EFFECT OF COVARIANCES IN A DENSITY MEASUREMENT OF SILICON SAMPLES BY HYDROSTATIC WEIGHING

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Abstract: A density standard realized by 1 kg silicon spheres has been used for measuring the density of solid samples by hydrostatic weighing. Details are given for the structure of the measurement system, procedure for the solid density measurement, and uncertainty evaluation. The density of a 1 kg silicon sample has been measured with a relative combined standard uncertainty of 1.2 parts in 10^7. When the effect of covariances in input quantities is taken into account, the relative combined uncertainty in measuring the density difference between the silicon spheres and the silicon sample may be reduced to 3.6 parts in 10^8. Uncertainty sources in the hydrostatic weighing and the effect of covariance in the input quantities are discussed.

Keywords: density standard, silicon crystal, hydrostatic weighing, correlation, covariance.

1. INTRODUCTION

Hydrostatic weighing is one of reliable methods for measuring the density of gases, liquids, and solids. Water is the most common material used for a density standard. Since the density of water is dependent on its isotopic compositions, Standard Mean Ocean Water (SMOW) is usually chosen for specifying isotopic compositions of water. The relative expanded uncertainty (k = 2) for the density of water having the same isotopic compositions with those of SMOW is estimated to be 8-9 parts in 10^7 [1].

Through a research for the determination of the Avogadro constant, \( N_A \), by the X-Ray Crystal Density (XRCD) method, the density of a 1-kg silicon sphere has been determined with a relative standard uncertainty of 7 parts in 10^7 [2], being much less than that of water. Such a silicon sphere is therefore useful for measuring the density of solid samples by hydrostatic weighing.

In the hydrostatic weighing system described here [3, 4], two 1-kg silicon spheres are used as a density standard. A 1 kg silicon sample is placed between the two standards. When the mass of the sample is measured relative to one of the silicon spheres, their masses are correlated. Since the diameters of the two silicon spheres were measured by optical interferometry, their volumes are also correlated. In this study the effect of the correlations has been taken into account for accurately evaluating the uncertainty in the density measurement by hydrostatic weighing.

2. HYDROSTATIC WEIGHING SYSTEM

Figure 1 shows a hydrostatic weighing system developed in this study. Two silicon spheres, S4 and S5, are used as the solid density standards. Their volumes have already been calibrated by optical interferometry [5-8]. Tridecane \( \text{(n-C_{13}H_{28}, \rho \approx 756 \text{ kg/m}^3 \text{ at } 20 \degree C)} \) is used as a working liquid, in which the two silicon spheres and the solid sample under study are weighed alternately with an electronic balance using a weight exchange mechanism. The reason for using the two silicon spheres is to cancel the effect of vertical density gradient in the liquid, which may be introduced by the temperature gradient and the gravity [9]. The two silicon spheres and the solid sample are independently...
placed on and removed from the cage by the weight exchange mechanism. As long as the level of the liquid is kept constant during the weight exchange, the buoyancy force acting on the cage can be canceled.

2.1. Procedure for the density measurement

When S4 is weighed in the liquid, as shown in figure 2, its apparent mass in the liquid is about 675 g because of the buoyancy force acting on the sphere. In order to operate the balance near 1 kg, a stainless steel weight with a mass of 325 g is placed on the balance pan with the automatic handler. At this state, the balance reading, \( B_{\text{in}} \), being a difference from 1 kg, is recorded. The sphere and the weight are then removed from the balance, and a 1 kg stainless steel weight is placed on the balance pan with the automatic handler, as shown in figure 3. At this state, the balance reading, \( B_{\text{in}} \), being a difference from 1 kg, is recorded. The relation of the forces acting in this system are therefore summarized to be

