

Analytical Method, 2015

Certification of reference materials of sodium tartrate dihydrate and potassium citric monohydrate for water content

Wei Liu, Haifeng Wang, Xifeng Gu, Can Quan, Xinhua Dai

Uncertainty budget

The uncertainty of water contents of CRMs were evaluated according to ISO Guide 35. The overall uncertainty was combined by the uncertainties of the coulometric method and the volumetric method, the uncertainty from the inhomogeneity and the instability. As seen in Part 1, the uncertainty from the inhomogeneity was evaluated according to ISO Guide 35. The uncertainty from the instability was evaluated according to ISO Guide 35 too (Part 2).

Part 1. Uncertainty from inhomogeneity

The candidate materials were packaged into 400 bottles. 15 bottles of were selected randomly for homogeneity test and each bottle was measured three times. The evaluation of homogeneity of CRM was evaluated by a one-way analysis of variance approach (ANOVA).

Q_1 : sum of square for variation between ampoules

Q_2 : sum of square for variation within ampoules

m : number of ampoules, 15

n : number of measurement for each ampoule, 3

N : sum of measurements, 45

ν_1 : degree of freedom, $m-1=14$

ν_2 : degree of freedom, $N-m=30$

MS_{between} : mean square between ampoules

MS_{within} : mean square within ampoules

s_{bb} : between-ampoule inhomogeneity standard deviation

$u_{\text{H,rel}}$: uncertainty from between-ampoule inhomogeneity

For sodium tartrate dihydrate, related results were listed as below.

$$x = \frac{\sum_{i=1}^m \bar{x}_i}{m} = 153.63 \text{ mg/g}$$

$$N = \sum_{i=1}^m n_i = 45$$

$$Q_1 = \sum_{i=1}^m n_i (\bar{x}_i - \bar{x})^2 = 3.747$$

$$Q_2 = \sum_{i=1}^m \sum_{j=1}^{n_i} (x_{ij} - \bar{x}_i)^2 = 4.086$$

$$F = \frac{Q_1 / \nu_1}{Q_2 / \nu_2} = 1.97$$

Where, the statistic of F (1.97) is less than the critical value $F_{(14, 30)}$ (2.04), which indicated that the CRMs were homogeneous.

Evaluation of uncertainty from homogeneity

According to ISO guide 35, the uncertainty from the homogeneity of CRMs for water content was evaluated.

For sodium tartrate dihydrate, $u_{H, rel}$ was evaluated as below.

$$S^2_{bb} = \frac{MS_{between} - MS_{within}}{n} = \frac{\frac{Q_1}{\nu_1} - \frac{Q_2}{\nu_2}}{n} = \frac{\frac{3.747}{14} - \frac{4.086}{30}}{3} = 0.0438 \text{ (mg/g)}^2$$

$$s_{bb} = \sqrt{0.0438} = 0.209 \text{ mg/g}$$

$$u_{H, rel} = 0.209 / 156.3 = 0.14\%$$

For potassium citrate monohydrate, the result of homogeneity test and its uncertainty contribution is listed below.

$$\bar{x} = \frac{\sum_{i=1}^m \bar{x}_i}{m} = 55.23 \text{ mg/g}$$

$$N = \sum_{i=1}^m n_i = 45$$

$$Q_1 = \sum_{i=1}^m n_i (\bar{x}_i - \bar{x})^2 = 0.1071$$

$$Q_2 = \sum_{i=1}^m \sum_{j=1}^{n_i} (x_{ij} - \bar{x}_i)^2 = 0.4771$$

$$F = \frac{Q_1 / \nu_1}{Q_2 / \nu_2} = 0.48$$

Where, the statistic of F (0.48) is less than the critical value $F_{(14, 30)}$ (2.04), which indicated that the CRMs were homogeneous.

