration

Temperature Calibration
ITS 90 & TPW

Bouraoui KHACHLOUF

Technical Sales Manager

Temperature & Humidity EMEA

FCAL: Temperature Calibration

Primary standards, thermometer readouts, probes, baths, industrial calibrators

TCAL - Temperature

Temperature Calibration Solutions

Temperature Verification System

FLUKE®
Calibration

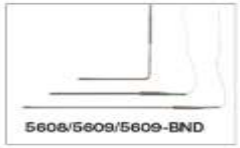
Temperature Source/ Supply

Field
- Field Dry-well
- Handheld Dry-well
- Micro-Bath
- TC Furnace

Bench
- Metrology Well


Temperature Reference

- Industrial PRT



- Thermistor probe

- Thermocouple

Temperature Readout

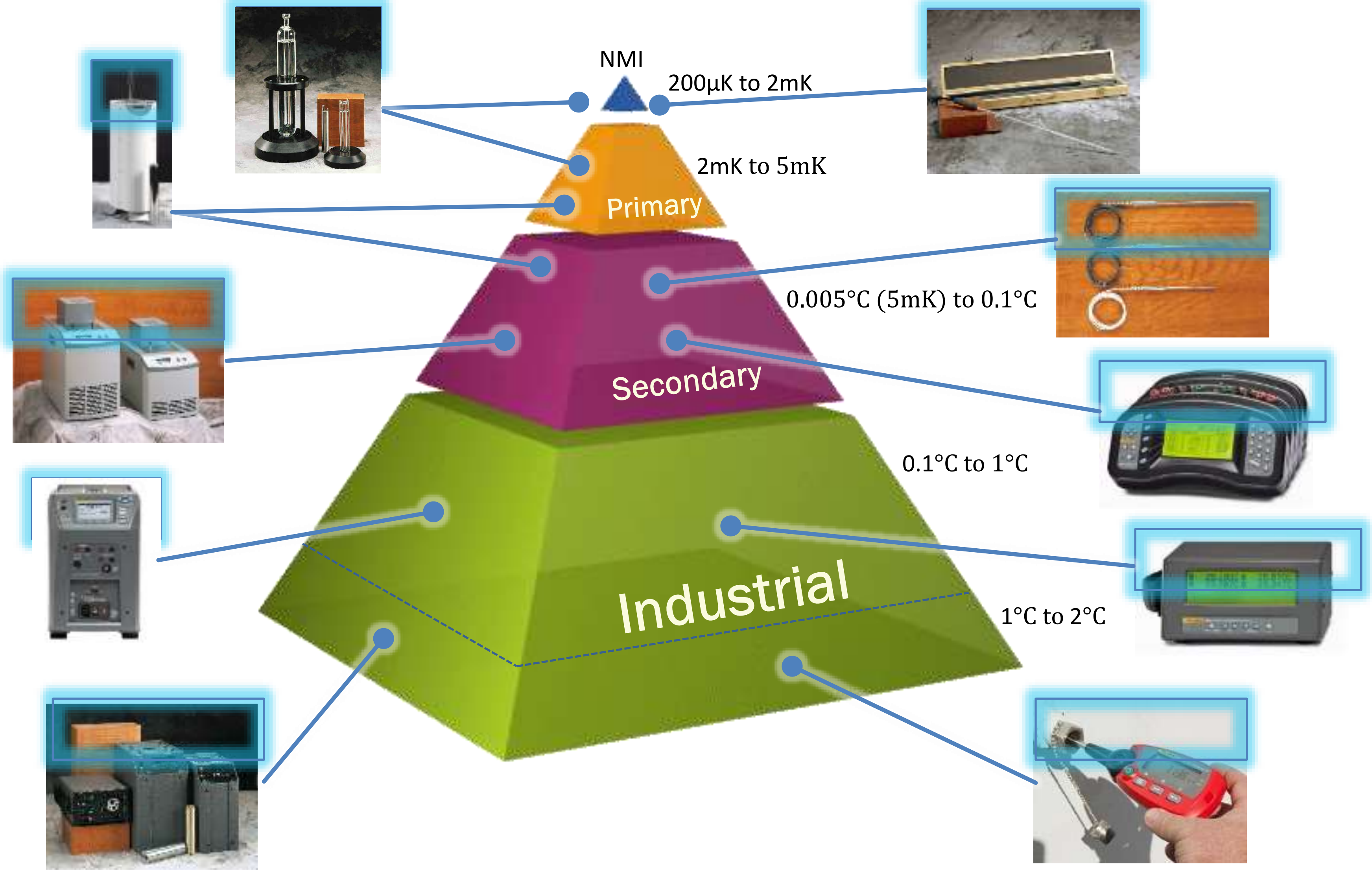
Field
- Handheld
- Stik

Bench
- 7526

- 1529

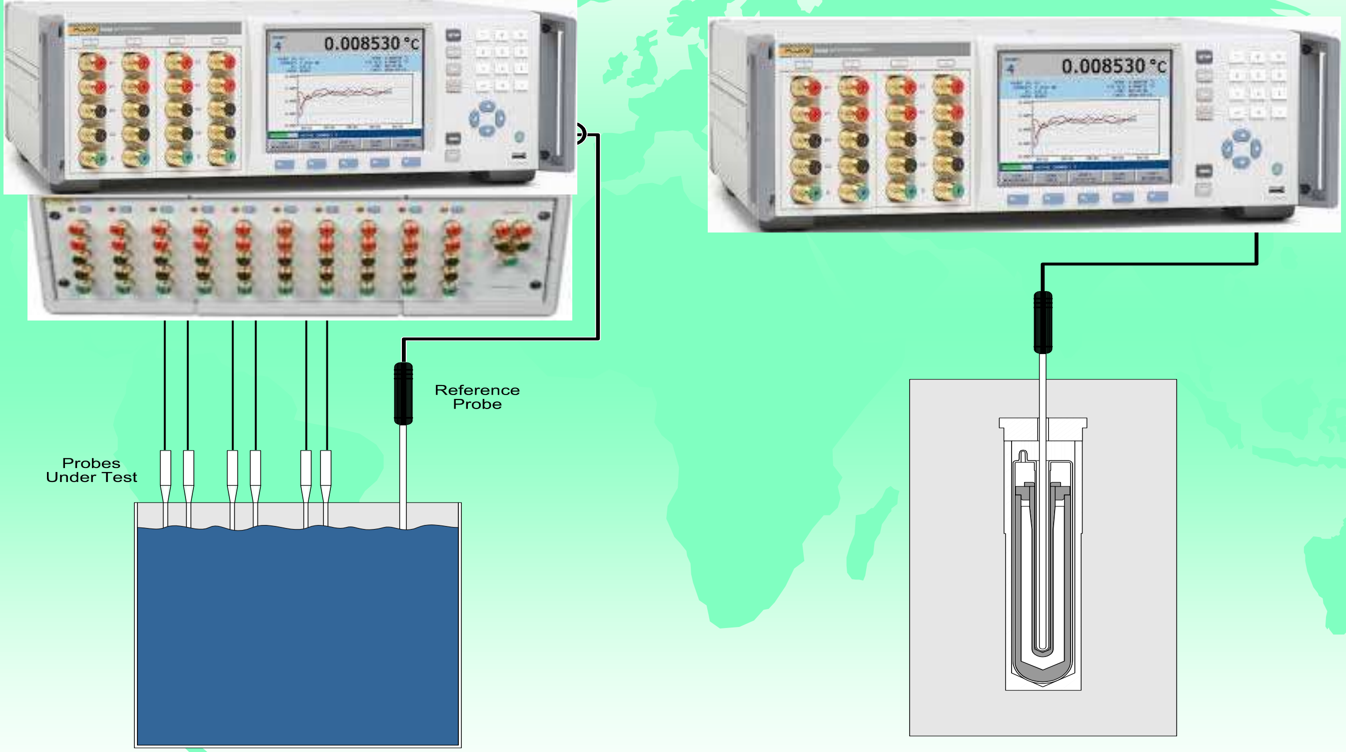
Temperature Products - Calibrators

FLUKE Calibration



Fixed Point vs. Comparison Calibrations

Two kinds of Calibration



Comparison Calibration

Fixed Point Calibration

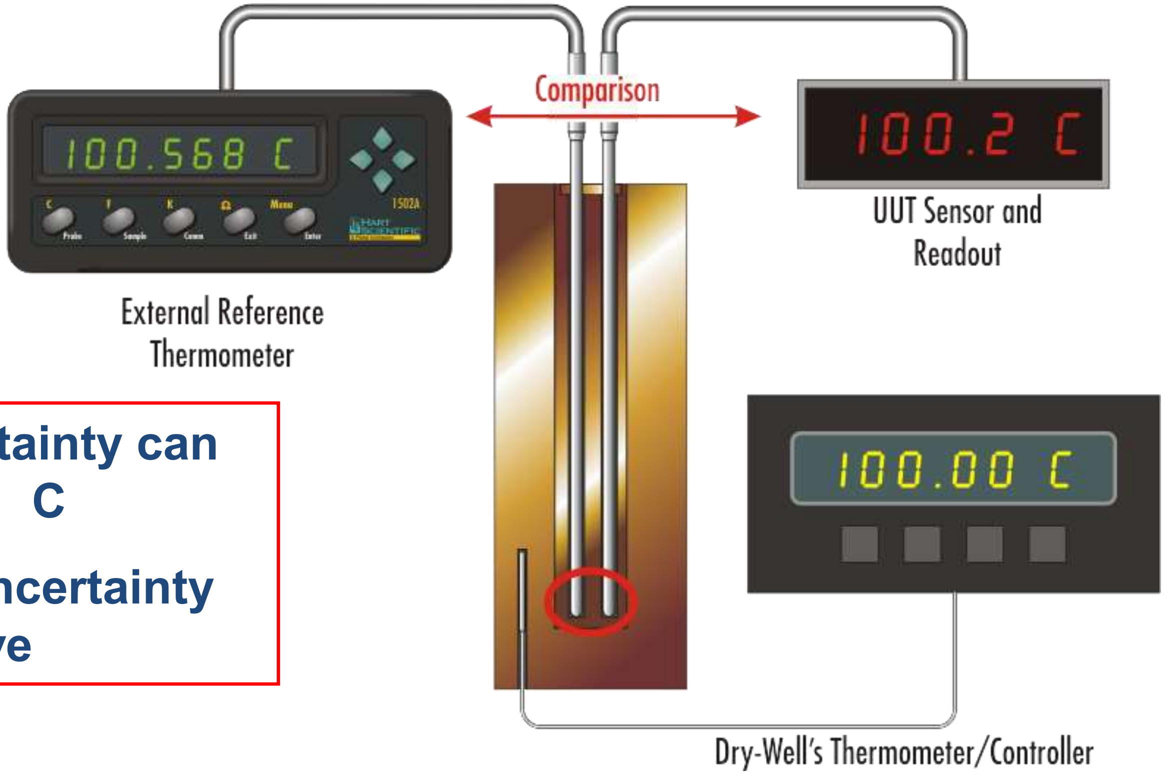
Fixed-Point vs. Comparison

- Fixed Point Method
  - Advantages
    - Follows ITS-90
    - Traceable without reference thermometer
    - Well Understood
    - Lowest Uncertainty
  - Disadvantages
    - Somewhat difficult
    - Expensive
    - Less flexible
    - Less Efficient
    - Thermometers must fit apparatus

- Comparison method
  - Advantages
    - Flexible
    - Efficient
    - Less Expensive
    - Less Difficult
  - Disadvantages
    - Requires reference thermometer
    - Data fitting not straightforward
    - Procedure can be complex
    - Larger uncertainty
    - May not follow ITS-90

Comparison Calibration with Built in Reference Thermometer

Comparison to External Reference Thermometer



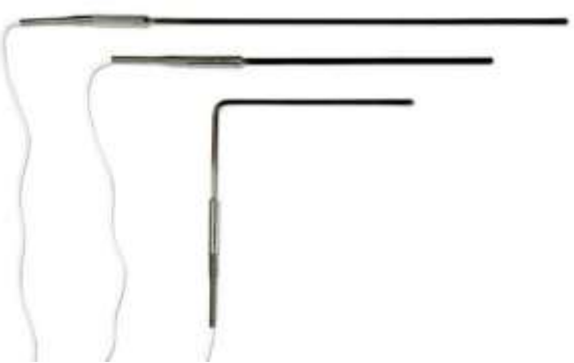
- Typical system uncertainty can be as good as ± 0.05 ° C

