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Progress Towards the Determination of the Relationship of Triple-Point Temperature versus Isotopic Composition of Neon

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Abstract Following the finalization of the work performed to establish the triple-point temperature versus isotopic composition relationship for protium (Metrologia **42**, 171 (2005)) adopted into the ITS-90 definition by the International Committee for Weights and Measures (CIPM) in 2005, and a preliminary exploration of the variability in the triple-point temperature of neon gas samples arising from differences in isotopic

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composition (Anal. Chem. **77**, 5076 (2005)), this article reports further progress toward the determination of a similar comprehensive relationship for neon, to be included in a future revision of the Technical Annexe to the '*mise en pratique*' of the kelvin. This progress article mainly concerns a set of gas samples used for the neon triple-point measurements during CCT-K2, and subsequently, others for which the isotopic compositions were recently measured. Recent high accuracy measurements of the corresponding thermal data are now available for many of these samples, but not yet for all of them.

Keywords Isotopes · ITS-90 · Kelvin *mise en pratique* · Neon · Triple-point temperature

1 Introduction

As summarized in [1], terrestrial neon predominately originates from two different sources: air, from which commercial neon is obtained, and mantle neon, from which the crustal neon found in natural gas wells is derived. The internationally recognized value for the isotopic composition of neon [2] is based on isotope measurements of neon in air.

Since the neon isotopic composition affects some of the properties of neon relevant to thermometry, namely, the temperature of the triple point, failure to specify its isotopic composition leads to ambiguity in the realized temperature. Although this fact has been recognized for a long time, the present definition of the International Temperature Scale of 1990 (ITS-90) simply states that the neon must be of "natural" isotopic composition. In fact, the isotopic variability of commercial gas limits the accuracy of the realization of the neon fixed point.

Consequently, a worldwide study concerning the variability of the isotopic composition of commercially available neon has been underway since 2003, initially within the framework of the European Research Project MULTICELLS [3], later within the framework of a EUROMET Project [4], and now merged into a Joint Research Project within the European Project iMERA [5]. The study is especially directed at the neon samples used in international intercomparisons of temperature standards carried out since 1978.

In 2005, the first results of isotope measurements performed in 2004 on six neon samples were published [1]. These results show two main features: all six samples probably originate from the distillation of neon derived from air, and there is an isotopic fractionation effect whose extent was totally unexpected by the specialists in the field. The sample-to-sample relative variability of the ^{22}Ne isotope amount fraction was ~1.8% of the nominal value $x(^{22}\mathrm{Ne}) = 9.25\%$,[1] corresponding to a variation in the triple-point temperature (T_{tp}) of about 0.3 mK, significantly higher than the state-of-the-art uncertainty of modern realizations of this fixed point [6]. The variability of the ^{21}Ne isotope, however, does not seem to be a problem for thermometry; since

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[1] We use the conventional notation for amount of substance $n(^{j}\mathrm{Ne})$, and the amount-of-substance fraction $x(^{j}\mathrm{Ne}) \equiv n(^{j}\mathrm{Ne})/\Sigma n(^{i}\mathrm{Ne})$.

normally only a small ^{21}Ne amount fraction, $x(^{21}\mathrm{Ne}) = 0.27\%$, is present, its relative influence is 1.5% that of ^{22}Ne.

The next step, for which preliminary results are reported in this article, was aimed at completing as far as possible the Ne isotopic analyses of the samples for which thermal data are currently available, specifically in connection with intercomparison exercises performed since 1978: the 'International Intercomparison of Triple Points in Sealed Cells' (1978–85, IMGC pilot) [7], the CCT-K2 (NRC pilot) [8] and related comparisons CCT K2.x [9], and the Euromet Star Intercomparison of sealed cells (PTB pilot) [10]. The recent sealed cell comparison [10] is the most important among these, because the thermal measurement uncertainty was substantially improved.

The final phase of these studies will be neon isotopic analyses and related thermal measurements on a different set of commercial neon samples of recent worldwide[2] production. The final goal is to verify the validity of the value of $\mathrm{d}T/\mathrm{d}x$ for ^{22}Ne that today can only be obtained from published values of T_{tp} of the pure neon isotopes, and to recommend to the Consultative Committee for Thermometry (CCT) an algorithm for the isotopic correction of the ITS-90 triple-point temperature of neon, similarly to that reported in 2005 for e-H_2 [11].