\[
\begin{align*}
\text{Balance reading when S4 is weighed in the liquid, } & \quad B_{\text{in}} = m_{325} - \rho_{\text{in,S4}}(t)V_{\text{S4}}(t)g_{\text{bal}} + m_{325} g_{\text{bal}} - \rho_{\text{at}}(t)\rho_{\text{at}}(t)g_{\text{bal}} - K_{\text{SS}} g_{\text{bal}} \\
& = m_{325} g_{\text{bal}} - \rho_{\text{at}}(t)\rho_{\text{at}}(t)g_{\text{bal}} - K_{\text{SS}} g_{\text{bal}}
\end{align*}
\]

where \( m_{325} \) and \( m_{1000} \) are the masses of the sphere S4, 325 g weight, and 1 kg weight, respectively, \( \rho_{\text{in,S4}}(t) \) and \( \rho_{\text{at}}(t) \) are the density of the liquid near the silicon sphere S4 at temperature \( t \) and density of air at temperature \( t_{\text{at}} \), respectively, \( V_{\text{S4}}(t) \) the volume of the sphere S4 at temperature \( t \), \( V_{\text{325}}(t) \) and \( V_{\text{1000}}(t) \) the volumes of the 325 g and 1 kg weights at temperature \( t_{\text{at}} \), respectively, \( g_{\text{bal}} \) and \( g_{\text{at}} \) the local accelerations due to gravity at the levels of silicon sphere S4 and the balance, respectively, and \( K \) the balance sensitivity. Since the vertical distance between the balance and the sphere S4 is about 1 m, the gravity gradient coefficient, \( C_{\text{SS}} = g_{\text{at}}/g_{\text{S4}} \), is estimated to be 0.999 9997.

In equation (1), the balance sensitivity \( K \), which is the relationship between the force and the balance reading, may be determined by loading and unloading a calibrated weight on the balance pan with the automatic handler. From equation (1), the liquid density near S4 is determined as

\[
\begin{align*}
\rho_{\text{liq,S4}}(t) &= (m_{325} + C_{\text{SS}}(m_{325} - m_{1000}) + K_{\text{SS}}(B_{\text{in}} - B_{\text{S4}})) \\
& \quad + C_{\text{SS}}\rho_{\text{at}}(t_{\text{at}})\left[V_{\text{325}}(20^\circ\text{C}) - V_{\text{325}}(20^\circ\text{C})\right] \\
& \quad \times [1 + \beta_{\text{SS}}(t_{\text{at}} - 20^\circ\text{C})] [V_{\text{S4}}(20^\circ\text{C})] \\
& \quad \times [1 + 3\alpha_{\text{SS}}(t - 20^\circ\text{C})]^{-1}
\end{align*}
\]

where \( \alpha_{\text{SS}} \) is the linear thermal expansion coefficient of silicon crystals, and \( \beta_{\text{SS}} \) the bulk thermal expansion coefficient of the stainless steel weight.

When S5 is weighed in the liquid, the liquid density near S5 is similarly given by

\[
\begin{align*}
\rho_{\text{liq,S5}}(t) &= (m_{325} + C_{\text{SS}}(m_{325} - m_{1000}) + K_{\text{SS}}(B_{\text{in}} - B_{\text{S5}})) \\
& \quad + C_{\text{SS}}\rho_{\text{at}}(t_{\text{at}})\left[V_{\text{325}}(20^\circ\text{C}) - V_{\text{325}}(20^\circ\text{C})\right] \\
& \quad \times [1 + \beta_{\text{SS}}(t_{\text{at}} - 20^\circ\text{C})] [V_{\text{S5}}(20^\circ\text{C})] \\
& \quad \times [1 + 3\alpha_{\text{SS}}(t - 20^\circ\text{C})]^{-1}
\end{align*}
\]

where \( m_{325} \) is the mass of the silicon sphere S5, \( B_{\text{SS}} \) the balance reading when S5 is weighed in the liquid, \( V_{\text{SS}}(t) \) the volume of S5 in the liquid at temperature \( t \), and \( C_{\text{SS}} = g_{\text{at}}/g_{\text{SS}} \) with \( g_{\text{SS}} \) being the acceleration due to gravity at the level of S5. From equations (2) and (3), the liquid density near a solid sample A is determined, assuming that any vertical density gradient in the liquid is linear, with

\[
\rho_{\text{liq,A}}(t) = \frac{\rho_{\text{liq,S4}}(t) + \rho_{\text{liq,S5}}(t)}{2}
\]

