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Further Progress Toward the Determination of $T_{\mathrm{tp}}-x\,(^{22}\mathrm{Ne})$

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Abstract Following the start of exploration of the problem of the effect of neon isotopes on the triple-point temperature in 2005, further progress was achieved in 2006–2008, and published in 2008. This paper summarizes the advances to date in our understanding as obtained from further work done in 2008–2009 on five basic aspects of the problem: new isotopic assays; new thermal measurements on neon of "natural" composition; the feasibility of obtaining a value of the slope $\mathrm{d}T_{\mathrm{tp}}/\mathrm{d}x\,(^{22}\mathrm{Ne})$ with an accuracy sufficient for the purpose; the possible occurrence of isotopic fractionation during the process of sealing the samples in the cells; and new thermal measurements

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on the pure isotopes ^{20}Ne and ^{22}Ne. The recently collected information is sufficient to state that it will eventually be possible to fulfill within 2010 the goal of recommending to the CCT solutions for the correction, with an accuracy fitting the purpose, of the neon isotopic effect on T_{tp}, to be included in a future revision of the Technical Annexe to the "mise en pratique" of the kelvin.

Keywords Cryogenic thermometry · Isotopes · ITS-90 fixed points · Neon

1 Introduction

Following two publications in 2005 and 2008 [1,2], a considerable amount of new activities have been developed.

Continuation of activities was prompted by the fact that the work performed until 2007 was not yet sufficient to prepare a proposal similar to that in 2005 [3] for hydrogen isotopic composition. The solution for neon is more complicated, for the following main reasons:

(a) The most accurate thermal results on "natural" neon then available, those performed at PTB, resulted in a value of the slope for the T_{tp} versus $x(^{22}\mathrm{Ne})$ (indicated as ^{22}x in the following) relationship that significantly deviates from the value obtained from a linear interpolation of the T_{tp} values for the pure ^{20}Ne and ^{22}Ne isotopes, as found in the (old) literature (see in [4]). No other dataset which existed at that time could have resolved this issue.

(b) The overall set of data produced at that date—even with the exclusion of a few data considered as outliers—did not allow a precise evaluation of the slope $\Delta T_{\mathrm{tp}}/\Delta^{22}x$, because of the limited range of ^{22}x for that data. In addition, a bootstrap evaluation for a 95 % CI for that set of data, and shown in the figure taken from [4], allows a too wide range for possible values of this slope.

(c) The best literature data on T_{tp} of pure isotopes came from only two works and were affected by an uncertainty of 1 mK to 1.5 mK ($k = 1$), compared with the present state of the art $\approx 50\,\mu$K (for $k \approx 2$). Only new measurements on pure isotopes can improve this situation.

(d) Concerning the analytical assay for ^{22}x, the IRMM claimed a relative uncertainty of 0.05 %, or $u(^{22}x) = 0.00005$, sufficient for thermometric purposes. However, the two sets of assays (between which there was no common sample) produced two clusters of $^{22}\mathrm{Ne}-^{20}\mathrm{Ne}$ compositions significantly separated from each other (see Fig. 1 in [2]): there was a suspicion about the actual uncertainty level.

(e) Experts in the field of isotopic mixtures warned that isotopic fractionation can easily take place during the natural-mixture manipulation necessary to fill the cells.

(f) The neon triple-point temperature, T_{tp}, was found to be affected by nitrogen impurity by Ancsin (as reported in [5]), with an effect of $-8\,\mu$K per μmol $\mathrm{N_2}$/mol Ne. Nitrogen was detected in some of the samples used for the measurements already performed. The above value, together with the literature value for the effect of

H_2 are presently taken as correct [5], but a second independent assessment is necessary to confirm these values.

(g) Some theoretical consideration should be useful to guide in the interpretation of the results.

