

# BEST PRACTICE FOR CHARACTERISATION OF CALIBRATION FURNACES

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**Abstract.** The CETIAT thermometry calibration laboratory has a wide range of temperature generators. Overflow baths, dry furnaces, furnaces as well as thermostatic chambers make it possible to achieve a temperature range from -90 °C to 1050 °C. Experience gained over more than ten years enables the teams to identify possible measurement difficulties. The thermal characterisation of portable furnaces is identified as risks in measurement errors. The diversity of CETIAT's calibration tools makes it possible to highlight these risks and to find solutions for them. Only a few documents deal with the characterisation of portable furnaces. The EURAMET guide "cg-13 form 3.0 (02/2015)" provides some elements. This lack of information on implementation in temperature calibration furnaces can lead to significant calibration errors. A calibration performed in a thermostated bath or dry furnace will give different results in both the correction and the associated uncertainty. The difference in the results obtained is such that the corrections do not overlap, even when taking into account the associated uncertainties. There is also the effect of the environment on the results. This study allows users of portable furnaces to reduce measurement errors due to poor implementation.

## 1 OBSERVATIONS

Sometimes, the calibration of a thermal probe requires the use of several temperature generators (thermostated baths, tubular furnaces, heat pipes, portable furnaces). This often results in discontinuity in sensor response corresponding to the change in source.

On the accuracy curve, this results in a jump which makes the analysis difficult, even if this difference is included within the measurement uncertainty. This gap in the correction of the sensor is tricky to model. This behaviour is explained by the change in the medium to which the probe is exposed. The thermal homogeneity, as well as the thermal profile to which the sheath of the sensor is subjected, is specific to each generator. We have observed this phenomenon on various generators in the course of our professional experience at different metrology laboratories.

This article aims to inform the user about these potential measurement errors relating to distinct comparison environments. The specific cases presented below will demonstrate the problem. They will serve as supports for the analysis of the effects encountered. Then, we suggest some elements of best practice.

## 2 The technological elements

### 2.1 Presentation of a portable calibration furnace

A dry calibration furnace is a temperature generator composed of a heating element, a bushing (or thermal equalisation block) and a regulation system. Various types of dry furnaces are shown in the picture (Figure 1).



Fig. 1. Photographs of different types of furnaces

The bushing consists of a thermal equalisation block into which the probes are introduced. This is the element which allows the transfer of heat (or cold) to the sensitive element of the probe. It is generally circular and pierced with borings for placing the sensors to be calibrated. Figure 2 shows on the left the first furnace made up of four bushings of different diameters, with only the bushing in the centre being removable. The centre furnace has a removable six-well bushing. The image on the right shows eight bushings with various boring configurations.

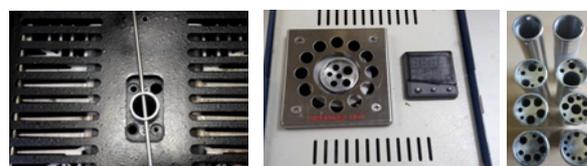


Fig. 2. Photographs of bushings for portable furnaces

The greater the thermal mass, the more thermally stable the bushing, but the stabilisation time will be longer. Responsibility for the optimisation of these effects depends on the manufacturer's expertise. The diameter and length of the bushing will depend on the portability of the furnace and its performance. However, the dimensions of the bushing must be adapted to those of

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the probes to be calibrated in order to minimize parasitic effects. Unfortunately, the probe to be calibrated is often inserted into a bushing of an unsuitable diameter due to a lack of availability.

Several heating technologies exist. The best furnaces have several thermal zones. Their locations and numbers will ensure the correct homogeneity of the bushing. Sometimes, a control probe is directly inserted into the bushing, allowing more precise control of the temperature. The supply of heat or cold is generated by resistive elements, Peltier coolers, or Stirling coolers etc.

For portability, dry calibration furnaces often have relatively short bushings. The bushings available on the market offer lengths varying from 80 to 200 mm. Its dimensions are compared to the 400 mm of immersion generally used in an overflow bath. The difference in immersion between these two types of temperature generators is the source of the jumps in response. For example, between a bath and a furnace, the sheath of the sensor is exposed to different thermal environments, leading to distinct responses for the same reference temperature.

## 2.2 Presentation of a temperature probe

A temperature probe is a system that provides an electrical value proportional to the temperature. It is generally composed of a sensitive element (Pt100, thermocouple, thermistor), a protective sheath (metal) and connecting wires. Figure 3 shows an example of a jacketed probe with its connection.