2 Isotopic Composition of Neon

Eight neon gas samples from different commercial suppliers and from various batches were supplied to the present (2006) studies, and used to realize the temperature standards at INRIM,[3] LNE-INM,[3] NIST, and NMIJ/AIST. They are listed in Table 1, with additionally a ninth sample used by IRMM as an internal standard. All future measurements of neon isotopes (of other samples) can be linked via this standard to previously obtained results. None of the samples reported here were included in the earlier (2004) study [1].

Prior to performing the isotope abundance ratio measurements, all the high-purity gas samples were checked at IRMM for chemical purity. A full description of the experimental method used by IRMM for the analytical assays can be found in [1]. The present isotopic data are given as *ion current* ratios—which are approximately equivalent to isotope amount ratios. There are presently no corrections for (minor) unknown systematic effects because no artificial isotope mixtures (primary measurement standards) of Ne are available. These corrections are normally a maximum of 5×10^{-4} of the ion current ratios [1].

Figure 1 presents the results of the neon isotope measurements of the nine new samples. In addition, the 2004 results (as published in 2005) [1] are reported together with a selection of archival data that are of direct relevance. Other data are not reported in the figure because their uncertainties are unknown or are much higher. The Compton [15] values for $n(^{22}\mathrm{Ne})/n(^{20}\mathrm{Ne})$ were relative to an arbitrarily chosen reference gas bottle, and so are not useful for comparison to other Ne isotope data. Kemp and

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[2] Concerning *absolute* measurements of the isotopic composition via artificial mixtures of neon isotopes, see Sect. 2.

[3] INRIM: until 2005, IMGC; LNE-INM: before 2005, INM-BNM.

Table 1 Samples measured in 2006 at IRMM (in chronological IRMM measurement order)

Manufacturer	Nominal chemical purity (%)	Chemical purity[a] (%)	Sample production year	Used by
SIO	99.99	99.995	1976	INRIM
Carbagas	>99.995	>99.995	2005	IRMM[b]
Matheson - A	>99.995	>99.995	1977	INRIM
NBS-101	99.995	99.991	1954	NIST
Matheson - B	>99.999	>99.995	1978	INRIM
Airgas/BOC	99.999	>99.995	2006	NIST
L'Air Liquide (Alphagaz)	99.999	99.992	1999	LNE-INM
Air Water	>99.999	99.994	2006	NMIJ-AIST
Iceblick	>99.999	>99.995	2003	NMIJ-AIST

[a] As measured at IRMM

[b] IRMM neon internal isotope standard

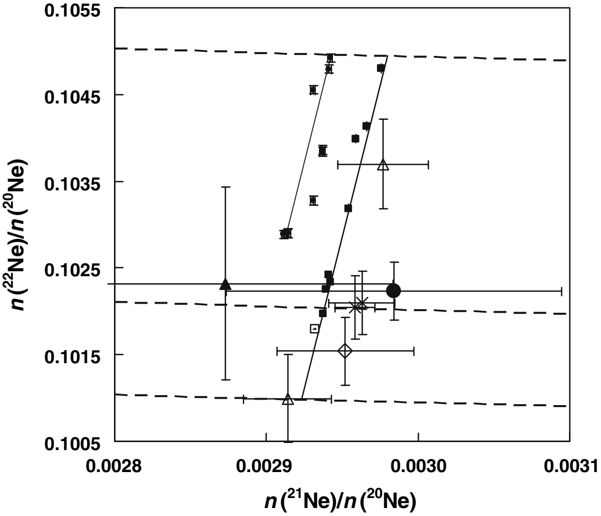

Fig. 1 Neon isotope ratios: 2006 samples (black squares –lowest open square: NBS-101), compared with previous data: 2004 samples [1] (black dots); Cameron and Walton [12] (1965) determination of IRM NBS-101 (open diamond); Furukawa [19] (black triangle); Tiggelman [14] (open triangles); Eberhardt et al. [26] (X, neon from purified air). IUPAC CAWIA currently recommended composition [2] (black circle). The diagonal lines are predicted mass-fractionation trends associated with the 2004 and 2006 ratio distributions. The central dotted line represents a line of constant relative atomic weight equal to 20.1797, the value currently recommended by the CAWIA [2]. Upper and lower dashed lines are upper and lower limits of atomic weight implied by the data: their range is 324 × 10^{-6} A_r, equivalent to $\Delta T_{tp} = 480\,\mu K$