When a 1-kg silicon sample A under study is weighed in the liquid, its volume at a temperature \( t \) is given by
Step 6a For measuring the density of a 1 kg silicon crystal, the balance reading when the sample A is weighed in the liquid, and the density of the sample A at temperature \( t \) is therefore given by

\[
\rho_A(t) = \frac{m_A}{V_A(t)}.
\]  

The density of the sample A under study is thus measured with respect to those of the silicon spheres. When the apparent mass of the solid sample in the liquid is different from 675 g, a different weight is placed on the balance so that the balance is operated near 1 kg. The procedure for a single density measurement of a solid sample therefore consists of the following steps:

Step 1 Measurements of the air temperature, pressure, and humidity for determining the air density \( \rho_{\text{air}} \). Measurement of the liquid temperature \( t \) and the pressure of the liquid.

Step 2 Calibration of the balance sensitivity \( K \) by loading and unloading a calibrated weight on the balance.

Step 3 Loading a 1 kg weight on the balance with \( \rho_{\text{air}} \) being the acceleration due to gravity at the level of sample A. The density of the sample A at temperature \( t \) is therefore given by

\[
\rho_A(t) = \frac{m_A}{V_A(t)}.
\]  

Step 4 Unloading the 1 kg weight and loading 325 g weight on the balance. Loading the silicon sphere \( S4 \), and recording balance reading \( B_{S4} \). This reading is used as a zero point in the hydrostatic weighing.

Step 5 Unloading \( S4 \) and loading \( S5 \) in the cage. Record the balance reading \( B_{S5} \) for determining the density \( \rho_{\text{liquid}} \) near \( S5 \).

Step 6a For measuring the density of a 1 kg silicon crystal, unloading \( S5 \) and loading the sample A in the cage. Recording the balance reading \( B_A \) for determining the sample density \( \rho_A \) at temperature \( t \).

Step 6b When the apparent mass of the sample A in the liquid is different from 675 g, unloading the 325 g weight and loading a different weight on the balance so that the balance is operated near 1 kg. Unloading \( S5 \) and loading the sample A in the cage. Recording the balance reading \( B_A \) for determining the sample density \( \rho_A \) at temperature \( t \).

For completing these steps 1 to 6, it usually takes 20 minutes. This system is presently used at the NMJJ for calibrating the density of silicon crystals and solid samples, such as stainless steel weights, glasses, metals, and polymers.

An example of density measurements of a 1 kg silicon crystal is shown in figure 4. A total of 61 density measurements were conducted in this example. Each data point was deduced from the procedure with steps 1 to 6. As can be seen from the figure, a very stable result was obtained for a total of about 20 hours. The density of the sample A was thus measured with a relative standard deviation of 1.8 \( \times 10^{-7} \).

### 2.2. Uncertainty in the hydrostatic weighing

When equations (2) to (6) used for deducing the density of a solid sample, \( \rho_A \), are expressed as a function with \( N \) input quantities

\[
\rho_A = f(x_1, x_2, \cdots, x_7)
\]

the combined variance of \( \rho_A \) is generally given by

\[
u^2(\rho_A) = \sum_{i=1}^{N} \left( \frac{\partial f}{\partial x_i} \right)^2 u^2(x_i) + 2 \sum_{i=1}^{N} \sum_{j=i+1}^{N} \frac{\partial f}{\partial x_i} \frac{\partial f}{\partial x_j} u(x_i) u(x_j)
\]

where \( u(\rho_A) \) is the combined standard uncertainty of \( \rho_A \), \( u(x_i) \) the standard uncertainty of the input quantity \( x_i \), and \( u(x_i) u(x_j) \) the covariance between the two input quantities \( x_i \) and \( x_j \). In equation (8), the first and second terms therefore correspond to the variance and covariance components, respectively [10].