The new studies involved several laboratories (INRIM, IRMM, NIST, NMIJ, NRC, VSL), from those who signed in 2006 a formal agreement [6], agreeing to contribute, at least for 2008–2010, to ongoing studies of neon isotopic problems. Specifically,

(1) To obtain for part of the already measured samples more data, in order to be able to compute a more reliable slope value of the T_{tp} versus ^{22}x relationship from the data on “natural” neon (items a and b above), new thermal measurements have been performed at INRIM, NMIJ, NRC, and VSL, and are underway at NIST, concerning item (a). For the first time, NMIJ is now linked to the thermal dataset through a two-way set of data INRIM–NMIJ, and NRC will, as soon as INRIM receives from NRC a cell of “natural” neon. INRIM already received a cell from MSL and will duplicate the PTB measurement.

(2) To find a new value of the slope of the T_{tp} versus ^{22}x relationship using pure isotopes, to be compared with the literature (item (c)), and to check the melting range of pure isotopes in the cells, batches of the purest commercially available ^{22}Ne and ^{20}Ne were bought by INRIM and NRC, sealed in new cells and measured with an uncertainty as low as $\approx 50\ \mu\mathrm{K}$. The first part of these measurements has already been completed at INRIM and NRC. In addition, NIST and NMIJ received INRIM ^{20}Ne cells—and ^{22}Ne cells within late spring 2010—to perform further measurements.

(3) To resolve the doubts about the actual uncertainty of the IRMM isotopic assays (item (d)), new assays have been performed by IRMM, in particular, linking the two former groups and also adding a few new samples. The recent measurements also included an assay of the impurities (both isotopic and chemical) of the INRIM batches of “pure” isotopes 99.99 % ^{20}Ne and 99.9 % ^{22}Ne—also measured by the producers/suppliers. NRC obtained assays from the supplier for its batches of “pure” isotopes.

These are still relative (uncalibrated) measurements. IRMM also bought a batch of both pure ^{22}Ne and pure ^{20}Ne for preparing artificial mixtures whose composition is to be obtained via the gravimetric method: this was not yet possible.

(4) To resolve the doubts about possible isotopic fractionation (item (d)) specific studies have been performed at INRIM and NMIJ.

(5) To help in better understanding of some experimental details that may be critical for the final assessment, INRIM, and NRC have put forward useful considerations, and NRC and NIST have performed calculations based on theoretical considerations.

Several of the results obtained were presented at the TEMPMEKO 2010 Conference and have been submitted to several journals. The aim of this paper is to provide an overview of the main findings and perspectives of these studies.

2 Main Results in the Period 2008–2010 and Discussion

The results of the studies performed in several laboratories in the period 2008–2010 can be summarized as follows, as relevant to the recommendation to the CCT of a slope value and a correction method.

2.1 Thermal Measurements on Neon Samples of "Natural" Composition

A decrease of the uncertainty of the realization of the triple points of the cells filled with natural neon has been obtained in most of the new measurements, and so a firmer link also established with prior measurements.

At INRIM, seven cells were measured [7]: four from INRIM (1Ne, 15Ne, E4Ne, Ec2Ne), one from PTB (Ne-12), one from NPL (Ne2), and one from NMIJ (Ne-2). The thermal results obtained on four of them (E4Ne, Ec2Ne, Ne-12, and Ne2) with their corresponding ^{22}Ne amount concentrations measured at IRMM [1,2] were found to show excellent agreement with previous measurements and to match the literature value for the slope $\mathrm{d}T_{\mathrm{tp}}/^{22}x$ (Fig. 1a). However, a basic difficulty remains in computing a reliable slope value directly from measurements on "natural" neon with the uncertainty required in thermometry. The reason is as follows. The total ^{22}Ne amount concentration span of the measured samples corresponds to a range of T_{tp} of 480 μK. If one wants to limit the uncertainty in the correction for the isotopic effect in neon to 5 μK (10 % contribution to the best total uncertainty budget today obtainable), this would require a relative uncertainty of ≈1 %, i.e., 0.0015 over, say, 0.147 $\mu\mathrm{K}(\mu\mathrm{mol}\,^{22}\mathrm{Ne/mol\,Ne})^{-1}$ (assuming the uncertainty on ^{22}x to be irrelevant), an interval (0.1455 to 0.1485) $\mu\mathrm{K}(\mu\mathrm{mol}^{22}\mathrm{Ne/mol\,Ne})^{-1}$. With a spread of the newly obtained T_{tp} values within ±30 μK (expanded uncertainty ($k{\approx}2$) $U = {\approx}50\,\mu\mathrm{K}$, the slope uncertainty arising from the linear fitting of data on "natural" neon has been reduced from 100 % [4] to ≈20 %, but this is still far from the required level.

