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# WMO CIMO Training Workshop on Metrology for the English-speaking countries of Region V (South-West Pacific)

Melbourne, 21<sup>st</sup> - 25<sup>th</sup> November 2011

## **Temperature**

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- Calibration
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- ✓ **Temperature** is the level of thermal energy of a body.
- ✓ Heat transfer only occurs spontaneously from a high temperature to a low temperature, never the reverse.
- ✓ Temperature is intensive and not extensive physical quantity:

t # t1+t2+t3+t4 !

- ✓ Not directly measurable
- Thermodynamical temperature: measured by primary thermometers linked with others physical parameters (PV=nRT); very expensive and quite difficult



- ✓ History:
  - Temperature scale is the closest approximation of thermodynamic temperature scale: difference between theoretical and practical scale ITS90 is between 0.1 mK and 0.5 mK
  - ➢ First defined in 1927, last scale in 1990
  - Expressed in Kelvin unit : t(°C)=T(K)-273.15

### ✓ How is the ITS-90 defined in range from 13.8K to1234.9K:

- Fixed points: Fixed points are physical systems with stable temperature during phase transition and they are reproducible and repeatable.
- > With a standard platinum resistance thermometer.

#### Interpolation relationships with reduced resistance W(T).

## **Fixed points of ITS-90**



- > 17 fixed points defines ITS-90
- The most known fixed points are phase changes of pure materials (triple point, freezing point and melting point)
- Meteorological range is between triple point of mercury and freezing point of indium.



No	Temp	perature	Material	Phase	
NO	Т <sub>90</sub> [К]	t <sub>90</sub> [°C]	Wateria	transition	
1	3 to 5	-270.15 to - 268.154	Не	V	
2	13.8033	-259.3467	e-H <sub>2</sub>	Т	
3	~17	~-256.15	e-H <sub>2</sub> (or He)	V (or P)	
4	~20.3	~-252.85	e-H <sub>2</sub> (or He)	V (or P)	
5	24.5561	-248.5939	Ne	Т	
6	54.3584	-218.7916	O <sub>2</sub>	Т	
7	83.8058	-189.3442	Ar	т	
8	234.3156	-38.8344	Hg	т	
9	273.16	0.01	H <sub>2</sub> O	т	
10	302.9146	29.7646	Ga	М	
11	429.7485	156.5985	In	F	
12	505.078	231.928	Sn	F	
13	629.677	419.527	Zn	F	
14	933.473	660.323	Al	F	
15	1234.93	961.78	Ag	F	
16	1337.33	1064.18	Au	F	
17	1357.77	1084.62	Cu	F	

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✓ Temperatures are determined in terms of the **ratio of resistance**  $R(T_{90})$  at temperature  $T_{90}$  and the resistance R(273.16K) at the triple point of water:

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

✓ Reference function  $W_r(T_{90})$  is defined:

Range from 13.8033 K to 273.16 KRange from 273.15 K to 1234.94K
$$\ln[W_r(T_{90})] = A_0 + \sum_{i=1}^{12} A_i \cdot \left[ \frac{\ln\left(\frac{T_{90}}{273.16}\right) + 1.5}{1.5} \right]^i$$
 $W_r(T_{90}) = C_0 + \sum_{i=1}^{9} C_i \cdot \left[ \frac{T_{90} - 754.15}{481} \right]^i$ 

Numeric values of reference function coefficients (Ai, Ci) and inverse reference function (Bi, Di) are available in literature.

✓ **Deviation function** is a deviation of reference function  $W_r(T_{90})$  and ratio  $W(T_{90})$  is defined:

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



The forms of deviation function **differ** according to different temperature range and used fixed points. In the temperature range from 13.8033 K to 1234.94 K three forms of deviation function are defined:

• from 83.8058 K to 273.16 K:

$$\Delta W(T_{90}) = a \cdot \left[ W(T_{90}) - 1 \right] + b \cdot \left[ W(T_{90}) - 1 \right] \cdot \ln(W(T_{90}))$$

• from 273.15 K to 1234.94 K:

$$\Delta W(T_{90}) = a \cdot [W(T_{90}) - 1] + b \cdot [W(T_{90}) - 1]^2 + c \cdot [W(T_{90}) - 1]^3 + d \cdot [W(T_{90}) - W(660.323^{\circ}C)]^2$$

Values of coefficients a, b, c,  $c_i$ , d depends on **temperature range** and **fixed point** used in **calibration** and they are results in calibration of standard platinum resistance thermometer.

