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Dissemination of Pt-Ir k-112 mass measurement standards to E0-74 working standards with sub-divisional methods

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Abstract: The mass value of the weights that are the mass unit's standard must be determined correctly, especially for E_0 no 74 as the national working standard. Sub-division is the method commonly used to determine the mass of these weights. The sub-division method involved three standard weights in this study, namely Pt-Ir K-112, E_0 no 74, and E_0 no 75. Pt-Ir was used as the measuring standard, while the E_0 class weights were test weights and check standard. The mass value of both weights was determined based on the mass value of Pt-Ir, which was analyzed using the weighted least squares method. The calibration obtained that the mass values of E_0 no 74 and E_0 no 75 were 1000,000046 g and 1000,000163 g, respectively. In measurement, the uncertainty value becomes a very important part of being stated. The uncertainty obtained for both E_0 weights is 0.036 mg.

Keywords: measurement, mass, sub-division method, Pt-Ir, measurement uncertainty.

1. Introduction

The use of weights and scales as tools for measuring mass for trade dates back thousands of years. Since then, mass standards and technology for weighing and measuring mass have evolved rapidly to meet society's ever-evolving and changing needs. In addition to its direct impact on commerce, mass measurement affects the scientific community and many manufacturing industries, including aerospace, aircraft, automotive, chemical, semiconductor, materials, nuclear, pharmaceutical, construction, and instrument manufacturing.

Many attempts have been made to create a uniform weighing system. However, it was not until 1875 that 17 countries signed the meter convention, which established the basis of the international system of units (SI) which ultimately provided the long-sought uniformity in weighing and measuring standards (Jabbour and Yaniv, 2001).

After the meter convention was finally approved an internationally recognized measurement system. The result is the creation of the first two measuring standards, namely the standard length and mass. For the scope of mass measurement, the highest standard recognized at that time was in the form of artefacts made of 90% Platinum and 10% Iridium alloys which were known as the International Prototype of Kilograms (IPK). After the IPK was chosen as the standard for measuring mass as the highest, copies were distributed to various countries as the national standard and then disseminated to the customers. This scheme also applies in Indonesia, which currently has a copy of Pt-Ir K-112 as the standard for measuring mass units with the highest caste. The mass value of the standard is disseminated through a long chain of dissemination involving the SNSU-BSN as the owner of the standard, calibration, or measurement laboratories that have mass standards with various accuracy classes until finally used by end-users in multiple fields.

As the highest standard, Pt-Ir is not used in calibration services carried out by SNSU-BSN. If this is done, the potential for changes to this standard value will be very large due to various types of contamination when used. For this reason, this standard is kept under strict conditions. Because of these conditions, another standard (referred to as working standard) is required whose mass value has been determined from this standard. This working standard is in the form of weights with an accuracy class of E_0. This article explains how disseminating mass values from a Pt-Ir to E0 weights no 74 using the sub-division method. This method involves another E_0 weight as a check standard. These weights are applied during dissemination to guarantee the quality of the measurements carried out.

1.1. Mass Traceability in Indonesia

A country will usually have an institution or agency responsible for providing a chain of traceability of units to primary standards. In Indonesia, this is regulated by law no. 14 of 2014 (Law of the Republic of Indonesia No. 20 of 2014, 2014). Following the above article, BSN has the duties and functions to manage the national standard of measurement units. Which is more specifically this task is carried out by the Directorate of National Standards for Units of Measurement (SNSU-BSN). In its role to ensure the traceability of measuring units, many activities are carried out, including calibration services, research, and the development of measuring standards. One of the units managed by SNSU-BSN is the mass unit.