Evaluation of uncertainty from homogeneity

According to ISO guide 35, the uncertainty from the homogeneity of CRMs for water content was evaluated. Since the statistic item F is less than 1, namely the variation between bottles is less than variation within bottle, the equation below was used to calculate u_{bb} as the upper limitation of variation between bottles.

$$u_{bb} = \sqrt{\frac{MS_{\text{within}}}{n}} \cdot \sqrt[4]{\frac{2}{V_{MS_{\text{within}}}}} = \sqrt{\frac{0.4771}{30}} \cdot \sqrt[4]{\frac{2}{30}} = 0.037 \text{ mg/g}$$

$$u_{H, \text{rel}} = 0.037/55.23 = 0.067\%$$

Part 2. Uncertainty from instability

The stability of two CRMs was tested by using the trend analysis according to ISO Guide 35. Table below listed the results in details for reference. CRMs were randomly selected and determined by the Karl Fischer coulometric method. And 1, 3, 6 and 12 months later, CRM were measured. The result for 0, 1, 3, 6 and 12 months was 156.22, 156.32, 156.25, 156.08 and 156.14 mg/g, respectively. The trend analysis was used to test the stability of the water content of CRM according to ISO Guide 35. The results indicated that the CRMs were stable during 12 months.

$$b_1 = \frac{\sum_{i=1}^n (X_i - \bar{X})(Y_i - \bar{Y})}{\sum_{i=1}^n (X_i - \bar{X})^2} = -0.013$$

$$b_0 = 156.26 \text{ mg/g}$$

$$s^2 = \frac{\sum_{i=1}^n (Y_i - b_0 - b_1 X_i)^2}{n - 2} = 0.00649$$

$$s(b_1) = \frac{s}{\sqrt{\sum_{i=1}^n (X_i - \bar{X})^2}} = \frac{0.0806}{9.654} = 0.008349$$

$$t_{0.05}(n-2)s(b_1) = 3.18 \times 0.008349 = 0.0265$$

b_1 : the slope of the fitted line of data (X_i, Y_i)

b_0 : the intercept

s : the standard deviation of the points among the line

$s(b_1)$: the uncertainty associated with the slope

$t_{0.05}(n-2)$: the Student's t -factor for $n-2$ degrees of freedom and $P=95\%$ (95% level of confidence)

$$|b_1| = 0.013 < t_{0.05}(n-2)s(b_1) = 0.0265$$

Results above showed that in 12 months, the slope is insignificant. Therefore, no

instability was observed.

The uncertainty from the long-term stability (u_{its}) was calculated as below.

$$u_{\text{its}} = s_b \cdot t = 0.008349 \text{ mg/g} \times 12 = 0.100 \text{ mg/g}$$

$$u_{\text{its, rel}} = (0.100 \text{ mg/g}) / (156.3 \text{ mg/g}) = 0.064\%$$

Similarly, the stability test of CRM of potassium citrate monohydrate for water content was performed. It showed that the statistic item of $|b_1|$ (0.0033) is less than $t_{0.05}(n-2)s(b_1)$ (0.0095), namely the water content of candidate was stable during the period of stability test. And the uncertainty from long-term stability (u_{its}) was 0.036 mg/g ($u_{\text{its, rel}}$, 0.065%).

Part 3. Uncertainty of the prepared value of the home-made water standard

The home-made water standard was used to calibrate both Karl Fischer coulometry and volumetry. Its uncertainty contributed to the uncertainty of both certification methods. The preparation procedure, determination of prepared value and uncertainty budget of the home-made water standard was listed in below for reference.

Two 20 mL crimp neck headspace vials with rubber septa (CNW Technologies GmbH, Düsseldorf, Germany) were dried at 95 °C for 24 h and then cooled to room temperature. The blank vial was filled with 10 mL of pure 1-octanol and then sealed. The other vial was filled with 10 mL of pure 1-octanol and weighed with a resolution of 0.0001 g. Then, about 0.16 g of deionized water with a resistivity of 18 MΩ·cm was added and the vial was sealed immediately. The mass of the added deionized water was weighed with a resolution of 0.0001 g. The water content of pure 1-octanol in the blank vial was determined using the coulometric method. The water content of this standard was calculated according to the following equation.