## **Types of contact thermometers**



Contact thermometers operate on the principle of change of characteristic as a result of change of temperature:



#### Liquid-in-glass:

Change of volume depending on the change of temperature.



(change of resistance):

Platinum resistance thermometer, thermistor

**Thermocouple**: Generation of thermoelectric voltage



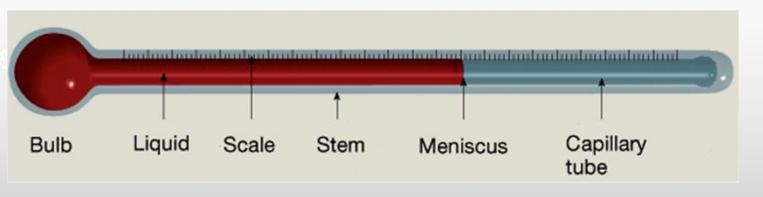
Thermograph: Mechanical change



### Liquid-in-glass thermometer







- One of the first thermometers
- Different accuracy classes (between mK and 10 K)
- Can be used in the range from –110°C to 600°C
- Simple to use
- As a result of difference in the volume expansion of glass and liquid within it as a change of temperature, the volume of liquid changes.



 $\checkmark\,$  Based on the thermal expansion and contraction of a liquid

Advantage	Disadvantage
no power source required	limited to applications where manual reading is acceptable
repeatable	cannot be digitised or automated
easy to use & cheap	careful handling is required
	mercury vapour is highly toxic

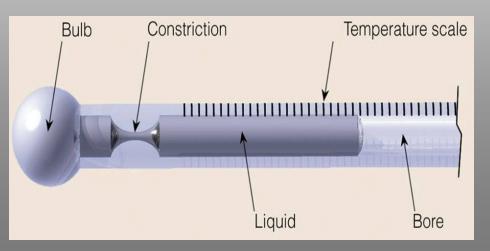
 $\checkmark$  Liquids and range:

Alcohol	-110°C to 100°C
Mercury	-38°C to 650°C
Mercury-thalium	-56°C to 650°C



The maximum thermometer is a mercury thermometer to measure the maximum temperature in a certain period.

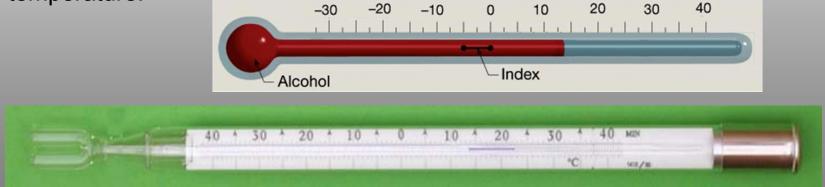
- This thermometer has a constriction in the capillary tube where the mercury is constricted between the bulb and the starting point of the scale.
- As air temperature rises, the mercury goes out of the bulb passing through the constriction.
- When air temperature falls, the mercury column breaks at the constriction. Thus mercury in the capillary tube cannot return to the bulb, and remains indicating the maximum temperature.



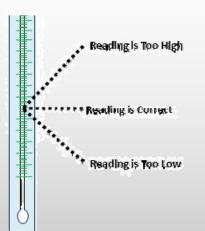


The minimum thermometer is a **spirit thermometer** to measure the minimum temperature in a certain period.

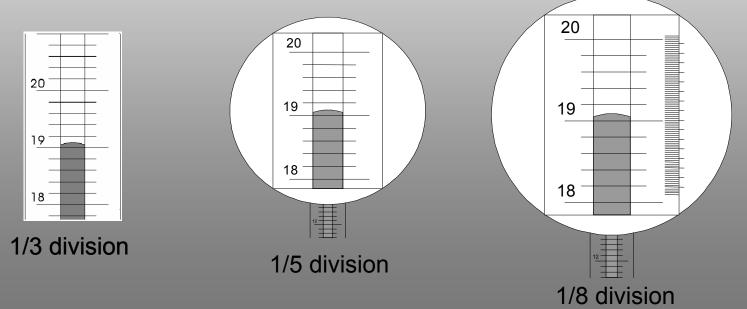
- > It has a dumbbell shaped index of colored glass in the spirit column.
- As air temperature falls, the index is dragged by the surface tension of the spirit and moves toward the bulb with the top of the column.
- When temperature rises, the index is left in its position because the spirit flows through it. As a result, the index remains indicating the minimum temperature.



