Statistical process control to monitor the impacts of extraordinary calibrations result on the mass dissemination

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Abstract. Based on the result of the Extraordinary Calibrations conducted by the Bureau of Weights and Measures (BIPM) using the international prototype of the kilogram (IPK) from the end of 2013 to 2014, it was found that the BIPM ‘as-maintained mass unit’ was offset by 35 μg from the mass of the IPK. This result is used by BIPM to create a model. A model has been used to estimate the correction of all 1 kg mass calibration certificates ever issued by BIPM in the period 1992 to 2014. The value of highest mass standard owned by Research Center for Metrology LIPI (RCM LIPI) also updated. This updated mass value affected the calibration results of RCM LIPI working standard by 34 μg. The statistical process control and control chart of check standard weights are used to monitor the impact of the updated mass on mass dissemination for RCM LIPI calibration customer.

1. Introduction

Research Centre for Metrology LIPI (RCM LIPI) is responsible for dissemination of mass measurement in Indonesia. The highest mass standards possessed by RCM LIPI is a mass standard made of stainless steel with a nominal mass of 1 kg. This mass standard traceable to SI through the Bureau of Weights and Measures (BIPM) and calibrated to the copy of international kilogram prototype (IPK) number 25.

The IPK is kept in a vault at the BIPM located in Sevres, France since 1889. The IPK has only been used during two measurement campaigns, namely the second and third periodic verifications (3rd PV) of national prototypes of the kilogram. In the 3rd PV, it was found that the mass of the six official copies had changed against the IPK by maximum 69 μg over 100 years \[1\].

To anticipate the kg redefinition, in 2013 the International Committee for Weights and Measures (CIPM) approved a two-phase a new calibration campaign so-called Extraordinary Calibration. from the end of 2013 to 2014. It was discovered that the BIPM as-maintained mass unit was offset by 35 μg \[2\]. The result has enabled BIPM to be re-evaluated the copy of IPK with the newest linkage to the IPK. This has enabled BIPM to model the evolution of the offset in the as-maintained mass unit to estimate corrections to all 1 kg mass calibration certificates issued by the BIPM during this 1992 to 2014\[3\] including mass standard RCM LIPI calibration certificate.

In this paper will be described the roles of 1 kg mass standard as the highest mass standards owned by RCM LIPI in the dissemination of mass in Indonesia and the implications for RCM LIPI calibration customers of the new mass value of the extraordinary calibrations result. A measurement assurance program is implemented to monitor changes in reference or working standard and the measurement process that affect the result. The check standards are used to monitor if a change in the value of the reference weight. The new value of check standard is tested for agreement with the accepted value using a statistical control technique based on t-statistic. To evaluate inconsistency of the two-mass values result is used the normalized error (\(E_n\)) number test. Based on the application of measurement assurance program that can be seen how much
extraordinary calibration results impact on the mass measurement dissemination conducted by RCM LIPI.

2. Mass standard and measurement assurance at RCM LIPI

The highest mass standards owned by RCM LIPI is a mass standard made of stainless steel with a nominal mass of 1 kg is designated as E0 92. It is calibrated to the Copy of IPK number 25 by BIPM every 5 years. There are two calibration results issued by BIPM, in 2007 and 2012. The last calibration is in 2012, the value of the mass was 999.999 807 g with a combined standard uncertainty, $u_c$, ($k = 1$) of 0.015 mg. In 2015, BIPM has updated the value as recommended by the CCM. The amended mass value was 999.999 773 g. It appears that there was a shift of 34 μg between the mass value before and after the amendment E0 92 is used for calibrating the stainless-steel mass standard number 74 (E0 74). To check E0 92 is used 1 kg mass standard number 75 (E0 75). The mass value dissemination scheme in the RCM LIPI can be seen in Fig.1.