$$x_{\text{cert}} = \frac{m_0 \cdot x_0 + \Delta m}{m_0 + \Delta m} = \frac{8.1024 \text{ g} \times 0.1517 \text{ mg/g} + 160.5 \text{ mg}}{8.1024 \text{ g} + 0.1605 \text{ g}} = 19.573 \text{ mg/g}$$

where, x_{cert} is the water content of the in-house water standard, mg/g; m_0 , is the mass of the pure 1-octanol, g; x_0 is the water content of the pure 1-octanol, mg/g; and Δm is the mass of the added water, mg. This water standard was used in calibration of the coulometric and volumetric methods. It was prepared in the morning and used the same day.

The relative humidity in the laboratory during the preparation of water standard is about 10 %rh. Therefore, the relative humidity in the headspace of the sealed vial of the freshly prepared water standard should be close to 10 %rh. The water in the water standard solution volatilized into the headspace of the sealed vial during the storage until the humidity in the upper room reached a saturated value (75 %rh), which resulted the decrease of water content of water standard. m_{vapor} is referred as the mass

of volatilized water. So the evaluation of uncertainty of water content of home-made water standard is performed according to the mathematical formula below:

$$x_{\text{cert}} = \frac{m_0 \cdot x_0 + \Delta m - m_{\text{vapor}}}{m_0 + \Delta m - m_{\text{vapor}}} \approx \frac{m_0 \cdot x_0 + \Delta m - m_{\text{vapor}}}{m_0} = x_0 + \frac{\Delta m}{m_0} - \frac{m_{\text{vapor}}}{m_0}$$

Since there is no correlation among the standard uncertainties in m_0 , x_0 , Δm and m_{vapor} , the combined variance of water content ($u_c^2(x)$) can be derived as:

$$u_c^2(x_{\text{cert}}) = \left(\frac{\partial x_{\text{cert}}}{\partial x_0}\right)^2 \cdot u^2(x_0) + \left(\frac{\partial x_{\text{cert}}}{\partial \Delta m}\right)^2 \cdot u^2(\Delta m) + \left(\frac{\partial x_{\text{cert}}}{\partial m_0}\right)^2 \cdot u^2(m_0) + \left(\frac{\partial x_{\text{cert}}}{\partial m_{\text{vapor}}}\right)^2 \cdot u^2(m_{\text{vapor}})$$

where $u_c(x_{\text{cert}})$, $u(x_0)$, $u(\Delta m)$, $u(m_0)$ and $u(m_{\text{vapor}})$ are the uncertainties of the water content of water standard, water content of pure 1-octanol, mass of added water and mass of volatilized water, respectively. The derivatives are referred to as sensitivity coefficients. Based on the mathematical formula above, these derivatives can be derived as below.

$$\frac{\partial x_{\text{cert}}}{\partial x_0} = 1; \quad \frac{\partial x_{\text{cert}}}{\partial \Delta m} = \frac{1}{m_0}; \quad \frac{\partial x_{\text{cert}}}{\partial m_0} = -\frac{\Delta m}{m_0^2}; \quad \frac{\partial x_{\text{cert}}}{\partial m_{\text{vapor}}} = -\frac{1}{m_0}.$$

$u(x_0)$:

$u(x_0)$ was a combination of A type uncertainty (u_A) and B type uncertainty (u_B). u_A was assessed by standard deviation of results (0.0046 mg/g). u_B was assessed according to the uncertainty of water content by the coulometric method. Since the volume of pure 1-octanol in each injection is 0.2-mL and the mass of water is only 24 μg , the relative uncertainty ($u_{B, \text{rel}}$) of water content by coulometry is estimated less than 5%.

$$u_B = 0.1517 \text{ mg/g} \times 5\% = 0.0076 \text{ mg/g}$$

$$u(x_0) = \sqrt{u_A^2 + u_B^2} = 0.0089 \text{ mg/g}$$

$u(\Delta m)$ and $u(m_0)$:

The expanded uncertainty (U) of the electronic balance given by the verification certificate is 0.0004 g ($k=2$). Therefore, $u(\Delta m)$ and $u(m_0)$ are assessed as 0.0002 g.