Parallax error : Parallax errors of many tenths of a division may easily occur in reading a Lig thermometer. To avoid them one must view the thermometer from exactly the same angle as was used for calibration, virtually always perpendicular to the liquid column.



### ✓ Interpolation error:







Range [°C]	Scale division [°C]	Scale division [mm]	Resolution	Expanded uncertainty
		$\Delta$ < 0.5	Δ	0.6 · ∆
-30°C÷40°C <sub>ord</sub>	0.2	0.5 < <u>∆</u> < 1	0.5 · ∆	0.3 · ∆
-40°C÷40°C <sub>min</sub> -30°C÷50°C <sub>max</sub>	0.5	1 ≤ ∆ < 2	0.25 · ∆	0.2 · ∆

 $\Delta \dots$  scale division [mm]

### ✓ Breakage in the liquid column



Several ways to reunite the column:

- Lightly tap the thermometer against the palm of your hand.
- Apply centrifugal force.
- Cool the bulb (dry ice, alcohol) so that all the liquid enters the bulb leaving none in the stem. The column may rejoin when it is warmed to room temperature.
- Heat the thermometer and drive the separated column into expansion chamber. (never fill the expansion chamber more than 2/3 full).

#### ✓ Emergent stem:

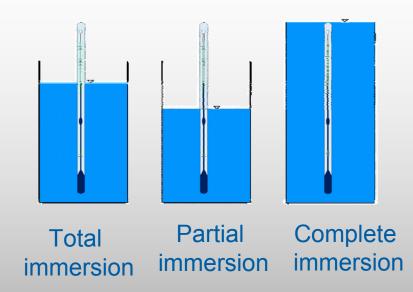
During calibration the thermometer must be correctly immersed in the calibration bath to ensure accurate measurements.

Partial immersion correction:

 $t_{corr} = t_{inst} + \Delta t$ 

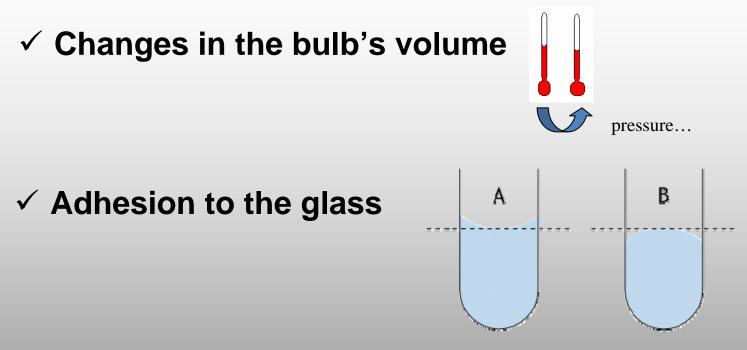
 $\Delta t = K \cdot n \cdot (Tc - Ts)$ 

- K the combined coefficient of expansion of the thermometric liquid and the glass. (Hg thermometers k=0.00016)
- **n** the number of scale degrees of the thermometer column between the surface of the liquid being measured and the meniscus of the liquid column.
- Tc the reading of the thermometer
- Ts average temperature of the emergent liquid column.









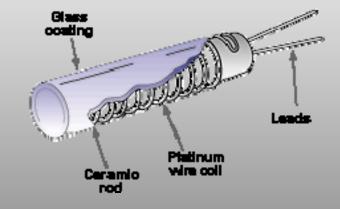
- ✓ Pollution of the liquid
- ✓ Glass expansion

#### Platinum resistance thermometer

- Based on the reversible change of the electrical resistance of a metal wire.
- Advantages of the platinum resistance thermometers:
  - ✓ Chemical stability
  - ✓ Available in a highly pure form
  - Excellent reproducibility of characteristics
  - ✓ Wide temperature range (–260°C to 1000°C)
  - ✓ Stable over time
  - ✓ Stable over temperature
  - ✓ Shallow slope (i.e.  $0.4\Omega/^{\circ}C$  for a 100 $\Omega$  PRT)
  - Relatively easy to measure
  - Relatively easy to calibrate

In general two types of resistance thermometers are defined:

- SPRT standard platinum resistance thermometer
- IPRT industrial platinum resistance thermometer





#### **Platinum resistance thermometer**



SPRT	IPRT
Capable of very high accuracy (mK)	Capable of moderate to high accuracy (10mK÷100mK)
Capable of wide temperature range	Capable of large temperature range
Extremely stable	Very stable
Relatively expensive to purchase	Relatively inexpensive to purchase
Relatively expensive to calibrate	Relatively inexpensive to calibrate
Extremely delicate	Less delicate





