ACTA IMEKO
ISSN: 2221-870X
September 2025, Volume 14, Number 3, 1 - 5

Potassium solution Certified Reference Material production at Inmetro

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ABSTRACT

This paper presents the production of a potassium solution Certified Reference Material (CRM), MRC 8757.0001. The CRM was produced by the formulation method, using a high-purity potassium chloride (KCl) certified for purity at Inmetro, in compliance with ISO 17034, ISO 33405, and internal standards. High-Performance Inductively Coupled Plasma Optical Emission Spectrometry (HP-ICP-OES) was used to assess the homogeneity, short-term stability (at 50 °C), and long-term stability (between 20 °C and 25 °C) of the CRM. The certified potassium mass fraction, with all masses corrected for air buoyancy, was (1002 ± 11) mg/kg, considering k = 2.02, at a 95 % confidence level. With both the formulation method as a characterisation strategy and HP-ICP-OES for certification studies, we achieved a 47 % reduction of the combined uncertainty when compared to previous procedures at Inmetro. This CRM supports Inmetro's objective to address the demand for metrological traceability of Brazilian organizations.

Section: RESEARCH PAPER

Keywords: calibration solution; uncertainty; metrological traceability; Certified Reference Material

Citation: A. C. Palacios Osorio, R. Casiano de Sena, M. Domínguez de Almeida, Potassium solution Certified Reference Material production at Inmetro, Acta IMEKO, vol. 14 (2025) no. 3, pp. 1-5. DOI: 10.21014/actaimeko.v14i3.1974

Section Editor: Daniel Ramos Louzada, PósMQI/PUC-Rio, Rio de Janeiro, Brazil

Received November 8, 2024; **In final form** September 1, 2025; **Published** September 2025

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1. INTRODUCTION

Calibration solutions are a specific type of Certified Reference Material (CRM) that underpin elemental determinations [1] for most instrumental techniques, such as Atomic Absorption Spectrometry (AAS), Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES), and Inductively Coupled Plasma Mass Spectrometry (ICP-MS). These CRMs are widely used by chemical analysis laboratories to calibrate their instruments and to provide accuracy, reliability, and metrological traceability to their measurement results, especially if the laboratory is accredited or seeking ISO 17025 accreditation [2].

Calibration solutions can be characterised using the mass or volume of the materials used in their preparation [3], also called the formulation method [4], which involves precise mass or volume measurements. ISO 33405 states that in order to apply this strategy, the purity of the material used for CRM preparation needs to be known and traceable to the International System of Units (SI). However, most CRM producers use commercially available substances with their purity based on the manufacturer's certificate, which lacks this traceability statement [5]. To achieve SI traceability of calibration solutions, the use of primary pure substances is essential [5], [6]. The characterisation of such primary pure substances usually requires the use of the mass balance approach—also called the primary difference method [6], [7]. This approach, which represents the realisation of the SI unit amount of substance, demands high technical skills and a great financial investment. As a result, it is undertaken only by a small number of National Metrology Institutes (NMIs) and Designated Institutes (DIs) [6], [7].

In order to support Inmetro's objective to address the demand for metrological traceability of Brazilian organizations, the Inorganic Analysis Laboratory (Labin) from the Chemical Metrology Department at Inmetro recently started to produce calibration solutions. Nevertheless, initial efforts revealed that the obtained measurement uncertainties needed significant improvement [8]. In order to achieve better results, in addition to the use of primary materials [9], we applied newer measurement methodologies, such as HP-ICP-OES.

HP-ICP-OES is an analytical method that employs exact matrix matching, internal standardisation, and a polynomial modelling of instrumental drift that improves accuracy and diminishes uncertainties [10], [11]. It has also found application within the metrological community [12], [13].

In this work, we detail the production and certification of MRC 8757.0001, a potassium single-element calibration solution. This CRM was gravimetrically prepared from a primary potassium chloride (KCl), assessed for purity at Inmetro [9], and was certified according to ISO 17034, ISO 33405, and Inmetro's internal standards. Homogeneity, as well as transportation and storage stability, were assessed using HP-ICP-OES. The potassium mass fraction was characterised by the formulation method and confirmed with HP-ICP-OES. To the best of our knowledge, this is the first time that these procedures have been applied in Brazil for the production of a CRM.

2. EXPERIMENTAL BACKGROUND

KCl-Inmetro-1 and KCl-Inmetro-2, assessed at Inmetro for purity [9], were used to prepare and certify the CRM. SRM 999b (NIST, USA) was used for accuracy assessment, and SRM 3153a (NIST, USA) was used as a strontium source. All other chemicals were of analytical grade.

