Report of the key CCQM-K48
Assay of potassium chloride

Draft B

(Final)

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1 ABSTRACT

The CCQM-K48 key comparison for determination of the purity of potassium chloride was organized jointly by the inorganic analysis and electrochemical analysis working groups of CCQM to test the abilities of the metrology institutes to measure the amount content of chloride in KCl. National Institute of Metrology P.R.China (NIM) acted as the coordinating laboratory of this comparison with SMU and NIST as co-coordinating laboratories. Seven NMIs participated in this key comparison. All participants used coulometry (except NMIJ which used gravimetry) and all of them analyzed the impurity bromine in the sample and made corrections. In general very good agreement of the results was observed, some possible problems were highlighted.

2   INTRODUTION

Pure materials are very important for traceability in chemical metrology, and pure material studies and comparisons are important activities in Electrochemical Analysis Working Group and Inorganic Analysis Working Group at all times. The EAWG with IAWG jointly organized a pilot study CCQM-P7 (CCQM 8) for determining the purity of KCl, NaCl and $K_2Cr_2O_7$ in 1998, NIST acted as the coordinating laboratory. In that pilot study, coulometry, 100%-sum impurity (instrumental techniques), gravimetry and titrimetry were used. The results showed that coulometry is the best method. As a primary method, coulometry is most suitably applied in determining the purity of pure material. Many NMIs increasingly use it for assay of primary reference material at the top of the national traceability chains, and it is also one of the main research topics in the EAWG.

Potassium chloride is an important chemical reagent, it is research foundation for pH, conductivity and mono-element standard solution, so a key comparison for determination the purity of KCl is necessary and CCQM has agreed to start a key comparison (CCQM-K48) for determination of the purity of potassium chloride.

The objective of this key comparison "Assay of Potassium Chloride" was to determine the amount content of Cl in potassium chloride. The participants were free to choose the analytical procedure, but the coulometric method was recommended for this comparison. The comparison tested the capabilities for assay of high purity materials and underpinned the claimed calibration and measurement capabilities of the participated national metrology institutes.

3   LIST OF PARTICIPANTS

Six institutes originally indicated interest in participating in the comparison. At last, seven institutes took part. Table 1 contains the full names of all participating NMIs and contact persons.

Table 1   List of participants

Institution	Country	Contact person
BAM Federal Institute for Materials Research and Testing	Germany	Martin Breitenbach
INMETRO Instituto Nacional de Metrologia, Normalização e Qualidade Industrial	Brazil	Paulo Paschoal Borges
KRISS Korean Research Institute of Standards and Science	Korea	Euijin Hwang
NIM National Institute of Metrology	China	Ma Liandi
NIST National Institute of Standards and Technology	USA	Kenneth W. Pratt
NMIJ National Metrology Institute of Japan	Japan	Dr. Akiharu HIOKI
SMU Slovak Institute of Metrology	Slovakia	Michal Mariassy

4   SAMPLES

The source of the sample is a 10 kg batch of commercial pure potassium chloride material. After being homogenised, a 500 g portion was selected from the middle fraction of the batch, and was homogenised again in a large bottle. This homogenised portion was then transferred to 15 glass bottles closed with silicone lined plastic caps for the comparison, each containing about 15 g of the material. The impurities were determined by ICP-MS.

Seven bottles were randomly selected from the set of 15 bottles. Two samples were sampled independently from these seven bottles. The content of chloride was determined by coulometry to check the homogeneity of the samples. No statistically significant heterogeneity was found based on F test, and the RSD was less than 0.0055%, no statistically significant difference was found.. Impurities were determined by ICP-MS, also no statistically significant difference was found from impurities in different samples.

The samples were sent to the participants by EMS on October 19, 2007 (except SMU, where the sample was sent on October 22 by DHL, and INMETRO, where the sample was sent on October 22 by TNT). All samples arrived to their destination without damage within two weeks. The dispatch dates and receipt dates are given in Table 2:

The deadline for reporting results was set to 15 March 2008 in order to prepare a presentation for discussion at the CCQM WG meeting in April 2008. All participants reported their results in time.

After results discussion on the joined meeting of IAWG and EAWG in April 2008 and draft A report discussion on the same meeting in October 2008, the draft B report was prepared and it was circulated to each participant before the CCQM WG meeting in April 2009, and after small revision the draft B final report is completed.

Table 2   Sample sent dates, receipt dates and report dates

Institute	Sample No.	Sample dispatch date	Sample receipt date	Date report sent
BAM	01	19 October 2007	23 October 2007	12 March 2008
INMETRO	03	22 October 2007	30 October 2007	15 March 2008
KRISS	02	19 October 2007	22 October 2007	15 March 2008
NIM	04			
NIST	05	19 October 2007	23 October 2007	14 March 2008
NMIJ	06	19 October 2007	22 October 2007	14 March 2008
SMU	07	22 October 2007	24 October 2007	13 March 2008

5   INSTRUCTIONS TO PARTICIPANTS

The instructions sent to the participants by e-mail consisted of technical protocol and results report form.