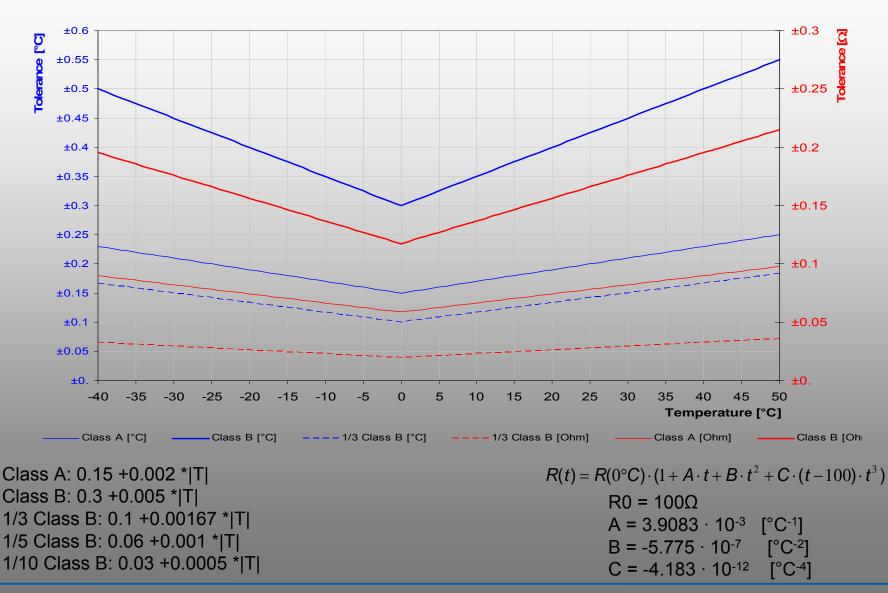
✓ Reference functions for IPRT (standard EN 60751):

- > From -200°C to 0°C:  $R_t = R_0 \left[ 1 + A \cdot t + B \cdot t^2 + C(t 100) \cdot t^3 \right]$
- > From 0°C to 661°C:  $R_t = R_0 \left[ 1 + A \cdot t + B \cdot t^2 \right]$

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#### **Resistance thermometer - standard EN 60751:**



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#### **Common error sources using Pt thermometers**



- Insulation Resistance: Error caused by the inability to measure the actual resistance of element. Current leaks into or out of the circuit through the sheath, between the element leads, or the elements.
- Stability: Ability to maintain R vs T over time as a result of thermal exposure.
- Repeatability: Ability to maintain R vs T under the same conditions after experiencing thermal cycling throughout a specified temperature range.
- Hysteresis: Change in the characteristics of the materials from which the RTD is built due to exposures to varying temperatures.
- Immersion depth: Error that results from the PRT sheath conducting heat into or out of the process.
- Lead Wire: Errors that occur because a 4 wire or 3 wire measurement is not used, this is greatly increased by longer connection wires.
  - 2 wire connection adds lead resistance in series with PRT element.
  - 3 wire connection relies on all 3 leads having equal resistance.
- Self Heating: Error produced by the heating of the PRT element due to the power applied.



- A thermograph is an instrument for recording the temperature of the surrounding air continuously and automatically.
- The sensor used is a bimetallic helix formed from aquaflex or coflex strips.
- Its one end is fixed while the other end is attached to a spindle to which the pen arm is screwed.
- When temperature changes, the curvature of the helix changes and this movement is recorded on a chart.

Thermograph properties:

- ➢ temperature range -30°C÷40°C
- very low maintenance
- easy reading and handling
- thermo-mechanic measuring principle
- typical accuracy ±0.3°C



#### Why calibrate?





#### **Equipment for temperature calibrations**



1. Temperature controlled medium:

Liquid baths in temperature range  $-50^{\circ}C$  to  $50^{\circ}C$ 



Climatic chambers in temperature range –20°C to 40°C





#### 2. A reference instrument



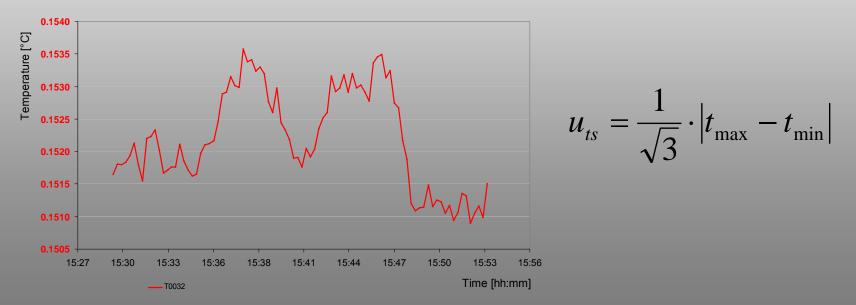
#### 3. Data acquiition unit (for reference and UUT)