KCl-Inmetro-1 was dried in a muffle furnace (Quimis, Brazil) for 4 h at 500 °C, and cooled in a desiccator. This salt was weighed (31.09682 g) on a calibrated analytical balance (Sartorius, Germany) and quantitatively transferred to a pre-weighed 50 L polypropylene container filled with about 12 L of Grade 1 [14] purified water (Millipore, USA). The mass was adjusted with Grade 1 water and sub-boiled (Duopur, USA) nitric acid (65 %, Merck, Germany), to obtain the desired mass (16287.88 g) and HNO_3 content (a volume fraction of 3 %). This solution was homogenised with a magnetic stirrer for 24 h. Each unit (n = 152) was prepared by adding 100 mL of the solution to a 125 mL capped high-density polyethylene bottle, protected with a polypropylene bag and an aluminised pouch.

Calibrant or control solutions were prepared by diluting muffle-dried KCl with HNO_3 of a volume fraction of 3 %, to obtain 100 g of a 1000 mg/kg potassium solution. In order to correctly apply HP-ICP-OES, the mass fraction variation of these solutions and subsequent dilutions was maintained below 0.5 %.

The HP-ICP-OES analysis was performed on an ICP-OES Optima 8300 (Perkin Elmer, USA) equipped with a Scott chamber, a crossflow nebulizer and an S10 autosampler. The ICP-OES conditions were a plasma, auxiliary, nebulizer, and pump flow rates of 12 L/min, 0.5 L/min, 0.9 L/min, and 1.0 mL/min, respectively, with an axial view and 1100 W. Data were acquired using peak area (5 points per peak), with an integration time of 0.010 s and a read time of 8.0 s. Potassium, K (3.6 mg/kg), was detected at 766 nm. Strontium, Sr (0.17 mg/kg), was used as an internal standard and was detected at 407 nm. Analysis was performed using five randomized runs, alternating between calibrants and samples or control solutions, to minimize instrumental drift.

3. CALCULATIONS

3.1. Gravimetric batch characterisation

All of the masses obtained to prepare the batch, calibrant, or control solutions were buoyancy corrected, using equation (1) [15], where m_c is the corrected mass, m_i is the experimental mass, d_ar is the air density (0.0012 g/cm^3 [15]), d_i is the density of KCl (2,13 gm/cm^3 [16]), and d_ref is 8.000 g/cm^3, the density of a weight made of stainless steel [15]:

$$m_\mathrm{c} = m_\mathrm{i} + d_\mathrm{ar} \cdot \left(\frac{m_\mathrm{i}}{d_\mathrm{i}} + \frac{m_\mathrm{i}}{d_\mathrm{ref}}\right). \tag{1}$$

The uncertainty of the measured masses was obtained from the balances' certificates. The uncertainties obtained from the reference documents were calculated using $u_\mathrm{i} = 10^{-y}/\sqrt{3}$ (rectangular distribution approach [17]), where u_i is an uncertainty and y are significant figures. The spreadsheet method proposed by Kragten was used to propagate these components and calculate the combined uncertainty of the corrected mass, $u(m_\mathrm{c})$, [18].

The potassium mass fraction, w_K, was calculated using equation (2), where m_KCl is the buoyancy-corrected KCl-Inmetro-1 mass (mg), m_sol is the buoyancy-corrected mass of the final solution, FE is the stoichiometric factor, P is the certified purity (mg/mg) of KCl-Inmetro-1. The stoichiometric factor (potassium fraction in KCl) was calculated using the standard atomic weights of the elements [19]. The mass fraction uncertainty, $u(w_\mathrm{K})$, was calculated using the relative approach [17].

$$w_\mathrm{K} = \frac{FE \cdot P \cdot m_\mathrm{KCl}}{m_\mathrm{sol}}. \tag{2}$$

Between-unit variation assessment (i.e. homogeneity) was performed using potassium mass fraction measurements in a single run. The results were analysed with one-way ANOVA (Analysis of Variance) to determine if the between-unit variation is not statistically significant at a 95 % confidence level.

Transportation stability was assessed using an isochronous design by studying the effects of multiple points in time (0, 5, 12, 19, 24, or 31 days) at a single storage condition (50 °C) on the potassium mass fraction. The results were analysed using simple linear regression to determine if the slope was not statistically significant (95 % confidence level).

