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The Impact of Isotopic Concentration, Impurities, and Cell Aging on the Water Triple-Point Temperature

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Abstract In 2005, the National Institutes of Standards and Technology (NIST) and Fluke's Hart Scientific Division initiated a study to validate the isotopic correction algorithm applied to the realization temperature of triple point of water (TPW) cells. Additionally, the study quantified the impact of water sample impurities on the TPW cell realization temperature. For this study, eight TPW cells containing water of the same nominal isotopic concentration as Vienna Standard Mean Ocean Water (VSMOW) were used. Five of the cells were manufactured with fused-quartz envelopes and the remaining three with borosilicate envelopes. One TPW cell of each type was uniquely designed so that water samples could be periodically removed to analyze the isotopic composition and to monitor any changes in water purity with time and thereby correlate changes in composition with changes in realization temperature. The borosilicate TPW cells gave an average drift of $-13\,\mu\text{K}\cdot\text{yr}^{-1}$ and the more stable fused-quartz TPW cells gave an average drift of $-2\,\mu\text{K}\cdot\text{yr}^{-1}$.

Keywords Isotopic concentration · ITS-90 · TPW (triple point of water) cell · Vienna standard mean ocean water (VSMOW) · Water impurities · Water triple point

1 Introduction

Based on the results of an international key comparison of triple-point-of-water (TPW) cells (CCT-K7) [1] and the subsequent CIPM Recommendation 2 (CI-2005) [2], a

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cooperative study between the National Institute of Standards and Technology (NIST) and the Hart Scientific Division of Fluke Corporation was undertaken in April 2005 to increase our understanding of the major influences on the TPW realization temperature. The results of the CCT-K7 for 21 participating national metrology institutes (NMIs) showed a disagreement of about ±85 μK, with only two NMIs making corrections for isotopic composition [1]. Motivated by this result and recent progress in understanding the effects of isotopic composition, the Technical Annex for the International Temperature Scale of 1990 (ITS-90) now specifies that the defined temperature of 273.16 K for the TPW applies when the liquid phase of water is of the same isotopic composition as that of Vienna standard mean ocean water (VSMOW) [2–4]. If all 21 NMI results in CCT-K7 are corrected for water isotopic composition, as determined from the estimated deviation in deuterium (D) concentration from VSMOW (δD) of the known sources of water used in the TPW cells, then the disagreement is reduced by about 40% to approximately ±60 μK [5,6]. The remaining disagreement is mainly attributable to the differences in impurities contained within the water of the TPW cells, though isotopic fractionation during manufacturing may be a contributor. This investigation explored how the use of the isotopic correction algorithm impacts the realization temperature difference between a group of TPW cells and compares those results with that of CCT-K7. Additionally, the study was designed to further our understanding of how the TPW realization temperature is affected by impurities introduced during manufacturing as well as the further dissolution (leaching) of impurities over time for different types of glass cells.

2 TPW Cell Measurements

2.1 TPW Cells

For this study, Hart Scientific manufactured five TPW cells with fused-quartz envelopes and three TPW cells with borosilicate glass envelopes [7]. Each TPW cell was filled with purified ocean water that was nominally equivalent in isotopic composition to VSMOW. The isotopic composition of the water contained in each of the manufactured TPW cells was analyzed and compared with that of the isotopic composition of the source water. One innovatively designed TPW cell of each type was manufactured with six blank ampoules formed as an integral part of the glass envelope; every six months, Hart Scientific filled one of the ampoules with cell water, flame sealed the ampoule neck to remove the ampoule, and then sent the sealed ampoule to NIST for impurity analysis (see [7] for a picture of the six ampoule TPW cell). The remaining six TPW cells were intercompared against NIST standards every six months from June 2005 through October 2006.