#### **Temperature controlled medium**



- ✓ Temperature of a calibration bath/chamber cannot be considered as completely stable in time and homogeneous all over its volume.
- Metrological characterization of calibration bath/chamber consists of two components:
  - 1. Time stability or short-term stability of a medium temperature. It strongly depends on type of regulation and flow of medium inside the bath.



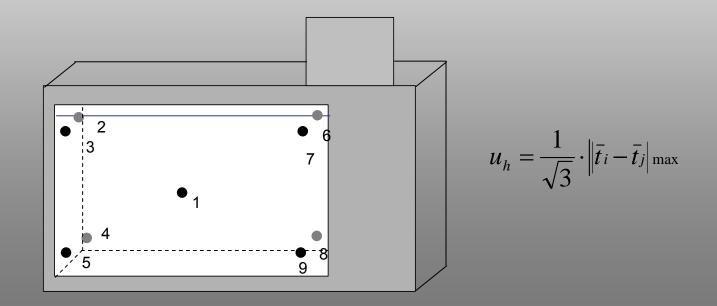
#### **Temperature controlled medium**



- **2. Spatial homogenity**: A gradient is observed as a change of a temperature reading of a thermometer according to a change of its position inside a calibration bath.
- Basic gradients that can be observed are vertical and horizontal gradient. A lot of calibration baths have either a cylindrical shape or equalizing blocks inside it is sometimes more appropriate to define axial and a radial gradient.

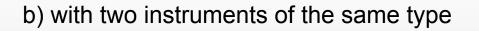
Spatial homogeneity can be measured using:

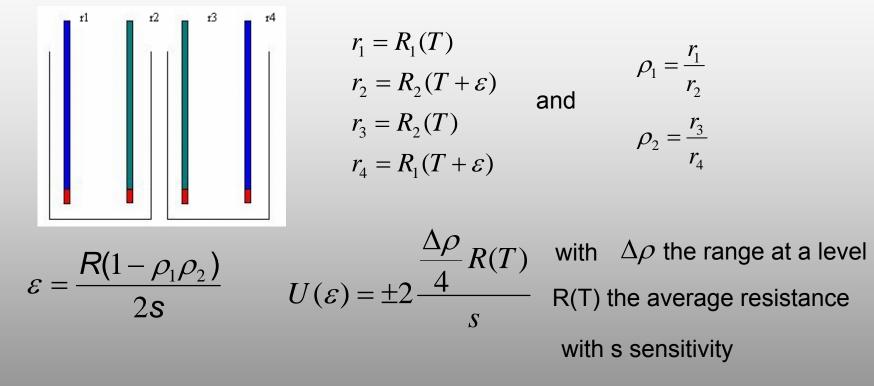
a) same type instruments



#### **Temperature controlled medium**





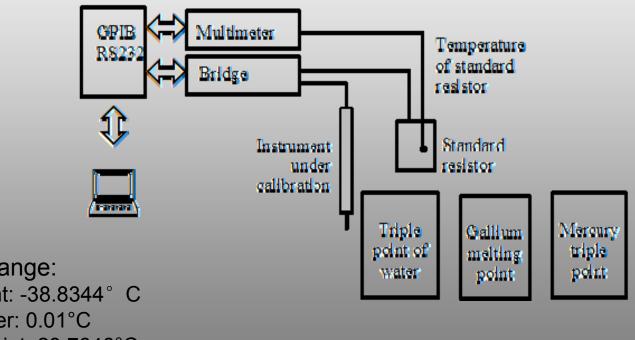


Time stability and spatial homogenity may represent a major contribution to the total uncertainty of a calibration procedure.

In order to decrease this uncertainty contributions equalizing blocks can be used in calibration baths.



**Fixed point method**: standard platinum resistance thermometers (SPRTs) at top uncertainty level (mK) are usually calibrated at fixed points, in accordance with ITS-90 definition.