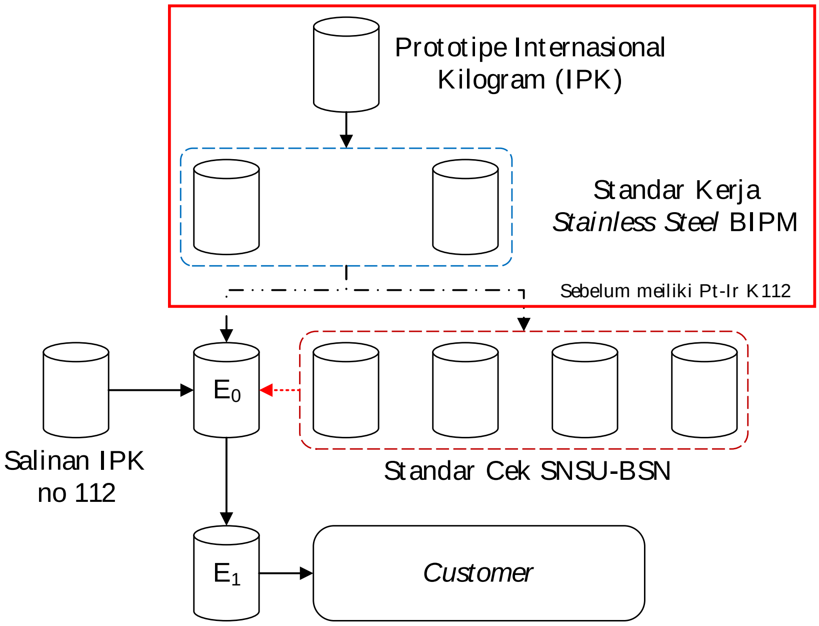

Figure 1. Mass unit traceability before and after possession of Pt-Ir K-112

Traceability of mass units is currently guaranteed through a national standard in the form of E_0 class weights which are checked or calibrated periodically to the BIPM every five years. In 2019 SNSU-BSN was given the mandate to manage a new standard, namely Pt-Ir.

The highest working standards belonging to SNSU-BSN in the form of E_0 class weights are traceable to the official copy of the IPK through two stainless steel work standards belonging to BIPM, which have been calibrated to the official copy of the IPK. With traceable values from the BIPM, SNSU-BSN disseminates mass values from work standards to industry, research institutions as well as calibration and testing laboratories. To reduce dependence on BIPM, through this study the E_0 weights were calibrated independently by SNSU-BSN using Pt-Ir K-112. The new traceability chain can be created by eliminating some sections in the mass traceability diagram in Figure 1.

1.2. Sub-Division Wealing

Many weighing designs are used to calibrate loads or weights in mass metrology. The weighing design depends on the accuracy class of weight being calibrated and the desired uncertainty requirements. The weighing design commonly used in SNSU-BSN for calibration of weights with high accuracy is the sub-division method. This method involves a set of weights that are compared according to a certain scheme. The weighing scheme involves at least two standards. The standard is compared against a group of loads with the same nominal value and various combinations of weights. Standard weights are treated like other test weights, added to each calibration scheme so that they can be used to verify values for the entire set of weights (Bell, 2004; Vâlcu and Dinu, 2009; Zelenka, 2009).

(a) (b)

Figure 1. (a) Pt-Ir national standard, and (b) E_0 class mass work standard no. 74.

In the dissemination of weights with this method used for the same nominal mass, this method is usually called the closed cycle method. This method involves three weights with the same nominal, which are all compared. The weight involved is standard, with two weights as test weights. In a case like this, one of the test weights is a mass standard used for quality assurance of measurements. These weights are always involved in every calibration process and stored again to stabilize the mass value. This stable mass value is used to check the measurement results. If an anomaly occurs when the measurement is carried out, the mass value of this check standard will shift.

The precision measurements carried out by SNSU-BSN always involve these check weights. For E_0 class weights, SNSU-BSN has 4 check standards, one of which is E_0 no 75. This standard is also traceable to BIPM through stainless steel weights such as E_0 no 74.