The subsequent measurements performed at INRIM on pure ^{22}Ne and ^{20}Ne [8] allowed dissipation of the uncertainties associated with the measurements on "natural" neon [7]. In fact, the four cells showed excellent consistency, within ≈30 μK, with the measurements on the pure isotopes, as shown in Fig. 1b, where also the results of the remaining three cells are reported again. For the NMIJ cell, see below; for the two INRIM cells, reasons for the discrepancy are believed to have been identified.

At NMIJ, a comparison was performed [9] between cell NMIJ Ne-5, known to have a T_{tp} value lower by 31 μK with respect to cell NMIJ Ne-2, and INRIM cell Ec2Ne, to double-check the fact, shown in Fig. 1, that the resulting T_{tp} value measured at INRIM does not match the ^{22}x amount concentration measured at IRMM [1,2]. The NMIJ results indicate a difference between Ne-2 and Ec2Ne of +50 μK($U = 64\,\mu$K), compatible with the value obtained at INRIM, +43 μK($U = 57\,\mu$K). Figure 2 shows a comparison of melting plateaux of both cells.

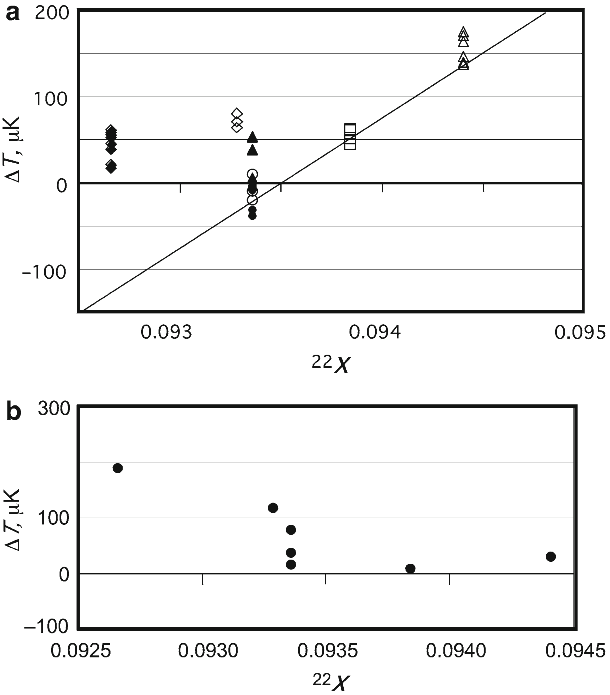


Fig. 1 (a) Liquidus point values for the seven cells using T_{LP} obtained from the data of each melt separately versus the measured amount concentration, ^{22}x [5], corrected for hydrostatic head. *Black diamonds* NMIJ Ne-2, *open diamonds* 1Ne, *black triangles* 15Ne, *open circles* E4Ne, *black circles* Ec2Ne, *open square* PTB Ne-12, *open triangles* NPL Ne2. Zero of dT arbitrary. The line has a slope 0.148 μK(μmol ^{22}Ne/molNe)$^{-1}$ taken from the literature on pure neon isotopes [2]: it is arbitrarily positioned to pass through the mean value of the results on the PTB cell (taken from [7]); (b) "Natural" neon samples measured in [7], compared with the straight line connecting the measured temperatures for ^{22}Ne and ^{20}Ne (baseline). From left to right: cell NMIJ Ne-2; INRIM cells (from top to bottom) 1Ne, 15Ne, E4Ne, and Ec2Ne; cell PTB Ne-12; cell NPL Ne2. Samples in cells 15Ne, E4Ne, and Ec2Ne were drawn, in that order, from the same gas bottle at subsequent times (taken from [8])