- Better than display uncertainty by nearly a factor of five

Equipment Overview

Industrial & Portable systems

- Industrial Calibration often takes place on the shop floor
  - Portable
  - Fast
  - Accommodate different sensor shapes



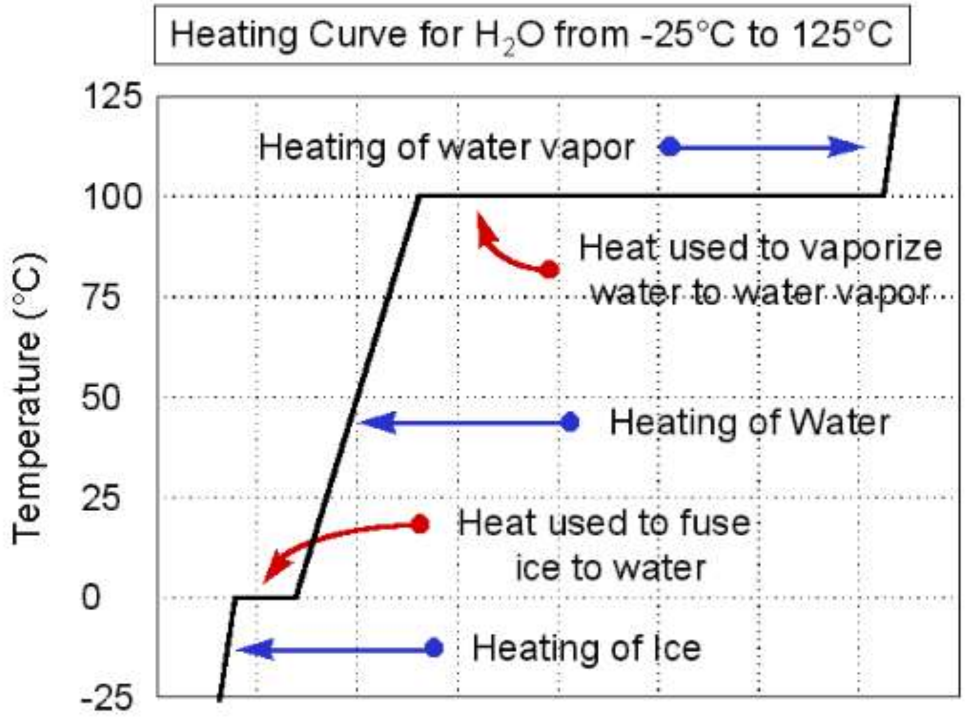
Secondary Laboratory systems

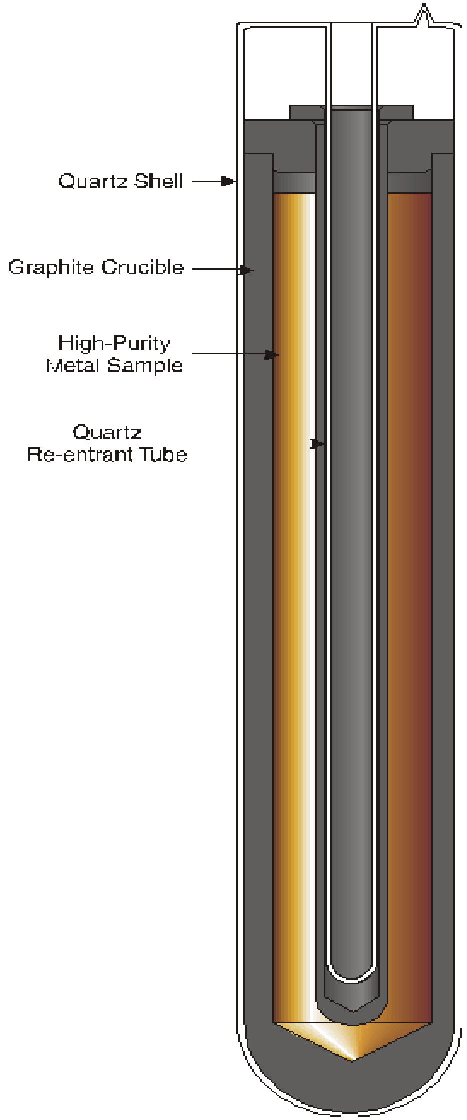
- Not as portable
- Mid-range accuracy
- Many heat sources and temperature ranges
- Large or small calibration volume

Fixed points Calibration

- Fixed points are phase change devices made from very pure metals

- The temperature of these fixed points is precisely defined and internationally agreed – ITS90





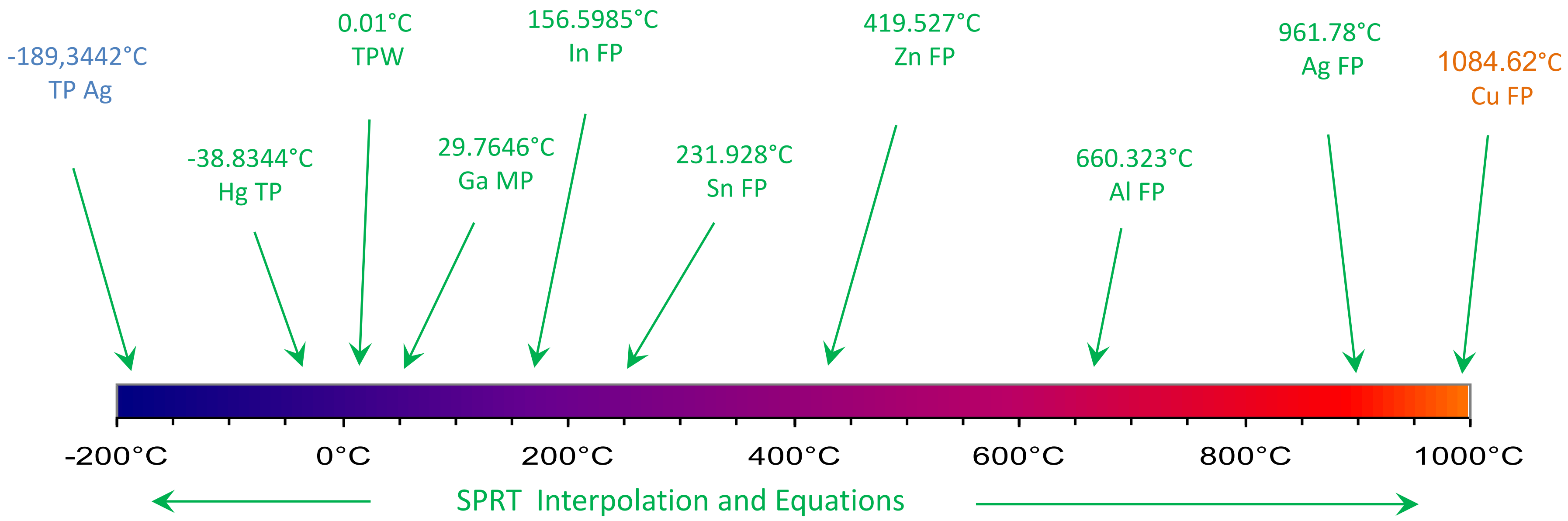
Equipment Overview

FLUKE®
Calibration

Primary Standards

- Solid World-Class Performance
- Fixed-Points, Furnaces, Readouts, Bridges, SPRTs
- “Best Uncertainty” Capability

ITS-90 : International Temperature Scale of 1990










ITS-90 Fixed Point Standards

FLUKE ® Calibration

Relied on by National Metrology Institutes, calibration labs, manufacturing and process worldwide

Model	Fixed Point	Assigned Value (°C)	Uncertainty mK, k=2
5960	TP Argon	–189.3442	0.25
5900	TP Mercury	– 38.8344	0.25
5901	TP Water	0.01	< 0.1
5943	Gallium Melt. P	29.7646	0.1
5904	Indium Fr P	156.5985	0.7
5905	Tin Fr P	231.928	0.8
5906	Zinc Fr P	419.527	1.0
5907	Aluminum Fr P	660.323	1.8
5908	Silver Fr P	961.78	4.5
5909	Copper Fr P	1084.62	12.0