2.2 Isotopic Water Measurements

The VSMOW isotopic ratios are: 0.00015576 for ^{2}H/^{1}H, 0.0020052 for ^{18}O/^{16}O, and 0.0003799 for ^{17}O/^{16}O. Water isotopic compositions are usually reported as per mil ($^{0}\!/_{00}$) deviations from VSMOW. The TPW cell isotopic correction can be calculated by

$$T_{\text{meas}} - T_{90}(\text{TPW}) = A(\text{D})\delta\text{D} + A(^{17}\text{O})\delta^{17}\text{O} + A(^{18}\text{O})\delta^{18}\text{O}, \tag{1}$$

where $A(\text{D}) = 628\,\mu\text{K}$, $A(^{17}\text{O}) = 57\,\mu\text{K}$, and $A(^{18}\text{O}) = 641\,\mu\text{K}$ are specified in the Technical Annex [3], and the isotopic deviations from VSMOW are defined as

$$\delta\text{D} = \left[\frac{(\text{D/H})_{\text{sample}} - (\text{D/H})_{\text{VSMOW}}}{(\text{D/H})_{\text{VSMOW}}}\right], \tag{2}$$

and similarly for δ^{18}O and δ^{17}O.

Most water analysis facilities give results for δD and δ^{18}O and require calculation of δ^{17}O by

$$\delta^{17}\text{O} = \left(1 + \delta^{18}\text{O}\right)^{0.528} - 1. \tag{3}$$

The value of $A(^{17}\text{O})$ is small enough that the uncertainty in calculating δ^{17}O is considered negligible [3,4].

Due to the global meteoric water line correlation of the δD, δ^{18}O, δ^{17}O values for precipitated water (e.g., rain, snow), it is possible to use an approximate equation to estimate the isotopic correction value based on just the δD value by

$$[T_{\text{meas}} - T_{90}(\text{TPW})] \approx (712\ \delta\text{D} - 0.8)\,\mu\text{K}, \tag{4}$$

with an estimated uncertainty (k=1) of 5 μK [5,6,8].

Table 1 gives the isotopic composition and the impact on the triple-point realization temperature of the finished TPW cells using Eqs. 1 and 4. Reference [7] contains the isotopic results for the source ocean water and manufacturing stages (e.g., filtering, distillation, and degassing) and information on how the sampling is performed. For all water samples, the water was tested at the University of Utah Stable Isotope Ratio Facility for Environmental Research (SIRFER) facility. SIRFER is part of the United States Geological Survey (USGS) water-testing program. The SIRFER measurements are traceable to NIST water standards with measurement uncertainties (k=1) of 1‰ for δ^{18}O and 0.1‰ for δD. Using Eq. 1, these uncertainties (k=1) contribute approximately 2 μK to the TPW realization temperature. The uniquely designed TPW cells G5004 and Q5015 (see Sect. 2.2) were retained at Hart Scientific to provide NIST water samples for impurity testing.

For the eight TPW cells, the range in the isotopic correction is approximately ±5 μK. The average isotopic composition temperature correction between the fused quartz (0.6 μK) and borosilicate (1.5 μK) TPW cells is not significantly different. Additionally, the maximum difference between the isotopic corrections using Eqs. 1 and 4 does not exceed 1.0 μK. Based on the isotopic measurements for the completed TPW cells, the results indicate that the Hart Scientific TPW cell manufacturing process is well controlled and does not significantly change the overall isotopic composition of the source water (0.7 μK).

Table 1 Isotopic composition of the water used to manufacture the TPW cells and resultant corrections to the realized temperature

TPW cell	δD ($^0/_{00}$)	δ^{18}O ($^0/_{00}$)	ΔT (μK), ±2 μK using δD and δ^{18}O	$\Delta T \pm 5$ μK (μK) estimated using δD
G5002	5	0.3	3.3	2.8
G5003	0	−0.5	−0.3	−0.8
G5004	2	0.5	1.6	0.6
Q5005	0.3	−0.5	−0.1	−0.6
Q5008	2	−0.3	1.1	0.6
Q5009	7	0.6	4.8	4.2
Q5011	−7	−1.6	−5.5	−5.8
Q5015	5	−0.7	2.7	2.8

The TPW cells G5004 and Q5015 were retained at Hart Scientific for providing NIST water samples for impurity testing. The standard uncertainty of the isotopic determination for δD is $0.5^0/_{00}$ and $0.3^0/_{00}$ for δ^{18}O

2.3 Impurity Measurements

In an effort to understand the cause of the remaining realization temperature differences between a group of TPW cells after applying isotopic corrections (e.g., CCT-K7) and whether fused-quartz envelopes can improve the long-term stability of a TPW cell, the impurities contained in TPW cells were measured at NIST every six months, using the ampoule cells fabricated by Hart Scientific. Due to the lower OH–content in fused quartz, fused quartz was chosen over fused silica. It is noted that, in 2004, Rod White proposed that TPW cells be made from vitreous silica (fused silica) as a possible material for reducing the dissolution rates of impurities [9].

