How accurate are temperature measurements in the ProboStat™? The influence of feedthrough junctions.

Application note
Update: 09 December 2013

Introduction

The ProboStat™ is normally equipped with one or more thermocouples to control and/or monitor the temperature close to the sample. The thermocouple legs are soldered onto mini-contacts that are connected to the electrical feedthroughs at the base unit of the cell. These feedthrough contacts are in turn soldered to compensation wires, and these are fed out of the base unit using proper thermocouple sockets with screw terminals.

One may suspect that this scheme can create thermoelectric voltages over the junctions, and that these may not cancel out in the readings of a thermocouple, especially if there are temperature differences between or through the feedthroughs. This situation is likely to worsen for instance when the base unit gets warm by heat radiation from the hot zone and is cooled by ambient air or water cooling.

In order to evaluate this, we investigated the difference between thermocouple readings taken through the feedthroughs as compared to a reference thermocouple fed out of the cell without the junctions. This study also gives an overview of typical temperatures in the cold part of the cell (top of the base unit) under various conditions.

Method

For the test we used a ProboStat™ version A-5 with thermocouple feedthroughs of type S, but with some feedthrough holes left open so as to enable that thermocouples could also be let out of the cell without junctions.

The thermocouple to be examined was of type S, manufactured using thermocouple quality Pt and Pt10Rh wires (from K.A. Rasmussen, Hamar, Norway) and a 50 cm, double bore, capillary alumina tube, as described in the ProboStat™ manual for a control thermocouple (TCC) of standard length. We connected the thermocouple to the appropriate electrical feedthroughs in the base unit of the ProboStat™. For comparison we used a thermocouple of type S manufactured in the same way and from the same batch of wires, however *not* soldered onto mini-contacts, but let out of the cell through the open feedthrough holes. It was directly connected to a temperature indicator. Thermal gradients in the ProboStat™ were thus assumed not to influence the readings of this reference thermocouple.

The tips of the test and the reference thermocouple were fixed side by side to the support tube at typical sample height inside the ProboStat™.

Additionally, a third thermocouple placed in the lower part of the cell monitored the temperature in the bottom of the cell, just above the rim of electrical feedthroughs in the outer chamber. This thermocouple was of type K and was also directly fed out of holes in the base unit and directly connected to a temperature indicator.

The test thermocouple controlled the furnace through a Eurotherm 2216i temperature controller, which also served as one temperature monitor. For the reference thermocouple and the thermocouple placed in the lower part of the ProboStat™, the temperatures were read by two Eurotherm 902P units. The resolution of all of the temperature reading units was 1°C. Figure 1 shows the setup and the electrical connections of the thermocouples.

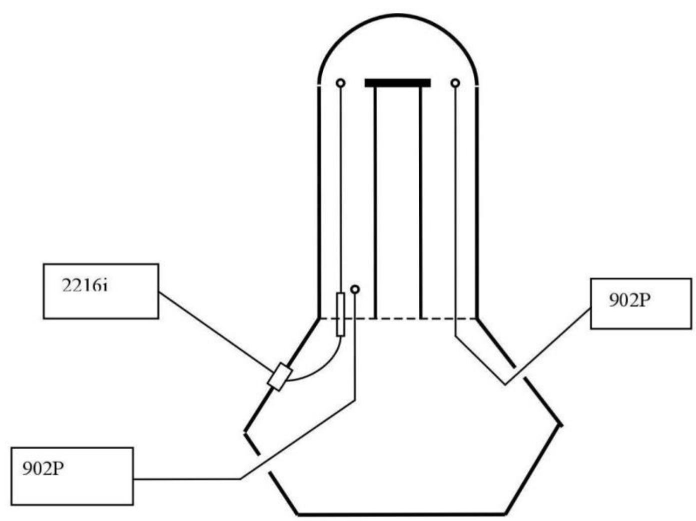

Figure 1. *Setup for testing of the influence of the feedthrough junctions. The test thermocouple under examination controlled the temperature (Eurotherm 2216i). The reference thermocouple and the thermocouple placed in the lower part of the cell were connected to two temperature reading monitors (Eurotherm 902P).*

The 1500 W tube furnace used was 50 cm long and with a working diameter of 40 mm. It was powered through a solid state thyristor.

To vary the temperature in the cold part and in turn the thermal gradient over the feedthroughs, the cell was fixed in two different heights in the furnace. At the lower position, the distance from the furnace to the outer tube flange was approximately 11 cm and at the upper position only 1 cm. Furthermore, the cooling water, entering with approximately ambient temperature, was either on or off.

The setting of the controller and thus the temperature of the test thermocouple was varied in the range 250 – 1000°C. It may be noted that the temperature controller has no problem of maintaining a stable temperature even when controlled from the internal cell thermocouple at temperatures as low as 250°C and lower. Following all changes in conditions, sufficient time was given to assure thermal steady state conditions throughout the whole cell, monitored in particular by means of the thermocouple in the lower part of the ProboStat™. Typically, more than two hours was required to reach thermal steady state in the cold zone.

Results

Temperatures in the cold zone of the cell

Cell position in the furnace	Water cooling	Temperature in cold zone, °C		
		Sample temperature		
		500°C	750°C	1000°C
Upper	No	47	76	Not established
	Yes	40	50	84
Lower	No	35	47	Not established
	Yes	35	Not established	55

Table 1. *Typical temperatures measured in the lower part of the cell under various conditions.*

Effect of feedthrough junctions

From monitoring the difference between the test and reference thermocouples, we find that the difference never exceeded 2°C at any temperature, and was mostly 0-1°C, regardless of position of the cell or whether or not cooling water was running.

This is thus largely independent of the temperature and gradients in the cold zone and base unit.

Conclusion

1. The temperatures in the cold zone - at the base unit and feedthroughs – range typically from 30 to 90°C under normal operating conditions. They may be expected to get higher at higher sample temperatures, especially if the base unit is close to the furnace opening or if cooling water is not used or has stopped running. It will also be higher on the pedestal inside the support tube than at the outer ring as measured here. The parts in the base unit exposed to these temperatures tolerate 200°C so there is much to go on.

2. These temperatures do not impose significant thermoelectric offsets through the feedthrough junctions used for thermocouples.

These conclusions apply to the outer chamber feedthroughs and to the normal operating range of type S thermocouples and to an accuracy range of 1°C. However, they indicate that the feedthroughs and connections used in the ProboStat™ are not causing significant offsets to temperature readings also on a broader basis. One may from this assume that calibration and ageing of thermocouples are more important issues for temperature accuracy than the junctions of the ProboStat™.