Kemp [16,17] report isotopic determinations measured at two separate laboratories for a single Matheson gas sample. The average ratios of the two data sets were: $n(^{21}\text{Ne})/n(^{20}\text{Ne}) = 0.00266(6)$ and $n(^{22}\text{Ne})/n(^{20}\text{Ne}) = 0.10556(39)$. These ratios are highly anomalous for any reported Ne from air separation, so unknown systematic errors are likely.

A few specific issues regarding the data in Fig. 1 need to be pointed out.

- One of the samples from Table 1 was the Isotope Reference Material (IRM) NBS-101 [13]. This IRM gas was never certified at the National Bureau of Standards (NBS, now NIST); however, in 1965, a sample of NBS-101 was included in a series of absolute isotopic determinations of commercial neon gases performed by Cameron and Walton [12] at the Oak Ridge National Laboratory. Within the reported uncertainties, that determination is in agreement with the ion current ratios given here, as measured by IRMM;
- IUPAC's Commission for Atomic Weights and Isotopic Abundances (CAWIA) recommended value of 0.00298 ± 0.00011 for the ratio $n(^{21}\mathrm{Ne})/n(^{20}\mathrm{Ne})$ in air is such that all IRMM present determinations, and those published in 2005 [1], are within the stated uncertainty. The distribution of the measured ratios from IRMM suggests, however, that the samples are slightly depleted in ^{21}Ne with respect to the CAWIA recommended value for air;
- The CAWIA recommended value for the ratio $n(^{22}\mathrm{Ne})/n(^{20}\mathrm{Ne}) = 0.10223 \pm 0.00031$ corresponds to a ^{22}Ne amount fraction that is lower than present samples and lower than all the values published in 2005 [1]. So, in contrast to the ^{21}Ne case, most of these samples appear enriched in ^{22}Ne. Literature values from earlier studies using commercial neon gases show some correspondence with the present (2006) results. For example, in Fig. 1, one of two samples used by Tiggelman in the 1970s for thermometric studies [14] had a composition comparable to some of the present samples of high ^{22}Ne content, while the second sample was entirely below the 2006 distribution;
- The present values of the *ion current* ratios are on a straight line ('mass fractionation line') nearly parallel to the line fitting the values published in 2005 [1] for the *isotope amount* ratios, but shifted by about $\Delta\{n(^{21}\mathrm{Ne})/n(^{20}\mathrm{Ne})\} = +0.000037$ if the shift is totally attributed to $n(^{21}\mathrm{Ne})$ or $\Delta\{n(^{22}\mathrm{Ne})/n(^{20}\mathrm{Ne})\} = -0.0026$ if the shift is totally attributed to $n(^{22}\mathrm{Ne})$. If the IRMM internal neon isotope standard is used as a reference, the ideal slope of the mass fractionation line would be $2[n(^{22}\mathrm{Ne})\ /n(^{20}\mathrm{Ne})]/[n(^{21}\mathrm{Ne})/n(^{20}\mathrm{Ne})] = 69.56$. A linear regression of the data concerning samples in Table 1 yields a slope of 72.37 ± 3.73, indicating that the data are consistent with a mass fractionation process.

When the results of Fig. 1 are compared to CAWIA's recommended isotope amount ratios for atmospheric neon, it can be seen that most $n(^{22}\mathrm{Ne})/n(^{20}\mathrm{Ne})$ ratios are clearly higher—by up to 2.7%. The full range of all reported isotope ratios for commercial neon implies relative variations in the molar mass of 324×10^{-6}, more than 10 times greater than the current CAWIA recommended uncertainty for the value of the relative atomic weight $A_\mathrm{r}(\mathrm{Ne}) = 20.1797 \pm 0.0006$ in air. Most of the contemporary samples measured are significantly heavier than this recommended value. The shift in the mass fractionation trends from the 2004 data [1] to the 2006 data (this work) remains to be understood, partially because the two sets of data are presently unrelated. The direct comparison of the IRMM internal reference gases will be performed at a later date and will be presented in a subsequent publication.