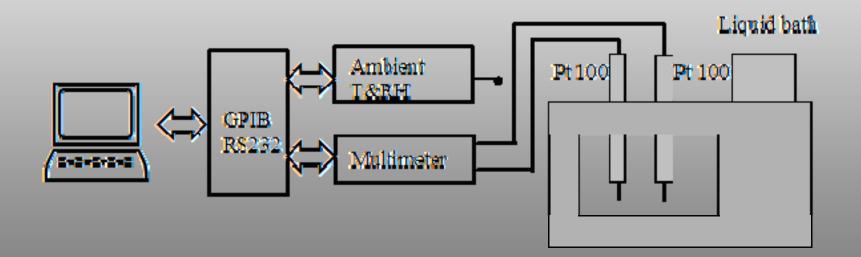


Meteorological range: Mercury triple point: -38.8344° C Triple point of water: 0.01°C Gallium melting point: 29.7646°C Indium freezing point: 156.5985° C



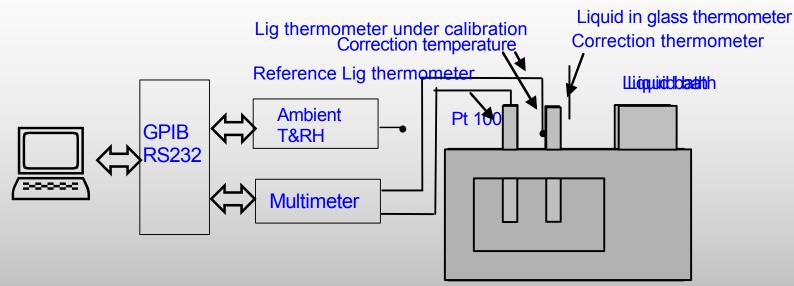
Industrial platinum resistance thermometers (IPRT), liquid-in-glass thermometers and thermistors are calibrated **by comparison** with laboratory's reference standard in a temperature controlled medium.

Typical calibration range: -40°C to 50°C (10°C increment) Number of measurement at each calibration point: 50 - 100



#### Liquid-in-glass thermometer calibration





#### Equipment used:

- ✓ Calibrated reference thermometer
- Liquid medium with known time stability and spatial homogenity

#### Measurement cycle:

- > 10 readings taken with reference thermometer
- > 10 readings taken with thermometer to be calibrated
- > 10 readings taken with reference thermometer

Assumptions:

- Calibration point: 20°C
- Operator able to interpolate to the fifth division and read with naked eye
- Immerged to the read degree
- Check at 0°C with melting ice

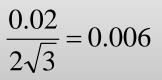
 $C = Tr - Tc + \delta T_{Rint\,erp} + \delta T_{Rcal} + \delta T_{Rdrift} + \delta T_{stem} + \delta T_{bath} + \delta T_{zero} + \delta T_{interp}$ 

- $\checkmark$  C: correction to be determined
- ✓ *Tr*. temperature reading of the reference thermometer
- ✓ Tc: temperature reading of the thermometer to be calibrated
- $\checkmark \delta T_{Rinterp}$ : correction linked to the interpolation of reference thermometer
- ✓  $\delta T_{Rcal}$ : correction linked to calibration of reference thermometer
- $\checkmark \delta T_{Rdrift}$ : correction linked to drift of reference thermometer bewteen two calib.
- $\checkmark \delta T_{stem:}$  correction linked with the emergent stem
- $\checkmark \delta T_{bath}$ : correction linked with the bath instability and uniformity
- ✓  $\delta T_{zero}$ : correction linked with the check of the zero
- $\checkmark \delta T_{interp}$ : correction linked with the interpolation of UUC





- > 10 readings of reference thermometer
  - Average = 20.22°C
  - Reading range = 0.02°C
  - Standard uncertainty = 0.006°C



- > 10 readings of thermometer to be calibrated
  - Mean = 20.02°C
  - Reading range = 0.02°C
  - Standard uncertainty =0.006°C

```
\frac{0.02}{2\sqrt{3}} = 0.006
```



δT<sub>Rinterp</sub>: operator is capable of interpolating the readings of reference themometer to the fifth of a division (scale division 0.1°C):

$$\delta T_{\text{Rinterp}} = 0;$$
  $u(\delta T_{\text{Rinterp}}) = \frac{0.1 \cdot \frac{1}{5}}{2 \cdot \sqrt{3}} = 0.006^{\circ}C$ 

δT<sub>Rcalib</sub>: Correction linked to calibration of reference thermometer. The certificate states correction of -0.1°C with expanded uncertainty 0.15°C:

$$\delta T_{\text{Rinterp}} = -0.1^{\circ}\text{C}; \quad u(\delta T_{\text{Rcal}}) = \frac{0.15}{2} = 0.075^{\circ}C$$