$u(m_{\text{vapor}})$:

During the preparation of water standard, the room temperature was 25 °C; the pressure of atmosphere was about 1 atm (101325 Pa); the relative humidity in the laboratory was 10 %rh. The saturated pressure of water at 25 °C is 3169 Pa. The volume of 1 mol water vapor is about 22400 mL. The molecular weight of water is 18 g/mol. The relative humidity in the headspace of sealed vial of water standard in equilibrium is 75 %rh. The volume of the headspace is about 12 mL. So the mass of volatilized water can be calculated as below:

$$\frac{3169 \text{ Pa}}{101325 \text{ Pa}} \times (75\% \text{rh} - 10\% \text{rh}) \times \frac{12 \text{ mL}}{22400 \text{ mL}} \times 18 \text{ g/mol} = 1.96 \times 10^{-4} \text{ g}$$

The actual mass of volatilized water is usually less than that in equilibrium. So

$u(m_{\text{vapor}})$ is estimated as 1.96×10^{-4} g.

Finally, $u_c(x_{\text{cert}})$ was calculated as below.

$$u_c^2(x_{\text{cert}}) = 1^2 \times (0.0089 \text{ mg/g})^2 + \left(\frac{1}{8.1024 \text{ g}}\right)^2 \times (0.0002 \text{ g})^2 + \left(\frac{0.1605 \text{ g}}{(8.1024 \text{ g})^2}\right)^2 \times (0.0002 \text{ g})^2 + \left(\frac{1}{8.1024 \text{ g}}\right)^2 \times (0.000196 \text{ g})^2$$

$$= (7.92 \times 10^{-5} + 6.09 \times 10^{-4} + 2.39 \times 10^{-7} + 5.85 \times 10^{-4} \text{ mg/g})^2$$

$$= 1.273 \times 10^{-3} (\text{mg/g})^2$$

$$u_c(x_{\text{cert}}) = \sqrt{1.273 \times 10^{-3} \text{ mg/g}^2} = 0.036 \text{ mg/g};$$

$$u_{c, \text{rel}}(x_{\text{cert}}) = \frac{0.036 \text{ mg/g}}{19.573 \text{ mg/g}} = 0.18\%$$

The uncertainty contribution from x_0 referred to $\left| \frac{\partial x_{\text{cert}}}{\partial x_0} \cdot u(x_0) \right|$ (0.0089 mg/g). The

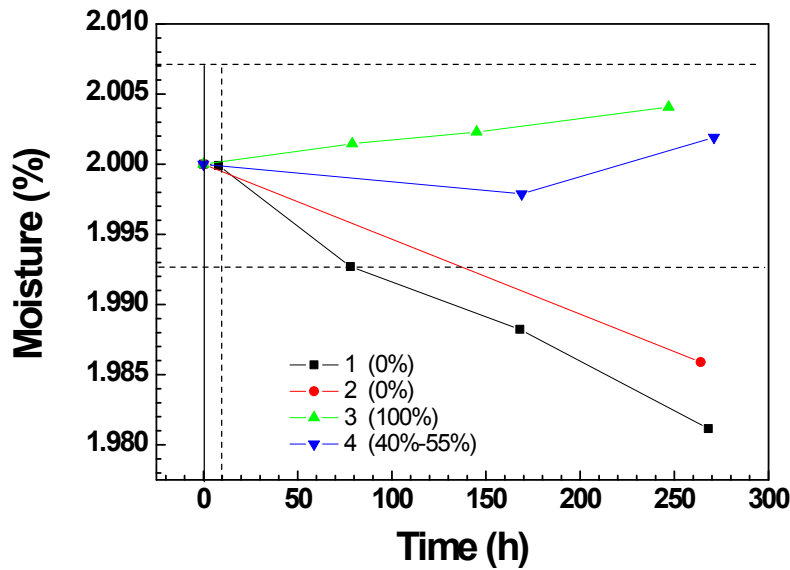
relative uncertainty contribution from Δm was $\frac{\partial x_{\text{cert}}}{\partial \Delta m} \cdot u(\Delta m)$ (0.0247 mg/g). Similarly,

the relative uncertainty contribution from m_0 and m_{vapor} was 0.00049 mg/g and 0.0242 mg/g, respectively. It indicated that the uncertainty contribution from mass of added water (Δm) and mass of water vapor (m_{vapor}) were main sources of uncertainty of the prepared value of this home-made water standard. Therefore, the water content of this standard prepared by the gravimetry was traceable to SI unit of the mass.