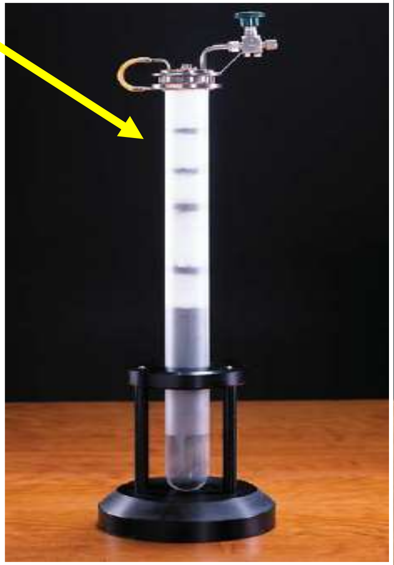
Argon triple point now offered

- 99.9999% purity
- Plateau duration > 30 hrs ,
- Multi-language display

Hart Fixed Point Cell Size Comparison

Open FP Cell

Metal-cased FP Cell



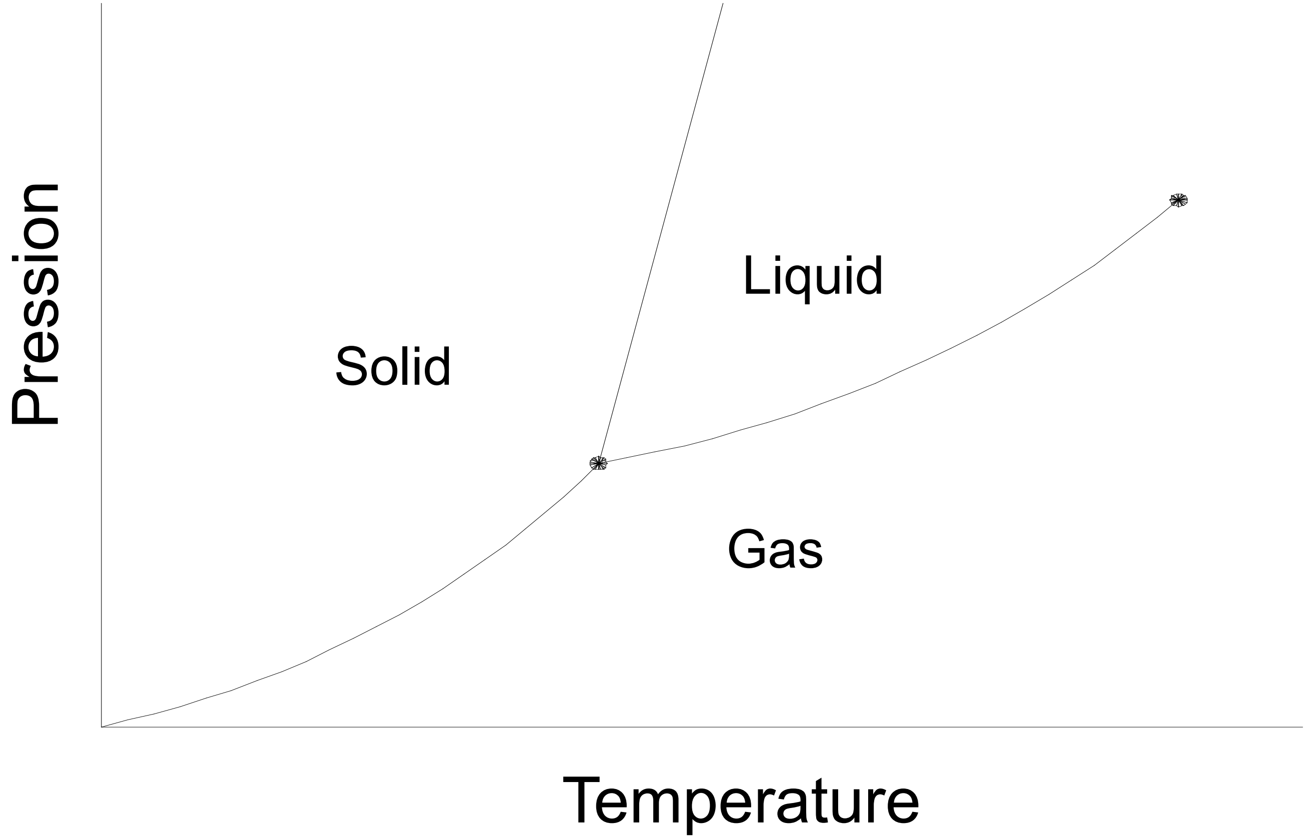
Full Size TPW

Mini TPW

Full Size Quartz

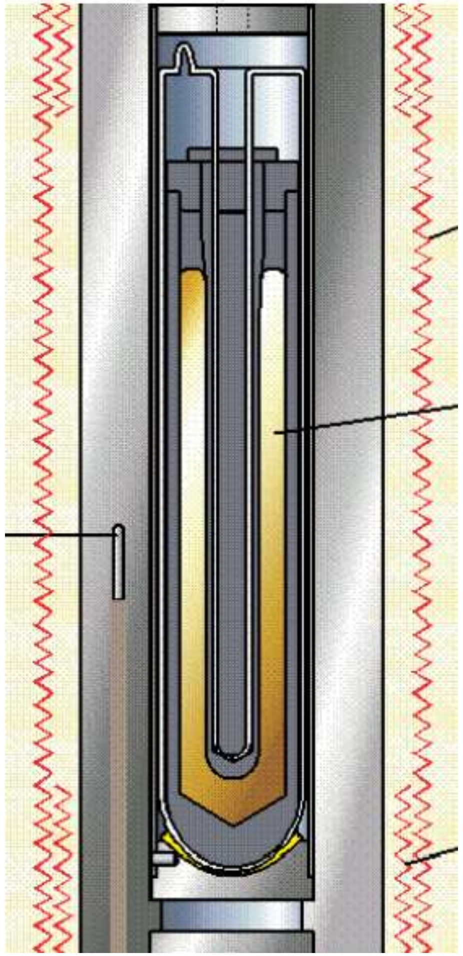
Mini Quartz

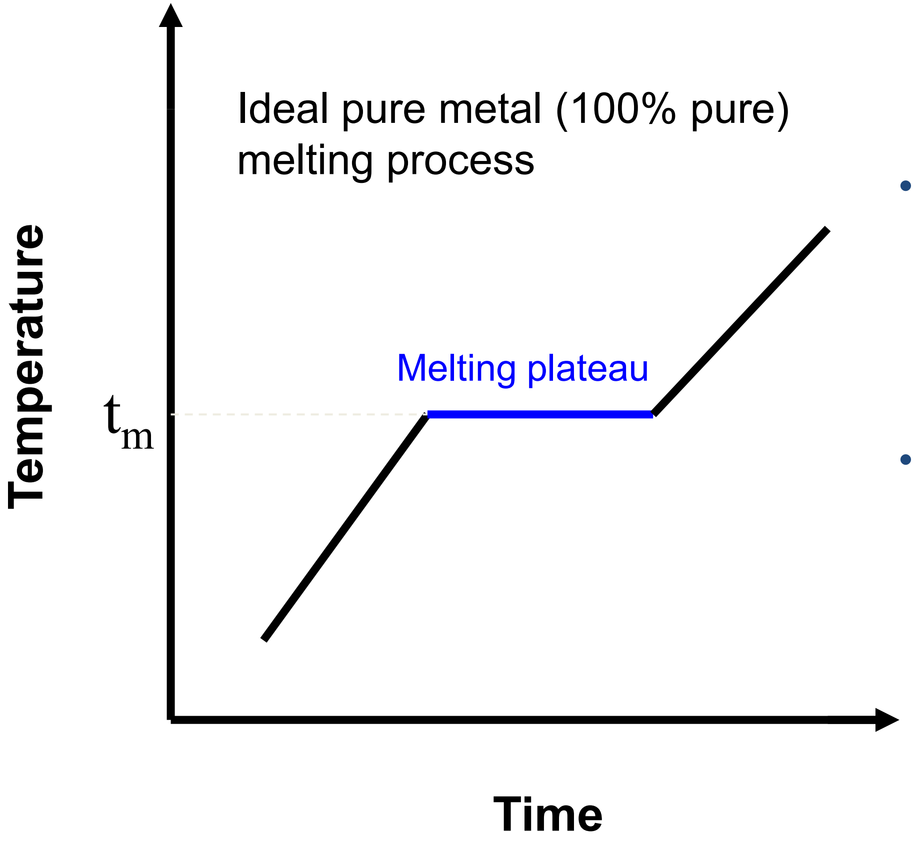
Phase Diagram for a Pure Substance



TP Argon	–189.3442
TP Mercury	– 38.8344
TP Water	0.01

Melting of an "Ideal" pure metal





- The melting of an ideal pure metal occurs at a unique temperature and involves the absorption or liberation of the latent heat of melting

- Before reaching the melting point, the temperature of the metal will rise by

$$\Delta t = \frac{Q}{n \cdot C_P}$$

- An amount of pure metal, k mol, will melt when it absorbs heat Q:

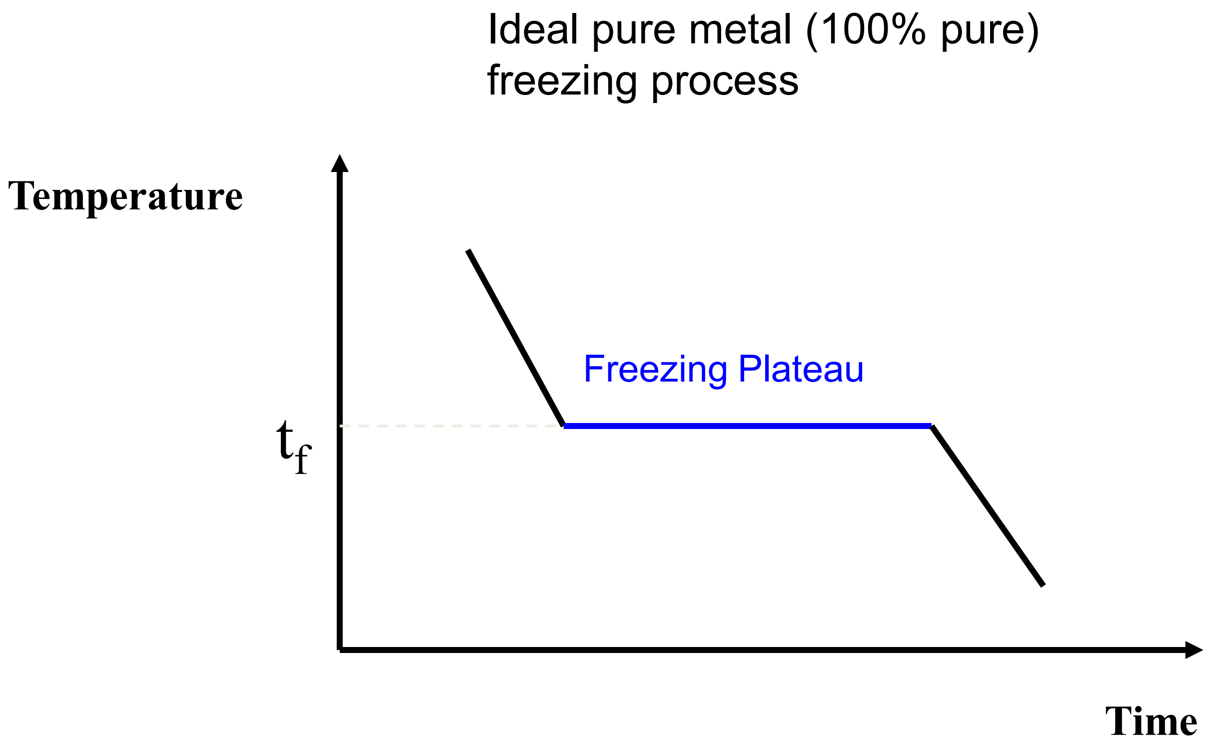
  Q = kL

  Where L is the mole latent heat of fusion

Gallium Melt. P	**29.7646**

Ideal Pure Metal Freezing Process

The freezing process is just the reverse of the melting process



Indium Fr P	156.5985
Tin Fr P	231.928
Zinc Fr P	419.527
Aluminum Fr P	660.323
Silver Fr P	961.78
Copper Fr P	1084.62

Fixed Point Cell Realization – Equipment

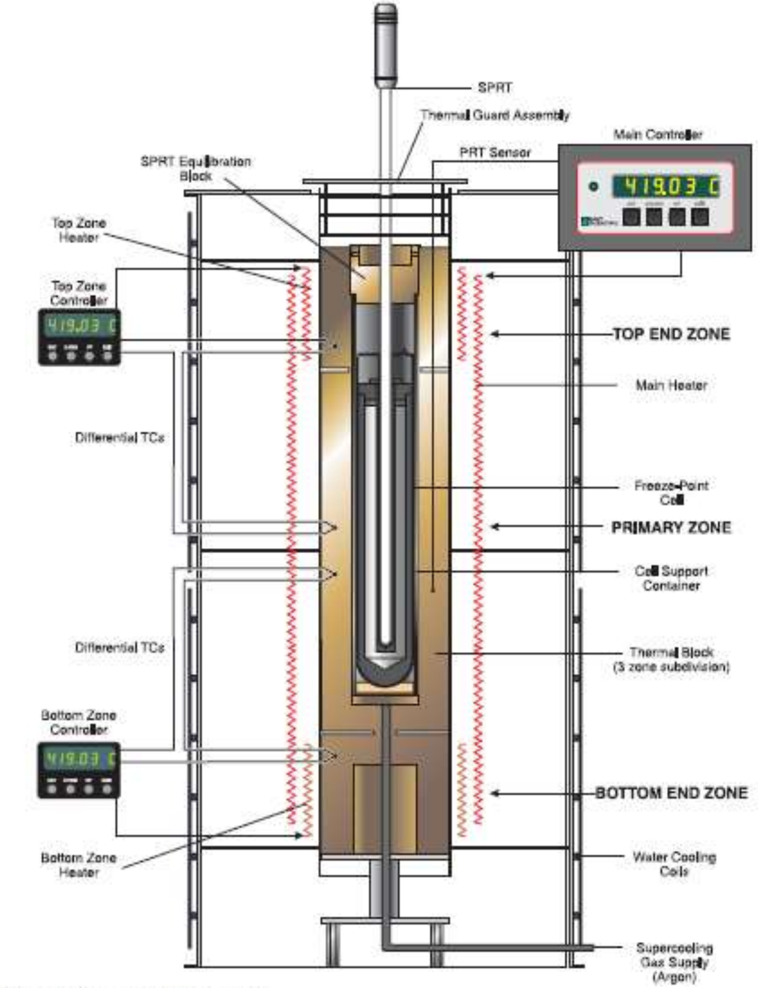


Figure 3. *9114 Heater Block Assembly*

FLUKE ®

Calibration

ITS-90 fixed-point cells

- Best cell uncertainties commercially available
- Every ITS-90 fixed point available from mercury to copper
- Plateaus last days (gallium for weeks and TPW for months)
- Manufactured and tested by Hart's primary standards scientists

Specifications

Model	Fixed Point	Style	Assigned Value (°C)	Outside Diameter	Inside Diameter	Total Outside Cell Height	Depth[2]	Cell Uncertainty (mK, k=2)	Certification (mK, k=2)[1]
5900	Mercury	Stainless Steel	-38.8344	31.8 mm	8.0 mm	417.6 mm	208 mm	0.2	0.25
5904	Indium	Traditional Quartz Glass	156.5985	48 mm	8 mm	282 mm	195 mm	0.7	0.7
5905	Tin	Traditional Quartz Glass	231.928	48 mm	8 mm	282 mm	195 mm	0.5	0.8
5906	Zinc	Traditional Quartz Glass	419.527	48 mm	8 mm	282 mm	195 mm	0.9	1.0
5907	Aluminum	Traditional Quartz Glass	660.323	48 mm	8 mm	282 mm	195 mm	1.3	1.8
5908	Silver	Traditional Quartz Glass	961.78	48 mm	8 mm	282 mm	195 mm	2.4	4.5
5909	Copper	Traditional Quartz Glass	1084.62	48 mm	8 mm	282 mm	195 mm	10.1	12.0
5924	Indium	Open Quartz Glass	156.5985	50 mm	8 mm	596 mm	195 mm	0.7	0.7
5925	Tin	Open Quartz Glass	231.928	50 mm	8 mm	596 mm	195 mm	0.5	0.8
5926	Zinc	Open Quartz Glass	419.527	50 mm	8 mm	596 mm	195 mm	0.9	1.0
5927A-L	Aluminum	Open Quartz Glass (long)	660.323	50 mm	8 mm	696 mm	195 mm	1.3	1.8
5927A-S	Aluminum	Open Quartz Glass (short)	660.323	50 mm	8 mm	596 mm	195 mm	1.3	1.8
5928	Silver	Open Quartz Glass	961.78	50 mm	8 mm	696 mm	195 mm	2.4	4.5
5929	Copper	Open Quartz Glass	1084.62	50 mm	8 mm	696 mm	195 mm	10	12.0
5943	Gallium	Stainless Steel	29.7646	38.1 mm	8.2 mm	250 mm	168 mm	0.1	0.1

[1]Certifications at lower uncertainties are available for national laboratories.
[2]Depth is measured from the bottom of the thermometer well to the top of the pure reference material.