Fig. 3. Photograph of a sheathed temperature probe

Figure 4 shows diagrammatically the different effects to which the sensitive element of the probe is subjected. To understand the response errors of the sensors placed in different furnaces, it is necessary to know the various parasitic effects.

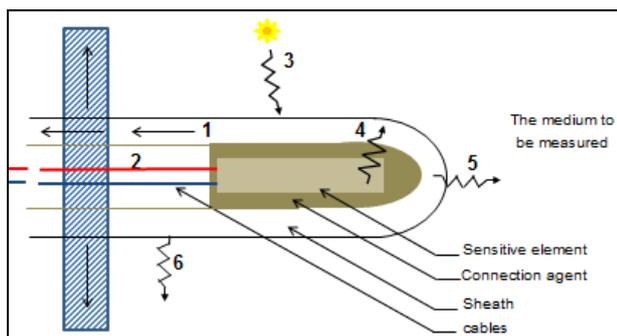


Fig. 4. Parasitic effects of a temperature probe

Marker 1 indicates the conductive flow along the sheath. This effect can be dominant, depending on the conductivity and geometry of the sheath. Losses by conduction in the wires are marked at 2. This is to be taken into account for negative temperatures. The effect of external radiation is identified at 3. It depends on the technology of the furnace, but it will appear, especially at high temperatures. Marker 4 identifies the effect of the insulation between the sensitive element and the sheath. This thermal resistance is related to the electrical insulation of the sensitive element, this depends on the manufacturing technology. Marker 5 shows the losses by convection from the sheath. Marker 6 shows the radiation of the sensor towards the medium to be measured.

Do not forget that the temperature sensor measures its own temperature. The temperature of the medium to be measured must be as close as possible to that of the sensitive element. The thermal coupling between the probe and the environment to be measured must be the best possible. The measurement must be carried out in a steady state. The less these elements are respected, the greater the measurement error will be.

## 3 The metrological elements

### 3.1 Presentation of specific examples

This section provides examples of industrial calibrations. The objective is to illustrate the immersion effect of a probe in a temperature generator to facilitate understanding and to evaluate the biases obtained. Please note that the deviations provided here are dependent on the sensors used, the generator and the environment. The corrections are intrinsic to the characteristics of each sensor. These examples will allow the reader to assess the risk of error due to the conditions of implementation.

Figure 5 shows an example of a calibration curve of a four-wire, jacketed, platinum, Pt100-type resistance probe, which has a diameter of 6 mm and a length of 400 mm. Calibration was carried out over seven years in a thermostated bath up to 150 °C and then in a furnace at 250 °C. The respective immersions of the bath and of the furnace are 390 mm and 190 mm.

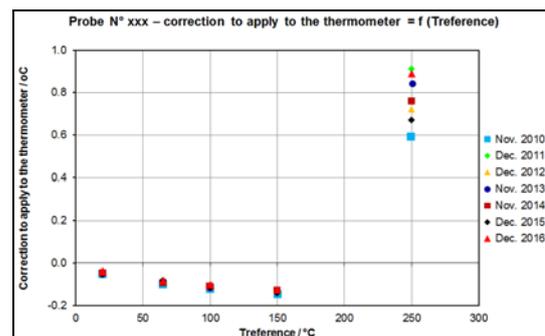


Fig. 5. Calibration history of a platinum probe

We can observe the difference between the response of the probe calibrated in a bath (from 30 °C to 150 °C) and in a furnace at 200 °C. In the bath, the accuracy error is reproducible, whereas in the furnace the correction becomes more significant and unstable over different years. This behaviour can be explained by thermal effects due to immersion of the probe.

Figure 6 shows an example of a calibration curve of a jacketed, T-type, thermoelectric couple with a 2 mm diameter over a 2 m length. The calibration was carried out over seven years in an overflow bath up to 200 °C and then in a furnace at 300 °C. The bath and furnace immersions were 390 mm and 270 mm respectively.