In general, the variances for all input quantities may be evaluated for estimating \( u(x_i) \), but it is rather difficult to evaluate all covariances between pairs of so many input quantities. In this work, the effect of dominant covariances are therefore evaluated: one is the covariance between the masses of the two silicon spheres, \( m_{S4} \) and \( m_{S5} \), and the other is the covariance between their volumes, \( V_{S4} \) and \( V_{S5} \). In the example of density measurement shown in figure 4, the mass of the 1 kg silicon sample was measured with respect to that of \( S4 \). This means that the mass of sample A, \( m_A \), is strongly correlated to \( m_{S4} \) and \( m_{S5} \). Following correlation coefficients were therefore taken into account for estimating the combined standard uncertainty in the density measurement:

\[
r(m_{S4}, m_{S5}) = u(m_{S4})u(m_{S5})/u(m_{S4})u(m_{S5})\]

\[
r(V_{S4}, V_{S5}) = u(V_{S4})u(V_{S5})/u(V_{S4})u(V_{S5})\]

\[
r(m_A, m_A) = u(m_A)^2/\{u(m_A)^2\}\]

\[
r(m_A, m_A) = u(m_A)^2/\{u(m_A)^2\}\]
These correlation coefficients are all positive in this case. In stead of analytical evaluation given in equation (8), the combined standard uncertainty may be deduced from numerical evaluation [10]. When a change in \( \rho_A \) due to a change in an input quantity \( x_i \) is expressed as

\[
Z_i = [f(x_1, \ldots, x_i + u(x_i), \ldots, x_N)] - f(x_1, \ldots, x_i - u(x_i), \ldots, x_N)] / 2
\]

the combined standard uncertainty in \( \rho_A \) is numerically obtained as

\[
u^2(\rho_A) = \sum_{i=1}^{N} Z_i^2 + 2\sum_{i<j=1}^{N} Z_i Z_j r(x_i, x_j)
\]

From equation (14), the combined standard uncertainty was estimated in this study.

One of difficulties in evaluating the uncertainty in the hydrostatic weighing described here is how to estimate the effect of non-linear density gradient in the liquid. As can be seen in equation (4), the density of the liquid near the solid sample, \( \rho_{A-L} \), may not be determined accurately if the vertical temperature gradient in the liquid has a relatively large non-linearity. In order to evaluate the effect, the differences in the measured solid densities determined from the hydrostatic weighing and from the pressure-of-flotation method [11, 12] were compared. Results of the measurements for five samples have shown that the relative difference in the densities determined by the two independent methods was as small as \( 3.0 \times 10^{-8} \) with a standard deviation of \( 3.3 \times 10^{-8} \). Since the result from the pressure-of-flotation method is considered to be almost free from the non-linear gradient in the liquid density, this difference is considered to be the magnitude of possible non-linear effect in the hydrostatic weighing.

Table 1 lists influence quantities in the hydrostatic weighing of the 1 kg silicon crystal. Major influence factors are attributed to the volume uncertainties, \( u(V_{SA}) \) and \( u(V_{SB}) \), and also to their covariance, \( u(V_{SA}, V_{SB}) \). It should be noted that the effects of covariances \( u(m_{SA}, m_{SB}) \) and \( u(m_{SB}, m_{SA}) \) on \( u(\rho_A) \) are negative because the sensitivity coefficient \( \partial f / \partial m_{SA} \) is negative and those for \( \partial f / \partial m_{SB} \) and \( \partial f / \partial m_{SA} \) are positive. The covariance between the masses of silicon sphere and the solid sample therefore contributes to reduce the combined standard uncertainty in \( \rho_A \), but the contribution is trivial in this case because the major uncertainty comes from the uncertainty in the volumes of S4 and S5. In this example, the density of the 1 kg silicon sample, \( \rho_A \), was thus determined with a relative combined standard uncertainty of \( 1.2 \times 10^{-8} \).