The technical protocol (appendix A) contained background information, timing of the comparison, and information on the participating institutes. Information on sample homogeneity and sample preparation for measurements was given. Any method or combination of methods can be used, but the coulometric method is recommended for this comparison. Some possible problems with measurement were highlighted. Participants were requested the results as amount content of chloride in KCl [mol/kg] and/or mass fraction of chloride expressed as KCl [kg/kg] and to provide uncertainty evaluation according to ISO Guide [1].

The results report form contained entries relating to the measurement results, detailed uncertainty evaluation and description of the measurement procedures.

6   METHODS OF MEASUREMENT

Six participants used coulometric titration for assay determination and reported more or less details on their procedure in their reports or additional information. Some details on measurements as derived from the reports are given in Table 3 and Table 4. Six participants introduced the solid samples directly into the coulometric cell; NMIJ used a special way to get the result for solid sample by gravimetry.

Table 3 Details on measurement methods used

Institute	Approx. sample mass/g	Sample dried	Corrected for buoyancy	Measurement method
BAM	0.15~0.27	yes	yes	Coulometry
INMETRO	0.3	yes	yes	Coulometry
KRISS	0.3	yes	yes	Coulometry
NIM	0.15	yes	yes	Coulometry
NIST	0.25	yes	yes	Coulometry
NMIJ	0.5	yes	yes	Gravimetry
SMU	0.5	yes	yes	Coulometry

Table 4 Details of Coulometric procedures used

Institute	Details of coulometric procedures				EP estimation	Initial titration
	Cell type	IC rinse	Cell volume /mL	Main current /mA		
BAM	vertical, 1 intermediate chamber (IC)	Yes	180	200	nonlinear regression	Yes
INMETRO	vertical, 1 IC	Yes	250	200	3rd order polynomial regr.	Yes
KRISS	horizontal, 2 IC	Yes	?	100	3rd order polynomial regr.	Yes
NIM	horizontal, 2 IC	Yes	180	100	3rd order polynomial regr.	Yes
NIST	horizontal, 2 IC	Yes	180	100	3rd order polynomial regr.	Yes
SMU	vertical, 1 IC	Yes	290	200	nonlinear regression	Yes

7   RESULTS AND DISCUSSION

7.1 Sum of halides (Cl+Br)

The reported values and uncertainties of all results before correction are summarized in Table 5, and arithmetic mean and median of all results before Br correction in Table 6. The same results are displayed graphically in Figure 1; coulometric results before correction in Figure 2.

Table 5 Sum of halides (Cl+Br) expressed as KCl result (KCl result before Br correction)

Institute	*Measurement date*	*Result /mol.kg^{-1}*	*n*	*SD /mol.kg^{-1}*	*u_c /mol.kg^{-1}*	*U /mol.kg^{-1}*	*k*
INMETRO	Mar 5~Mar 14, 08	13.41116	6	0.00221	0.00146	0.00292	2
KRISS	Mar 7~Mar 14, 08	13.41255	7	0.00080	0.00076	0.00152	2
BAM	Jen 23~Feb 12, 08	13.41277	7	0.00176	0.00098	0.00196	2
SMU	Mar 6~Mar 10, 08	13.41281	6	0.00082	0.00088	0.00176	2
NIM	Nov 7~Nov 13, 07	13.41289	6	0.00055	0.00062	0.00124	2
NIST	Feb 19~ Feb 27, 08	13.4149	10	0.00054	0.00083	0.0017	2
NMIJ	Feb 22, 08	13.4156	6	0.0022	0.0009	0.0019	2

