Acid Deposition Monitoring Network in East Asia (EANET)

Report of the Inter-laboratory Comparison Project 2009

12th Inter-laboratory Comparison Project on Wet Deposition

5th Inter-laboratory Comparison Project on Dry Deposition

11th Inter-laboratory Comparison Project on Soil

10th Inter-laboratory Comparison Project on Inland Aquatic Environment

February 2011
Network Center for EANET

Contents

1. INTRODUCTION

The inter-laboratory comparison project (round robin analysis survey) 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 objectives of the project are, through the evaluation of analytical results, analytical equipment and its operating condition and other practices,

(i) to recognize the analytical precision and accuracy of the measurement in each participating laboratory,

(ii) to give an opportunity to improve the quality of the analysis on wet deposition, dry deposition monitoring (filter pack method), soil monitoring and inland aquatic monitoring of EANET,

(iii) to improve reliability of analytical data through the assessment of suitable analytical methods and techniques.

The inter-laboratory comparison project is implemented by the Network Center of EANET (NC) annually for the following items:

a. Wet Deposition
b. Dry Deposition
c. Soil
d. Inland aquatic Environment

This report presented the results of the 12th inter-laboratory comparison project on wet deposition, 5th inter-laboratory comparison project on dry deposition, 11th inter-laboratory comparison project on soil, and 10th inter-laboratory comparison project on inland aquatic environment.

The number of laboratories from each country that participated in each of the projects was shown in Figure 1.1.

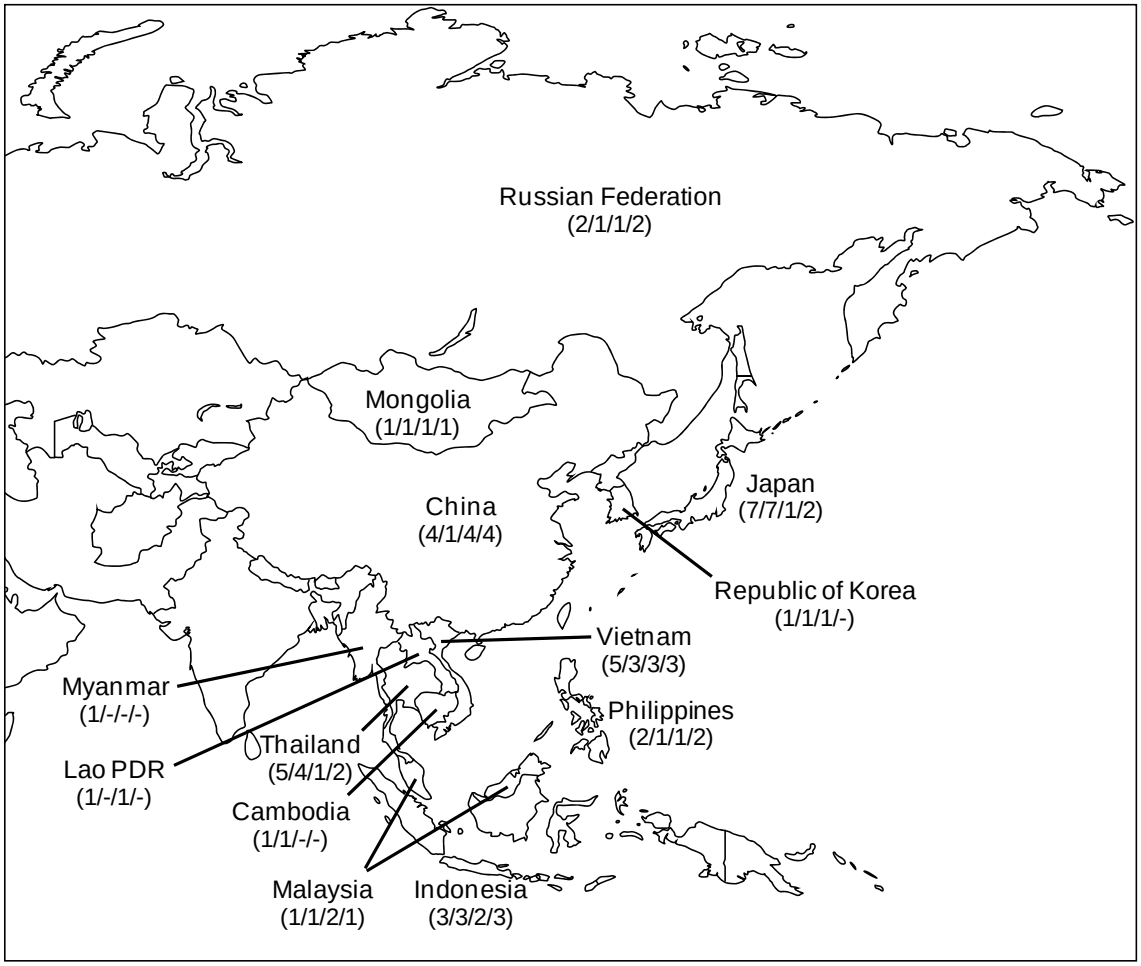


Figure 1.1 Number of participating laboratories in 2009

* The parenthesis in the figure shows the number of laboratories of each country (Wet/Dry/Soil/Inland aquatic environment)

2. 12th INTER-LABORATORY COMPARISON PROJECT ON WET DEPOSITION

2.1 Introduction

In the 12th Inter-laboratory comparison project on wet deposition, artificial rainwater samples containing known concentrations of major ions were prepared and distributed to the participating countries of EANET by the Network Center (NC). The measured values of pH, Electric Conductivity (EC) and concentrations of major ions submitted by the participating countries were compared with the prepared values and were treated statistically.

The NC shipped the artificial rainwater samples to laboratories in charge of chemical analysis in EANET on 1 October 2009. Their analytical results were required to be submitted to the NC by 28 February 2010.

2.2 Procedures

2.2.1 Participating Laboratories

The NC distributed the artificial rainwater samples to 34 laboratories in charge of chemical analysis in 13 countries of EANET. All the participating laboratories submitted their analytical results to the NC. A list of the participating laboratories with the abbreviated name and code were shown in Appendix 2.1.

2.2.2 Description of samples

Two kinds of artificial rainwater samples (one with high ions concentration and one with low ions concentration) were distributed to the laboratories. A description of the samples was given in Table 2.1.

Table 2.1 Description of artificial rainwater samples

Artificial rainwater sample	Quantity of sample	Container	Number of samples	Note
No. 091w (high concentration sample) No. 092w (low concentration sample)	100mL each	Polypropylene bottle 100mL	One bottle each	Fixed quantity of reagents are dissolved in deionized water

The prepared values of analytical parameters in the artificial rainwater samples were described in Table 2.2.

Table 2.2 Prepared values/concentrations of analytical parameters*

	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
No. 091w (high concentration)	4.52	4.23	57.8	46.7	81.9	59.9	9.1	29.0	14.8	57.5
No. 092w (low concentration)	4.92	1.66	19.9	22.0	34.0	24.0	5.9	9.3	7.0	21.2

* For 100 times diluted samples.

2.2.3 Analytical Methods and Data Checking Procedures

Before the measurement, the samples have to be diluted 100 times accurately with deionized water in each laboratory according to the specified procedure.

All participating laboratories were expected to analyze the diluted samples for the following 10 parameters; pH, EC, concentrations of SO_4^{2-}, NO_3^-, Cl^-, Na^+, K^+, Ca^{2+}, Mg^{2+} and NH_4^+.

The laboratories were required to apply the analytic methods and data checking procedures that were 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 were listed in Table 2.3.

Table 2.3 Analytical methods specified in the manual

Parameter	Analytical method
pH	Glass Electrode Method (preferably with the Electrode of non-leak inner cell)
EC	Conductivity Cell Method
SO_4^{2-} NO_3^- Cl^-	Ion Chromatography (preferably with suppressor) Spectrophotometry
Na^+ K^+ Ca^{2+} Mg^{2+}	Ion Chromatography Atomic Absorption Spectrometry Atomic Emission Spectrometry
NH_4^+	Ion Chromatography Spectrophotometry (Indophenol Blue Method)

Checking analytical results was performed using the calculation of ion balance (R_1) and total electric conductivity agreement (R_2).

Calculation of ion balance (R_1)

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

$$\mathbf{A}\ [\mu eq\ /L] = \sum n\ C_{Ai}\ [\mu mol\ /L] = 2C\ (SO_4^{2-}) + C\ (NO_3^-) + C\ (Cl^-)$$

n, C_{Ai}: electric charge of ion and concentration [μmol /L] of anion "i".

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

$$\mathbf{C}\ [\mu eq\ /L] = \sum n\ C_{Ci}\ [\mu mol\ /L] = 10^{(6-pH)} + C\ (NH_4^+) + C\ (Na^+) + C\ (K^+) + 2C\ (Ca^{2+}) + 2C\ (Mg^{2+})$$

n, C_{Ci}: electric charge of ion and concentration [μmol /L] of cation "i".

(3) Calculation of ion balance (R_1)

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

(4) R_1 calculated by the above equation was compared with allowable ranges in Table 2.4. If R_1 was out of the range, re-measurement, check with standard solutions, and/or inspection of calibration curves were required.

Table 2.4 Allowable ranges for R_1 in different concentration ranges

C+**A** [μeq/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) was calculated as follows;

$$\Lambda \text{ calc [mS /m]} = \{349.7 \times 10^{(6-pH)} + 80.0 \times 2C(SO_4^{2-}) + 71.5 \times C(NO_3^-) + 76.3 \times C(Cl^-) + 73.5 \times C(NH_4^+) + 50.1 \times C(Na^+) + 73.5 \times C(K^+) + 59.8 \times 2C(Ca^{2+}) + 53.3 \times 2C(Mg^{2+})\} / 10000$$

C: Molar concentrations [μmol/L] of ions in the parenthesis; each constant value was ionic equivalent conductance at 25 degrees centigrade.

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

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

(3) R_2 calculated by the above equation was compared with allowable ranges in Table 2.5. If R_2 was out of the range, re-measurement, check with standard solutions, and/or inspection of calibration curves were required.

Table 2.5 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)

2.3 Results

The NC received the analytical results from 34 laboratories in the participating countries of EANET. The original data submitted by the laboratories were shown in Appendix 2.2 and 2.3. Basic statistics of submitted data summarized in Table 2.6 were calculated for each parameter of the artificial rainwater samples such as: Average (Va), Minimum (Min.), Maximum (Max.), Standard deviation (S.D.), and Number of data (N). The outliers which are apart from the average greater than a factor of 3 of S.D. were not included for the statistics calculation. As shown in Table 2.6, Va agreed with prepared value (Vp) fairly well. The range of Va/Vp was between -4.6% to 1.3% for the sample No. 091w, and -3.3% to 4.6% for the sample No. 092w.

Table 2.6 Summary of analytical results of the artificial rainwater samples (Reported data after removing outliers)

Sample No. 091w

Constituents	Prepared (Vp)	Average (Va)	Va/Vp *1 %	S.D.	N	Min.	Max.
pH	4.52	4.55	0.7	0.11	34	4.25	4.77
EC [mS/m]	4.23	4.03	-4.6	0.11	33	3.62	4.23
SO_4^{2-} [µmol/L]	57.8	57.7	-0.1	2.62	33	51.6	64.8
NO_3^- [µmol/L]	46.7	46.0	-1.4	1.28	33	42.5	49.8
Cl^- [µmol/L]	81.9	80.0	-2.3	2.98	32	74.5	88.3
Na^+ [µmol/L]	59.9	59.1	-1.4	1.61	32	55.7	63.8
K^+ [µmol/L]	9.1	9.1	-0.3	0.67	33	8.1	11.3
Ca^{2+} [µmol/L]	29.0	29.4	1.3	2.07	33	27.2	35.9
Mg^{2+} [µmol/L]	14.8	14.7	-0.9	0.88	34	12.8	17.1
NH_4^+ [µmol/L]	57.5	56.8	-1.3	3.36	34	47.3	63.3

Sample No. 092w

Constituents	Prepared (Vp)	Average (Va)	Va/Vp *1 %	S.D.	N	Min.	Max.
pH	4.92	4.99	1.3	0.14	33	4.73	5.33
EC [mS/m]	1.66	1.62	-2.2	0.05	33	1.51	1.72
SO_4^{2-} [µmol/L]	19.9	20.0	0.3	1.05	33	18.5	23.9
NO_3^- [µmol/L]	22.0	21.3	-3.3	1.13	33	18.3	23.5
Cl^- [µmol/L]	34.0	33.0	-3.0	1.66	32	30.3	38.5
Na^+ [µmol/L]	24.0	23.5	-2.0	1.56	33	18.6	27.5
K^+ [µmol/L]	5.9	6.0	2.1	0.56	34	4.9	7.5
Ca^{2+} [µmol/L]	9.3	9.7	4.6	1.62	33	5.3	14.0
Mg^{2+} [µmol/L]	7.0	7.3	4.0	0.61	33	6.2	8.6
NH_4^+ [µmol/L]	21.2	21.9	3.1	2.18	34	17.0	27.4

Note: *1, (Va-Vp)/Vp x 100

The Data Quality Objective for accuracy (hereafter referred to as DQO) was specified in the QA/QC program of the EANET for every parameter to be within ±15% of deviation from Vp. In this report, analytical data of the artificial rainwater samples were compared with Vp, and the data exceed DQO were marked with flags. Flag "E" was put to the data exceed DQO within a factor of 2 (±15% to ±30%), and flag "X" was put to the data exceed DQO more than a factor of 2 (over ±30%).

A set of data for each sample was evaluated by the Data Checking Procedures described in chapter 2.2.3. The flag "I" and the flag "C" were put to the data sets with poor ion balance and poor conductivity agreement, respectively.

The results were evaluated by the following three aspects:
i) Comparison of concentration dependence on level of their concentration
- sample No. 091w (high concentrations) and No. 092w (low concentrations),
ii) Comparison of individual parameters,
iii) Comparison of circumstances of chemical analysis in each participating laboratory.

Evaluation of analytical data on both the sample No. 091w and No. 092w was presented in "2.3.1 Evaluation of laboratories' performance (by sample)", evaluation of analytical data for each constituent was presented in "2.3.2 Evaluation of laboratories' performance (by analytical parameters)", and evaluation of analytical data by the circumstances of chemical analysis such as analytical method used, experience of personnel in charge, and other analytical condition were presented in "2.3.4 Information on Laboratories".

2.3.1 Evaluation of laboratories' performance (by sample)

1) High Concentration Sample No. 091w

The number and percentage of flagged data for the high concentration sample No. 091w were shown in Table 2.7. Eleven analytical data out of 338 exceeded DQO within a factor of 2 and were flagged by "E". One analytical data out of 338 exceeded DQO more than a factor of 2 flagged by "X". Data flagged by "E" and "X" shared about 3.6 percent of all the submitted data for sample No. 091w.
The data normalized by prepared value in each parameter were shown in Figure 2.1.