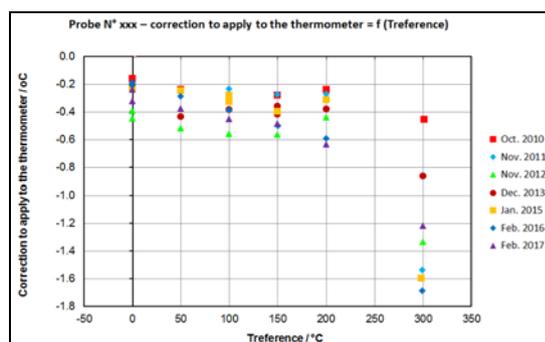


Fig. 6. Calibration history of a thermocouple

We observe a difference in the behaviour of the thermoelectric couple between the range of 0 °C to 200 °C and the set point of 300 °C. This evolution corresponds to the change in the temperature generator. In the bath, the response of the thermoelectric couple is relatively stable (0.4 °C), but it becomes random in the furnace (1.4 °C). This may be due to an immersion effect coupled with the heterogeneity of the sensor.

### 3.2 Analysis of the results

The calibration examples presented show a discontinuity in the evolution of the calibration correction corresponding to the change in temperature generator. Although it is generally included in the measurement uncertainty, this phenomenon induces a difficulty in modelling the sensor response. This modelling is part of the second step of a calibration as recommended in definition 2.39 (6.11) of the JCGM 200:2012 (VIM).

For identical temperatures, the measurement error depends on the geometry of the probe and on the exposure medium. The difference is not the same for varying sensors introduced into the same generator. The quantification of this error is not possible in advance; it depends both on the furnace and the jacketing of the sensor (length, diameter, material).

This effect is also found on calibration baths with different immersions. The explanation stands mainly from the type of sensor used and the conditions of its implementation.

On some calibration histories the response may be continuous despite a change in heat source. It is difficult to predict the behaviour of a probe subjected to different heat generations. Generally, the spread of the responses to a change of furnace is properly taken into account in the estimation of measurement uncertainties. In other words, changing the comparison medium to one with less efficient thermal characteristics will not only increase the measurement uncertainty but also the spread of the results.

In these examples, we can observe that there is an effect related to the temperature generators used. The response error can be attributed to differences in the characteristics of the generators, to the difference in the immersion of the sensor, or to the thermal coupling between the probe and the generator.

This last point is too often overlooked. In a bath, the thermal coupling is optimal because the heat transfer fluid is in direct contact with the sheath of the sensor. In a dry furnace it is relative, because between the diameter of the sheath and the boring there must be clearance to allow the introduction of the sensor. From then on, an air gap is created in this space thus introducing a thermal resistance.

Care should be taken to minimize this air gap between the bushing and the sheath. The EURAMET cg-13 Version 3.0 (02/2015) Calibration of Temperature Block Calibrators recommends a clearance of 0.5 mm (for  $t < 660$  °C) between the diameter of the boring and that of the probe sheath. This recommendation is important to ensure a thermal coupling of the sensitive element with the comparison medium.

The immersion of the sensor is an important explanatory element. If the sensitive element of the probe is not sufficiently immersed in the temperature generator, then the parasitic effects of conduction, convection or even radiation will disrupt the measurement.

The best practice provided in this article for the use of portable calibration furnaces can also be used for calibrations in other types of temperature generators (baths, heat pipes, etc.).

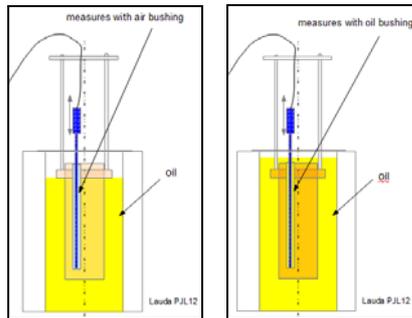
## 4 Elements of explanation

### 4.1 Effect thermal coupling

To evaluate the effect of thermal coupling, we performed tests using a bushing immersed in a thermostated bath. The immersion of the bushing was adjustable. A probe positioning system was also used to vary the immersion of the probes in the borings of the bushing.

The diagrams (in Figure 7) show the two configurations tested. To the left the borings of the bushing are in the air, on the right the oil is flush with the top of the

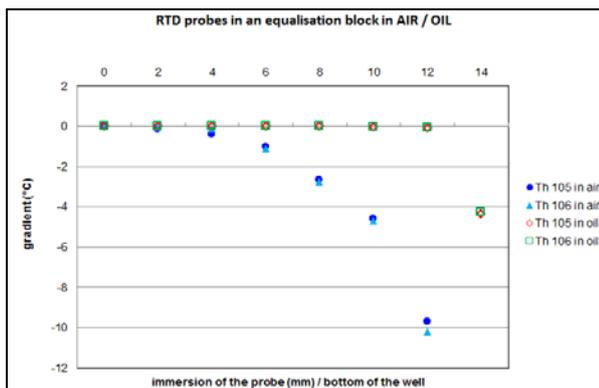
equalisation block of which the borings are filled with silicone oil. The oil allows good thermal coupling.