The uncertainties of the isotope ratios presented here are considered nominally equivalent to those presented in the 2004 study [1]. They were estimated from a combined relative standard uncertainty of $\sim 5 \times 10^{-4}$ for either ratio, which yields ratio uncertainties of $u[n(^{22}\mathrm{Ne})/n(^{20}\mathrm{Ne})] \approx 5 \times 10^{-5}$ and $u[n(^{21}\mathrm{Ne})/n(^{20}\mathrm{Ne})] \approx 1.5 \times 10^{-6}$. These uncertainties are considered provisional until such time as a set of calibrated artificial mixtures can be prepared.

To summarize, the CAWIA recommended isotopic composition for Ne in air seems inconsistent with that of 11 of the 15 commercial gas samples taken from a dozen different origins and measured thus far. In addition, in order to obtain *absolute* determinations of the isotope amount ratios, artificial (i.e., gravimetrically prepared) isotope mixtures are required in the future. At present, there are no certified or otherwise traceable IRMs available to provide *absolute* neon isotope amount ratios. Such traceability is necessary to the thermometric correction, which should be based on physical states of known composition and/or molar mass.

3 Thermal Data for Neon Isotopic Mixtures

Only a few high-accuracy determinations of the triple-point temperature have been published for pure neon isotopes. Since it is only the differences in temperature between the various compositions that are relevant to this work, the problem of the different temperature scales used by various authors is alleviated. Figure 2 reports the data obtained from [18,19]; the older work of Clusius et al. [20] is not considered here. The residuals of the linear fit do not exceed 1 mK, compared with the uncertainty stated in the two articles of ±1.5 mK and ±1 mK, respectively. For the two works, the corresponding slopes $\mathrm{d}T_{\mathrm{tp}}/\mathrm{d}x(^{22}\mathrm{Ne})$ are $0.147\,\mu\mathrm{K} \cdot (\mu\mathrm{mol} \cdot \mathrm{mol}^{-1})^{-1}$ and $0.150\,\mu\mathrm{K} \cdot (\mu\mathrm{mol} \cdot \mathrm{mol}^{-1})^{-1}$, respectively.

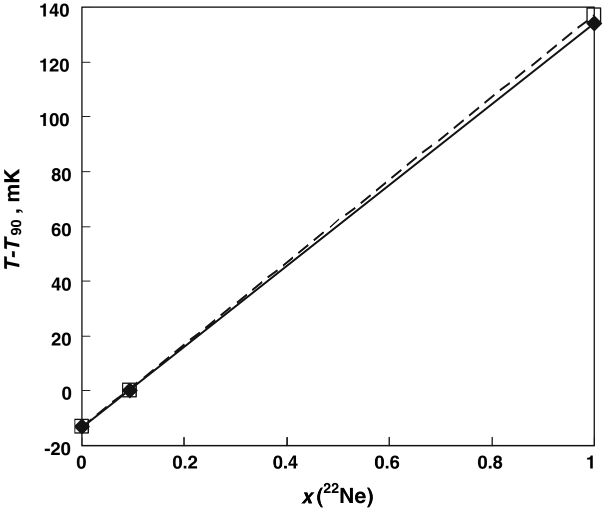

Fig. 2 Linear models for observed triple-point temperature difference versus mole fraction of ^{22}Ne. Isotopic data from [18] (dotted line and open squares: $\Delta T/\mathrm{mK} = 150 \cdot x(^{22}\mathrm{Ne}) - 13$) and [19] (solid line and black diamonds: $\Delta T/\mathrm{mK} = 147 \cdot x(^{22}\mathrm{Ne}) - 13$): the zero of the vertical scale is the "natural" compositions that are assumed to be equivalent. The effect of ^{21}Ne is negligible

Table 2 Neon samples generating data and their uncertainties ($k = 1$) considered in Fig. 3