ITS 90 Fixed Point – Open Cells-

Open cells allow users to minimize the uncertainty from pressure corrections by regulating cell pressures themselves.

"open" metal fixed-point cells include a high-quality valve for connecting to a precision pressure-handling system within your lab. Using such a system, the cell can be evacuated, charged, and purged several times with a pure inert gas, then charged again to a regulated pressure level while measurements are made with the cell.

Once assembled and tested, each Fluke ITS-90 open cell further undergoes more rigorous testing in our lab, unlike cells from some manufacturers who provide their open cells as a kit of parts, without any test data.

Because open cells allow users to measure the pressure within the cell, uncertainties due to pressure corrections may be minimized. Use of open cells is now being suggested by the CCT, and open cells can be used for demanding temperature-versus-pressure applications, as well as precision SPRT calibrations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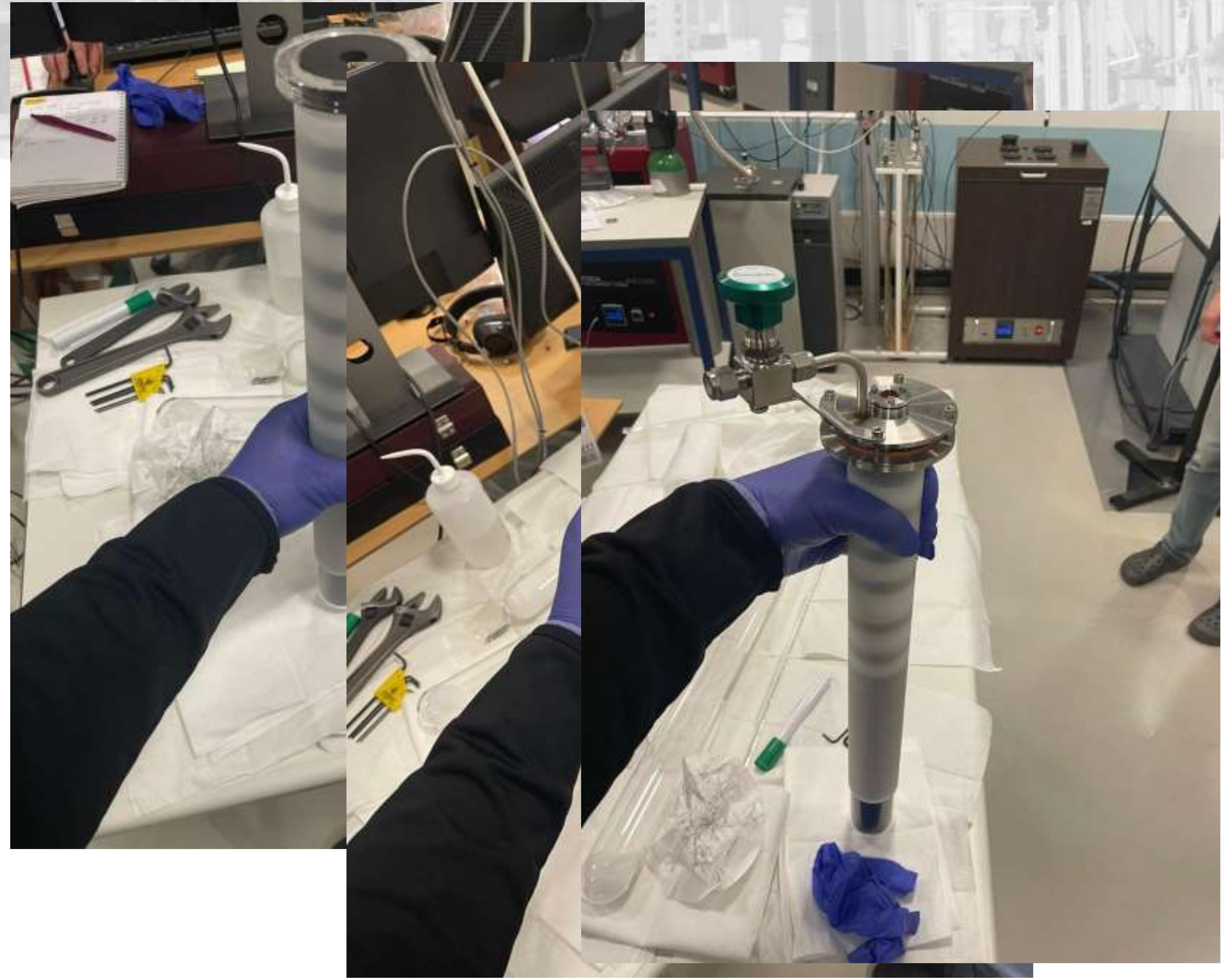
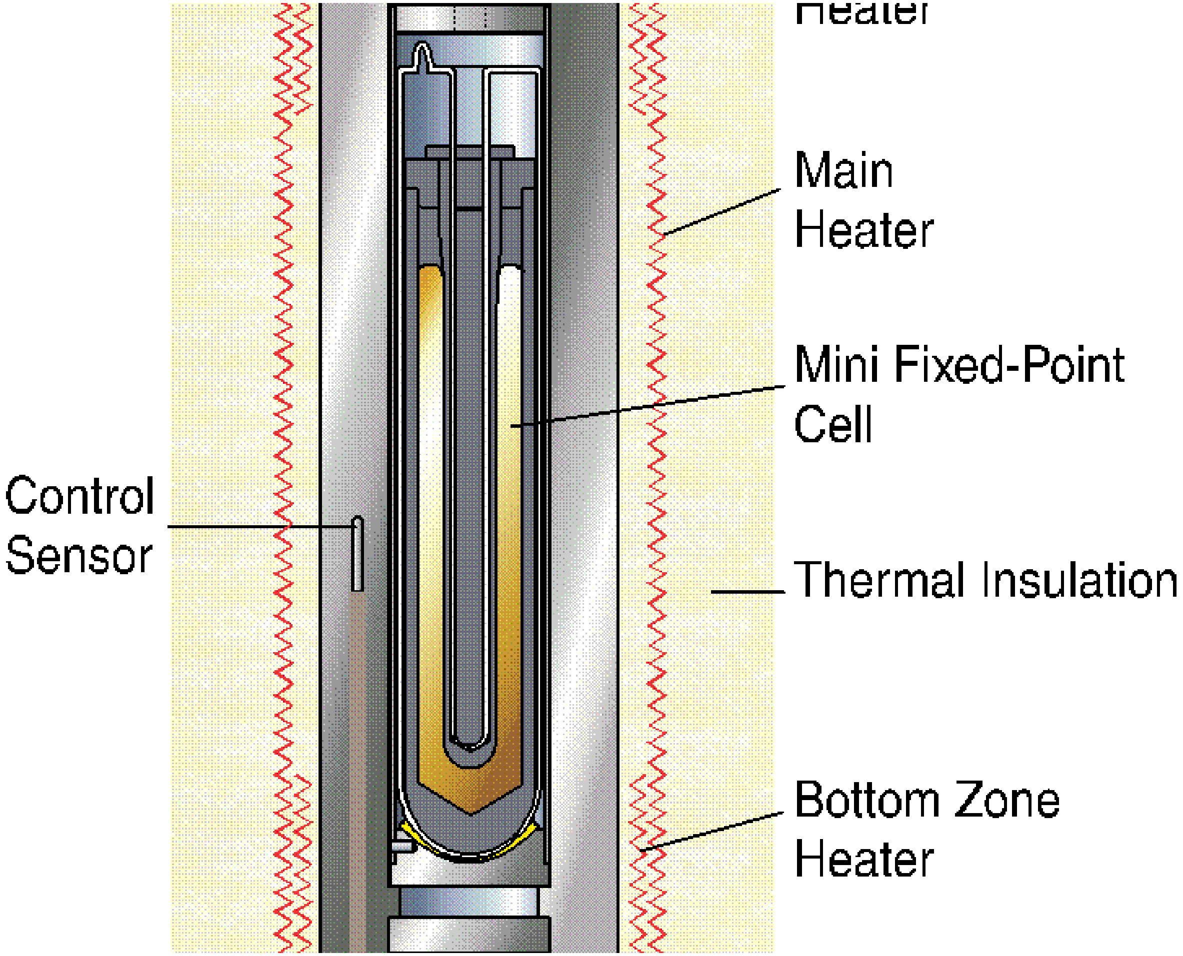


Fixed point mini-cells and fixed point cells with metal shell



Fixed Point Cells – Types

Full Sized Cells

- Pros
  - Best Uncertainty (±0.07 mK - ±2.4 mK)
  - Large Immersion 195 mm
  - Longer Plateaus 8 – 48 hrs
- Cons
  - Maintenance Apparatus has large footprint
  - Higher Cost

Mini Cells

- Pros
  - Lower cost
  - Smaller footprint
  - Easy to get full set in a small area
- Cons
  - Less Immersion 140 – 156 mm
  - Shorter Plateaus 5 – 8 hrs
  - Higher Uncertainties 0.2 mK – 4.0 mK