The home-made water standards stored in varied humidity had been measured during a period of 11 days. The result is shown in figure below. Sample 1, 2, 3 and 4 with a water content of about 2.0% were prepared at August 26th. Sample 1 and 2 were stored in a desiccator containing phosphorus pentoxide where the humidity is close to 0%. Sample 3 was stored in a desiccator containing the deionized water of a quarter of the total volume where the humidity is close to 100%. Sample 4 was stored in the air where the humidity ranged from 40% to 55%. The room temperature ranged from 26 °C to 31 °C. The prepared value of water content was not equal to 2% exactly, but for the convenience of comparison all the water content results (prepared and measured) was modified proportionally to make the initial value of water content equal to 2%. For example, the water content of sample 1 was modified as below.

$$\text{Modified water content} = \frac{\text{Measured water content}}{\text{Prepared water content}} \times 2\% = \frac{\text{Measured water content}}{2.0574} \times 2\%$$

Time (h)	Water content (%)	RSD (%)	Modified water content (%)
0	2.0574	/(Prepared value)	2.000
8	2.0573	0.47	1.9999
78	2.0499	0.42	1.9927
168	2.0453	0.13	1.9882
268	2.0380	0.19	1.9811



(The measured water content is the mean of three replicates. Coulometric method: DL 39 Karl Fischer titrator with a electrode without diaphragm; Coulomat AG anolyte; water mass of each injection is about 0.3 mg)

It was shown that the water content of water standard stored in a humidity of 0% decreased slowly and 70 h later the decrease of the water content was more than its expanded uncertainty ($U=0.072$ mg/g). Sample 2 was measured for the first time 11 days after the preparation. The results showed that even if the septum was not pierced, the water also volatilized from the crimp neck vial through the septum. The water content of water standard stored in a humidity of 100% and (40%-55%) varied slightly.

These results showed that the variation of the water content of water standards stored in varied humidity is less than their uncertainty within 10 h. In fact, in this study the water standard was prepared in the morning and used during the after working day. The interval of time is about 10 h.

Part 4. Uncertainty of water content by the coulometric method

The evaluation of the uncertainty of the coulometric method was introduced by the example of sodium tartrate dihydrate. The uncertainty was combined by Type A and Type B uncertainty. Type A uncertainty arose from the statistic and was equal to the RSD. For sodium tartrate dihydrate with a water content of 156.22 mg/g, RSD is 0.141%. Type B uncertainty is the non-statistical uncertainty and deduced according to its mathematical formula.

$$x' = \frac{Q \times 18.015}{96485 \times 2m} \cdot f_1 \cdot f_2$$

Since there is no correlation among the standard uncertainties in Q , m and $f_1 \cdot f_2$, the combined variance of water content ($u_{B,rel}^2(x')$) can be derived as:

$$u_{B,rel}^2(x')^2 = u_r(Q)^2 + u_r(M)^2 + u_r(F)^2 + u_r(m)^2 + u_r(f_1 f_2)^2$$