Neon cell #	Used in	Provided by	Measured by
Ne-12	Star[a]	PTB	PTB
1Ne 2-VI-77	CCT[c], K2[b], Star	INRIM	INRIM, KCDB[d], PTB
3Ne 6-II-79	CCT[c], K2[b], Star	INRIM	INRIM, KCDB[d], PTB
Ne02/1	Star, MULTICELLS[e]	LNE-INM	PTB, NMi-VSL
Ne-2	K2, Star	NPL	KCDB, PTB
Ne-F17	Star	NRC	PTB
7Ne 7-IV-86	Star	INRIM	INTiBS, PTB
11Ne 10-IV-86	Star	INRIM	PTB
12Ne 21-X-99	Star	INRIM	PTB
E1Ne-22-XII-99	Star	INRIM	PTB
Ne-7	K2, Star	PTB	KCDB, PTB
Cell 12	K2	NRC	KCDB
BCMMT97-5	K2	LNE-INM	KCDB
E3Ne 29-IX-00	MULTICELLS	INRIM	NMi-VSL
Eb1Ne 08-VI-01	MULTICELLS	INRIM	INTiBS
Ec2Ne 17-I-02	MULTICELLS	INRIM	INTiBS

[a] Euromet Star Comparison [10]: PTB uncertainty 45–61 μK
[b] CIPM CCT-K2 [8] uncertainty 140–220 μK (INM 520 μK)
[c] CCT Comparison [7]
[d] As published on the BIPM KCDB (kcdb.bipm.org)
[e] European Project MULTICELLS [3]: INTiBS uncertainty 59–71 μK; NMi-VSL uncertainty 150 μK

Many of the 2006 samples, and all the 2004 samples, have already been used in thermal measurements aimed at determining T_{tp}. However, at present, not all of these measurements can be *directly* related to one another. Several of them can presently only be related *indirectly* to the others through measurements using different thermometers of uncertain temporal stability, leading to much larger uncertainties that render the data ineffective in determining the value of dT/dx.

Table 2 lists the fixed-point cells taken into consideration for Fig. 3 of this article, which shows the corresponding results obtained thus far concerning the T_{tp} versus ^{22}Ne amount fraction $x(^{22}\text{Ne})$ relationship for commercial neon gases. In Fig. 3, no fit of the present data has been attempted: a linear approximation is reported instead, to show the extent of the compatibility with the present results, based on data from [18,19] for pure isotopes.

4 Discussion and Conclusions

Currently, not all gas samples with an accurate isotopic analysis have corresponding thermal data available of sufficient accuracy. Hence, some samples in Table 1 cannot yet provide data points for Fig. 3. A few gas samples showed inconsistent thermal or isotopic composition values and have been flagged for further analysis.

The impurities that might influence the triple-point temperature of neon, and contribute to the dispersion of the thermal data, are helium, hydrogen, and nitrogen. Helium, generally the major impurity in neon, was found to have negligible influence ($<$0.1 mK) on T_{tp} [21]. Hydrogen was reported [21] to have negligible influence, but,

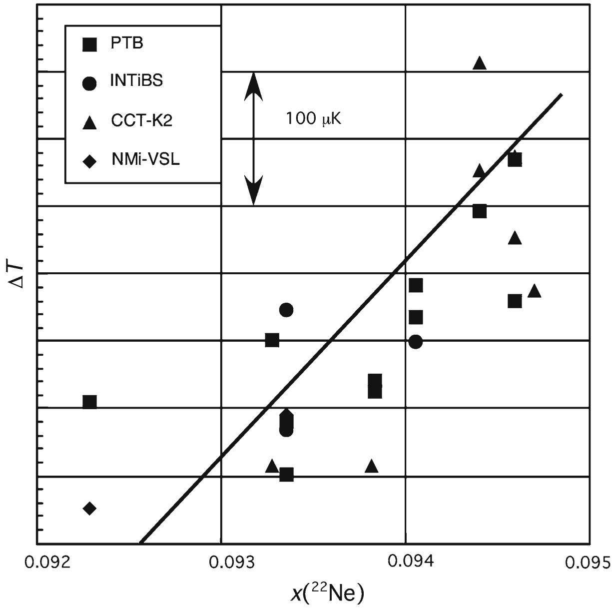

Fig. 3 Temperature differences between cells—zero is arbitrary—for the full set of data as a function of the ^{22}Ne isotope amount fraction; their uncertainties are reported in Table 2. The linear approximation of Fig. 2 is reported, using an average slope of 148 mK (vertical position arbitrarily chosen)