Figures 1 and 2 give the significant impurities found for the ampoules taken from the borosilicate TPW cell G5004 and fused-quartz TPW cell Q5015, respectively. The water samples were tested by Lee Yu of the NIST Analytical Chemistry Division for the existence of elemental impurities using inductively coupled plasma/mass spectrometry (ICP-MS). Further details on the NIST water impurity testing facility are found in [10]. For any given sample tested, the sum of all of the other elements found in the water contributed less than 0.1% of the total impurities. One of the borosilicate cell water samples was blind-tested twice to investigate the repeatability of the impurity measurements. The two results (given in mass fraction) were within 2 $\text{ng} \cdot \text{g}^{-1}$ of each other for the K and Na impurities and within 10 $\text{ng} \cdot \text{g}^{-1}$ of each other for the Si and B impurities.

As shown in Fig. 1 (results converted by binary analysis to molar fraction), the element K did not change for the four samples and Na changed by a factor of about 4.5 between the first two samples and then reached a steady state. The major impurity components found are elements inherently contained in borosilicate glass (e.g., B and Si). Over time, the quantity of B changed by a factor of four between the first two samples and then reached a steady state. More importantly, the quantity of Si is increasing at an estimated constant rate of 94 $\text{nmol} \cdot \text{mol}^{-1} \cdot \text{yr}^{-1}$ (Fig. 1 trendline). The dissolution rates of Si, B, and Na are similar to that found by Ledieu et al. in 2004 [11]. Furthermore, Ledieu et al. determined through the use of small-angle x-ray

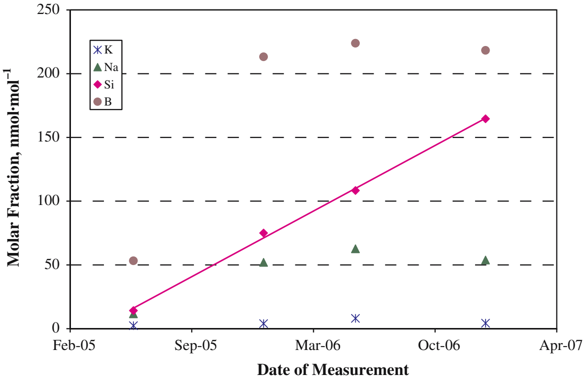

Fig. 1 Significant impurities from four borosilicate ampoules from TPW cell G5004. The line is a linear fit through the Si impurities equivalent to an increase of 94 nmol · $\text{mol}^{-1} \cdot \text{yr}^{-1}$

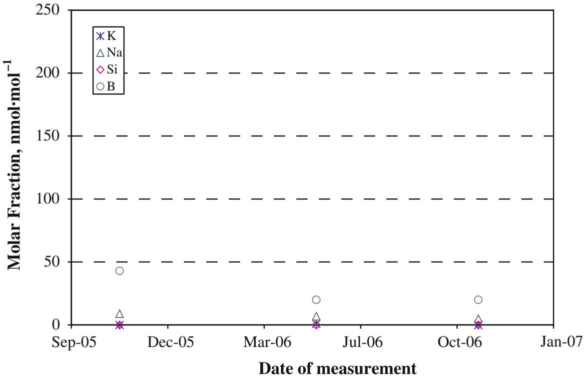

Fig. 2 Significant impurities from three fused-quartz ampoules from TPW cell Q5015

scattering that, due to the solubility of B and Na in water, an initial nanometer-scale porous network is formed at the borosilicate surface. This hydrated porous network can geometrically increase the borosilicate glass/water interfacial area, resulting in the creation of non-bridging Si bonds that allow the dissolution of Si over time. Additionally, the amount of boron oxide (B_2O_3) and sodium oxide (Na_2O) contained in the borosilicate glass strongly influences the duration of dissolution and whether the silica gel layer (passivation layer that prevents further dissolution) is reconstructed or not.

