

The Network Center for the Acid Deposition Monitoring Network in East Asia

Report of the Inter-laboratory Comparison Project 2005 on Wet Deposition

8th Attempt

November 2006

Acid Deposition and Oxidant Research Center

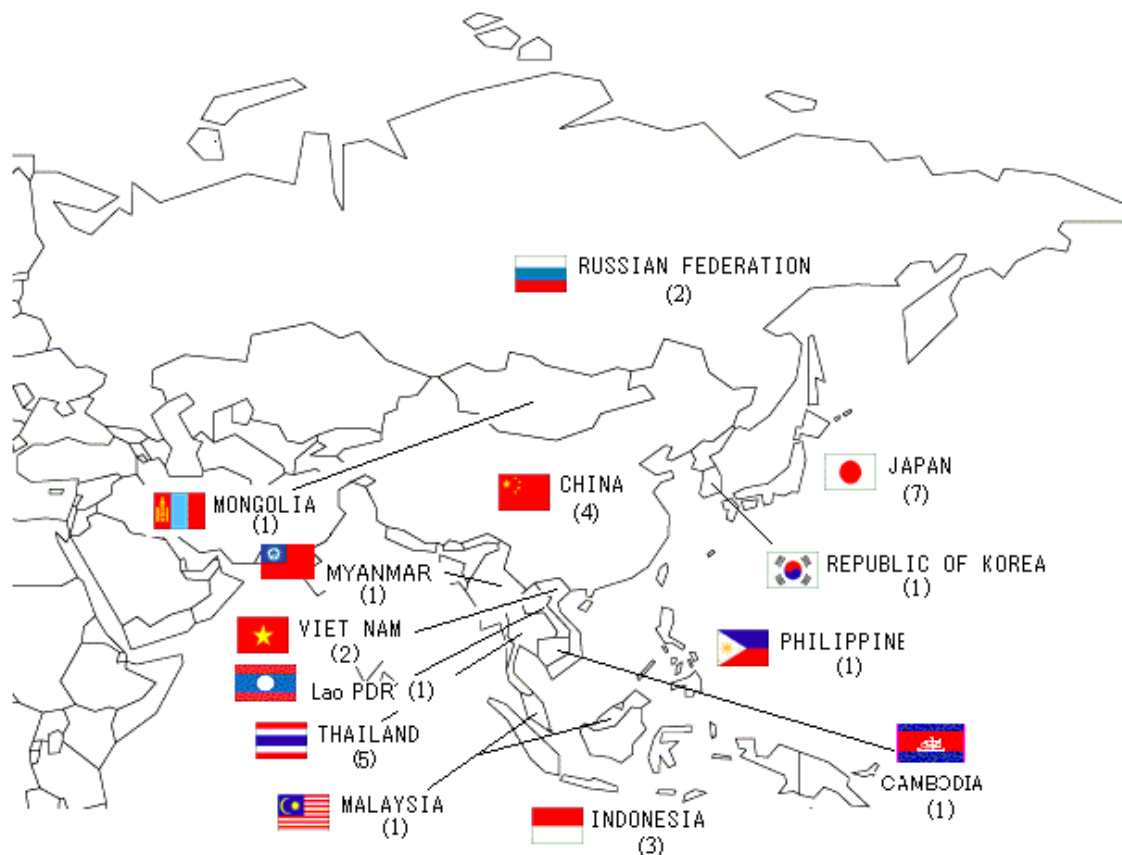
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1. INTRODUCTION

This inter-laboratory comparison project (round robin analysis survey of uniformly prepared artificial rainwater samples) was conducted among the analytical laboratories in participating countries of the Acid Deposition Monitoring Network in East Asia (EANET), based on the Quality Assurance / Quality Control (QA/QC) Program of EANET. The purposes of this project are, through the evaluation of analytical results, analytical equipment and its operating condition, (i) to recognize the analytical precision and accuracy of the data in each participating laboratory, and give an opportunity to improve the quality of the analysis on wet deposition monitoring, and (ii) to improve reliability of analytical data through the assessment of suitable analytical methods and techniques.

Artificial rainwater samples contained major ions, were prepared and distributed by the Network Center (NC) in November 2005. All of the participating laboratories submitted their analytical data to NC. Obtained data for pH, EC and concentrations of SO_4^{2-} , NO_3^- , Cl^- , Na^+ , K^+ , Ca^{2+} , Mg^{2+} and NH_4^+ were compared with prepared values and statistically treated. List of the participating laboratories, individual analytical data with their laboratory's short name, and various statistical parameters are included in this report.



The some laboratories out of EANET submitted data in this project but these data weren't included in statistics. (see APPENDIX2-3)

* Figure in parenthesis indicates the number of laboratories of each country (30 laboratories from 13 countries)

Fig.1 Laboratories participated in the Inter-comparison project 2005 of the EANET

2. PROCEDURE

2.1 Participating Laboratories

The laboratories in charge of chemical analysis in 13 countries of EANET participated in this survey. The Network Center (NC) shipped the artificial rainwater samples to these laboratories, and all of them submitted their analytical data to NC. The names of the participating laboratories are presented in **APPENDIX 1**.

2.2 Artificial Rainwater Samples

Two kinds of artificial rainwater samples (of both higher concentration and lower concentration) were distributed to the laboratories (See Table 1).

Table 1 Outline of artificial rainwater samples

Artificial rainwater samples	Amount of each sample	Container	Number of samples	Note
No.051 (higher concentration) No.052 (lower concentration)	Approximately 100mL	Poly-propylene bottle 100mL	One bottle each	Known amount of reagents are dissolved in deionized water

Before the measurement, each laboratory should accurately dilute distributed samples by 100 times under the specified procedure (2.3 Analytical Method).

The information on the analytical precision and accuracy on individual parameters can be obtained through the statistical treatment of submitted analytical data of 100 times diluted samples.

All participating laboratories were expected to analyze samples and submit the data with the units listed in Table 2 on ten parameters: pH, Electric Conductivity (EC), concentrations of sulfate, nitrate, chloride, sodium-ion, potassium-ion, calcium-ion, magnesium-ion and ammonium. The participating laboratories were informed that concentration of each parameter was prepared within prescribed range (Table 2).

Table 2 Reporting units and concentration range of analytical parameters*

Analytical parameter	Reporting Units		Range of Values
pH	pH Unites	-	4.0 – 5.5
EC	milli siemens/meter	(mS/m)	1.0 – 10.0 mS/m
SO ₄ ²⁻	micro mole/liter	(μmol/L)	5 – 100 μmol/L
NO ₃ ⁻	micro mole/liter	(μmol/L)	5 – 100 μmol/L
Cl ⁻	micro mole/liter	(μmol/L)	5 – 150 μmol /L
Na ⁺	micro mole/liter	(μmol/L)	1 – 100 μmol /L
K ⁺	micro mole/liter	(μmol/L)	1 – 50 μmol /L
Ca ²⁺	micro mole/liter	(μmol/L)	1 – 50 μmol /L
Mg ²⁺	micro mole/liter	(μmol/L)	1 – 50μmol /L
NH ₄ ⁺	micro mole/liter	(μmol/L)	1 – 50μmol /L

* For 100 times diluted samples.

2.3 Analytical Procedure

Distributed samples shall be diluted by 100 times accurately in each laboratory according to the specified procedure before the measurement.

Dilution procedure was carried out according to the following procedures;

- (1) Keep the received samples and the deionized water for dilution (electric conductivity less than 0.15 mS/m) in a laboratory for one to two hours until they become at the room temperature.
- (2) Prepare 3 solutions for laboratory analysis from received sample No.051. Take 10mL of the sample No.051 by transfer pipette, and dilute it accurately to 1L by deionized water by following scheme:
 - (i) Wash a 1L volumetric flask with cap more than three times by deionized water.
 - (ii) Fill deionized water into the volumetric flask of (i) about 80% of the flask.
 - (iii) Take 10mL of sample solution using transfer pipette and put into the volumetric flask of (ii). Add the deionized water up to the marked line of the flask. After well mixture, put the diluted solution into the clean 1L polyethylene bottle.
 - (iv) Repeat the same procedure of (i) to (iii) another two times with new flasks, and prepare the three diluted samples (a, b and c)
- (4) Prepare similar 3 solutions for analysis from samples No.052. Take 10mL of the sample No.052 by transfer pipette, and dilute accurately by deionized water same as described in b) and prepare the three diluted sample (d, e, and f).

Note 1) Carry out the measurements /analysis of solution samples as soon as possible after make a calibration curve

Note 2) Calculate the analytical result data of the concentrations of diluted samples.
Measure each samples 3 times for individual parameters.

2.4 Analytical Method and Data Checking Procedures

Participating laboratories were expected to use analytical methods and data checking procedures that are specified in the “Technical Manual for Wet Deposition Monitoring in East Asia” and “Quality Assurance/Quality Control (QA/QC) Program for Wet Deposition Monitoring in East Asia”. Analytical methods specified in the manual are listed in Table 3.

Table 3 Analytical methods specified in the manual

Parameter	Analytical method
pH	Glass Electrode
EC	Conductivity Cell
SO ₄ ²⁻ NO ₃ ⁻ Cl ⁻	Ion Chromatography Spectrophotometry
Na ⁺ K ⁺ Ca ²⁺ Mg ²⁺	Ion Chromatography Atomic Absorption/Emission Spectrometry
NH ₄ ⁺	Ion Chromatography Spectrophotometry (Indophenol Blue)

The checking of analytical results are performed with help of calculation of ion balance (R₁) and total electric conductivity agreement (R₂)

Calculation of ion balance (R₁)

(1) Total anion (**A**) equivalent concentration (μeq /L) is calculated by summing the concentrations of all anions (C: μmol /L).

$$\mathbf{A} (\mu\text{eq} /\text{L}) = \sum n C_{\text{Ai}} (\mu\text{mol} /\text{L}) = 2C (\text{SO}_4^{2-}) + C (\text{NO}_3^-) + C (\text{Cl}^-)$$

n, C_{Ai}: electric charge of ion and concentration (μmol /L) of anion “i”.

(2) Total cation (**C**) equivalent concentration (μeq /L) is calculated by summing the concentrations of all cations (C: μmol /L).

$$\mathbf{C} (\mu\text{eq} /\text{L}) = \sum n C_{\text{Ci}} (\mu\text{mol} /\text{L}) = 10^{(6-\text{pH})} + C (\text{NH}_4^+) + C (\text{Na}^+) + C (\text{K}^+) + 2C (\text{Ca}^{2+}) + 2C (\text{Mg}^{2+})$$

n, C_{Ci}: electric charge of ion and concentration (μmol /L) of cation “i”.

(3) Calculation of ion balance (R₁)

$$R_1 = 100 \times (\mathbf{C}-\mathbf{A}) / (\mathbf{C}+\mathbf{A})$$

(4) R_1 , which is calculated using the above equation, should be compared with standard values in Table 4. If R_1 is out of the range, re-measurement, check with standard solutions, and/or inspection of calibration curves were recommended.

Table 4 Allowable ranges for R_1 in different concentration ranges

C+A ($\mu\text{eq} / \text{L}$)	R_1 (%)
< 50	± 30
50 – 100	± 15
> 100	± 8

(Reference) "Technical Documents for Wet Deposition Monitoring in East Asia (2000)"

Comparison between calculated and measured values of electrical conductivity (R_2)

(1) Total electric conductivity (Λ calc) should be calculated as follows;

$$\Lambda \text{ calc (mS /m)} = \{349.7 \times 10^{(6-\text{pH})} + 80.0 \times 2C (\text{SO}_4^{2-}) + 71.5 C (\text{NO}_3^-) + 76.3 C (\text{Cl}^-) + 73.5 C (\text{NH}_4^+) + 50.1 C (\text{Na}^+) + 73.5 \times C (\text{K}^+) + 59.8 \times 2C (\text{Ca}^{2+}) + 53.3 \times 2C (\text{Mg}^{2+})\} / 10000$$

C: Molar concentrations ($\mu\text{mol} / \text{L}$) of ions in the parenthesis; each constant value is ionic equivalent conductance at 25°C.

(2) Ratio (R_2) of calculations (Λ calc) to measurements (Λ meas) in electric conductivity should be calculated as follows;

$$R_2 = 100 \times (\Lambda \text{ calc} - \Lambda \text{ meas}) / (\Lambda \text{ calc} + \Lambda \text{ meas})$$

(3) R_2 , which is calculated using the above equation, should be compared with standard values in Table 5. If R_2 is out of the range, re-measurement, check with standard solutions, and/or inspection of calibration curves were recommended.

Table 5 Allowable ranges for R_2 in different ranges of EC

Λ meas (mS/m)	R_2 (%)
< 0.5	± 20
0.5 – 3	± 13
> 3	± 9

(Reference) "Technical Documents for Wet Deposition Monitoring in East Asia (2000)"

3. RESULTS

The Network Center received the data on analytical results of artificial rainwater samples from 30 laboratories in the participating countries of EANET. General statistics of obtained data summarized in Table 6 were calculated for each constituent of the artificial rainwater samples such as: Average, Minimum (Min.), Maximum (Max.), Standard deviation (S.D.), and Number of data (N). Outlying data, which are apart from the average greater than a factor of 3 of S.D. were not included for this calculation. As shown in Table 6, average of submitted data were fairly well agreed with the prepared values/concentrations within a range of -3.1% (EC) to $+1.8\%$ (NH_4^+) for the sample No.051, and -6.7% (K^+) to 5.0% (Mg^{2+}) for the sample No.052. But there are a few laboratories that submitted measured values with considerable differences forward prepared concentrations.

