

## **FINAL REPORT**

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### **Field**

Amount of substance

### **Subject**

Comparison of primary standards of sulphur dioxide in nitrogen

### **Participants**

China (NIM), Korea (KRISS), Japan (CERI),

### **Background**

Reference gases of sulphur dioxide in air (or nitrogen) are used for the calibration of analyzers used to measure sulphur dioxide levels in atmospheric air or exhaust gas from mobile emission sources and fixed sources such as industry. Sulphur dioxide is one of the most important substances in view of environmental and human health problems.

The key comparison CCQM-K1 of sulphur dioxide in nitrogen at nominal amount fractions of 100 and 1000  $\mu\text{mol/mol}$  was executed from 1996 to 1997. Recently preparation techniques for reference gases have improved and the equivalence level is expected to have improved. This comparison APMP.QM-K1.d was intended to be a rerun of the comparison CCQM-K1 in APMP region using the same protocol. CERI (NRLM/CITI), KRISS and NIM (NRCCRM) from APMP (Asia Pacific Metrology Program) region participated in this comparison. The nominal amount fraction of the standards used for the comparison was 90 to 100  $\mu\text{mol/mol}$ . This comparison is linked to CCQM-K1 as described in this report.

## Conduct of the comparison

The Chemicals Evaluation and Research Institute, Japan (CERI) prepared primary standards of sulphur dioxide in nitrogen for distribution to each participating laboratory. The standards were prepared using the procedure defined for gravimetric preparation of gas standards [ISO 6142] with necessary modifications to prevent reactions of sulphur dioxide in cylinders.

The pure sulphur dioxide used to prepare the standards is a reference material (NMIJ CRM 3402-a, NMIJ: National Metrology Institute of Japan). The purity is  $99.99962\% \pm 0.00047\%$  ( $k = 2$ ). The sulphur dioxide was put into a 10 L cylinder. The mass of sulphur dioxide is obtained by weighing the 10 L cylinder before and after the filling. Then a balance of high purity nitrogen was added in the cylinder. The mass of nitrogen was measured by weighing the cylinder. The nitrogen was analysed for sulphur dioxide and found to be less than 5 nmol/mol. The 10 L cylinders were manufactured from aluminium, with a passivated inner surface that minimises the reactivity of the cylinder walls with contents. Tests carried out previously at CERI confirmed that sulphur dioxide-in-nitrogen standards contained in them were stable over the time-scales required for this comparison. The pressure in the cylinders was approximately 10 MPa when distributed.

The participants measured the concentration of sulphur dioxide in the cylinder received with respect to their own standards of sulphur dioxide in nitrogen. The methods reported are given in **Table 1**. After the completion of the comparison, the standards were returned to CERI where they were re-analysed by comparison with primary standards that had not been distributed. These measurements showed no significant change in the sulphur dioxide amount fraction within the estimated uncertainty of the measurements. The combined standard uncertainty estimated by CERI for the gravimetric value of the sulphur dioxide amount fraction was 0.10  $\mu\text{mol/mol}$ . CERI reanalyzed the returned cylinders from each participant and evaluated the instability of the cylinders during the comparison. Deviations of sulphur dioxide concentration between analytical values measured before and after the shipment to the participants were 0.08, 0.15 and -0.07  $\mu\text{mol/mol}$ . The instability result of the delivered standards is the major component of the whole uncertainty of the delivered standards but it is small enough in this comparison.

**Table 1.** Conditions of APMP.QM-K1.d: comparison of sulphur dioxide in nitrogen

Laboratory	Cylinder number	Analytical method	Standard	Calibration model	No. of measurements	Total no. of sub-measurements
CERI	CPB-19101	ND-IR	gravimetric	2 point bracketing	5	15
KRISS	CPB-19099	ND-IR	gravimetric	2 independent single point calibration	7	42
NIM	CPB-19100	UV-FL	gravimetric	4 single point calibration	3	18

NDIR: non-dispersive infrared analyzer  
UV-FL: Pulsed UV fluorescence SO<sub>2</sub> analyzer

### Uncertainty of the distributed standards

Each distributed standard materials was prepared using gravimetric method. The uncertainty of the standard gases was estimated by combining the uncertainty of weighing including the expansion of a gas cylinder by pressure, purity analysis of the component gases and the stability of gases in a cylinder.