Where $u_r(Q)$, $u_r(M)$, $u_r(F)$, $u_r(m)$ and $u_r(f_1:f_2)$ was the relative uncertainty of amount of charge, molecular weight of water, *Faraday's* constant, mass of samples and correction coefficient ($f_1:f_2$), respectively. The amount of charge was the integration of the current with respect to time. Since the current and time were measured with high accuracy, $u_r(Q)$ is low and it can be omitted. Similarly, $u_r(M)$ and $u_r(F)$ could be omitted. The maximum permitted error (MPE) of the electronic balance given by the verification certificate is 0.005 mg ($k=2$). Therefore, the standard uncertainty of mass was assessed as 0.0029 mg. Considering the mass was determined by the subtraction, the $u(m)$ is evaluated as 0.0041 mg, namely $u_r(m)$ was 0.021%, considering the mass per injection of 20 mg. For $u_r(f_1:f_2)$, it is difficult to quantify the value of f_1 and f_2 directly, so the home-made water standard was applied to assess the systematic error. The $f_1:f_2$ (1002.4 mg/g) was equal to the reciprocal of recovery (997.6 mg/g). $u_r(f_1:f_2)$ equal to the relative uncertainty of recovery was combined by the Type A uncertainty (RSD of recovery) and Type B uncertainty which was mainly come from the uncertainty of water content of home-made water standard (u_r 0.018%). Since the contribution of statistic of data has been evaluated in Type A uncertainty of the coulometric method, $u_r(f_1:f_2)$ is approximately equal to the uncertainty of water content of home-made water standard ($u_r=0.18\%$).

$$u_{r,B} = \sqrt{0^2 + 0^2 + 0^2 + u_r(m)^2 + u_r(f_1:f_2)^2} = \sqrt{0.021^2 + 0.18^2} = 0.181\%$$

$$u_{r,coulomat} = \sqrt{u_A^2 + u_B^2} = \sqrt{0.141^2 + 0.181^2} = 0.23\%$$

Finally, the relative standard uncertainty of coulometry for sodium tartrate dihydrate ($u_r(x')$) is 0.23% ($k=2$).

Similarly, the relative standard uncertainty of coulometry for potassium citrate monohydrate ($u_r(x')$) is 0.20% ($k=2$).

Part 5. Uncertainty of water content by the volumetric method

The evaluation of the uncertainty of the volumetric method was introduced by the example of sodium tartrate dihydrate. The uncertainty was combined by Type A and Type B uncertainty. Type A uncertainty arose from the statistic and was equal to the RSD. For sodium tartrate dihydrate with a water content of 156.34 mg/g, RSD is 0.205%. Type B uncertainty is the non-statistical uncertainty and deduced according to its mathematical formula.

$$x = \frac{V_{TS} \cdot m_R \cdot x_{cert}}{m_S \cdot V_{TR}}$$

where V_{TS} is the volume of the reagent required to titrate the added water in the sample, mL; m_R is the mass of home-made water standard, g; x_{cert} is the water content of home-made water standard; m_S is the mass of the sample, g; V_{TR} is the volume of the reagent required to titrate the added water in home-made water standard, mL.

The combined variance of water content ($u_{B,rel}^2(x)$) can be derived as:

$$u_{B,rel}^2(x) = u_r(m_S)^2 + u_r(m_R)^2 + u_r(V_{TS})^2 + u_r(V_{TR})^2 + u_r(x_{cert})^2$$

Where $u_r(m_S)$, $u_r(m_R)$, $u_r(V_{TS})$, $u_r(V_{TR})$, and $u_r(x_{cert})$ were the relative uncertainty of mass of the sample, mass of home-made water standard, volume of the reagent in sample, volume of the reagent in home-made water standard, and water content of home-made water standard, and, respectively.

The uncertainty of sample mass determined by the balance with a readability of 0.001 mg was assessed as 0.0041 mg, and thus $u_r(m_S)$ was 0.021%. The uncertainty of home-made water standard mass determined by the balance with a readability of 0.01 mg was assessed as 0.122 mg, and thus $u_r(m_R)$ was 0.079%. For $u_r(V_{TS})$ and $u_r(V_{TR})$, the readability of volume is 0.0005mL, and thus the uncertainty of volume is evaluated as 0.001 mL. The volume of reagent was about 1.5 mL and the relative uncertainty ($u_r(V_{TS})$ or $u_r(V_{TR})$) is 0.064%. As shown above, the relative uncertainty of water content of home-made water standard ($u_r(x_0)$) was 0.18%.

$$u_{B,rel}(x) = \sqrt{0.079^2 + 0.021^2 + 0.064^2 + 0.064^2 + 0.18^2} = 0.218\%$$

$$u_{c,rel}(x) = \sqrt{u_{A,rel}^2 + u_{B,rel}^2} = \sqrt{0.205\%^2 + 0.218\%^2} = 0.30\%$$

Finally, the relative standard uncertainty of volumetry ($U_r(x)$) is 0.30% ($k=2$).

