WATER THERMOSTATIC BATH TO COMPARE GALLIUM CELLS

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ABSTRACT

In general, gallium cells can be realised in any water thermostatic bath, however, some manufactures have developed air furnaces or heat-cooling ovens (with peltier cells and heating resistors) to avoid mechanic vibrations, electromagnetic interference, and to allow for easier and dedicated operation mode. Generally, all of these devices are dedicated and they are used with only one cell. As we want to compare two different gallium cells, we have developed a water thermostatic bath, which allows realising simultaneously two or more gallium melting points, with cells up to 50 cm long.

1. INTRODUCTION

The gallium melting point is one of seventeen definition fixed points of the International Temperature Scale of 1990 (ITS-90) (1). Between the triple point of argon (-189,3442°C) and the freezing point of silver (961,78°C) the ITS-90 is based in interpolation equations applied to standard platinum resistance thermometers (SPRT's), in which a resistance ratio $W = R(T)/R(0{,}01°C)$ is obtained by measuring the resistance at the temperature T and at 0,01°C (water triple point). The resistance ratio $W = R(T)/R(0{,}01°C)$ at the definition temperature of gallium melting point (29,7646°C) is used as an ITS-90 criteria. This criteria defines a SPRT as interpolation standard in the Scale, where $W \geq 1{,}11807$. Thus, it's important to realise the gallium melting point in appropriate devices. There are different baths and furnaces to perform it. The peltier cell ovens are the most recent and modern apparatus to realise the gallium fixed point. They are made for only one cell with defined dimensions and there are gallium cells with different sizes.

In the comparison between cells it is interesting to keep them in the same thermal enclosure. It is more practical and it defines the temperature of the process, i.e., in this case the duration of the gallium melting point. During the melting of gallium, the main problems are: electromagnetic interference, mechanical vibrations, and failure in temperature control, temperature instability and thermal gradient. It will be described how these problems were treated, as well as thermal stability and gradient tests, measurement equipment, bath dimensions etc.

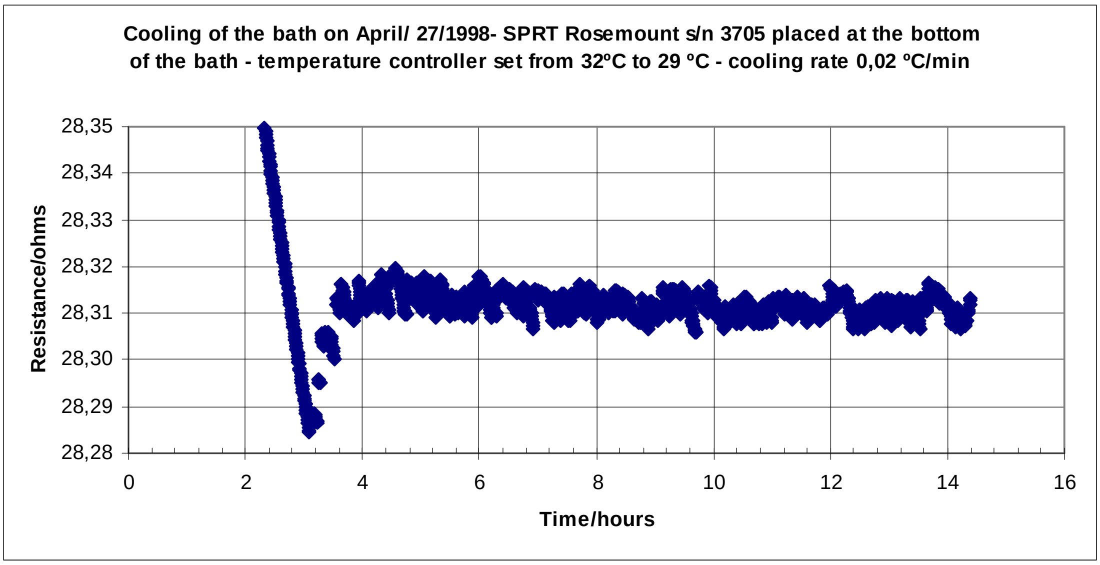
2. STRUCTURE, FACILITIES AND DIMENSIONS OF THE BATH

This thermostatic bath is based in a glass vase filled with water stirred by an air pump. The air is pumped through plastic tubes, bubbled in the periphery with softened vibrations. This is obtained by passing the air through a plastic chamber (a recipient) before it goes into the vase. The heating is done by a heating element of 175 W controlled by electronic circuits (one circuit to control and another to cut-out).

The main PID electronic circuit commands a tiristor (triac). An electronic circuit with PWM technique, which permits the elimination of spikes in the power line, switches the tiristor. It was also possible to eliminate electrical noises and interference, keeping grounded the external metallic structure of the heating resistor.