The relative standard uncertainties of distributed standards are given in **Table 2**.

**Table 2.** The standard uncertainty of distributed standards

components	relative standard uncertainty
purity analyses	0.01 %
weighing	0.03 %
stability (6 months)	0.09 %
Total	0.10 %

### Results and link to CCQM-K1

The results submitted by the three participants are shown in **Table 3** and **Figure 1**. In **Figure 1**, the results are plotted in terms of their deviation from the gravimetric value. Expanded uncertainties are calculated using a coverage factor  $k = 2$ , as an approximation of 95% confidence intervals.

Three participants submitted results that were within 0.6% of the relevant reference value. In these cases, the estimated uncertainty was larger than the deviation from the reference value. The agreement of the results in this key comparison is good.

The Mutual Recognition Arrangements (MRA) require that the results of a Key Comparison carried out by a Regional Metrology Organisation (RMO) should be linked to those of the corresponding key comparison through the participation of one or more NMIs in both phases of the comparison. In this case, CERI have participated in both phases of the Key Comparison and form the link. A small additional uncertainty contribution has been added to allow for their offset from the KCRV (as described in Annex 1).

### Coordinators

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### Completion Date

August 6, 2006.

**Table 3.** Results of key comparison APMP.QM-K1d sulphur dioxide in nitrogen

**Key comparison APMP.QM-K1d**

**MEASURAND:** Amount-of-substance fraction of sulphur dioxide in nitrogen

**NOMINAL VALUE:** 90 - 100  $\mu\text{mol/mol}$

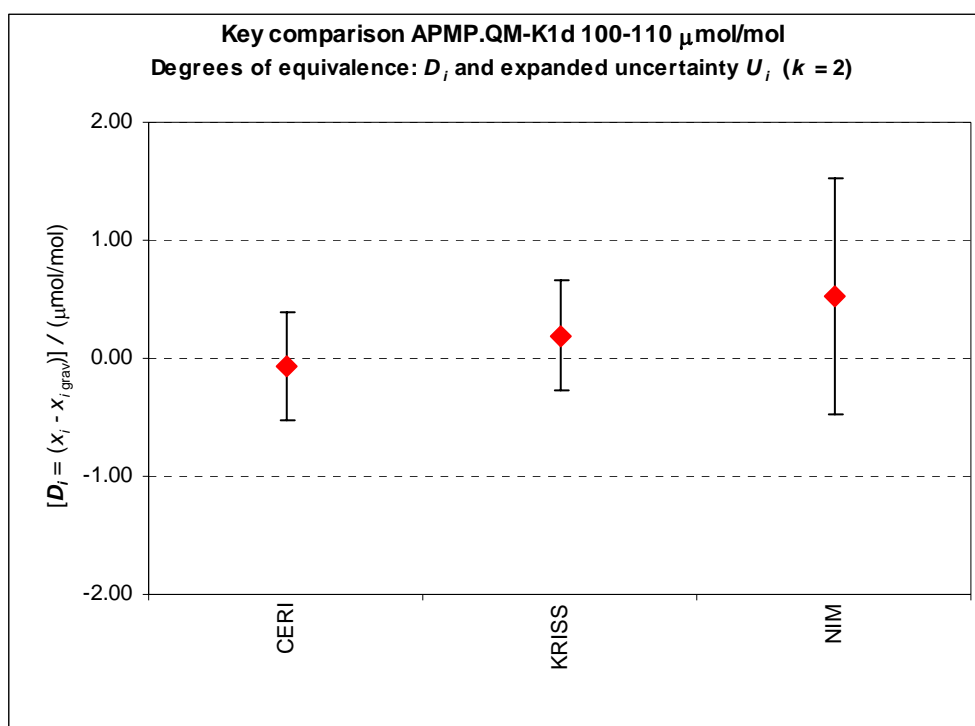
$x_i$ : result of measurement carried out by laboratory  $i$

$u_i$ : combined standard uncertainty of  $x_i$

$x_{i\text{grav}}$ : gravimetric value of the sulphur dioxide amount-of-substance fraction in the cylinder received by laboratory  $i$