**Table 6 Summary of analytical results of the artificial rainwater samples
(Reported data after removing of outliers)**

Constituents	Prepared (Vp)	Average (Va)	Va/Vp (%)	S.D.	N	Min.	Max.
[Sample No.051]							
pH	4.66	4.65	-0.3	0.15	29	4.16	4.82
EC(mS/m)	3.32	3.22	-3.1	0.11	28	2.94	3.59
SO_4^{2-} ($\mu\text{mol/L}$)	43.7	42.4	-3.0	1.84	27	37.5	45.8
NO_3^- ($\mu\text{mol/L}$)	40.3	39.5	-1.9	1.39	26	36.4	43.7
Cl^- ($\mu\text{mol/L}$)	68.5	66.9	-2.4	3.29	27	57.9	74.1
Na^+ ($\mu\text{mol/L}$)	56.5	56.7	0.3	5.92	27	40.5	73.8
K^+ ($\mu\text{mol/L}$)	6.9	6.7	-2.6	1.14	27	4.4	9.2
Ca^{2+} ($\mu\text{mol/L}$)	23.2	23.6	1.7	1.68	27	20.6	28.0
Mg^{2+} ($\mu\text{mol/L}$)	11.7	11.7	0.2	0.71	26	10.7	13.5
NH_4^+ ($\mu\text{mol/L}$)	40.9	41.6	1.8	5.38	27	28.9	54.5
[Sample No.052]							
pH	5.05	5.11	1.3	0.25	30	4.60	6.21
EC(mS/m)	1.05	1.06	0.7	0.10	29	0.91	1.53
SO_4^{2-} ($\mu\text{mol/L}$)	14.4	14.1	-2.1	0.80	27	12.4	15.7
NO_3^- ($\mu\text{mol/L}$)	13.2	13.1	-0.9	0.82	27	11.3	15.6
Cl^- ($\mu\text{mol/L}$)	15.3	15.0	-2.0	1.21	26	12.8	18.0
Na^+ ($\mu\text{mol/L}$)	10.3	10.1	-2.3	1.83	27	4.6	13.9
K^+ ($\mu\text{mol/L}$)	3.0	2.8	-6.7	0.63	25	1.8	4.4
Ca^{2+} ($\mu\text{mol/L}$)	7.6	7.7	1.8	1.41	27	3.9	10.9
Mg^{2+} ($\mu\text{mol/L}$)	3.1	3.3	5.0	0.63	26	2.3	5.2
NH_4^+ ($\mu\text{mol/L}$)	13.6	14.2	4.1	2.93	27	5.4	23.2

(Note) Prepared: Value or concentration, which was calculated from the amount of chemicals, used for the preparation of samples.

Va/Vp (%): {Average(Va) / Prepared (Vp) - 1} X 100

The Data Quality Objectives (DQOs) of EANET was specified by the QA/QC program of the EANET for every constituent to be within $\pm 15\%$ of deviation from prepared value. In this report, analytical data on the artificial rainwater samples were compared with the prepared value/concentration and evaluated by the excess of DQOs criteria: the flag "E" was put to the data that exceed DQOs by a factor of 2 ($\pm 15\% \sim \pm 30\%$), and the flag "X" was put to the data that exceed DQOs more than a factor of 2 ($< -30\%$ or $> 30\%$).

A set of data for each sample was evaluated by the data checking procedures described in chapter 2.4. The flag "I" and the flag "C" were added to the data sets with a poor ion balance and conductivity agreement, respectively.

The results were evaluated by the three aspects:

- i) Comparison of concentration dependence on level of their concentration
 - sample No.051 (higher concentrations) and No.052 (lower concentrations),
- ii) Comparison of individual parameters,
- iii) Comparison of circumstances of analysis in each participating laboratory.

Evaluation of data on both the sample No.051 and No.052 is presented in "3.1 Comparison by Sample", evaluation of data for each constituent is presented in "3.2 Analytical Parameter", and evaluation of data by the circumstances of analysis such as analytical method used, experience of personnel, and other analytical condition is presented in "3.3 Circumstance of Sample Analysis".

3.1 Comparison by Sample

Sample No.051 (higher concentrations)

Table 7 Numbers of flagged data for the Sample No.051 (higher concentrations)

Characterization of data	pH	EC	SO ₄ ²⁻	NO ₃ ⁻	Cl ⁻	Na ⁺	K ⁺	Ca ²⁺	Mg ²⁺	NH ₄ ⁺	Total
With flag E*	0	1	0	1	1	2	4	1	1	4	15
With flag X**	1	0	1	0	1	1	4	0	1	2	11
Data within DQOs	29	29	27	26	26	24	19	26	25	21	252
Flagged(%)	3.3	3.3	3.6	3.7	7.1	11.1	29.6	3.7	7.4	22.2	9.4

(Total data=278)

*E : Value exceeded the DQO by a factor of 2

**X : Value exceeded the DQO more than a factor of 2

For sample No.051 (higher concentrations), 15 analytical data out of 278 exceeded the DQOs by a factor of 2 and flagged by "E". 11 analytical data out of 278 exceeded the DQOs more than a factor of 2 and flagged by "X". Data flagged by "E" and "X" shared about 9.4 percents of all reported data for sample No.051. Especially there were many results with flags for measured values of K⁺ and NH₄⁺ (Table 8)

Table 8 Analytical Results of Sample No.051

Lab.ID	pH	EC (mS/m)	SO ₄ ²⁻ (µmol/L)	NO ₃ ⁻ (µmol/L)	Cl ⁻ (µmol/L)	Na ⁺ (µmol/L)	K ⁺ (µmol/L)	Ca ²⁺ (µmol/L)	Mg ²⁺ (µmol/L)	NH ₄ ⁺ (µmol/L)	R1	R2
KH01	X 6.79	2.89	X 7.1		X 27.0							
CN01	4.70	3.24	42.3	40.4	69.3	55.0	7.3	24.2	12.4	38.6	-0.1	-0.2
CN02	4.63	3.28	45.0	43.7	74.1	59.1	6.3	22.6	12.2	39.5	-2.4	2.5
CN03	4.70	3.30	43.2	40.3	68.9	54.8	6.9	25.0	11.1	40.8	-0.2	-0.8
CN04	4.70	3.20	43.0	40.7	67.0	56.8	7.2	24.5	11.3	40.6	0.6	0.6
ID01	4.16	3.07	43.2	40.6	64.6	54.7	X 9.1	21.0	12.8	X 54.5	I 14.2	C 24.1
ID02	4.74	3.14	45.8	38.6	69.1	56.1	6.0	23.9	11.7	E 51.6	1.0	2.3
ID03	4.52	3.00	41.9	38.4	65.5	49.4	6.7	23.6	11.7	41.5	2.8	7.8
JP01	4.71	3.26	42.6	40.2	63.2	56.0	7.1	22.6	11.0	42.4	1.0	-1.5
JP02	4.68	3.35	42.6	40.0	69.6	56.1	6.6	23.9	11.6	42.1	0.5	-1.1
JP03	4.74	3.22	43.5	39.9	67.8	56.1	7.0	23.9	11.6	40.7	-0.4	-0.7
JP04	4.69	3.24	43.9	40.5	68.4	55.6	6.3	22.8	11.1	39.6	-1.8	-0.1
JP05	4.73	3.16	39.2	37.6	E 57.9	55.5	6.4	22.7	11.7	40.8	4.5	-2.4
JP06	4.71	3.23	41.9	39.4	66.8	57.0	7.1	23.9	11.7	39.1	1.0	-0.8
JP07	4.75	3.21	42.9	38.9	64.5	57.6	6.9	24.1	12.1	42.6	2.1	-1.0
LA01	4.37	3.24										
MY01	4.63	2.94	43.3	39.9	68.5	56.5	6.9	22.1	11.1	39.0	-0.7	6.1
MN01	4.74	3.22	45.1	41.6	68.8	58.6	E 8.4	23.2	12.7	46.1	0.6	1.0
PH01	4.67	3.25	42.0	38.0	64.7	54.3	E 4.9	24.7	10.9	E 33.2	-0.5	-1.6
KR01	4.31	3.59	43.5	36.4	71.5	E 40.5	X 4.4	23.8	X 6.4	43.4	0.7	6.9
RU01	4.62	3.32	43.1	39.7	65.0	59.0	6.1	22.1	11.1	40.4	1.3	0.1
RU02	4.70	3.20	37.5	E 48.6	64.9	59.6	E 8.5	21.0	11.5	E 33.9	-0.4	-1.2
TH01	4.70	3.14	40.9	38.7	66.3	57.7	7.0	25.3	13.0	41.2	4.0	1.2
TH02	4.71	3.18	41.4	38.2	69.1	56.1	6.2	22.0	10.7	X 53.9	2.8	1.0
TH03	4.45	3.18	42.9	39.9	67.6	51.7	X 4.7	26.1	10.7	40.8	3.2	8.2
TH04	4.79	3.33	41.0	39.2	60.3	E 72.6	7.8	E 28.0	E 13.5	44.6	I 10.5	-2.1
TH05	4.82	3.14	42.8	39.5	68.9	52.8	X 4.7	26.6	12.2	43.4	-0.1	-0.8
VN01	4.68	3.26	42.1	38.4	63.8	56.6	6.8	22.8	11.9	41.0	2.2	-0.9
VN02	4.77	3.19	38.3	38.9	69.1	X 73.8	E 8.2	20.6	11.4	E 28.9	1.9	-2.7
MM01	4.67	E 3.91										

E:Value exceeded the DQO(±15) by a factor of 2

I:Poor ion balance (R1)

X:Value exceeded the DQO(±15) more than a factor of 2

C:Poor conductivity agreement (R2)

Sample No.052 (lower concentrations)

Table 9 Number of flagged data for the Sample No.052 (lower concentrations)

Characterization of data	pH	EC	SO ₄ ²⁻	NO ₃ ⁻	Cl ⁻	Na ⁺	K ⁺	Ca ²⁺	Mg ²⁺	NH ₄ ⁺	Total
With flag E*	1	0	0	1	2	1	9	8	5	4	31
With flag X**	0	2	1	0	2	3	5	3	4	3	23
Data within DQOs	29	28	27	26	24	23	12	16	17	20	222
Flagged(%)	3.3	6.7	3.6	3.7	14.3	14.8	53.8	40.7	34.6	25.9	19.6

(Total data=276)

*E : Value exceeded the DQO by a factor of 2

**X : Value exceeded the DQO more than a factor of 2

For sample No.052 (lower concentrations), 31 analytical data out of 276 exceeded the DQOs by a factor of 2 and flagged by "E". 23 analytical data out of 276 exceeded the DQOs more than a factor of 2 and flagged by "X". Data flagged by "E" and "X" shared up to 19.6 percents of all reported data for sample No.052. Many data on K⁺, Ca²⁺, Mg²⁺ and NH₄⁺ were marked with flags E or flags X. (Table 10)

Evaluation

The ratio of the flagged data for sample No.051 was 9.4 percent, and the No.041 (2004) was 10.7 percent. Both of them had almost same concentration for each ion. For the sample with low concentration, the ratio of flagged data in the sample No.052 was 19.6 percent and the No.042 (2004) was 13.0 percent. The difference of the rate of data satisfied DQOs between the higher and lower concentration sample spread larger than that of last project.

Comparing the results in 2005 and in 2004, the ratio of flagged data on 7 constituents decreased in higher concentration sample. Especially the ratio of Ca²⁺ decreased more than 10 percent. On the other hand, the ratio of Na⁺, K⁺ and NH₄⁺ increased. The ratio of flagged data on 4 constituents in lower concentration decreased or same in comparison of the results in 2005 with that in 2004. Especially the ratio of SO₄²⁻ decreased significantly. On the other hand, the ratio of K⁺ increased 5 times.