The main sensor of the PID electronic circuit is an industrial platinum resistance thermometer with nominal value 100Ω (pt-100) and the sensor of the cut-out circuit is a transistor, which activates an electric relay if the temperature reaches 32,5° — in this circuit the histeresis phenomena occurs — allowing for the bath to be turned-off for one hour or more (see graph 1).

Graph 1

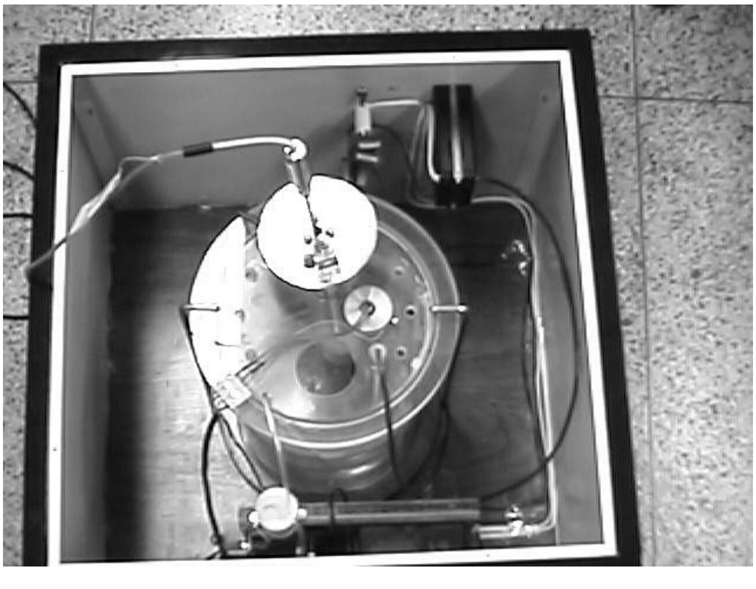


The vase, controls and other circuits are mounted in a metallic cabinet with 4 wheels and on the metallic surface there are holes for cells and six pre-heating wells, allowing for the realisation and comparison of at least two gallium melting points in the same device .On the top of the glass vase there is an acrylic cap with eight holes: six holes for pre-heating wells and two to receive the cells. (see picture 1) — the vase dimensions are: diameter : 225 mm, depth: 380 mm.

Picture 1

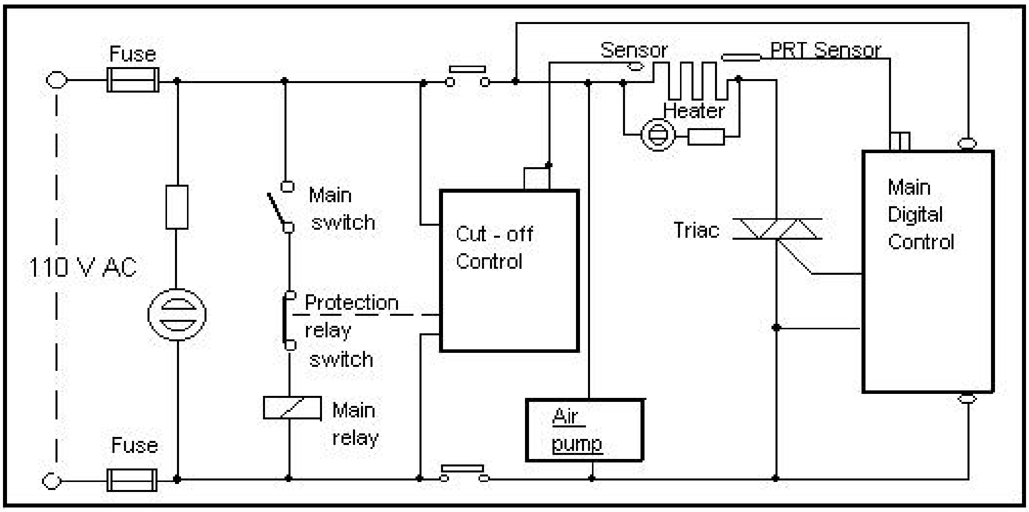
The bath is mounted into a metallic box with the following dimensions: height: 850 mm, width: 600 mm and length:600 mm. The main controller, the indicator lamps and the power switch are localised on the instrument frontal panel (see picture 2).