Table 2.7 Number of flagged data for the Sample No. 091w (High concentrations)

Charactarization of data	pH	EC	SO_4^{2-}	NO_3^-	Cl^-	Na^+	K^+	Ca^{2+}	Mg^{2+}	NH_4^+	Total
Data within DQO	34	33	33	33	32	33	32	30	33	33	326
Data with flag E*1	0	1	1	0	1	1	1	4	1	1	11
Data with flag X*2	0	0	0	0	0	0	1	0	0	0	1
Flagged data [%]	0.0	2.9	2.9	0.0	3.0	2.9	5.9	11.8	2.9	2.9	3.6

(Total data = 338)

Note: *1, Data exceeded DQO within a factor of 2; *2, Data exceeded DQO more than a factor of 2

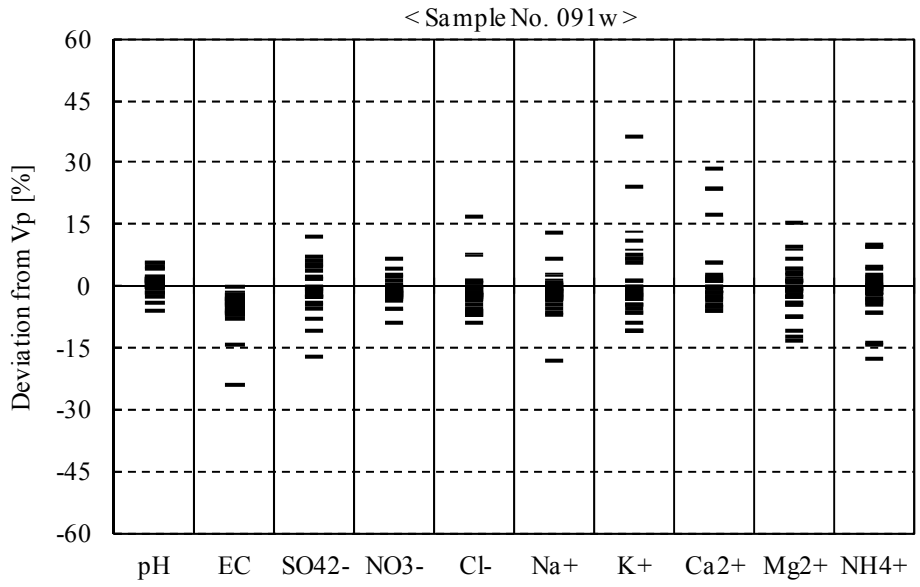


Figure 2.1 Distribution of the data normalized by prepared value in each parameter for sample No. 091w

The parameter which had most flags was Ca^{2+}. The analytical data submitted by the participating laboratories were shown in Table 2.8 with flags.

Table 2.8 Analytical Results of Sample No. 091w

Lab. ID*1	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	R_1 %	R_2 %
KH01	4.77	3.95	61.3	46.0	80.6	57.7	8.1	28.6	15.0	60.1	-4.0	-1.8
CN01	4.49	4.01	57.2	46.1	80.4	56.5	8.1	29.6	15.0	57.1	0.5	3.1
CN02	4.55	4.09	58.8	45.2	79.2	58.3	8.7	29.5	15.4	58.8	0.4	0.8
CN03	4.52	4.09	60.8	45.3	79.6	58.0	8.1	29.8	14.4	57.2	-0.9	1.8
CN04	4.45	4.15	59.9	45.0	79.1	58.5	9.1	29.3	15.1	57.0	1.0	3.1
ID01	4.61	3.89	56.9	45.5	80.2	58.0	8.8	28.5	15.0	49.3	-2.5	0.4
ID02	4.51	4.09	59.1	49.8	76.0	57.2	9.0	29.4	15.2	62.9	1.1	2.4
ID03	4.77	E 3.22	56.2	45.9	79.4	59.2	10.1	27.7	14.7	54.9	-2.5	6.8
JP01	4.57	4.15	56.3	46.4	80.3	60.0	9.2	28.1	14.2	57.0	-0.3	-1.1
JP02	4.56	4.23	57.3	46.0	82.8	59.4	9.1	28.5	14.6	57.9	-0.7	-1.2
JP03	4.58	4.14	57.1	45.8	80.5	58.9	9.2	29.0	13.7	57.2	-0.7	-1.1
JP04	4.58	4.12	56.4	46.2	81.3	58.6	8.5	28.6	14.2	56.6	-1.0	-1.0
JP05	4.59	3.94	56.4	45.6	78.2	58.4	9.0	28.9	14.6	57.6	0.2	0.8
JP06	4.62	4.05	54.6	46.4	81.3	60.3	9.1	29.4	14.7	53.7	-0.3	-1.4
JP07	4.59	3.96	57.6	45.5	82.8	60.4	8.9	28.3	14.6	56.2	-1.4	1.2
LA01	4.45	3.95	57.8	46.0	82.6	59.3	8.9	27.5	13.7	57.6	-0.1	5.2
MY01	4.60	4.03	56.5	46.2	80.4	61.4	9.7	30.6	13.7	53.8	-0.2	-0.2
MN01	4.50	4.13	56.3	45.4	76.3	59.2	8.9	27.9	13.2	55.6	0.7	0.3
MM01	4.33	3.97	60.6	48.6	87.9	58.6	8.6	28.6	14.4	59.0	0.2	C 10.6
PH01	4.59	4.02	64.8	47.7	88.3	63.8	9.8	27.5	13.0	E 47.3	-7.7	1.7
PH02	4.44	4.04	61.9	48.0	74.5	67.7	9.7	E 35.8	15.8	58.6	5.6	6.6
KR01	4.25	3.99	56.5	46.1	79.9	55.7	9.9	28.3	15.0	58.3	5.5	C 12.1
RU01	4.57	4.06	55.5	45.9	81.4	58.5	8.5	27.2	15.3	55.5	-0.8	-0.4
RU02	4.55	4.09	53.2	45.8	77.0	59.1	8.3	28.8	16.2	56.4	2.7	-0.6
TH01	4.62	4.05	57.2	45.7	80.1	60.2	8.7	30.7	14.7	56.5	0.0	-0.7
TH02	4.56	4.08	57.3	44.2	76.5	61.7	9.0	30.7	14.5	59.9	2.7	0.5
TH04	4.41	4.09	60.9	46.8	76.9	59.6	8.8	27.3	12.8	49.5	-1.8	4.2
TH05	4.50	4.07	55.2	44.0	76.3	58.0	8.7	E 34.0	16.1	56.5	5.0	2.0
TH06	4.56	4.04	56.4	42.5	77.2	60.8	9.6	29.5	E 17.1	63.3	4.5	1.1
VN01	4.54	4.17	58.9	47.2	82.7	59.6	8.9	28.6	14.1	57.5	-1.5	0.3
VN02	4.50	3.97	57.5	46.6	79.8	59.1	8.8	28.5	14.1	55.2	-0.3	3.1
VN03	4.73	3.62	E 47.8	---	---	59.7	E 11.3	29.0	15.4	62.9		
VN04	4.70	3.94	51.6	46.7	E 95.8	55.9	X 12.4	E 35.9	14.4	57.4	0.1	0.3
VN05	4.58	3.98	61.4	45.5	80.0	E 49.0	10.3	E 37.3	14.9	55.7	-0.5	2.4
Vp	4.52	4.23	57.8	46.7	81.9	59.9	9.1	29.0	14.8	57.5	0.0	0.0
N of data	34	34	34	33	33	34	34	34	34	34		
Within DQO	34	33	33	33	32	33	32	30	33	33		
Flag E	0	1	1	0	1	1	1	4	1	1		
Flag X	0	0	0	0	0	0	1	0	0	0		

Note: "E", Value exceeded the DQO (Accuracy, ±15); "X", Value exceeded the DQO (Accuracy, ±15) more than a factor of 2;
"I", Poor ion balance (R_1); "C", Poor conductivity agreement (R_2); "---", Not measured; "Vp", Prepared values of parameters;
*1. The abbreviated name and code are given in Appendix 2.1

2) Low Concentration Sample No. 092w

The number and percentage of flagged data for the low concentration sample No. 092w were shown in Table 2.9. Twenty eight analytical data out of 338 exceeded the DQO within a factor of 2 and were flagged by "E". Eight analytical data out of 338 exceeded the DQO more than a factor of 2 and were flagged by "X". Data marked with flags shared up to 10.7 percent of all the submitted data for sample No. 092w.
The normalized data by prepared value in each parameter were shown in Figure 2.2.

Table 2.9 Number of flagged data for the Sample No. 092w (low concentrations)

Charactarization of data	pH	EC	SO_4^{2-}	NO_3^-	Cl^-	Na^+	K^+	Ca^{2+}	Mg^{2+}	NH_4^+	Total
Data within DQO	34	33	32	31	32	32	28	24	28	28	302
Data with flag E*1	0	1	1	2	1	1	6	5	5	6	28
Data with flag X*2	0	0	1	0	0	1	0	5	1	0	8
Flagged data [%]	0.0	2.9	5.9	6.1	3.0	5.9	17.6	29.4	17.6	17.6	10.7

(Total data = 338)

Note: *1, Data exceeded DQO within a factor of 2; *2, Data exceeded DQO more than a factor of 2

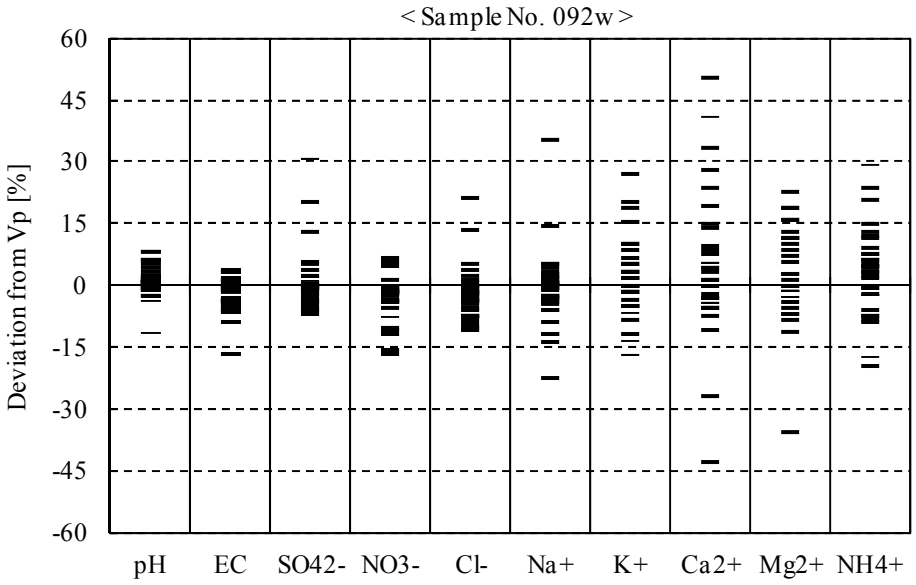


Figure 2.2 Distribution of the data normalized by prepared value for each parameter for sample No. 092w

Analytical data of Cations had a tendency to be marked with flags in comparison with anions. The analytical data submitted by the participating laboratories were shown in Table 2.10 with flags.

Table 2.10 Analytical Results of Sample No. 092w

Lab. ID*1	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	R_1 %	R_2 %
KH01	4.35	1.55	20.0	21.1	32.0	23.0	5.1	9.7	7.7	22.2	I 16.5	C 28.6
CN01	4.88	1.59	19.9	21.2	33.0	23.1	5.4	10.6	7.8	23.1	3.9	3.9
CN02	4.94	1.60	20.0	20.8	32.4	24.8	5.9	E 11.5	E 8.3	23.9	6.3	2.6
CN03	4.90	1.68	20.9	21.1	32.9	24.2	5.2	9.8	7.0	22.5	1.2	0.4
CN04	4.87	1.68	21.0	21.7	33.2	25.2	6.0	E 10.7	7.8	23.7	4.2	2.7
ID01	5.18	1.57	20.1	21.7	33.5	22.9	6.5	9.7	7.0	19.6	-3.5	-3.6
ID02	5.02	1.58	20.3	22.0	30.3	21.2	5.6	E 11.9	7.8	22.1	2.6	0.1
ID03	5.22	E 1.38	19.1	21.6	32.6	25.2	E 6.8	10.1	7.2	21.2	0.8	2.4
JP01	4.98	1.71	19.8	21.6	33.5	23.3	6.3	8.9	6.7	20.7	-1.5	-3.8
JP02	4.95	1.64	19.7	21.8	33.7	23.4	5.9	9.1	6.9	21.1	-0.7	-0.7
JP03	4.95	1.65	19.8	21.6	33.6	23.7	6.2	10.0	6.8	20.7	0.3	-0.7
JP04	4.95	1.71	20.3	23.2	E 41.2	23.4	5.6	9.0	6.9	22.3	-5.4	-0.3
JP05	5.03	1.59	19.9	21.9	34.0	24.1	5.9	9.3	6.9	21.6	-1.3	-0.7
JP06	5.08	1.59	19.7	21.7	33.3	24.4	6.1	9.8	7.1	21.7	0.0	-1.8
JP07	4.99	1.60	19.3	21.2	32.8	24.0	5.9	9.4	6.8	20.7	0.3	-1.0
LA01	4.86	1.60	20.4	22.3	35.2	24.2	5.8	8.8	6.2	21.9	-1.3	4.0
MY01	5.03	1.57	18.8	21.1	34.3	25.0	6.1	9.6	6.5	21.9	0.8	-0.5
MN01	4.88	1.66	19.2	21.2	31.3	24.1	E 4.9	8.8	X 4.5	19.4	-1.5	-1.5
MM01	5.33	1.63	20.6	23.0	35.8	23.4	5.6	9.4	6.9	23.6	-5.3	-5.9
PH01	4.91	1.65	18.7	19.7	31.0	23.8	6.5	X 5.3	7.4	E 17.0	-1.8	-3.4
PH02	4.80	1.59	E 23.9	23.5	31.5	X 32.5	6.4	X 18.5	E 8.6	22.5	I 12.2	12.2
KR01	4.80	1.72	19.5	21.6	33.5	20.7	E 6.8	9.0	7.6	E 25.6	4.1	2.5
RU01	4.86	1.61	19.1	21.8	34.7	24.4	5.6	8.8	7.2	19.6	0.4	2.7
RU02	4.93	1.67	18.5	21.9	32.1	22.6	6.0	8.6	7.8	19.9	1.1	-2.2
TH01	4.98	1.66	19.5	21.4	33.2	24.5	5.8	E 11.1	7.5	22.2	3.4	-1.0
TH02	4.95	1.51	18.6	19.4	30.4	24.2	5.8	8.3	6.4	22.8	3.6	1.6
TH04	4.73	1.69	19.9	21.5	31.5	27.5	E 7.1	9.3	6.5	E 17.5	4.9	4.9
TH05	5.32	1.64	18.9	20.3	31.5	23.8	5.5	X 13.1	E 8.1	20.8	4.1	-7.5
TH06	5.00	1.56	20.0	19.6	32.7	23.2	5.7	10.2	7.9	E 26.2	4.6	1.8
VN01	4.96	1.66	20.3	21.6	30.7	24.7	6.2	9.0	7.0	21.0	1.0	-1.8
VN02	4.91	1.67	19.8	21.5	34.4	22.6	E 7.5	8.9	6.9	19.3	-1.2	-0.5
VN03	5.13	1.55	20.6	---	---	21.2	6.0	E 6.8	6.6	E 27.4		
VN04	5.23	1.58	22.5	E 18.3	38.5	21.9	E 7.0	X 14.0	E 8.1	E 24.4	0.8	0.2
VN05	4.97	1.64	X 26.0	E 18.5	32.7	E 18.6	6.1	X 12.4	E 8.3	22.8	-1.8	2.2
Vp	4.92	1.66	19.9	22.0	34.0	24.0	5.9	9.3	7.0	21.2	0.0	0.0
N of data	34	34	34	33	33	34	34	34	34	34		
Within DQO	34	33	32	31	32	32	28	24	28	28		
Flag E	0	1	1	2	1	1	6	5	5	6		
Flag X	0	0	1	0	0	1	0	5	1	0		