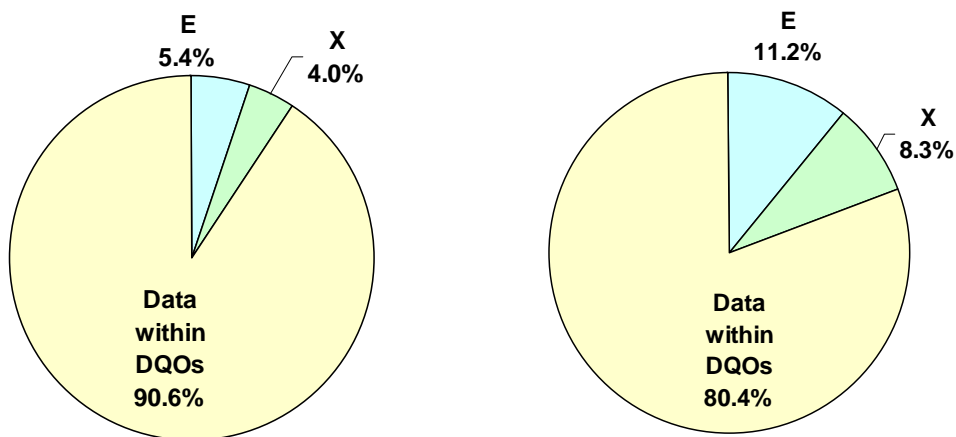


Fig.2 Percentage of flagged data for Sample No.051 and No.052

Table 10 Analytical Results of Sample No.052

Lab.ID	pH	EC (mS/m)	SO ₄ ²⁻ (µmol/L)	NO ₃ ⁻ (µmol/L)	Cl ⁻ (µmol/L)	Na ⁺ (µmol/L)	K ⁺ (µmol/L)	Ca ²⁺ (µmol/L)	Mg ²⁺ (µmol/L)	NH ₄ ⁺ (µmol/L)	R1	R2
KH01	E 6.21	1.14	X 4.4		X 8.0							
CN01	5.08	1.06	14.2	13.9	16.4	11.1	E 2.4	E 9.0	3.2	13.0	0.4	-0.3
CN02	4.99	1.12	13.5	13.1	17.1	11.5	E 2.3	E 8.9	3.4	13.8	4.4	-0.2
CN03	5.04	1.05	15.6	13.5	15.2	11.3	3.1	E 9.0	3.3	14.4	2.1	2.7
CN04	5.07	0.99	14.5	13.8	15.8	11.5	X 1.9	8.4	3.2	13.8	0.3	3.2
ID01	4.91	1.04	13.8	13.0	13.6	9.1	X 4.4	X 5.3	3.0	14.6	2.5	4.1
ID02	5.20	1.01	15.1	12.8	16.1	X 5.0	E 2.5	8.2	2.9	X 23.2	0.1	0.5
ID03	4.80	0.92	13.2	12.1	13.9	X 4.6		8.4		X 5.4		
JP01	5.11	1.07	13.7	13.0	13.4	10.4	2.6	E 6.4	E 2.6	14.8	-0.2	-4.9
JP02	5.00	1.08	14.2	12.6	15.2	9.8	2.6	7.5	3.2	13.0	0.5	-0.4
JP03	5.08	1.05	14.4	13.1	15.2	10.2	3.0	7.7	3.1	13.8	-0.2	-0.9
JP04	5.06	1.08	14.3	13.1	14.9	9.5	E 2.5	7.3	2.7	13.1	-2.5	-2.9
JP05	5.14	1.04	14.3	13.2	15.1	E 8.4	2.6	7.1	E 2.6	14.2	-4.6	-3.5
JP06	5.11	1.03	14.0	13.0	15.1	10.4	3.1	7.9	3.1	13.8	0.9	-1.1
JP07	5.12	1.04	13.7	12.4	13.6	10.2	3.0	8.0	3.0	13.4	2.5	-3.2
LA01	5.47	1.01										
MY01	5.03	0.91	13.7	12.8	15.9	11.1	3.1	7.7	2.9	12.5	1.0	7.2
MN01	5.05	1.04	15.7	13.9	E 18.0	9.1	E 3.6	8.4	X 5.2	13.9	-0.5	4.1
PH01	5.04	1.08	13.0	12.3	13.4	9.1	X 1.8	E 9.7	2.9	E 10.2	3.5	-4.0
KR01	4.60	X 1.53	12.4	14.9	X 22.6	9.7	3.2	X 3.9	E 2.3	12.6	0.6	2.0
RU01	5.00	1.10	14.5	13.4	15.2	11.6	E 2.3	7.2	2.8	13.8	0.1	-0.6
RU02	5.00	1.11	15.2	E 15.6	16.3	10.7	E 3.7	6.9	3.0	E 15.9	-1.8	1.4
TH01	5.12	0.98	14.2	12.7	14.5	11.6	2.9	E 9.1	E 3.6	13.6	4.8	2.0
TH02	5.23	0.97	12.8	11.3	14.7	9.7	2.6	6.8	2.7	X 20.0	5.1	-2.2
TH03	5.03	1.04	14.2	12.8	14.8	10.3	E 2.2	8.7	X 4.1	E 15.8	6.1	2.3
TH04	5.28	1.07	13.4	12.7	E 12.8	X 13.9	E 3.8	X 10.8	X 4.1	E 16.3	I 13.8	-4.6
TH05	5.11	1.03	14.3	12.8	14.7	10.2	X 1.8	8.3	X 4.4	15.6	4.0	0.0
VN01	5.06	1.06	13.5	12.4	13.5	10.6	3.0	E 6.0	3.3	13.8	1.7	-3.1
VN02	5.18	1.02	15.2	13.1	15.3	11.0	X 0.0	E 6.1	E 3.9	13.8	-6.7	-3.3
MM01	5.32	X 1.91										

E: Value exceeded the DQO(±15) by a factor of 2

I: Poor ion balance (R1)

X: Value exceeded the DQO(±15) more than a factor of 2

C: Poor conductivity agreement (R2)

3.2 Analytical Parameter

The general overviews of data were presented below in Figures and Tables for each analytical parameter. The results received from each laboratory were normalized by prepared values to evaluate their deviation. The numbers of flagged data were indicated in the Tables for each analytical parameter.

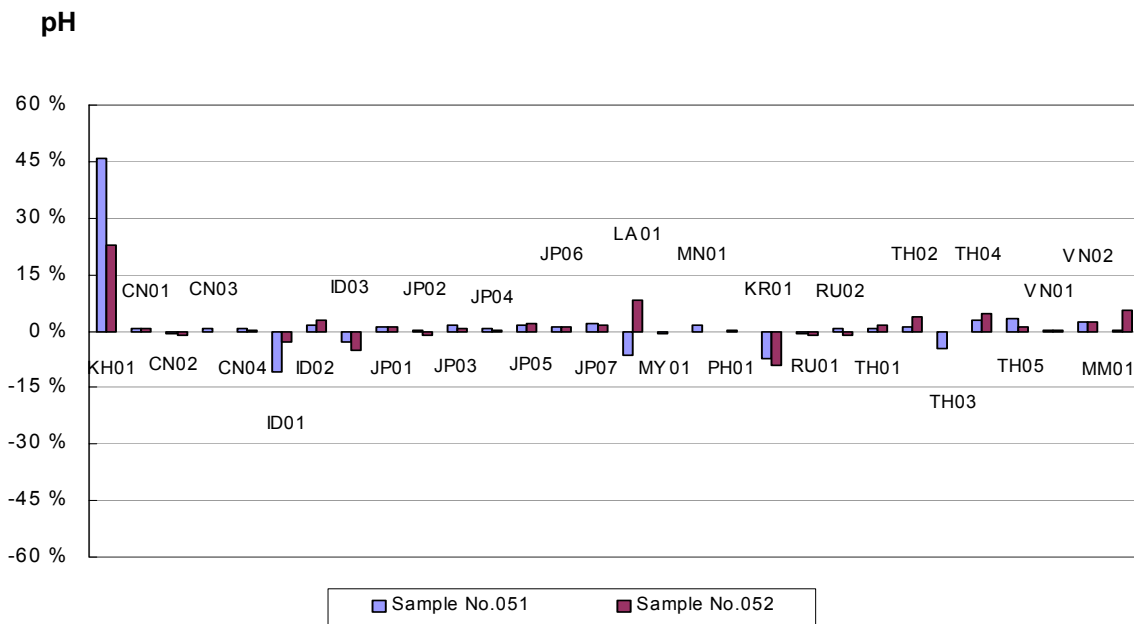


Fig.3 Distribution of pH data normalized by prepared value

All participating laboratories used pH meter with glass electrode for measurement of pH. All of obtained data except KH01 was satisfied the DQOs of the QA/QC program of the EANET. Many laboratories submitted slightly higher pH values than prepared value in both samples. The relative standard deviations of the pH values for sample No.051 and No.052 were 3.2% and 4.9%, respectively.

EC

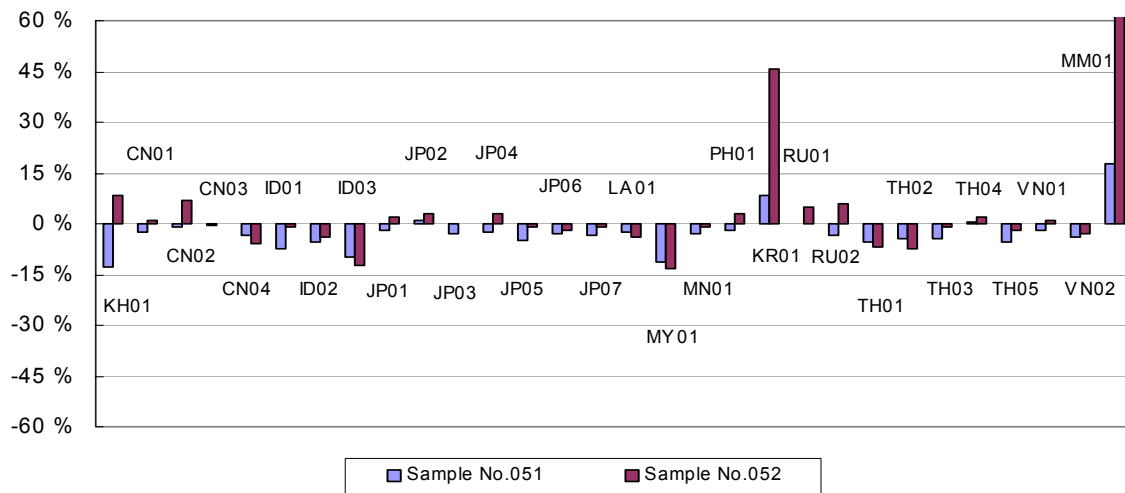


Fig.4 Distribution of EC data normalized by prepared value

All participating laboratories used conductivity cell for the measurement of EC. Almost of obtained data were satisfied the DQOs of the QA/QC program of the EANET. However, Lab.MM01 reported the data flagged by “E” in higher sample and ”x” in lower sample. 16 laboratories reported lower data than prepared value for both samples.

SO₄²⁻

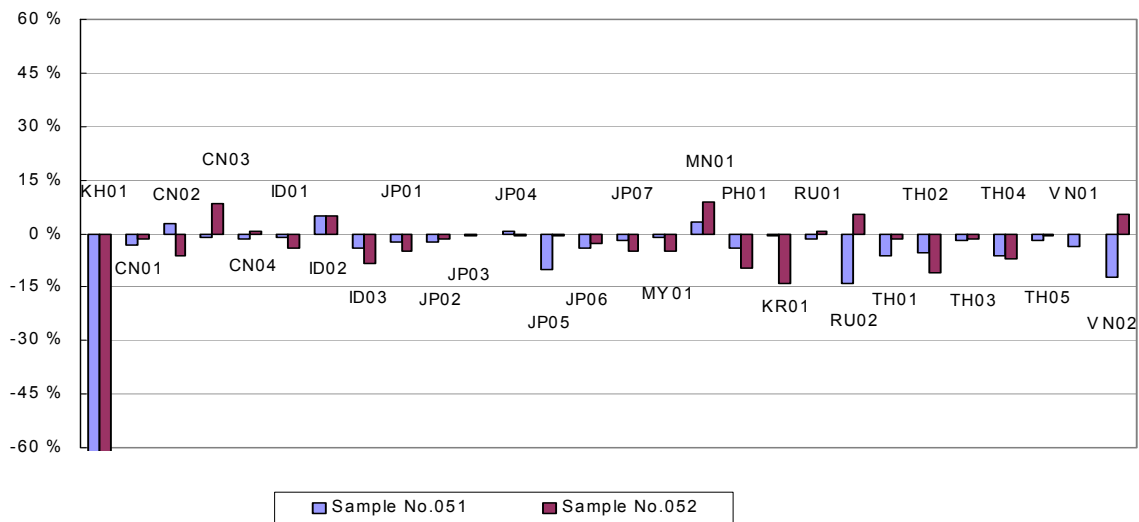


Fig.5 Distribution of SO₄²⁻ data normalized by prepared concentration

All of the participating laboratories used ion chromatography for the determination of SO₄²⁻ except for two laboratories (Lab.RU02, KH01), which used Nephelometry and Turbidimetric method.

Results of analysis of SO₄²⁻ by 27 participating laboratories had no flag in both of samples.

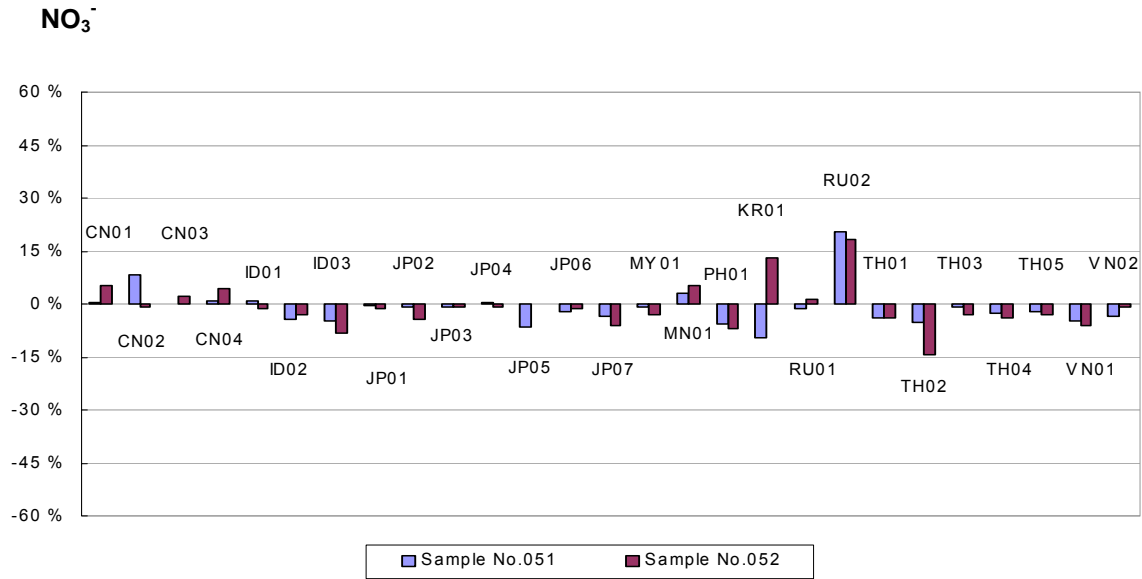


Fig.6 Distribution of NO₃⁻ data normalized by prepared concentration

All of the participating laboratories used ion chromatography for the determination of NO₃⁻ except for one laboratory (Lab.RU02), which used Colorimetry.

The ratio of flagged data in both samples decreased and no data both have “X” flag. The data of the Lab.RU02 (obtained by Colorimetry) were flagged “E” in both samples.

