# THE FINAL REPORT FOR CCM.M-K7: KEY COMPARISON OF 5 kg, 100 g, 10 g, 5 g and 500 mg STAINLESS STEEL MASS STANDARDS

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## ABSTRACT

In order to show equivalence in mass standards calibration among National Metrology Institutes (NMIs) of member countries of the "Comité international des poids et mesures" (CIPM), key comparisons (KC) of mass standards have been carried out under the auspices of the "*Comité Consultatif pour la Masse et les Grandeurs Apparentées*" (CCM). This key comparison of 5 kg, 100 g, 10 g, 5 g and 500 mg stainless steel mass standards was based on the decision of the CCM during the 12th meeting held in 2010 at the Bureau International des Poids et Mesures (BIPM). KRISS (Republic of Korea) and PTB (Germany) acted as pilot laboratory and co-pilot laboratory, respectively. The results were evaluated with the Monte Carlo method using measurement values based on participants' reference standards calculated following the recent BIPM amendments in 2015. Regarding participant results, VNIIM (100 g and 5 g) were not consistent with the key comparison reference values within their expanded uncertainties with the coverage factor, $k$ = 2.

## 1. INTRODUCTION

The key comparison, CCM.M-K7 was based on the decision of the CCM during its 12th meeting held in 2010 at BIPM. The comparison was piloted by KRISS and supported by PTB as co-pilot laboratory. The comparison was organized in two petals, and used one set of 5 kg, 100 g, 10 g, 5 g and 500 mg stainless steel standards in each petal.

Ten laboratories measured a set of travelling standards between April 2014 and March 2015.

All results presented in this final report are based on the BIPM amendments announced officially at the 14th CCM meeting in 2015. On May 2015, CCM Working Group on the Dissemination of the kilogram (WGD-kg, chairperson: Chris Sutton) agreed with the opinion of the pilot that the results reported by all participants should be derived using values for their reference standards calculated following the recent amendments to the BIPM mass calibration certificates [1]. All participants agreed with this request; however, since the request came after the submission of participants' initial reports, it was required that participants revise these initial reports to reflect the correction and resubmit them to the pilot laboratory.

## 2. PARTICIPANTS

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Under the permission of CCM, the participants were recommended by the Technical Committee of Mass and Related Quantities (TCM) of each Regional Metrology Organization (RMO) within its assigned quota by the pilot laboratory. The quota in each RMO was estimated from the historical records of participation in previous CCM key comparisons [2-7]. The pilot laboratory recommended each TCM chair to consider the following conditions in choosing a candidate: 1) CCM membership[1], 2) Ability to transport artifacts via hand-carry, and 3) Capability of managing regional KC of CCM.M-K7. Both pilot and copilot laboratories were also counted in the assigned quota.

As a result of these recommendations, ten NMIs took part in this key comparison. Among the participants, one represented intra-Africa metrology system (AFRIMET, Chairperson: Alaaeldin A. Eltawil) and one represented Euro-Asian Cooperation of National Metrology Institutions (COOMET, Irina Kolozinska); there were two representatives each for Sistema Interamericano de Metrología (SIM, Francisco García) and Asia Pacific Metrology Programme (APMP, Tokihiko Kobata); there were four representatives for European Association of National Metrology Institutes (EURAMET, Nieves Medina) members. The participating laboratories are listed in Table 1 and the volunteered candidates for organizing a regional key comparison of CCM.M-K7 are emphasized in italic type.

**Table 1.** Participant laboratories of the comparison

| National Institute of Metrology | Acronym | Country | RMO |
|---|---|---|---|
| Korea Research Institute of Standards and Science | KRISS | Republic of Korea | APMP |
| Physikalisch-Technische Bundesanstalt | PTB | Germany | EURAMET |
| *National Institute of Metrology* | *NIM* | *China* | *APMP* |
| *National Institute for Standards* | *NIS* | *Egypt* | *AFRIMET* |
| *Istituto Nazionale di Ricerca Metrologica* | *INRIM* | *Italy* | *EURAMET* |
| *Centro Nacional de Metrologia* | *CENAM* | *Mexico* | *SIM* |
| The Federal State Unitary Enterprise "All-Russian D.I.Mendeleev Research Institute for Metrology" | VNIIM | Russia | COOMET |
| Centro Espanol de Metrologia | CEM | Spain | EURAMET |
| Federal Institute of Metrology | METAS | Switzerland | EURAMET |
| National Institute of Standards and Technology | NIST | United States of America | SIM |

## 3. TRANSFER STANDARDS

The transfer standards were monitored regularly for more than six months at the pilot laboratory. Two sets were selected to circulate in two petals, both of which showed excellent stability without any significant deviation during monitoring. Each set consists of five weights, which were marked differently on the surface for identification. Table 2 shows the relevant technical data for each weight provided by pilot and co-pilot laboratories. Other quantities were

[1] As of the date in choosing participant, Egypt was not a member of CCM. On July 2013, the Working Group Dissemination (WGD-kg) interpreted that any non-member of CCM (Egypt) can participate in Key Comparison according to CIPM document MRA-D-05, *Measurement comparisons in the CIPM MRA*.

determined according to the standard procedure of the participating institute. For example, the coefficient of volume expansion for stainless steel material was assumed to be 0.000 045 $K^{-1}$.

The transportation case was an aluminum box with separate holes for each standard (Fig. 1). The weights were wrapped in clean dust-free paper and fixed at their positions by such paper stuffed into the holes. The dimensions of the box are 27 cm × 33 cm × 18 cm and the total weight is approximately 8 kg.

**Fig**. 1. Transfer standards and transportation case.

**Table 2.** Transfer standards

| | Nominal | Marking | Volume[1] / $cm^3$ (Uncertainty, *k*=2) | Center of Mass / mm (Uncertainty, *k*=2) | Magnetic susceptibility[2] |
|---|---|---|---|---|---|
| | 5 kg | A | 624.25 (0.06) | 64.7 (1.2) | 0.000 5 |
| | 100 g | C | 12.485 5 (0.000 8) | 17.6 (2.0) | 0.000 4 |
| Petal A | 10 g | C | 1.248 6 (0.000 5) | 9.1 (0.5) | 0.002 |
| | 5 g | A | 0.624 3 (0.000 4) | 7.3 (0.3) | 0.003 |
| | 500 mg | (B) | 0.062 6 (0.000 2) | - | - |
| | 5 kg | C | 623.89 (0.06) | 64.6 (1.2) | 0.003 |
| | 100 g | D | 12.479 1 (0.000 8) | 17.8 (2.0) | 0.000 1 |
| Petal B | 10 g | D | 1.249 0 (0.000 5) | 9.2 (0.5) | 0.003 |
| | 5 g | C | 0.624 2 (0.000 4) | 7.4 (0.3) | 0.003 |
| | 500 mg | (C) | 0.062 6 (0.000 2) | - | - |

[1] provided by the co-pilot lab, PTB.
[2] measured with a commercial susceptometer (Sartorius, Model: YSZ01C, Germany).

## 4. CIRCULATION OF THE TRANSFER STANDARDS

Table 3 shows the final schedule confirmed by all participants for the circulation of transfer standards. In principle, the transfer standards were to be hand-carried from one participant to the next. However, transportation by courier or diplomatic bag was allowed according to mutual agreement between participants. By consideration of all participants' requests, the schedule and sequence of circulation were decided as shown in the footnote of Table 3. The circulation was carried out on time without any serious delay according to the planned schedule for each participant. The pilot laboratory arranged the ATA-Carnet for customs clearance when crossing international borders.

**Table 3.** The sequence of circulation and measurement schedule

| Start Date | End Date | Petal A | Petal B |
|---|---|---|---|
| - | - | - | Korea |
| April 15, 2014 | June 1, 2014 | Korea[1] | Germany |
| June 1, 2014 | July 15, 2014 | Egypt[2] | Switzerland |
| July 15, 2014 | September 30, 2014[3] | Russia | Spain |
| September 30, 2014 | November 15, 2014 | Mexico | Italy |
| November 15, 2014 | January 15, 2015[4] | USA[5] | China[2] |
| January 15, 2015 | March 30, 2015[4] | Korea | Korea |

[1] Transported to Egypt by a diplomatic bag.
This method of transportation was initially proposed by NIST.
[2] Not applicable for ATA-Carnet
[3] Consideration of summer vacation (Aug. 1 ~ 30)
[4] Consideration of end of year in solar and lunar calendars.
[5] Special request for the available date of the laboratory.

## 5. MASS COMPARATORS USED BY PARTICIPANTS

The weighing instruments used by participating laboratories are listed in Table 4.