Using Raoult's law of dilute solutions for a binary water and Si solution analysis, and assuming that the Si is insoluble in the ice phase, this rate of Si contamination of 94 nmol · $\text{mol}^{-1} \cdot \text{yr}^{-1}$ is equivalent to $-10\,\mu\text{K} \cdot \text{yr}^{-1}$ [12]. This value is in close agreement with results published by Hill (NRC) that estimated the total impurity leaching effect for borosilicate envelope TPW cells to be approximately $-14\,\mu\text{K} \cdot \text{yr}^{-1}$ [13] and $-4\,\mu\text{K} \cdot \text{yr}^{-1}$ [14]. As discussed further in Sect. 3, it appears that Raoult's Law may be a good estimate for determining the impact on the TPW realization temperature for water-Si solutions.

The magnitude of leaching of Si from the borosilicate envelope into the water sample (shown in Fig. 1) is attributable to the cleaning technique applied to the borosilicate envelope [15]. Based on the large amount of impurities attributable to ocean salts and glass leaching in the first borosilicate ampoule, several suggestions were made to Hart Scientific to reduce these elements in future TPW cells fabricated from borosilicate. These changes were implemented for cells of serial number 5005 and greater.

As shown in Fig. 2, the fused-quartz envelope TPW cell gave water samples with significantly smaller amounts of ocean salt elements [K, Na ($<10\ \mathrm{nmol \cdot mol^{-1}}$)] and glass dissolution [Si, B ($<50\ \mathrm{nmol \cdot mol^{-1}}$)], compared to that of the borosilicate sample. The quantity of salt elements was a factor of 10 smaller for the fused-quartz TPW cell than that of the borosilicate TPW cell. Unlike the borosilicate ampoule samples, there does not appear to be any increased dissolution of impurities from the fused quartz into the water sample. The existing Si and B found in the water probably originated from the borosilicate still. The results imply that the fused-quartz envelope (shown in Fig. 2) is more stable than a borosilicate envelope (shown in Fig. 1).

The results given in Figs. 1 and 2 are considered to be indicators of the type, magnitude, and change with time of the dominant impurity species observed for each cell type. Section 3 explores whether these chemical analyses can reliably predict changes in the TPW cell realization temperature. Fused-silica envelope TPW cells were not tested. Fused silica is manufactured using a process that is different from that for fused quartz (e.g., OH–content), and impurities from a fused-silica envelope may differ substantially from the results presented here.

2.4 TPW Cell Comparison Measurements

The TPW cells were intercompared every six months (roughly corresponding to the water impurity analysis) to determine if any change in the impurity concentrations could be correlated with the differences in realization temperatures. The cells were tested by direct comparison with a standard platinum resistance thermometer (SPRT) measured with an Automatic Systems Laboratories (ASL) F900 resistance ratio bridge, as described in [16]. Uncertainties ($k=1$) in the direct comparisons between one of the test TPW cells and the reference cell are estimated to be $10\ \mu\mathrm{K}$. A description of the direct comparison uncertainty budget is found in [17]. The temperature realization drift in the NIST TPW cell 1041 due to a change in impurities is estimated to be $-5\ \mu\mathrm{K \cdot yr^{-1}}$. This drift is based on direct comparison measurements (made at least once per year since 1999) of the NIST TPW cell 1040 made against other NIST reference TPW cells, where for every direct comparison a new TPW cell and a previously compared TPW cell is measured. These ongoing comparisons allow the NIST TPW cells' realization temperatures to be linked to the CCT-K7 results. The results given in Fig. 3 and Table 2 (Sect. 3) reflect an adjustment for the temperature realization drift in the NIST TPW cell 1040.