on the contrary, an initial slope of the liquidus line of −5 μK per μmol of H_2 per mole of Ne has been observed [22,23], though, as stated in [21], it is questionable if the method used in the latter references applies to the condition of neon in a thermometric cell. Nitrogen was reported in [21] to decrease the liquidus point temperature, T_{LP}, by −8 μK per μmol of N_2 per mole of Ne for concentrations of nitrogen lower than about 50 μmol of N_2 per mole of Ne; however, in neon purer than about 99.995%, nitrogen rarely seems to be a significant chemical impurity, with its concentration unlikely to be more than 2 μmol · mol^{-1}. Therefore, although the use of the cryoscopic constant ($k_c = 0.0668\ \mathrm{K}^{-1}$) [24] would indicate a T_{LP} depression of −15 μK per μmol of chemical impurities per mole of neon, the depression itself and its related uncertainty ($k = 1$) seem more likely to be limited to below 10–20 μK for all gas samples of Table 2.

The dT/dx slope value, estimated thus far by using different sets of available data, is at present much too dependent on the pivoting effect of some of the data values. As a consequence, preliminary results are reported without a tentative slope value. Instead, we only show in Fig. 3, the degree of compatibility of the present measurements with a linear approximation, as obtained from [18,19], based on pure isotopes. While this indicates our progress with respect to the work published in [1], it is not yet a conclusive study of the best estimate of slope required to correct samples differing in isotopic composition from a given reference composition. Given such a composition $x_s(^{22}\mathrm{Ne})$ with an assigned triple-point temperature T_0, corrections may be applied via

$$T_{\mathrm{tp}} = T_0 + \frac{\mathrm{d}T_{\mathrm{tp}}}{\mathrm{d}x\left({}^{22}\mathrm{Ne}\right)}\left(x\left({}^{22}\mathrm{Ne}\right) - x_s\left({}^{22}\mathrm{Ne}\right)\right).$$

Alternatively, corrections could also be applied in terms of the relative atomic weight $A_{\mathrm{r}}(\mathrm{Ne})$ relative to that of a known standard, $\mathrm{Ne_s}$, via

$$T_{\mathrm{tp}} = T_0 + \frac{\mathrm{d}T_{\mathrm{tp}}}{\mathrm{d}A_r\,(\mathrm{Ne})}\left(A_r\,(\mathrm{Ne}) - A_r\,(\mathrm{Ne_s})\right).$$

The work foreseen in the introduction of this article to finalize the analysis of the $T-x$ relationship for ^{22}Ne will require more analytical assays of neon samples and more thermal measurements of sufficiently low uncertainty before a proposal can be forwarded to the CCT for addition to the ‘mise en pratique’ ITS-90 Technical Annexe. For neon, the contents of this proposal are more complex than they were for hydrogen.

On the one hand, the lack of a correction would force metrologists to add an uncertainty component for the unknown isotopic composition of neon to account for the whole range of variability of the triple-point temperature due to sample-to-sample isotopic composition variability. Based on the available data, this full range amounts to 480 μK. Assuming a rectangular probability distribution, this would correspond to an uncertainty $u_{\mathrm{iso}} \approx 140\,\mu\mathrm{K}$, which is too high when compared to the state-of-the-art *total* uncertainty for the remaining components that is normally less than 100 μK.

On the other hand, in order to perform this correction, each laboratory requires an isotopic composition assay of their neon samples, unless a reference material is used—a scheme not presently allowed by the ITS-90 [25]. Only one analytical laboratory is presently known worldwide with proven ability to measure the isotope amount ratio $n(^{22}\mathrm{Ne})/n(^{20}\mathrm{Ne})$ with the accuracy needed by thermal metrology (the variability of the isotope amount ratio $n(^{21}\mathrm{Ne})/n(^{20}\mathrm{Ne})$ has negligible influence on the triple-point temperature). Unless other opportunities to obtain an assay become available, this would restrict the ability of any metrological laboratory to obtain the lowest possible uncertainty for its realization of the triple point of neon by correcting for the isotopic composition.

Similarly, the CIPM CCT-K2 data published on the KCDB could be updated to account for the sample-to-sample variability in the isotopic composition of e-$\mathrm{H_2}$, but more time is needed to be able to account for neon isotopic variations as well.

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