**Table 4.** Weighing Instruments of participants

| Nominal | NMI | Manufacturer | Model | Capacity | Resolution |
|---|---|---|---|---|---|
| 5 kg | KRISS | Mettler-Toledo | AT10005 | 10 011 g | 10 µg |
| | PTB | Mettler-Toledo | AX10005 | 10 kg | 10 µg |
| | NIM | Mettler-Toledo | AT10005 | 10 011 g | 10 µg |
| | NIS | Sartorius | CC10000U-L | 10 kg | 10 µg |
| | INRIM | Mettler-Toledo | AT10005 | 10 011 g | 10 µg |
| | CENAM | Mettler-Toledo | AT10005 | 10 011 g | 10 µg |
| | VNIIM | Sartorius | CC10000S | 10 011 g | 100 µg |
| | CEM | Mettler-Toledo | AT10005 | 10 011 g | 10 µg |
| | METAS | Mettler-Toledo | AT10006 | 10 011 g | 10 µg |
| | NIST | Mettler-Toledo | AX10005 | 10 kg | 10 µg |
| 100 g | KRISS | Mettler-Toledo | M-one | 1001.5 g | 0.1 µg |
| | PTB | Mettler-Toledo | HK1000 | 1 kg | 0.1 µg |
| | NIM | Mettler-Toledo | M-one | 1001.5 g | 0.1 µg |
| | NIS | Mettler-Toledo | AT1006 | 1 kg | 1 µg |
| | INRIM | Mettler-Toledo | AT106 | 100 g | 1 µg |
| | CENAM | Mettler-Toledo | AX1005 | 1 kg | 10 µg |
| | VNIIM | Sartorius | CC1000S-L | 1 kg | 1 µg |
| | CEM | Mettler-Toledo | M-one | 1001.5 g | 0.1 µg |
| | METAS | Mettler-Toledo | M-one | 1001.5 g | 0.1 µg |

| Nominal | NMI | Manufacturer | Model | Capacity | Resolution |
|---|---|---|---|---|---|
| | NIST | Mettler-Toledo | AT1005 | 1 kg | 10 µg |
| 10 g | KRISS | Mettler-Toledo | AT106 | 100 g | 1 µg |
| | PTB | Mettler-Toledo | AX107 | 110 g | 0.1 µg |
| | NIM | Sartorius | CCR10 | 10.1 g | 0.1 µg |
| | NIS | Mettler-Toledo | AT21 | 20 g | 1 µg |
| | INRIM | Mettler-Toledo | AT106 | 100 g | 1 µg |
| | CENAM | Mettler-Toledo | AX106 | 100 g | 1 µg |
| | VNIIM | Sartorius | CCE 66 | 61 g | 1 µg |
| | CEM | Mettler-Toledo | M-one | 1001.5 g | 0.1 µg |
| | METAS | Mettler-Toledo | AT106H | 111 g | 1 µg |
| | NIST | Mettler-Toledo | AX106 | 100 g | 1 µg |
| 5 g | KRISS | Mettler-Toledo | a5 | 5.1 g | 0.1 µg |
| | PTB | Mettler-Toledo | AX107 | 110 g | 0.1 µg |
| | NIM | Mettler-Toledo | a5 | 6.1 g | 0.1 µg |
| | NIS | Sartorius | CC6 | 6 g | 0.1 µg |
| | INRIM | Mettler-Toledo | UMX5 | 5 g | 0.1 µg |
| | CENAM | Mettler-Toledo | UMT5 | 5 g | 0.1 µg |
| | VNIIM | Mettler-Toledo | UMX5 | 5 g | 0.1 µg |
| | CEM | Mettler-Toledo | a5 | 5.1 g | 0.1 µg |
| | METAS | Mettler-Toledo | a5 | 5.1 g | 0.1 µg |
| | NIST | Mettler-Toledo | UMT5 | 5 g | 0.1 µg |
| 500 mg | KRISS | Mettler-Toledo | a5 | 5.1 g | 0.1 µg |
| | PTB | Sartorius | CCE6 | 6 g | 0.1 µg |
| | NIM | Mettler-Toledo | a5 | 6.1 g | 0.1 µg |
| | NIS | Sartorius | CC6 | 6 g | 0.1 µg |
| | INRIM | Mettler-Toledo | UMX5 | 5 g | 0.1 µg |
| | CENAM | Mettler-Toledo | UMT5 | 5 g | 0.1 µg |
| | VNIIM | Mettler-Toledo | UMX5 | 5 g | 0.1 µg |
| | CEM | Mettler-Toledo | a5 | 5.1 g | 0.1 µg |
| | METAS | Mettler-Toledo | a5 | 5.1 g | 0.1 µg |
| | NIST | Mettler-Toledo | UMT2 | 2 g | 0.1 µg |

Note: Based on the information provided by participants without any verification. Certain commercial equipment, instruments, or materials are identified in this paper to foster understanding. Such identification does not imply recommendation or endorsement by any of the participating organizations nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

## 6. BIPM AMENDMENTS

Table 5 shows the reported previous and revised values and standard uncertainties of the national prototype kilogram used by each participant according to the BIPM amendments. All except NIST revised their first reports according to the amendments. NIST used 102, a new prototype which was not affected by the BIPM shift and therefore not involved in the amendments.

**Table 5.** BIPM amendments to certificates of national prototypes of kilogram. The units are mg.

| NMI | No. Prototype | Old Mass - 1 kg | Old Standard Uncertainty | New Mass - 1 kg | New Standard Uncertainty |
|---|---|---|---|---|---|
| KRISS | 72 | 0.485 | 0.007 | 0.449 | 0.003 |
| PTB | 70 | -0.207 | 0.007 | -0.242 | 0.003 |
| NIM | 60 | 0.358 | 0.006 | 0.327 | 0.003 5 |
| NIS | 58 | -0.167 | 0.007 | -0.201 | 0.003 |
| INRIM | 76 | 0.17 | 0.005 | 0.156 | 0.003 |
| CENAM | 96 | 0.206 | 0.007 | 0.172 | 0.003 |
| VNIIM | 12 | 0.135 | 0.007 | 0.099 | 0.003 |
| CEM | 3 | 0.128 | 0.007 | 0.092 | 0.003 |
| METAS | 38 | 0.256 | 0.005 | 0.251 | 0.003 |
| NIST | (102) | - | - | -0.132 | 0.003 |

**Table 6.** The measured mass value and its standard uncertainties reported by the participants. Data are given as the difference between the mass of the travelling standards and the nominal value in mg.

| Petal / Participant | | 5 kg | | 100 g | | 10 g | | 5 g | | 500 mg | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | $m_i$ | $u_i$ | $m_i$ | $u_i$ | $m_i$ | $u_i$ | $m_i$ | $u_i$ | $m_i$ | $u_i$ |
| Petal A | Marking | A | | C | | C | | A | | (B) | |
| | KRISS | 0.135 | 0.073 | 0.017 9 | 0.002 5 | 0.011 6 | 0.001 5 | 0.007 7 | 0.000 43 | 0.001 93 | 0.000 27 |
| | NIS | 0.10 | 0.30 | 0.002 6 | 0.005 0 | 0.009 | 0.002 6 | 0.011 2 | 0.002 0 | 0.002 4 | 0.000 7 |
| | VNIIM | 0.41 | 0.20 | 0.002 9 | 0.003 0 | 0.009 4 | 0.001 7 | 0.010 9 | 0.000 9 | 0.000 54 | 0.000 75 |
| | CENAM | 0.12 | 0.20 | 0.022 5 | 0.005 7 | 0.008 5 | 0.001 7 | 0.008 8 | 0.001 1 | 0.000 89 | 0.000 71 |
| | NIST | -0.138 | 0.085 | 0.005 9 | 0.004 2 | 0.005 8 | 0.002 4 | 0.007 1 | 0.001 2 | 0.000 62 | 0.000 30 |
| | KRISS | 0.040 | 0.073 | 0.012 6 | 0.002 5 | 0.010 0 | 0.001 5 | 0.008 5 | 0.000 43 | 0.002 10 | 0.000 27 |
| Petal B | Marking | C | | D | | D | | C | | (C) | |
| | KRISS | -0.271 | 0.073 | 0.010 9 | 0.002 5 | 0.012 0 | 0.001 5 | 0.007 9 | 0.000 43 | 0.002 30 | 0.000 27 |
| | PTB | -0.355 | 0.056 | 0.003 1 | 0.001 5 | 0.006 8 | 0.000 5 | 0.007 7 | 0.000 4 | 0.002 16 | 0.000 20 |
| | METAS | -0.170 | 0.146 | 0.008 7 | 0.004 5 | 0.006 3 | 0.002 0 | 0.008 9 | 0.001 5 | 0.002 7 | 0.000 8 |
| | CEM | -0.440 | 0.2 | 0.007 6 | 0.002 6 | 0.009 3 | 0.001 1 | 0.009 | 0.000 87 | 0.002 41 | 0.000 48 |
| | INRIM | -0.335 | 0.085 | 0.009 9 | 0.004 2 | 0.009 4 | 0.001 9 | 0.008 7 | 0.001 2 | 0.002 0 | 0.000 6 |
| | NIM | -0.40 | 0.07 | 0.010 2 | 0.002 4 | 0.006 8 | 0.001 8 | 0.008 7 | 0.001 0 | 0.000 9 | 0.000 8 |
| | KRISS | -0.310 | 0.073 | 0.005 6 | 0.002 5 | 0.012 5 | 0.001 5 | 0.010 1 | 0.000 43 | 0.002 90 | 0.000 27 |

## 7. REPORTED RESULTS FROM PARTICIPANTS

Table 6 shows the measured mass value ($m_i$) and standard uncertainties ($u_i$) provided by the participants.[2] The results of the laboratories are given as the mass differences in reference to the nominal value of the transfer standard.