![Fig.1. Mass value dissemination scheme](image1)

The check standard is calibrated against E0 92, and the result is compared to its historical value. For quality assurance is applied statistical process control [4]. Deviation from accepted value of check standard may indicate a change in E0 92. The value of check standard obtained from the monitored processes is plotted on a chart. A central line is drawn, indicating the mean ($\bar{x}$) of 8 measurements of the check standard and designated limits are indicated within which the results of measurements are expected to be randomly distributed, based on statistical considerations. The designated limit is valid for the duration of the check standard’s calibration interval. The system is in control when the individual values are within the warning limits. The system is considered to be out of control if an excessive number of values are present outside warning limit or control limits[5][6].

The values of E0 75 is designed as check standard, are plotted on a chart. The record values are obtained from the monitored processes. A control chart of E0 75 can be seen in Fig.2.

![Fig.2. Control chart of E0 75](image2)
The calibration interval of E0 75 is specified for 5 years, the designated limits are valid from 2012 to 2017 and determined based on a value of E0 92 that has not been updated. In 2015, BIPM has updated the value of E0 92. Based on the updated value, the measurement results of E0 75 were a shift and out of control. In Fig.3, it is seen that the E0 75 value starts out from the designated limits since 2015, i.e. since the value of E0 92 was updated by the BIPM. This indicates that the change of mass value in E0 92 due to extraordinary calibration result had an impact on E0 74 mass value significantly.

Fig.3. The out of control values of E0 75

3. Mass dissemination of E1 class

In disseminating the mass value of the E0 weight to the E1 class weights, RCM LIPI uses a closed-cycle, subdivision and multiplication methods. The closed-cycle method is used to calibrate two weights on one reference weight having the same nominal value. In this case, E0 74 is used to calibrate 1 kg E1 class weight and 1 kg E1 199 which is designated as check standard. Due to the amendment of the calibration certificate by the BIPM in 2015 and caused a shift in the mass value of E0 74, the mass value of E1 class weight calibration also shifted. Fig.4 shows a shift in the conventional mass of E1 class weights with the nominal mass of 10 kg, 1 kg, 100g, 10g and 0.001g.

Fig.4. The shift mass values of E1 class weight

4. Result and discussion

To see the compatibility between the two values of E1 class weight in each nominal mass, $E_n$ number test[7] is done. The results can be seen in Table 1.

The calibration interval period of E1 class check standards is 3 years. The designated limits were determined in 2013 and data used to determine the limits were traceable to E0 92 mass value before the amendment. On the chart, it appears that the check standard data record does not indicate any deviation from the designated limits. This is in accordance with the results of $E_n$ number test where the result is less than one.
Table 1. Compatibility between the two values of E1 class weight in each nominal mass

| Nominal /g | Before Amendment | Amendment |
|------------|------------------|-----------|
|            | Conventional Mass /g | Uncertainty /mg (k = 2) | Conventional Mass /g | Uncertainty /mg (k = 2) |
| 10 000     | 9 999 999 89      | 0.80      | 9 999 999 55      | 0.80      | 0.30 |
| 1 000      | 999 999 73        | 0.06      | 999 999 69        | 0.06      | 0.47 |
| 100        | 999 999 97        | 0.010     | 999 999 53        | 0.010     | 0.28 |
| 10         | 10 000 009        | 0.004     | 10 000 009        | 0.004     | 0.00 |
| 0.001      | 0.000 999 2       | 0.000 5   | 0.000 999 2       | 0.000 5   | 0.00 |

The values of check standard obtained from the monitored processes are plotted on a chart. The control chart of check standard weights 199-1kg and 199-0.001g can be seen in Fig.5.

5. Conclusion
The updated mass of E0 92 had affected the mass measurement result of E0 74 significantly. But, the result does not have a significant impact on the calibration service of E1 class weight for RCM LIPI customer. Statistical process control is very effective for monitoring changes in the measurement process. The impact of the mass value change on the reference standard can be monitored by deviation of check standard from accepted value. The designated limit on the control chart should always be up-to-date to monitor the measurement process and provide quality assurance on the measurement results.

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