δT<sub>Rdrift</sub>: Correction linked to reference thermometer drift: Assume no drift, uncertainty contribution equal to interpolation

$$\delta T_{\text{Rdrift}} = 0;$$
  $u(\delta T_{\text{Rdrift}}) = 0.006^{\circ}C$ 

 $\succ \delta T_{steam}$ : Assume the reference thermometer and thermometer to be calibrated are immersed to the degree read. In practice small part of the column emerges from the bath to be read.

$$\delta T_{\text{steam}} = 0$$
  $u(\delta T_{\text{steam}}) = 0.006^{\circ}C$ 

 $\succ \delta T_{bath}$ : Correction linked the time stability and spatial homogenity of calibration bath.

$$\delta T_{\text{bath}} = 0$$
  $u(\delta T_{\text{bath}}) = \sqrt{u_{ts}^2 + u_h^2} = 0.010^{\circ}C$ 

δT<sub>zero</sub>: Correction linked to the change of zero value of the thermometer to be calibrated before and after calibration. (Uncertainty linked to the interpolaton)

$$\delta T_{zero} = 0$$
  $u(\delta T_{zero}) = 0.006^{\circ}C$ 

δT<sub>interp</sub>: Correction linked to the interpolation of instrument under calibration. Operator is capable of interpolating the readings of reference themometer to the fifth of a division (scale division 0.1°C):

$$\delta T_{\text{interp}} = 0;$$
  $u(\delta T_{\text{interp}}) = \frac{0.1 \cdot \frac{1}{5}}{2 \cdot \sqrt{3}} = 0.006^{\circ} C$ 

## **Uncertainty budget of Lig thermometer calibration**



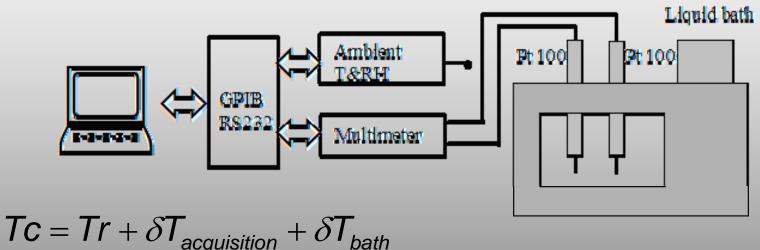
Quantity	Symbol	Value/ corr. [°C]	Standard uncert. [°C]	Prob. Iaw	Sensitivity coefficient	Uncertainty contribution [°C]
Reference thermometer reading	δTr	20.22	0.006	Rect	1	0.006
Instrument under calibration reading	δΤϲ	20.02	0.006	Rect	1	0.006
Reference thermometer interpolation	$\delta T_{Rinterp}$	0	0.006	Rect	1	0.006
Reference thermometer calibration	$\delta T_{Rcal}$	-0.1	0.075	Normal	1	0.075
Reference thermometer drift	$\delta T_{Rdrift}$	0	0.006	Rect	1	0.006
Reference thermometer emerging steam	$\delta T_{steam}$	0	0.006	Rect	1	0.006
Bath stability and homogenity	$\delta T_{bath}$	0	0.010	Normal	1	0.010
Change in zero value	$\delta T_{zero}$	0	0.006	Rect	1	0.006
Interpolation of instrument under calibration	δT <sub>interp</sub>	0	0.006	Rect	1	0.006
Correction		0.10				0.078

Result: 0.10°C±0.16°C (k=2)

#### **Resistance thermometer calibration**



- > By comparison with our reference standard  $Pt_{100}$
- 7 temperature calibration points in the range from -20°C to 40°C (10°C step)
- Liquid bath without thermal block
- Using multimeters for data acquisition



- $\checkmark$  *Tc*: temperature of the thermometer to calibrate
- $\checkmark$  *Tr*: temperature of the reference
- $\checkmark$   $\delta T_{acquisition}$ : correction linked with the data acquisition
- $\checkmark \delta T_{bath}$ : correction linked with the liquid bath



- Repeatability of measurements during a calibration
  - At least 50 (typically 100) measurements at each calibration point
  - U<sub>RAtype</sub>= 0.002°C - Standard deviation
- Correction linked to calibration of reference thermometer. The certificate states correction polinoma cofficients and expanded uncertainty 0.29°C
  - Corrections must be applied
  - $u_{calib} = \frac{0.029}{2} = 0.0145^{\circ}C$ - Calibration expanded uncertainty: 0.029 °C (k=2)
- $\succ$  Drift of working standard is based on the maximal difference between 2 calibrations (0.01°C)

$$\boldsymbol{J}_{drift} = \frac{0.01}{\sqrt{3}} = 0.0058^{\circ}\boldsymbol{C}$$