Note: "E", Value exceeded the DQO (Accuracy, ±15); "X", Value exceeded the DQO (Accuracy, ±15) more than a factor of 2;
"I", Poor ion balance (R_1); "C", Poor conductivity agreement (R_2); "---", Not measured; "Vp", Prepared values of parameters;
*1. The abbreviated name and code are given in Appendix 2.1

3) Comparison of High and Low Concentration Sample

The percentage of flagged data for the high concentration sample and the low concentration sample were shown in Figure 2.3.
The percentage of the data within the DQO for the sample No. 091w and 092w were 96.4% and 89.3% respectively. The difference between both samples was 7.1%. In this project, the total number of flagged data was 48 (E: 39, X: 9) among the whole set of 676 data.

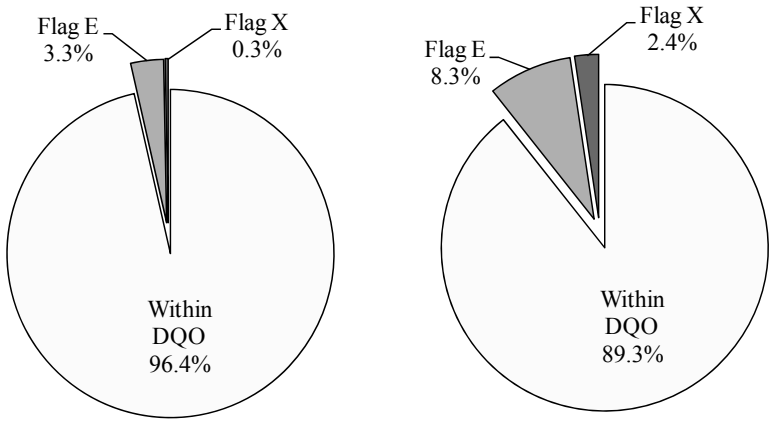


Figure 2.3 Percentage of flagged data for Sample No. 091w and No. 092w (Left: No. 091w, Right: No. 092w)

4) The number of laboratory (by number of flags)

The number of laboratory by number of flags was shown in Figure 2.4. The number of laboratory without flagged data was 17, which corresponds to 50.0% of all the analytical data submitted by participating laboratories.

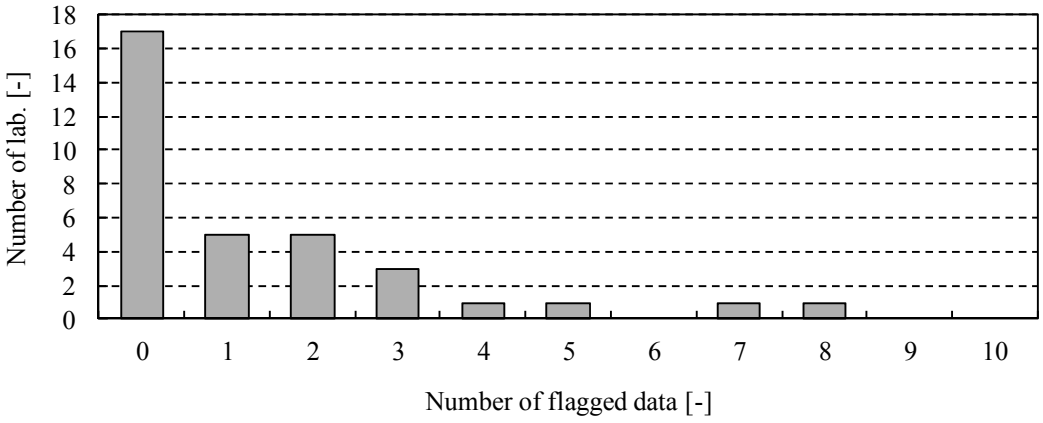


Figure 2.4 Histogram of the number of laboratory (by number of flags)

2.3.2 Evaluation of laboratories' performance (by analytical parameter)

The data normalized by Vp were shown in Figures 2.5 through 2.24 for each parameter. In scatter diagrams (lower figures), bold line means the prepared values of sample No. 091w and 092w, broken lines and dotted lines showed the values of Vp±15% and Vp±30% respectively.

1) pH

All participating laboratories used pH meter with glass electrode method for the measurement of pH. All the obtained data satisfied the DQO of the QA/QC program of EANET.

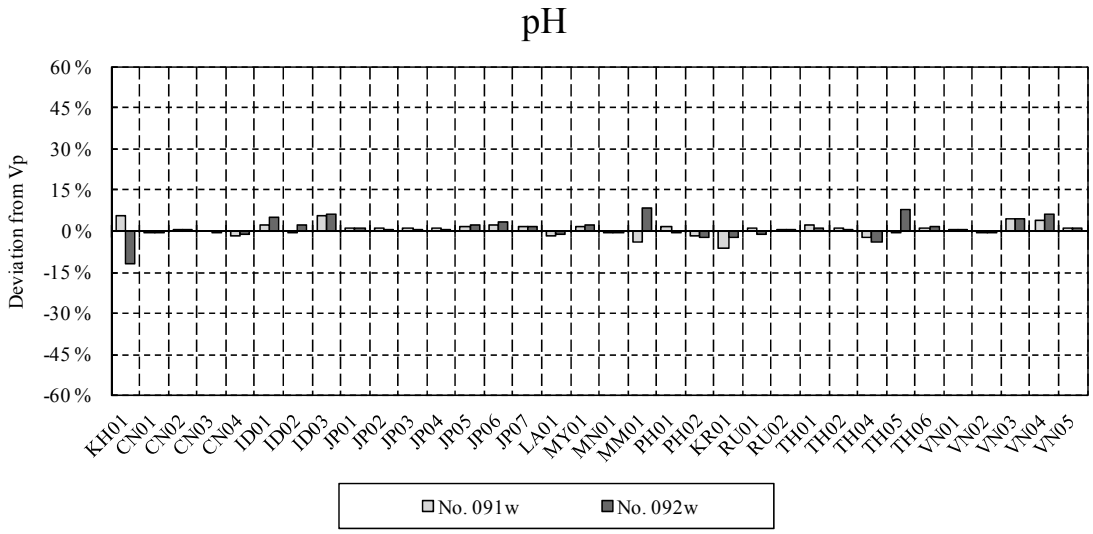
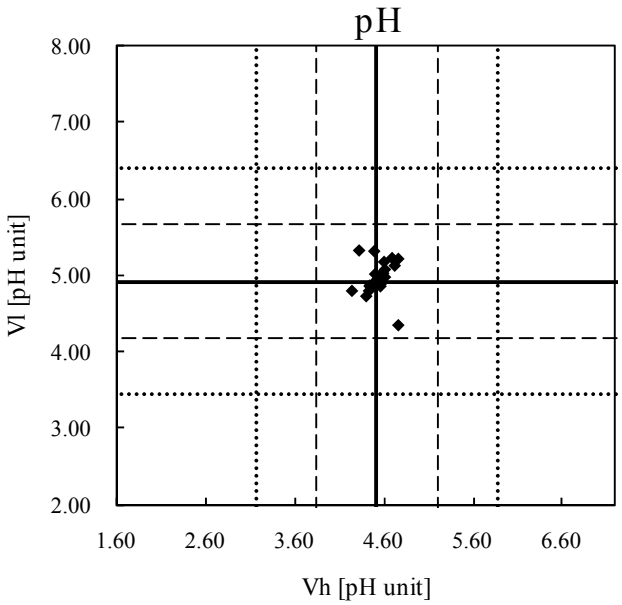


Figure 2.5 Deviation from prepared value for pH (normalized by prepared value)



(Vh: Values for high conc. sample, Vl: Values for low conc. sample)

Figure 2.6 Scatter diagram for pH

2) EC

All participating laboratories used conductivity cell method for the measurement of EC. One data submitted by ID03 exceeded the DQO in both sample.

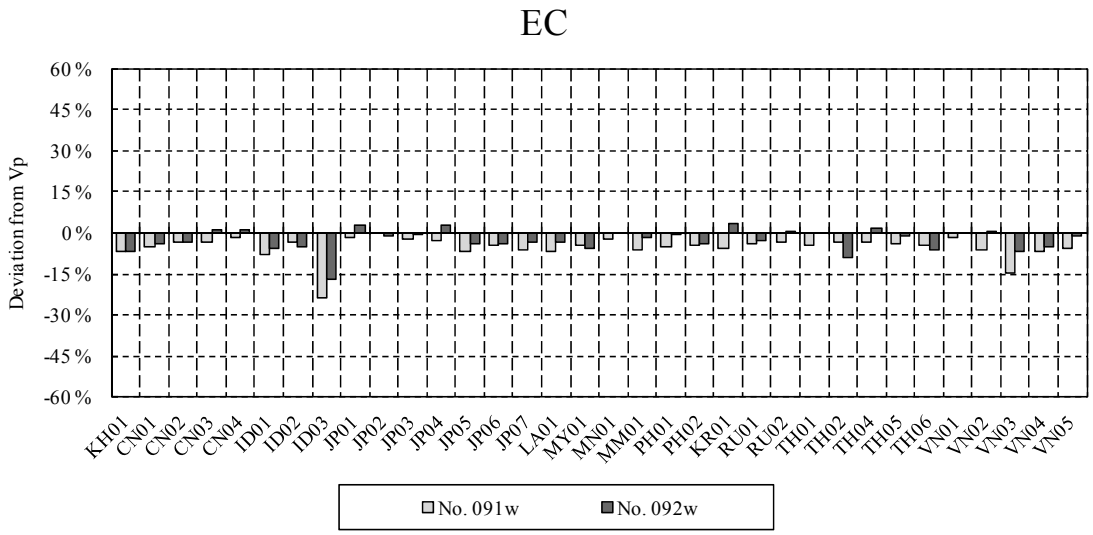
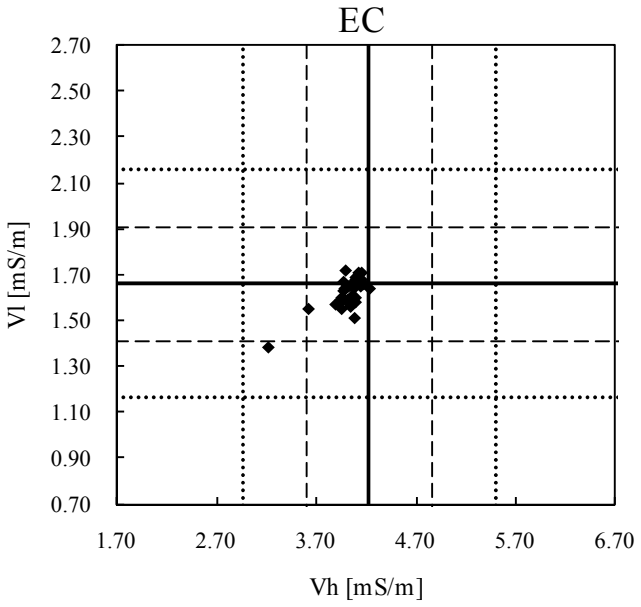


Figure 2.7 Deviation from prepared value for EC (normalized by prepared value)



(Vh: Values for high conc. sample, Vl: Values for low conc. sample)

Figure 2.8 Scatter diagram for EC

3) SO_4^{2-}

The data of sample No. 091w from VN03 and the data of sample No. 092w from PH01 exceeded the DQO and were marked with flag “E”. Additionally, the data of sample No. 092w from VN05 exceeded the DQO more than a factor of 2 and was marked with flag “X”.