Similarly, the relative standard uncertainty of volumetry for potassium citrate monohydrate ($U_r(x)$) is 0.44% ($k=2$).

Part 6. Uncertainty combination

The evaluation of the expanded uncertainty of CRM was introduced by the example of sodium tartrate dihydrate.

$$u_{rel}^2 = u_{H,rel}^2 + u_{I,ts,rel}^2 + u_{c,rel(coulometric)}^2 + u_{c,rel(volumetric)}^2 +$$

$$u_{rel}^2 = 0.14\%^2 + 0.064\%^2 + 0.23\%^2 + 0.30\%^2$$

$$u_{rel} = 0.41\%$$

$$U_r = 0.82\% (k=2)$$

$$U = 1.3 \text{ mg/g } (k=2)$$

where, u_{rel} , $u_{H,rel}$, $u_{I,ts,rel}$, $u_{c,rel(coulometric)}$ and $u_{c,rel(volumetric)}$ was the combined relative uncertainty of certified value, that from homogeneity and stability, the coulometric method and the volumetric method, respectively. U_r and U was the expanded relative uncertainty and expanded uncertainty of certified value, respectively.

Similarly, the uncertainty budget of potassium citrate monohydrate was listed below.

$$u_{rel}^2 = u_{H,rel}^2 + u_{I,ts,rel}^2 + u_{c,rel(coulometric)}^2 + u_{c,rel(volumetric)}^2$$

$$u_{rel}^2 = 0.067\%^2 + 0.065\%^2 + 0.20\%^2 + 0.44\%^2$$

$$u_{rel} = 0.50\%$$

$$U_r = 1.00\% (k=2)$$

$$U = 0.6 \text{ mg/g } (k=2)$$

Uncertainty budget of three CRMs of water content

CRM No.	Item	Coulometric		Volumetric		
Sodium tartrate dihydrate	$u_{A,rel}$	<i>RSD</i>	0.141%	<i>RSD</i>	0.205%	
	$u_{c,rel}$	$u_{B,rel}$	$u_{m,rel}$	0.021%	$u_{mS,rel}$	0.079%
			$u_{flf2,rel}$	0.18%	$u_{mR,rel}$	0.021%
					$u_{VTS,rel}$	0.064%
					$u_{VTR,rel}$	0.064%
					$u_{cert,rel}$	0.18%
				0.181%	0.218%	
	$u_{c,rel}$	0.23%		0.30%		
	$u_{H,rel}$	0.14%				
	$u_{Its,rel}$	0.064%				
	$u_{c,rel}$	0.41%				
	$U_r(k=2)$	0.82%				
	$U(k=2)$	1.3 mg/g				
	Potassium citrate monohydrate	$u_{A,rel}$	<i>RSD</i>	0.072%	<i>RSD</i>	0.377%
$u_{c,rel}$		$u_{B,rel}$	$u_{m,rel}$	0.009%	$u_{mS,rel}$	0.009%
			$u_{flf2,rel}$	0.18%	$u_{mR,rel}$	0.10%
					$u_{VTS,rel}$	0.072%
					$u_{VTR,rel}$	0.072%
					$u_{cert,rel}$	0.18%
				0.181%	0.23%	
$u_{c,rel}$		0.20%		0.44%		
$u_{H,rel}$		0.067%				
$u_{Its,rel}$		0.065%				
$u_{c,rel}$		0.50%				
$U_r(k=2)$		1.00%				
$U(k=2)$		0.6 mg/g				