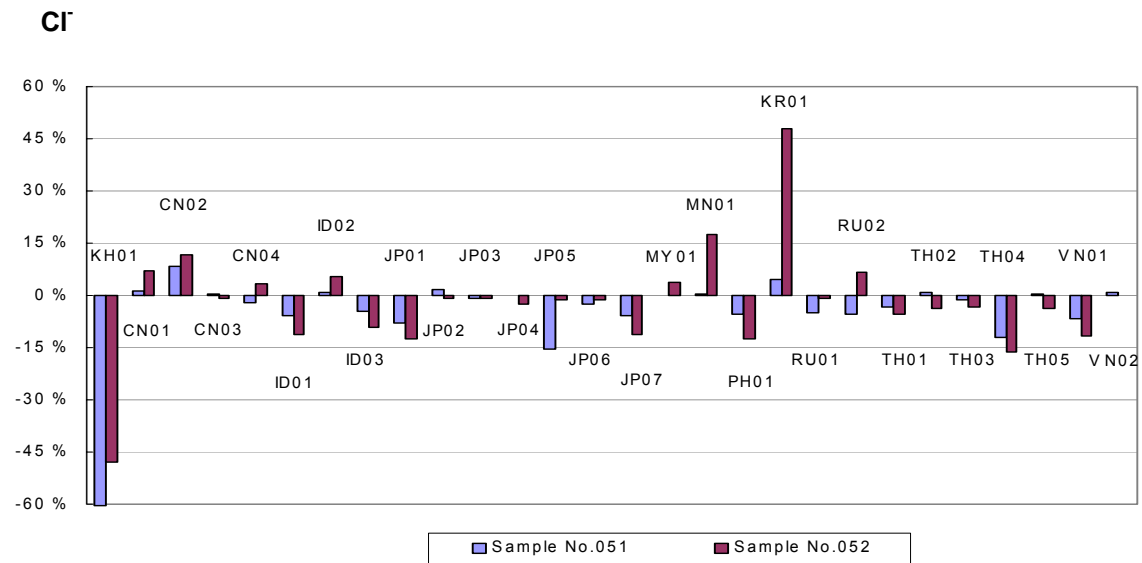


Fig.7 Distribution of Cl⁻ data normalized by prepared concentration

25 laboratories used ion chromatography for the determination of Cl⁻. The Lab.RU02 and KH01 used titration method for the determination of Cl⁻.

Comparing the result of last year, the ratio of flagged data for No.051 decreased in half.

It seemed that contamination from deionized water might affect the data of Lab.MN01 in lower concentration sample analysis.

Na⁺

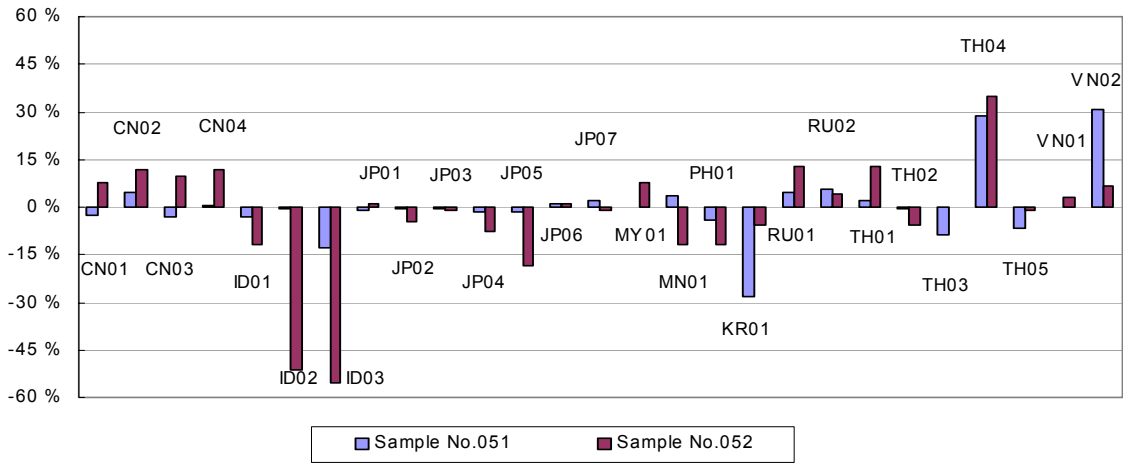


Fig.8 Distribution of Na⁺ data normalized by prepared concentration

22 laboratories used ion chromatography, 3 laboratories used atomic absorption spectrometry (Lab.ID01, ID02, KR01), and 2 Laboratory used flame (emission) photometry (Lab.RU01, RU02) for the determination of Na⁺.

The concentrations of the sample No.051 and No.052 were 0.85 and 0.50 times lower than that of the sample No.041 and No.042 (2004), respectively. Lab.ID03 has flagged data for lower sample. The concentration of the lowest standard solution (std01) is higher than the value of lower concentration sample. It seemed that there was caused by the problem in drawing calculation curve.

K⁺

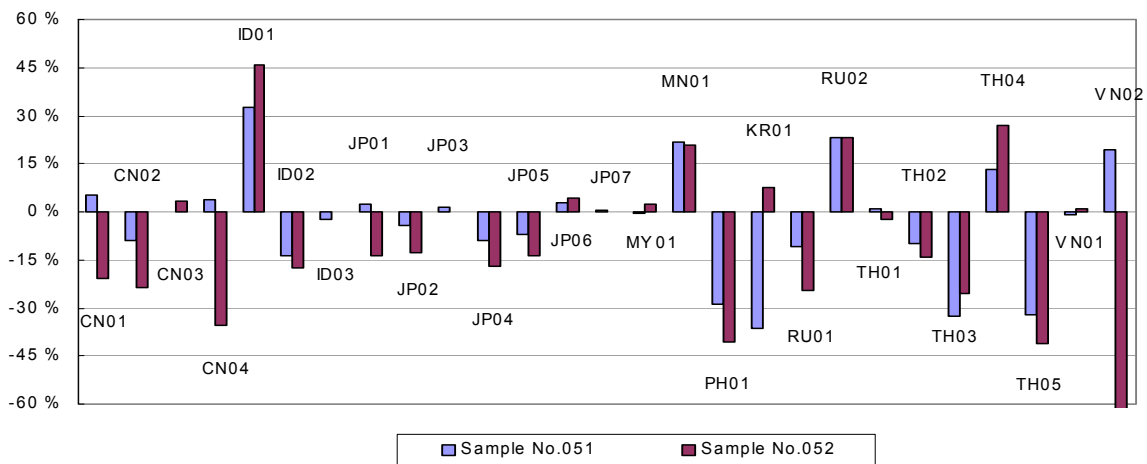


Fig.9 Distribution of K⁺ data normalized by prepared concentration

22 laboratories used ion chromatography, 3 laboratories used atomic absorption spectrometry and 2 laboratories used flame (emission) photometry, for the determination of K⁺.

The ratio of flagged data in the sample of lower concentration is around 2 times higher than the one of higher concentration. Comparing with the 2004 project, the flagged ratio for higher concentration sample increased up to 5 times.

There are 7 laboratories with flagged data for both samples.

The data of Lab.ID01 exceeded the prepared value of 30% higher for both samples.

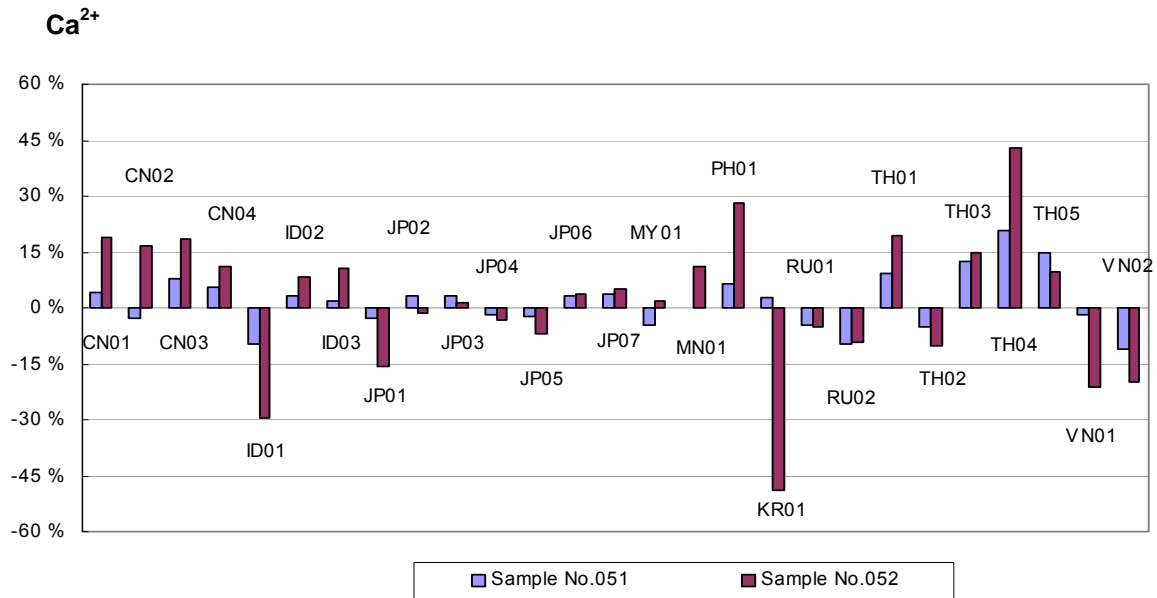


Fig.10 Distribution of Ca²⁺ data normalized by prepared concentration

22 laboratories used ion chromatography, 5 laboratories used atomic absorption spectrometry for the determination of Ca²⁺. The almost half of data measured by ion chromatography were flagged.

Comparing with 2004 project, flagged ratio decreased in higher concentration sample and increased in lower concentration samples. There are large differences in the flagged ratios between the higher and lower concentration samples. The flagged ratio of No.052 was 10 times higher than the one of No.051. Normalized percentages by prepared value tended to be in same directions in each laboratory.

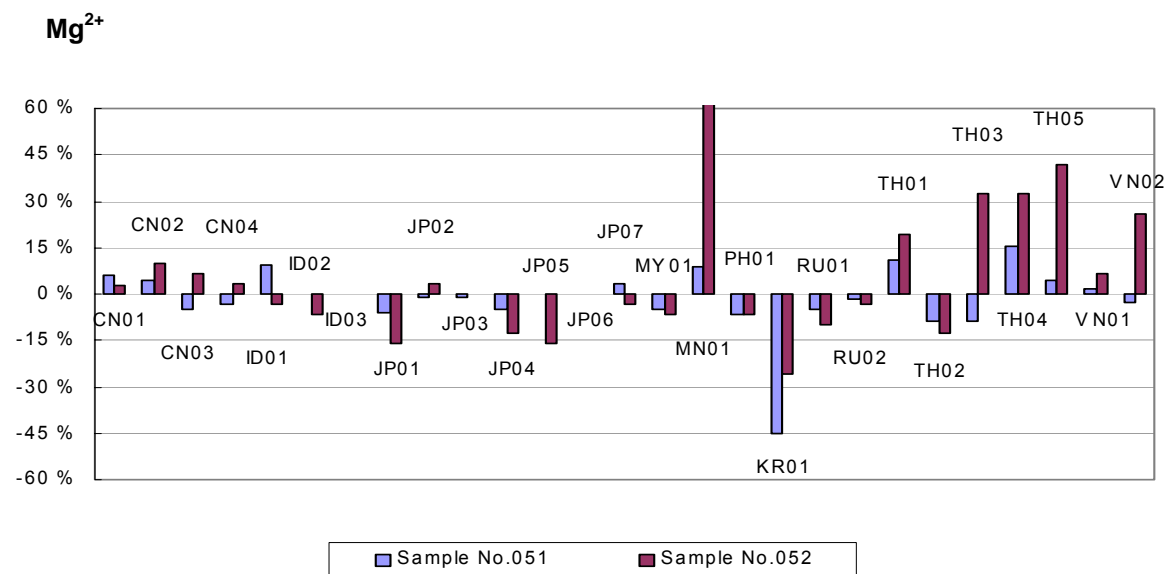


Fig.11 Distribution of Mg²⁺ data normalized by prepared concentration

Ion chromatography and atomic absorption spectrometry were used in the analysis of Mg^{2+} same as for Ca^{2+} .

As same as Ca^{2+} , there are large differences in flagged ratios between No.051 and No.052. All data with flag "X" had positive analytical errors.

The ratio of flagged data in sample No.052 increased comparing with last project in spite of the concentration of sample No.052 ($3.1\mu mol/L$) was slightly higher than for sample No.042 ($2.7\mu mol/L$) of last year project.

Reported value from Lab.MN01 was approximately 1.5 times higher than the prepared value for No.051 samples.

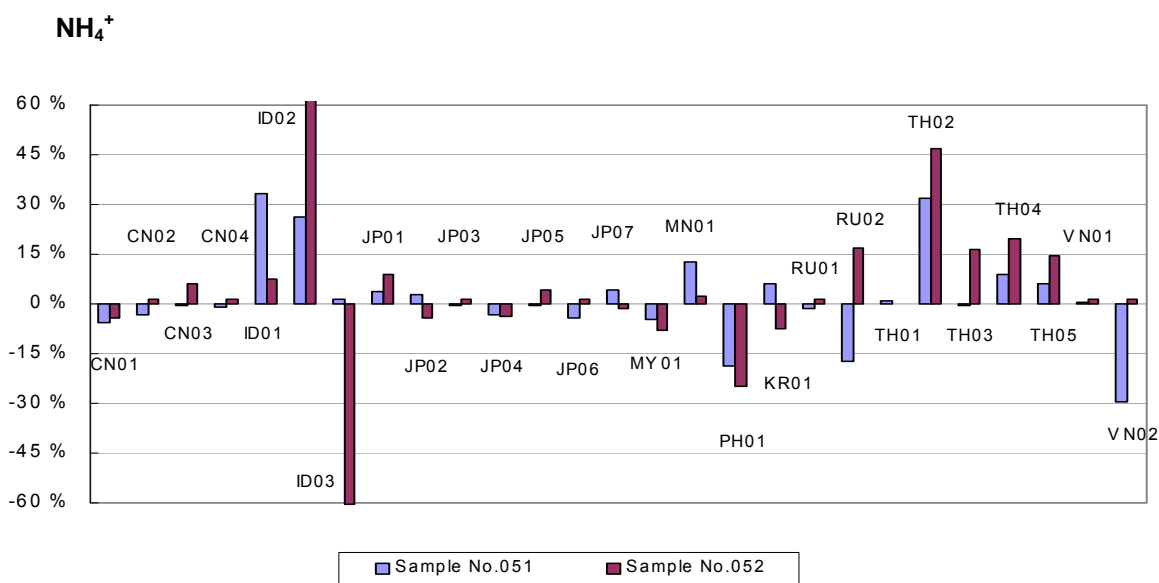


Fig.12 Distribution of NH_4^+ data normalized by prepared concentration

25 laboratories used recommended analytical method of EANET for the determination of NH_4^+ : 22 laboratories used ion chromatography; 3 laboratories (Lab.ID02, PH01, KR01) used spectrometry (Indophenol blue); 2 laboratories used Colorimetry. (Lab.RU01, RU02)

While the concentrations were almost same as of last project, the flagged ratios were calculated as higher than for last project in both samples.

