Bureau International des Poids et Mesures

Guide to the Realization of the ITS-90

Cryogenic Fixed Points

APPENDIX 2: *Recorded Data*

Consultative Committee for Thermometry
under the auspices of the
International Committee for Weights and Measures

APPENDIX 2

Recorded Data

Essential data

Considering the conditions for measuring the reference melting curves and determining the thermal parameters as described in Appendix 1, it is recommended to obtain the following data for the evaluation of the thermal properties of the investigated Sealed Triple-Point Cell (STPC):

- One reference melting curve covering at least the range from 5 % to 95 %. Each heat pulse should not cause a melting of more than 10 % of the fixed-point sample. The preferred set of F values is: 5 %, 10 %, 20 %, …, 80 %, 90 %, 95 %.
- Equilibrium melting temperatures $T_{equ}(F)$ at $F = 50$ % and $F = 90$ % as well as the temperature width $\Delta T_{80\%} = T_{equ}(F = 90\ \%) - T_{equ}(F = 10\ \%)$ of the melting curve. ($T_{equ}(F)$ is the equilibrium temperature at fraction F, which may be obtained as the asymptotic value by fitting the thermal recovery by a superposition of exponential components [Wolber and Fellmuth 2013].) For hydrogen STPCs, the F range should be chosen so that the observed melting temperatures are not depressed by the spin-conversion catalyst.
- Repeatability of $T_{equ}(F = 50\ \%)$ and $T_{equ}(F = 90\ \%)$ based on the measurement of at least three independent melting curves. If the evaluation of the melting curves is based on the extrapolation to the liquidus point, the measurement of the complete shape for all three curves is required. Also the repeatability of the liquidus-point temperature T_{LP} is important.
- Total heat of fusion Q_{HF} deduced from each melting including the uncertainty of its determination. The Q_{HF} values obtained have to be evaluated as an additional overall check concerning the parasitic heat load during the measurement of a melting curve. For hydrogen STPCs, the heat supplied in the pre-melting range has to be included carefully.
- Internal thermal resistance at least at $F = 50$ % and $F = 90$ % and the heating power used for its measurement. If R_{cs} is significantly larger than 1 K/W, it has to be determined at all fractions of sample melted. This applies also to the measurement of the "self heating".
- Time constant τ for the recovery of the metallic body of the STPC after the heat pulses at least at $F = 50$ % and $F = 90$ % determined by fitting the first thermometer readings after the heat pulses applying an exponential law. For these fractions of sample melted, the recovery behaviour in the second part should be characterized by the recovery period $t_{r,x\ \mu K}$, which is necessary to obtain thermal equilibrium within the desired range of x μK. The determination of this parameter requires the use of sufficient post-pulse recovery time until the true equilibrium temperature is definitely reached. If necessary, an extrapolation to the asymptotic T_{equ} value, based on fitting the thermal recovery by a superposition of exponential components, see [Wolber and Fellmuth 2013], may be an alternative.

- For the reference melting curve, the static and dynamic temperature-measurement errors should be estimated considering the heat load, the internal thermal resistances and the recovery time. The method for determining the heat load and its magnitude should be given.
- Heat capacity C_c of the entire STPC assembly below and above the triple-point temperature (since C_c is dominated by the heat capacity C_{cw} of the metallic parts, there is not much difference whether the filling is in the solid or liquid state, so the measurements below and above should yield approximately equal values).
- For hydrogen, the check concerning the ortho-para conversion into the equilibrium composition should be documented. If known, the volume ratio of the catalyst and the liquid fixed-point sample as well as the parameters of the spin-conversion catalyst should be given.

Useful additional data

The following additional data and information is useful:

- It should be described how the STPC was suspended in the cryostat and how the isothermal shield surrounding it was controlled (relative to the STPC or absolute).
- The amount of the sealed substance should be provided whenever possible in order to check the obtained value of the heat of fusion.
- Thermometer-resistance values measured with two measuring currents at least at one fraction F of sample melted. These data allow calculating the thermal resistance R_w between the sensor element of the thermometer and the heat sink of the STPC. (The correction of the "self heating" itself is of course mandatory for any comparison.)
- Measurement of the overheating (not meant as referenced to some true temperature, but simply observing the reading of the thermometer) during a continuously performed melt because this allows to see tips, bumps, and irregular melting particularly at the end. (This is not a by-product of the first melt because after the cooldown, the sample may not be properly located in the STPC.)
- Total thermal isolation resistance R_e between the STPC and its environment (isothermal shield) as well as influence of changes in the settings, especially the temperature of the isothermal shield. The magnitude of this influence can be estimated if the thermal resistances R_{cs} and R_e are known. This allows to check whether the relation $\Delta T_{stat} / \Delta T_e \approx R_{cs} / R_e$ is fulfilled.
- Dependence of the thermal recovery on further experimental conditions: temperature and period of the annealing of the solid phase prior to a melt, freezing conditions, and temperature gradients existing in the STPC after heating (use of heaters located at different parts of an STPC having a stainless-steel wall).
- Dependence of the temperature range of the melting curve on the freezing conditions, as well as on the temperature and time of the annealing of the sample, occurred prior to the triple point measurement. This includes an evaluation of the beginning of the melting curve with respect to the influence of effects as pre-melting and surface supercooling. The effect of a slow refreezing is of special interest because it is known that for some fixed-point samples, the refreezing may reduce the width of the melting curve. (Since the freezing conditions may have a dramatic effect on the recovery and the shape of the

melting curve, see [Wolber and Fellmuth 2008], full measurements of the melting-curve shape are recommended for such an investigation.)

- Magnitude of supercooling and data on the recovery from supercooling. This information may be important for evaluating the quality of the fixed-point substance or its interaction with the internal parts of the STPC.
- Influence of the heating power and the duration of the heat pulses as well as overheating.

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