**Fig. 7.** Diagrams of the two configurations of tests of thermal coupling

The probes have a diameter of 3 mm and a length of 300 mm. The internal diameter of the borings is 6 mm.

The thickness of the air gap at the diameter is 3 mm. This configuration allows the evaluation of the thermal coupling between the probe and the bushing. Figure 8 shows the impact of the air interface relative to that of silicone oil.

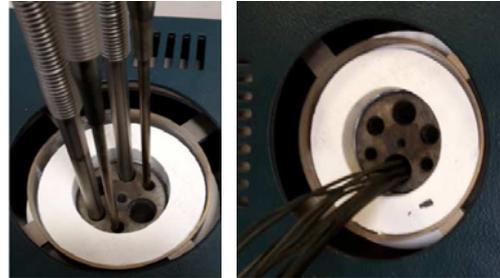


**Fig. 8.** Graphic of the effect of thermal coupling in oil or air

Despite the small diameter of the probes tested, Figure 8 shows two distinct responses. For maximum immersion up to shrinkage of 4 cm the impact of the heat transfer fluid is negligible. Beyond this, the impact of the air gap is visible with a bias of 5 °C for shrinkage of 50 mm. It is therefore important to adjust the diameter of the probe as closely as possible to that of the bushing boring. The optimum configuration is the maximum immersion of the probe in the bushing along its length and in an adjusted boring. Unfortunately, too often the sheath of the probe is twisted, or the bushing does not have the right diameter, or the bushing is too short.

The photographs (Figure 9) present typical cases encountered. The bushing does not have enough suitable borings (left picture). The response of the sensors may be dependent on their position in the equalisation block borings, as a function of the quality of the thermal coupling. In addition, in this case, the furnace will have difficulty in regulating properly due to the thermal pumping induced by the large number of probes in relation to the thermal mass of the bushing.

In the picture on the right, a large number of probes are introduced into the same boring. Since the sensors are twisted, the contact between the ducts and the bushing leaves too much room for air.



**Fig. 9.** Photographs of the calibration configuration

In both photographs the thermal coupling between the sheath(s) and the bushing is not correct. The free space favours the creation of an air gap, which is the source of thermal resistance. The measurement will be biased. The shorter the bushing is in relation to the sheath, the more parasitic effects will be visible.

In a dry furnace, when the diameter of the boring is too large compared to that of the probe (greater than 0.5 mm), one solution consists of reducing the air gap, either by making an over-sheath (Figure 10), or by adding tubes of conductive material (copper) (Figure 11) or a heat transfer fluid (silicone oil etc.). The choice of solution depends on the calibration programme and should be characterised.



**Fig. 10.** Photograph of an over-sheath



**Fig. 11.** Photograph showing the use of a copper tube.

In Figure 10, the 50 mm white cable sensor is introduced into a metallic over-sheath fitted to the boring of the bushing. This is the *de luxe* solution. Figure 11 shows an bushing with the insertion of copper tubes to replace the air. In this case, the difficulty is to find the tubes of adequate diameter and thickness in order to reduce the volume of air.

#### 4.2 Immersion effect

To evaluate the effect of immersion we carried out tests using a thermostated overflow bath and a furnace having

a 150 mm bushing. Two types of probes have been used to cover a wide range of sensors. One probe is chosen for its large exchange surface and another for more common dimensions. The sheaths are introduced into borings of adjusted diameter, in order to maximize the thermal coupling.

A series of tests is carried out with a 4-wire, metal, sheathed RTD (Pt100) type probe with a diameter of 6 mm and a length of 500 mm. Figure 12 shows the photograph of the assembly. The probe is immersed in the 190 mm furnace (150 mm in the aluminium bushing). We can observe from the photograph that a large part of the sheath is in contact with the environment. The exchange area over a length of 310 mm is approximately 60 cm<sup>2</sup>. This configuration will make it possible to demonstrate the influence of the environmental conditions and the immersion effect on the differences found between the reference probe and the test probe (calibration corrections).