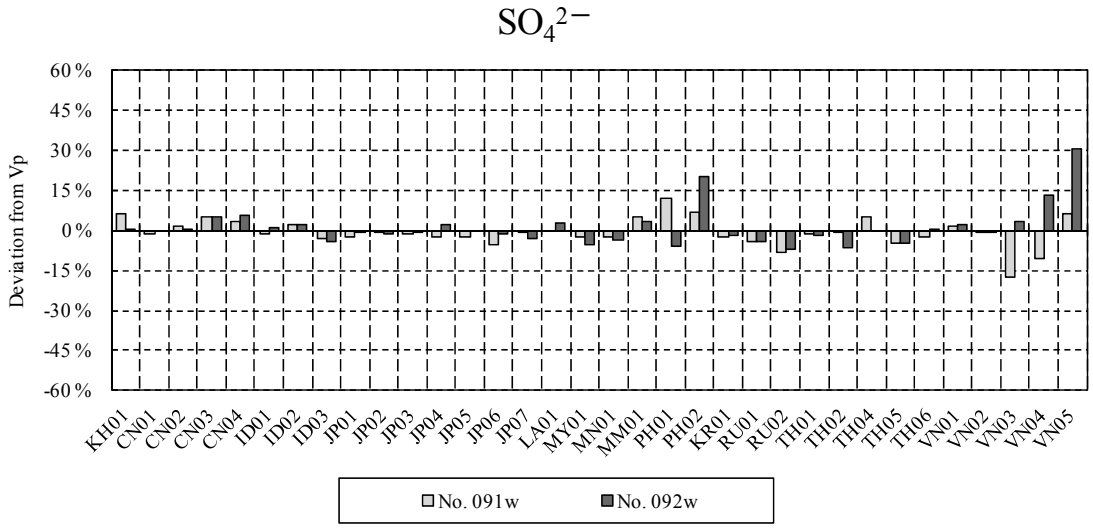
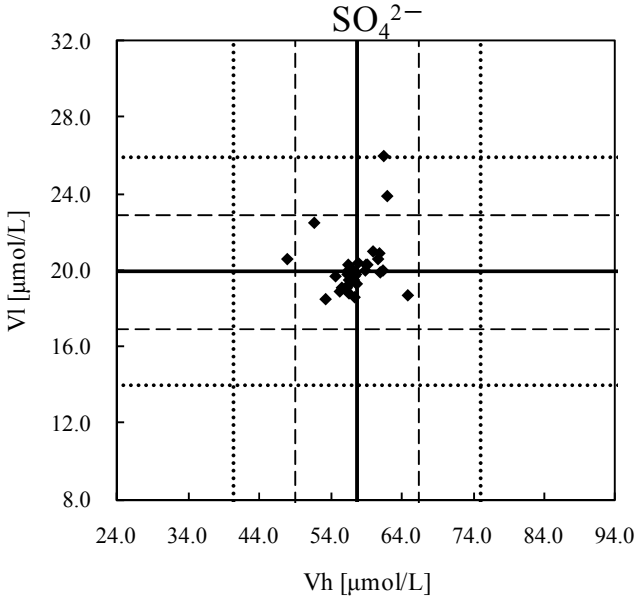


Figure 2.9 Deviation from prepared value for SO_4^{2-} (normalized by prepared value)



(Vh: Values for high conc. sample, Vl: Values for low conc. sample)

Figure 2.10 Scatter diagram for SO_4^{2-}

4) NO_3^-

All the data of sample No. 091w satisfied the DQO. The data of sample No. 092w from VN04 and VN05 exceeded the DQO and were marked with flag "E".

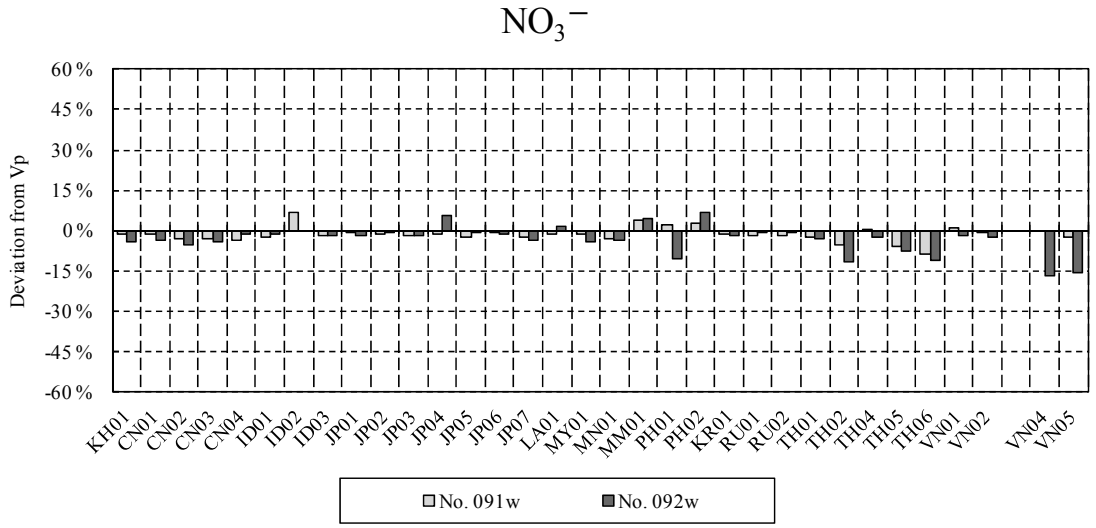
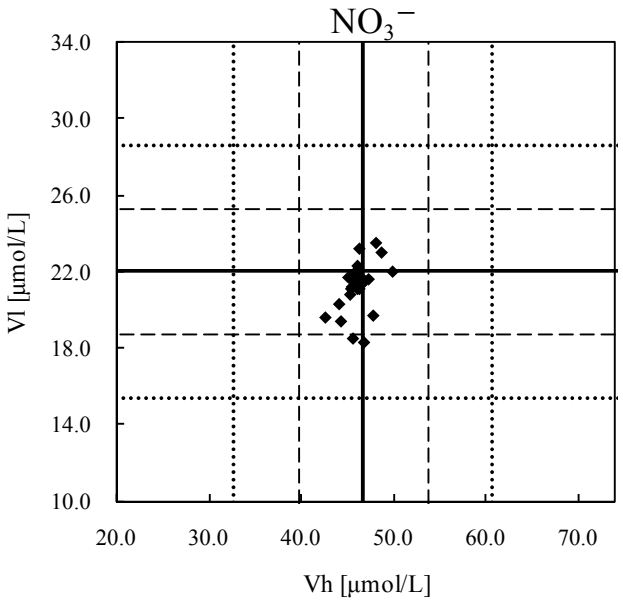


Figure 2.11 Deviation from prepared value for NO_3^- (normalized by prepared value)



(Vh: Values for high conc. sample, Vl: Values for low conc. sample)

Figure 2.12 Scatter diagram for NO_3^-

5) Cl^-

The data of sample No. 091w from VN04 and the data of sample No. 092w from JP04 exceeded the DQO and were marked with flag “E”.

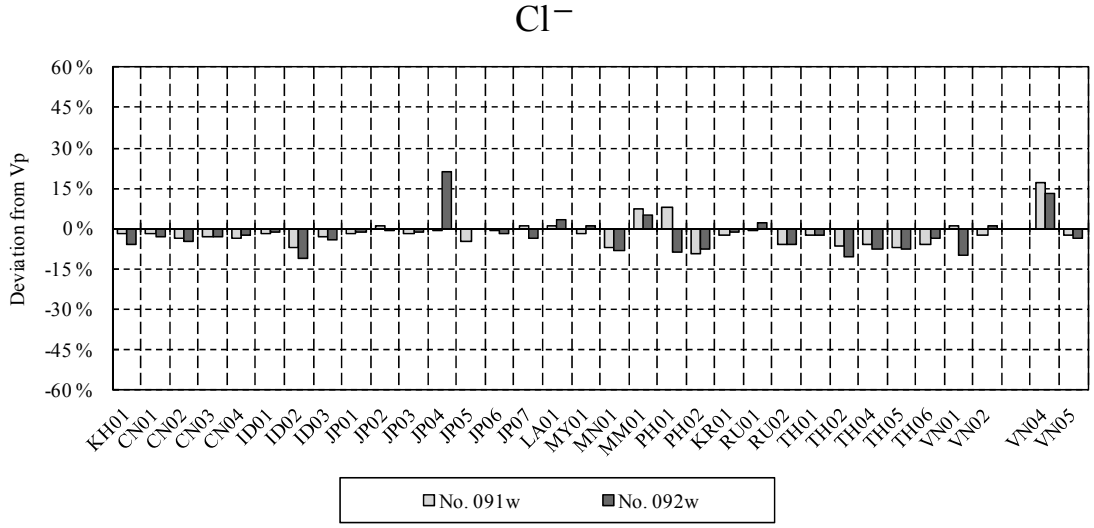
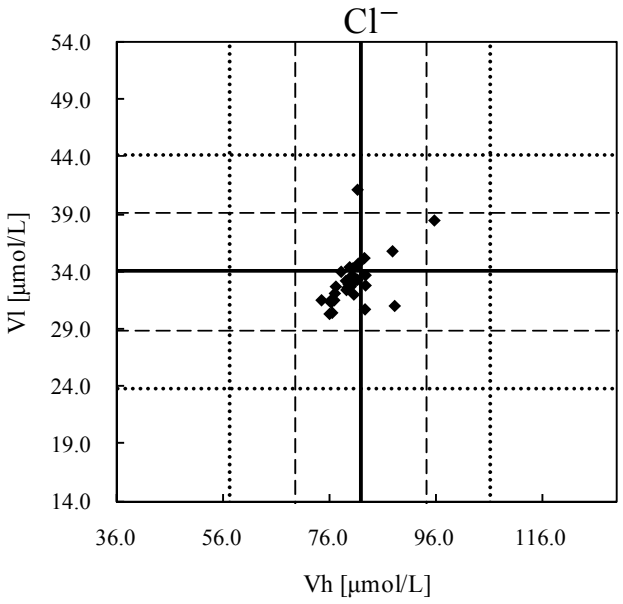


Figure 2.13 Deviation from prepared value for Cl^- (normalized by prepared value)



(Vh: Values for high conc. sample, Vl: Values for low conc. sample)

Figure 2.14 Scatter diagram for Cl^-

6) Na^+

The data of sample No. 091w and 092w from VN05 exceeded the DQO and were marked with flag “E”. Additionally, the data of sample No. 092w from PH02 exceeded the DQO more than a factor of 2 and was marked with flag “X”.

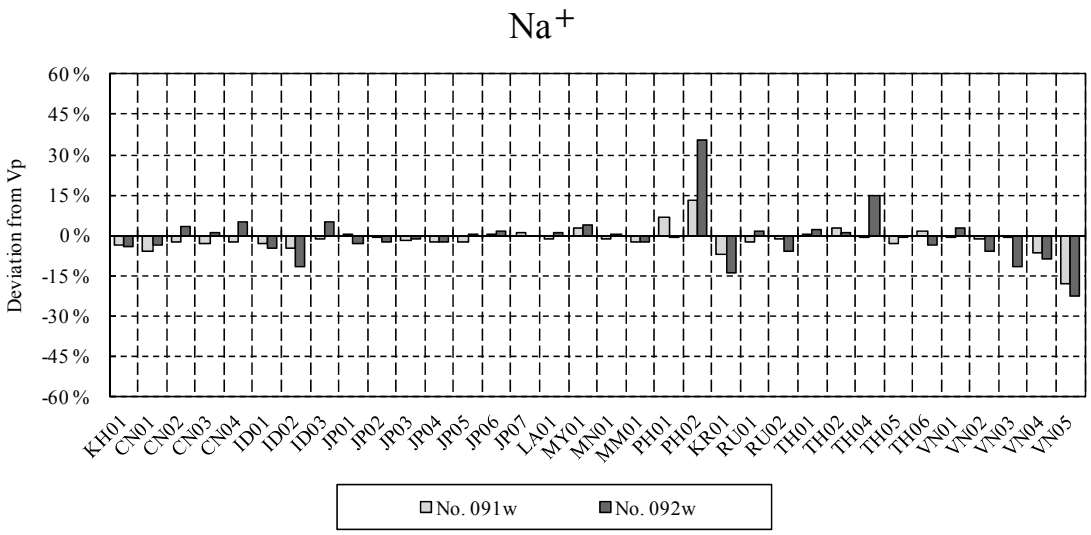
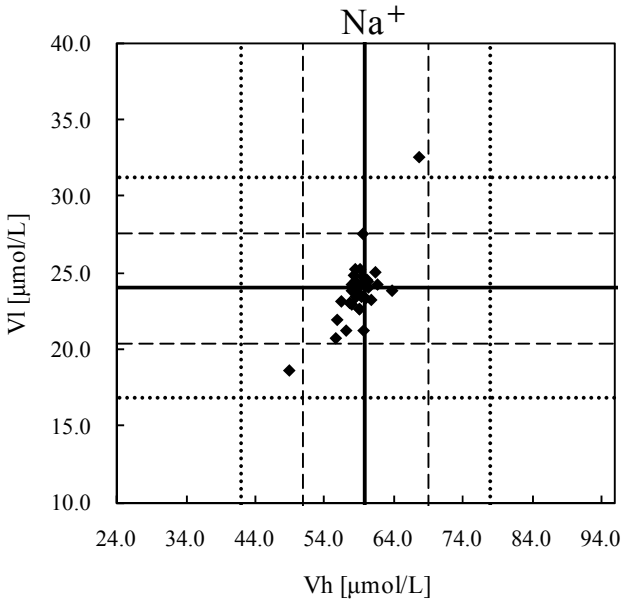


Figure 2.15 Deviation from prepared value for Na^+ (normalized by prepared value)



(Vh: Values for high conc. sample, Vl: Values for low conc. sample)

Figure 2.16 Scatter diagram for Na^+

7) K^+

The data of sample No. 091w from VN03 and the data of sample No. 092w from 6 laboratories (ID03, MN01, KR01, TH04, VN02 and VN04) exceeded the DQO and were marked with flag "E". Additionally, the data of sample No. 091w from VN04 exceeded the DQO more than a factor of 2 and was marked with flag "X".

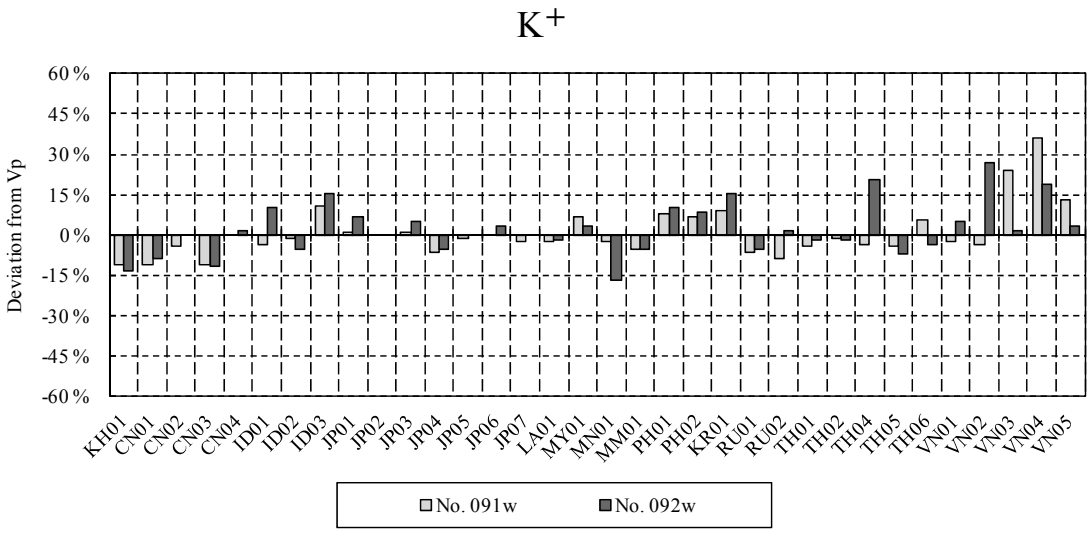
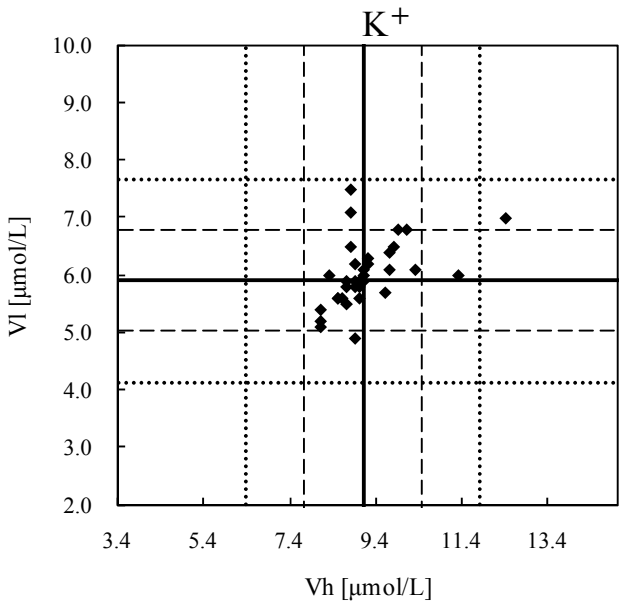


Figure 2.17 Deviation from prepared value for K^+ (normalized by prepared value)



(Vh: Values for high conc. sample, Vl: Values for low conc. sample)

Figure 2.18 Scatter diagram for K^+

8) Ca^{2+}

The data of sample No. 091w from 4 laboratories (PH02, TH05, VN04 and VN05) and the data of sample No. 092w from 5 laboratories (CN02, CN04, ID02, TH01 and VN03) exceeded the DQO and were marked with flag "E". Additionally, the data of sample No. 092w from 5 laboratories (PH01, PH02, TH05, VN04 and VN05) exceeded the DQO more than a factor of 2 and were marked with flag "X".