Overall Evaluation

The prepared concentrations of ions in the samples No.051 were approximately at the same level as the sample of project 2004 in the range of 0.60 to 1.20 times of 2004 values. The concentrations of ions in the sample No.052 were in the range of 0.50 to 1.14 times of the project 2004.

The relative standard deviations (R.S.D) of the sample No.051 and No.052 are shown in the figure 13. Whereas the R.S.D of anions except Cl^- in both samples was lower than the project 2004, those of cations except Ca^{2+} and Mg^{2+} were higher comparing with the project 2004.

Comparing the results by DQOs with that of last project, the flagged ratios of the sample No.041 (2004) and the sample No.051 (with higher concentration) were 10.7% and 9.4%, respectively. As for the lower concentration sample, the flagged ratio of flagged data in 2004

and 2005 were 13.0% and 19.6%, respectively. The number of flagged data of anions for higher concentration sample decreased. On the other hand, the number of flagged data of cations increased though the concentrations of prepared value were almost same as previous one. And the results of Ca^{2+} for higher concentration sample were flagged only one data.

There are some laboratories having problems about drawing calibration curve in the determination of the ions and the measurement of pH and EC in this project as same as past project. The person in charge of analysis should confirm the more suitable calibration curve drawn on the chart along technical manual. And the reliability of the calibration should be examined before the analysis of the rain samples by using the working standard to avoid the acquisition of low-trust data.

(Relative standard deviation (%) = (Standard deviation / Average) x100; Reported data after removing the outliers)

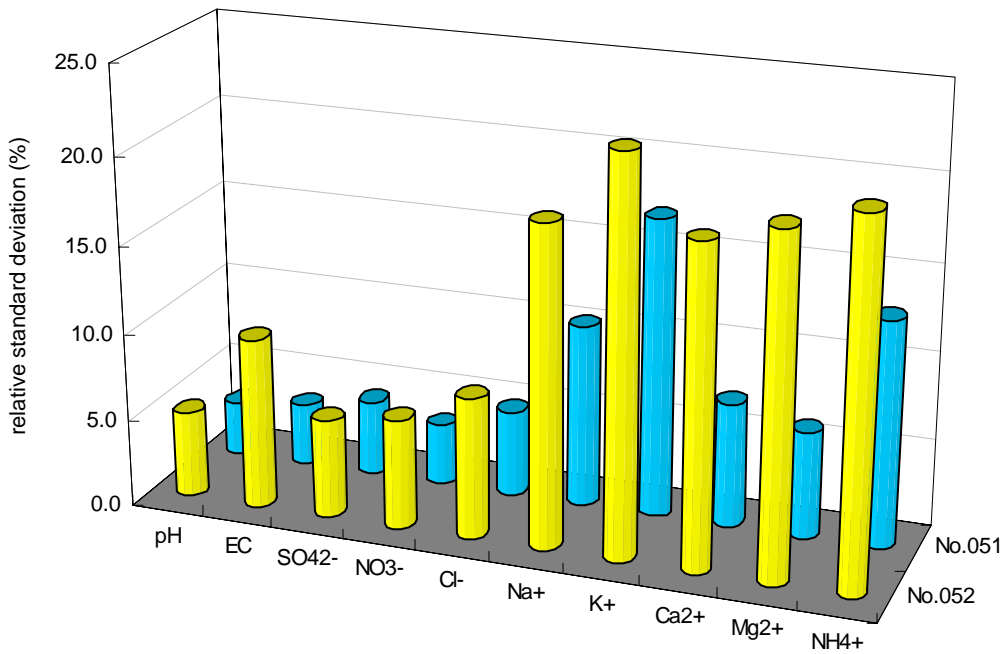


Fig.13 Relative standard deviation of each constituent data

3.3 Circumstances of Sample Analysis

Methods Used

As shown in Fig.14, the most of participating laboratories used recommended methods of EANET. Regarding the determination of anions, 25 laboratories used ion chromatography. One laboratory used Nephelometry, Colorimetry, and Titration in the determination of SO_4^{2-} , NO_3^- and Cl^- , respectively. And another laboratory used Turbidimetric method and Titration in the determination of SO_4^{2-} and Cl^- , respectively.

As for determination of the cations, 22 of 27 laboratories used ion chromatography. 3 (Na^+ , K^+) and 5 (Ca^{2+} , Mg^{2+}) laboratories used Atomic Absorption Spectrometry. Two laboratories used Emission Spectrometry (Na^+ , K^+). Regarding the NH_4^+ , three laboratories used Indophenol Spectrophotometry, and two laboratories used the Colorimetry. The relationship between used analytical methods and flagged data couldn't be detailed because data of no recommended methods were too few.

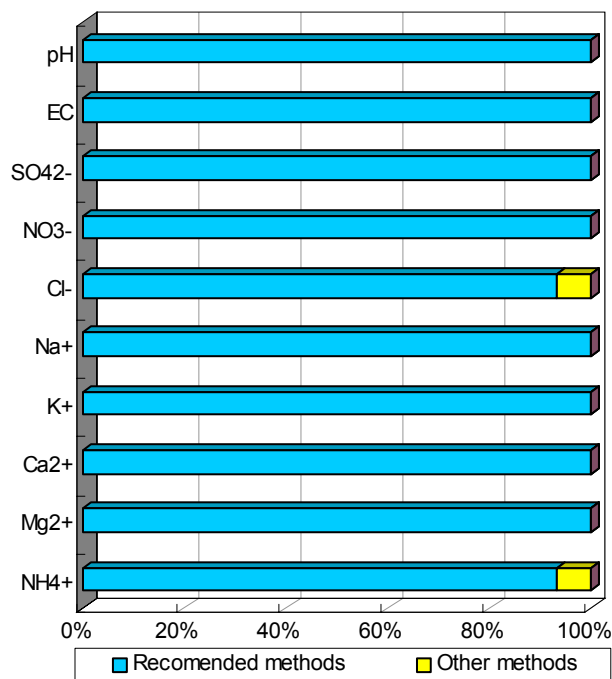


Fig.14 Ratio of recommended method used in the project

Table 11 List of methods

Code	Method
0	pH meter with electrode
1	Conductivity cell
2	Titration
3	Atomic Absorption Spectrometry
4	Emission Spectrometry
5	Ion chromatography
6	Inductively Coupled Plasma - Atomic Emission Spectrometry (ICP - AES)
7	Spectrophotometry
8	Indophenol Spectrophotometry (NH ₄ ⁺)
9	Inductively Coupled Plasma - Mass Spectrometry (ICP - MS)
10	Graphite Furnace Atomic Absorption spectrometry (GFAA)
11	Other method

Table 12 Number of laboratories used different analytical method

SampleNo.051

Method	pH	EC	SO ₄ ²⁻	NO ₃ ⁻	Cl ⁻	Na ⁺	K ⁺	Ca ²⁺	Mg ²⁺	NH ₄ ⁺
0	30(1)									
1		30(1)								
2					2(1)					
3						3(1)	3(2)	5	5(1)	
4						2	2(1)			
5			26	26	26(1)	22(2)	22(5)	22(1)	22(1)	22(3)
6										
7			2(1)	1(1)						2(1)
8										3(2)
9										
10										
11										
Flagged E	0	1	0	1	1	2	4	1	1	4
Flagged X	1	0	1	0	1	1	4	0	1	2

Sample No.052

Method	pH	EC	SO ₄ ²⁻	NO ₃ ⁻	Cl ⁻	Na ⁺	K ⁺	Ca ²⁺	Mg ²⁺	NH ₄ ⁺
0	30(1)									
1		30(2)								
2					2(1)					
3						3(1)	3(2)	5(2)	5(1)	
4						2	2(2)			
5			26	26	26(3)	22(3)	22(10)	22(9)	22(8)	22(5)
6										
7			2(1)	1(1)						2(1)
8										3(1)
9										
10										
11										
Flagged E	1	0	0	1	2	1	9	8	5	4
Flagged X	0	2	1	0	2	3	5	3	4	3

Reverse mesh is recommended method of EANET (): Number of data flagged by "E" or "X"

Number of method is shown in Table 11

Number of staff in charge of measurement

The number of staff in charge of measurement on rainwater samples is described in Table 13. In 18 laboratories only one person carried out measurement of rainwater samples. In 9 laboratories two persons carried it. In 3 laboratories three persons carried it. There was no laboratory where more than 4 people carried out measurement.

In the laboratories that 3 persons carried out measurement, their responsibilities were separated according to the methods used for analysis such as pH-EC-NH₄⁺, anions and cations (PH01, RU01), pH-EC, anions and cations (MY01).

Relationship between staff number and flagged data couldn't be identified.

Table 13 Staff in charge of measurement

Lab.ID	Total	pH	EC	SO ₄ ²⁻	NO ₃ ⁻	Cl ⁻	Na ⁺	K ⁺	Ca ²⁺	Mg ²⁺	NH ₄ ⁺
KH01	1	A	A	A		A					
CN01	1	A	A	A	A	A	A	A	A	A	A
CN02	2	A	A	B	B	B	B	B	B	B	B
CN03	1	A	A	A	A	A	A	A	A	A	A
CN04	1	A	A	A	A	A	A	A	A	A	A
ID01	2	A	A	B	B	B	A	A	A	A	B
ID02	2	A	A	A	A	A	B	B	B	B	B
ID03	1	A	A	A	A	A	A	A	A	A	A
JP01	1	A	A	A	A	A	A	A	A	A	A
JP02	1	A	A	A	A	A	A	A	A	A	A
JP03	1	A	A	A	A	A	A	A	A	A	A
JP04	1	A	A	A	A	A	A	A	A	A	A
JP05	1	A	A	A	A	A	A	A	A	A	A
JP06	1	A	A	A	A	A	A	A	A	A	A
JP07	1	A	A	A	A	A	A	A	A	A	A
LA01	1	A	A								
MY01	3	A	A	B	B	B	C	C	C	C	C
MN01	2	A	B	B	B	B	A	A	A	A	A
PH01	3	A	A	B	B	B	C	C	C	C	A
KR01	1	A	A	A	A	A	A	A	A	A	A
RU01	3	A	A	B	B	B	C	C	C	C	A
RU02	2	A	A	B	B	B	B	B	B	B	B
TH01	2	A	B	B	B	B	A	A	A	A	A
TH02	1	A	A	A	A	A	A	A	A	A	A
TH03	1	A	A	A	A	A	A	A	A	A	A
TH04	1	A	A	A	A	A	A	A	A	A	A
TH05	2	A	A	B	B	B	B	B	B	B	B
VN01	2	A	A	B	B	B	A	A	A	A	A
VN02	1	A	A	A	A	A	A	A	A	A	A
MM01	2	A	B								

"A", "B", and "C" represent individuals of staff in each laboratory who are in charge of measurement.

Reverse mesh: Flagged data of "E" or "X" in sample No.051 and/or sample No.052.

Reverse mesh with dark are flagged data of both sample No.051 and No.052

Years of experience (Acid rain)

According to information obtained through this project, clear evidence of data quality improvement was not found in terms of “years of experience of the staff”. (Table 14)

The average years of the experience along each analysis were in the range from 4.90 (EC) to 6.37 (Na⁺ - Mg²⁺). The average in 2004 project was the range from 5.98 to 6.98. The reason why the average in this year was lower than that in last project was that the person in charge in 8/30 laboratories (ID03, JP02, JP06, JP07, MY01, TH03, LA01, MM01) was the their first experience for analysis of acid rain in this project.

Table 14 Years of experience

Lab.ID	pH	EC	SO ₄ ²⁻	NO ₃ ⁻	Cl ⁻	Na ⁺	K ⁺	Ca ²⁺	Mg ²⁺	NH ₄ ⁺
KH01	4	4	4		4					
CN01	14	14	14	14	14	14	14	14	14	14
CN02	7	7	4	4	4	4	4	4	4	4
CN03	7	7	7	7	7	7	7	7	7	7
CN04	10	10	10	10	10	10	10	10	10	10
ID01	6	6	6	6	6	6	6	6	6	6
ID02	2	2	2	2	2	2	2	2	2	2
ID03	1	1	1	1	1	1	1	1	1	1
JP01	21	21	21	21	21	21	21	21	21	21
JP02	1	1	1	1	1	1	1	1	1	1
JP03	3	3	3	3	3	3	3	3	3	3
JP04	3	3	3	3	3	3	3	3	3	3
JP05	2	2	2	2	2	2	2	2	2	2
JP06	1	1	1	1	1	1	1	1	1	1
JP07	1	1	1	1	1	1	1	1	1	1
LA01	1	1								
MY01	1	1	1	1	1	1	1	1	1	1
MN01	8	8	8	8	8	8	8	8	8	8
PH01	5	5	4.5	4.5	4.5	9	9	9	9	5
KR01	6	6	6	6	6	6	6	6	6	6
RU01	8	8	8	8	8	8	8	8	8	8
RU02	2	2	33	33	33	33	33	33	33	33
TH01	8	3	3	3	3	8	8	8	8	8
TH02	9	9	9	9	9	9	9	9	9	9
TH03	1	1	1	1	1	1	1	1	1	1
TH04	4	4	4	4	4	4	4	4	4	4
TH05	3	3	3	3	3	3	3	3	3	3
VN01	11	11	20	20	20	11	11	11	11	11
VN02	2	2	2	2	2	2	2	2	2	2
MM01	1	1								

Unit: year

Reverse mesh: Flagged data of “E” or “X” in sample No.051 and/or sample No.052

Reverse mesh with dark are flagged date of both sample No.051 and No.052

1 year means experience with one year or less

The number of flagged data in laboratories.