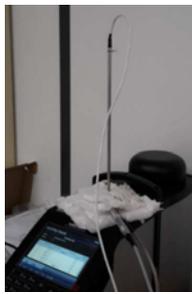


Fig. 12. Photograph showing the installation of the probe in the portable furnace.

The first tests allow us to identify the temperatures of interest. Over the temperature range of -40 °C to 90 °C, the effect of immersion is more significant for low temperatures.

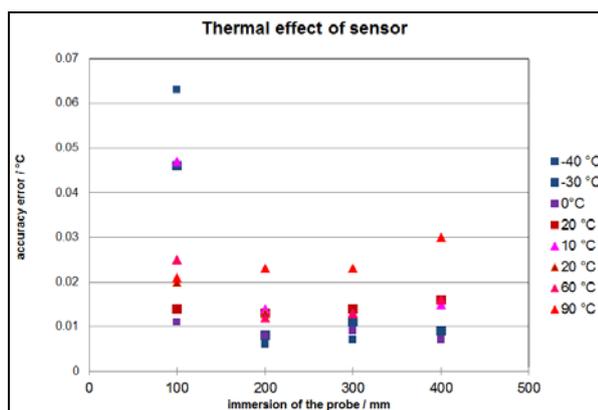


Fig. 12. Graph showing the immersion effect of a sensor

A slight immersion effect on accuracy errors is observed. On the other hand, the errors are proportional to the temperature. The lower the temperature, the greater the measurement error for an immersion of 100 mm. Let us continue our investigations into low temperatures.

We carried out tests with the same probe in two different configurations. Firstly, the probe is placed in an overflow bath with two different immersions; then, it is

positioned at the same immersion in different generators. These two tests allow the analysis of the effects of thermal coupling and immersion. Unfortunately, the bath cannot generate temperatures below -80 °C. Figure 13 shows the results.

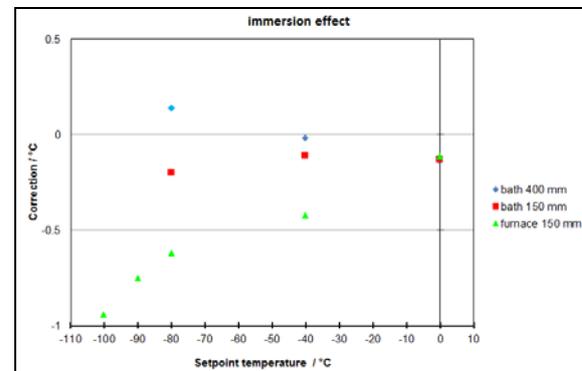


Fig. 13. Graph showing immersion effect on a large probe

In this graph, we obtain three different responses for temperatures from -100 °C to 0 °C. As the temperature decreases, the more the difference between responses increases. The observed correction varies from 0.02 °C to 0.94 °C for -100 °C. At 0 °C the different configurations have little impact.

The best thermal coupling is obtained with immersion in a thermostated bath. This configuration makes it possible to test the immersion effect. The response for immersion of 400 mm is linear for the lowest values. For that of 150 mm the response is shifted towards negative corrections. The heat is supplied via the sheath, the exchange area of which is not negligible (approximately 60 cm<sup>2</sup>). The fin effect along the sheath and the conduction promotes the transit of the heat flow towards the sensitive element. The correction measured at -80 °C is 0.14 °C for an immersion of 400 mm (blue diamonds), against -0.2 °C at a submersion of 150 mm (red squares). This represents a difference of 0.34 °C between the two configurations. This error is mainly due to immersion of the sensor.

The effect of thermal coupling is evaluated by comparing two different generators for the same immersion. Since the useful length of the furnace bushing is 150 mm, this immersion is chosen. For this immersion, the bath measurements (red squares) have lower corrections than those in the furnace (green triangles). This shows that for the same immersion, the difference obtained with the reference probe is stronger in the furnace. This result can be explained by the poorer thermal coupling in the furnace. In a bath, the probe is dipped directly into a comparison medium which has a very good heat homogeneity. In the furnace, there is necessarily some clearance between the sheath of the probe and the boring of the bushing. There is a thermal discontinuity from the air that creates a buffer zone. This error is mainly due to thermal coupling.