Table 6 Arithmetic mean and Median of All Results before Br correction

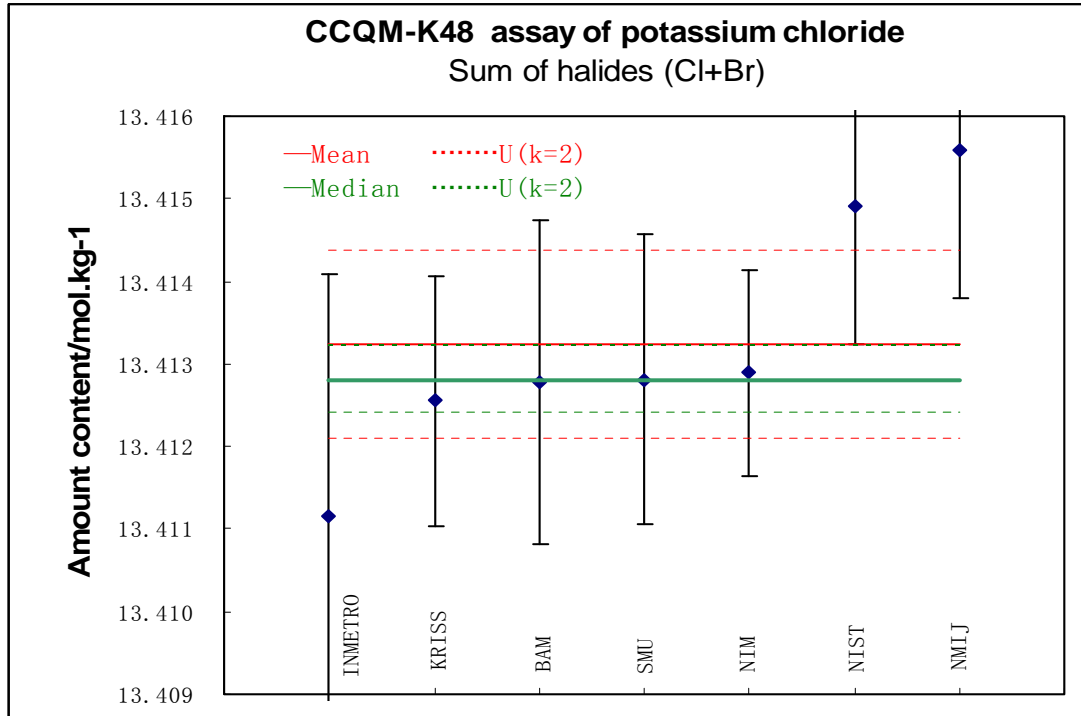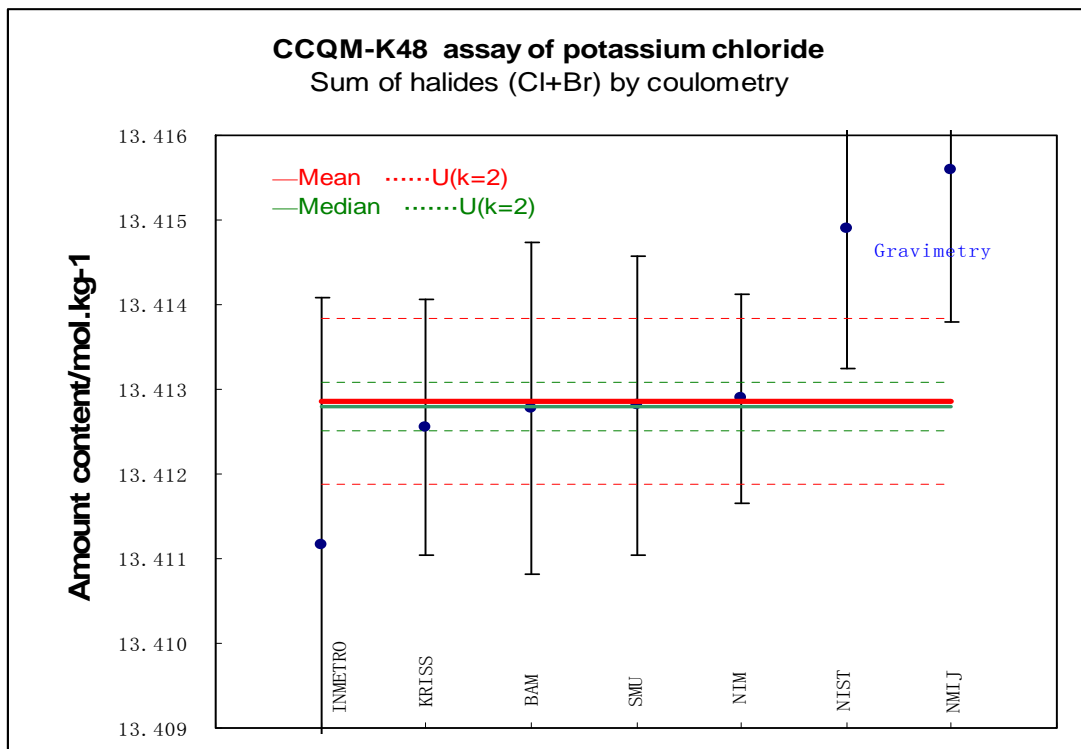|	Value (mol.kg^{-1})	standard uncertainty (mol.kg^{-1})	expanded uncertainty k=2 (mol.kg^{-1})
All results mean ($\bar{x}$)	13.41324	0.00057	0.00114
All results median	13.41281	0.00020	0.00040
Coulometric results mean ($\bar{x}$)	13.41285	0.00049	0.00098
Coulometric results median	13.41279	0.00014	0.00028

Figure 1

Figure 2

Note: NMIJ result by gravimetry is not included in calculation.

7.2 Chloride amount content

The reported values and uncertainties of all final results with Br correction are summarized in Table 7 and arithmetic mean and median of all results with Br correction for possible KCRV in Table 8. The same results are displayed graphically in Figure 3, coulometric results only in Figure 4.