2.2 Analytical Assay for Isotopic Composition

At IRMM, two new series of assays were performed on 18 samples of "natural" neon in 2009. These assays have been compared indirectly with previous IRMM assays. Due to the fact that the spectrometer is un-calibrated, the calibration factor can change from run to run; however, it has been assumed as reasonable to expect that it should instead reproduce from sample to sample of a run within the stated uncertainty, given that the composition spread is small in this respect and the total time taken for the assays is short. Using this criterion, and the combined uncertainty of the pair of assays, the pair ratios have been computed: confirmation of the claimed uncertainty was obtained in many cases, but for six samples its variation for ^{22}Ne exceeds the expected reproducibility. These are coincident with outlying data in the T_{tp} versus ^{22}x plot. See also Sect. 2.4. A double check of some of these samples is underway in other laboratories worldwide, and a conclusive publication is then foreseen.

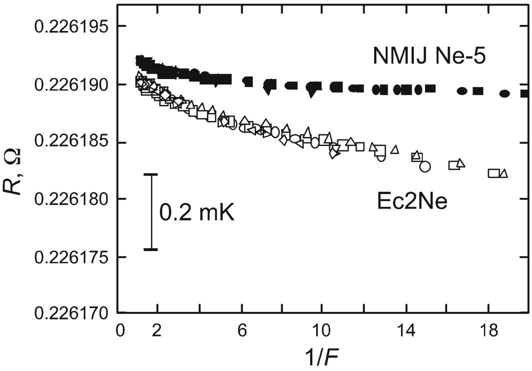


Fig. 2 Melting plateaux as measured in several runs at NMIJ for cell NMIJ Ne-5 and cell INRIM Ec2Ne, containing "natural" neon samples of different origin (taken from [9])

2.3 Measurements on Pure ^{20}Ne and ^{22}Ne

At INRIM, measurements on one cell filled with ^{20}Ne and one cell filled with ^{22}Ne have been finalized [8].

At NRC, measurements have been finalized [10] on two cells filled with material from two batches of ^{20}Ne and one cell filled with ^{22}Ne: ^{22}Ne is originating from a batch different than the INRIM one; for one ^{20}Ne batch, a suspicion exists that the batch, sold by different companies, is from the same origin as the INRIM one, since it has the same analysis certificate as an INRIM one (not measured yet).

The two batches used at INRIM carry manufacturer/supplier analyses, and they were additionally measured at IRMM. For ^{20}Ne, the purity was found to be very high, with low chemical impurity (N_2 and H_2) content, so the correction of the measured value of T_{LP} is very small. For ^{22}Ne, the IRMM value is discrepant and quite higher than the manufacturer's value, chemical impurities being very low: the IRMM value was taken as correct, resulting in an isotopic—and chemical—correction of $+331\,\mu$K.

Two batches of ^{20}Ne and one batch of ^{22}Ne were used at NRC carrying a manufacturer/supplier analysis of both chemical and isotopic impurities (overall isotopic purity for ^{20}Ne and for the specific amounts of ^{20}Ne and ^{21}Ne for ^{22}Ne; for the latter also an assay of the chemical impurities and of the overall isotopic purity was available). For ^{20}Ne, the purity was found to be very high, with low chemical impurity (N_2) content, so the correction of the measured value of T_{LP} is very small. For ^{22}Ne, the assay values for ^{20}Ne and ^{21}Ne are not so low, while the chemical impurity level (N_2 and H_2) is very low: the resulting isotopic—and chemical—correction of $+150\,\mu$K, quite different than for the INRIM one, is a useful difference.