$u_{i\text{grav}}$ : combined standard uncertainty of  $x_{i\text{grav}}$

Lab $i$	$x_i$ $\mu\text{mol/mol}$	$u_i$ $\mu\text{mol/mol}$	$x_{i\text{grav}}$ $\mu\text{mol/mol}$	$u_{i\text{grav}}$ $\mu\text{mol/mol}$	Date of measurement	$\Delta x$ $(\mu\text{mol/mol})$
CERI	98.13	0.208	98.20	0.10	2005-11	-0.07
KRISS	96.88	0.21	96.69	0.10	2006/7/5-10	0.20
NIM	98.5	0.49	97.98	0.10	2006-01,02	0.53



**Figure 1.** Results of key comparison APMP.QM-K1d sulphur dioxide in nitrogen

**Annex 1 - Degrees of Equivalence**

**Key comparison APMP.QM-K1d**

**MEASURAND: Amount-of-substance fraction of Sulphur dioxide in nitrogen**

**NOMINAL VALUE: 90-100  $\mu\text{mol/mol}$**

**Key comparison reference value: the value  $x_{i\text{grav}}$  is taken as the reference value for laboratory  $i$ .**

The degree of equivalence of each laboratory with respect to the reference value is given by a pair of numbers:  $D_i = (x_i - x_{i\text{grav}})$  and  $U_i^2 = 2^2(u_i^2 + u_{i\text{grav}}^2)$ , its expanded uncertainty ( $k = 2$ ), both expressed in  $\mu\text{mol/mol}$ . This comparison has been linked to CCQM-K1d through the participation of CERI (/NRLM/NMIJ). An additional uncertainty contribution of 0.1 mmol/mol has been added in quadrature to allow for the offset from the KCRV of CERI in this comparison.

	$D_i$	$U_i$
	$\mu\text{mol/mol}$	
CERI	0.07	0.47
KRISS	0.19	0.48
NIM	0.52	1.00

## Annex 2

### Contact persons

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Report from CERI

– Results for SO<sub>2</sub> in nitrogen –

Laboratory : Chemicals Evaluation and Research Institute, Japan  
 Cylinder number : CPB-19101

NOMINAL COMPOSITION

- Sulfur dioxide : 90 to 100 μmol/mol  
 - nitrogen : balance

Measurement No. 1	Date	Result (μmol/mol)	stand. deviation (% relative)	number of sub-measurements
SO <sub>2</sub>	14/11/2005	98.08	0.063	3

Measurement No. 2	Date	Result (μmol/mol)	stand. deviation (% relative)	number of sub-measurements
SO <sub>2</sub>	15/11/2005	98.14	0.072	3

Measurement No. 3	Date	Result (μmol/mol)	stand. deviation (% relative)	number of sub-measurements
SO <sub>2</sub>	16/11/2005	98.20	0.101	3

Measurement No. 4	Date	Result (μmol/mol)	stand. deviation (% relative)	number of sub-measurements
SO <sub>2</sub>	17/11/2005	98.10	0.062	3

Measurement No. 5	Date	Result (μmol/mol)	stand. deviation (% relative)	number of sub-measurements
SO <sub>2</sub>	18/11/2005	98.15	0.066	3

Results:

Analyte	Result (assigned value)	Coverage factor	Assigned expanded uncertainty
SO <sub>2</sub>	98.13	2	0.42

Reference Method:

Instruments for SO<sub>2</sub> measurement  
Principles : Non Dispersive Infra Red (NDIR)  
Make : Shimadzu cooperation.  
Type : URA-107  
Data collection : output of integrator recording

Calibration Standards:

Preparation : Gravimetric method

Purity analysis ;

SO<sub>2</sub> : certified by NMIJ(National Metrology Institute of Japan)

N<sub>2</sub> : The impurities in N<sub>2</sub> are determined by analyses and the amount of the major component is conventionally determined by,

$$X_{pure} = 1 - \sum_{i=1}^N x_i$$

where:

$x_i$  = mole fraction of impurity  $i$ , determined by analysis

$N$  = number of impurities likely in the final mixture

$X_{pure}$  = mole fraction 'purity' of the 'pure' parent gas

Impurities in the component gases: shown in table 1

SO<sub>2</sub> : NMIJ performed analysis

Impurities present in the balance gas (or in other components): shown in table 2

N<sub>2</sub>: CERI performed analysis

The impurities are less than the specifications by the manufacture. When a possible component is not detected, the concentration is estimated that it is half of the limit of detection. The standard uncertainties are uncertainty of analysis or estimated as  $1/\sqrt{3}$  of the half of the limit of detection.