Mini Fixed Point Calibration System

Fixed Point Cells – Construction and Optional Certification

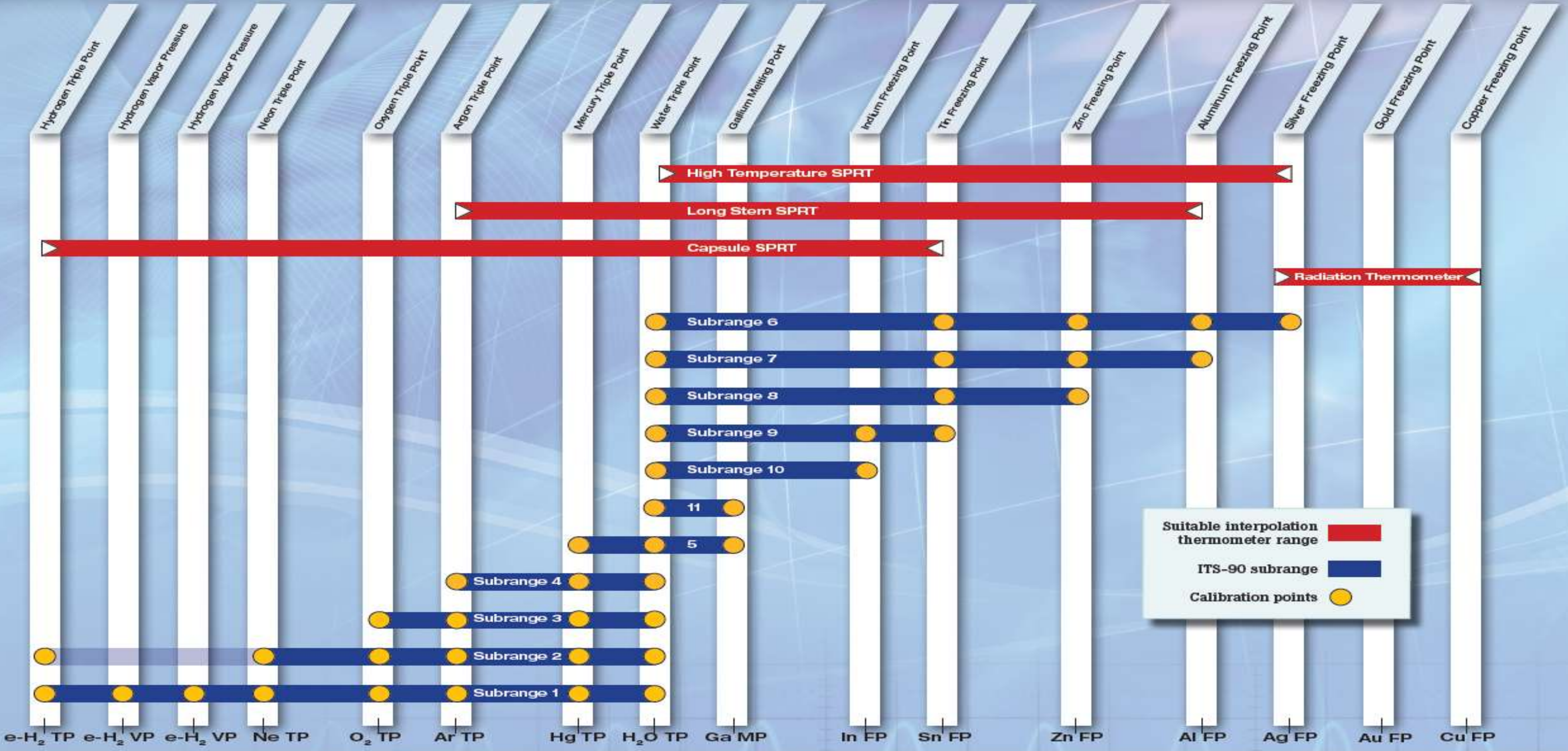
FLUKE ® Calibration

- Only the best materials are used:
  - 99.9999%+ pure metals
  - 99.999%+ pure graphite crucibles
  - 99.9999%+ pure Argon fill gas
- Built with the best manufacturing processes:
  - Extra cleaning of containers
  - Vacuum systems to ensure no contaminants during build
- Optional certification of cells:
  - Traceable to International System of Units (SI)
  - Best commercially available uncertainties

Fixed Point Cells – Care and Handling

- Fixed Point Cells are delicate instruments
- Most cannot be shipped***
- Keep clean using high purity alcohol
- Oils from the skin can damage them
- When not in use, store in upright position
  - Preferably in a padded container
  - Preferably in a low traffic area

Defining Temperatures of the ITS-90



	e-H_2 TP	e-H_2 VP	e-H_2 VP	Ne TP	O_2 TP	Ar TP	Hg TP	H_2O TP	Ga MP	In FP	Sn FP	Zn FP	Al FP	Ag FP	Au FP	Cu FP
t_{90} °C	-259.3467	-256.15	-252.85	-248.5939	-218.7916	-189.3442	-38.8344	0.01	29.7646	156.5985	231.928	419.527	660.323	961.78	1064.18	1084.62
T_{90} K	13.8033	17	20.3	24.5561	54.3584	83.8058	234.3156	273.16	302.9146	429.7485	505.078	692.677	933.473	1234.93	1337.33	1357.77

Components of ITS-90

SPRTs

Standard Platinum Resistance Thermometers

Definition of interpolation instruments, such as standard platinum resistance thermometers, vapor pressure thermometers, volume-interpolated gas thermometerstant

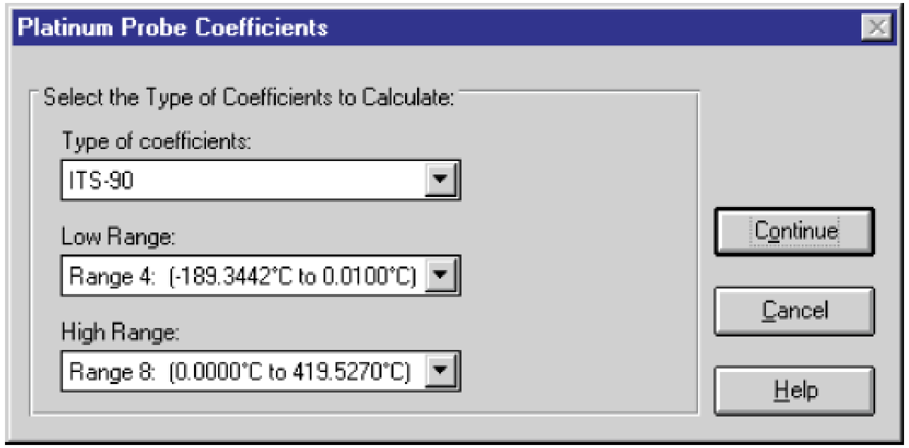
Sensors

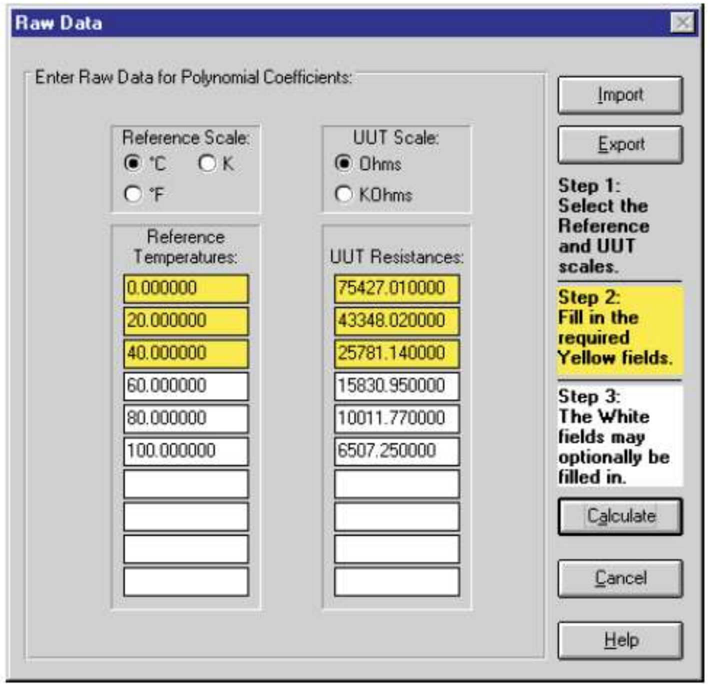
FLUKE ® Calibration

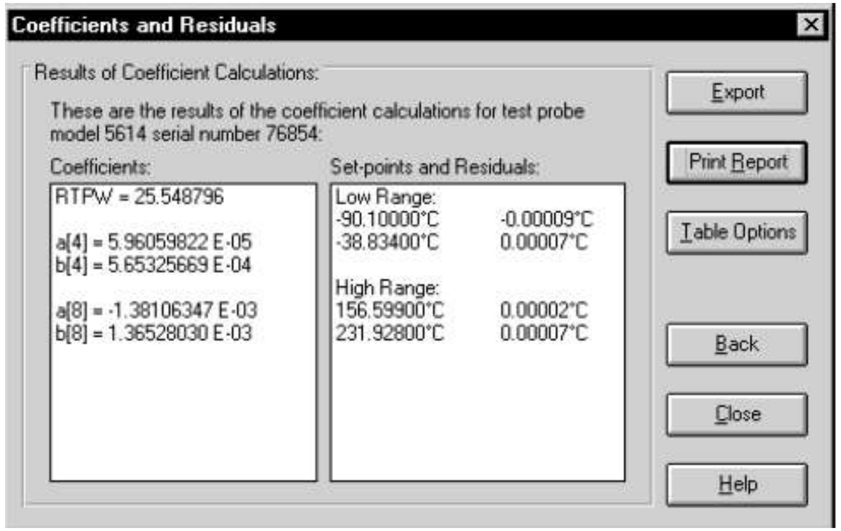
- Sensors – Calibrations Vary
  - Calibrated by Fixed Point, Comparison, standard curve fit.
  - Characterized using ITS-90, Poly, CVD, IEC DIN, Steinhart-Hart, etc.

CALIBRATION POINT			TEMPERATURE	MEASURED	UNCERTAINTY
(point °C)	(type)	(SN)	t90(°C)	RESISTANCE	(mK)
-197.000	Comp	N/A	-197.000	4.67299	±6.0
-80.000	Comp	N/A	-80.000	17.30656	±6.0
-38.834	Comp	N/A	-38.834	21.58569	±6.0
0.010	Comp	N/A	0.010	25.57102	±4.0
In	FP	44013	156.599	41.16238	±6.0
Sn	FP	45001	231.928	48.39812	±7.0
Zn	FP	S9007	419.527	65.68455	±9.0
Al	FP	17082	660.323	86.31876	±14.0

Components of ITS-90







Interpolation equations

Define interpolation equations relating the measured property of instruments to temperatures

$$W(T_{90}) = \frac{R(T_{90})}{R(273.16\ \text{K})}$$

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

$$T_{90}(W_r) = 273.15 + D_0 + \sum_{i=1}^{9} D_i \left[\frac{W_r(T_{90}) - 2.64}{1.64} \right]^i$$

ITS-90 coefficients

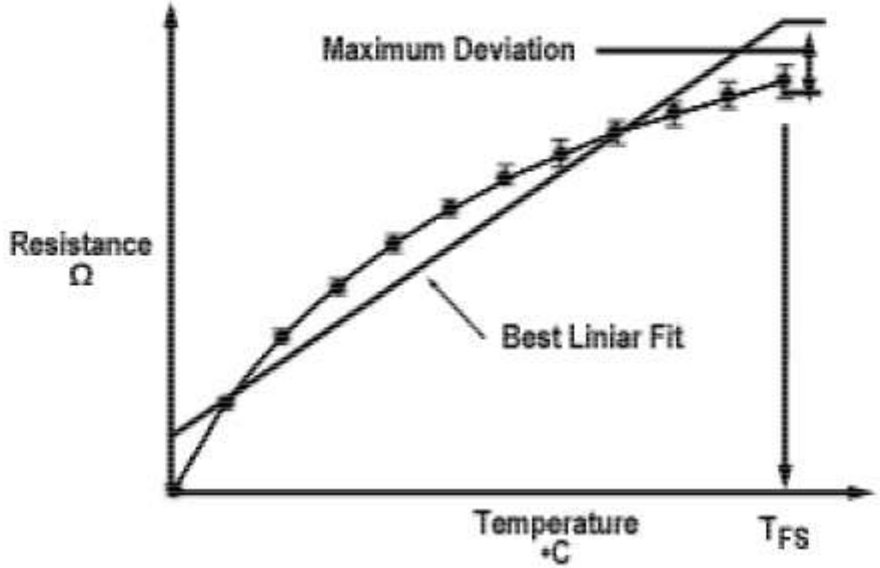
FLUKE® Calibration

Report Number:
B1202017

Fluke Calibration, American Fork
Primary Temperature Lab
Report of Calibration

The PRT was calibrated at the following temperatures with the associated uncertainties. The uncertainty evaluation accounts for all known uncertainties present at the time of calibration including long-term behavior of the calibration system, measurement noise, and any short-term effects of the PRT. The uncertainties are reported at the calibration temperatures only. The uncertainties at intermediate temperatures can be computed from these values and the ITS-90 propagation of error curves. The uncertainties are reported at a coverage factor of 2 (k=2).

CALIBRATION POINT (point °C)	(type)	(SN)	TEMPERATURE t90(°C)	MEASURED RESISTANCE	UNCERTAINTY (mK)
-197.000	Comp	N/A	-197.000	18.2097	±6.0
-100.000	Comp	N/A	-100.000	59.2614	±10.0
-38.834	Comp	N/A	-38.834	84.1391	±6.0
0.010	Comp	N/A	0.010	99.6745	±4.0
In	FP	44013	156.599	160.4537	±6.0
Sn	FP	S7005	231.928	188.6586	±6.0
Zn	FP	S9007	419.527	256.0466	±9.0
Al	FP	47007	660.323	336.4755	±14.0



The following values were determined for the RTPW and the coefficients of the pertinent deviation functions of the ITS-90. For best results, the RTPW value shown should be used as a baseline value for determining the stability of the PRT. The user should maintain a record of RTPW values measured as a routine operation and use these values when computing temperature.

