# A FIRST REVIEW OF

# CALIBRATION DEVICES ACCEPTABLE

# FOR METROLOGY LABORATORY

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### Abstract:

CIMO-XIII and CIMO Management Group requested to strengthen further the services of the Regional Instrument Centres (RICs), especially those in developing countries, In this regard, different workshops on Metrology have been conducted. During this meetings it became clear that only a few of the Laboratory had traceability to a recognized international standard and that it is a turning point between mechanical and electronic devices. It was also shown that methods and instruments in use are very different from a country to another. To standardize metrology laboratory, a list of acceptable calibration devices, references and generators, is presented for the main surface parameters such as pressure, temperature and humidity.

### Résumé:

La 13ème session de la CIMO et le Management Group demandèrent un renforcement des fonctions des Centres Régionaux d'Instrumentation, en particulier ceux présents dans les pays en voie de développement. Dans ce but, différents ateliers de Métrologie ont été organisés. Pendant ces rencontres, il a été démontré que peu de laboratoires garantissent la traçabilité aux étalons internationaux et que nous sommes à un tournant entre les capteurs mécaniques aux capteurs électroniques. Il a été aussi mis en valeur le fait que les instruments et méthodes sont très différents d'un pays à un autre. Pour normaliser les laboratoires d'étalonnage, une liste de systèmes utilisables pour l'étalonnage comme des références ou des générateurs est présentée pour les principaux paramètres d'observation de surface tels que la pression, la température et l'humidité.

### 1. Introduction

Pressure, temperature and humidity are important atmosphere's state parameters. Meteorological requirements for temperature measurements primarily relate to:

- the surface;
- the upper air;
- the surface levels of the sea and lakes.

These measurements are required for input to numerical weather forecast models, for agriculture, hydrology or climatology. Requirements on these measurement are described in the CIMO guide [1]. This paper will try to present some devices acceptable for Metrology Laboratory and especially for pressure, temperature and humidity measurement.

### 2. Pressure

The important decision to make is to determine what type of standard is appropriate for the calibration laboratory. The most important consideration in selecting a reference instrument is traceability. In pressure terms, traceability is defined as the ability to trace the calibration of a given measurement either directly or indirectly to standards of mass and length. The top choice is between primary and secondary laboratory standards.

A primary pressure standard is a pressure measuring instrument, which can reduce pressure measurements into measurements of mass, length and temperature and gravity. Examples are pressure balances or dead weight testers and primary mercury barometers. A secondary standard is an instrument which must be calibrated to relate the output directly to pressure. Both standards have advantages and disadvantages ([2] & [3]).

### 2.1. Primary Pressure Standards

The basic principle (see fig 1.) of the pressure balance consists of a vertical piston (P) freely rotating within a cylinder (C). The two elements of good quality define a surface called 'effective area'. The pressure to be measured is applied of the piston, creating an upward vertical force. This force is equilibrated by the gravitational downward force due to masses (M) submitted to the local gravity and placed on the top of the piston. The primary mercury barometer must operate in high vacuum above mercury column, contain highly-pure mercury at constant temperature and be located in pollution safe environment (see fig 2.).



Figure 1: Pressure balance principle



Figure 2: Example of a dead weigh gauge

### Advantages:

- Pressure balances are traceable to measurements of mass and length and therefore more directly to national standards.
- They have good long term stability.
- High accuracy, high reproducibility, reliable and stable instrument.

## Disadvantages:

- Time consuming operation for day-to-day routine work.
- Measurements must be corrected for temperature, local gravity and air buoyancy which can increase probability for errors as the corrections are applied.
- Mercury barometers potentially represent a health and environmental hazard.

## 2.2. Secondary Pressure Standards

Secondary pressure standards may be secondary mercury barometers or electronic barometers (quartz, see Fig. 3) with good long term stability. With recent development of high quality electronic barometers measuring uncertainty was reduced to few Pascals level.



Figure 3: Example of a quartz barometer

### Advantages:

- Faster and easier to use.
- Easy to adapt to automatic operation.
- Generally less expensive.

## Disadvantages:

- Pressure measurements cannot be reduced to measurements of mass, length or temperature.
- Higher measurement uncertainty.

Primary and secondary standard calibration devices are typically used and maintained by the calibration laboratory within an organization. With primary or secondary pressure standards laboratory can then either directly calibrate working standards, calibrate travelling standards or calibrate field measuring instruments.

### 2.3. Travelling pressure standards

Travelling pressure standard is typically used for on-site calibration of barometers. Due to mercury barometer transportation problems it is more convenient to use an electronic barometer (see fig 4.) as a travelling standard. Travelling standards must be checked against reference standard before an after on-site measurements to ensure metrological properties.