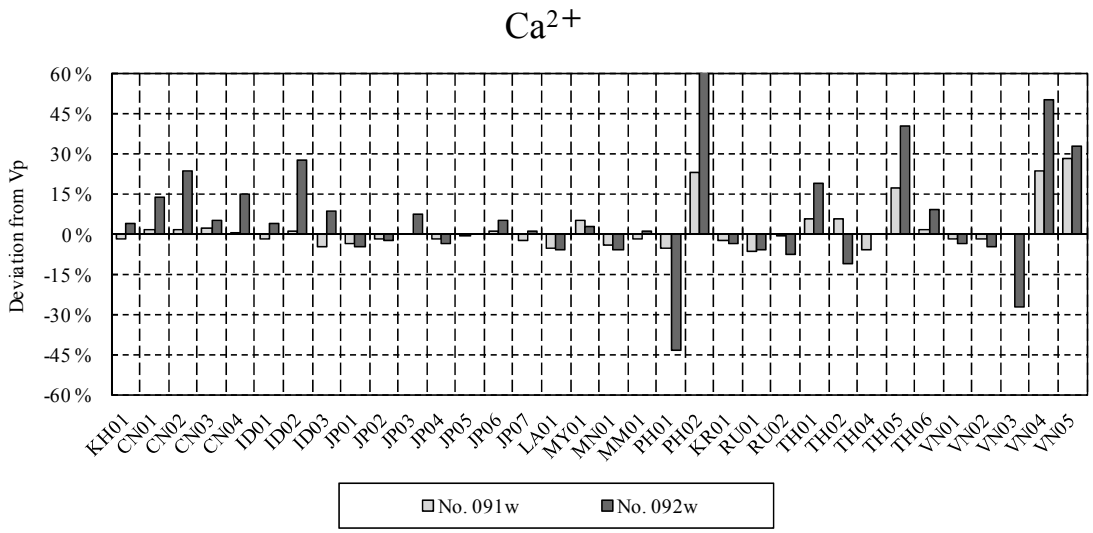
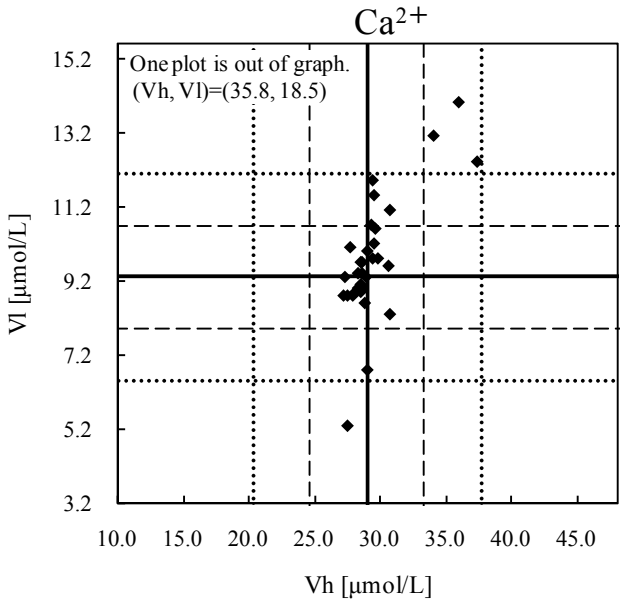


Figure 2.19 Deviation from prepared value for Ca^{2+} (normalized by prepared value)



(Vh: Values for high conc. sample, Vl: Values for low conc. sample)

Figure 2.20 Scatter diagram for Ca^{2+}

9) Mg^{2+}

The data of sample No. 091w from TH06 and the data of sample No. 092w from 5 laboratories (CN02, PH02, TH05, VN04 and VN05) exceeded the DQO and marked with flag "E". Additionally, the data of sample No. 092w from MN01 exceeded the DQO more than a factor of 2 and was marked with flag "X".

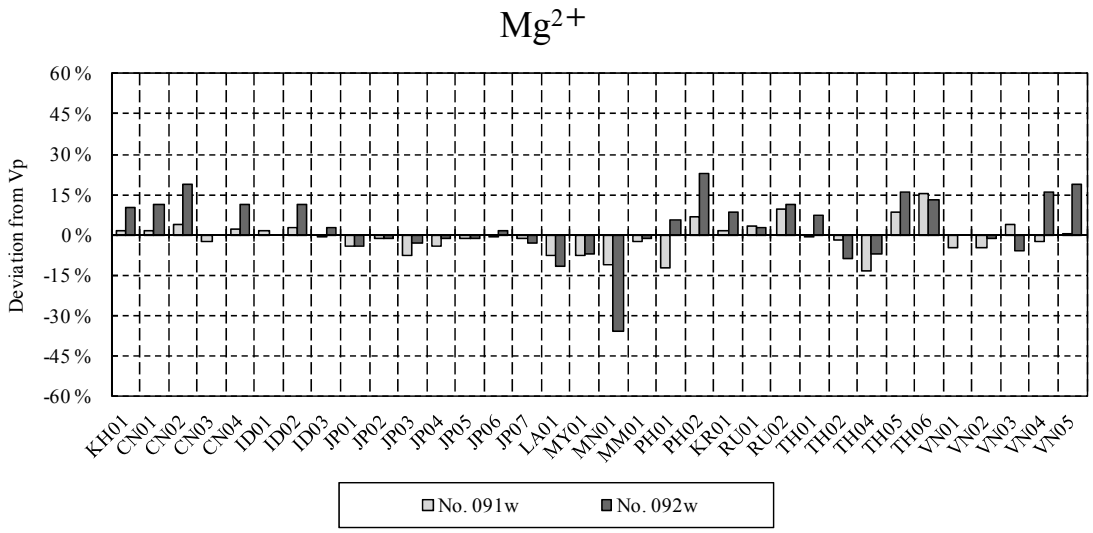
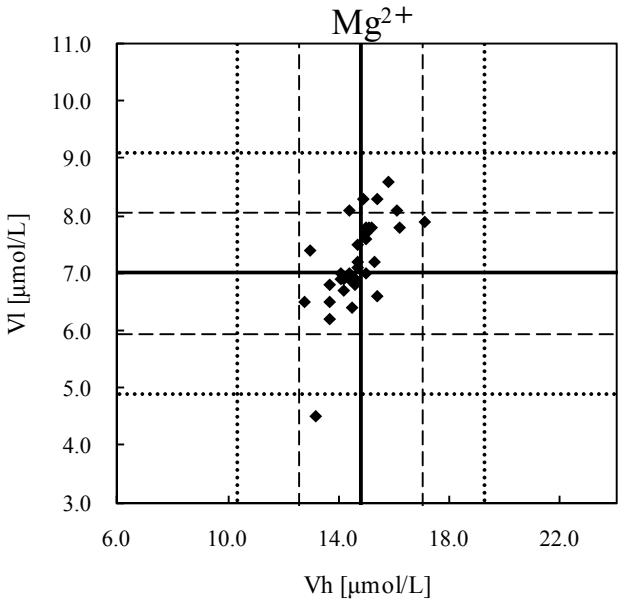


Figure 2.21 Deviation from prepared value for Mg^{2+} (normalized by prepared value)



(Vh: Values for high conc. sample, Vl: Values for low conc. sample)

Figure 2.22 Scatter diagram for Mg^{2+}

10) NH_4^+

The data of sample No. 091w and the data of sample No. 092w from 6 laboratories (PH01, KR01, TH04, TH06, VN03 and VN04) exceeded the DQO and marked with flag "E".

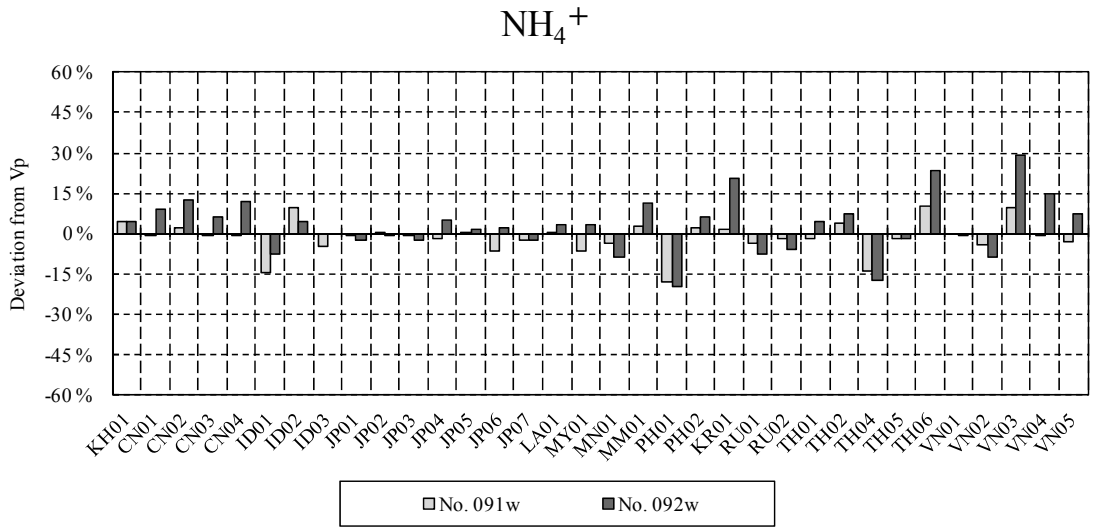
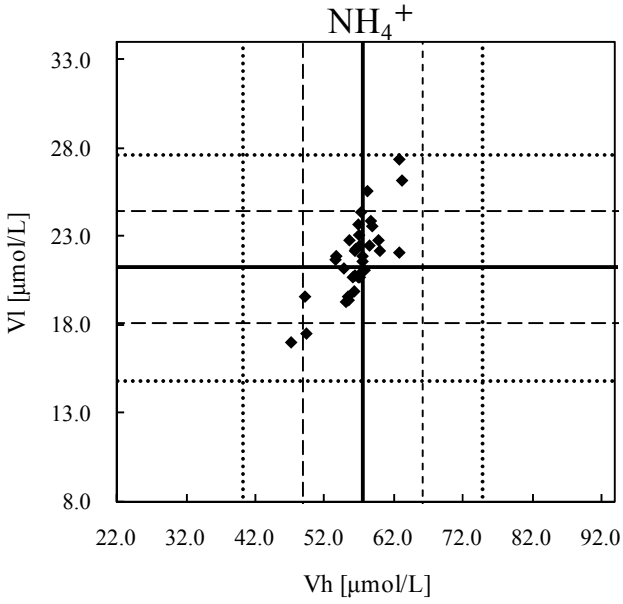


Figure 2.23 Deviation from prepared value for NH_4^+ (normalized by prepared value)



(Vh: Values for high conc. sample, Vl: Values for low conc. sample)

Figure 2.24 Scatter diagram for NH_4^+

For further statistical evaluation, the NC applied the Z-score to the data of all parameters. Detail of the Z-score evaluation was shown in Appendix 2.4.

2.3.3 Overall Evaluation

The concentrations of the analytical parameters in the samples for this survey were fixed on the basis of the statistics of monitoring data on wet deposition in EANET. Compared to the samples for the survey in 2008, concentrations of ions in the sample No. 091w and 092w were slightly high.

The relative standard deviations (R.S.D.) of each parameter for the sample No. 091w and No. 092w were shown in the Figure 2.25. The R.S.D. of Ca^{2+} for sample No. 092w was the highest in this survey. The R.S.D. values for sample No. 092w were higher than those for sample No. 091w on every parameter.

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

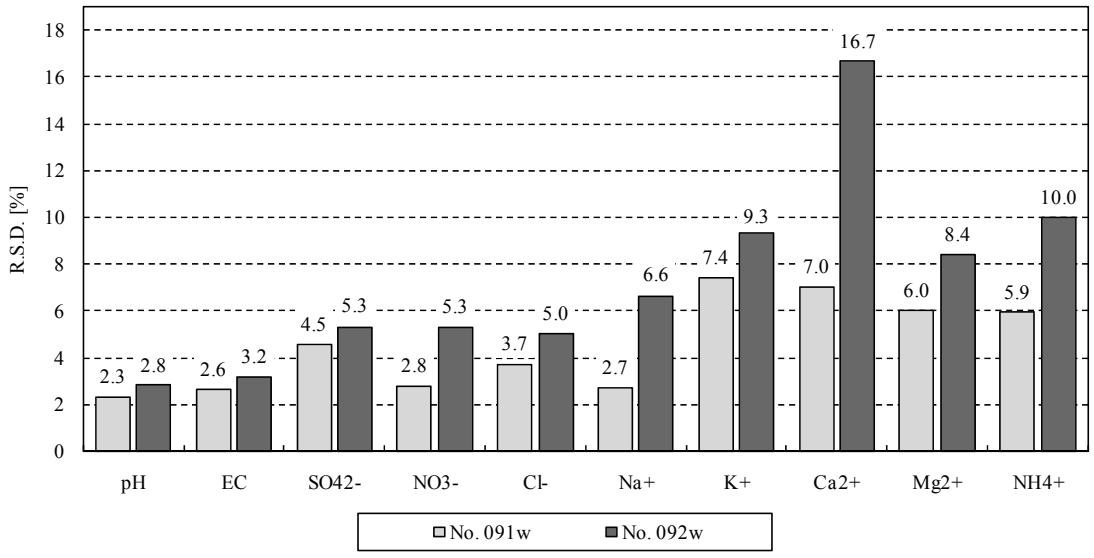


Figure 2.25 Relative standard deviations (R.S.D.) of each constituent

Some laboratories had problems in calibration curves for the determination of ion concentrations in this project as same as past projects. The person in charge of analysis needs to confirm that the calibration curves were suitable for accurate determination analysis. The reliability of the calibration curves have to be examined before the analysis of the samples by analyzing the Standard Reference Materials (SRMs) to avoid the acquisition of low quality data. In addition, the staffs in charge of analysis have to take care to prepare and save a potion of the standard solution for drawing the calibration curves.