- Thermal coupling with the surroundings
  - Tests at different immersion depth
  - Rectangular law
- Self-heating of reference thermometer
  - Corrections are applied
  - From the measurement of self heating

$$u_{coupling} = \frac{0.0055}{\sqrt{3}} = 0.0032^{\circ}C$$

 $u_{heat} = 0.002^{\circ}C$ 



- Resolution of calibrated instrument
- Repeatability: maximum standard deviation during the calibration
- > Self-heating: with 2 electric currents (1mA and  $\sqrt{2}$  mA)

$$R(0mA) = 2 \cdot R(1mA) - R(\sqrt{2}mA)$$

Uncertainty due to time stability and spatial homogenity of calibration bath.

$$u(T_{bath}) = \sqrt{u_{ts}^2 + u_h^2} = 0.010^{\circ}C$$



- ► Uncertainty due to calibration of multimeter  $u_{dmm} = 0.0013^{\circ}C$
- ► Uncertainty due to time stability of multimeter  $u_{ts} = 0.0009^{\circ}C$
- ➢ Uncertainty due to ambient temperature influence  $U_{ambient} = 0.0002°C$
- > Multimeter resolution least significant bit LSB  $u_{resol} = 0.0001^{\circ}C$
- $\blacktriangleright \text{ Multimeter additional noise } u_{resol} = 0.0001 \,^{\circ}C$
- Uncertainty linked to the connection: The reference probe is four wire connected to multimeter and the uncertainty associated with the connection is taken to be negligible.



- Data supposed to be independent or non-correlated
- Expanded uncertainty:

$$u_c^2 = \sum u_i^2$$
$$U = 2 \cdot u_c$$

Example of the result:

T <sub>s</sub>	T <sub>x</sub>	T <sub>k</sub>	U
[°C]	[°C]	[°C]	[°C]
-19.87	-20.04	0.17	0.05
-9.82	-9.82	0.00	0.05
0.15	0.21	-0.06	0.05
10.15	10.08	0.07	0.05
20.18	20.07	0.11	0.05
30.17	30.21	-0.04	0.05
40.07	40.26	-0.19	0.05

Legend

Ts

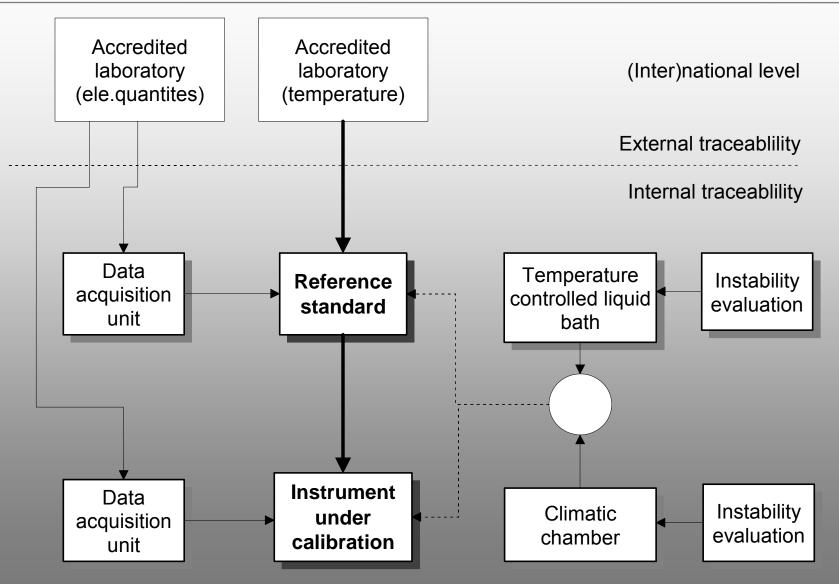
T<sub>x</sub> T<sub>k</sub>

U

- Value determined by the reference instrument
- Value determined by the instrument under calibration
- The correction
  - The expanded measurement uncertainty of the correction

#### **Traceability chart**







- Neither an easy definition or measurement but a lot of different types of sensors
- Choice according the use
- In meteorology, the more common are:
  - Liquid-in-glass thermometer
  - Resistance thermometer



No adjustment

Accept or reject according the requirements (class A, B...)