Based on the data of Table 6 and Reference 8, a chi-squared test was performed to evaluate the consistency of reported results in each petal. Table 7 shows the observed chi-squared ($\chi_{obs}$) value in each petal. By consideration of the chi-squared values (Petal A: 9.49 and Petal B: 11.07) at the degree of freedom (Petal A: 4, Petal B: 5) in each petal, the consistency check can be done as indicated in Table 7. Inconsistencies were found in the scale of 100 g, 5 g and 500 mg of Petal A and 10 g of Petal B. Although individual messages were sent to some related participants in order to resolve these inconsistencies, they were unable to be resolved even after updating the values.

**Table 7.** Observed chi-squared values of the results reported by the participants in Table 6.

| Scale | Petal A | Petal B |
|---|---|---|
| 5 kg | 8.7 | 3.1 |
| 100 g | 20 (Fail) | 9.3 |
| 10 g | 3.9 | 21 (Fail) |
| 5 g | 11.7 (Fail) | 6.5 |
| 500 mg | 18.2 (Fail) | 6.0 |

## 8. ANALYSIS OF RESULTS

The pilot laboratory allowed the participants to check and update their reported values for 2 months for the initial report, 6 months for the revised report due to BIPM amendment, and >1 month for individual checking. In order to link results of participants of both Petal A and B, the differences between results reported by $i$-th participant and pilot laboratory ($\Delta m_i$) were calculated as follows,

$$\Delta m_i = m_i - (m_{PL} + \varepsilon_{drift} + \varepsilon_{reprod}) \qquad (1)$$

$$m_{PL} = \frac{m_{PL,i} + m_{PL,i+1}}{2} \qquad (2)$$

Where,

$m_{PL}$, the mean value of the results of the pilot laboratory measurements closest in time to the measurement of participant $i$

$m_{PL,i}$, the measured mass value reported by pilot laboratory before the measurement of $i$-th participant

[2] In preparation of Draft A, WGD-kg chair (Chris Sutton) directed the Pilot lab to handle data reporting in the following way: If a participant confirmed the reported data in a separate communication to the Pilot lab after reporting the data, any forthcoming updates could not be accepted as the final data.

$m_{PL,i+1}$, the measured mass value reported by pilot laboratory after the measurement of $i$-th participant

$\varepsilon_{drift}$, an error due to the possible drift of the travelling standards

$\varepsilon_{reprod}$, an error due to the reproducibility of travelling standards at the pilot laboratory by changing some conditions of measurement

The error due to the possible drift of the travelling standards was estimated by the difference between the measurements of the pilot laboratory before sending the travelling standard and upon its return. The largest of the two possible values of this drift was taken as representative for all participants. This drift error was assumed to be centered on zero with a uniform (rectangular) probability density function (pdf) [7].

Expressions for the expectation ($E(\varepsilon_{drift})$) and standard uncertainty ($u_{drift}$) of drift are given in Eq. (3). Only for 5 kg standard in Petal A, the drift was a little bit larger than that of Petal B possibly due to the noticeable mark on the top surface of the standard. This mark was reported just after calibration at NIS.

$$E(\varepsilon_{drift}) = 0\text{, and } u_{drift} = \frac{|m_{PL,i+1} - m_{PL,i}|}{\sqrt{12}} \quad (3)$$

The transfer standards were monitored from July 2013 to March 2014 at the pilot laboratory and then re-checked for reproducibility after returning to the pilot lab. The standard uncertainty of reproducibility ($u_{reprod}$) was estimated in the same way as the drift error, assuming a uniform (rectangular) distribution.

Table 8 shows the summary of standard uncertainties due to drift and reproducibility errors used in this analysis. Because of the inconsistencies found in the reported data and in order to consider relevant correlations, the pilot laboratory decided to determine the key comparison reference value (KCRV) by using the median (cf. Eq. (4)) in combination with the Monte Carlo method as recommended by Cox [8]. The median is a robust estimator (insensitive to outliers) and was used as the reference value in previous CCM key comparisons [2-4, 6, 7].

In order to comply with a recent official decision of WG Strategy of the CCM,[3] the pair-wise degrees of equivalence between two different participants is omitted in this report though it can be readily calculated from the reported data.

**Table 8.** The standard uncertainty of drift ($u_{drift}$) and reproducibility ($u_{reprod}$) errors. The units are mg.

| Nominal value | $u_{drift}$ | $u_{reprod}$ |
|---|---|---|
| 5 kg | 0.027 | 0.056 |
| 100 g | 0.001 5 | 0.001 5 |
| 10 g | 0.000 46 | 0.000 69 |
| 5 g | 0.000 70 | 0.000 39 |
| 500 mg | 0.000 17 | 0.000 12 |

[3] Refer, *CCM Guidelines for approval and publication of the final reports of key and supplementary comparisons*, CCM-WGS document (2014)

## 8.1 Numerical Simulation by Monte Carlo Method (MCM)

The pilot laboratory developed its own MCM program executable under the run-time engine of National Instrument's LabVIEW by faithfully following the recommendation of Joint Committee for Guides in Metrology (JCGM) supplement document in our numerical experiment [9, 10]. The program executable with a run-time engine was distributed to all participants, and it is available to the reader by request to the pilot laboratory.

The enhanced version of the pseudo-random number generator (PRNG) of the Wichmann-Hill (WH) routine was implemented for sampling of data because some commercial random number functions like that in Microsoft's Excel have failed randomness tests [11]. WH routine has a period of $2^{121}$ (~36 decimal digits) which is quite suitable for estimating uncertainties in studies such as this comparison.

## 8.2 Input Data for Monte Carlo Method (MCM)

In the MCM numerical simulation, the input data listed in Tables 6 and 8 were used. A summary of the input quantities for the application of the Monte Carlo method is also given in Annex A.

In order to simulate the probability density function (pdf) producing the results and standard uncertainties reported by the participants, the shape of the pdf was assumed to be a normal (Gaussian) distribution; for both errors of drift and reproducibility, this uniform distribution was applied. From the resulting pdfs of the numerical simulation, the mean values were taken as the best estimation for the corresponding estimator of KCRV or other quantity (e.g. mass difference between two laboratories), and the standard deviations were taken as the standard uncertainty of the quantity, as performed in CCM.M-K6 [7].

The pilot laboratory measured two sets of travelling standards. The correlation coefficient between mass measurements done by the same laboratory was considered as $r(m_{\mathrm{KRISS},i},\ m_{\mathrm{KRISS},j}) = 0.3$ for any pair of them. The correlation coefficient indicated above, was estimated as 0.3 due to the fact that the variance contribution of the type B uncertainty is around 30 % with regard to the variance of the travelling standard estimated by the pilot laboratory. The Cholesky Factorization routine was used to realize this correlation effect for four measurements of pilot laboratory in each scale [9, 12].

## 8.3 Key Comparison Reference Value (KCRV) and Degree of Equivalence (DoE)

The median of the mass difference set, $\{\Delta m_i\}$ between results provided by participant and results reported by pilot laboratory was calculated as the best estimator of reference value for this key comparison.

$$m_{KCRV} = \mathrm{median}\{\Delta m_{i,j}\} \qquad (4)$$

Where, $i$ index of participants, and $j$ index of iterations.

The Degrees of Equivalence (DoE) is the degree to which the measured value of a participant is consistent with the KCRV. This is expressed by the deviation $D_i$ from the KCRV and the expanded uncertainty of this deviation compared at the 95 % level of confidence [9, 13] ($U(D_i)$ or 95 % coverage interval). DoE were estimated in each step of MCM by using Eq. (5) and the shortest 95 % coverage interval according to [9]. Consistence with the KCRV is given when the coverage interval of the $D_i$ includes the value zero. Although the corresponding distributions are asymmetric (cf. Annex B), the $E_{n,i}$ number was calculated using Eq. (6) in order to give a normalized value for the degree of equivalence. Here, $u_{D_i}$ denotes the standard uncertainty of $D_i$. The standard deviation of the Di was taken as an estimate of the standard uncertainty $u_{D_i}$ [9].

$$D_i = \text{mean}\{\Delta m_{i,j} - m_{KCRV,j}\} \quad (5)$$

$$E_{n,i} = \frac{|D_i|}{2 \cdot u_{D_i}} \quad (6)$$

Where, *i* index of participants, and *j* index of iterations.

**8.4 Simulation Result**

Table 9 shows the mean of each KCRV, its standard uncertainty, and the shortest 95% coverage interval obtained from $1\times10^6$ iterations. The results did not depend greatly on the number of iterations for values larger than $1\times10^5$. Figure 2 and Table 9 show graphical demonstrations and the results of numerical simulations for $DoE_i$, the mass difference between KCRV ($m_{KCRV}$) and *i*-th participant's result ($\Delta m_i$).