Storage stability was assessed using a classical design, by studying the potassium mass fraction under storage conditions (between 20 °C and 25 °C) for 188 to 538 days. Measurements were performed in monthly intervals. The results were also analysed with simple linear regression to assess the statistical significance of the slope.

Repeated subsampling was also assessed by analysing the potassium mass fraction of the same unit, maintained under storage conditions, in monthly intervals. Simple linear regression was used for data analysis.

Stratified random sampling was used for unit selection in all of the aforementioned studies. Data was assessed for outliers using the interquartile range approach [20].

The standard uncertainty associated with heterogeneity was calculated using ANOVA results and equation (3) [4], in which MQ_b is the between-unit mean square, MQ_w is the within-unit mean square, and n is the number of subsamples

$$u_\mathrm{h} = \frac{MQ_\mathrm{b} - MQ_\mathrm{w}}{n}. \tag{3}$$

The standard uncertainty, associated with the transportation stability of the material (u_sts), was calculated according to equation (4) [4], in which s_b is the standard error for the estimated slope, and t is the duration of the study in days. The same equation was used to calculate the standard uncertainty associated with storage stability (u_lts):

$$u_{\mathrm{sts}} = s_{\mathrm{b}} \cdot t \, . \quad (4)$$

The combined standard uncertainty (u_{c}) was calculated using equation (5), in which u_{ch} is the characterisation standard uncertainty, and the other uncertainty components were previously described:

$$u_{\mathrm{c}} = \sqrt{u_{\mathrm{h}}^2 + u_{\mathrm{ch}}^2 + u_{\mathrm{sts}}^2 + u_{\mathrm{lts}}^2} \, . \quad (5)$$

To determine the appropriate coverage factor (k), effective degrees of freedom, ϑ_{eff}, were calculated using equation (6) [17], in which u_i is an uncertainty component (long-term stability, short-term stability, characterisation, or homogeneity), and ϑ_i represents the degrees of freedom of each uncertainty component:

$$\vartheta_{\mathrm{eff}} = \frac{u_{\mathrm{c}}^4}{\sum_{i=1}^{n} \frac{u_i^4}{\vartheta_i}} \, . \quad (6)$$

To compare potassium mass fractions obtained from two different methods, we used equation (7) [4], where w_1 and w_2 are mass fractions obtained with different methods, k is the coverage factor, $u(w_1)$ and $u(w_2)$ are uncertainties of those methods:

$$|w_1 - w_2| \leq k \cdot \sqrt{u_{w_1}^2 + u_{w_2}^2} \, . \quad (7)$$

4. RESULTS

4.1. Homogeneity and stability assessment

The assessment criteria, results, and standard uncertainties obtained for homogeneity and stability studies are summarised in Table 1. A non-significant between-unit variation for an ANOVA and a non-significant slope for a linear regression were considered adequate for these assessments.

A one-way ANOVA for homogeneity assessment confirmed that the potassium mass fraction was not significantly different between the units (Table 1), hence, the batch was considered homogeneous. Furthermore, to identify trends arising from processing, a simple linear regression was applied to the homogeneity data using the potassium mass fraction as the dependent variable and the processing order as the independent variable. A non-significant slope (p-value 0.31, 95 % confidence level) indicated that there were no processing trends.

Three types of stability were assessed to ensure the integrity of the CRM over time and under different conditions: transportation, storage, and repeated subsampling of the same unit. For transportation stability assessment, a linear regression model was constructed with the duration of the study as the independent variable and the potassium mass fraction as the dependent variable. The results are plotted in Figure 1a, where the abscissa represents the time in days that a unit was submitted at the studied temperature of 50 °C, and the ordinate is the potassium mass fraction with error bars representing a 95 % Confidence Interval (CI). A non-significant slope (Table 1) showed that the potassium mass fraction remained stable at 50 °C for 31 days.

Table 1. Certification studies for MRC 8757.0001.

Study	Statistical assessment	Assessment result	Uncertainty result	ϑi
Homogeneity	Anova	Non-significant between-unit variation ($F_{\mathrm{calculated}}$ 1.73 < F_{critical} 2.12)	2.3 mg/kg	40
Transportation stability (50 °C)	Linear regression	Non-significant slope p-value 0.31, 95 % confidence level	3.7 mg/kg	10
Storage stability (between 20 °C and 25 °C)	Linear regression	Non-significant slope p-value 0.21, 95 % confidence level	2.4 mg/kg	43

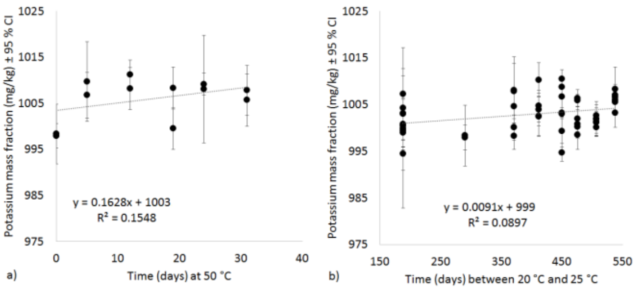

Figure 1. Certification studies for a) transport and b) storage stability.