1.3. Sub-Division Weighing Solution

The solution to the sub-division weighing case is to construct a set of calibration equations, which are linearly related to the observed mass difference d_i, several weighing combinations M, from a load of N. The correction for the nominal mass of the load, c_j, can be written in matrix form as

$$A \cdot C = D \tag{1}$$

where $A = a_{ij}$ is a dimension matrix $M \times N$, called matrix design
$C = c_j$ is a column matrix with dimension $N \times 1$, is called the correction matrix
$D = d_i$ is a column matrix with dimension $M \times 1$, is called the deflection matrix

The difference in mass between the reference weights and the unknown weights is measured in a weighing design. In mass calibration, the weighing design can determine corrections from many standards at once. One or a set of standard combinations that are considered to be known for their correction can be used as a constraint value to solve the calibration equation. Here, the calibration value constraint for one or more mass standards is expressed as

$$c_r = d_r \tag{2}$$

Equation (2) is added to equation (1). because the value of the restraints is fixed, which the reference standard gives value. The deflection of the weighing results can be related to the uncertainty, which is described by the variances of the $V(D)$ matrix. When a mass comparator has a variance difference of ${\sigma_i}^2$ is required in a calibration system that follows equation (1). This can be done conventionally by introducing a weighted matrix W, which is assumed to be an $M \times M$ diagonal matrix with

$$W_{ii} = {s_{\mathrm{norm}}^2}\big/{\sigma_i^2}\,;\;\; W_{ij} = 0\ (i \neq j) \tag{3}$$
$$(\sigma^2)^{-1} = \frac{W}{s_{\mathrm{norm}}}$$

where $\sigma^2 = V(D)$

The form of s_{norm}^2 can be viewed as an arbitrary factor whose only purpose is to create a dimensionless weighted matrix. As can be seen, this form can eliminate both the least squares estimate, C', from the correction and the variances of the V(C') matrix from this estimate. In this case $s_{\mathrm{norm}}^2 = \sigma s^2$ from different scales.

The least-squares estimate and its variance, the least-squares estimate of the correction of a weighing can be expressed as

$$C'_W = (A^T \cdot W \cdot A)^{-1} \cdot A^T \cdot W \cdot D \tag{4}$$

And the variance associated with the least-squares estimate of this correction is

$$\begin{aligned} V(C'_W) &= <(C'_W - C_W) \cdot (C'_W - C_W)^T> \\ &= (A_W^T \cdot A_W)^{-1} \cdot A_W^T \cdot V(D_W) \cdot A_W \\ &\qquad \cdot (A_W^T \cdot A_W)^{-1} \end{aligned} \tag{5}$$

where $A_W = W^{-1/2} \cdot A$ and $D_W = W^{-1/2} \cdot D$.

C0W has the minimum variance of a linearly unbiased estimator if

$$V(D_W) = \sigma^2 I \tag{6}$$

where σ^2 is a positive number.

For that, equation (5) can be simplified to:

$$V(C_W') = \sigma^2 (A_W^T \cdot W \cdot A_W)^{-1} \tag{7}$$

From equation (3), if $\sigma^2 = s_{\text{norm}}^2$, so that this is a normalization factor on the weighting matrix, then the arbitrary factor can be lost from $V(C_W')$

$$V(C_W') = (A^T \cdot (\sigma^2) \cdot A)^{-1} \tag{8}$$

as well as from C_W' Equation (4).

Considering the estimation of uncertainty and inaccuracy in the correction, variance can be obtained based on the square of the standard deviation value taken over a long period from the scale (pooled standard deviation) (Pendrill, 1992).

The sub-division method has its advantages and disadvantages compared to other methods. The advantages of this method include minimizing the use of standards, generating a data set that provides important statistical information about the scales' daily measurements and performance, and data redundancy. While the disadvantages are that it requires a relatively complex algorithm to analyze the measured data, it requires placing the number of loads on the weighing pan (this can cause problems for instruments with poor eccentricity characteristics or automatic balances designed for single load comparisons) (Davidson et al., 2004).

2. Experimental

The closed cycle weighing scheme is designed to calibrate weights with the same nominal mass. The closed-cycle weighing scheme includes three weights, namely one standard weight and two calibrated weights. The two calibrated weights consist of one weight to be determined, and the other is the checking standard. These two weights are in the same class. In this study, the standard used is Pt-Ir K-112 which has traceability to BIPM.

