

COMPARISON OF TRIPLE POINT OF WATER CELLS THROUGH THEIR FREEZING CURVES

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Complete freezing curves were obtained for five water triple point cells [1]. The triple points spread over a temperature range of 0.6 mK. The data was fitted vs. $1/F$ in order to estimate the triple point of the ideally pure water. The model used was capable of estimating the ideal value by extrapolating all the measured data to $1/F \rightarrow 0$ and they coincide within ± 0.03 mK.

Experimental

Initial cell preparation: The thermometer well of the cells was filled with ethyl alcohol and a 6 mm of diameter brass bar (pre cooled in liquid nitrogen) was inserted into the thermometer well for one minute. A, hardly perceptible, solid shell formed around the well whose apparent thickness was 1 mm to 2 mm. All five cells were prepared the same day.

The cells were placed in a commercial liquid bath and the temperature was controlled to 14.0 ± 0.5 mK below the triple point temperature. The solidification process lasted approximately 28 days. The liquid fraction of the sample (F) was considered to be proportional to the time of the freezing, i.e.

$$F = 1 - \frac{d}{D} \quad , \quad (1)$$

where d is the number of freezing days and D is the total number of freezing days required to entirely freeze the samples.

Temperature was measured with a long stem, fused-silica sheathed, 0.25Ω platinum resistance thermometer. Its apparent resistance (R_t/R_s) was measured with an AC thermometric bridge (F18) with a standard resistor (R_s) with nominal value of 1Ω . Measurements were corrected to zero current values.

Results

Figure 1 shows the measured data.

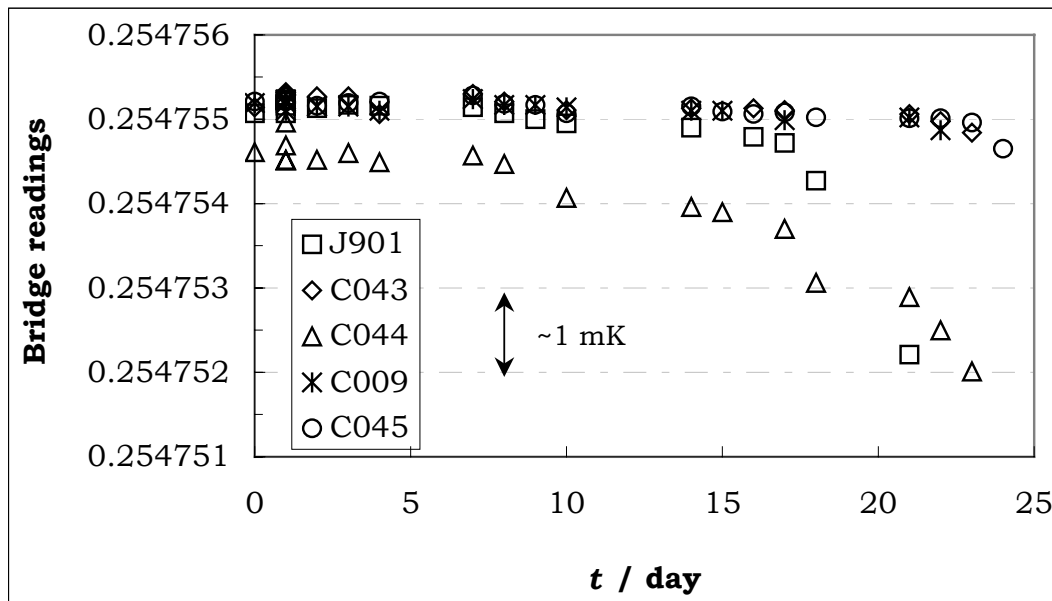


Figure 1. Freezing curves of the five samples.



Several models [2-4] can be used to analyze the data. One of those models is the $1/F$ one

$$T(F) = T_0 - \frac{x}{AF} \quad (2)$$

where T_0 is the melting temperature (in kelvin) of the pure substance, x is the amount of substance fraction (mol fraction) of impurity and A is the cryoscopic constant (its value for water is 0.0097 K^{-1} [5].)