The resulting measured difference, $T_{tp}(^{22}\text{Ne}) - T_{tp}(^{20}\text{Ne})$, corrected for the effects of the estimated concentrations of the amount of chemical and isotopic impurities, was found to be (in parentheses, the *expanded* uncertainty U):

- at INRIM 0.146 60(7) K (or 0.146 27(10) K if uncorrected for these effects),
- at NRC 0.146 58(10) K (or 0.146 43(12) K if uncorrected for these effects).

The agreement of the INRIM and NRC corrected values is excellent and is believed to also indicate that the measured values of the isotopic-impurity concentrations are basically correct.

At NIST, new measurements on existing neon cells are underway, new cells are being prepared using neon gas with known isotopic composition, and measurements are planned using new INRIM-cells of pure isotopes ^{20}Ne and ^{22}Ne. Results from these efforts, however, are not yet available.

2.4 Isotopic Fractionation

Reasons exist for isotopic fractionation to occur during gas handling. If the gas is throttled through a small orifice (like a leaking valve) in a short time, the lighter isotope is likely to be transferred in a larger proportion with respect to the heavier isotope, making the concentration of ^{22}Ne in the sample smaller with respect to an assay performed before the gas is drawn, and leaving the rest of the bottle enriched in ^{22}Ne, relevant to samples drawn subsequently.

Typically, a small amount of gas is drawn each time from a high-pressure cylinder and transferred through the gas handling system to which the cells to be filled are attached for initial conditioning. One may want, in general, to minimize the risks of contaminating the gas drawn due to small residual leaks in the gas inlet system, and so it is normal practice to open the cylinder valve only a little and for the shortest time possible. In addition, the pressure regulator might also be considered as a small orifice in certain circumstances.

Isotopic fractionation might also occur as a result of some types of filling technique. For example, at INRIM, cryogenic condensation is used: cold helium vapors condense neon from the filling system in the cell, waiting until the pressure of the residual gas in the system is $<\approx 5$ kPa (the minimum detectable from the pressure gauges), quite below the triple-point pressure of neon, 43.4 kPa [7].

There are therefore reasons why samples sealed from gas of the same bottle may show different isotopic compositions. A theoretical study is not simple, and it may be of little help in being applied to actual experimental conditions. However, since this effect depends on subsequent drawing of samples (no effects possible from adsorption on the bottle walls or from leaks of air), one might be able to detect part of it from the time sequence of the results.

At INRIM, the T_{LP} values obtained for cells containing samples drawn in subsequent times from the same gas cylinder were compared [7]: the study was essentially inconclusive, with only a small probability that some effects, limited to about 55 μK($U = 42$ μK) in total, had occurred.

Concerning NMIJ, due to the large discrepancy of the NMIJ cell, it was thought that the ^{22}Ne concentration amount in the NMIJ cells might have changed from that of the original Ne gas bottle assayed by IRMM. One may consider the possibility of inducing a change of the ^{22}Ne concentration with the NMIJ filling system for preparation of sealed cells. To check this possibility, the isotopic composition of Ne samples extracted from three different positions of the NMIJ filling system was assayed at the University of Tokyo. There was no significant difference of the isotopic composition among the three Ne samples. The origin of the deviation from T_{tp} versus ^{22}x relationship for the NMIJ cells has not been yet clarified [9].