Table 1 Purity table for high-purity SO<sub>2</sub>

component	analyzed mole fraction	standard uncertainty	distribution	Analytical Methods
N <sub>2</sub>	3.1	1.8	Normal	GC-PID
O <sub>2</sub>	2.1	1.2	Normal	GC-PID
CH <sub>4</sub>	0.21	0.12	Rectangular	GC-FID
C <sub>3</sub> H <sub>8</sub>	0.68	0.39	Rectangular	GC-FID
H <sub>2</sub> O	70.2	1.2	Normal	Karl-Fischer
SO <sub>2</sub>	99.99265	2.4		

Table 2 impurity of balance gases ( μmol/mol )

component	Specifications by manufacturer	distribution	analyzed mole fraction	standard uncertainty
H <sub>2</sub>	<= 0.05	rectangular	<= 0.05	0.014
O <sub>2</sub>	<= 0.05	rectangular	<= 0.05	0.014
CO	<= 0.05	rectangular	<= 0.01	0.003
CO <sub>2</sub>	<= 0.05	rectangular	<= 0.01	0.003
CH <sub>4</sub>	<= 0.1	rectangular	<= 0.01	0.003
C <sub>3</sub> H <sub>8</sub>	<= 0.05	rectangular	<= 0.01	0.003
NO <sub>x</sub>	<= 0.005	rectangular	<= 0.005	0.001
SO <sub>2</sub>	<= 0.005	rectangular	<= 0.005	0.001
H <sub>2</sub> O	<= 0.5	rectangular	<= 1	0.289
N <sub>2</sub>	>= 999999		999999.4	0.29

Instrument Calibration:

Table 1 concentration of PSMs

Component	Concentration ( μmol/mol )	
	R <sub>1</sub>	R <sub>2</sub>
SO <sub>2</sub>	100.74	81.54

This procedure is for the determination of SO<sub>2</sub> in a sample using NDIR.

- 1) Inject the calibration standard (R<sub>1</sub>) into NDIR. Record the output.
- 2) Inject the sample to be tested in same manner as the calibration standard. Record the output.
- 3) Inject the calibration standard (R<sub>2</sub>). Record the output.
- 4) Calculate the concentration of SO<sub>2</sub> using the formula below.

$$Y = \frac{A(E - D) + B(C - E)}{(C - D)}$$

where Y: Concentration of sample  
A: Concentration of standard (R<sub>1</sub>)  
B: Concentration of standard (R<sub>2</sub>)  
C: Standard (R<sub>1</sub>) output  
D: Standard (R<sub>2</sub>) output  
E: Sample output

Following above procedure, 3 measurements are repeated subsequently in a day and iterated for 5 days.

Sample Handling:

Stabilization : none  
Pressure : 100 kPa  
Sample flow : 1.0 l/min  
Sample line temperature : room temperature 25°C)  
Dilution : none

Uncertainty:

Uncertainty source	Estimate $x_I$	Assumed distribution	Standard uncertainty $u(x_i)$	Sensitivity coefficient $c_I$	Contribution to standard uncertainty $u_I(y)$
Repeatability of analysis	98.13	normal	0.073	1	0.073
Reference gas R1 preparation	100.74	normal	0.10	0.864	0.0864
Reference gas R1 stability	100.74	rectangular	0.20	0.864	0.1728
Reference gas R2 preparation	81.54	normal	0.08	0.136	0.0109
Reference gas R2 stability	81.54	rectangular	0.16	0.136	0.0218
total					0.208