The detailed histograms of KCRV and participants' DoE for each standard can be found in Annex B. From the histogram, the probability density function for each physical quantity can be derived.

**Table 9.** The result of the KCRV (median) evaluated by numerical simulation. The units are mg.

| Nominal value | Mean | Standard Uncertainty | Shortest 95% Coverage Interval |
|---|---|---|---|
| 5 kg | -0.036 1 | 0.081 | [-0.196, 0.121] |
| 100 g | -0.002 1 | 0.002 8 | [-0.007 6, 0.003 2] |
| 10 g | -0.003 4 | 0.001 4 | [-0.006 2, -0.000 7] |
| 5 g | 0.000 08 | 0.000 77 | [-0.001 44, 0.001 57] |
| 500 mg | -0.000 57 | 0.000 33 | [-0.001 23, 0.000 05] |

**9. SUMMARY AND CONCLUSIONS**

This report summarizes the procedure and analysis of CCM.M-K7, a Key Comparison of 5 kg, 100 g, 10 g, 5 g and 500 mg stainless steel mass standards. Under the permission of CCM, the pilot laboratory was not involved in the nomination of participants directly, but accepted the recommendations for participants from each Regional Metrology Organization (RMO). This methodology gives the RMO's some responsibility in controlling various metrology projects.

Two sets of transfer standards were prepared, monitored, and circulated to the participant laboratories. No serious problems were encountered during the course of the key comparison.

All participants reported the results of measurements using values for their reference standards calculated following the recent amendments to the BIPM mass calibration certificates.

The median of the mass difference between results reported by participant laboratories and the pilot laboratory was taken as the key comparison reference value. All analyses were done by a computer program developed by the pilot lab to implement the Monte Carlo simulation method. In order to validate the calculation of the degrees of equivalence between results of participants and the KCRV, data analysis was repeated at the co-pilot laboratory using independently developed MCM simulation code; no significant differences were found between the values calculated by these two laboratories.

Finally, one of ten laboratories, VNIIM, was not consistent with the key comparison reference values at 100 g and 5 g within their expanded uncertainties with a coverage factor, $k = 2$, as shown in Figure 2 and Table 10.

## ACKNOWLEDGEMENTS

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M. Borys and F. Scholz would like to thank Michael Hämpke for his excellent measurements and his support for the evaluation of the results.

S. Lee would like to express his thanks to all participant laboratories and to several individuals for their help in making this comparison run smoothly: CCM President (Philippe Richard) and WGD-kg chair (Chris Sutton) for their prompt and insightful interpretations of protocol and for kindly providing urgent decisions on some critical issues; Luis Omar Becerra (CENAM) for his help on the analysis of CCM.M-K6; Michael Borys (PTB) and J. W. Chung (KRISS) for transferring their expertise of KC analysis and providing detailed comments; and Patrick Abbott (NIST) for his review of written English in this report.

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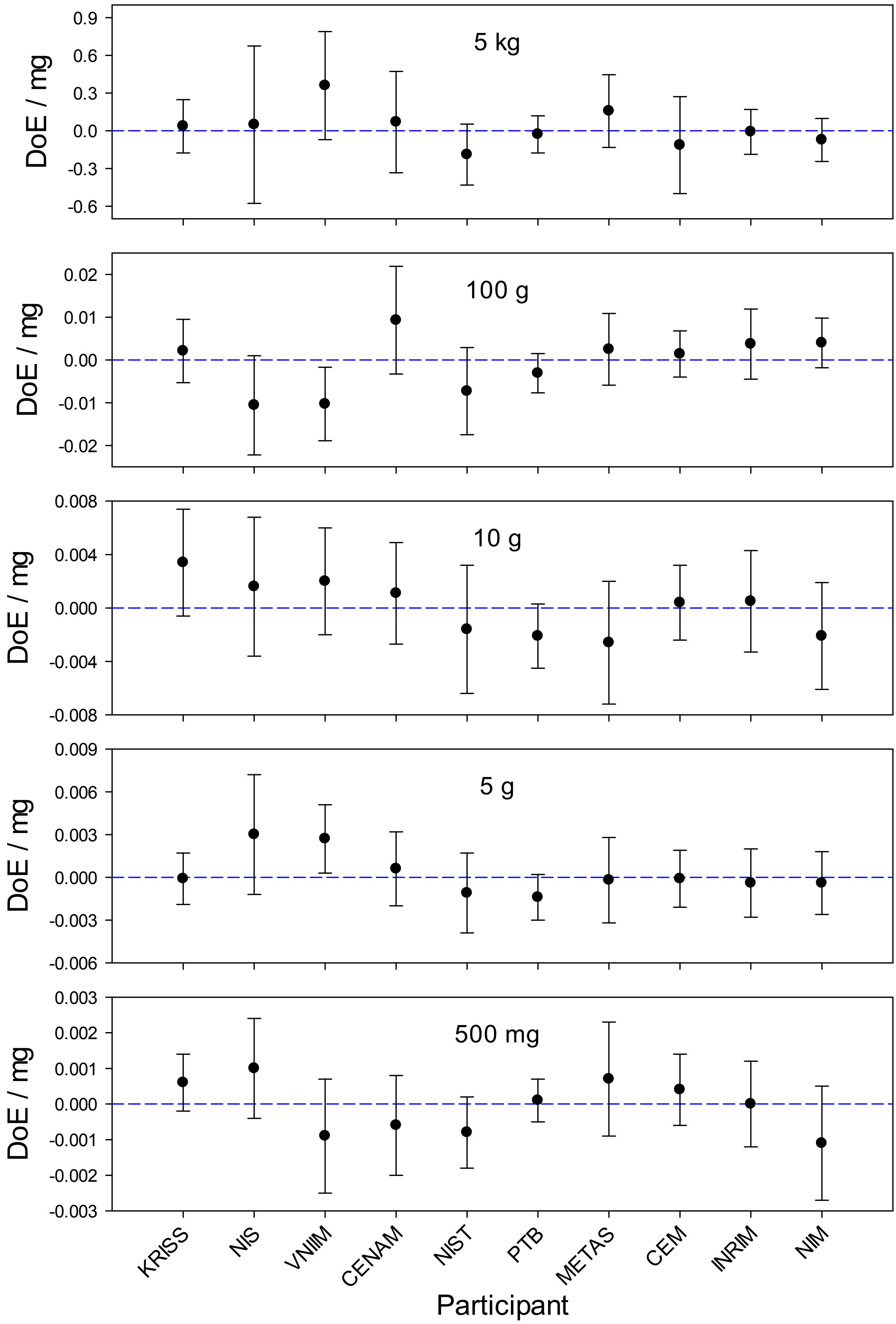

**Figure** 2. Degree of Equivalence for 5 kg, 100 g, 10 g, 5 g, and 500 mg standards.

**Table 10.** The mean, its standard uncertainty ($k$ = 1), the shortest 95% confidence interval and $E_n$ number of DoE. The units are mg.