In contrast, the check standard is the weight class E_0 no 75. These two standards are used to determine the mass value of the weight E_0 no 74. The dissemination process of mass values is carried out using the Mettler Toledo AX1006 mass comparator with environmental conditions acquired using the climate station Klimet A30 (Meteolabor AG, 2004; Mettler Toledo, 2014).

The closed-cycle weighing scheme is represented by equation (9).

$$\begin{aligned} R &= C_s + a_1 \\ C_s &= T + a_2 \\ T &= R + a_3 \end{aligned} \tag{9}$$

where R is the reference weight; C_s is standard check, and; T is a weight whose mass value is unknown.

This method is solved by weighted least-squares analysis. For this reason, it is necessary to construct a weighing matrix that represents equation (8) by adding a constraint value, namely a_0. The value of a_0 is the conventional mass of the standard mass of Pt-Ir K-112 After equation (4.3) is added to the restraint value, the new equation can be written as follows,

$$\begin{aligned} &(a); \quad R && && = a_0 \\ &(b); \quad R && -C_s && = a_1 \\ &(c); && +C_s && -T = a_2 \\ &(d); \quad -R && && +T = a_3 \end{aligned} \tag{10}$$

To get the solution of the least-squares approach, referring to equation (1), then the equation is redefined as,

$$X \cdot \vec{M} = \vec{a} \tag{11}$$

where X is the weighing design matrix; $\vec{M}$ is the solution vector; and $\vec{a}$ is the observer vector or the deflection vector.

The X matrix is arranged based on the weighing design shown in equation (9). Furthermore, the weighing design equation is determined by the value of the matrix components. The value of the matrix components in the closed-cycle weighing scheme can be seen in Table 1. The matrix components in the table are worth 1, 0, and 1. This value is defined based on the order of weighing substitutions performed on each row of mass comparison.

Table 1. Weighing design matrix closed-cycle weighing scheme.

Equation	R	C_s	T
$R = a_0$	1	0	0
$R - C_s = a_1$	1	-1	0
$C_s - T = a_2$	0	1	-1
$-R + T = a_3$	-1	0	1

Based on Table 1, the X, $\vec{M}$ and $\vec{a}$ matrices are arranged as shown in equation (12).

$$X_{cc} = \begin{bmatrix} 1 & 0 & 0 \\ 1 & -1 & 0 \\ 0 & 1 & -1 \\ -1 & 0 & 1 \end{bmatrix}; \ \vec{M}_{cc} = \begin{bmatrix} M_R \\ M_{C_s} \\ T \end{bmatrix} \text{ and } \vec{M}_{cc} = \begin{bmatrix} a_0 \\ a_1 \\ a_2 \\ a_3 \end{bmatrix} \tag{12}$$

The mass of the weights whose value is unknown is solved using the weighted least-squares analysis method. From equation (11) and the relationship between the weights, W, and the variance-covariance matrix, V, which is shown in equation (3), a new equation can be drawn up as follows:

$$\vec{M} = (X^T \cdot V_a^{-1} \cdot X)^{-1} \cdot X^T \cdot V_a^{-1} \cdot \vec{a} \tag{12}$$

The values of X and $\vec{a}$ have been described in equation (12). Meanwhile, V_a is a matrix whose diagonal value is determined based on the uncertainty value of the head weight and the standard deviation of the pooled mass comparator at the nominal weighing. The head weight uncertainty value is obtained from a calibration certificate issued by BIPM in 2019. The pooled standard deviation value is the value collected from periodic checking of the mass comparator as a form of quality assurance in calibration activities within a certain period.