Results for Nominal Current Calibration

RTPW = 99.6745 Ω

a4 = -1.320865 E-05
b4 = -2.063356 E-06
a7 = -8.464574 E-05
b7 = 7.092116 E-05
c7 = -3.463376 E-05

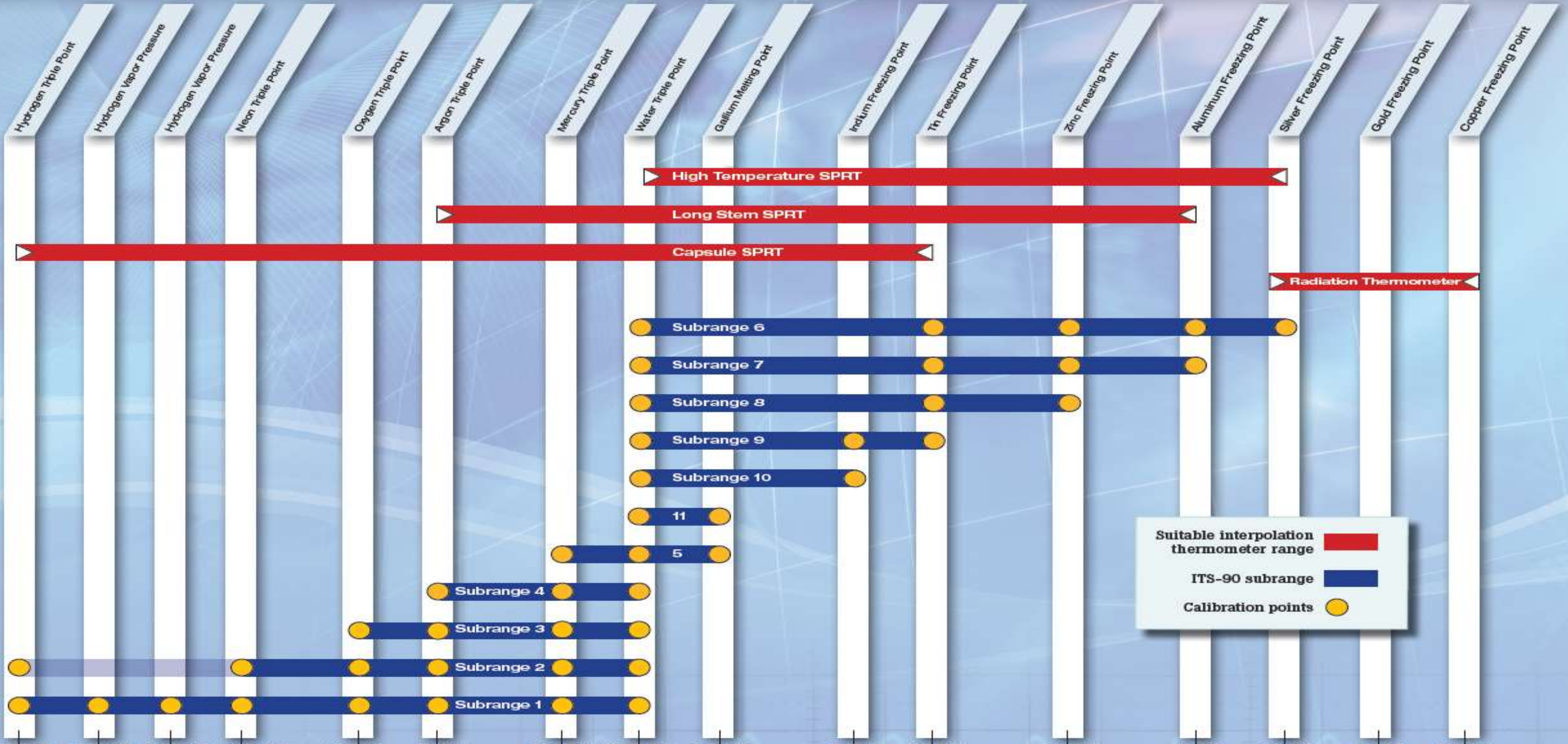
ITS-90 interpolation table

Model: 5626 Serial: 2958 — ITS-90 Temperature vs. Resistance Table

Temp (°C)	Resistance (ohms)	dR/dT (ohms/°C)	Temp (°C)	Resistance (ohms)	dR/dT (ohms/°C)	Temp (°C)	Resistance (ohms)	dR/dT (ohms/°C)
-200.00	16.92073	0.42902	**-143.00**	41.36284	0.42142	**-86.00**	65.00680	0.40913
-199.00	17.34975	0.42970	**-142.00**	41.78426	0.42114	**-85.00**	65.41593	0.40897
-198.00	17.77944	0.43030	**-141.00**	42.20540	0.42086	**-84.00**	65.82490	0.40880
-197.00	18.20974	0.43082	**-140.00**	42.62626	0.42058	**-83.00**	66.23370	0.40864
-196.00	18.64056	0.43126	**-139.00**	43.04684	0.42030	**-82.00**	66.64234	0.40848
-195.00	19.07182	0.43165	**-138.00**	43.46714	0.42003	**-81.00**	67.05082	0.40832
-194.00	19.50347	0.43197	**-137.00**	43.88718	0.41976	**-80.00**	67.45914	0.40816
-193.00	19.93544	0.43223	**-136.00**	44.30694	0.41950	**-79.00**	67.86731	0.40800
-192.00	20.36767	0.43245	**-135.00**	44.72643	0.41923	**-78.00**	68.27531	0.40785
-191.00	20.80012	0.43261	**-134.00**	45.14567	0.41897	**-77.00**	68.68316	0.40769
-190.00	21.23273	0.43273	**-133.00**	45.56464	0.41871	**-76.00**	69.09085	0.40754
-189.00	21.66545	0.43281	**-132.00**	45.98335	0.41846	**-75.00**	69.49839	0.40738
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]

Defining Temperatures of the ITS-90

FLUKE Calibration



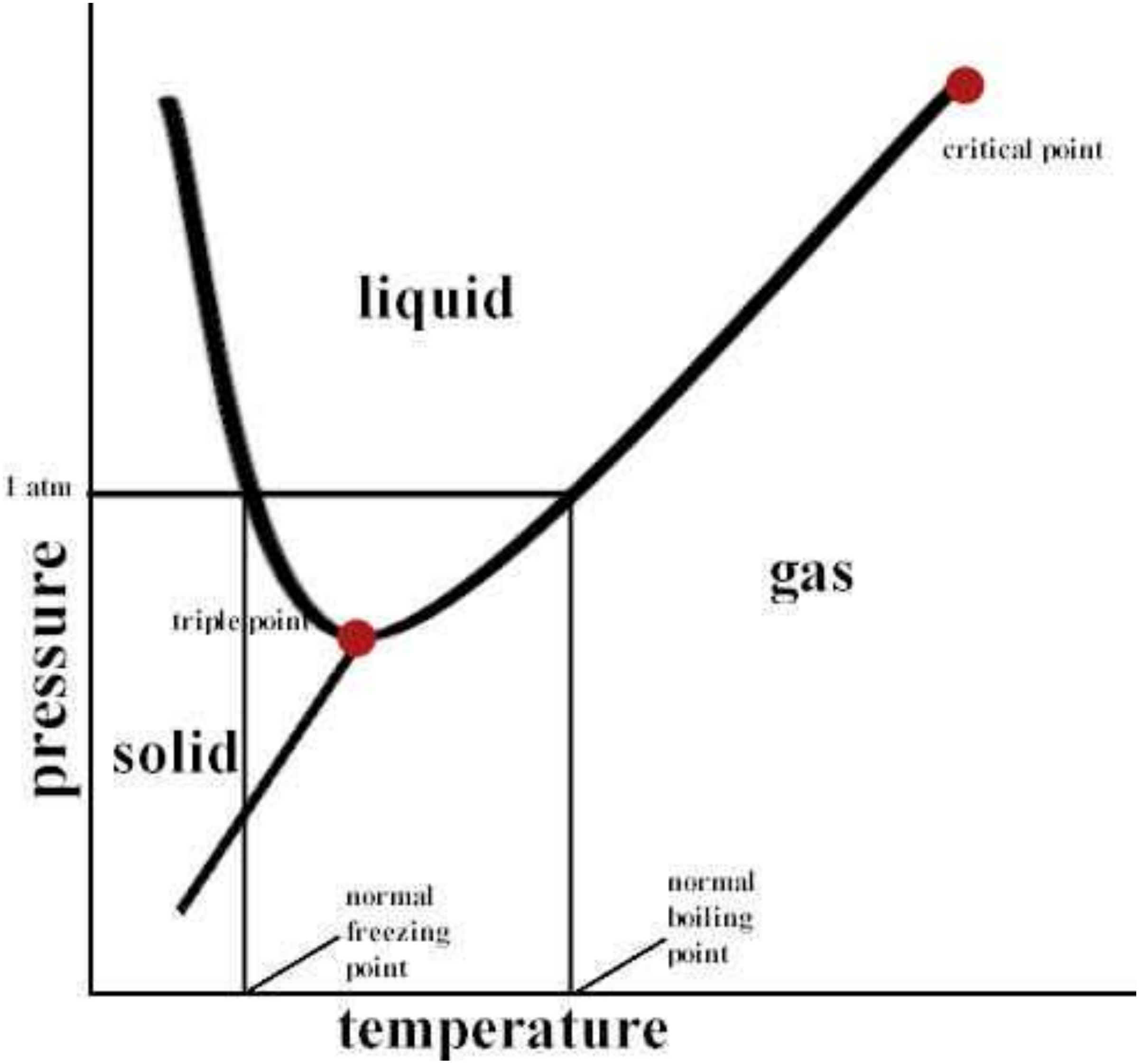
	$e\text{-}H_2$ TP	$e\text{-}H_2$ VP	$e\text{-}H_2$ VP	Ne TP	O_2 TP	Ar TP	Hg TP	H_2O TP	Ga MP	In FP	Sn FP	Zn FP	Al FP	Ag FP	Au FP	Cu FP
t_{90} °C	-259.3467	-256.15	-252.85	-248.5939	-218.7916	-189.3442	-38.8344	0.01	29.7646	156.5985	231.928	419.527	660.323	961.78	1064.18	1084.62
T_{90} K	13.8033	17	20.3	24.5561	54.3584	83.8058	234.3156	273.16	302.9146	429.7485	505.078	692.677	933.473	1234.93	1337.33	1357.77

TPW cell

Definition of Triple Point of Water [TPW]

- The triple point is, in thermodynamics, a point of the phase diagram which corresponds to the coexistence of three states (liquid, solid and gaseous) of a pure body. It is unique and only seen at a given temperature and pressure

The triple point of water (TPW) is the only thermometric fixed point used in definitions of both the thermodynamic temperature and the international temperature scale. The unit of thermodynamic temperature, Kelvin, is defined as the fraction 1/273.16 of the thermodynamic temperature of the TPW. It is also a defining fixed point on the International Temperature Scale of 1990 (ITS-90)



TPW Cell Realization – Equipment

- The traditional way with LN2 or dry ice and a lot of skill and patience

The smart way the quick stick

The easy way, with automation

The traditional way with dry ice

With dry ice fill the cell to form a coat of ice

Monitor the formation of ½ space ice mantles

Prevent the formation of an ice bridge that nay break the cell by heating this ice bridge

[*TPW Video*]

Place the cell in a Cup of water to eliminate the parallax and measure exactly the thickness of the ice mantle

[redacted]

“Quick Stick” Immersion Freezer

FLUKE ® Calibration

- Fast – about 45 minutes
- Simple – fill the cup with alcohol and either dry ice or liquid nitrogen