| Nominal | | KRISS | NIS | VNIIM | CENAM | NIST | PTB | METAS | CEM | INRIM | NIM |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 5 kg | Mean | 0.036 | 0.049 | 0.359 | 0.069 | -0.189 | -0.029 | 0.157 | -0.114 | -0.009 | -0.074 |
| | Standard Uncertainty | 0.107 | 0.295 | 0.215 | 0.203 | 0.120 | 0.075 | 0.148 | 0.194 | 0.091 | 0.086 |
| | Confidence Interval | [-0.178 3, 0.260 0] | [-0.540 0, 0.641 2] | [-0.033 2, 0.786 3] | [-0.334 4, 0.483 5] | [-0.429 1, 0.021 8] | [-0.190 8, 0.115 5] | [-0.112 8, 0.460 1] | [-0.510 6, 0.262 7] | [-0.201 2, 0.177 4] | [-0.256 8, 0.077 7] |
| | $E_n$ Number | 0.17 | 0.08 | 0.83 | 0.17 | 0.79 | 0.19 | 0.53 | 0.29 | 0.05 | 0.43 |
| 100 g | Mean | 0.002 1 | -0.010 6 | -0.010 3 | 0.009 3 | -0.007 3 | -0.003 1 | 0.002 5 | 0.001 4 | 0.003 7 | 0.004 0 |
| | Standard Uncertainty | 0.003 7 | 0.005 8 | 0.004 3 | 0.006 3 | 0.005 1 | 0.002 3 | 0.004 2 | 0.002 7 | 0.004 1 | 0.002 9 |
| | Confidence Interval | [-0.004 8, 0.010 0] | [-0.022 1, 0.000 2] | [-0.018 7,-0.001 7] | [-0.001 6, 0.022 1] | [-0.017 5, 0.001 7] | [-0.007 8, 0.000 8] | [-0.005 4, 0.011 5] | [-0.003 4, 0.007 3] | [-0.003 5, 0.012 4] | [-0.000 8, 0.009 9] |
| | $E_n$ Number | 0.28 | 0.91 | 1.18 | 0.74 | 0.71 | 0.68 | 0.30 | 0.27 | 0.45 | 0.70 |
| 10 g | Mean | 0.003 4 | 0.001 6 | 0.002 0 | 0.001 1 | -0.001 6 | -0.002 1 | -0.002 6 | 0.000 4 | 0.000 5 | -0.002 1 |
| | Standard Uncertainty | 0.002 0 | 0.002 7 | 0.002 0 | 0.001 9 | 0.002 5 | 0.001 2 | 0.002 2 | 0.001 4 | 0.001 9 | 0.002 0 |
| | Confidence Interval | [-0.000 2, 0.007 5] | [-0.003 6, 0.007 1] | [-0.001 6, 0.006 1] | [-0.002 6, 0.005 1] | [-0.006 8, 0.003 0] | [-0.004 5, 0.000 1] | [-0.007 0, 0.001 3] | [-0.002 3, 0.003 3] | [-0.003 3, 0.004 6] | [-0.006 2, 0.001 4] |
| | $E_n$ Number | 0.83 | 0.30 | 0.50 | 0.28 | 0.33 | 0.87 | 0.60 | 0.15 | 0.13 | 0.53 |
| 5 g | Mean | -0.000 1 | 0.003 | 0.002 7 | 0.000 6 | -0.001 1 | -0.001 4 | -0.000 2 | -0.000 1 | -0.000 4 | -0.000 4 |
| | Standard Uncertainty | 0.000 9 | 0.002 1 | 0.001 2 | 0.001 3 | 0.001 4 | 0.000 8 | 0.001 5 | 0.001 | 0.001 2 | 0.001 1 |
| | Confidence Interval | [-0.002 0, 0.001 8] | [-0.000 9, 0.007 2] | [ 0.000 3, 0.005 1] | [-0.001 9, 0.003 3] | [-0.003 9, 0.001 4] | [-0.003 0, 0.000 0] | [-0.003 2, 0.002 8] | [-0.002 2, 0.002 0] | [-0.003 0, 0.002 1] | [-0.002 7, 0.001 8] |
| | $E_n$ Number | 0.04 | 0.71 | 1.12 | 0.24 | 0.39 | 0.89 | 0.06 | 0.04 | 0.15 | 0.17 |
| 500 mg | Mean | 0.000 6 | 0.001 0 | -0.000 9 | -0.000 6 | -0.000 8 | 0.000 1 | 0.000 7 | 0.000 4 | 0.000 0 | -0.001 1 |
| | Standard Uncertainty | 0.000 4 | 0.000 7 | 0.000 8 | 0.000 7 | 0.000 4 | 0.000 3 | 0.000 8 | 0.000 5 | 0.000 6 | 0.000 8 |
| | Confidence Interval | [-0.000 2, 0.001 5] | [-0.000 3, 0.002 5] | [-0.002 5, 0.000 5] | [-0.002 1, 0.000 8] | [-0.001 7,-0.000 0] | [-0.000 4, 0.000 8] | [-0.000 8, 0.002 3] | [-0.000 5, 0.001 4] | [-0.001 2, 0.001 1] | [-0.002 8, 0.000 3] |
| | $E_n$ Number | 0.67 | 0.64 | 0.57 | 0.38 | 0.91 | 0.23 | 0.43 | 0.39 | 0.03 | 0.70 |

## ANNEX A

### Input quantities

| $X_i$ | Distribution |
|---|---|
| $m_{\mathbf{KRISS\_1}}$ | $N(\mu,\sigma^2)$ |
| $m_{\mathbf{KRISS\_2}}$ | $N(\mu,\sigma^2)$ |
| $m_{\mathbf{KRISS\_3}}$ | $N(\mu,\sigma^2)$ |
| $m_{\mathbf{KRISS\_4}}$ | $N(\mu,\sigma^2)$ |
| $m_{\mathbf{NIS}}$ | $N(\mu,\sigma^2)$ |
| $m_{\mathbf{VNIIM}}$ | $N(\mu,\sigma^2)$ |
| $m_{\mathbf{CENAM}}$ | $N(\mu,\sigma^2)$ |
| $m_{\mathbf{NIST}}$ | $N(\mu,\sigma^2)$ |
| $m_{\mathbf{PTB}}$ | $N(\mu,\sigma^2)$ |
| $m_{\mathbf{METAS}}$ | $N(\mu,\sigma^2)$ |
| $m_{\mathbf{CEM}}$ | $N(\mu,\sigma^2)$ |
| $m_{\mathbf{INRIM}}$ | $N(\mu,\sigma^2)$ |
| $m_{\mathbf{NIM}}$ | $N(\mu,\sigma^2)$ |
| $\varepsilon_{drift,A/B}$ | $R(a,b)$ |
| $\varepsilon_{reprod,A/B}$ | $R(a,b)$ |

$N(\mu,\sigma^2)$ = Normal distribution with best estimate $\mu$ and variance $\sigma^2$,

$R(a,b)$ = Rectangular distribution with lower and upper limits *a* and *b*

### Correlations

The results $m_{\mathbf{KRISS}\_i}\,(i = 1, ..., 4)$ are correlated with the correlation matrix:

$$\begin{pmatrix} 1 & 0.3 & 0.3 & 0.3 \\ 0.3 & 1 & 0.3 & 0.3 \\ 0.3 & 0.3 & 1 & 0.3 \\ 0.3 & 0.3 & 0.3 & 1 \end{pmatrix}$$

All other input quantities are considered to be uncorrelated.

**Parameters of input quantities for the nominal value, 500 mg**

| $X_i$ | Distribution | Expectation $\mu$ | Standard dev. $\sigma$ | Lower limit $a$ | Upper limit $b$ |
|---|---|---|---|---|---|
| $m_{\mathbf{KRISS\_1}}$ | $N(\mu,\sigma^2)$ | 0.001 93 | 0.000 27 | | |
| $m_{\mathbf{KRISS\_2}}$ | $N(\mu,\sigma^2)$ | 0.002 10 | 0.000 27 | | |
| $m_{\mathbf{KRISS\_3}}$ | $N(\mu,\sigma^2)$ | 0.002 30 | 0.000 27 | | |
| $m_{\mathbf{KRISS\_4}}$ | $N(\mu,\sigma^2)$ | 0.002 90 | 0.000 27 | | |
| $m_{\mathbf{NIS}}$ | $N(\mu,\sigma^2)$ | 0.002 4 | 0.000 7 | | |
| $m_{\mathbf{VNIIM}}$ | $N(\mu,\sigma^2)$ | 0.000 54 | 0.000 75 | | |
| $m_{\mathbf{CENAM}}$ | $N(\mu,\sigma^2)$ | 0.000 89 | 0.000 71 | | |
| $m_{\mathbf{NIST}}$ | $N(\mu,\sigma^2)$ | 0.000 62 | 0.000 30 | | |
| $m_{\mathbf{PTB}}$ | $N(\mu,\sigma^2)$ | 0.002 16 | 0.000 20 | | |
| $m_{\mathbf{METAS}}$ | $N(\mu,\sigma^2)$ | 0.002 7 | 0.000 8 | | |
| $m_{\mathbf{CEM}}$ | $N(\mu,\sigma^2)$ | 0.002 41 | 0.000 48 | | |
| $m_{\mathbf{INRIM}}$ | $N(\mu,\sigma^2)$ | 0.002 0 | 0.000 6 | | |
| $m_{\mathbf{NIM}}$ | $N(\mu,\sigma^2)$ | 0.000 9 | 0.000 8 | | |
| $\varepsilon_{drift,A/B}$ | $R(a,b)$ | | | -0.000 294 | 0.000 294 |
| $\varepsilon_{reprod,A/B}$ | $R(a,b)$ | | | -0.000 207 | 0.000 207 |

**Parameters of input quantities for the nominal value, 5 g**

| $X_i$ | Distribution | Expectation $\mu$ | Standard dev. $\sigma$ | Lower limit $a$ | Upper limit $b$ |
|---|---|---|---|---|---|
| $m_{\mathbf{KRISS\_1}}$ | $N(\mu,\sigma^2)$ | 0.007 7 | 0.000 43 | | |
| $m_{\mathbf{KRISS\_2}}$ | $N(\mu,\sigma^2)$ | 0.008 5 | 0.000 43 | | |
| $m_{\mathbf{KRISS\_3}}$ | $N(\mu,\sigma^2)$ | 0.007 9 | 0.000 43 | | |
| $m_{\mathbf{KRISS\_4}}$ | $N(\mu,\sigma^2)$ | 0.010 1 | 0.000 43 | | |
| $m_{\mathbf{NIS}}$ | $N(\mu,\sigma^2)$ | 0.011 2 | 0.002 0 | | |
| $m_{\mathbf{VNIIM}}$ | $N(\mu,\sigma^2)$ | 0.010 9 | 0.000 9 | | |
| $m_{\mathbf{CENAM}}$ | $N(\mu,\sigma^2)$ | 0.008 8 | 0.001 1 | | |
| $m_{\mathbf{NIST}}$ | $N(\mu,\sigma^2)$ | 0.007 1 | 0.001 2 | | |
| $m_{\mathbf{PTB}}$ | $N(\mu,\sigma^2)$ | 0.007 7 | 0.000 4 | | |
| $m_{\mathbf{METAS}}$ | $N(\mu,\sigma^2)$ | 0.008 9 | 0.001 5 | | |
| $m_{\mathbf{CEM}}$ | $N(\mu,\sigma^2)$ | 0.009 | 0.000 87 | | |
| $m_{\mathbf{INRIM}}$ | $N(\mu,\sigma^2)$ | 0.008 7 | 0.001 2 | | |
| $m_{\mathbf{NIM}}$ | $N(\mu,\sigma^2)$ | 0.008 7 | 0.001 0 | | |
| $\varepsilon_{drift,A/B}$ | $R(a,b)$ | | | -0.001 21 | 0.001 21 |
| $\varepsilon_{reprod,A/B}$ | $R(a,b)$ | | | -0.000 675 | 0.000 675 |