Figure 2 shows the suitability of the above model. Line **A** shows the data for cells C009, C043 and C045, and lines **B** and **C** shows the data for cells J901 and C044 respectively.

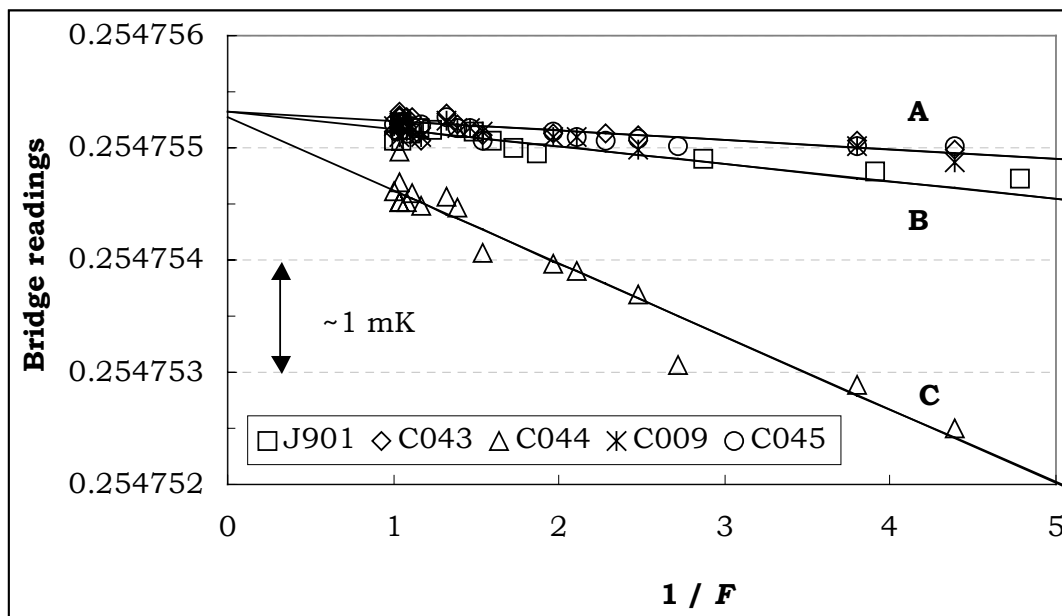


Figure 2: Bridge readings vs. $1/F$. The straight lines, and especially that they all intersect at $1/F=0$, show the usefulness of this particular model.

The parameters (T_0 and x) were adjusted by least square fitting for each cell. The resulting values are shown in table 1. The continuous lines (A, B and C) in figure 2 were calculated using these parameters.

Table 1: Adjusted parameters of the $1/F$ model. The first column identifies the cell, the second column is the calculated impurity concentration, the third column is the corresponding bridge reading $[Rt/Rs(T_0^i)]$ for the estimated T_0^i and the last column is the difference between the calculated (T_0^i) value for a given cell and the average of the five T_0 values (T_0^*).

Cell	$x * 10^6$	$Rt/Rs (T_0^i)$	$[(T_0^i - T_0^*)/mK$
J901	1.5	0.25475532	+0.03
C043	0.8	0.25475532	+0.03
C044	6.3	0.24575427	-0.02
C009	0.8	0.24575526	-0.03
C045	0.6	0.24575526	-0.03
$Rt/Rs (T_0^*) \rightarrow$		0.24575529	-

Conclusion

Five water triple point cells were used in these freezing experiments. Each cell has different impurity concentration, yet the used $1/F$ model was able to predict a unique value for water triple point within a total scatter range of ± 0.03 mK, no matter what the sample purity.

References

- [1] Méndez Lango, E. Proceedings Tempmeko 2001. In press.

- [2] Méndez Lango, E. Universidad Autónoma Metropolitana-Iztapalapa, 2000, PhD Thesis.
- [3] Méndez-Lango, E. CCT 1999, Work Document CCT99-12.
- [4] Mangum B.W., Bloembergen, P., Fellmuth B., Marcarino P., Pokhodun, A.I. CCT 1999, Work Document CCT99-11.
- [5] Mangum B.W., Furukawa, G.T. NIST Technical Note 1265, 1990 176 p.