Table 7 Result (amount content of chloride)

Institute	*Result* /mol.kg⁻¹	*n*	*SD* /mol.kg-1	u_c /mol.kg⁻¹	*U* /mol.kg⁻¹	*k*
INMETRO	13.41077	6	0.00221	0.00146	0.00292	2
KRISS	13.41214	7	0.00080	0.00076	0.00152	2
BAM	13.41226	7	0.00176	0.00098	0.00196	2
NIM	13.41229	6	0.00055	0.00062	0.00124	2
SMU	13.41230	6	0.00082	0.00088	0.00176	2
NIST	13.4145	10	0.00054	0.00083	0.0017	2
NMIJ	13.4151	6	0.0022	0.0009	0.0019	2

Table 8 Possible estimators of KCRV

Potential KCRV	Value (mol.kg⁻¹)	standard uncertainty (mol.kg⁻¹)	expanded uncertainty k=2 (mol.kg⁻¹)
All results, mean（$\bar{x}$）	13.41277	0.00057	0.00114
All results, median （*KCRV*）	**13.41229**	**0.00012**	**0.00024**
Coulometric results mean（$\bar{x}$）	13.41238	0.00049	0.00098
Coulometric results median	13.41228	0.00007	0.00014

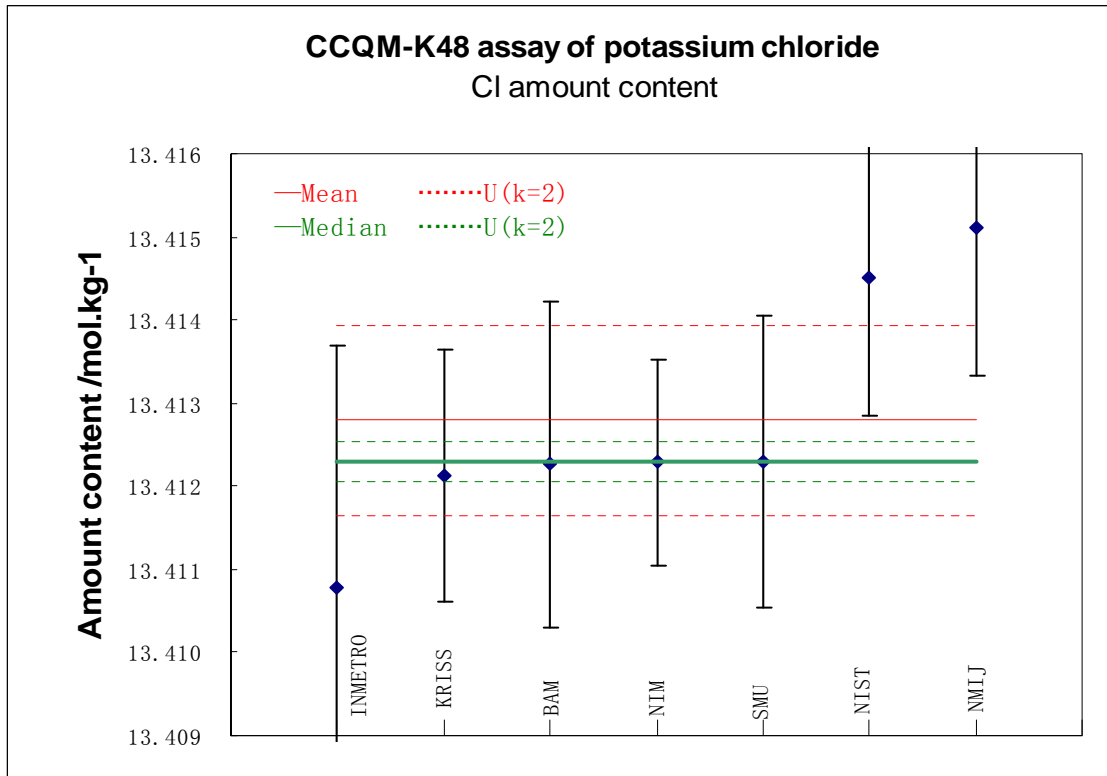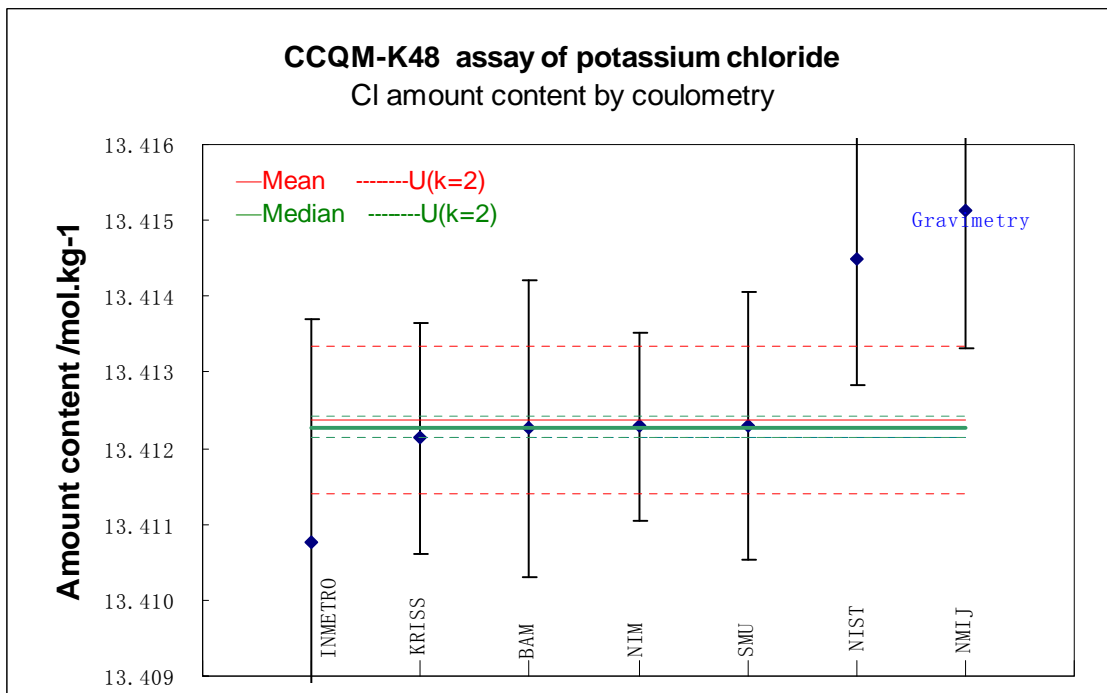Note：*It was proposed to use the median as the KCRV.