Consistently forms a uniform mantle

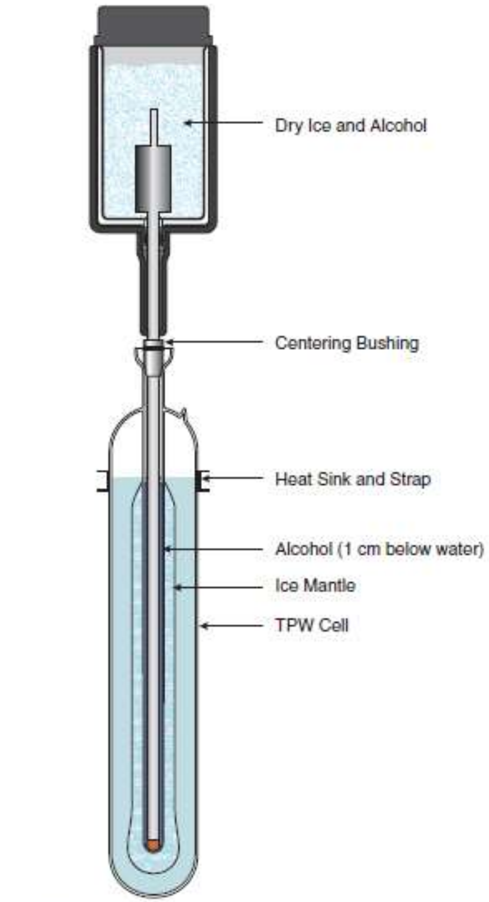
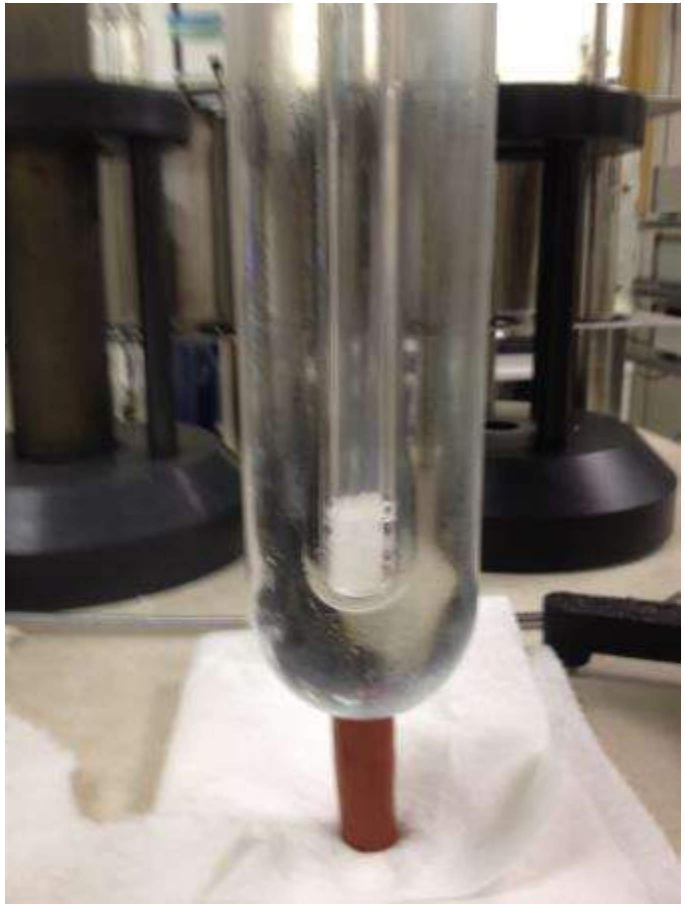


Figure 3 Freezing Process

Full Sized TPW Cell Realization Steps

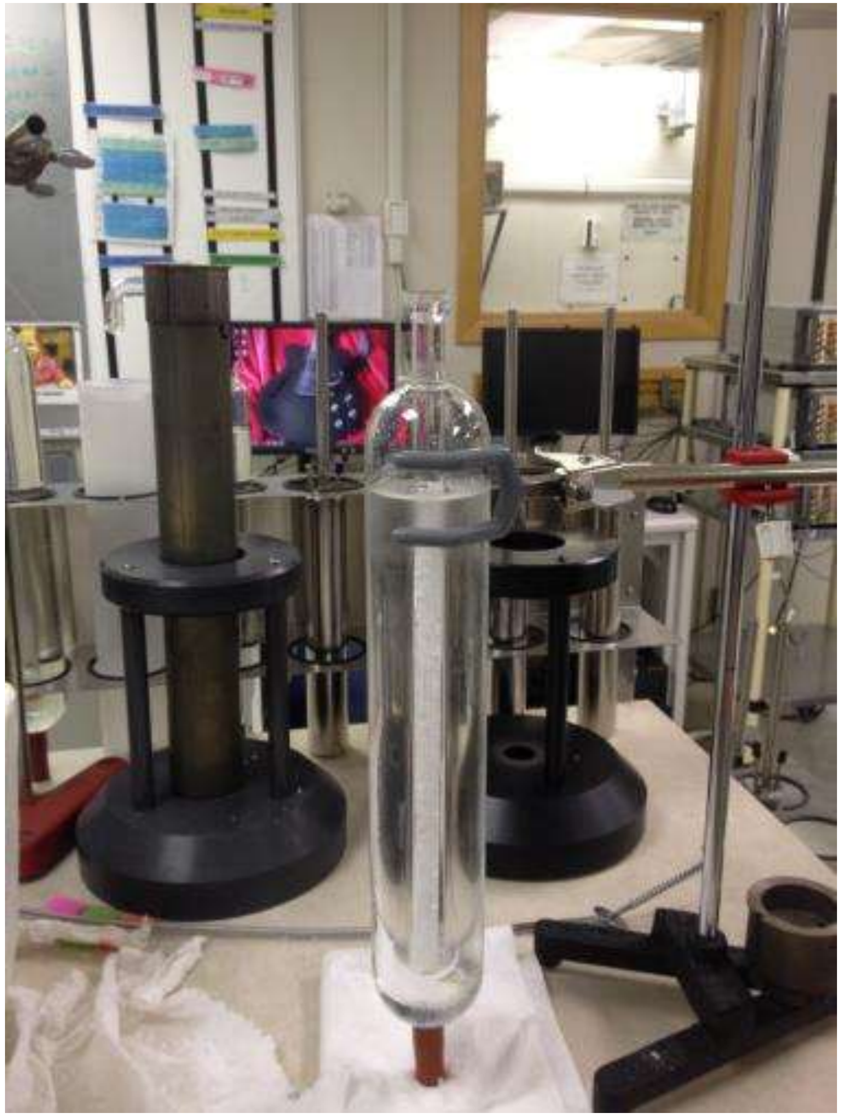
- *Both "Quick Stick" and "Entrant Well Cooling" methods start in the same manner*

- *Start a bulb in the bottom of the cell*
  - *Pre-chill cell*
  - *Dry entrant well*
  - *Place a few drops of alcohol in the entrant well*
  - *Add dry ice to the bulb*



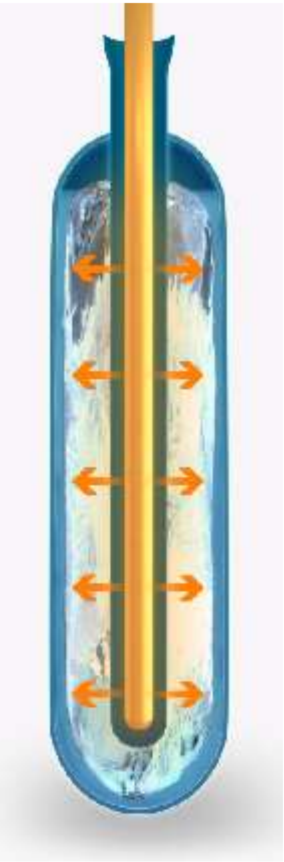
TPW Realization – Entrant Well Cooling Steps

- Fill entrant well with dry ice
- Continue to add dry ice to well until ice mantle is to desired size
- Carefully pour unused dry ice out of entrant well
- Place cell in maintenance bath 0.004°C to 0.007°C



TPW Realization – Entrant Well Cooling Drawbacks

- Introduces more stress to the mantle
- Delays use of cell
- 48 to 72 hours before cell is stabilized



To reduce the size of the ice mantle use a metal bar with room temperature and obtain a uniform and free appearance

TPW Realization – Quick Stick Steps

- While bulb is forming fill cup of "Quick Stick" with dry ice
- Add alcohol to make a slushy consistency

TPW Realization – Quick Stick Steps Continued

- After bulb is formed, place "Quick Stick" in cell and add alcohol to entrant well to bring it to the water level in the cell

TPW Realization – Quick Stick Steps Continued

- Add dry ice to “Quick Stick” cup every 15-20 minutes until mantle is desired size
- Remove “Quick Stick” and place cell in bath

Steps Quick Stick LN2 - Alternative

- Place the metal Rods in LN2 (Liquid Nitrogen)
- Fill the cell with acetone
- Insert a cold bar in the entrant well
- LN2 evaporates and transfers the cold to form an ice mantle
- Replace every 30 seconds with another cold bar (15 - 20) times
- Empty the acetone inlet and replace it with water + ice for thermal contact and place the cell in a Maintenance bath 0.004°C to 0.007°C

Full Sized TPW Cell Realization Methods

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Calibration

"Quick Stick"

- Pros
  - Less Labor intensive
  - Less stress in the ice = quicker time to use
    - About 24 hours
- Cons
  - Extra equipment required

Entrant Well Cooling

- Pros
  - No extra equipment needed
- Cons
  - Labor intensive
  - Longer time until it can be used
    - About 72 hours
  - Needs more attention to ensure cells don't break

Maintenance of the Triple Point Water

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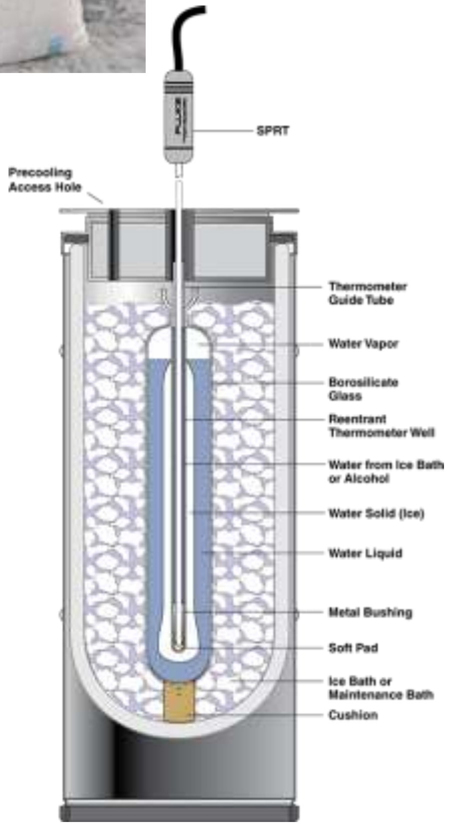
Calibration

Crushed ice:

Drain water and add ice every day
Typically provides a TPW lifetime of up to three weeks
Do not allow ice to freeze across top of TPW cell as it will break!

Liquid bath (Hart 7312 or 7012)

Maintain temperature: **0.004°C to 0.007°C**
Typically provides a longer TPW lifetime of up to two months



Automated mini TPW

- Fully automated operation
- < 0.0005° C uncertainty
- > 12-hour plateaus
- Self-calibration via water triple point cell
- Intrinsic standard

Realization of the Mini TPW cell using 9210 apparatus

FLUKE ® Calibration

Insert the TPW cell into the 9210 apparatus

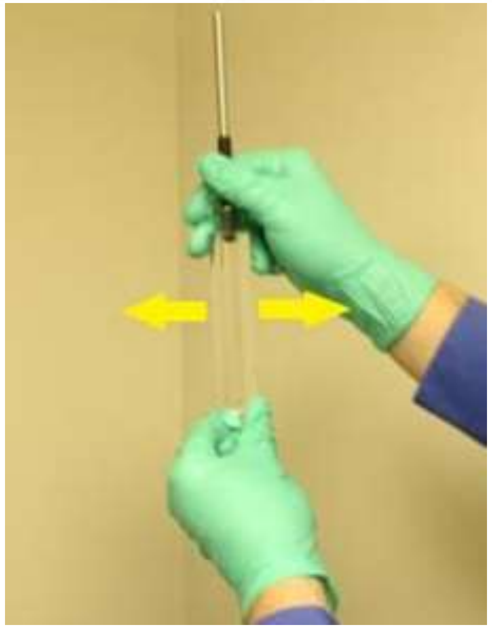
Add ethanol Alcohol to cover the TPW cell

Realization of the Mini TPW cell using 9210 apparatus

Set 9210 program to “freeze” mode

Take the cell out of the 9210, and the water is under supercool

Realization of the Mini TPW cell using 9210 apparatus



Shake the cell, the water starts to freeze, and ice spread through whole cell

←

Put the cell back into the 9210, and set 9210 program to “maintenance” mode

→

Mini TPW cell and Apparatus

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Calibration

- Realize TPW automatically, no dry ice or LN_2 needed
- Maintain for 1 day
- Used for monitoring reference thermometer Rtpw drift
- Used as temperature calibration system "checker"

[9210 TPW video]

TPW Cell Maintenance

Full Sized Cells

- Maintenance Baths
  - Can maintain cell for up to 60 days
- Make sure the mantle is free inside the cell
  - Insert a room temperature glass rod for 5 minutes into the entrant well
  - Tilt to a 45° angle
  - Repeat until mantle turns freely

Mini Cells

- 9210 Maintenance
  - 6-8 hours
  - Automatically melts
- No need to free mantle

Practical aspects of TPW

- Does not need recalibration
- Check reference probes for drift
- Valuable history – record all TPW measurements
- Most accurate temperature standard
- Easy to realise
- Check on return from calibration

QUESTION?