2.3.4 Information on Laboratories

1) Number of analysts and their experience

Number of analysts and years of their experience were shown in Table 2.11 and Table 2.12 respectively. In the Table 2.11, the letters of “A”, “B” and “C” mean individuals of analysts in each laboratory who carried out analyses. In 17 laboratories, same analyst carried out the analyses for all parameters. Clear relationship between the number of analysts and flagged data was not suggested.

Table 2.11 Number of analysts

Lab. ID	Total	pH	EC	SO_4^{2-}	NO_3^-	Cl^-	Na^+	K^+	Ca^{2+}	Mg^{2+}	NH_4^+
KH01	2	A	A	B	B	B	B	B	B	B	B
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	2	A	A	B	B	B	B	B	B	B	B
ID01	2	A	A	A	A	A	B	B	B	B	B
ID02	1	A	A	A	A	A	A	A	A	A	A
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	A	A	A	A	A	A	A	A
MY01	3	A	A	B	B	B	C	C	C	C	C
MN01	3	A	A	B	B	B	C	C	C	C	C
MM01	1	A	A	A	A	A	A	A	A	A	A
PH01	3	A	A	B	B	B	C	C	C	C	A
PH02	2	A	A	B	B	B	B	B	B	B	B
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	3	A	A	B	B	B	C	C	C	C	B
TH01	2	A	A	A	A	A	B	B	B	B	B
TH02	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
TH06	1	A	A	A	A	A	A	A	A	A	A
VN01	3	A	A	B	B	B	C	C	C	C	C
VN02	2	A	A	B	B	B	B	B	B	B	B
VN03	3	A	A	B	---	---	C	C	C	C	B
VN04	2	A	A	B	B	B	B	B	B	B	B
VN05	2	A	A	B	B	B	B	B	B	B	B

Note: Light mesh, Analytic data of sample No. 091w or No. 092w was marked with flag "E" or "X";
Dark mesh, Analytic data of both samples were marked with flag "E" or "X";
"---", Not measured

Total of 178 data out of 328 were analyzed by the analysts whose experience was less than 5 years. The number corresponds to 52.7% of all the submitted data. Clear relationship between the years of experience and flagged data was not suggested.

Table 2.12 Years of experience

Lab. ID	pH	EC	SO_4^{2-}	NO_3^-	Cl^-	Na^+	K^+	Ca^{2+}	Mg^{2+}	NH_4^+
KH01	2	2	5	5	5	5	5	5	5	5
CN01	18	18	18	18	18	18	18	18	18	18
CN02	11	11	1	1	1	1	1	1	1	1
CN03	11	11	11	11	11	11	11	11	11	11
CN04	1	1	14	14	14	14	14	14	14	14
ID01	10	10	10	10	10	10	10	10	10	10
ID02	8	8	8	8	8	8	8	8	8	8
ID03	2	2	2	2	2	2	2	2	2	2
JP01	6	6	6	6	6	6	6	6	6	6
JP02	3	3	3	3	3	3	3	3	3	3
JP03	2	2	2	2	2	2	2	2	2	2
JP04	2	2	2	2	2	2	2	2	2	2
JP05	2	2	2	2	2	2	2	2	2	2
JP06	3	3	3	3	3	3	3	3	3	3
JP07	2	2	2	2	2	2	2	2	2	2
LA01	3	3	3	3	3	3	3	3	3	3
MY01	3	3	7	7	7	4	4	4	4	4
MN01	4	4	11	11	11	4	4	4	4	4
MM01	4	4	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
PH01	1	1	2	2	2	5	5	5	5	1
PH02	2	2	2	2	2	2	2	2	2	2
KR01	12	12	12	12	12	12	12	12	12	12
RU01	6	6	11	11	11	12	12	12	12	6
RU02	6	6	37	37	37	18	18	18	18	37
TH01	12	7	7	7	7	12	12	12	12	12
TH02	2	2	2	2	2	2	2	2	2	2
TH04	8	8	8	8	8	8	8	8	8	8
TH05	6	6	3	3	3	3	3	3	3	3
TH06	8	8	6	6	6	6	6	6	6	6
VN01	1	1	25	25	25	16	16	16	16	16
VN02	4	4	5	5	5	5	5	5	5	5
VN03	3	3	4	---	---	6	6	6	6	4
VN04	4	4	4	4	4	4	4	4	4	4
VN05	4	4	2	2	2	2	2	2	2	2

Note: Light mesh, Analytic data of sample No. 091w or No. 092w was marked with flag "E" or "X";
Dark mesh, Analytic data of both samples were marked with flag "E" or "X";
"---", Not measured

2) Analytical instruments

As shown in Figure 2.26, most of the participating laboratories used the specified methods described in the "Technical Manual for Wet Deposition Monitoring in East Asia". Laboratory of RU02 did not used the specified methods for the analyses of SO_4^{2-}, Cl^- and NH_4^+. (SO_4^{2-}: Nephelometry, Cl^-: Titrimetry, NH_4^+: Spectrophotometry without Indophenol) The specified methods were shown in Table 2.3.
Analytical methods used for the measurement in the participating laboratories were shown in Table 2.13.

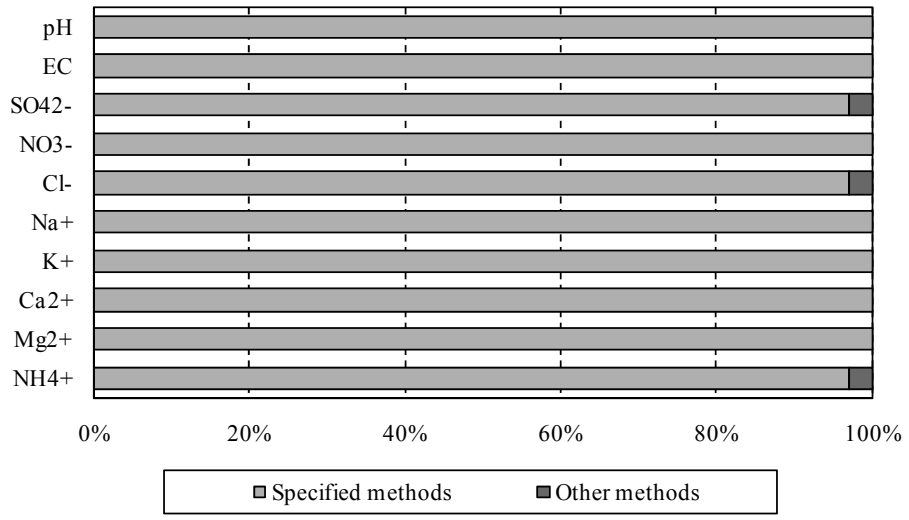


Figure 2.26 Percentage of laboratories that used the specified methods

Table 2.13 Analytical method used for the measurement in the participating laboratories

Lab. ID	SO_4^{2-}	NO_3^-	Cl^-	Na^+	K^+	Ca^{2+}	Mg^{2+}	NH_4^+
KH01	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography
CN01	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography
CN02	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography
CN03	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography
CN04	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography
ID01	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography
ID02	Ion chromatography	Ion chromatography	Ion chromatography	Ion chromatography	Ion chromatography	Ion chromatography	Ion chromatography	Ion chromatography
ID03	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography
JP01	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography
JP02	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography
JP03	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography
JP04	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography
JP05	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography
JP06	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography
JP07	Ion chromatography	Ion chromatography	Ion chromatography	Ion chromatography	Ion chromatography	Ion chromatography	Ion chromatography	Ion chromatography
LA01	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography
MY01	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography
MN01	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography
MM01	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography
PH01	Ion Chromatography	Ion Chromatography	Ion Chromatography	Atomic Absorption Spectrometry	Atomic Absorption Spectrometry	Atomic Absorption Spectrometry	Atomic Absorption Spectrometry	Spectrophotometry (Indophenol)
PH02	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography
KR01	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography
RU01	Ion Chromatography	Ion Chromatography	Ion Chromatography	Atomic Emission Spectrometry	Atomic Emission Spectrometry	Atomic Absorption Spectrometry	Atomic Absorption Spectrometry	Spectrophotometry (Indophenol)
RU02	Nephelometry	Spectrophotometry	Titrimetry	Atomic Emission Spectrometry	Atomic Emission Spectrometry	Atomic Absorption Spectrometry	Atomic Absorption Spectrometry	Spectrophotometry (Other)
TH01	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography
TH02	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography
TH04	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography
TH05	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography
TH06	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography
VN01	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography
VN02	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography
VN03	Spectrophotometry	---	---	Atomic Absorption Spectrometry	Atomic Absorption Spectrometry	Atomic Absorption Spectrometry	Atomic Absorption Spectrometry	Spectrophotometry (Indophenol)
VN04	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography
VN05	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography	Ion Chromatography

Note: "---", Not measured; "n.i.", No information

3) Date of Analysis

Figure 2.27 shows the histogram of "Start date" and "Finish date" of analysis in the participating laboratories. In total, 37% of all the submitted data was determined in January 2010, and 10% was determined after the deadline of data submission in this project.

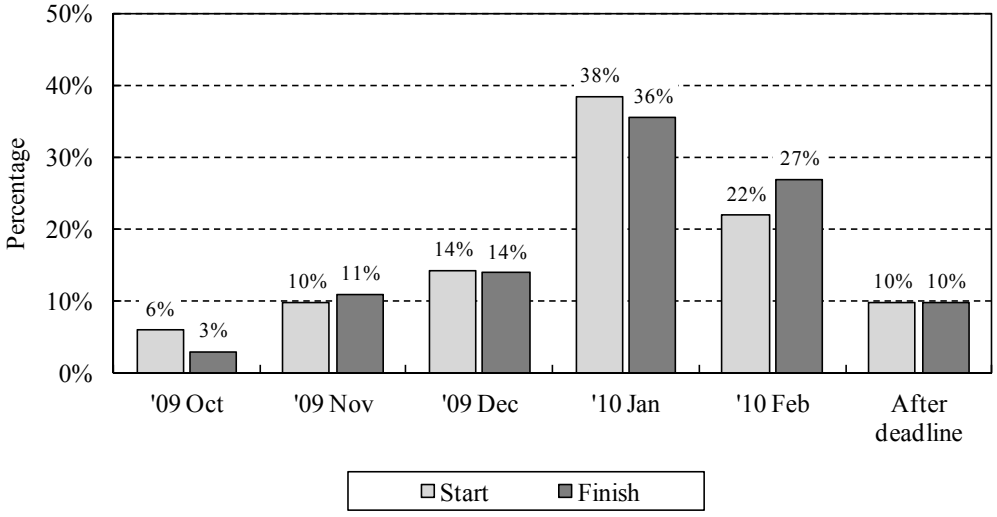


Figure 2.27 Histogram of Start date and Finish date of analysis

Figure 2.28 shows how many days were needed to determine the analytical data in the participating laboratories. Most analytical data were obtained within less than 3 days.

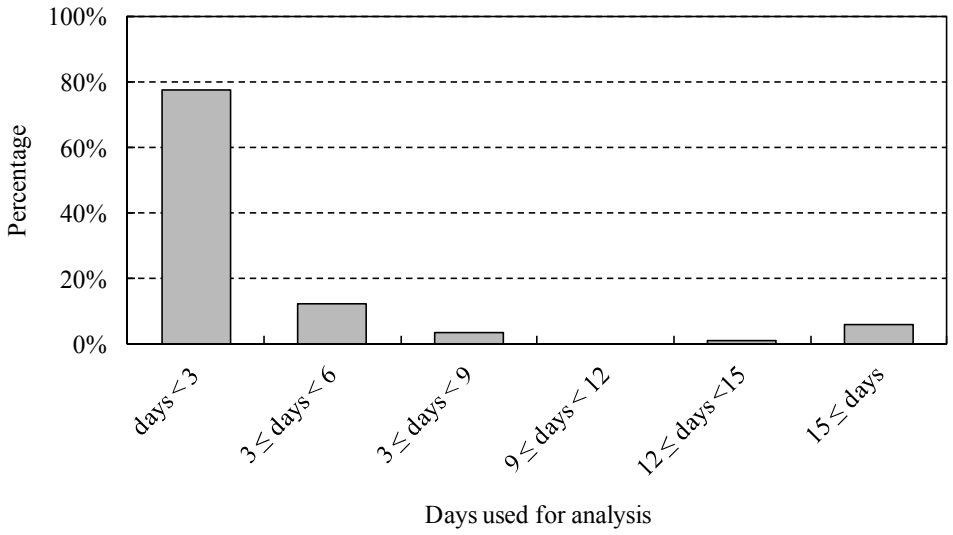


Figure 2.28 Histogram of Days used for analysis

2.4 Comparison with past surveys

Since the beginning of EANET, inter-laboratory comparison on wet deposition reached the 12th survey. The results showing the percentages of flagged data and percentage of data that satisfied the DQO were shown in Figure 2.29.

The percentages of data within DQO for the sample No. 091w and No. 092w were 96.4% and 89.3% respectively. These were the highest percentages since the beginning of inter-laboratory comparison on wet deposition. The accuracy of the measurement on wet deposition monitoring might have improved due to the continuous efforts and experiences in participating laboratories.