Figure 3

Figure 4

Note: NMIJ result by gravimetry is not included in calculation.

7.3 Formulas

Arithmetic mean

$$\overline{x} = \frac{\sum_{i=1}^{n} x_i}{n} \ ;$$

Expanded uncertainty of mean

$$U_{KCRV(mean)} = 2 \times \sqrt{\frac{\sum_{i=1}^{n} (x_i - \overline{x})^2}{n \times (n-1)}}$$

Median

$$median = average\left(x_{\frac{n}{2}} + x_{\frac{n}{2}+1}\right), \ \text{n= even number;} \ median = x_{\frac{n+1}{2}}, \ \text{n=odd number}$$

Expanded uncertainty of median [2]

$$U_{KCRV(median)} = 2 \times \frac{1.858}{\sqrt{n-1}} \cdot median\{|x_i - KCRV|\}$$

7.4 Impurity analysis

The impurity Br detected by all participants are summarized in Table 9, and other impurities detected by BAM, NIM and NIST are summarized in Table 10 and Table 11.

Table 9 Impurity Br detected

Institute	Amount content / mol.kg^{-1}	Mass fraction /mg.kg^{-1}	Expanded uncertainty	Measurement method
BAM	0.00051	41	10 % ?	ICP-MS
INMETRO	0.00039	31.32	1.3%	Ion Chromatography
KRISS	0.000417	33.32	8% ?	Ion Chromatography
NIM	0.00060	48	11%	ICP-MS
NIST	0.00044	35	11%	XRF
NMIJ	0.00048	38.6	2.6%	Ion Chromatography
SMU	0.00051	41	18%	Ion Chromatography
Average	0.00048	38. 3		
RSD	15%	15%		

Table 10 Mass fraction of impurities detected

Institute	NIM		BAM		NIST		NMIJ	
	value / mg.kg⁻¹	Expanded uncertainty	value /mg.kg⁻¹	Expanded uncertainty	value / mg.kg⁻¹	Expanded uncertainty	value / mg.kg⁻¹	Expanded uncertainty
method	ICP-MS		ICP-MS		XRF		Ion Chromatography	
Na	93	5.4%	116	30 %				
Rb	27	9.3 %	32	10 %	120	36%		
Pb	0.93		1	30 %				
I			<1				undetected	

Note: Little iodine was found in the sample, so we considered the iodine had no effect on the results, that means the sum of halides included Cl and Br only.

Table 11 Mass fraction of other impurities detected with ICP-MS by NIM

elements	value / mg.kg⁻¹	elements	value / mg.kg⁻¹	elements	value / mg.kg⁻¹
Li	0.013	Cu	1.0	Sb	0.003
Be	$<6.7e^{-3}$	Zn	3.4	Cs	0.001
B	3.1	Ga	0.006	Ba	0.24
Mg	15	Ge	$<3.8e^{-3}$	La	0.0009
Al	1.2	As	0.53	Ce	0.008
Ca	5.1	Se	0.10	W	0.004
Sc	$<8e^{-4}$	Sr	0.40	Au	0.59
Ti	0.67	Y	0.002	Hg	0.12
V	0.66	Zr	0.009	Tl	0.010
Cr	2.2	Nb	0.0008	Bi	$<1.4e^{-3}$
Mn	0.44	Mo	0.030	Th	0.0006
Fe	1.2	Cd	$<6.1e^{-3}$	U	0.002
Co	0.010	In	0.001		
Ni	1.4	Sn	0.007		

7.5 Discussion

Based on the final results from all of the participants, BAM, NIM, KRISS and SMU are very good agreement, and results from NIST and NMIJ are somewhat higher than the reference value.

NIST has lot of experience in this field (coulometry research and pure material determination), and Dr. Kenneth W. Pratt [3] discussed the relationship between the sample mass and the result, and also gave potential possibilities for a positive bias in this key comparison.

For more discussion, the coordinating laboratory NIM have showed the potential possibilities for the bias in this key comparison as follows, which had been discussed at

the IAWG+EAWG meeting in October 2008.