Picture 2

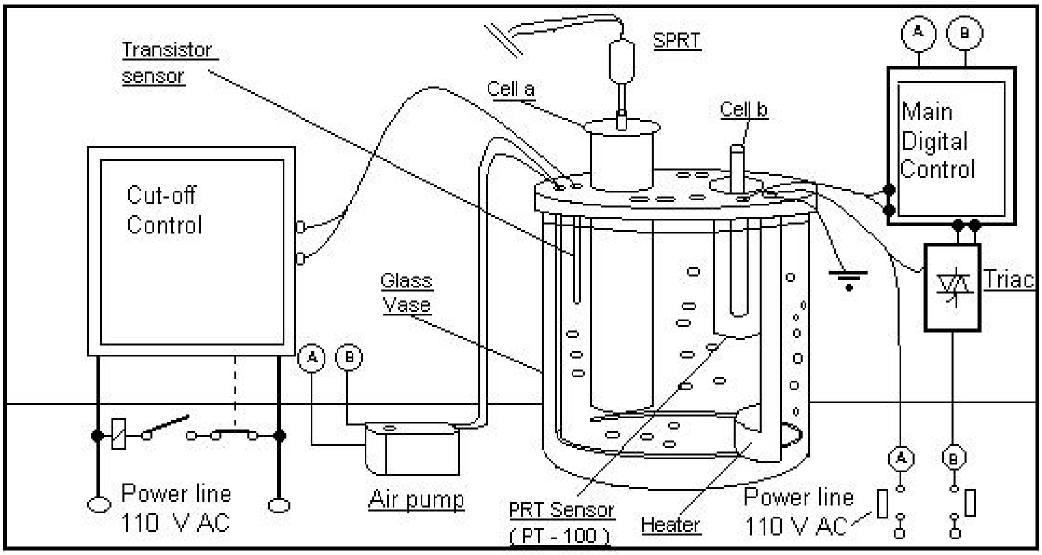


The schematic circuits are shown in two figures: the first one (picture 3) includes the electrical circuit diagram and the second figure (picture 4) is the drawing with electrical connections, circuits and main structure of the water thermostatic bath.

Picture 3



Picture 4



3. MEASURING EQUIPMENT TO TEST THE BATH

The measurements were perform initially (in 1997) with 6 Liquid-in-glass thermometers (subdivision = 0,1°C), manufactured by Thermoschneider (Germany), scale: -4°C to 38,5°C. In 1998, an AC Bridge from Automatic System Laboratory, model F18 with a standard resistor from H.Tinsley (100Ω s/n 236063) immersed in a controlled oil bath Guildline model 9732 VT was used. During the measurements the temperature in oil was 20,01°C ±0,01°C. The monitoring sensor was the SPRT from Rosemount model 162 CE, s/n 3705.

4. METHODOLOGY AND RESULTS

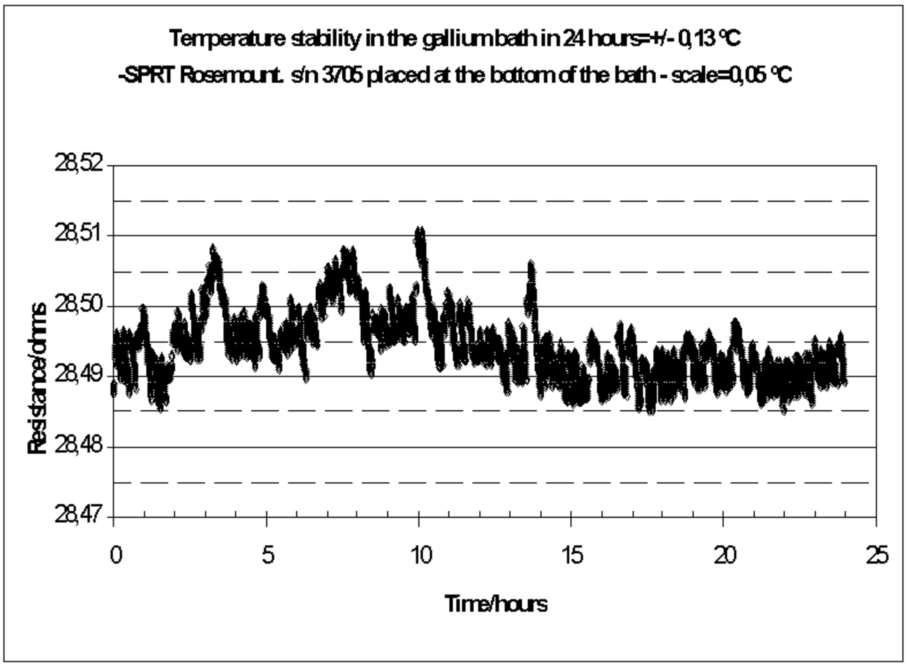
Six liquid-in-glass-thermometers were distributed to account for the stability and thermal gradient along the bath. Four thermometers were positioned close to the bottom of the vase (two thermometers were near the heating resistor) and two were positioned 4 centimetres bellow the water surface. The maximum difference found between the six thermometers was 0,10°C and the instability during 8 hours was 0,14°C.

All the liquid-in-glass thermometers were calibrated with uncertainties of ±0,03°C (k=2). Thus, we can calculate the expanded uncertainty of this measurements as *U* = ±0,20°C (k=2).