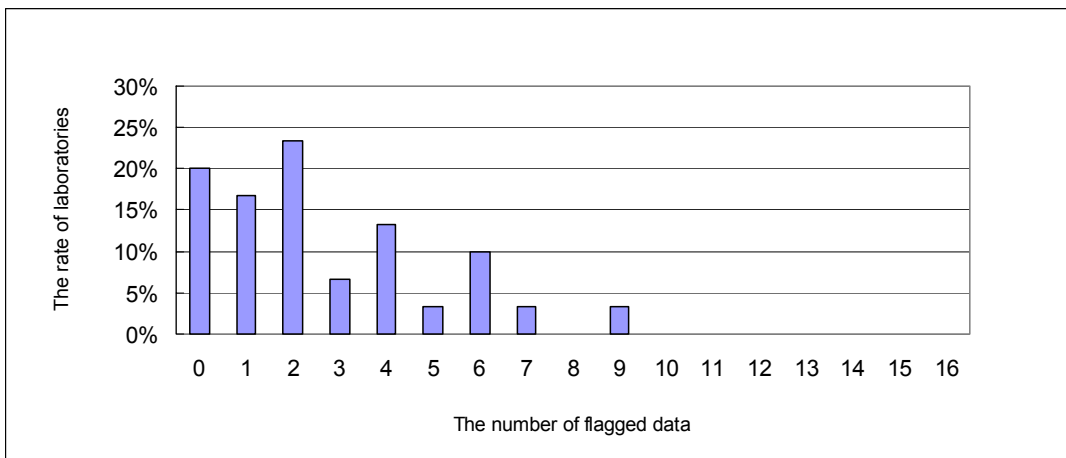


Fig.15 The distribution of laboratories with the number of flagged data

Table 15 Number of flagged data in each laboratory.

Number of flagged data	Number of laboratories	Share
0	6	20%
1	5	17%
2	7	23%
3	2	7%
4	4	13%
5	1	3%
6	3	10%
7	1	3%
8	0	0%
9	1	3%
10	0	0%
11	0	0%
12	0	0%
13	0	0%
14	0	0%
15	0	0%
16	0	0%

In this project, the total number of flagged data was 80(E46, X34) among the whole set of 554 data. The attribution of flagged data in each laboratory was presented in Table 15.

The number of laboratories without flagged data was 6, which was equivalent to about 20% of the all-participating laboratories. Comparing with last project, the number of laboratories with best analytical results was decreased. On the other hand, there was one laboratory that had 9 flagged data.

Water temperature at measurement (pH and EC)

As described in Table 16, most of the participating laboratories measured pH and EC at temperature around 25 °C as condition recommended by EANET. There were no flagged data in the measurement of pH and EC except some 3 laboratories. Regulated measurement condition may be one of the possible causes of the excellent results

Table 16 Water temperature at measurement (pH and EC)

lab.ID	pH		EC	
	No.051	No.052	No.051	No.052
KH01	25	25	25	25
CN01	25	25	25	25
CN02	18	18	18	18
CN03	25.0	25.0	25.0	25.0
CN04	24.6	25.3	25.1	24.8
ID01	25	25	25	25
ID02	25.0	25.0	25.0	25.0
ID03	25	25	25	25
JP01	24.4-25.1	24.6-25.1	24.4-25.1	24.6-25.1
JP02	25.0	25.0	25.0	25.0
JP03	25.0	25.0	25.0	25.0
JP04	25.0	24.3	25.0	24.3
JP05	25.0	25.0	25.0	25.0
JP06	25.0	25.0	25.0	25.0
JP07	25.0	25.0	25.0	25.0
LA01	25	25	25	25
MY01	25	25	25	25
MN01	25	25	25	25
PH01	25	25	25	25
KR01	25	25	25	25
RU01	25	25	25	25
RU02	25	25	25	25
TH01	25	25	25	25
TH02	25	25	25	25
TH03	25	25	25	25
TH04	25	25	25	25
TH05	25	25	25	25
VN01	25	25	25	25
VN02	24	23	24	23
MM01	23	25	23	25

Unit: degrees centigrade

Reverse mesh with light are flagged data of "E"

Reverse mesh with dark are flagged date of "X"

4. COMPARISON OF 1st, 2nd, 3rd, 4th, 5th, 6th, 7th AND 8th SURVEY

The inter-laboratory comparison surveys were carried out 8 times, so far their results with the rations of flagged data are shown in Fig. 16. The rate of data that satisfied the required quality objectives (DQOs) increased from 75-78% to 84-93% until the 4th (2001) survey. The data quality seemed to be improved by accumulating experiences. But on the 5th project (2002), the satisfied ratios of DQOs in the higher concentration sample and the lower concentration sample decreased because the ion concentrations were set lower than that of previous one. The satisfied rate of DQOs increased from the 6th (2003) survey to the 7th (2004) survey newly.

In the higher concentration sample (correspond to the sample No.051 of 8th project), the number of data within DQOs increased for 8th project. On the other hand, the number of data within DQOs of the lower concentration sample (correspond to the sample No.052 for 8th project) decreased.

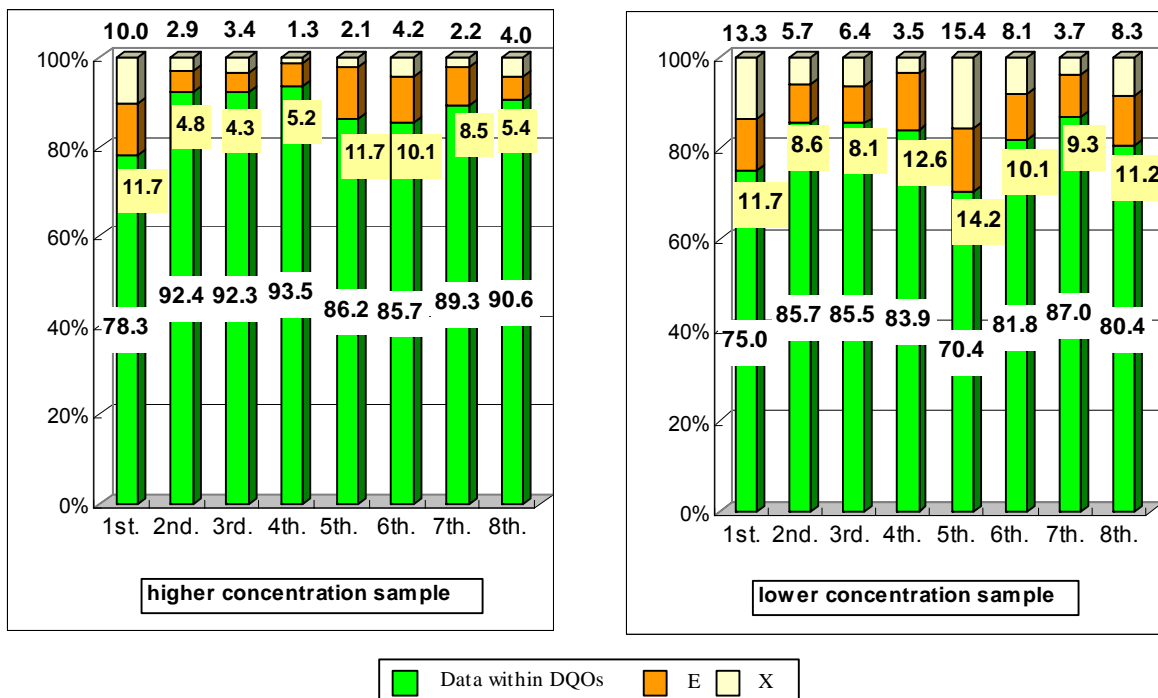


Fig. 16 Comparison of 1st, 2nd, 3rd, 4th, 5th, 6th, 7th and 8th inter-laboratory comparison project

The comparison for each parameter in inter-laboratory comparison project on wet deposition year by year shows in Fig.17. And the prepared values of each parameter in artificial rainwater of inter-laboratory comparison project of EANET are described in Table17.

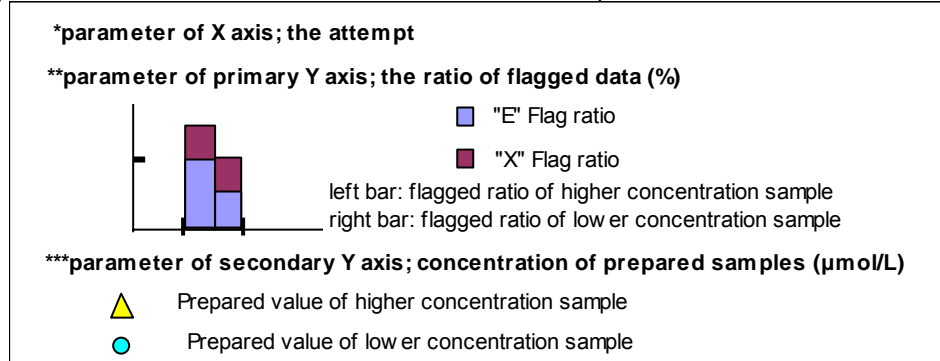
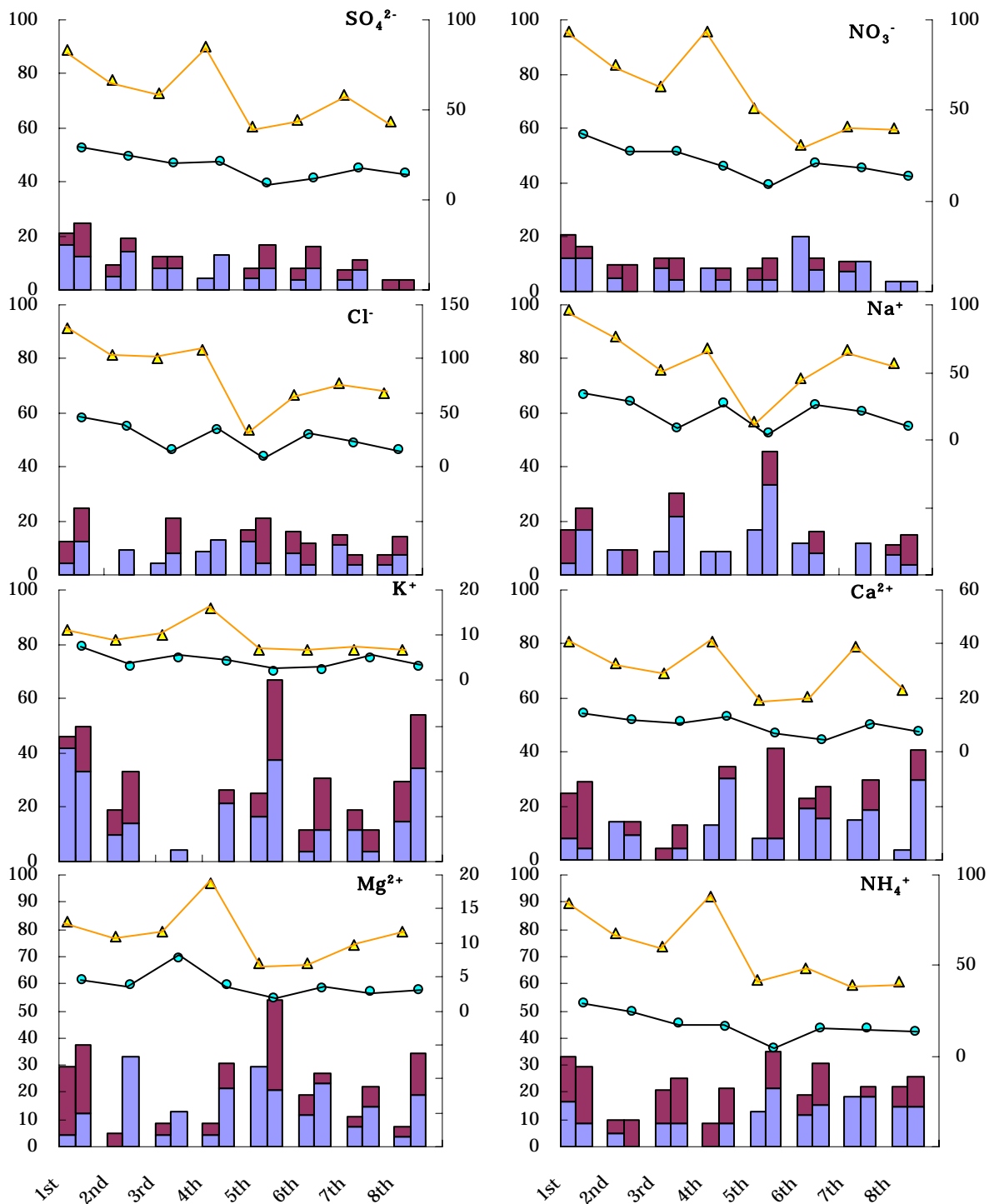


Fig.17 Comparison for each parameter in inter-laboratory comparison project

Table 17 The prepared values of each parameter in artificial rainwater of inter-laboratory comparison projects of EANET

		pH (-)	EC (mS/m)	SO ₄ ²⁻ (μmol/L)	NO ₃ ⁻ (μmol/L)	Cl ⁻ (μmol/L)	Na ⁺ (μmol/L)	K ⁺ (μmol/L)	Ca ²⁺ (μmol/L)	Mg ²⁺ (μmol/L)	NH ₄ ⁺ (μmol/L)
1998	No.1	4.05	7.94	83.5	93.3	129	95.8	11.1	41.1	13.1	84.8
	No.2	4.51	2.82	29.1	36.1	45.1	33.5	7.42	14.3	4.6	29.5
1999	No.1	4.14	6.38	67	75.0	104	77.0	8.9	33.0	11.0	68.0
	No.2	4.59	2.30	24.0	27.0	38.0	28.0	3.2	12.0	3.8	25.0
2000	No.1	4.10	6.23	59.7	63.3	101.3	51.3	9.9	29.4	11.7	60.5
	No.2	4.85	1.55	20.1	27.5	15.5	8.7	4.9	11.0	7.8	18.2
2001	No.11	4.10	7.45	85.0	93.3	108.4	68.4	15.8	41.1	18.7	87.8
	No.12	4.82	1.76	21.5	19.4	34.4	27.4	4.00	13.2	3.7	16.7
2002	No.021	4.30	3.75	40.3	51.0	33.7	13.7	6.92	19.1	7.02	42.4
	No.022	5.15	0.69	8.88	8.49	9.13	5.13	1.98	6.6	1.75	4.54
2003	No.031	4.52	3.44	44.7	30.9	66.0	46.1	6.9	20.5	7.0	48.3
	No.032	4.80	1.48	12.0	21.3	29.6	25.6	2.5	4.4	3.4	15.1
2004	No.041	4.60	3.94	58.6	41.4	76.7	66.7	6.9	38.9	9.8	39.4
	No.042	5.00	1.33	17.6	18.4	22.5	20.5	5.0	10.0	2.7	15.1
2005	No.051	4.66	3.32	43.7	40.3	68.5	56.5	6.9	23.2	11.7	40.9
	No.052	5.05	1.05	14.4	13.2	15.3	10.3	3.0	7.6	3.1	13.6