Table 2 shows an uncertainty evaluation for the same solid sample. In this case, the combined standard uncertainty of the density difference, \( \Delta \rho = \rho_A - (\rho_{SA} + \rho_{SB})/2 \), is evaluated. Evaluation of such a density difference is especially useful for determining the Avogadro constant, where the densities of many silicon samples are measured with respect to those of reference silicon spheres [2, 13]. As can be seen in the table, the uncertainty in the volumes, \( u(V_{SA}) \) and \( u(V_{SB}) \), does not contribute to the uncertainty in \( \Delta \rho \). Consequently, the density difference \( \Delta \rho \) was determined with a relative combined standard uncertainty of \( 3.6 \times 10^{-8} \). If the covariances between the masses \( m_{SA}, m_{SB}, m_{A} \), and \( m_A \) are not taken into account, the relative combined standard uncertainty increases to \( 5.4 \times 10^{-8} \), showing importance of evaluating covariances in input quantities. If the effect of non-linear gradient in the liquid density is

<table>
<thead>
<tr>
<th>Uncertainty source</th>
<th>( u(x_i) )</th>
<th>( Z_i^2/(kg/m^3)^2 ) or 2Zr( (x_i, x_j)/(kg/m^3)^2 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass</td>
<td></td>
<td></td>
</tr>
<tr>
<td>( m_{SA} )</td>
<td>16.0 ( \mu g )</td>
<td>0.33 \times 10^{-4}</td>
</tr>
<tr>
<td>( m_{SB} )</td>
<td>16.0 ( \mu g )</td>
<td>0.33 \times 10^{-4}</td>
</tr>
<tr>
<td>Covariance between ( m_{SA} ) and ( m_{SB} )</td>
<td></td>
<td>0.64 \times 10^{-4}</td>
</tr>
<tr>
<td>( m_{S000} )</td>
<td>28.0 ( \mu g )</td>
<td>0.00 \times 10^{-4}</td>
</tr>
<tr>
<td>( m_{S25} )</td>
<td>51.0 ( \mu g )</td>
<td>0.00 \times 10^{-4}</td>
</tr>
<tr>
<td>( m_A )</td>
<td>16.4 ( \mu g )</td>
<td>0.63 \times 10^{-4}</td>
</tr>
<tr>
<td>Covariance between ( m_A ) and ( m_{SA} )</td>
<td></td>
<td>(-0.88 \times 10^{-4})</td>
</tr>
<tr>
<td>Covariance between ( m_A ) and ( m_{SB} )</td>
<td></td>
<td>(-0.85 \times 10^{-4})</td>
</tr>
<tr>
<td>Volume</td>
<td></td>
<td></td>
</tr>
<tr>
<td>( V_{SA} )</td>
<td>0.000 052 cm(^3)</td>
<td>1.99 \times 10^{-4}</td>
</tr>
<tr>
<td>( V_{SB} )</td>
<td>0.000 064 cm(^3)</td>
<td>3.01 \times 10^{-4}</td>
</tr>
<tr>
<td>Covariance between ( V_{SA} ) and ( V_{SB} )</td>
<td></td>
<td>2.12 \times 10^{-4}</td>
</tr>
<tr>
<td>( V_{S000} )</td>
<td>0.020 cm(^3)</td>
<td>0.00 \times 10^{-4}</td>
</tr>
<tr>
<td>( V_{S25} )</td>
<td>0.025 cm(^3)</td>
<td>0.00 \times 10^{-4}</td>
</tr>
<tr>
<td>Non-linear liquid density gradient</td>
<td></td>
<td>0.49 \times 10^{-4}</td>
</tr>
<tr>
<td>Standard deviation of the mean</td>
<td></td>
<td>0.17 \times 10^{-4}</td>
</tr>
</tbody>
</table>

\( u_A(\rho) \) | 0.000 28 kg/m\(^3\) |

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<tr>
<th>Uncertainty source</th>
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<td>0.15 \times 10^{-4}</td>
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</tr>
<tr>
<td>Covariance between ( m_A ) and ( m_{SA} )</td>
<td></td>
<td>(-0.59 \times 10^{-4})</td>
</tr>
<tr>
<td>Covariance between ( m_A ) and ( m_{SB} )</td>
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</tr>
<tr>
<td>Volume</td>
<td></td>
<td></td>
</tr>
<tr>
<td>( V_{SA} )</td>
<td>0.000 052 cm(^3)</td>
<td>0.00 \times 10^{-4}</td>
</tr>
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</tbody>
</table>

\( u_A(\Delta \rho) \) | 0.000 084 kg/m\(^3\) |
removed, the relative combined standard uncertainty in the measurement of $\Delta \rho$ further reduces to $2.0 \times 10^{-8}$.