1. Metallic impurities in Ag anode and AgCl clinging on Ag anode may effect the efficiency of electric current to be less than 100%, causing a positive bias.

2. The coulometric instruments must be calibrated before this determination to avoid the system errors. And it is encouraged to calibrate the coulometric system every year, althouh it is very stable.

3. The occluded Ag^+ (as $AgNO_3$ solution) in the precipitated AgCl may be the reason for high results, because the loss of Ag^+ will prolong the time for electrolysis.

4. On the other way Cl^- co-precipitation/adsorption on AgCl may cause low results .

5. If there is a large excess of electrolytic Ag^+ from Ag anode when AgCl forms, the occlusion/adsorption of Ag^+ (as $AgNO_3$) in the precipitated AgCl may easily happen. And the Cl co-precipitation on AgCl can easily happen if a large amount of sample is added, as the concentration of Cl^- will be high.

6. NIM has noticed the above problems for many years. In order to avoid occluded Ag^+ in AgCl or Cl^- co-preciption on AgCl, we dissolve the solid KCl sample by pure water and then add the solution into the anode compartment by a pipe step by step, instead of adding the solid KCl sample in to the anode cell at one time.

7. If the above discussions are potential reasons for bias of coulometric result, it can happen easier in gravimetry, because locally high concentration of Ag^+ (as $AgNO_3$ solution) cannot be avoided. So the result of NMIJ (by gravimetry) may be higher due to this reason, but this assumption has to be tested.

8. This reaction must be performed in the dark, because the light can cause the loss of Ag^+ by reduction to Ag and leads to a positive bias.

9. Loss of Ag^+ or Cl^- into the SiO_2 gel plug would also cause biased results.

8　EQUIVALENCE STATEMENTS

The equivalence statements have been calculated according to the BIPM guidelines. The degree of equivalence (and its uncertainty) between a NMI result and the KCRV is calculated according to the following equations:

$$d_i = \left(x_i - x_{ref}\right) \qquad\qquad u^2(d_i) = u^2\left(x_i\right) + u^2\left(x_{ref}\right)$$

Where d_i is the degree of equivalence between the NMI result x_i and the KCRV x_{ref}, and $U(d_i)$ is the expanded uncertainty $(k = 2)$ of the d_i calculated by combining the standard uncertainty $u(d_i)$ of the NMI result x_i and the standard uncertainty $u\left(x_{ref}\right)$ of the KCRV x_{ref}. The equivalence statements for CCQM-K48 are given in Table 12.

Table 12 Equivalence Statements of Cl amount content for CCQM-K48

| Participant	Reported Value （x_i） /mol/kg	Standard uncertainty （$u(x_i)$）/mol/kg	d_i /(mol/kg)	$U(d_i)$ /mol/kg
INMETRO	13.41077	0.00146	-0.00152	0.00293
KRISS	13.41214	0.00076	-0.00015	0.00154
BAM	13.41226	0.00098	-0.00003	0.00197
NIM	13.41229	0.00062	0.00000	0.00126
SMU	13.41230	0.00088	0.00001	0.00178
NIST	13.4145	0.00083	0.0022	0.0017
NMIJ	13.4151	0.0009	0.0028	0.0019

9　CONCLUSIONS

Good agreement between the participating laboratories for measurement of potassium chloride was observed. The median of all results is proposed for the KCRV (amount content of chloride in KCl 13.41229 mol/kg, associated expanded uncertainty 0.00024 mol/kg). The use of median and its uncertainty based on median of the absolute deviations (MAD) was agreed as the KCRV at the IAWG+EAWG meetings in April and October 2008. The consistent results in CCQM-K48 are better than those in CCQM-P7. The suitability of coulometry for assay of high purity materials was again demonstrated.