Figure 4 : An example electronic barometer

### 2.4. Calibration systems

When establishing pressure calibration laboratory several options regarding pressure standards must be discussed. There are different levels of pressure standards and traceability issues available to be implemented in calibration laboratory:

- 1. Using primary standards: Traceability must be maintained through periodic recalibrations to base SI units with smallest uncertainty level. Reference standards are then used for dissemination of value or, in some cases, calibration of instruments on smallest uncertainty level. Highly pure medium (nitrogen) for pressure generation combined with a pressure regulator generates calibration points in working standard comparison calibration. Working standards are then used for filed measuring instruments on a daily bases.
- 2. Using secondary standards (see fig 5.): A secondary standard must be traceable directly through pressure calibrations to the national or international level. The measuring system must also include similar pressure medium like in previous case. Secondary standards can be used for working standards calibration purposes or field measuring instruments calibrations.

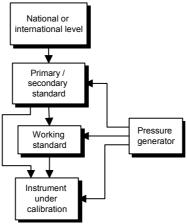


Figure 5: Traceability scheme using primary or secondary standards

3. Using working standards (see fig 6.): A working standard must be traceable directly pressure calibrations to the national or international level. Barometric chamber (or other pressure generator) can be used for comparison calibrations of instruments (see fig 7.).

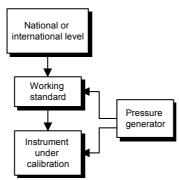


Figure 6: Traceability scheme using working standard

Financial investments in calibration equipment increases when uncertainty approaches beneath 10 Pascal level. From financial perspective is very important to recognize different approaches to establish and maintain traceable of pressure calibrations especially in developing countries.





Figure 7: example of a generator (Left) and of a barometric chamber (Right)

### 3. Temperature

#### The International Temperature Scale 3.1.

To link physical laws with reality, an international scale of temperature has been defined. A scale of temperature is made up of:

- a thermometer, a sensor with an output depending on temperature;
- an interpolation function that links the sensor's output with temperature;
- fixed points of temperature to define the interpolation function.

The International Temperature Scale, first defined in 1927, has been reviewed to reduce the difference between absolute temperature and temperature in the scale. The last scale was defined in 1990 hence ITS 90. The triple point of water is the major point of definition (t=0.01 °C).

ITS 90 consists of different areas and sub-areas with their own definition of T90. For the common parts, the definitions coexist.

ITS 90 enables easy and reproducible measurements of temperature.

ITS 90 is defined with a thermometer with platinum resistance, the interpolation functions are expressed with reduced resistance W(T), against the resistance at the triple point of water

$$W(T) = \frac{R(T)}{R(273.16K)}$$
 and against the reference function: W(T)=Wr(T)+\(\sup W(T)\). efined by some fixed points.

ITS 90 is defined by some fixed points.

The following spreadsheet sums up the different fixed points that define the ITS 90, at the atmospheric pressure (except for the triple point):

Température		BODY1	POINT
T90 (K)	T90 (°C)		
83,8058	-189,3442	Ar	T
234,3156	-38,8344	Hg	Т
273,16	0,01	H2O	Т
302,9146	29,7646	Ga	М
429,7485	156,5985	In	F

- e-H2: according to molecular composition 1
- 2 V: saturated vapour pressure
  - T: triple point between fluid, vapour and gas
  - G: thermometer with gas
  - F.M: freezing or melting point

Table 1. Fixed points of the ITS 90

The range acceptable in meteorology is between the triple point of mercury and the melting point of gallium (see fig 8.). To cover a wider range, it is also possible to use Argon and Indium point, but their respective temperature are far from typical meteorological temperature.

Between the triple point of hydrogen (13,8033K) and the freezing point of silver (961.78 °C) temperature according ITS 90, T90, is defined with a platinum resistance thermometer, calibrated against specific fixed points and using the interpolation functions.

The interpolation is made with this reference function, defined in the standard EN 60751: 1995, with t in °C:

from -200 to 0 °C:

$$R_{t} = R_{0} \Big[ 1 + A \cdot t + B \cdot t^{2} + C \big( t - 100 \big) \cdot t^{3} \Big]$$
 from 0 to 850 °C: 
$$R_{t} = R_{0} \Big( 1 + A \cdot t + B \cdot t^{2} \Big)$$

$$R_{t} = R_{0} \left( 1 + A \cdot t + B \cdot t^{2} \right)$$

	A	3.90802.10 <sup>-3</sup> °C <sup>-1</sup>
ĺ	В	-5.802.10 <sup>-7</sup> °C <sup>-2</sup>
ĺ	C	-4.27350.10 <sup>-12</sup> °C <sup>-4</sup>

These EIT 90 points are available and useable both as a standard and as a generator. But to be used some needs accessories such as bath (mercury), liquid nitrogen (water)...



