Technical Note

Melting Point Apparatus - MPD -2 and MPD -3

The most important specification of the Melting Point Apparatus is the accuracy of the apparatus over a wide range of the temperature. The temperature of the samples in a capillary tube must be detected and showed in the display accurately. The temperature value displayed in the machine relies on the sensor accuracy, the location of the sensor, and the heat transfer rate.

The MPD-2 and MPD-3 use an aluminum column for the heating compartment and the two heating elements directly heat the aluminum column for better control of the temperature. The sensor employed is a PT 100 sealed inside stainless steel tubes and screwed tightly against the chamber. The location of the sensor is 3 mm underneath the capillary tube location to minimize the difference between the temperature of the inside capillary and that of the machine (see Figure 1)

Our experimental data showed that the MPD-2 and MPD-3 have very good accuracy (<±0.5 °C) ranging from 35°C to 400°C when using a less than 5°C/minute ramping rate. The good linearity between the display and real temperature gives us a chance to do the simple calibration for the machine if necessary.

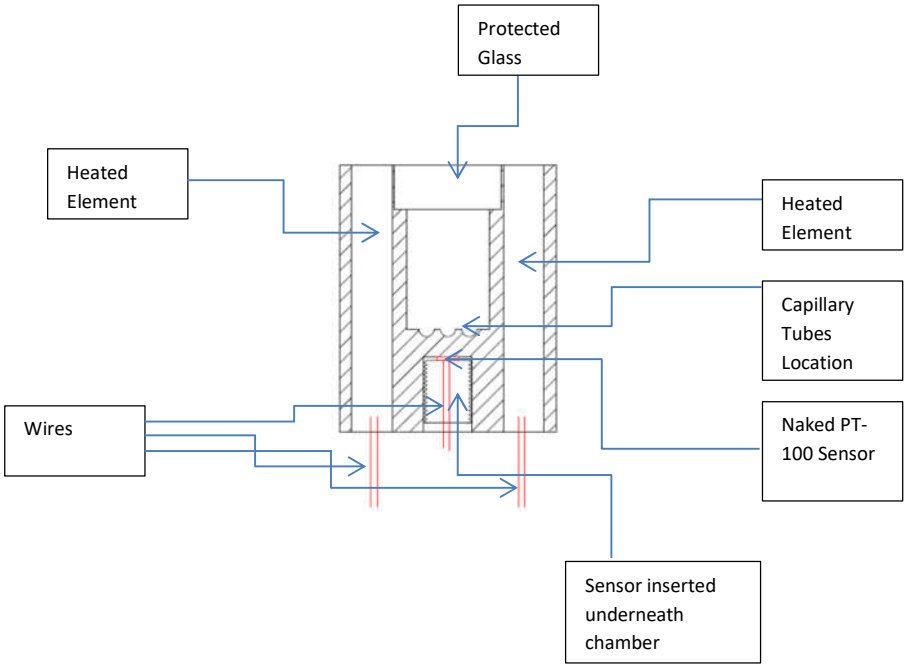

Figure 1 the Structure of the Heating Compartment

Method to monitor the real temperature of the sample capillary tube:

Instrument: Fluke 51-II Thermometer

Sensor: Type of TT-K-36 is inserted into the capillary tube with thermal compound (see Figure 2)

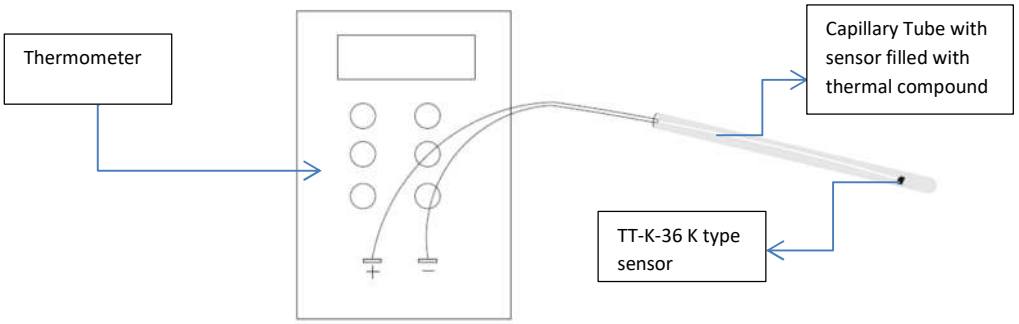

Figure 2 Thermometer with TT-K-36 sensor

Results:

1. There is very good linearity between the display and real temperature ranging from 35°C to 400°C (R^2=1, see Figure 3).
2. The maximum difference between the display and real temperature is + 0.4°C and the minimum difference is -0.2°C.

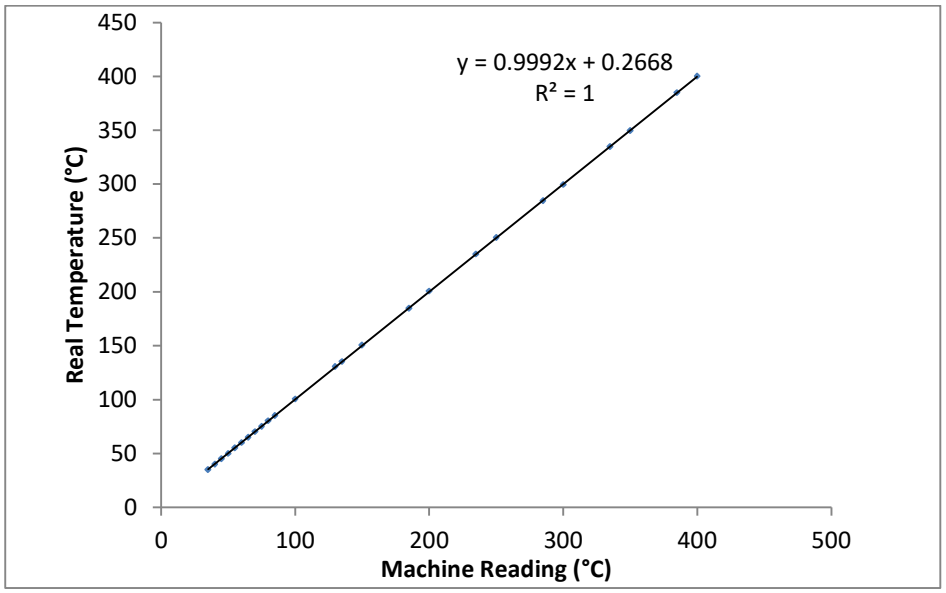

Figure 3 the Accuracy of the MPD-3 ranging from 35 - 400°C

3. When the ramping rate is high, the instant difference (< 2 sec) between the display and real temperature becomes high, indicating the slow speed rate must be set at lower than 5°C/minute to achieve accurate value.

Calibration

There is no need to do the calibration in normal case since we have calibrated all the machines and issued a calibration certificate. However, in some circumstance, users may need to do their own calibration.

There are two types of the calibration: 1) external probe calibration and 2) standard chemical calibration.

1. External Probe Calibration:

Calibration can be done in stable temperature status. Insert the capillary containing the external probe to sample location. Set the fast stage temperature at the desired point, press “start/stop’ button to run the machine. After the machine reach temperature (machine will beep), use a small screw to adjust the micro-potentiostat in the “calibration inlet” to the correct value, and then press the “start/stop” button twice to terminate the run. Or during the slow ramping, press the “read” button to hold the run for 2 minute, and machine will beep at this time and then adjust micro-potantiostat to the correct value.

2. Standard Chemical Calibration:

To calibrate the machine with known melting point of a type of test material, when the chemical starts to melt, press the “read” button and wait further about 2 minutes until the machine starts beeping. At this time, the reading will be fluctuated within +/- 0.5, adjust the temperature reading to the correct reading using a small screw through “calibration” inlet.