In order to link these intercomparisons with the results of CCT-K7, all results are with respect to the CCT-K7 key comparison reference value (KCRV) through NIST TPW cell 1041. TPW cell 1041 was tested against TPW cell 1040 (the CCT-K7 NIST transfer cell) before and after the CCT-K7 measurements were performed. The

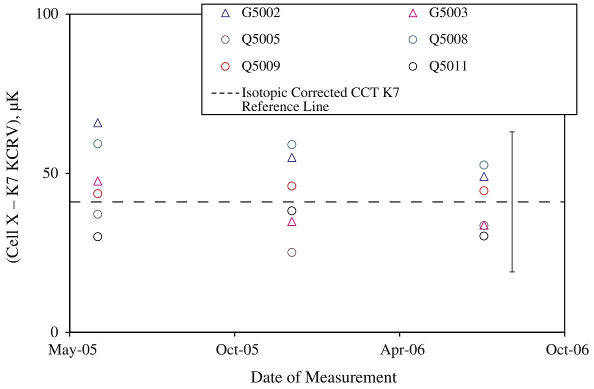

Fig. 3 Results of the six isotopically corrected TPW cells' (borosilicate and fused-quartz) realization temperatures with respect to the CCT-K7 KCRV (non-isotopic corrected value). The dashed line is the recalculated CCT-K7 reference line using estimated δD values for each CCT-K7 TPW cell. The uncertainty bar (k=1) is the combined uncertainty for the direct comparison measurements and linkage to CCT-K7

Table 2 Linear drift in TPW cell realization temperatures due to changes in impurities

	Borosilicate TPW cells			Fused-quartz TPW cells				
	G5002	G5003	Average	Q5005	Q5008	Q5009	Q50011	Average
Drift ($\mu K \cdot yr^{-1}$)	−14	−12	−13	−3	−6	1	0	−2
Std. dev. residual (μK)	1	3	3	6	2	1	5	4

combined uncertainty (k=1) for the direct comparison measurements and linkage to the CCT-K7 is estimated to be 22 μK. Additionally, the water isotopic composition was tested at the time of manufacture of both TPW cells 1040 and 1041. Since neither TPW cell 1040 nor 1041 contained VSMOW-equivalent water, each TPW cell required an isotopic adjustment of 72 μK. Figure 3 gives the results of the TPW cells (including 1041) with respect to the CCT-K7 KCRV (non-isotopic-corrected value). In this case, all of the TPW cell results are corrected to VSMOW isotopic values. In the calculation of the CCT-K7 KCRV, only 2 of the 21 TPW cells were corrected for isotopic variations from VSMOW [1]. Using estimated δD values for the known water sources [6] of the TPW cells (as described in Sect. 1) and using Eq. 4, we have obtained a new reference value for CCT-K7 by correcting all of the CCT-K7 TPW cell results for isotopic variation from VSMOW. The dashed line in Fig. 3 is the isotopically corrected CCT-K7 reference line, while the zero line is the CCT-K7 KCRV.

Figure 3 gives the results of the TPW cells with respect to the CCT-K7 KCRV (non-isotopic-corrected value). All of the results for the six tested TPW cells are isotopically corrected to VSMOW. The dashed line is the recalculated CCT-K7 reference line using estimated δD values for each CCT-K7 TPW cell. The combined uncertainty (k=1) for the direct comparison measurements and linkage to the CCT-K7 is estimated to be 22 μK.

3 Correlations

As evidenced in Fig. 3, the realization temperatures of the six TPW cells changed as a function of time relative to the CCT-K7 KCRV. For all six of the TPW cells, the range is 56 μK, 49 μK, and 41 μK for the June 2005, January 2006, and August 2006 measurements, respectively. The average difference between the borosilicate and fused-quartz TPW cells is 12 μK, 2 μK, and 3 μK for the June 2005, January 2006, and August 2006 measurements, respectively. This decrease in the range of TPW realization temperature values is attributed to the change in the realization temperatures of the borosilicate TPW cells due to the change in Si impurity concentration, while the fused-quartz TPW cell realization temperatures are nearly constant in time. Analysis of the direct comparison results shows that the realization temperature drifts in the borosilicate TPW cells are larger than those of the fused-quartz cells. Table 2 gives the linear fit for all six TPW cells and the average for each TPW cell-type. The average drift is $-13\ \mu\text{K}\cdot\text{yr}^{-1}$ for the borosilicate TPW cells and $-2\ \mu\text{K}\cdot\text{yr}^{-1}$ for the fused-quartz TPW cells. The drift in the borosilicate TPW cells is close to the value of $-14\ \mu\text{K}\cdot\text{yr}^{-1}$ from [14]. Both of the borosilicate TPW cells showed decreasing drifts that were nearly identical, while the fused-quartz TPW cells showed drifts that were increasing, decreasing, and zero.