Figure 8 : Cells available in Météo-France, from left to right: gallium, mercury and water cells

## 3.2. Standard Platinum Resistance Thermometer (SPRT)

But it is not necessary to provide 3 or 5 cells to do temperature calibration. It is possible to use a Standard Platinum Resistance Thermometer (or SPRT, see fig 9). As it is call, this thermometer is made of platinum. And the purity of a thermometer's platinum wire is critical to meeting ITS-90 requirements. Maintaining that purity over the life of the thermometer impacts long-term stability. The tube of the SPRT should be properly sealed to prevent contamination of the platinum sensor. The tube is made of quartz glass or iron steel.

The SPRT could be available with is own high precision numerical display. But it is also possible to connect this device to a multimeter or a resistor bridge. In all cases connexion are done with a 4-wire to limit the influence of lead resistance.



Figure 9: example of an SPRT

### 3.3. Means of calibration

## Definition ([2])

A calibration is the set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material and the corresponding values realized by standards.

For temperature, calibrations are achieved thanks to generators, that create the comparing surroundings.

There are 4 types of generators:

- generators using cryogenics, with nitrogen baths, for the very cold temperatures;
- baths, from -80 to 180 °C;
- kilns for high temperatures ; (not use for meteorology calibration)
- climatic chamber.

### Baths:



Figure 10: Bath with mixing

To obtain the best stability, both special fluid and thermal block of equalization are used in a bath (see fig10).

Alcohol, water or oil are used according to the wanted range of temperature.

There are 2 types of baths: bath with overflow, especially for the liquid-in-glass thermometers or with mixing.

### Climatic chambers

Used when it is impossible to immerge the sensors in a fluid (thermohygrometer...), the calibration is made in the air inside a climatic chamber.

See Humidity for more details.

## 4. Humidity

Humidity is poorly measured in the field with typical uncertainties of 15% RH. For this reason the calibration of devices within the laboratory is not as onerous as that for pressure and temperature and the 95% uncertainty target for a calibration laboratory should be of the order of 2% RH.

Humidity calibration systems are comprised of two stages. Firstly, a stable stream or body of humidified air must be produced. Then, the humidity of the reference parcel of air must be determined. In some systems, such as two pressure or two temperature humidity generators, the devices are considered to be 'first principles' devices and the calculation of RH or dew point is derived from pressure and temperature measurements. In other systems the RH or dew point is measured, typically with a dew point hygrometer or an aspirated psychrometer.

## 4.1. Humidity Generators

"Two-pressure" humidity generation process involves saturating air with water vapor at a known temperature and pressure. The saturated high-pressure air flows from the saturater, through a pressure reducing valve, where the air is isothermally reduced to test pressure at the test temperature. System uncertainty is dependent on the accurate measurement of temperature and pressure and the stability of these measurements. The claimed uncertainty of commercially available systems is approximately 0.5 % RH. They offer good stability and reproducibility with an initial unit cost of around €60,000. These systems have a wide range of dew points from -20 oC to +50 oC.

Several manufacturers also offer Two temperature – two pressure humidity generators which extend the range of operation over that obtained from a two pressure system. The cost of these systems is considerably more and their uncertainty similar to that of two pressure systems (see fig 11).



Figure 11. Thunder Scientific Model 1200 Two Pressure Humidity Generator (left) and GE Humilab split stream system.

Alternatively, split stream humidity generators combine two air streams, one fully saturated while the other is 'dry'. Control of the ratio of the two streams determines the RH of the combined stream. Split stream systems can produce very low dew points (down to -100 oC DP) limited only by the dew point of the dry air stream. They possess good stability and reproducibility. Since flow rate is rarely a traceable measurement, the determination of dew point or RH is usually made with an associated dew point hygrometer. Typically, the uncertainty of these combined systems is 1% RH at ambient. Typically split stream systems cost €30,000 - 50,000.

These systems can produce a range of low RH suitable for the assessment of sondes.

## 4.2. Climatic Chambers

Climatic chambers (see fig 12) are divided into two types, those that control the humidity of a parcel of air by controlling the temperature of a body of water within the chamber separately from that of the chamber air temperature, and those that mix two streams of air, one saturated and one dry into the chamber. In the former, the temperature of the water body sets the dew point whilst the air temperature sets the ambient temperature. These systems have a limited range of humidities due the limited temperature range of liquid water at atmospheric pressure. Typically, 20-90% RH at 25 °C. There are known problems with temporal and spatial stability with environmental chambers. The chambers are also slow to change dew points and have long settling times, of the order of  $\frac{1}{2}$  an hour.