Table 3 lists influence quantities in the density measurement of a 1 kg stainless steel. Since the apparent mass of this sample in the liquid was about 900 g, a 100 g weight with mass $m_{100}$ and a volume $V_{100}$ was placed on the balance during the hydrostatic weighing of the sample. As can be seen in this table, the number of influence quantities is much more than that for density measurements of 1 kg silicon crystals because different weights were used in the hydrostatic weighing: 325 g weight for the two silicon spheres and 100 g weight for the solid sample. In this case, the largest uncertainty source lies in the uncertainty in the air density measurement. The density of the 1 kg stainless steel weight, $\rho_s$, was thus determined with a relative combined standard uncertainty of $1.2 \times 10^{-6}$. Precise measurements of the volumes of stainless steel weights are especially important for calibrating the volumes of air-density artifacts [14]. They have nearly the same masses, but different volumes. By weighing them in air, the density of air is directly measured from their apparent masses in air, providing much better uncertainty than that obtained from air pressure, temperature, and humidity measurements [15].

Table 4 lists examples of density measurements for different materials. Solid samples with different masses and densities have been measured by the hydrostatic weighing system described in section 3.1. The uncertainty in the density measurement depends on the mass and density of each sample, but the uncertainty in the volume is almost the same for any solid samples except for 1 kg silicon crystals. As can be seen from equation (5), hydrostatic weighing determines the volume of the sample from mass measurement in air and from apparent mass measurement in liquid. Since the uncertainty in the mass of a combination of weights is almost the same for any measurement described here, the combined standard uncertainty in the volume determination of the solid sample is almost equal to or less than 0.0002 cm$^3$ = 0.2 mm$^3$. This means that when the volume of the solid sample is larger, better uncertainty is obtained.

3. DISCUSSIONS

In this work, examples of density measurements for silicon crystals, metals, glasses, and polymers have been presented. Best uncertainties were obtained for 1 kg silicon crystals because their mass, volume, and thermal expansion coefficient are nearly the same with those of silicon spheres used as a solid density standard. Uncertainty evaluations for 1 kg silicon crystals have therefore shown that $u_r(\rho) = 1.2 \times 10^{-8}$ and $u_r(\Delta \rho) = 3.6 \times 10^{-8}$. For other materials, the uncertainty depends on their mass and density, but the combined standard uncertainty in their volume determination, $u_r(V)$, is approximately equal to or less than 0.2 mm$^3$.

Such a capability in the density measurement is presently used at the NMIJ for the determination of the Avogadro constant, $N_A$, by the x-ray crystal density (XRCD), where both the hydrostatic weighing method and the pressure-of-flotation method are used for measuring density differences between silicon crystals.

In the field of material science, density measurements for solid samples with small volumes, such as 1 cm$^3$ or less, are often needed. Further reduction of uncertainty in the volume determination is therefore being considered at the NMIJ for measuring the density of small samples. As can be seen in figure 7, one of the serious uncertainty sources lies in a scatter in the density measurement. The most serious cause of this scatter is attributed to the instability in the effect of meniscus at the suspension rod. One of
possible ways to reduce the scatter is to implement a magnetic coupling used in magnetic suspension densimeters [16-20]. The magnetic suspension densimeter is a device developed to measure the density of fluids. When the coil suspended from the balance is kept in air and the permanent magnet is kept in the liquid, the buoyancy forces acting on solid materials in the liquid may be measured without the effect of meniscus. Such a principle will be useful for measuring the volume and density of new materials.

REFERENCES