Should a secondary or industrial temperature lab own a TPW cell?

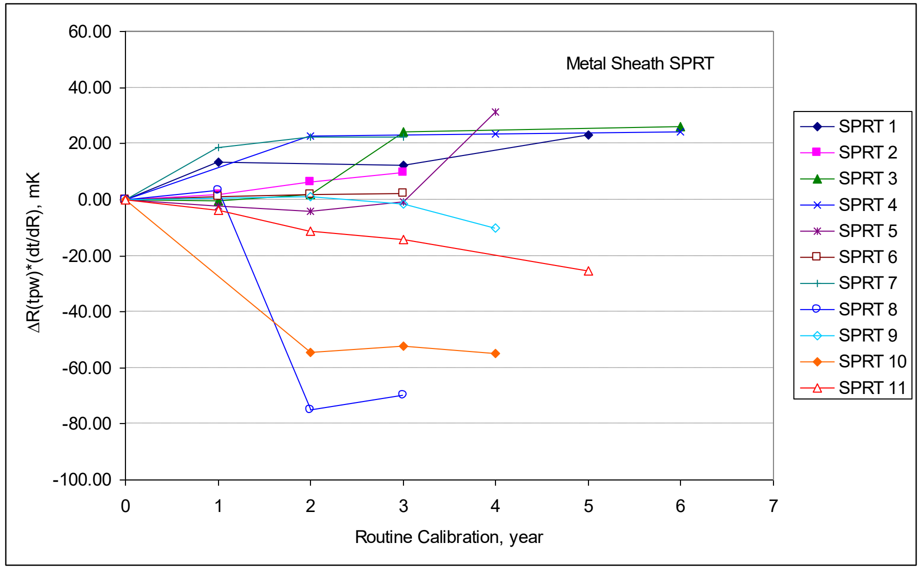
Reference Thermometer

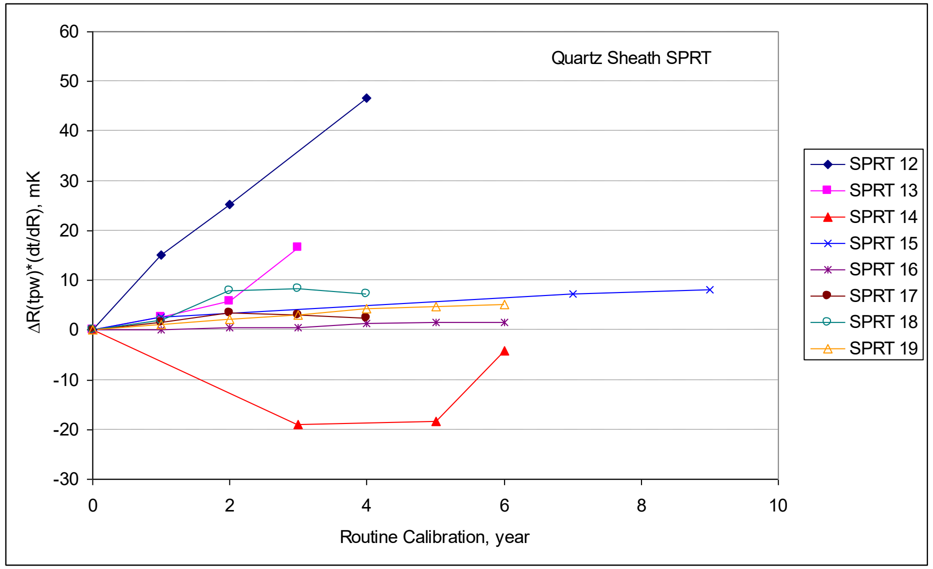
- Temperature calibration laboratories not using fixed-point standards use some level of reference or standard thermometer instead against which units under test (UUTs) are calibrated.
- Reference thermometers are typically sent to primary or upper level calibration laboratories for routine calibration. Often the calibration interval for a reference thermometer is one year.
- It is difficult for many users to observe and track the drift of the reference thermometer between calibrations.

Reference Thermometers

- Upon investigation of re-calibration results, it was found that the resistances at the triple point of water (Rtpw) of some reference thermometers were significantly different between the two calibrations.
- In some cases we found, the “bad” reference thermometers had been in use as standards for calibration work. The users did not know when the thermometers drifted significantly.
- Measurement instruments, including readouts and reference resistors, may also drift over some time period.
- If the reference thermometer drifts beyond the acceptable limit during the calibration interval, the process uncertainties may be compromised requiring recall of calibration work.

Examples of SPRT Calibration Results

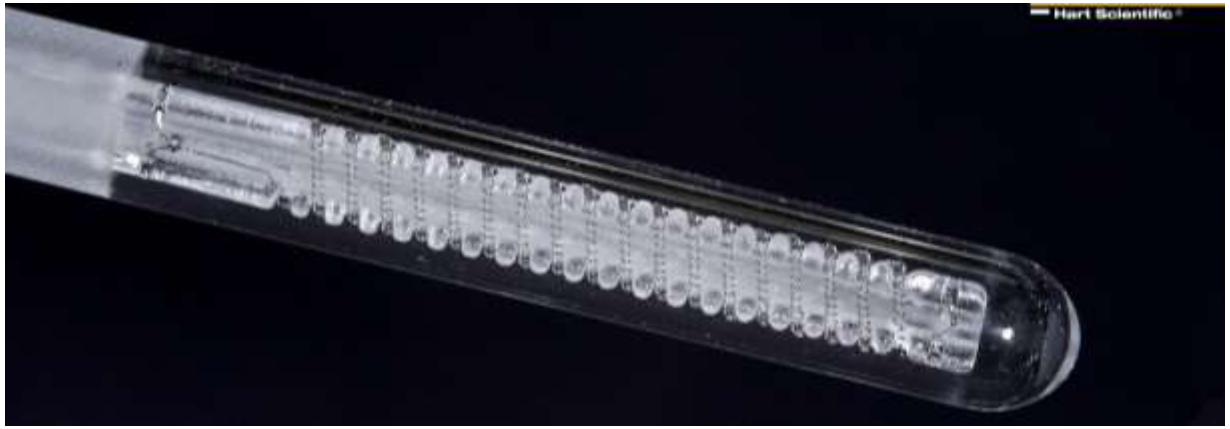




R(tpw) drift of selected metal-sheath SPRTs during routine calibration intervals

R(tpw) drift of selected quartz-sheath SPRTs during routine calibration intervals

Reference Probes



- Specified for stability, not accuracy
- Stability is affected by time and use
- Stability must be verified
- Problems with the reference will be noted before calibration errors arise
- There is a simple method available

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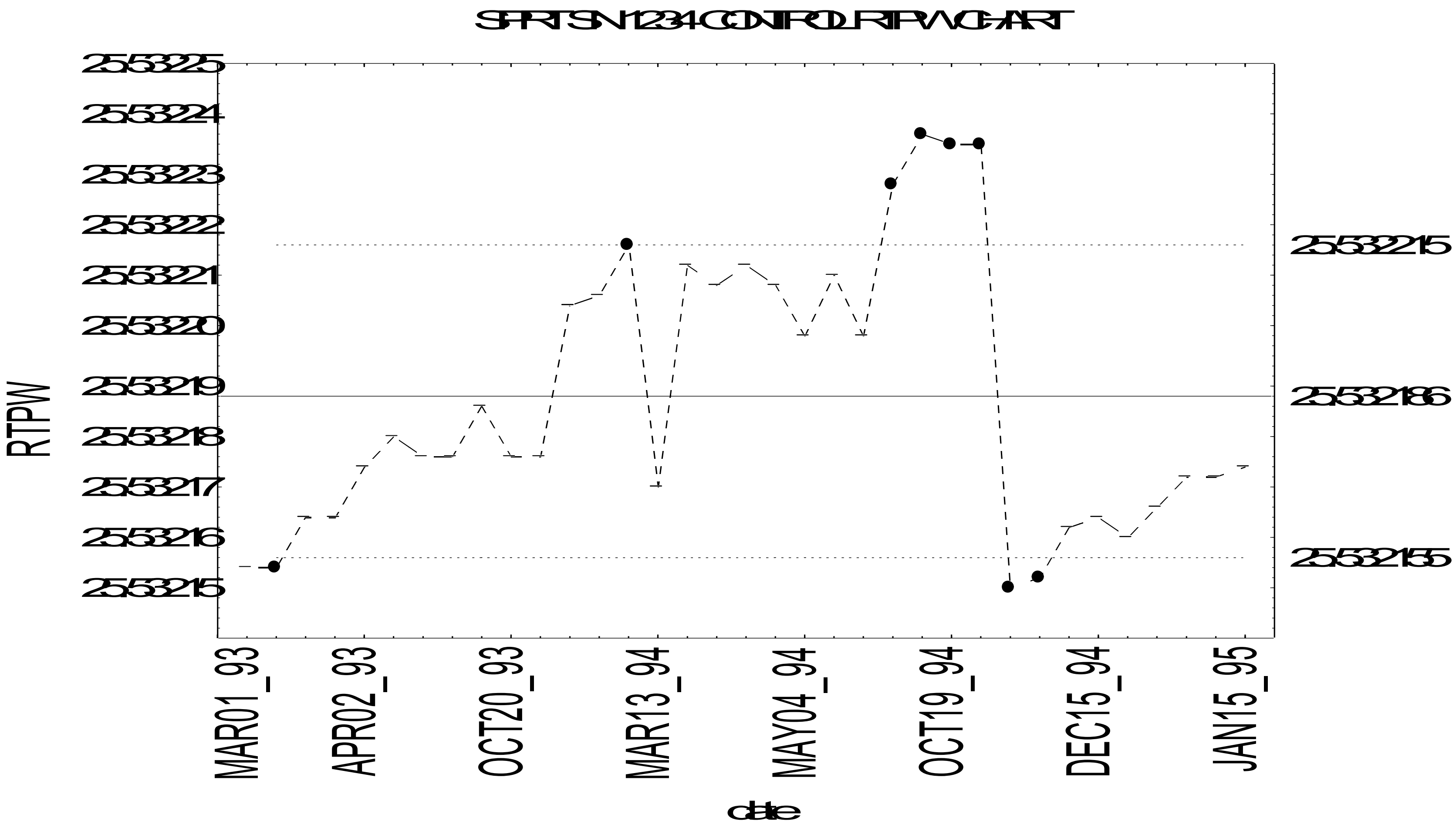
Calibration

Control Chart of Reference Probes



Summary on Reference thermometer

- Long-term stability of metal sheath SPRTs can be comparable to that of fused silica SPRTs.
- Many factors affect the performance of these two types of SPRTs.
- All SPRTs must be handled extremely carefully.
- Evaluation of Rtpw and W can help determine possible causes of observed drift.
- Reference thermometers should be checked regularly against a TPW cell.
- Resistance ratio W(t) should be used in temperature measurement to reduce uncertainty.
- Maintaining a control chart can indicate when a reference thermometer should be re-calibrated. It can also indicate equipment errors or failures.

Standard Checker for a temperature calibration system

- All components of the calibration system may drift over time.
- Changes in the thermometer readout or reference resistor can also produce apparent drift of the measured R(tpw) of the reference thermometer.
- Consider other possible problems that might affect temperature measurements, such as due to environmental conditions or electrical interference.
- When drift is observed, it is important to be able to identify which instrument or instruments are not stable.
- The mini TPW system could be used as a check standard for the entire temperature calibration system.

Questions?