For systems employing a dry and saturated stream the range of RH is greater and the change in RH is faster. Typically, the measurement of chamber RH is made with an aspirated wet/dry bulb pair incorporated into the chamber. Uncertainty can be reduced by placing a dew point hygrometer in the chamber, or extracting air from the chamber for analysis by a dew point hygrometer.





Figure 12. Heraus Votsch 4030 Environmental Chamber (left), split stream chamber (right).

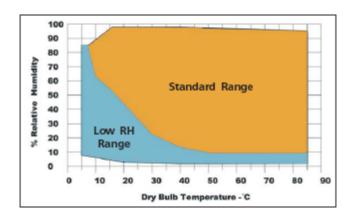


Figure 13. Typical RH versus temperature range for a climatic chamber.

It can be seen in Figure 13 that climatic chambers are suitable for producing surface level RH and are unsuited for evaluating sondes etc. Typically these systems cost £20,000 with an added dew point hygrometer costing £15,000. Typical uncertainties for these systems is 2% RH when a dew point mirror device is incorporated.

### 4.3. Salt Solutions Generator

Several NMHS employ saturated salt solutions to calibrate RH probes. The dry salt is spread about 3 mm deep in a shallow tray that occupies most of the bottom of an airtight box. Water is added to moisten the salt. The instrument is then laid on a grid supported above the tray. Electronic sensors can be inserted through a hole in the box which is made reasonably airtight with a split rubber bung. A wide range of salt solutions are available (Table 2). Their time to stabilize is poor and the solutions degrade with time.

Table 2. Nominal RH produced by various salt solutions at various temperatures [4].

Salt/Temperature °C	5.0	10.0	15.0	20.0	25.0
Lithium chloride	11.3	11.3	11.3	11.3	11.3
Magnesium chloride	33.6	33.5	33.3	33.1	32.8
Potassium carbonate	43.1	43.1	43.1	43.2	43.2
Sodium bromide	63.5	62.2	60.7	59.1	57.6
Sodium chloride	75.7	75.7	75.6	75.7	75.3
Potassium chloride	87.7	86.8	85.9	85.1	84.3
Potassium sulphate	98.5	98.2	97.9	97.6	97.3

Due to the static air parcel above the salt solution the humidity must be monitored with an impedance type humidity probe. This increases the uncertainty of the calibration. These systems are cheap, but salt consumables must be changed regularly to maintain low uncertainties and therefore costs can be high. Typical uncertainties for these systems is 5% RH for fresh salt solutions.



Figure 14. The salt solution system at Meteo France for the calibration of field humidity probes.

# 4.4. Approximate Uncertainties of Measurement techniques

The relative uncertainties of various measurement techniques are displayed in figure 15. A reference laboratory in a NMHS should have a chilled mirror device or a first principles humidity generator as a reference standard. Further decreases in uncertainty by purchasing a gravimetric device are not justified given the high uncertainty of humidity probes in the field.

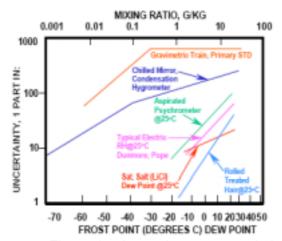


Figure 15. Approximate uncertainties of various humidity measuring techniques.

### 4.5. Recommendations

- 1. The target uncertainty for the production and measurement of humidity within a calibration laboratory should be 2 % RH or 0.3 °C in dew point.
- 2. RICs should have a humidity generator or chamber capable of generating air streams or parcels of air within the range 5 % RH at 15 °C to 90 % at 40 °C. The streams or parcels should be stable to within ± 0.2% RH per minute.
- 3. If the source of humidity is not a first principles device, then measurement of the humidity should be with a dew point hygrometer with an 95 % uncertainty of 0.2 °C or better.
- 4. Calibration of field humidity probes should be done with a view to maintaining a low overall uncertainty. The whole of measurement cycle, including inspection frequency, must be considered and appropriate levels of calibration uncertainty applied.

### 5. Conclusion

This first review of acceptable devices for metrology laboratory has been prepared to explain what are nowadays some possibilities in terms of calibration. Of course this first review is not exhaustive and represent only a small part of pressure, temperature and humidity calibration possibilities. Every other remaining devices, standard, generator not presented here are still acceptable is ever it is managed by his owner. Please remember that metrology is the science of measurement and that this science begins with the expression of the uncertainty joined with the result.

## 6. Bibliography

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