**Parameters of input quantities for the nominal value, 10 g**

| $X_i$ | Distribution | Expectation $\mu$ | Standard dev. $\sigma$ | Lower limit $a$ | Upper limit $b$ |
|---|---|---|---|---|---|
| $m_{\mathbf{KRISS\_1}}$ | $N(\mu,\sigma^2)$ | 0.011 6 | 0.001 5 | | |
| $m_{\mathbf{KRISS\_2}}$ | $N(\mu,\sigma^2)$ | 0.010 0 | 0.001 5 | | |
| $m_{\mathbf{KRISS\_3}}$ | $N(\mu,\sigma^2)$ | 0.012 0 | 0.001 5 | | |
| $m_{\mathbf{KRISS\_4}}$ | $N(\mu,\sigma^2)$ | 0.012 5 | 0.001 5 | | |
| $m_{\mathbf{NIS}}$ | $N(\mu,\sigma^2)$ | 0.009 | 0.002 6 | | |
| $m_{\mathbf{VNIIM}}$ | $N(\mu,\sigma^2)$ | 0.009 4 | 0.001 7 | | |
| $m_{\mathbf{CENAM}}$ | $N(\mu,\sigma^2)$ | 0.008 5 | 0.001 7 | | |
| $m_{\mathbf{NIST}}$ | $N(\mu,\sigma^2)$ | 0.005 8 | 0.002 4 | | |
| $m_{\mathbf{PTB}}$ | $N(\mu,\sigma^2)$ | 0.006 8 | 0.000 5 | | |
| $m_{\mathbf{METAS}}$ | $N(\mu,\sigma^2)$ | 0.006 3 | 0.002 0 | | |
| $m_{\mathbf{CEM}}$ | $N(\mu,\sigma^2)$ | 0.009 3 | 0.001 1 | | |
| $m_{\mathbf{INRIM}}$ | $N(\mu,\sigma^2)$ | 0.009 4 | 0.001 9 | | |
| $m_{\mathbf{NIM}}$ | $N(\mu,\sigma^2)$ | 0.006 8 | 0.001 8 | | |
| $\varepsilon_{drift,A/B}$ | $R(a,b)$ | | | -0.000 796 | 0.000 796 |
| $\varepsilon_{reprod,A/B}$ | $R(a,b)$ | | | -0.001 19 | 0.001 19 |

**Parameters of input quantities for the nominal value, 100 g**

| $X_i$ | Distribution | Expectation $\mu$ | Standard dev. $\sigma$ | Lower limit $a$ | Upper limit $b$ |
|---|---|---|---|---|---|
| $m_{\mathbf{KRISS\_1}}$ | $N(\mu,\sigma^2)$ | 0.017 9 | 0.002 5 | | |
| $m_{\mathbf{KRISS\_2}}$ | $N(\mu,\sigma^2)$ | 0.012 6 | 0.002 5 | | |
| $m_{\mathbf{KRISS\_3}}$ | $N(\mu,\sigma^2)$ | 0.010 9 | 0.002 5 | | |
| $m_{\mathbf{KRISS\_4}}$ | $N(\mu,\sigma^2)$ | 0.005 6 | 0.002 5 | | |
| $m_{\mathbf{NIS}}$ | $N(\mu,\sigma^2)$ | 0.002 6 | 0.005 0 | | |
| $m_{\mathbf{VNIIM}}$ | $N(\mu,\sigma^2)$ | 0.002 9 | 0.003 0 | | |
| $m_{\mathbf{CENAM}}$ | $N(\mu,\sigma^2)$ | 0.022 5 | 0.005 7 | | |
| $m_{\mathbf{NIST}}$ | $N(\mu,\sigma^2)$ | 0.005 9 | 0.004 2 | | |
| $m_{\mathbf{PTB}}$ | $N(\mu,\sigma^2)$ | 0.003 1 | 0.001 5 | | |
| $m_{\mathbf{METAS}}$ | $N(\mu,\sigma^2)$ | 0.008 7 | 0.004 5 | | |
| $m_{\mathbf{CEM}}$ | $N(\mu,\sigma^2)$ | 0.007 6 | 0.002 6 | | |
| $m_{\mathbf{INRIM}}$ | $N(\mu,\sigma^2)$ | 0.009 9 | 0.004 2 | | |
| $m_{\mathbf{NIM}}$ | $N(\mu,\sigma^2)$ | 0.010 2 | 0.002 4 | | |
| $\varepsilon_{drift,A/B}$ | $R(a,b)$ | | | -0.002 59 | 0.002 59 |
| $\varepsilon_{reprod,A/B}$ | $R(a,b)$ | | | -0.002 59 | 0.002 59 |

**Parameters of input quantities for the nominal value, 5 kg**

| $X_i$ | Distribution | Expectation $\mu$ | Standard dev. $\sigma$ | Lower limit $a$ | Upper limit $b$ |
|---|---|---|---|---|---|
| $m_{\mathbf{KRISS\_1}}$ | $N(\mu,\sigma^2)$ | 0.135 | 0.073 | | |
| $m_{\mathbf{KRISS\_2}}$ | $N(\mu,\sigma^2)$ | 0.040 | 0.073 | | |
| $m_{\mathbf{KRISS\_3}}$ | $N(\mu,\sigma^2)$ | -0.271 | 0.073 | | |
| $m_{\mathbf{KRISS\_4}}$ | $N(\mu,\sigma^2)$ | -0.310 | 0.073 | | |
| $m_{\mathbf{NIS}}$ | $N(\mu,\sigma^2)$ | 0.10 | 0.30 | | |
| $m_{\mathbf{VNIIM}}$ | $N(\mu,\sigma^2)$ | 0.41 | 0.20 | | |
| $m_{\mathbf{CENAM}}$ | $N(\mu,\sigma^2)$ | 0.12 | 0.20 | | |
| $m_{\mathbf{NIST}}$ | $N(\mu,\sigma^2)$ | -0.138 | 0.085 | | |
| $m_{\mathbf{PTB}}$ | $N(\mu,\sigma^2)$ | -0.355 | 0.056 | | |
| $m_{\mathbf{METAS}}$ | $N(\mu,\sigma^2)$ | -0.170 | 0.146 | | |
| $m_{\mathbf{CEM}}$ | $N(\mu,\sigma^2)$ | -0.440 | 0.2 | | |
| $m_{\mathbf{INRIM}}$ | $N(\mu,\sigma^2)$ | -0.335 | 0.085 | | |
| $m_{\mathbf{NIM}}$ | $N(\mu,\sigma^2)$ | -0.40 | 0.07 | | |
| $\varepsilon_{drift,A/B}$ | $R(a,b)$ | | | -0.046 7 | 0.046 7 |
| $\varepsilon_{reprod,A/B}$ | $R(a,b)$ | | | -0.096 9 | 0.096 9 |