Figure 5 Degrees of equivalence d_i and expanded uncertainty U (d_i) (k=2)
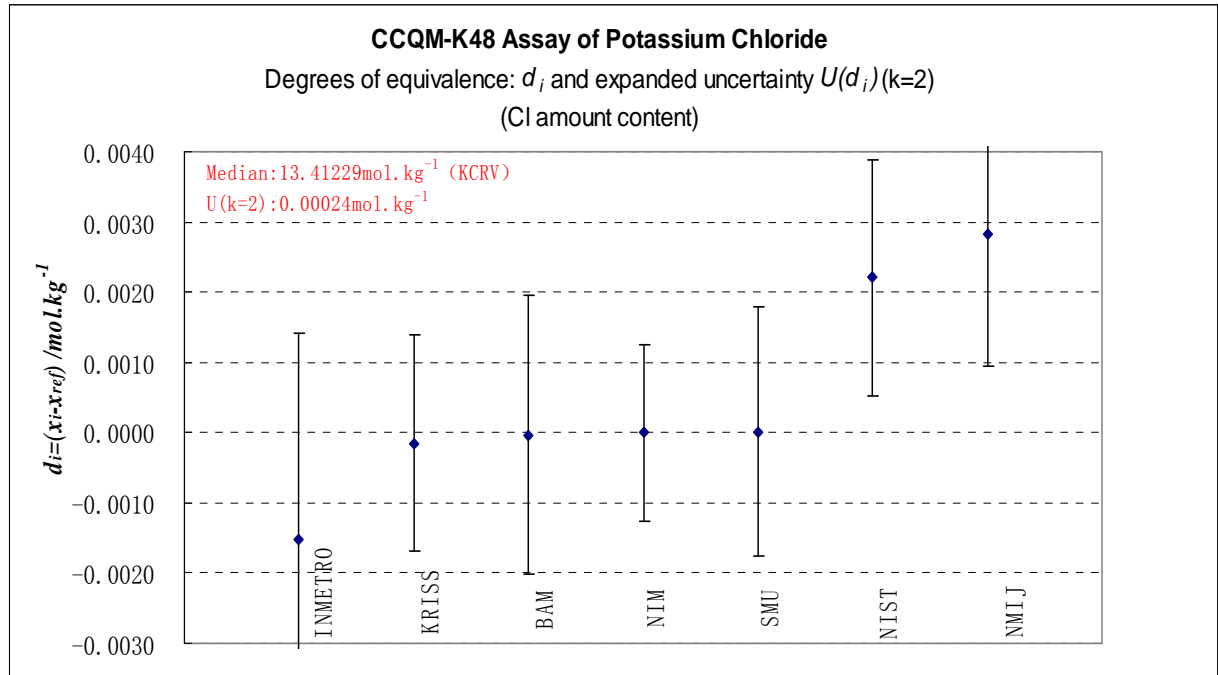
10 ACKNOWLEDGEMENTS

11 REFERENCES

[1] BIPM, ISO, IEC, OIML, Guide to the expression of Uncertainty in Measurement (1995) 1st ed., ISO, Geneva.

[2] Müller J.W., Possible Advantages of a Robust Evaluation of Comparisons. J. Res. Natl. Inst. Stand. Technol. 105, 551-555 (2000)

[3] K.W.Pratt: "Coulometry at NIST:$K_2Cr_2O_7$ and KCl", EAWG workshop on coulometry, April 2008,

http://www.bipm.org/wg/CCQM/EAWG/Restricted/April_2008/Pratt_EAWG_Apr08_coulometry.ppt

Appendix A – Technical Protocol

CCQM-K48 Assay of potassium chloride
Technical Protocol

INTRODUCTION

Pure materials are very important for traceability in chemical metrology, and pure material studies and comparisons are important activities in Electrochemical Analysis Working Group and Inorganic Analysis Working Group at all times. The EAWG with IAWG jointly organized a pilot study CCQM-P7(CCQM 8) for determining the purity of KCl, NaCl and $K_2Cr_2O_7$ in 1998, NIST acted as the coordinating laboratory. In this pilot study, coulometry, 100%-sum impurity (instrumental techniques), gravimetry and titrimetry were used. The results showed that coulometry is the best method. As a primary method, coulometry is most suitably applied in determining the purity of pure material. Many NMIs increasingly use it for assay of primary reference material at the top of the national traceability chains, and it is also one of the main research topics in the EAWG.

Potassium chloride is an important chemical reagent, it is research foundation for pH, conductivity and mono-element standard solution, so a key comparison for determination the purity of KCl is necessary. CCQM had agreed that this should proceed to a following key comparison (CCQM-K48) for determination of the purity of potassium chloride, NIM (National Institute of Metrology P.R.China, former NRCCRM) is acting as the coordinating laboratory of this comparison with SMU and NIST as co-coordinating laboratories.

Scope:

The comparison will test the capabilities and methods used for assay of high purity materials and underpin the claimed calibration and measurement capabilities of the institutes.

Time schedule

Dispatch of the samples: 20, October 2007
Deadline for receipt of the report: 15, March 2008
Results discussion: IAWG-EAWG/CCQM meeting in April 2008
Draft A report discussion: IAWG-EAWG/CCQM meeting in October 2008

Samples

Sample preparation:
The source of the sample is from a 10 kg batch of commercial pure potassium chloride material. After being homogenised, a 500 g portion was selected from the middle fraction of the batch, and was homogenised again in a large bottle. This homogenised portion was then transferred to 15 glass bottles closed with silicone lined plastic caps for the comparison. The impurities were determined by ICP-MS. The assay is in the range of 99.9 % to 100 % of the theoretical value.

Homogeneity test:
Seven bottles were randomly selected from the set of 15 bottles. Two samples were sampled independently from these seven bottles. The content of chloride was determined by coulometry to check the homogeneity of the samples. No statistically significant heterogeneity was found based on F test, and the RSD was less than 0.004 %, no statistically significant difference was found.. Impurities were determined by ICP-MS, also no statistically significant was found from difference impurities in different samples.
The sample mass used for homogeneity tests at NIM was 150 mg. Please pay attention that you do not use less than 150 mg in your analyses for the key comparison.