For the storage stability assessment, we constructed a similar regression model, including monthly assays of the potassium mass fraction from selected units, as well as the results obtained from the homogeneity and storage stability units maintained under storage conditions. These results are summarised in Figure 1b, where the abscissa represents the time in days that a unit was maintained at the studied temperature (between 20 °C and 25 °C), and the ordinate is the potassium mass fraction with error bars representing a 95 % CI. The regression analysis also showed a non-significant slope (Table 1), confirming that the potassium mass fraction remained stable at the selected storage conditions.

The repeated subsampling of the same unit kept under storage conditions was also evaluated with a linear regression model, in which the variability of the potassium mass fraction was assessed over time (days). A non-significant slope (p-value of 0.18, 95 % confidence level) was also obtained. Since this assessment is not required under ISO 33405 uncertainty budget, and storage stability was already accounted for, we did not include the repeated sampling in the combined uncertainty calculation.

4.2. Characterisation

To apply the formulation method thoroughly, in addition to the requirements of ISO 17034 and ISO 33405, we considered the need for the buoyancy correction of the experimentally obtained KCl-Inmetro-1 and batch masses. According to the International Organization of Legal Metrology (OIML) [15], buoyancy correction may be applied if the uncorrected value exceeds a specification. In our case, this specified value was a significant difference between corrected and non-corrected values. To do so, we calculated the potassium mass fraction obtained by using the non-corrected experimental masses (Section 2) and the mass fraction obtained from the buoyancy-corrected data (Table 2). When applying equation (7) (considering a $k = 2$ coverage factor at a 95 % confidence level), we found a significant difference between both values, and we concluded that buoyancy correction is necessary to apply the formulation method for calibration solution characterisation.

To calculate the potassium mass fraction, we used the formulation method and the input quantities summarised in Table 2. After applying equation (2), we obtained a mass fraction of 1000.587 mg/kg with an uncertainty of 0.034 mg/kg. As

Table 2. Input quantities for MRC 8757.0001 gravimetric characterisation.

Input quantity	**Value**	**Standard uncertainty**
Buoyancy corrected KCl-Inmetro-1 mass	31110.960 mg	00.040 mg
Buoyancy corrected batch mass	16.30495 kg	0.000071 kg
Purity of KCl-Inmetro-1	0.999904 mg/mg	0.000030 mg/mg
Stoichiometric factor	0.5244483	0.0000071

stated in ISO 33405, the confirmation by an independent measurement procedure is advisable to provide confidence in the results. The mean potassium mass fraction obtained from HP-ICP-OES measurements, obtained during the long-term stability study, served as this confirmation value: 1003.2 mg/kg with a standard uncertainty of 1.9 mg/kg. We compared this result with the one obtained with the formulation method using equation (7). The difference between the absolute value of both mass fractions (2.6 mg/kg) was less than their combined standard uncertainty (4.2 mg/kg) multiplied by a coverage factor (k = 2.18, 95 % confidence level), therefore, there was a non-significant difference between the two results. According to ISO 33405, the value obtained from the formulation method is sufficient for characterisation, since the confirmation method usually has a greater uncertainty. Nevertheless, we preferred a more conservative approach, and combined both results. The potassium mass fraction in the CRM was 1001.9 mg/kg, and was calculated using the mean of the formulation and HP-ICP-OES mass fractions. The characterisation uncertainty was obtained using the summation in quadrature of both uncertainties (formulation and HP-ICP-OES), resulting in 1.9 mg/kg.

4.3. Improvement of uncertainties

The application of HP-ICP-OES had an important role in diminishing the uncertainties obtained in the certification studies of calibration solutions performed at Inmetro. When we applied this approach, we obtained a significant improvement across all uncertainty components. Figure 2 summarises the results obtained for MRC 8757.0001 and HP-ICP-OES, alongside another calibration solution prepared and certified at Inmetro using conventional ICP-OES and external calibration [8], [21]. In that figure, each uncertainty contribution is expressed as a relative value, with conventional ICP-OES results in light gray bars and HP-ICP-OES results in black bars. Applying this highly accurate measurement procedure significantly reduced all sources of uncertainty, with the most substantial improvement observed in the characterisation uncertainty, which decreased from 0.76 % to 0.19 %. As a result, the relative combined uncertainty for MRC 8757.0001 was reduced by 47 %.