An attempt was made to correlate the direct comparison drifts in the TPW cell realization temperatures with an estimated temperature realization drift based on water sample impurity analysis. An estimated overall change of $-41\ \mu\text{K}$ for the borosilicate TPW cells and 3 μK for the fused-quartz TPW cells from June 2005 to January 2007 results from applying Raoult's law of dilute solutions and the data from Figs. 1 and 2 to a binary analysis of the impurities (K, Na, B, and Si).

For the borosilicate TPW cell water sample, only the Si impurity is increasing at a constant $94\ \text{nmol}\cdot\text{mol}^{-1}\cdot\text{yr}^{-1}$, which is equivalent to an estimated temperature drift of $-10\ \mu\text{K}\cdot\text{yr}^{-1}$. The other impurities (K, Na, and B) reached a steady state after the first six months. The estimate of $-10\ \mu\text{K}\cdot\text{yr}^{-1}$ for Si from the simple binary analysis is in close agreement with that of the direct comparison measurement results of $-13\ \mu\text{K}\cdot\text{yr}^{-1}$ as well as previous published values of $-14\ \mu\text{K}\cdot\text{yr}^{-1}$ and $-4\ \mu\text{K}\cdot\text{yr}^{-1}$ [13,14]. Based on these results, it is clear that using Raoult's law of dilute solutions as a correction algorithm for Si impurity concentrations is a possibility that merits further exploration. The ICP-MS detection of Si works well only if the water source is pure and if multiple tests are performed over time to generate relative differences as a function of time [18–20].

For the fused-quartz TPW cell water sample, the impurity results from Fig. 2 show no obvious leaching of any impurities and in Table 2 there is no clear pattern in the realization temperature drifts. Based on the drift-rate results determined from the direct comparisons and the lack of any significant change in impurity concentrations, the realization temperatures of TPW cells manufactured with properly cleaned fused-quartz envelopes appear more stable than those of TPW cells manufactured with borosilicate envelopes.

4 Conclusions

Based on the results of the direct comparisons, the use of the isotopic correction algorithm decreased the differences in the realization temperatures of the VSMOW-filled TPW cells. For TPW cells with isotopically characterized water, the uncertainty of the correction is small enough to insignificantly impact the overall temperature realization uncertainty budget. For TPW cells with no isotopic analysis, the results based on only the δD value can be used to make a correction to the realization temperature with a slightly larger increase in uncertainty.

The TPW cell realization temperature drift as a function of time due to impurities leaching from the glass envelopes was estimated to be $-13\ \mu\mathrm{K}\cdot\mathrm{yr}^{-1}$ for the borosilicate TPW cells and $-2\ \mu\mathrm{K}\cdot\mathrm{yr}^{-1}$ for the fused-quartz TPW cells. These results are in close agreement with estimates based on the ICP-MS impurity results of $-10\ \mu\mathrm{K}\cdot\mathrm{yr}^{-1}$ for the borosilicate TPW cells. NIST will continue to directly compare a subset of the six TPW cells over the next several years to determine if the drift rate is constant and if the fused-quartz envelope TPW cells continue to be more stable than the borosilicate envelope TPW cells.

Further work with intentionally doped water samples is needed to empirically understand how the individual impurities impact the realization temperature of the TPW. However, it is useful to test the water for impurities to understand what impurities are added during the manufacturing process (e.g., cleaning procedure) and whether any improvements should be implemented in that process. For example, as shown in results from the water impurity analysis of the fused-quartz ampoules, appropriate actions were taken in the present Hart Scientific TPW cell manufacturing process to minimize the amount of impurities in the water contained in the TPW cells.

Without an easy and accurate way to test the TPW cell water for an increase in impurities as a function of time, we suggest that a gallium triple-point cell replace the TPW as a reference point for SPRT resistance measurement in the next temperature scale. The gallium triple-point (Ga TP) cell has intrinsic advantages over a TPW cell with respect to isotopic variations and gallium is unlikely to react with a polytetrafluoroethylene (PTFE) container [21,22]. As an enhancement of the planned continuance of testing the TPW cells, NIST will incorporate the Ga TP into the direct comparison measurements.

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Disclaimer

Certain commercial equipment, instruments or materials are identified in this paper in order to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the NIST.

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