## ANNEX B

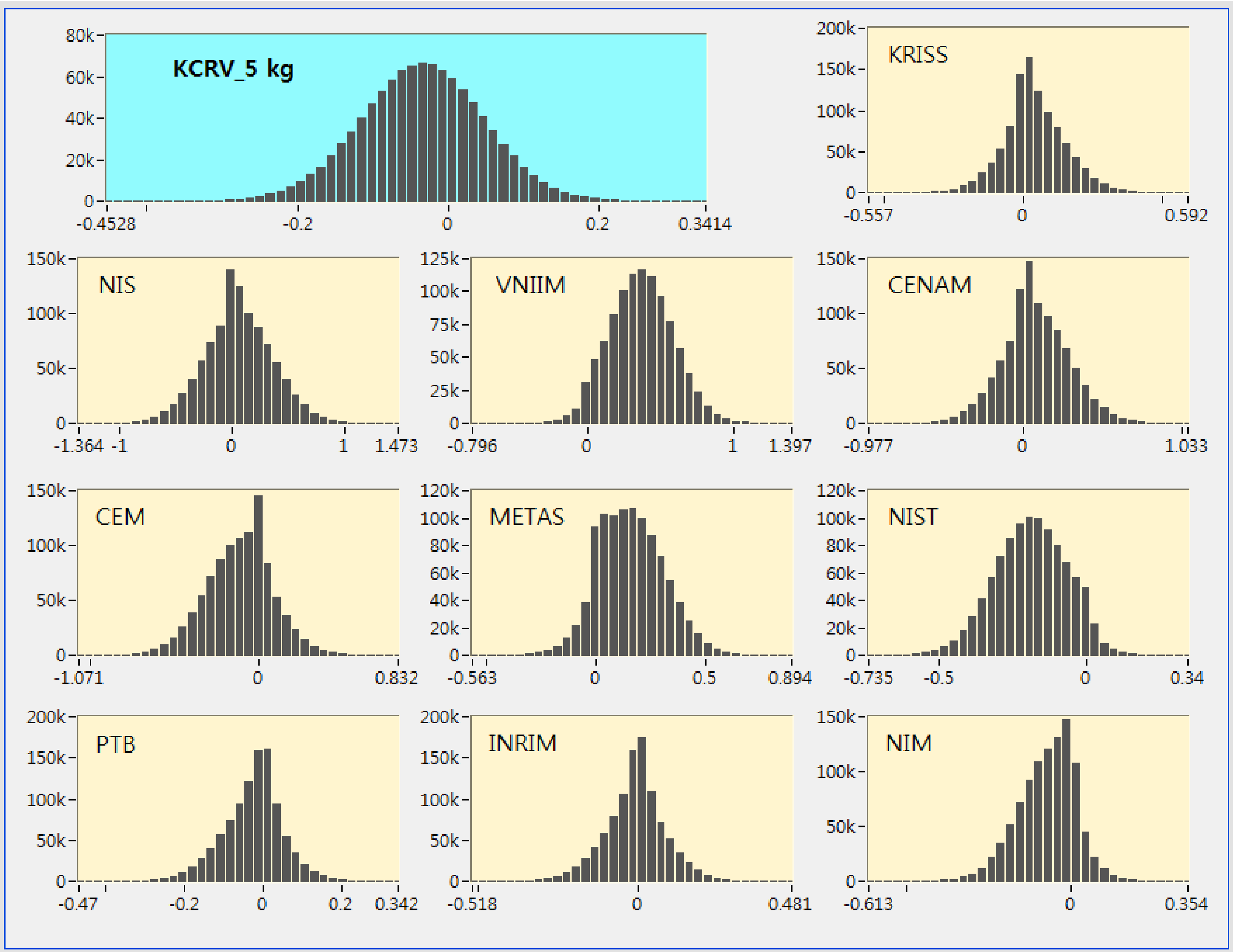


**Fig**. 3. The histograms of KCRV and DoE for 5 kg standard

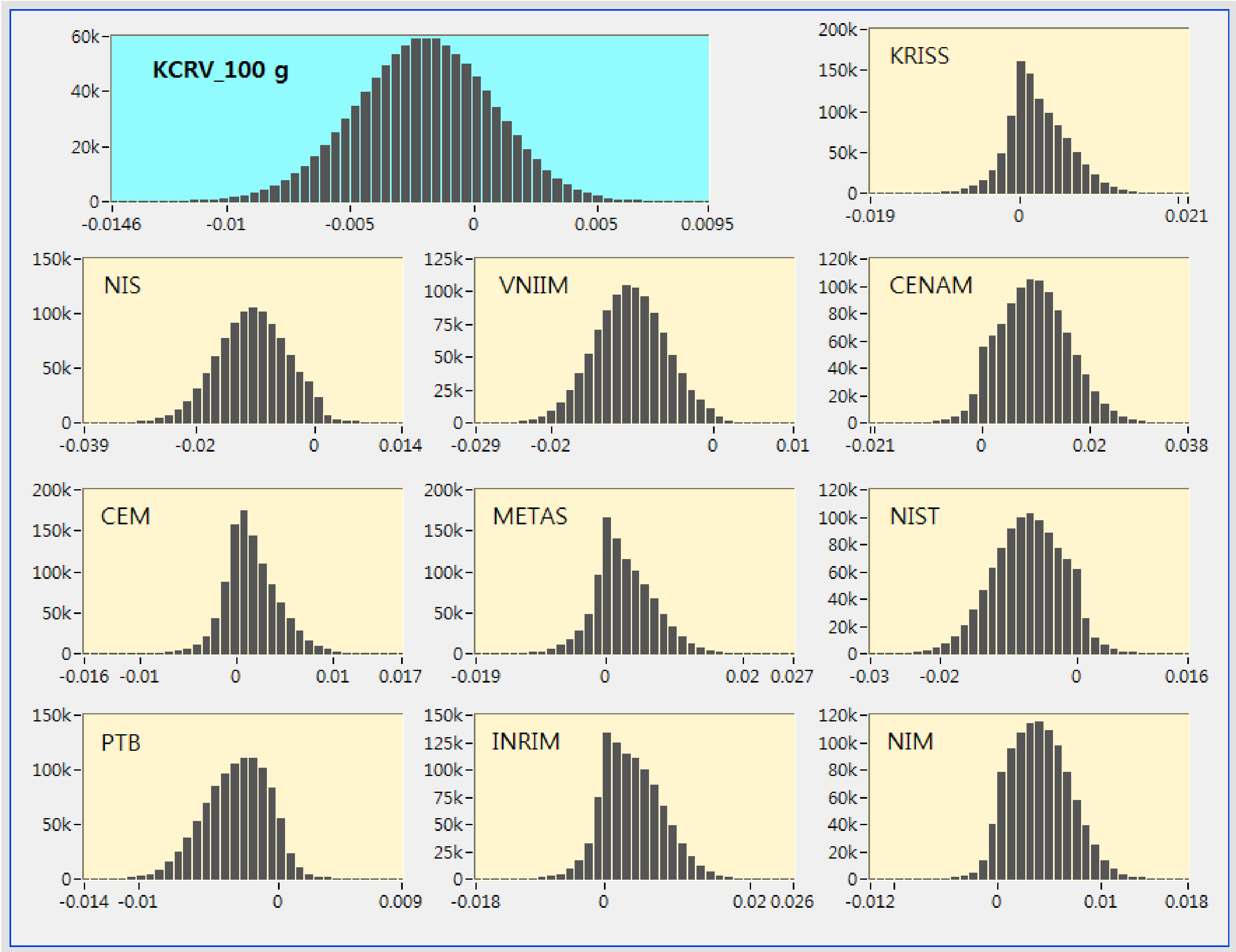


**Fig**. 4. The histograms of KCRV and DoE for 100 g standard

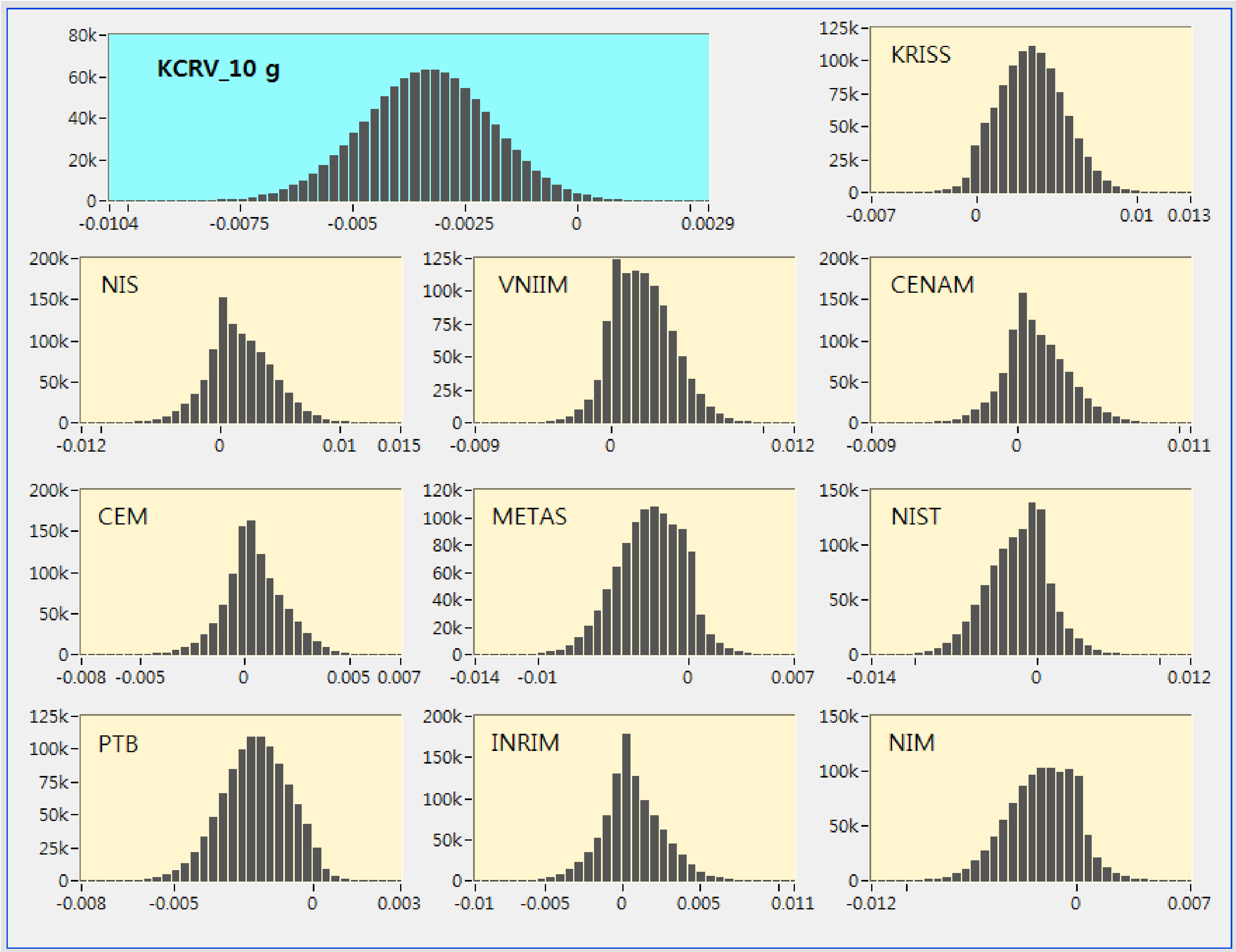