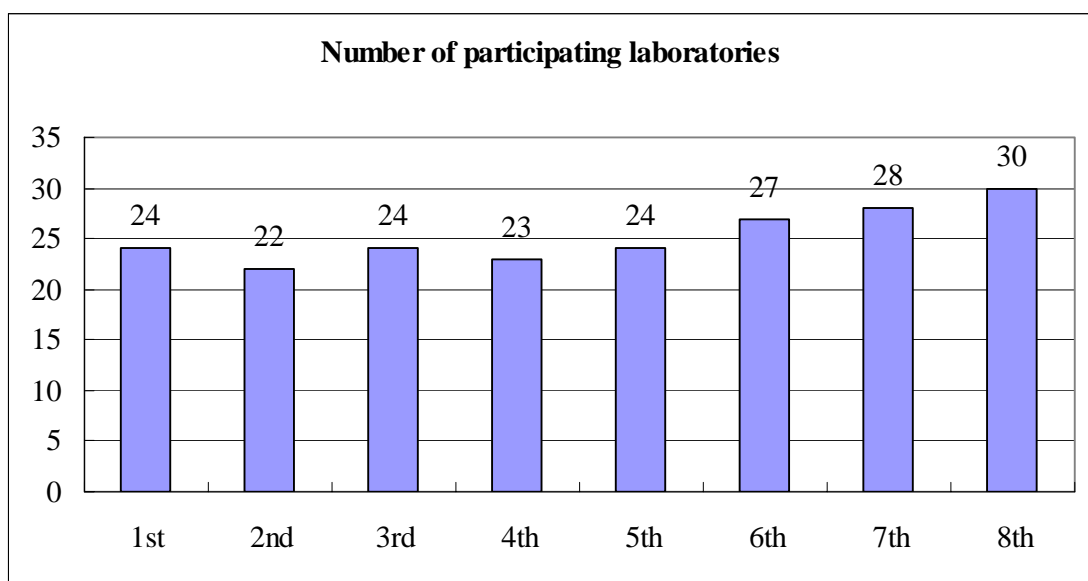


Fig. 18 The number of participating laboratories in the Inter-laboratory comparison project on wet deposition

Cambodia and Lao PDR participated from 6th Inter-laboratory comparison project on wet deposition. And Myanmar participated this survey from project 2005.

5. RECOMENDATION FOR IMPROVEMENT OF MEASUREMENT PRECISIONS

The following fundamental matters should be taken into account in measurement, analysis, and data control processes.

5.1 Fundamental measurement and analysis matters

Clearance from contamination of the apparatus, materials and reagents used for measurement and analysis must be confirmed beforehand.

- ▶ Blank values of target substances should be as low as possible.
- ▶ Measurement and analysis should be conducted by persons who are well trained.
- ▶ To maintain high analytical quality, **SOPs** must be prepared for the management of apparatus, reagents, and procedure of operation.
- ▶ Other details on measurement and analysis of samples are as follows.

1) Deionized water

- ▶ Water with a conductivity less than 0.15mS/m is acceptable for measurements, analyses, dilution of precipitation samples and cleaning.

2) Certified materials and certified samples

- ▶ The measurements are evaluated by comparison of measured results of samples and certified materials.
- ▶ In order to assure the reliability of measurements, the certified solutions and materials should be used as much as possible.

3) Pretreatment of samples at analytical laboratory

- ▶ Conductivity and pH should be measured as soon as possible after sample receiving, and checking agreement of samples and sample list.
- ▶ Effort should be made to start analysis of the other parameters within a week of sample arrival in the laboratory and to complete the data sets by measuring EC, pH and all other chemical parameters.

4) Adjustment of analytical instruments

- ▶ Each of the analytical instruments must be calibrated when they are used, and they should be adjusted as appropriate.

5.2 Evaluation of reliability

1) Sensitivity fluctuation of analytical instruments

While numerous samples are measured, measurements should be continued after confirming that the sensitivity fluctuation is within the prescribed range.

a) For example, Ion chromatography

- ▶ A new calibration should be performed not more than 30-sample measurements.
- ▶ Reference materials should be measured after the calibration. It should also be done once or twice before the next calibration.
- ▶ Control charts should be applied for the measurement of the reference materials.
- ▶ Standard solutions and reference solutions must be prepared from different stock solutions in order to be independent.
- ▶ If the results of the control solutions are outside of 3 standard deviations, or out of 15 % from the expected value, the reasons should be found and corrections will be made, and reference solution will be measured again.
- ▶ If the retention time changes slowly while the separator column is deteriorating, then

adequate actions could be taken as appropriate. If it changes significantly in a relatively short time, the reasons should be found and removed, then the reference material must be measured again.

5.3 Data control

1) Data check in analysis organizations

- ▶ When the sensitivity of instruments is not stable, when the results of duplicate analyses or re-measurements are significantly different, or when the ratio of a theoretical value to that for determined data in ion balances and electrical conductivity is significantly different from 1.0, measurement should be repeated since reliability is low.
- ▶ When samples seem to be obviously contaminated, these data should be treated as unrecorded data.
- ▶ Abnormal or unrecorded data can corrupt research results. So, careful checks are needed to avoid data of inadequate quality. When abnormal or unrecorded data appear, the process should be carefully reviewed to prevent the occurrence of the same problem in the future.

6. REFERENCES

- 1) Guidelines for Acid Deposition Monitoring in East Asia
March 2000 adopted at:
The Second Interim Scientific Advisory Group Meeting of Acid Deposition Monitoring Network in East Asia.
- 2) Technical Documents for Wet Deposition Monitoring in East Asia
 - Technical Manual for Wet Deposition Monitoring in East Asia
 - Quality Assurance / Quality Control (QA/QC) Program for the Preparatory-Phase Wet Deposition Monitoring in East Asia, March 2000 adopted at:
The Second Interim Scientific Advisory Group Meeting of Acid Deposition Monitoring Network in East Asia.
- 3) Report of the Inter-laboratory Comparison Project 1998
(Round robin analysis survey 1st Attempt) November 1999.
- 4) Report of the Inter-laboratory Comparison Project 1999
(Round robin analysis survey 2nd Attempt) October 2000
- 5) Report of the Inter-laboratory Comparison Project 2000
(Round robin analysis survey 3rd Attempt) October 2001
- 6) Report of the Inter-laboratory Comparison Project 2001
(Round robin analysis survey 4th Attempt) November 2002
- 7) Report of the Inter-laboratory Comparison Project 2002
(Round robin analysis survey 5th. Attempt) November 2003
- 8) Report of the Inter-laboratory Comparison Project 2003
(Round robin analysis survey 6th. Attempt) November 2003
- 9) Report of the Inter-laboratory Comparison Project 2004
(Round robin analysis survey 7th Attempt) November 2005

7. CONTACT INFORMATION

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Dr. Tsuyoshi OHIZUMI	Dept. Head, Atmospheric Research Department E-mail: ohizumi@adorc.gr.jp

APPENDIX 1 Participating laboratories

<u>Countries/Laboratories</u>	<u>Code</u>
<u>1. Cambodia</u>	
1) Department of Pollution Control, Ministry of Environment	(KH01)
<u>2. CHINA</u>	
2) Zhuhai Environmental Monitoring Station	(CN01)
3) Environmental Monitoring Station of Xiamen	(CN02)
4) Xi'an Environmental Monitoring Station	(CN03)
5) Chongqing Institute of Environmental Science	(CN04)
<u>3. INDONESIA</u>	
6) Analysis Division, Meteorological and Geophysical Agency (BMG)	(ID01)
7) Center for Environmental Impact Control Facilities (PUSARPEDAL) Environmental Impact Management Agency (BAPEDAL)	(ID02)
8) Indonesian National Institute of Aeronautic and Space (Lapan)	(ID03)
<u>4. JAPAN</u>	
9) Hokkaido Institute of Environmental Sciences	(JP01)
10) Nagano Research Institute for Health and Pollution	(JP02)
11) Gifu Prefectural research Institute of health and Environmental Science	(JP03)
12) Kochi Prefectural Environmental Research Center	(JP04)
13) Shimane Prefectural Institute of Public Health and Environmental Science	(JP05)
14) Okinawa Research Institute of health and Environment	(JP06)
15) Acid Depositon and Oxidant Research Center (ADORC)	(JP07)
<u>5. LAO PDR</u>	
16) Environment Quality Monitoring Center, Environment Research Institute, Science Technology and Environment Agency	(LA01)
<u>6. MALAYSIA</u>	
17) Division of Environmental Health, Department of Environmental Monitoring	(MY01)
<u>7. MONGOLIA</u>	
18) Central Laboratory of Environmental Monitoring, National Agency for Meteorology, Hydrology and Environmental Monitoring, Ministry of Nature and Environment	(MN01)
<u>8. PHILIPPINES</u>	
19) Research and Development Division, Environmental Management Bureau (EMB), Department of Environment and Natural Resources (DENR)	(PH01)

<u>Countries/Laboratories</u>	<u>Code</u>
<u>9. Republic of KOREA</u>	
20) Atmospheric Chemistry Division, National Institute of Environment Research (NIER)	(KR01)
<u>10. RUSSIA</u>	
21) Limnological Institute of the Russian Academy of Sciences/Siberian Branch (RAS/SB)	(RU01)
22) Primorskii Environmental Monitoring Center of Roshydromet (Laboratory for Monitoring of Atmosphere and Soil Pollution)	(RU02)
<u>11. THAILAND</u>	
23) Research and Training Centre (ERTC), Department of Research and Environmental Quality Promotion	(TH01)
24) Pollution Control Department (PCD) Ministry of Natural Resources and Environment (MONRE)	(TH02)
25) School of Energy & Materials, King Mongkut's University of Technology Thonburi	(TH03)
26) Meteorological Observation Division, Thailand Meteorological Department (TMD)	(TH04)
27) Chemistry Department, Science Faculty, Chiangmai University (CMU)	(TH05)
<u>12. VIET NAM</u>	
28) Institute of Meteorology and Hydrology (IMH), Hydrometeorological Service of Viet Nam (HMS)	(VN01)
29) Middle of Central regional Hydro-Meteorological Observatory National Hydro-Meteorological Center (NHMS)	(VN02)
<u>13. Myanmar</u>	
30) Department of meteorology and Hydrology (DMH)	(MM01)
<u>Other laboratories</u>	
<u>(Some laboratories in India participated in this survey. Results are shown APPENDIX 2-3)</u>	
31) Indian Institute of Chemical Technology (IICT)	(IN01)
32) National Physical Laboratory (NPL)	(IN02)

APPENDIX 2 Original data

Sample No.051 (higher concentrations)

	pH	EC	SO ₄ ²⁻	NO ₃ ⁻	Cl ⁻	Na ⁺	K ⁺	Ca ²⁺	Mg ²⁺	NH ₄ ⁺
	-	mS/m	μmol/L	μmol/L	μmol/L	μmol/L	μmol/L	μmol/L	μmol/L	μmol/L
KH01	6.79	2.89	7.1		27.0					
CN01	4.70	3.24	42.3	40.4	69.3	55.0	7.3	24.2	12.4	38.6
CN02	4.63	3.28	45.0	43.7	74.1	59.1	6.3	22.6	12.2	39.5
CN03	4.70	3.30	43.2	40.3	68.9	54.8	6.9	25.0	11.1	40.8
CN04	4.70	3.20	43.0	40.7	67.0	56.8	7.2	24.5	11.3	40.6
ID01	4.16	3.07	43.2	40.6	64.6	54.7	9.1	21.0	12.8	54.5
ID02	4.74	3.14	45.8	38.6	69.1	56.1	6.0	23.9	11.7	51.6
ID03	4.52	3.00	41.9	38.4	65.5	49.4	6.7	23.6	11.7	41.5
JP01	4.71	3.26	42.6	40.2	63.2	56.0	7.1	22.6	11.0	42.4
JP02	4.68	3.35	42.6	40.0	69.6	56.1	6.6	23.9	11.6	42.1
JP03	4.74	3.22	43.5	39.9	67.8	56.1	7.0	23.9	11.6	40.7
JP04	4.69	3.24	43.9	40.5	68.4	55.6	6.3	22.8	11.1	39.6
JP05	4.73	3.16	39.2	37.6	57.9	55.5	6.4	22.7	11.7	40.8
JP06	4.71	3.23	41.9	39.4	66.8	57.0	7.1	23.9	11.7	39.1
JP07	4.75	3.21	42.9	38.9	64.5	57.6	6.9	24.1	12.1	42.6
LA01	4.37	3.24								
MY01	4.63	2.94	43.3	39.9	68.5	56.5	6.9	22.1	11.1	39.0
MN01	4.74	3.22	45.1	41.6	68.8	58.6	8.4	23.2	12.7	46.1
PH01	4.67	3.25	42.0	38.0	64.7	54.3	4.9	24.7	10.9	33.2
KR01	4.31	3.59	43.5	36.4	71.5	40.5	4.4	23.8	6.4	43.4
RU01	4.62	3.32	43.1	39.7	65.0	59.0	6.1	22.1	11.1	40.4
RU02	4.70	3.20	37.5	48.6	64.9	59.6	8.5	21.0	11.5	33.9
TH01	4.70	3.14	40.9	38.7	66.3	57.7	7.0	25.3	13.0	41.2
TH02	4.71	3.18	41.4	38.2	69.1	56.1	6.2	22.0	10.7	53.9
TH03	4.45	3.18	42.9	39.9	67.6	51.7	4.7	26.1	10.7	40.8
TH04	4.79	3.33	41.0	39.2	60.3	72.6	7.8	28.0	13.5	44.6
TH05	4.82	3.14	42.8	39.5	68.9	52.8	4.7	26.6	12.2	43.4
VN01	4.68	3.26	42.1	38.4	63.8	56.6	6.8	22.8	11.9	41.0
VN02	4.77	3.19	38.3	38.9	69.1	73.8	8.2	20.6	11.4	28.9
MM01	4.67	3.91								
IN01	6.44	3.22	41.0	42.7	98.6	61.2	6.2	23.3	3.9	43.1
IN02	4.54	16.80				53.5	7.0	17.0	11.0	8.1
Prepared value	4.66	3.32	43.7	40.3	68.5	56.5	6.9	23.2	11.7	40.9
Data count	32	32	29	28	29	29	29	29	29	29
Average	4.77	3.65	41.1	40.0	66.6	56.7	6.7	23.4	11.2	40.5
Minimum	4.16	2.89	7.1	36.4	27.0	40.5	4.4	17.0	3.9	8.1
Maximum	6.79	16.80	45.8	48.6	98.6	73.8	9.1	28.0	13.5	54.5
Standard deviation	0.50	2.37	6.68	2.21	9.98	5.80	1.11	2.02	1.82	8.05