2.5 Calculations Based on Theoretical Considerations and Other Cell Issues

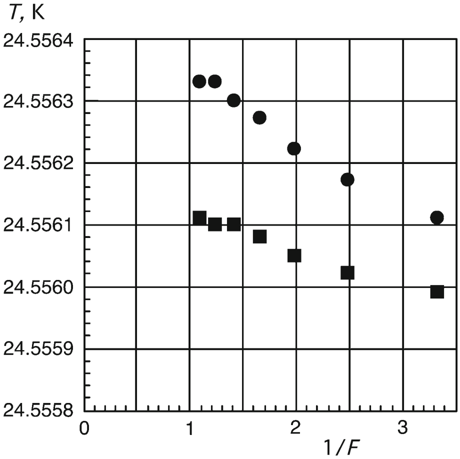
Similar to Fig. 2, showing meltings of one NMIJ and one INRIM cell, Fig. 3 shows an example of the melting plateaux obtained at VSL on cells 14Ne and 5Ne (of INRIM production), containing samples of "natural" neon of different origin. Overall, the melting range of cells of different models, defined as the temperature difference between melted fraction $F = 10\,\%$ and the liquidus point, was found ranging from less than 100 μK up to about 300 μK, depending on the cell model and on the thermal history of the sample.

At NRC, studies have been performed on two issues concerning isotopic mixtures [10]. For "natural" neon and for the cell models considered (condensed sample of a vertical cylindrical form with thickness $>\approx 1$ mm, examples of melting behavior in Fig. 4), evidence was found that the melting range is affected not only by the thermodynamic behavior of the mixture, causing typically a difference between the solidus and liquidus point of 100 μK, but also by isotopic fractionation for the changing melted fraction. These issues however do not appear to affect the value of the liquidus-point temperature.

At both NIST and NRC [10], calculations have been performed on the ideal-solution phase boundaries based on the known pure isotope triple-point temperatures, $T\,(^{20}\text{Ne})$ and $T\,(^{22}\text{Ne})$ and their respective enthalpies of fusion (see Fig. 5). The calculated slopes of the liquidus lines agree within the margins expected given the small variations in the assumed input parameters.

At INRIM, only cell model (c) was presently used for pure isotopes [8], where the inner copper body is made of many wires, 0.8 mm in diameter, closely packed and

Fig. 3 Typical melting plateaux as measured at VSL on cell INRIM 5Ne (*black squares*) and INRIM cell 14 Ne (*black dots*) containing "natural" neon samples of different origin (courtesy of A. Peruzzi)



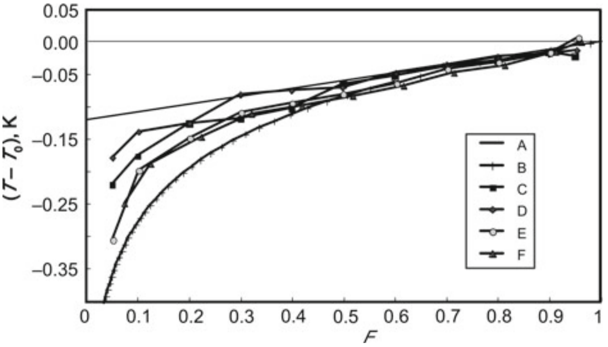


Fig. 4 Modeled temperature curves for the phase transition of neon of "natural" isotopic composition together with actual measurements of neon fixed points. (*A*) Modeling of thermodynamic equilibrium, (*B*) Scheil model, (*C*) melt after slow freeze, taken from [12], (*D*) melt after slow refreeze, taken from [12], (*E*) melt after fast freeze, taken from [12], and (*F*) measurement of a fixed point at NRC with shorter waiting times than for curves (*C*), (*D*), and (*E*) (taken from [10])