3. Results and Discussion

The measured value and its uncertainty generally express the calibration results. The measured value here is the mass of the E_0 weights. The mass of these weights is obtained by completing the calculation using equation (12) from the weighing data. However, it is necessary first to define the value of the V_a matrix. By knowing the uncertainty value of the head weight of 12 mg and the pooled standard deviation of the AX1006 mass comparator at a nominal weighing of 1 kg, then V_a can be expressed as follows,

$$V_a = \begin{bmatrix} 144 & 0 & 0 & 0 \\ 0 & 25 & 0 & 0 \\ 0 & 0 & 25 & 0 \\ 0 & 0 & 0 & 25 \end{bmatrix} \tag{13}$$

With the known value of the V_a matrix, equation (12) can be solved easily. The solution is the mass value of the three weights involved in the weighing scheme. The results show that the conventional masses of E_0 no 74 and E0 no 75 are 1000,000046 g and 1000,0000163 g, respectively.

In calibration, the uncertainty value becomes very important in addition to the measurement result value. The uncertainty value states the range of values the measurement results are. In addition, the measurement uncertainty shows the quality of the measurement results, where the smaller the uncertainty, the better the measurements made. Many factors need to be considered in determining the value of measurement uncertainty. By the complex analytical method, the sub-division weighing scheme must evaluate several factors contributing to this uncertainty: the estimated value of the solution vector solution, least squares regression error, and the value of the check standard control. The calculation of the uncertainty value is based on the Guide to the expression of uncertainty in measurement (GUM) (JCGM-100, 2008). Figure 2 and Table 2 are budget uncertainty with the sub-division method, respectively.

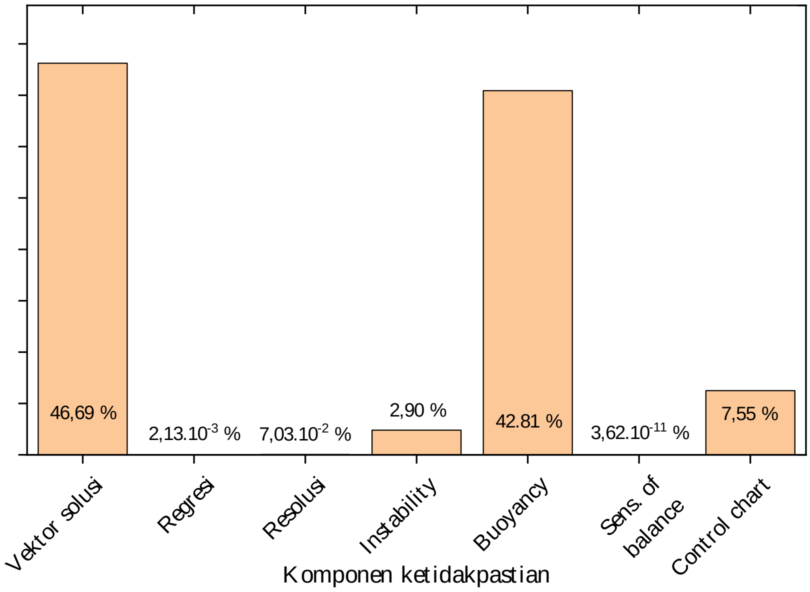

Figure 2. The proportion of contribution to calibration uncertainty E_0 no 74.

Table 2. Calibration uncertainty budget E_0 no 74.

Komponen	Unit	Dist.	U_i	d	ν_i	c_i	$(\frac{U_i}{d} \times c_i)^2$	$\frac{(u_i \times c_i)^4}{\nu_i}$
Vek. Solusi	µg	Norm.	$12,44$	1	397	1	$154,67$	$60,27$
Regresi	µg	Norm.	$8,40 \cdot 10^{-2}$	1	1	1	$7,06 \cdot 10^{-3}$	$4,98 \cdot 10^{-5}$
Resolusi	µg	Rect.	$0,41$	$1,73$	$1 \cdot 10^6$	1	$1,11$	$1,23 \cdot 10^{-12}$
Instability	µg	Rect.	$3,10$	$1,73$	60	1	$3,20$	$1,03 \cdot 10^{-9}$
Buoyancy	µg	Norm.	$11,91$	1	100	$1,06 \cdot 10^{-6}$	$159,32$	$253,83$
Sens. Komp.	µg	Norm.	$5 \cdot 10^{-7}$	1	9	1	$2,5 \cdot 10^{-13}$	$1,43 \cdot 10^{-23}$
Grafik Kendali	µg	Norm.	5	1	20	1	25	$1,43 \cdot 10^{-23}$
Sums							$342,308$	$345,35$
Combined uncertaity							$18,50$	
Effective degree of freedom							339	
Coverage factor							$1,97$	
Expanded uncertainty							36	µg