4.4. Value assignment

The results for the uncertainty of homogeneity, transportation stability, storage stability, and characterisation of MRC 8757.0001 were summarised in Table 1 and described in Section 4.2. The major contribution to the combined uncertainty of the potassium mass fraction in MRC 8757.0001 was transportation stability (Figure 2).

Using characterisation, homogeneity, transportation and storage stability standard uncertainties, and the equations provided in Section 2, the combined standard uncertainty was 5.3 mg/kg. The certified potassium mass fraction in the batch was 1002 mg/kg, with an expanded uncertainty of 11 mg/kg, considering a coverage factor of $k = 2.02$ and a confidence level of 95 %.

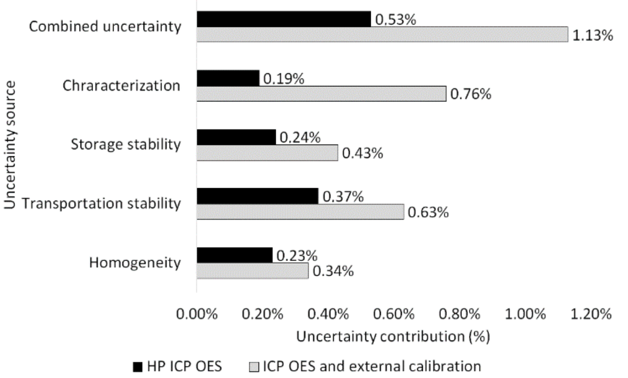

Figure 2. Uncertainties obtained for another CRM with ICP-OES and external calibration [21], and for MRC 8757.0001 and HP-ICP-OES

4.5. Comparison with international efforts to achieve traceability

The production and certification process of this potassium calibration solution was reviewed and approved within the CIPM MRA (International Committee for Weights and Measures Mutual Recognition Arrangement), and was recognised as Calibration and Measurement Capability (CMC) SIM-QM-BR-00000NQT-1 [22]. CMCs are directly related to the ability of an NMI to characterise and assign traceable values to CRMs. They also attest to the Institute's technical competence and help address measurement challenges in their countries [23]. Therefore, Brazilian laboratories now have a reliable and accessible primary standard that is a traceability source for the measurement of potassium. Currently, only eight countries, including Brazil, have a similar CMC for potassium [23], thus, the availability of MRC 8757.0001 contributes to increasing the worldwide supply of reliable traceability sources for chemical measurements.

5. CONCLUSION

MRC 8757.0001 (Figure 3), a potassium calibration solution CRM, was successfully produced and certified at Inmetro according to ISO 17034, ISO 33405, and Inmetro's internal standards.

The CRM is homogeneous and stable under transportation (50 °C) and storage conditions (between 20 °C and 25 °C) and can be sampled repeatedly. The certified potassium mass fraction was 1002 mg/kg, with an expanded uncertainty of 11 mg/kg, considering a coverage factor of $k = 2.02$ and a confidence level of 95 %. The certified value has metrological traceability to the SI using an internally certified primary material and through gravimetric preparation.

The CRM provides metrological traceability for potassium measurement results obtained using AAS, ICP-OES, ICP-MS, and other calibration-based techniques.

The use of a primary material, the formulation approach, and HP-ICP-OES reduced uncertainties by up to 47 %, improving the production and certification processes of calibration solutions at Inmetro.

Encouraged by these results, the Inorganic Analysis Laboratory at Inmetro plans to produce more calibration solutions and multi-elemental solutions that are strategic for measurements performed at Brazilian laboratories, required by food, health, and environmental legislation, further strengthening Brazil's metrological capabilities and its role in the global measurement system.

Figure 3. MRC 8757.0001 Potassium calibration solution.

AUTHORS' CONTRIBUTION

A. C. P. Osorio, M. D. Almeida, and R.C. Sena contributed to conceptualization and formal data analysis. A. C. P. Osorio contributed to project administration, investigation, methodology development, and original draft writing. M. D. Almeida and R.C. Sena contributed to validation, as well as draft reviewing and editing.

ACKNOWLEDGEMENT

A. C. P. Osorio would like to thank the National Support Program for Metrology, Quality and Technology Development (Pronametro/Inmetro) for financial support.

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