Other tests were carried out with more usual probes of 3 mm diameter and length of 300 mm. For this study, we compared different types of sensors (Pt100 and type N

TC) introduced into three different types of furnaces. These furnaces had bushings of different lengths (150, 170 and 190 mm). In parallel, these probes were calibrated in an overflow bath for five different immersions (130, 150, 170, 190 and 300 mm). In Figures 12 and 13 we find the same type of results. The measurement error increases inversely to the temperature.

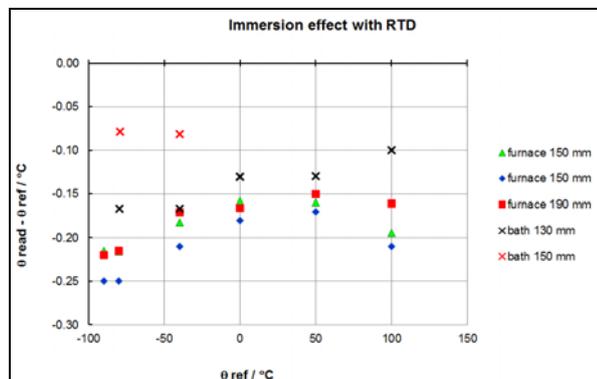


Fig. 14. Graph showing immersion effect of a RTD probe

The results in Figure 12 come from a Pt100-type probe introduced firstly into two different immersion furnaces and then immersed in an overflow bath two-immersion. For negative bath temperatures, the corrections are lower for the maximum immersion (150 mm). The sensor response for a 130 mm immersion is close to that of the furnace at 190 mm.

The corrections obtained in the furnace for 150 mm are the highest absolute values for it. For a Pt100 probe with a diameter of 3 mm and a length of 300 mm, the previous results with lower values are obtained, because the exchange surface area is smaller.

Figure 13 shows the results for an N-type thermoelectric couple with the same sheath dimensions as for the Pt100. These tests were carried out on three different furnaces and in an overflow bath for five immersions of the probe. It was observed that the greater the furnace immersion, the better the thermal coupling. The corrections found are the lowest in absolute values. For measurements in the bath at  $-80\text{ }^\circ\text{C}$ , a difference is noticed between total immersion (300 mm) and the other immersions tested. This effect is certainly due to the heterogeneity of the sensor which coincides with the immersion effect.

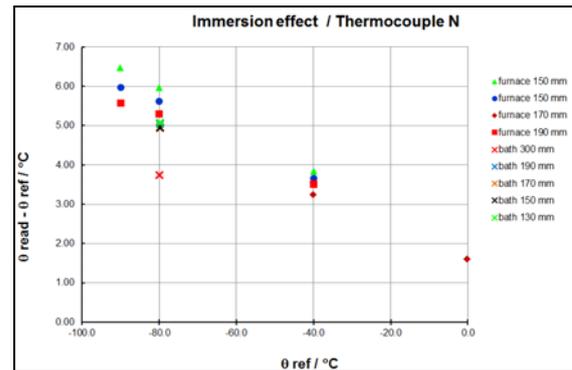


Fig. 15. Graph showing immersion effect of a thermocouple probe

## 5 Best practice conclusion

To conclude, we have shown the importance of a good thermal coupling between the jacketed probe and the bushing of a dry furnace. The greater the immersion of the sensor sheath, the more accurate the response will be.

We can restate here an empirical rule, but which gives good results, which consists of having an immersion of at least 10 times the length of the sensitive element. For a coiled platinum resistance probe with a sensitive element measuring approximately 10 mm, the immersion length will be at least 100 mm.

Care should be taken to ensure that the whole sheath is immersed as far as possible in the medium to be measured. The thermal coupling between the medium to be measured and the probe must be optimised in order to limit the presence of air or insulating materials. Heat transfer fluid or conductive materials (over-sheath) may be used.

We recommend that you calibrate the probes at the same immersion in the temperature generators in order to obtain a continuous response. This does not prevent the implementation of complementary measures for the best immersions.

The importance of the immersion in the temperature generator should not be forgotten. The more that the medium for comparison is thermally homogeneous, stable and sufficiently long to submerge the sensor sheath completely, the better the quality of the sensor response. Unfortunately, these criteria are often in the conflict with requirements of portable furnaces!

## References

1. JCGM 200: 2012 International vocabulary of metrology – Basic and general concepts and associated terms (VIM) 3rd edition
2. Calibration of Temperature Block Calibrators EURAMET cg-13 Version 3.0 (02/2015) Previously EA-10/13