Sample No.052 (lowerer concentrations)

	pH	EC	SO ₄ ²⁻	NO ₃ ⁻	Cl ⁻	Na ⁺	K ⁺	Ca ²⁺	Mg ²⁺	NH ₄ ⁺
	-	mS/m	µmol/L	µmol/L	µmol/L	µmol/L	µmol/L	µmol/L	µmol/L	µmol/L
KH01	6.21	1.14	4.4		8.0					
CN01	5.08	1.06	14.2	13.9	16.4	11.1	2.4	9.0	3.2	13.0
CN02	4.99	1.12	13.5	13.1	17.1	11.5	2.3	8.9	3.4	13.8
CN03	5.04	1.05	15.6	13.5	15.2	11.3	3.1	9.0	3.3	14.4
CN04	5.07	0.99	14.5	13.8	15.8	11.5	1.9	8.4	3.2	13.8
ID01	4.91	1.04	13.8	13.0	13.6	9.1	4.4	5.3	3.0	14.6
ID02	5.20	1.01	15.1	12.8	16.1	5.0	2.5	8.2	2.9	23.2
ID03	4.80	0.92	13.2	12.1	13.9	4.6		8.4		5.4
JP01	5.11	1.07	13.7	13.0	13.4	10.4	2.6	6.4	2.6	14.8
JP02	5.00	1.08	14.2	12.6	15.2	9.8	2.6	7.5	3.2	13.0
JP03	5.08	1.05	14.4	13.1	15.2	10.2	3.0	7.7	3.1	13.8
JP04	5.06	1.08	14.3	13.1	14.9	9.5	2.5	7.3	2.7	13.1
JP05	5.14	1.04	14.3	13.2	15.1	8.4	2.6	7.1	2.6	14.2
JP06	5.11	1.03	14.0	13.0	15.1	10.4	3.1	7.9	3.1	13.8
JP07	5.12	1.04	13.7	12.4	13.6	10.2	3.0	8.0	3.0	13.4
LA01	5.47	1.01								
MY01	5.03	0.91	13.7	12.8	15.9	11.1	3.1	7.7	2.9	12.5
MN01	5.05	1.04	15.7	13.9	18.0	9.1	3.6	8.4	5.2	13.9
PH01	5.04	1.08	13.0	12.3	13.4	9.1	1.8	9.7	2.9	10.2
KR01	4.60	1.53	12.4	14.9	22.6	9.7	3.2	3.9	2.3	12.6
RU01	5.00	1.10	14.5	13.4	15.2	11.6	2.3	7.2	2.8	13.8
RU02	5.00	1.11	15.2	15.6	16.3	10.7	3.7	6.9	3.0	15.9
TH01	5.12	0.98	14.2	12.7	14.5	11.6	2.9	9.1	3.7	13.6
TH02	5.23	0.97	12.8	11.3	14.7	9.7	2.6	6.8	2.7	20.0
TH03	5.03	1.04	14.2	12.8	14.8	10.3	2.2	8.7	4.1	15.8
TH04	5.28	1.07	13.4	12.7	12.8	13.9	3.8	10.8	4.1	16.3
TH05	5.11	1.03	14.3	12.8	14.7	10.2	1.8	8.3	4.4	15.6
VN01	5.06	1.06	13.5	12.4	13.5	10.6	3.0	6.0	3.3	13.8
VN02	5.18	1.02	15.2	13.1	15.3	11.0	0.0	6.1	3.9	13.8
MM01	5.32	1.91								
IN01	7.45	1.32	13.8	14.8	18.3	20.1	6.9	10.0	1.8	14.4
IN02	4.87	0.58				9.4	3.3	4.7	2.6	2.6
Prepared value	5.05	1.05	14.4	13.2	15.3	10.3	3.0	7.6	3.1	13.6
Data count	32	32	29	28	29	29	28	29	28	29
Average	5.18	1.08	13.7	13.1	15.1	10.4	2.9	7.7	3.2	13.8
Minimum	4.60	0.58	4.4	11.3	8.0	4.6	0.0	3.9	1.8	2.6
Maximum	7.45	1.91	15.7	15.6	22.6	20.1	6.9	10.8	5.2	23.2
Standard deviation	0.48	0.20	1.92	0.88	2.30	2.55	1.11	1.53	0.69	3.54

APPENDIX 3 Deviation from prepared value

Deviation from prepared value (Va/Vp): {Average (Va) / Prepared value (Vp) - 1} x 100 (%)

Sample No.051 (higher concentration)

Lab. ID	pH	EC	SO ₄ ²⁻	NO ₃ ⁻	Cl ⁻	Na ⁺	K ⁺	Ca ²⁺	Mg ²⁺	NH ₄ ⁺
KH01	45.7	-13.0	-83.8		-60.6					
CN01	0.9	-2.4	-3.2	0.2	1.2	-2.7	5.4	4.3	6.0	-5.6
CN02	-0.6	-1.2	3.0	8.4	8.2	4.6	-9.1	-2.6	4.3	-3.4
CN03	0.9	-0.6	-1.1	0.0	0.6	-3.0	0.0	7.8	-5.1	-0.2
CN04	0.9	-3.6	-1.6	1.0	-2.2	0.5	3.8	5.6	-3.4	-0.7
ID01	-10.7	-7.5	-1.1	0.7	-5.7	-3.2	32.6	-9.5	9.4	33.3
ID02	1.7	-5.4	4.8	-4.2	0.9	-0.7	-13.5	3.0	0.0	26.2
ID03	-3.0	-9.6	-4.1	-4.7	-4.4	-12.6	-2.5	1.7	0.0	1.5
JP01	1.1	-1.8	-2.5	-0.2	-7.7	-0.9	2.5	-2.6	-6.0	3.7
JP02	0.4	0.9	-2.5	-0.7	1.6	-0.7	-4.2	3.0	-0.9	2.9
JP03	1.7	-3.0	-0.5	-1.0	-1.0	-0.7	1.4	3.0	-0.9	-0.5
JP04	0.6	-2.4	0.5	0.5	-0.1	-1.6	-8.8	-1.7	-5.1	-3.2
JP05	1.5	-4.8	-10.3	-6.7	-15.5	-1.8	-7.1	-2.2	0.0	-0.2
JP06	1.1	-2.7	-4.1	-2.2	-2.5	0.9	2.9	3.0	0.0	-4.4
JP07	1.9	-3.3	-1.8	-3.5	-5.8	1.9	0.3	3.9	3.4	4.2
LA01	-6.2	-2.4								
MY01	-0.6	-11.4	-0.9	-1.0	0.0	0.0	-0.6	-4.7	-5.1	-4.6
MN01	1.7	-3.0	3.2	3.2	0.4	3.7	21.7	0.0	8.5	12.7
PH01	0.2	-2.1	-3.9	-5.7	-5.5	-3.9	-28.8	6.5	-6.8	-18.8
KR01	-7.5	8.1	-0.5	-9.7	4.4	-28.3	-36.2	2.6	-45.3	6.1
RU01	-0.9	0.0	-1.4	-1.5	-5.1	4.4	-11.0	-4.7	-5.1	-1.2
RU02	0.9	-3.6	-14.2	20.6	-5.3	5.5	23.2	-9.5	-1.7	-17.1
TH01	0.9	-5.4	-6.4	-4.0	-3.2	2.1	1.0	9.1	11.1	0.7
TH02	1.1	-4.2	-5.3	-5.2	0.9	-0.7	-10.1	-5.2	-8.5	31.8
TH03	-4.5	-4.2	-1.8	-1.0	-1.3	-8.5	-32.6	12.5	-8.5	-0.2
TH04	2.8	0.3	-6.2	-2.7	-12.0	28.5	13.0	20.7	15.4	9.0
TH05	3.4	-5.4	-2.1	-2.0	0.6	-6.5	-32.3	14.7	4.3	6.1
VN01	0.4	-1.8	-3.7	-4.7	-6.9	0.2	-1.0	-1.7	1.7	0.2
VN02	2.4	-3.9	-12.4	-3.5	0.9	30.6	19.3	-11.2	-2.6	-29.3
MM01	0.2	17.8								
IN01	38.2	-3.0	-6.1	5.9	43.9	8.2	-10.0	0.4	-66.4	5.4
IN02	-2.6	406.0				-5.3	1.4	-26.8	-5.6	-80.3
Minimum	-10.7	-13.0	-83.8	-9.7	-60.6	-28.3	-36.2	-26.8	-66.4	-80.3
Maximum	45.7	406.0	4.8	20.6	43.9	30.6	32.6	20.7	15.4	33.3
Average	2.3	10.0	-5.9	-0.8	-2.8	0.4	-2.7	0.7	-3.9	-0.9

Sample No.052 (lower concentration)

Lab. ID	pH	EC	SO ₄ ²⁻	NO ₃ ⁻	Cl ⁻	Na ⁺	K ⁺	Ca ²⁺	Mg ²⁺	NH ₄ ⁺
KH01	23.0	8.6	-69.4		-47.7					
CN01	0.6	1.0	-1.4	5.3	7.2	7.8	-21.0	18.9	2.9	-4.4
CN02	-1.2	6.7	-6.3	-0.8	11.8	11.7	-23.7	16.7	9.7	1.5
CN03	-0.2	0.0	8.3	2.3	-0.7	9.7	3.3	18.6	6.5	5.9
CN04	0.4	-5.7	0.7	4.5	3.3	11.7	-35.7	11.1	3.2	1.5
ID01	-2.8	-1.0	-4.2	-1.5	-11.1	-11.7	46.0	-29.7	-3.2	7.4
ID02	3.0	-3.8	4.9	-3.0	5.2	-51.5	-17.7	8.3	-6.5	70.6
ID03	-5.0	-12.4	-8.3	-8.3	-9.2	-55.3		10.5		-60.3
JP01	1.2	1.9	-4.9	-1.5	-12.4	1.0	-13.7	-15.5	-16.1	8.8
JP02	-1.0	2.9	-1.4	-4.5	-0.7	-4.9	-12.7	-1.3	3.2	-4.4
JP03	0.6	0.0	0.0	-0.8	-0.7	-1.0	0.0	1.3	0.0	1.5
JP04	0.2	2.9	-0.7	-0.8	-2.6	-7.8	-17.0	-3.4	-12.9	-3.7
JP05	1.8	-1.0	-0.7	0.0	-1.3	-18.4	-13.7	-7.0	-16.1	4.4
JP06	1.2	-1.9	-2.8	-1.5	-1.3	1.0	4.3	3.6	0.0	1.5
JP07	1.4	-1.0	-4.9	-6.1	-11.1	-1.0	0.0	5.1	-3.2	-1.5
LA01	8.3	-3.8								
MY01	-0.4	-13.3	-4.9	-3.0	3.9	7.8	2.3	1.8	-6.5	-8.1
MN01	0.0	-1.0	9.0	5.3	17.6	-11.7	21.0	10.9	67.7	2.2
PH01	-0.2	2.9	-9.7	-6.8	-12.4	-11.7	-40.7	28.0	-6.5	-25.0
KR01	-8.9	45.7	-13.9	12.9	47.7	-5.8	7.7	-49.1	-25.8	-7.4
RU01	-1.0	4.8	0.7	1.5	-0.7	12.6	-24.7	-4.9	-9.7	1.5
RU02	-1.0	5.7	5.6	18.2	6.5	3.9	23.3	-9.2	-3.2	16.9
TH01	1.4	-6.7	-1.4	-3.8	-5.2	12.6	-2.3	19.2	19.4	0.0
TH02	3.6	-7.6	-11.1	-14.4	-3.9	-5.8	-14.3	-10.3	-12.9	47.1
TH03	-0.4	-1.0	-1.4	-3.0	-3.3	0.0	-25.7	14.7	32.3	16.2
TH04	4.6	1.9	-6.9	-3.8	-16.3	35.0	27.0	42.8	32.3	19.9
TH05	1.2	-1.9	-0.7	-3.0	-3.9	-1.0	-41.3	9.7	41.9	14.7
VN01	0.2	1.0	-6.3	-6.1	-11.8	2.9	1.0	-21.4	6.5	1.5
VN02	2.6	-2.9	5.6	-0.8	0.0	6.8	-100.0	-19.9	25.8	1.5
MM01	5.3	81.9								
IN01	47.6	25.3	-4.0	12.4	19.6	94.7	129.4	31.4	-43.0	5.9
IN02	-3.6	-44.8				-8.8	9.3	-37.6	-16.5	-81.3
Minimum	-8.9	-44.8	-69.4	-14.4	-47.7	-55.3	-100.0	-49.1	-43.0	-81.3
Maximum	47.6	81.9	9.0	18.2	47.7	94.7	129.4	42.8	67.7	70.6
Average	2.6	2.6	-4.5	-0.4	-1.1	0.8	-4.6	1.5	2.5	1.2