Distribution:
Each participant will receive one numbered bottle containing about 15 g of material. The sample number will be the same as the laboratory number. Shipment to all participants will be performed at the same time. The bottles are shipped in a cardboard box by courier. The contents will be marked "**potassium chloride**" for research purposes; please be attentive of possible customs delays, etc. The measurement protocol is sent by e-mail.

The participants will be informed of the date of dispatching of the samples. Participants must confirm the receipt of the sealed samples, by filling in the return receipt table and sending it to the NIM contact person by e-mail, fax or mail. If there is any damage, please contact us immediately, and NIM will mail out another bottle.

Handling and storing instructions:

The sample should be stored in a dark, dry place at laboratory temperature in the original container until used.

Sample preparation for measurement

The material should be dried at 500 °C for 6 h without crushing or grinding the material. After drying, it should be placed in a desiccator with silica gel or other desiccant, and cooled to room temperature before weighing.

The mass of the samples should be corrected for buoyancy.

The density of the potassium chloride sample is 1.984 g/cm^3.

The quantity of sample to be used in the assay is not less than 150 mg.

Measurand and measurement method:

Potassium chloride assay based on chloride content. The results will be reported as amount content of chloride in KCl [mol/kg] and/or mass fraction of chloride expressed as KCl [kg/kg]. The mass fraction of chloride expressed as KCl in the sample is in the range 0.999 to 1.000 (99.9 % to 100 %). Any method or combination of methods can be used, but the coulometric method is recommended for this comparison.

Reporting

The report should be sent to the coordinating laboratory before 15 March 2008, preferentially by e-mail. The coordinator will confirm the receipt of each report to the participant. If the confirmation does not arrive within one week, please contact the coordinator to identify the problem.

A template for the report will be enclosed (Excel spreadsheet). If possible, the requested data should be entered into the corresponding boxes. If this is not possible, the format can be modified or the data can be reported in another form.

Information requested:

1. The results will be reported as amount content of chloride in KCl [mol/kg] and/or mass fraction of chloride expressed as KCl [kg/kg], to be accompanied by a full uncertainty budget.

   For example:

   If the chloride in KCl was determined by coulometry, the amount of Cl (after correction for Br and/or I) divided by the sample mass (corrected for air buoyancy) is the amount content of chloride [mol/kg] in the KCl sample. Such as: 13.4XXX mol/kg. In another way, the chloride content can also be expressed as mass fraction [kg/kg] of KCl, where the reported KCl mass fraction is based on the content of chloride in the sample (i.e."KCl" is the assumed form of the Cl present). Such as: 0.99XXX kg/kg. However, this alternate way includes a contribution to the uncertainty from the uncertainties of the molar masses of Cl and K.

2. Information on impurities determined (value, uncertainty, method), the correction included in the reported chloride content - value and uncertainty of this correction.

3. Participants using coulometry should also report, as an information value, the

coulometric result uncorrected for impurities.

4. Participants using indirect assay ("100 % - trace components" approach) should also report, as an information value, the sum of the mass fractions of K and Cl.

5. A detailed description of the measurement procedure is to be given (for coulometry this should include the following: cell description, volume of electrolyte in working chamber, the number of stages used in the titration and the current used for each stage, evaluation procedure for the endpoint, examples of the titration curve for initial and final endpoint determination), and the equipment used.

6. The complete measurement equation has to be given, as well as the values of the constants (suggested Faraday constant: 96485.3399(24) C mol^{-1}) used and variables (raw data) for at least one measurement. The data should enable the recalculation of the result of this measurement. If trace element correction is used, the relevant data must be included here also.

7. At least six determinations should be performed. Please state all the individual results, not only the final mean value. The uncertainty budget must include instrumental sources of uncertainty (mass, time, voltage, volume, ...) as well as chemical ones (endpoint estimation, equilibria, impurities, purity of calibration standards, ...) plus the relevant uncertainties for any trace element corrections. The uncertainty calculations should conform to the ISO document: *Guide to the Expression of Uncertainty in Measurement* (1995) 1st ed., ISO, Geneva. Both Type A and Type B uncertainty components and a summary of how they are calculated have to be included. The reported uncertainty should be expressed as a combined standard uncertainty and as an expanded uncertainty calculated using a coverage factor, *k*, of 2

8. In order to facilitate comparisons of your measured masses (for assay measurements), please also provide either (1) the air density used for each buoyancy correction, or (2) the air temperature, humidity and pressure in your laboratory at the time of each mass measurement.

9. Report the details of the procedure used (a separate text file or official report may be used).

10. If the results are reported as mass fraction, state also the molar mass of KCl you used and the uncertainty in this molar mass.

Reference value

The reference value will be agreed upon at the combined meeting of the Inorganic Analysis Working Group (IAWG) and Electrochemical Analysis Working Group (EAWG) in October 2008.

Participation

Organisations which are a national metrological institute (NMI), or an appropriate designated laboratory in accordance with the CIPM MRA, are invited to participate in this key comparison.

The Draft A Report based on the reported results will be prepared and sent to the participants for comments and will be discussed at the October 2008 at the combined meeting of the IAWG and EAWG.

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