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THE TRIPLE POINT OF XENON: A CANDIDATE FIXED-POINT REFERENCE TEMPERATURE

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Abstract: The triple point of xenon has recently been shown to be a suitable fixed point for incorporation into the next revision of the International Temperature Scale as a means to reduce the non-uniqueness in the important 84 K to 273 K range. We summarize the results, which illustrate that the xenon triple point itself is highly reproducible, with a standard deviation of 48 µK for the eight melts of this study, and a total realization uncertainty of just 76 µK.

Keywords: international temperature scale, fixed point

1. INTRODUCTION

The triple point of xenon has long been recognized [1] as a potentially useful calibration point for the International Temperature Scale (ITS). Future revisions of the ITS will need to address the requirements of precision resistance thermometry, even if a definition of the kelvin based on the Boltzmann constant is adopted. It is anticipated that future scale definitions will retain the “two part” scheme in use today in the ITS-90, namely the combination of reference functions, which relate temperature to the thermometer’s resistance ratio, and fixed point temperatures.

In this short paper we summarize the results of an exhaustive study of the thermometric properties of a fixed point constructed using ultra-high purity xenon [3], which is important due to its desirable placement nearly halfway between the triple point of argon and the triple point of water.

2. XENON MELTING CURVES

The NRC experimental program for xenon began with an examination of triple point cells filled with gas obtained by Ancsin in the mid-1980s: a new generation of sealed cells was produced from these old cylinders (AP84, L87, and M86, respectively). The results obtained were similar to those reported by Ancsin: the behaviour (broad melting range, variation in liquidus points among different samples) reported by previous investigators was reproduced using archival gas samples.

Some years ago, two nearly empty bottles of laser-grade xenon supplied by Spectra Gases in 1985 and 1986 were obtained. New triple point cells (SG85 and SG86, respectively) filled from these bottles were found to have higher liquidus points and narrower melting ranges than any of the other xenon samples.

Through extensive consultation with Spectra Gases, a new cylinder of ultra-high-purity (99.99999%) xenon with natural isotopic composition (the composition obtained through the liquefaction of air free of any intentional alterations of the isotopic composition with respect to the air from which it was derived) was obtained in 2003, and a new sealed cell (SG03) was produced. This new sample has the flattest melting curve obtained to date, supporting the hypothesis that the historical difficulties with xenon triple point realizations were purity-related rather than attributable to isotopic effects.

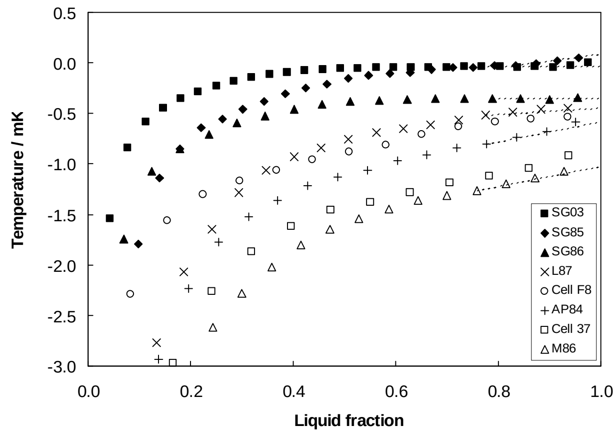

Figure 1: Summary of the melting curves for the various xenon samples of this study. The new ultra-high-purity sample (SG03, solid squares) has a melting range within ±10 µK from 50% to 90% liquid fraction, indicative of a first-quality fixed-point realization. The dashed lines are linear extrapolations used to estimate liquidus point temperatures.

Figure 1 is a summary of the adiabatic melting curves obtained sequentially, using a single capsule-style standard platinum resistance thermometer (CSPRT) to characterize the plateau for each of the eight xenon triple point cells. The symbols represent the equilibrium values measured after the application of a heating pulse directly to the sealed cell; total heats of fusion for the samples can be inferred from the amount of heat required to complete the melt, and range from 52 J (cell AP84) to 140 J (cell F8). Normalizing the abscissa to the liquid fraction (0% when the sample is completely solid, 100% when melting has finished) allows direct visual comparison of the thermal quality of the different sample gases. The coordinate axis in the figure quantifies temperature differences for each sample (in mK) with respect to the triple point of the new ultra-high-purity sample, SG03.

The flattest curve is for reference sample SG03 (solid squares), which has the second-highest liquidus point temperature and a very narrow melting range: the temperature measurements from 50% to 90% melt fraction are within ±10 μK, yielding a very flat plateau of a quality suitable for a defining fixed point of the ITS. For such flat melting plateaux, the value near 50% melted fraction is often the best estimate of the liquidus point, avoiding both the influences of crystal defects, etc., at low melted fractions and the thermal influences sometimes manifest at large melted fractions.