Coverage factor: 2

Expanded uncertainty: 0.42  $\mu\text{mol/mol}$

## Report from KRISS

### – Results for SO<sub>2</sub> in nitrogen –

Laboratory : KRISS  
Cylinder number : CPB19099

#### NOMINAL COMPOSITION

- Sulfur dioxide : 90 to 100 .μ mol/mol  
- Nitrogen : balance

Measurement No.	Date	Result (μmol/mol)	stand. deviation (% relative)	number of sub-measurements
1	2006/7/5	96.87	0.02	6
2	2006/7/6	96.87	0.04	6
3	2006/7/6	96.90	0.02	6
4	2006/7/7	96.87	0.02	6
5	2006/7/7	96.89	0.03	6
6	2006/7/10	96.89	0.02	6
7	2006/7/10	96.87	0.02	6

#### Results:

Analyte	Result (assigned value) μmol/mol	Coverage factor	Assigned expanded uncertainty μmol/mol
SO <sub>2</sub>	96.88	2	0.42

#### Reference Method:

SO<sub>2</sub> analyzer (Ultramat 6, Siemens) was used for this measurement.

We made a new auto sampling system using one regulator to remove adsorption problems on the regulator. Sample and zero gases (pure nitrogen) were introduced into analyzer for 4 min alternatively. We used ABA method to correct instrumental drift. Sample and reference gases were determined 6 times, respectively. And gas flow rate was controlled to 400 ml/min by MFC.

#### Calibration Standards:

We used Al cylinders (Luxfer, Au) with stainless steel valve.

The calibration standards were prepared by gravimetric method in our institute as follow.

2 % mol/mol → 2,000 μmol/mol → 100 μmol/mol (2 cylinders)

Purity of SO<sub>2</sub> source gas was determined by impurity analysis. Overall uncertainty of the 100 μmol/mol standards including purity of the source gas, weighing uncertainty, and manufacturing uncertainty was about 0.2 %.

#### Instrument Calibration:

SO<sub>2</sub> analyzer was calibrated with zero gas and 100 μmol/mol standard gas prepared by gravimetric method. And ABA method was used.

#### Sample Handling:

After receiving sample cylinder, cylinder was stood at room temperature with reference cylinders before measurements.

Uncertainty:

Quantity	Value ( $\mu\text{mol/mol}$ )	Standard Uncertainty ( $\mu\text{mol/mol}$ )	Sensitivity coefficient	Corr.- coeff.	Index (%)
Meas. 1	96.87	0.02	0.14	0.01	0.0
Meas. 2	96.87	0.04	0.14	0.02	0.0
Meas. 3	96.90	0.02	0.14	0.01	0.0
Meas. 4	96.87	0.02	0.14	0.01	0.0
Meas. 5	96.89	0.03	0.14	0.02	0.0
Meas. 6	96.89	0.02	0.14	0.01	0.0
Meas. 7	96.87	0.02	0.14	0.01	0.0
Uncertainty of PRM	0	0.15	1.0	0.55	30.7
Uncertainty of reproduceability	0	0.73	1.0	0.83	69.1

Results :

Quantity: 96.88  $\mu\text{mol/mol}$

Coverage factor: 2.0

Expanded uncertainty: 0.42  $\mu\text{mol/mol}$

## CCQM Bilateral Comparisons Sulfur dioxide in nitrogen Measurement report

Laboratory : NRCCRM  
Cylinder number : CPB19100

**NOMINAL COMPOSITION**

- sulphur dioxide : 90 to 100  $\mu\text{mol/mol}$   
- nitrogen : balance

Measurement No. 1	Date	Result ( $\mu\text{mol/mol}$ )	stand. deviation (% relative)	number of sub-measurements
SO <sub>2</sub>	Jan. 12, 2006	98.4	0.1	6

Measurement No. 2	Date	Result $\mu\text{mol/mol}$	stand. deviation (% relative)	number of sub-measurements
SO <sub>2</sub>	Jan. 20, 2006	98.7	0.1	6

Measurement No. 3	Date	Result ( $\mu\text{mol/mol}$ )	stand. deviation (% relative)	number of sub-measurements
SO <sub>2</sub>	Feb. 11, 2006	98.5	0.1	6

**Results:**

Analyte	Result (assigned value) $\mu\text{mol/mol}$	Coverage factor	Assigned expanded uncertainty
SO <sub>2</sub>	98.5	2	1%

**Reference Method:**

Pulsed fluorescence SO<sub>2</sub> analyzer was used to measure the sulphur dioxide in nitrogen.