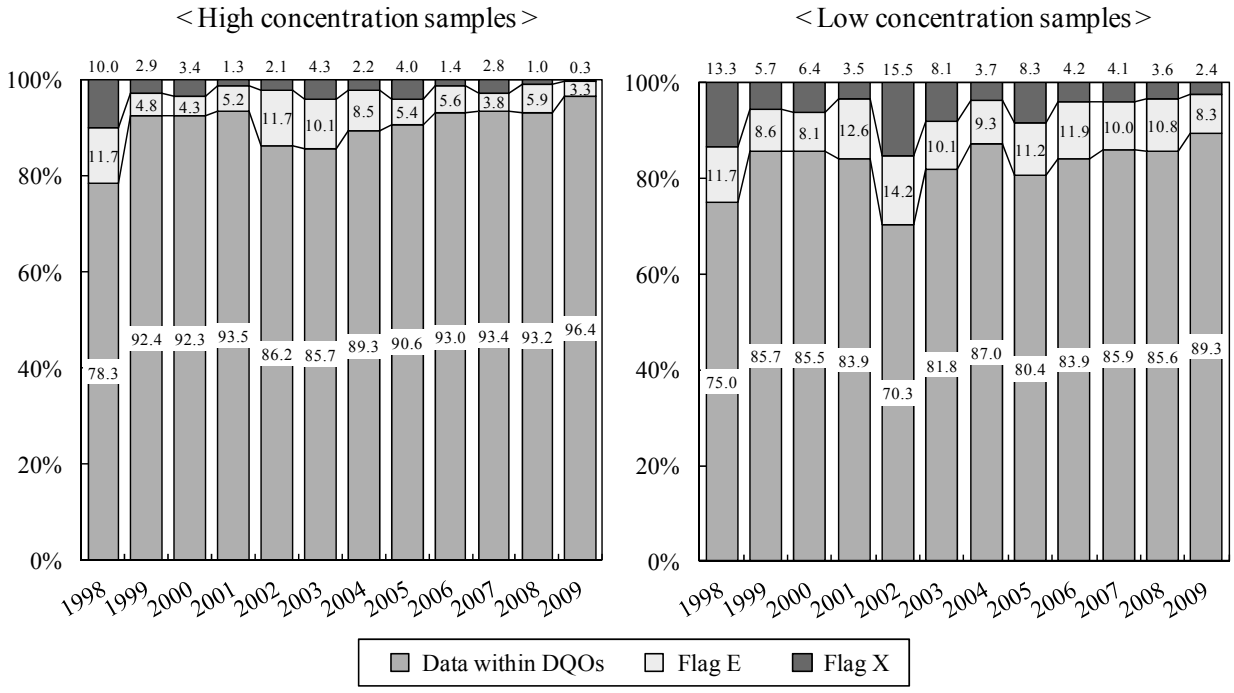


Figure 2.29 Comparison of results from the inter-laboratory comparison projects

Figure 2.30 showed the trend of the prepared values and the percentage of flagged data. Although the percentages of data marked with flags “E” and “X” decreased in most parameters compared with past surveys, the percentages were relatively high in cations as ever.

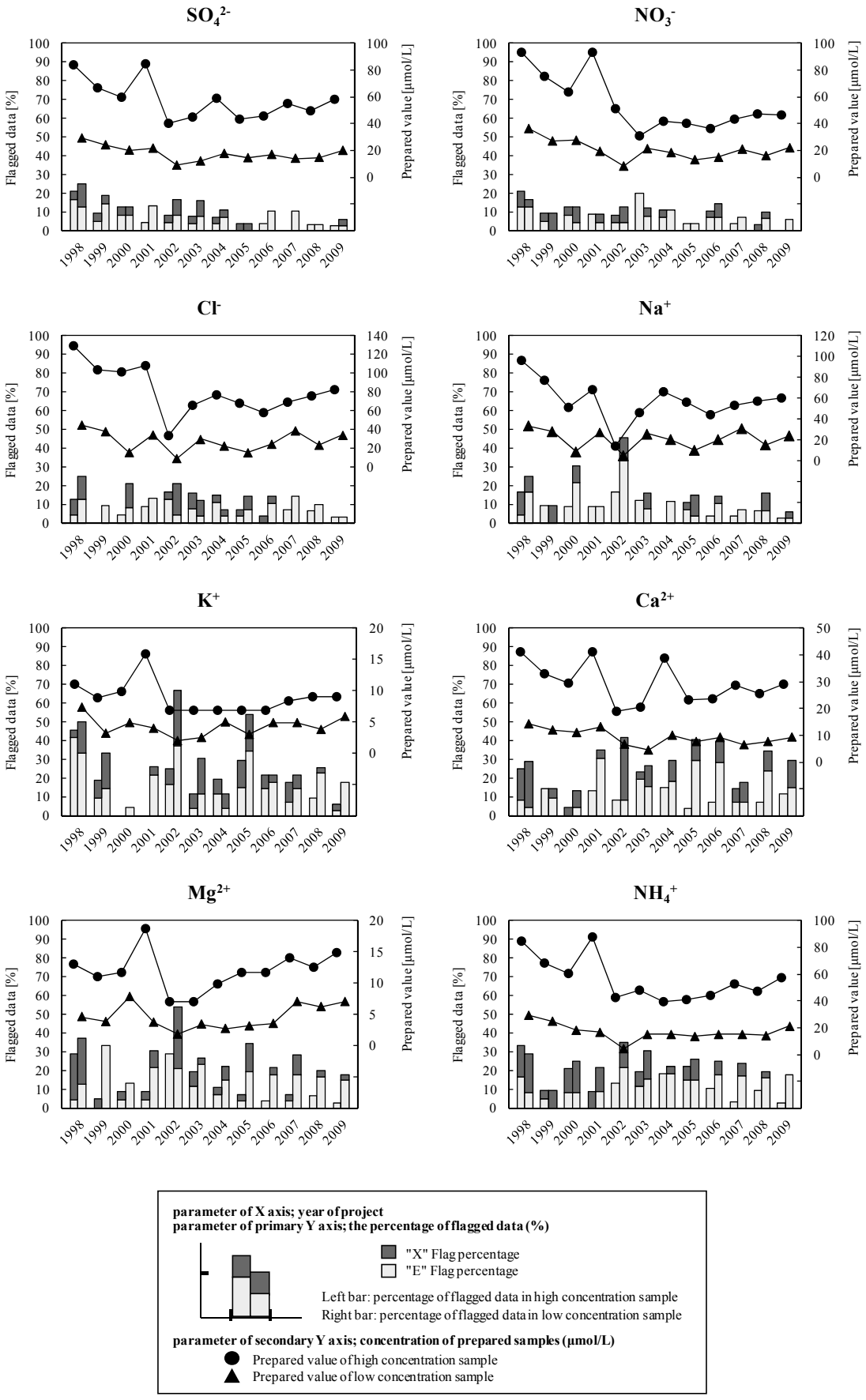


Figure 2.30 Comparison for each parameter in inter-laboratory comparison project

As shown in the figure 2.31, the number of data has increased since the beginning of the project. The total number of data reached 676 in this survey because the laboratory of MM01 started submitting analytical data of ions, and the number of participating laboratory increased in Viet Nam.

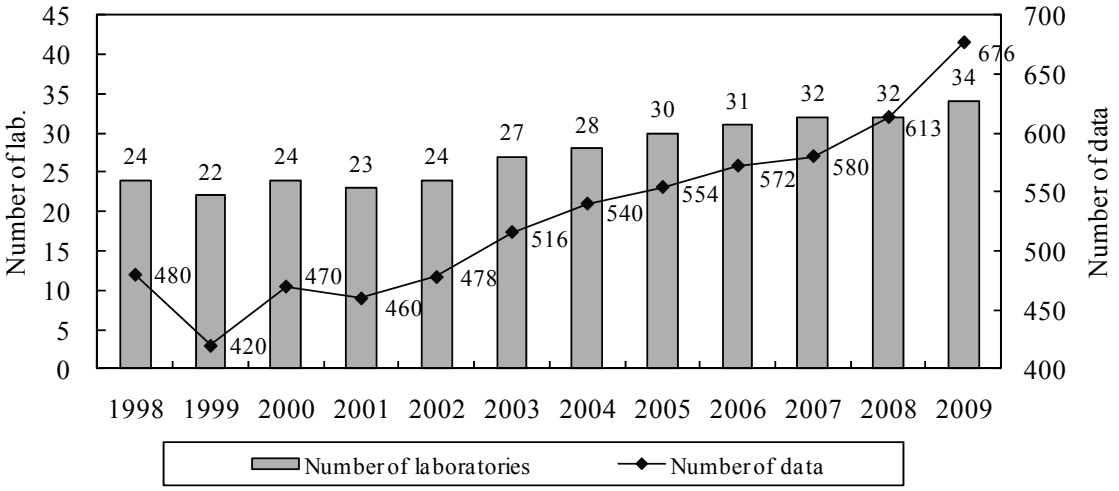


Figure 2.31 The number of participating laboratories and data in the inter-laboratory comparison projects on wet deposition

2.5 Recommendations for improvements

The fundamental matters for QA/QC on measurements and analyses of samples were described on the page 22 through 29 of the "Quality Assurance/Quality Control (QA/QC) Program for Wet Deposition Monitoring in East Asia".
Additionally, the NC showed the following matters for the improvement of data accuracy.

2.5.1 Measurement and Analysis

►After drawing calibration curves for the determination of ion concentrations, accuracy check by analyzing Reference Materials is recommended.
►Ranges of calibration curves need to cover the concentrations of the samples.

2.5.2 Data control

►After determining all the analytical parameters, data check by calculating R_1 and R_2 values are important. If the values exceed their allowable ranges, the data set is doubtful and reanalysis shall be carried out after rechecking analytical instruments and analytical procedures.

References

1) Second Interim Scientific Advisory Group Meeting of Acid Deposition Monitoring Network in East Asia. 2000. Guidelines for Acid Deposition Monitoring in East Asia

2) Second Interim Scientific Advisory Group Meeting of Acid Deposition Monitoring Network in East Asia. 2000. Technical Documents for Wet Deposition Monitoring in East Asia

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4) Acid Deposition and Oxidant Research Center. 2000. Report of the Inter-laboratory Comparison Project 1999 (Round robin analysis survey), 2nd Attempt.

5) Acid Deposition and Oxidant Research Center. 2001. Report of the Inter-laboratory Comparison Project 2000 on Wet Deposition, 3rd Attempt.

6) Acid Deposition and Oxidant Research Center. 2002. Report of the Inter-laboratory Comparison Project 2001 on Wet Deposition, 4th Attempt.

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9) Acid Deposition and Oxidant Research Center. 2005. Report of the Inter-laboratory Comparison Project 2004 on Wet Deposition, 7th Attempt.

10) Acid Deposition and Oxidant Research Center. 2006. Report of the Inter-laboratory Comparison Project 2005 on Wet Deposition, 8th Attempt.

11) Network Center for EANET. 2007. Report of the Inter-laboratory Comparison Project 2006

12) Network Center for EANET. 2008. Report of the Inter-laboratory Comparison Project 2007

13) Network Center for EANET. 2009. Report of the Inter-laboratory Comparison Project 2008

Appendix 2.1 Participating laboratories

CAMBODIA	**Code**
1) Department of Pollution Control, Ministry of Environment	**(KH01)**
CHINA	
2) Zhuhai Environmental Monitoring Station	**(CN01)**
3) Xiamen Environmental Monitoring Station	**(CN02)**
4) Xi'an Environmental Monitoring Station	**(CN03)**
5) Chongqing Institute of Environmental Science	**(CN04)**
INDONESIA	
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)**
JAPAN	
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 Deposition and Oxidant Research Center (ADORC)	**(JP07)**
LAO PDR	
16) Environment Quality Monitoring Center, Environment Research Institute, Science, Technology and Environment Agency	**(LA01)**
MALAYSIA	
17) Division of Environmental Health, Department of Chemistry	**(MY01)**
MONGOLIA	
18) Central Laboratory of Environmental Monitoring, National Agency for Meteorology, Hydrology and Environmental Monitoring, Ministry of Nature and Environment	**(MN01)**
MYANMAR	
19) Department of Meteorology and Hydrology (DMH)	**(MM01)**

PHILIPPINES	**Code**
20) Research and Development Division, Environmental Management Bureau (EMB-CO), Department of Environment and Natural Resources (DENR)	**(PH01)**
21) Environmental Management Bureau CAR (EMB-CAR),	**(PH02)**
REPUBLIC OF KOREA	
22) Atmospheric Chemistry Division, National Institute of Environment Research (NIER)	**(KR01)**
RUSSIA	
23) Limnological Institute, Russian Academy of Sciences, Siberian Branch (LI/RAS/SB)	**(RU01)**
24) Primorsky Center for Environmental Monitoring, Roshydromet (PCEM)	**(RU02)**
THAILAND	
25) Research and Training Centre (ERTC), Department of Research and Environmental Quality Promotion	**(TH01)**
26) Pollution Control Department (PCD) Ministry of Natural Resources and Environment (MONRE)	**(TH02)**
27) Meteorological Observation Division, Thailand Meteorological Department (TMD)	**(TH04)**
28) Chemistry Department, Science Faculty, Chiangmai University (CMU)	**(TH05)**
29) Khon Kaen University (KKU)	**(TH06)**
VIET NAM	
30) Environmental Laboratory – Center for Environmental Research – Vietnam Institute of Meteorology, Hydrology and Environment (IMHEN)- MoNRE – Vietnam	**(VN01)**
31) Middle of Central Regional Hydrometeorological Observatory, National Hydrometeorological Center (NHMS)	**(VN02)**
32) Sub-Institute of Hydrometeorology and Environment of South Vietnam (SIHYMETE)	**(VN03)**
33) Center for Hydrometeorological and Environmental Networks, National Hydrometeorological Service of Vietnam, MoNRE, Vietnam	**(VN04)**
34) Southern Region Hydrometeorological Center, National Hydrometeorological Service of Vietnam, MoNRE, Vietnam	**(VN05)**

Appendix 2.2 Analytical Results submitted by the laboratories

Appendix Table 2.2.1 Analytical data concerning high conc. sample No. 091w

Sample No. 091w (High concentrations)