When the melting curves are less ideal and exhibit broadening due to the influence of impurities, the liquidus point is often determined by extrapolation to a liquid fraction of unity. The dashed lines in Figure 1 indicate the slopes of the melting curves near the liquidus point for the samples that have been analyzed for chemical impurities.

3. UNCERTAINTY BUDGET FOR XENON

The measurement-related standard uncertainty for the xenon triple point determination using the cell filled with ultra-high purity gas is shown in Table 1.

Uncertainty budgets such as Table 1 are routinely produced when realizing each of the defining fixed points of the ITS-90, although the specific details and the magnitude of the components vary from material to material. If xenon were assigned a triple point temperature in a future revision of the scale, Table 1 could be used as a complete uncertainty statement for its realization, and would be the only uncertainty required to characterize calibrations at 161.4 K.

Uncertainty components, Type B	μK
Chemical impurities	2
Isotopic composition	2
Determination of fixed-point value	10
Hydrostatic effect; gas pressure	22
Heat flux	10
CSPRT self-heating correction	7
Accuracy of resistance bridge ratio	13
Standard resistor	2
Uncertainty propagated from the TPW	50
Uncertainty components, Type A	
Repeatability (melt-to-melt)	48
CSPRT stability	10
Combined standard uncertainty	76

Table 1: The measurement-related standard uncertainty for the xenon triple point determination using cell SG03.

Since the xenon triple point temperature is not defined in ITS-90, however, the uncertainty associated with our assignment of 161.40596 K must include other unavoidable components, such as the calibration uncertainty of the thermometers and indeed the variability in interpolated temperature associated with scale non-uniqueness among a set of calibrated thermometers.

The complete uncertainty budget for the xenon triple point temperature determination on ITS-90 is summarized in Table 2, including the measurement-related components from Table 1, the propagated uncertainty from the calibration fixed points, and an estimate of the CSPRT non-uniqueness inferred from measurements made on a set of seven calibrated thermometers. The combined standard uncertainty for the assigned triple point temperature of 161.40596 K is 0.32 mK, limited by the calibration uncertainty and, to a lesser extent, the CSPRT non-uniqueness, rather than by the quality of the xenon melting curve for cell SG03.

Uncertainty components, Type B	μK
Measurement uncertainty	76
Propagated calibration uncertainty	291
Uncertainty components, Type A	
CSPRT non-uniqueness	120
Combined standard uncertainty	324

Table 2. The standard uncertainty for the xenon triple point determination. The propagated calibration uncertainty and the CSPRT non-uniqueness components dominate over the measurement uncertainty in the realization of the triple point of cell SG03.

The assigned xenon triple point temperature and uncertainty of 161.40596 K ± 0.32 mK were obtained using the deviation equation and calibration temperatures appropriate to the full low-temperature range (13.8 K to 273 K) of the ITS-90; a discussion of the consequences for use in other, more restricted, subranges is given in [3].

4. CONCLUSIONS

We have shown a very high-quality melting curve obtained for a new sample of ultra-high-purity xenon gas. With such a high-purity sample, we have demonstrated that a very flat plateau results with a melting range within ±10 μK from 50% to 90% of the melted fraction. It is clear that the triple point of xenon is suitable for incorporation into the next revision of the ITS as a means to reduce the thermometer non-uniqueness in the important 84 K to 273 K range. With a total realization uncertainty of only 76 μK, significant reduction in the non-uniqueness of the ITS would result from simply replacing the triple point of mercury by the triple point of xenon and the propagated calibration uncertainty would be improved as well, particularly for the 84 K to 273 K range. The possibility of adding a range terminating at 161 K could also prove advantageous.

In the interim, we have determined that the triple point of xenon is 161.40596 K ± 0.32 mK (k=1) on the ITS-90. We anticipate that this fixed point will benefit platinum resistance thermometry by acting as a secondary calibration point and by providing a very reproducible temperature to evaluate the non-uniqueness of the ITS-90 near 161 K for both capsule-style and long-stem platinum resistance thermometers.

As with most fixed points, sample purity remains the overriding concern, but we have demonstrated that high-purity xenon gas is commercially obtainable. Krypton, in particular, is the key impurity to be minimized and this is particularly important since it is normally the dominant impurity in commercial research grade xenon. Isotopic effects, once thought to be a limiting factor, have been shown to be negligible. Although confirmation of the influence of isotopic variations on the triple point temperature remains a worthwhile endeavour, the lack of such confirmation should not preclude xenon from becoming a defining fixed point of the ITS.

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