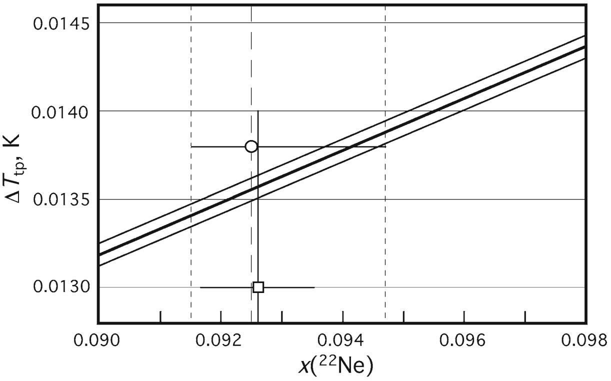


Fig. 5 Calculated phase boundaries for a ^{20}Ne-^{22}Ne ideal binary solution based on pure isotope triple-point temperatures from [10] and enthalpies of fusion given by $1.65RT_{\mathrm{tp}}$: solidus slope $y = 0.14589\,x$, liquidus slope $y = 0.14721\,x$, linear slope (^{20}Ne–^{22}Ne)$y = 0.14655\,x$ The present IUPAC CAWIA recommended value for $x(^{22}\mathrm{Ne})$ (*dashed line*) is shown along with the current estimated upper and lower bounds for commercial gas (*point lines*). The data for commercial Ne is shown from Furukawa [13] (*open square*) and from Hill and Fahr (*open circle*). The $x(^{22}\mathrm{Ne})$ for the commercial gas sample in [10] is assumed to be equivalent to the IUPAC value. The origin for ΔT corresponds to pure ^{20}Ne and is assumed to be equivalent for both data sets (courtesy of W. Tew)

leaving an undisturbed vertical cylindrical 1 mm thick space between the packed wires and the wall of the cell. With this type of cell, a somewhat larger melting range has been observed, which in principle could be explained qualitatively by the presence of a certain amount of fragmented interface constrained in narrow spaces; this is preliminarily believed to produce a double effect, a smaller effect of the isotopic fractionation effect and a lower start of the melting temperature—the latter in similarity to what has been clearly explained for the catalyst in *e*-H_2 cells. The above issues would not affect the value of the liquidus point.

3 Conclusions and Pending Work

As a general consideration, while the ITS-90 defines the Ne isotopic composition via the ambiguous term, "natural" neon, all practical sources of gas are those derived from air by commercial suppliers. Therefore, the Commission for the Atomic Weights and Isotopic Abundance (CAWIA) of the International Union of Pure and Applied Chemistry (IUPAC) recommended values for the isotopic composition of atmospheric neon not necessarily the same as that of *commercial* gas, as indicated in the relevant note of the CAWIA last document. Thus, the use of the term "commercial" should be preferred to the term "natural."

The slope values obtained from the new measurements on pure isotopes are, first of all, very close to Furukawa's value (0.1470(14)K for $k = 1$); second, in both cases the values are compatible with each other. Therefore, we are quite confident that, with the supplement of the NIST and NMIJ measurements, it can be demonstrated that the slope value is now known largely with sufficient accuracy for resolving the problem of the dispersion of the T_{tp} value of "natural" neon of commercial origin, a problem that would be difficult to resolve by relying only on measurements of samples of "natural" composition. This will allow the CCT to discuss possible solutions, similarly to what was already done in 2005 for H_2 and H_2O [11]. Basically, there are two possible solutions: the first resorting to the choice of a reference value for the "natural" composition, e.g., using the IUPAC CAWIA recommended value; the second taking advantage of the fact that the ^{20}Ne T_{tp} is only $\approx$ 14 mK lower than that of "natural" neon—the accuracy of the slope value would suffice for this larger correction; or both.

Some supplementary studies would be desirable, to increase the confidence in the present results.

Considering how critical is the precision in the knowledge of the impurity content, either isotopic—namely for ^{22}Ne—or chemical, further measurements on different batches of different impurity contents would be desirable. In this respect also double checks on the existing values would increase confidence in their accuracy.

Further studies on the effect of different cell models would allow the detection of possible systematic errors due to cell geometry and thermal resistance, which could affect the liquidus-point temperature.

A double check of the effect of some chemical impurities, namely, He, H_2, and N_2 would increase the reliability of their correction. This would also require collaboration from the analytical laboratories to obtain reliable assays for these impurities. One alternative solution could involve the routine use of existing devices able to filter out all chemical impurities except the noble gases to a relative level below 10^{-7}.

Finally, some laboratories could perform further thermal measurements to complete or double check the link between existing cells containing samples of "natural" neon. These studies are useful to allow an accurate future correction of the measured values with some of these old cells.

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