Components	SO <sub>2</sub>
Instruments	Pulsed fluorescence SO <sub>2</sub> analyzer Thermo Environmental instrument, Inc
Model	43C
Flow rate (sampling)	1L/min
Average time	60s
Range	(0~100) $\mu\text{mol/mol}$

**Calibration Standards:**

Four sulphur dioxide in nitrogen gas mixtures were used as calibration standards to analyse the sample. The calibration standards were prepared by gravimetric method according to ISO 6142, and the detail information was listed in table 1. The impurities of complementary gas and impurities of components interested were determined with a standard normalized method by gas chromatography instrument. Experiments showed that the impurities of the material gases have no effects to the results within the measurement uncertainties. So the purity of pure gases used for preparation was taken from the certificates of producer. Their uncertainties were calculated by type B evaluation.

**Table 1 Calibration Standards**

Components	Assigned value (x)					Relative standard uncertainty ( $u(x)$ ),%
	( $\mu\text{mol/mol}$ )					
Cylinder number	202348	210572	202361	202008		
Sulphur dioxide in nitrogen	97.26	98.08	95.89	96.97		0.5

**Instrument Calibration:**

One point calibration method was used to analyse the sample mixture. Concentrations of four standards mixture listed in table 1. The sample was measured in four different days based upon the six calibration standards. One measurement sequence was in the order of standard A - sample - standard A' - standard B - sample - standard B' - .....- standard D - sample - standard D'. After averaging two response values of the same calibration standard, the concentration of sample mixture was calculated by direct comparison based on the following equation (1).

$$C_{sample,i} = \frac{A_{sample,i}}{A_{st,i}} \cdot C_{st,i} \quad (1)$$

$C_{sample,i}$  is the concentration of component i in sample mixture to be calculated,

$A_{sample,i}$  is the response value of component i in sample mixture,

$A_{st,i}$  is the response value of component i in the calibration standard mixture,

$C_{st,i}$  is the concentration of component i in the calibration standard mixture.

Temperature and pressure were not corrected during the calibration procedure.

**Sample Handling:**

Sample cylinder after arrival was stored in the room temperature. Sample and standard gas were all directly led to pulsed fluorescence SO<sub>2</sub> analyzer by a regulator, a mass flow control meter and a Teflon pipe. Before each sample injection, the pipe system was purged for five minutes.

**Uncertainty:**

For sulfur dioxide component, we established three types of uncertainties:

- Standard mixture uncertainty, which mainly depend upon the gravimetric method uncertainty
- stability uncertainty
- Analytical uncertainty

The Gravimetric method uncertainty contributions included:

- Balance uncertainty
- Buoyancy of cylinders

- Impurity of gases
- Absorption

The stability uncertainty is evaluated according to the past experimental results.

The analytical uncertainty was evaluated by repeatability of the measurements.

The relative standard uncertainty of sulphur dioxide component in sample mixture was evaluated by equation (3)

$$u^2(C_{sample,i},\%) = u_{gravi}^2 + u_{stab}^2 + u_{repea}^2 \quad (2)$$

The relative standard uncertainty of standard mixture included the gravimetric method uncertainty, stability uncertainty and analytical uncertainty. The relative standard uncertainty of repeatability was the RSD% of the four concentration values of the component, which were determined in four different days.

The amount of each contribution to the measurement uncertainty was listed in table 2.

The absolute standard uncertainty was calculated by:

$$u(C_{sample,i}) = u(C_{sample,i},\%) \cdot C_{sample,i} \quad (3)$$

Expanded uncertainty can be calculated with a confidence interval 95% and a coverage factor  $k= 2$ . The expanded uncertainty was:

$$U_i = k \cdot u(C_{sample,i},\%)$$

**Table 2 Uncertainty Evaluation**

Uncertainty source	Estimate (relative,%) $X_i$	Assumed distribution	Standard uncertainty (relative,%) $u(x_i)$	Sensitivity coefficient $c_I$	Contribution to standard uncertainty $U_i(y)$ (relative,%)
Gravimetric method	0.9	Rectangle	0.5	1	0.5
Stability	0.5	Normal	0.2	1	0.2
Analytical	0.2	Normal	0.1	1	0.1

Coverage factor: 2

Expanded uncertainty: 1%