In calculating measurement uncertainty with a complex weighing combination, several uncertainty components are obtained in a more complicated way. The value of the solution vector component is obtained from the calculation of the weighing design matrix and the variance-covariance matrix, which stores information about the head-weight uncertainty and the standard deviation of the mass comparator. The estimated uncertainty value for the solution vector component is calculated based on the variances of the weighted matrix.

Meanwhile, the regression uncertainty component is determined based on the analysis of the weighing residuals. The uncertainty component of this regression is determined based on equation 7.

$$U_{reg} = (A^T A)^{-1} \sigma^2 \tag{13}$$

with

$$\sigma^2 = \sum_{i=1}^{N} (a_i - \langle a_i \rangle)^2 \tag{13}$$

Where σ^2 is the variance of the calibration data obtained by residual analysis. The residual here is the difference in value between the deflection of the weighing result shown by the mass comparator indicator (a_i) and the mass of the calibration weight ($\langle a_i \rangle$).

Another component of uncertainty contributed by the mass comparator is the resolution and sensitivity of the comparator. The smaller the mass comparator's reading ability, the more accurate the measurements are made, which means the smaller the error that may occur when using the tool. In this E_0 dissemination, a mass comparator with a readability of 1 μg is used. Meanwhile, the sensitivity component of the mass comparator is related to the ability of the mass comparator to detect very small changes in mass. This sensitivity value is usually obtained from repeated weighing by including weights with a small nominal mass. The sensitivity value of the AX1006 mass comparator at the weighing point of 1 kg is 5.00×10^{-7} mg.

Even though the handling of the measuring standard is strictly maintained, changes in the mass value of the measuring standard can still occur. The uncertainty component of instability needs to be taken into account to anticipate changes in the value of this measuring standard. The value is estimated according to the instability of Pt-Ir no K46, which is 3.10×10^{-3} mg.

In high-accuracy measurements, the uncertainty component due to the air's buoyancy becomes crucial. This component can be dominant when measurements are made with high fluctuations in air density. Therefore, strict environmental conditioning is a must to reduce the contribution of this component's uncertainty. The component of air buoyancy uncertainty is calculated by looking at fluctuations in air density during the calibration process. The air density fluctuation during weighing is 5.04×10^{-5} kg.m^{-3}.

Combining all uncertainty components summarized in Table 2 produces an uncertainty value of 0.036 mg at a 95% confidence level. The proportion of each component of uncertainty can be seen in Figure 2. From the graph, it can be seen that there are two components with the most dominant uncertainty contribution, namely the vector solution (46.69 %) and the correction for buoyancy (42.81 %). Other components have a contribution of less than 8%.

4. Conclusion

The weights E_0 no 74, as the highest working standard owned by SNSU-BSN, must have traceability that can be guaranteed. Calibration is the method used to ensure the chain traceability of the weights. This article describes a method used to determine the mass value. In this method, there is a measurement quality assurance parameter by applying check weights in another measuring standard with the same accuracy class. The calibration obtained the results in the form of a standard mass value of E0 no 74 of 1000,000046 g with an uncertainty of 0.036 mg.

In determining the value of measurement uncertainty, several uncertainty components are taken into account from the measurement instruments used, measuring standards, and calibration environmental conditions. From the evaluation carried out, the uncertainty component of the vector solution represents the uncertainty value of the measuring standard used and the air buoyancy component that describes the influence of the environmental conditions of measurement, and both have the most dominant contribution.

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