The average difference between the set-point of the electronic controller and the average temperature measured by the standard thermometers was 0,5°C.

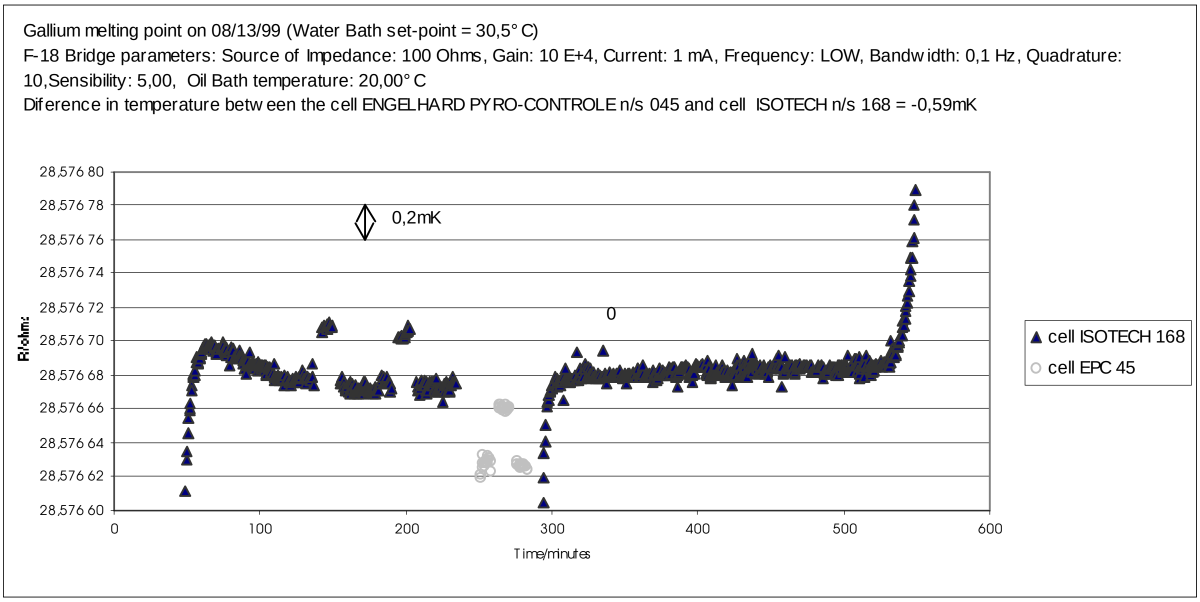
The SPRT 3705 was used to account for the bath stability at the bottom for 24 hours. The results are in graph 2.

Graph 2



The graph 3 show a comparison between two gallium cells performed in the water thermostatic bath. The melting point in the reference cell (ISOTECH 168) was realised in 10 hours approximately.

Graph 3


Gallium melting point on 08/13/99 (Water Bath set-point = 30,5° C)
F-18 Bridge parameters: Source of Impedance: 100 Ohms, Gain: 10 E+4, Current: 1 mA, Frequency: LOW, Bandwidth: 0,1 Hz, Quadrature: 10,Sensibility: 5,00, Oil Bath temperature: 20,00° C
Diference in temperature between the cell ENGELHARD PYRO-CONTROLE n/s 045 and cell ISOTECH n/s 168 = -0,59mK

5. CONCLUSIONS

The temperature of the gallium melting point is 29,7646°C and with the controller set-point adjusted to 30,0°C, an average temperature of 30,5°C ±0,2°C is obtained (including the instability and the thermal gradient (thermal non-homogeneity)). This is sufficient to perform the gallium melting point during 10 hours or more (it depends on the cell and the set-point temperature, of course).

It is possible to perform 2 or more gallium melting points in this same glass — it is necessary to change the caps and the heating resistor.

Due to the use of a air pump, it is not necessary to use a criostatic bath to refrigerate the water, because the stirring made by the helix of a motor increases the temperature up to approximately 35°C.

In case of failure in the main electronic control or a short-circuit in the tiristor, the cut-out circuit turns-off the power line when the temperature is approximately 32,5°C. Only 2 hours later the circuit is turned on. Thus, during this time, the melting of gallium continues to be performed.

It is possible to improve this bath changing the main electronic control (an industrial type) to other more accurate and the glass vase to a stainless steel vase to install the heating resistor along the external surface of the vase.

Apart from the technical advantages mentioned above, this device, due to its simplified construction, also proved to be cost saving (less than US$ 600.00, concerning only to the used materials)

REFERENCES

(1) Preston Thomas H., Quinn T.J. and Bloenbergen P., Supplementary Information for the International Temperature Scale of 1990, BIPM, 1990, pp 36..43

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