**Fig**. 5. The histograms of KCRV and DoE for 10 g standard

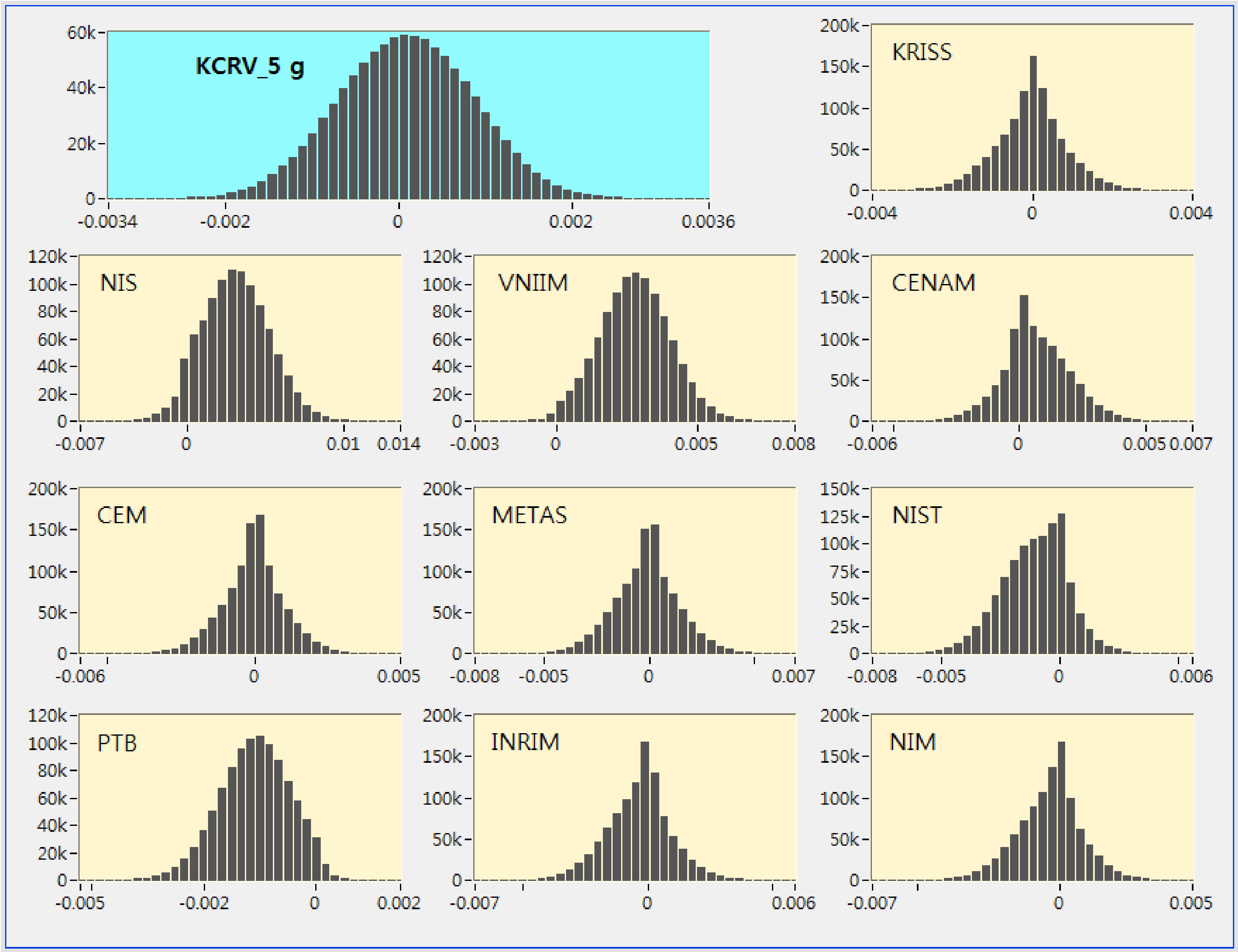


**Fig**. 6. The histograms of KCRV and DoE for 5 g standard

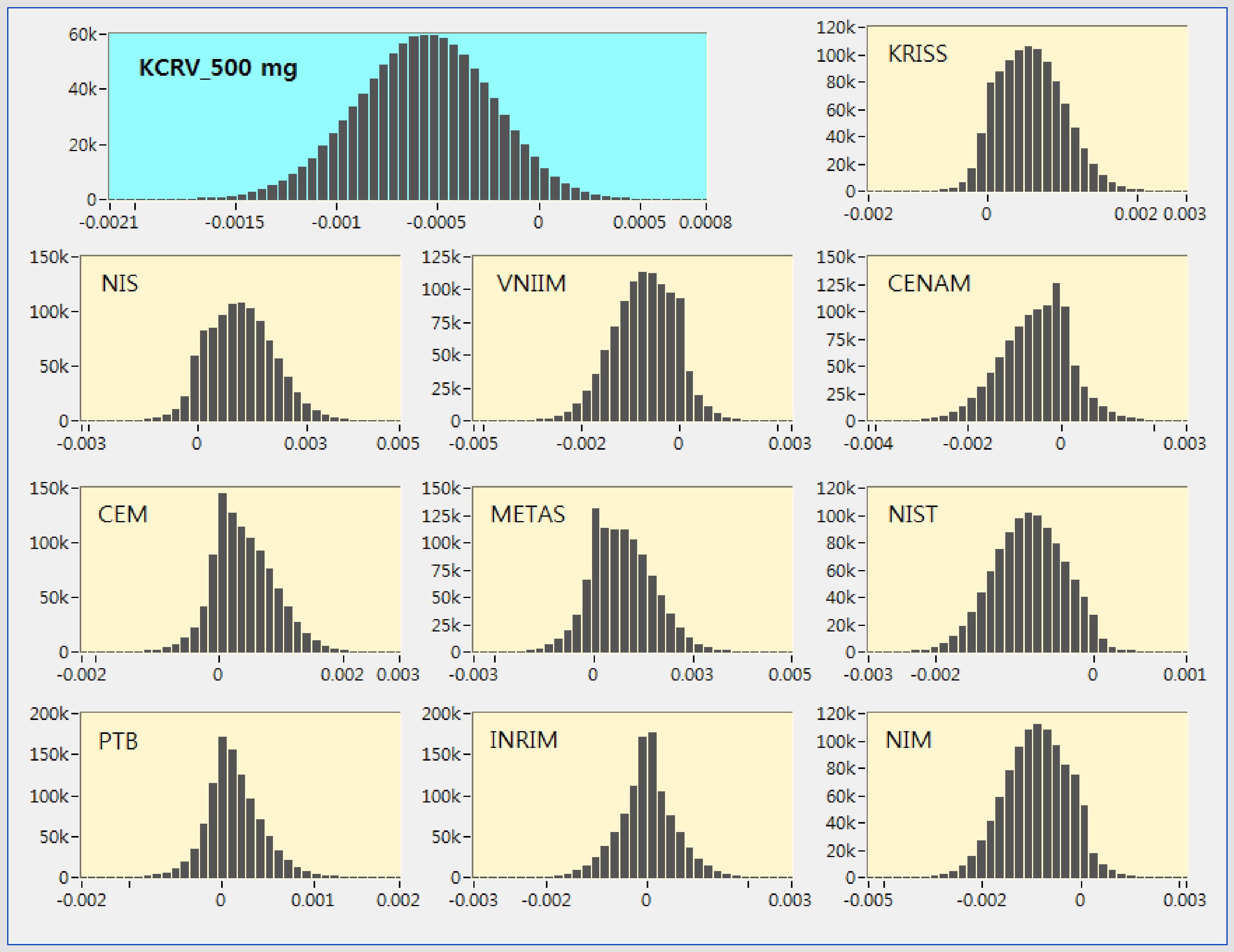


**Fig**. 7. The histograms of KCRV and DoE for 500 mg standard