Lab. ID	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	4.77	3.95	61.3	46.0	80.6	57.7	8.1	28.6	15.0	60.1
CN01	4.49	4.01	57.2	46.1	80.4	56.5	8.1	29.6	15.0	57.1
CN02	4.55	4.09	58.8	45.2	79.2	58.3	8.7	29.5	15.4	58.8
CN03	4.52	4.09	60.8	45.3	79.6	58.0	8.1	29.8	14.4	57.2
CN04	4.45	4.15	59.9	45.0	79.1	58.5	9.1	29.3	15.1	57.0
ID01	4.61	3.89	56.9	45.5	80.2	58.0	8.8	28.5	15.0	49.3
ID02	4.51	4.09	59.1	49.8	76.0	57.2	9.0	29.4	15.2	62.9
ID03	4.77	3.22	56.2	45.9	79.4	59.2	10.1	27.7	14.7	54.9
JP01	4.57	4.15	56.3	46.4	80.3	60.0	9.2	28.1	14.2	57.0
JP02	4.56	4.23	57.3	46.0	82.8	59.4	9.1	28.5	14.6	57.9
JP03	4.58	4.14	57.1	45.8	80.5	58.9	9.2	29.0	13.7	57.2
JP04	4.58	4.12	56.4	46.2	81.3	58.6	8.5	28.6	14.2	56.6
JP05	4.59	3.94	56.4	45.6	78.2	58.4	9.0	28.9	14.6	57.6
JP06	4.62	4.05	54.6	46.4	81.3	60.3	9.1	29.4	14.7	53.7
JP07	4.59	3.96	57.6	45.5	82.8	60.4	8.9	28.3	14.6	56.2
LA01	4.45	3.95	57.8	46.0	82.6	59.3	8.9	27.5	13.7	57.6
MY01	4.60	4.03	56.5	46.2	80.4	61.4	9.7	30.6	13.7	53.8
MN01	4.50	4.13	56.3	45.4	76.3	59.2	8.9	27.9	13.2	55.6
MM01	4.33	3.97	60.6	48.6	87.9	58.6	8.6	28.6	14.4	59.0
PH01	4.59	4.02	64.8	47.7	88.3	63.8	9.8	27.5	13.0	47.3
PH02	4.44	4.04	61.9	48.0	74.5	67.7	9.7	35.8	15.8	58.6
KR01	4.25	3.99	56.5	46.1	79.9	55.7	9.9	28.3	15.0	58.3
RU01	4.57	4.06	55.5	45.9	81.4	58.5	8.5	27.2	15.3	55.5
RU02	4.55	4.09	53.2	45.8	77.0	59.1	8.3	28.8	16.2	56.4
TH01	4.62	4.05	57.2	45.7	80.1	60.2	8.7	30.7	14.7	56.5
TH02	4.56	4.08	57.3	44.2	76.5	61.7	9.0	30.7	14.5	59.9
TH04	4.41	4.09	60.9	46.8	76.9	59.6	8.8	27.3	12.8	49.5
TH05	4.50	4.07	55.2	44.0	76.3	58.0	8.7	34.0	16.1	56.5
TH06	4.56	4.04	56.4	42.5	77.2	60.8	9.6	29.5	17.1	63.3
VN01	4.54	4.17	58.9	47.2	82.7	59.6	8.9	28.6	14.1	57.5
VN02	4.50	3.97	57.5	46.6	79.8	59.1	8.8	28.5	14.1	55.2
VN03	4.73	3.62	47.8	---	---	59.7	11.3	29.0	15.4	62.9
VN04	4.70	3.94	51.6	46.7	95.8	55.9	12.4	35.9	14.4	57.4
VN05	4.58	3.98	61.4	45.5	80.0	49.0	10.3	37.3	14.9	55.7
Prepared value	**4.52**	**4.23**	**57.8**	**46.7**	**81.9**	**59.9**	**9.1**	**29.0**	**14.8**	**57.5**
Number of data	34	33	33	33	32	32	33	33	34	34
Average	4.55	4.03	57.7	46.0	80.0	59.1	9.1	29.4	14.7	56.8
Minimum	4.25	3.62	51.6	42.5	74.5	55.7	8.1	27.2	12.8	47.3
Maximum	4.77	4.23	64.8	49.8	88.3	63.8	11.3	35.9	17.1	63.3
Standard deviation	0.11	0.11	2.62	1.28	2.98	1.61	0.67	2.07	0.88	3.36

Note: The outliers judged by 3S.D. method were painted with light mesh and were excluded from statistics;
"---", Not measured

Appendix Table 2.2.2 Analytical data concerning low conc. sample No. 092w

Sample No. 092w (Low concentrations)

Lab. ID	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	4.35	1.55	20.0	21.1	32.0	23.0	5.1	9.7	7.7	22.2
CN01	4.88	1.59	19.9	21.2	33.0	23.1	5.4	10.6	7.8	23.1
CN02	4.94	1.60	20.0	20.8	32.4	24.8	5.9	11.5	8.3	23.9
CN03	4.90	1.68	20.9	21.1	32.9	24.2	5.2	9.8	7.0	22.5
CN04	4.87	1.68	21.0	21.7	33.2	25.2	6.0	10.7	7.8	23.7
ID01	5.18	1.57	20.1	21.7	33.5	22.9	6.5	9.7	7.0	19.6
ID02	5.02	1.58	20.3	22.0	30.3	21.2	5.6	11.9	7.8	22.1
ID03	5.22	1.38	19.1	21.6	32.6	25.2	6.8	10.1	7.2	21.2
JP01	4.98	1.71	19.8	21.6	33.5	23.3	6.3	8.9	6.7	20.7
JP02	4.95	1.64	19.7	21.8	33.7	23.4	5.9	9.1	6.9	21.1
JP03	4.95	1.65	19.8	21.6	33.6	23.7	6.2	10.0	6.8	20.7
JP04	4.95	1.71	20.3	23.2	41.2	23.4	5.6	9.0	6.9	22.3
JP05	5.03	1.59	19.9	21.9	34.0	24.1	5.9	9.3	6.9	21.6
JP06	5.08	1.59	19.7	21.7	33.3	24.4	6.1	9.8	7.1	21.7
JP07	4.99	1.60	19.3	21.2	32.8	24.0	5.9	9.4	6.8	20.7
LA01	4.86	1.60	20.4	22.3	35.2	24.2	5.8	8.8	6.2	21.9
MY01	5.03	1.57	18.8	21.1	34.3	25.0	6.1	9.6	6.5	21.9
MN01	4.88	1.66	19.2	21.2	31.3	24.1	4.9	8.8	4.5	19.4
MM01	5.33	1.63	20.6	23.0	35.8	23.4	5.6	9.4	6.9	23.6
PH01	4.91	1.65	18.7	19.7	31.0	23.8	6.5	5.3	7.4	17.0
PH02	4.80	1.59	23.9	23.5	31.5	32.5	6.4	18.5	8.6	22.5
KR01	4.80	1.72	19.5	21.6	33.5	20.7	6.8	9.0	7.6	25.6
RU01	4.86	1.61	19.1	21.8	34.7	24.4	5.6	8.8	7.2	19.6
RU02	4.93	1.67	18.5	21.9	32.1	22.6	6.0	8.6	7.8	19.9
TH01	4.98	1.66	19.5	21.4	33.2	24.5	5.8	11.1	7.5	22.2
TH02	4.95	1.51	18.6	19.4	30.4	24.2	5.8	8.3	6.4	22.8
TH04	4.73	1.69	19.9	21.5	31.5	27.5	7.1	9.3	6.5	17.5
TH05	5.32	1.64	18.9	20.3	31.5	23.8	5.5	13.1	8.1	20.8
TH06	5.00	1.56	20.0	19.6	32.7	23.2	5.7	10.2	7.9	26.2
VN01	4.96	1.66	20.3	21.6	30.7	24.7	6.2	9.0	7.0	21.0
VN02	4.91	1.67	19.8	21.5	34.4	22.6	7.5	8.9	6.9	19.3
VN03	5.13	1.55	20.6	---	---	21.2	6.0	6.8	6.6	27.4
VN04	5.23	1.58	22.5	18.3	38.5	21.9	7.0	14.0	8.1	24.4
VN05	4.97	1.64	26.0	18.5	32.7	18.6	6.1	12.4	8.3	22.8
Prepared value	**4.92**	**1.66**	**19.9**	**22.0**	**34.0**	**24.0**	**5.9**	**9.3**	**7.0**	**21.2**
Number of data	33	33	33	33	32	33	34	33	33	34
Average	4.99	1.62	20.0	21.3	33.0	23.5	6.0	9.7	7.3	21.9
Minimum	4.73	1.51	18.5	18.3	30.3	18.6	4.9	5.3	6.2	17.0
Maximum	5.33	1.72	23.9	23.5	38.5	27.5	7.5	14.0	8.6	27.4
Standard deviation	0.14	0.05	1.05	1.13	1.66	1.56	0.56	1.62	0.61	2.18

Note: The outliers judged by 3S.D. method were painted with light mesh and were excluded from statistics;
"---", Not measured

Appendix 2.3 Normalized Data

Deviation from prepared value (Va/Vp): (Average (Va) / Prepared value (Vp) – 1) x 100 [%]

Appendix Table 2.3.1 Deviation% from prepared values of high conc. sample No. 091w

Sample No. 091w (High concentrations)

Lab. ID	pH	EC	SO_4^{2-}	NO_3^-	Cl^-	Na^+	K^+	Ca^{2+}	Mg^{2+}	NH_4^+
	%	%	%	%	%	%	%	%	%	%
KH01	5.5	-6.6	6.1	-1.5	-1.6	-3.7	-11.0	-1.4	1.4	4.5
CN01	-0.7	-5.2	-1.0	-1.3	-1.8	-5.7	-11.0	2.1	1.4	-0.7
CN02	0.7	-3.3	1.7	-3.2	-3.3	-2.7	-4.4	1.7	4.1	2.3
CN03	0.0	-3.3	5.2	-3.0	-2.8	-3.2	-11.0	2.8	-2.7	-0.5
CN04	-1.5	-1.9	3.6	-3.6	-3.4	-2.3	0.0	1.0	2.0	-0.9
ID01	2.0	-8.0	-1.6	-2.6	-2.1	-3.2	-3.3	-1.7	1.4	-14.3
ID02	-0.2	-3.3	2.2	6.6	-7.2	-4.5	-1.1	1.4	2.7	9.4
ID03	5.5	-23.9	-2.8	-1.7	-3.1	-1.2	11.0	-4.5	-0.7	-4.5
JP01	1.1	-1.9	-2.6	-0.6	-2.0	0.2	1.1	-3.1	-4.1	-0.9
JP02	0.9	0.0	-0.9	-1.5	1.1	-0.8	0.0	-1.7	-1.4	0.7
JP03	1.3	-2.1	-1.2	-1.9	-1.7	-1.7	1.1	0.0	-7.4	-0.5
JP04	1.3	-2.6	-2.4	-1.1	-0.7	-2.2	-6.6	-1.4	-4.1	-1.6
JP05	1.5	-6.9	-2.4	-2.4	-4.5	-2.5	-1.1	-0.3	-1.4	0.2
JP06	2.2	-4.3	-5.5	-0.6	-0.7	0.7	0.0	1.4	-0.7	-6.6
JP07	1.5	-6.4	-0.3	-2.6	1.1	0.8	-2.2	-2.4	-1.4	-2.3
LA01	-1.5	-6.6	0.0	-1.5	0.9	-1.0	-2.2	-5.2	-7.4	0.2
MY01	1.8	-4.7	-2.2	-1.1	-1.8	2.5	6.6	5.5	-7.4	-6.4
MN01	-0.4	-2.4	-2.6	-2.8	-6.8	-1.2	-2.2	-3.8	-10.8	-3.3
MM01	-4.2	-6.1	4.8	4.1	7.3	-2.2	-5.5	-1.4	-2.7	2.6
PH01	1.5	-5.0	12.1	2.1	7.8	6.5	7.7	-5.2	-12.2	-17.7
PH02	-1.8	-4.5	7.1	2.8	-9.0	13.0	6.6	23.4	6.8	1.9
KR01	-6.0	-5.7	-2.2	-1.3	-2.4	-7.0	8.8	-2.4	1.4	1.4
RU01	1.1	-4.0	-4.0	-1.7	-0.6	-2.3	-6.6	-6.2	3.4	-3.5
RU02	0.7	-3.3	-8.0	-1.9	-6.0	-1.3	-8.8	-0.7	9.5	-1.9
TH01	2.2	-4.3	-1.0	-2.1	-2.2	0.5	-4.4	5.9	-0.7	-1.7
TH02	0.9	-3.5	-0.9	-5.4	-6.6	3.0	-1.1	5.9	-2.0	4.2
TH04	-2.4	-3.3	5.4	0.2	-6.1	-0.5	-3.3	-5.9	-13.5	-13.9
TH05	-0.4	-3.8	-4.5	-5.8	-6.8	-3.2	-4.4	17.2	8.8	-1.7
TH06	0.9	-4.5	-2.4	-9.0	-5.7	1.5	5.5	1.7	15.5	10.1
VN01	0.4	-1.4	1.9	1.1	1.0	-0.5	-2.2	-1.4	-4.7	0.0
VN02	-0.4	-6.1	-0.5	-0.2	-2.6	-1.3	-3.3	-1.7	-4.7	-4.0
VN03	4.6	-14.4	-17.3	---	---	-0.3	24.2	0.0	4.1	9.4
VN04	4.0	-6.9	-10.7	0.0	17.0	-6.7	36.3	23.8	-2.7	-0.2
VN05	1.3	-5.9	6.2	-2.6	-2.3	-18.2	13.2	28.6	0.7	-3.1
Number of data	34	34	34	33	33	34	34	34	34	34
Average	0.7	-5.2	-0.6	-1.4	-1.8	-1.5	0.8	2.1	-0.9	-1.3
Minimum	-6.0	-23.9	-17.3	-9.0	-9.0	-18.2	-11.0	-6.2	-13.5	-17.7
Maximum	5.5	0.0	12.1	6.6	17.0	13.0	36.3	28.6	15.5	10.1

Note: "---", Not measured

Appendix Table 2.3.2 Deviation% from prepared values of low conc. sample No. 092w

Sample No. 092w (Low concentrations)

Lab. ID	pH	EC	SO_4^{2-}	NO_3^-	Cl^-	Na^+	K^+	Ca^{2+}	Mg^{2+}	NH_4^+
	%	%	%	%	%	%	%	%	%	%
KH01	-11.6	-6.6	0.5	-4.1	-5.9	-4.2	-13.6	4.3	10.0	4.7
CN01	-0.8	-4.2	0.0	-3.6	-2.9	-3.8	-8.5	14.0	11.4	9.0
CN02	0.4	-3.6	0.5	-5.5	-4.7	3.3	0.0	23.7	18.6	12.7
CN03	-0.4	1.2	5.0	-4.1	-3.2	0.8	-11.9	5.4	0.0	6.1
CN04	-1.0	1.2	5.5	-1.4	-2.4	5.0	1.7	15.1	11.4	11.8
ID01	5.3	-5.4	1.0	-1.4	-1.5	-4.6	10.2	4.3	0.0	-7.5
ID02	2.0	-4.8	2.0	0.0	-10.9	-11.7	-5.1	28.0	11.4	4.2
ID03	6.1	-16.9	-4.0	-1.8	-4.1	5.0	15.3	8.6	2.9	0.0
JP01	1.2	3.0	-0.5	-1.8	-1.5	-2.9	6.8	-4.3	-4.3	-2.4
JP02	0.6	-1.2	-1.0	-0.9	-0.9	-2.5	0.0	-2.2	-1.4	-0.5
JP03	0.6	-0.6	-0.5	-1.8	-1.2	-1.3	5.1	7.5	-2.9	-2.4
JP04	0.6	3.0	2.0	5.5	21.2	-2.5	-5.1	-3.2	-1.4	5.2
JP05	2.2	-4.2	0.0	-0.5	0.0	0.4	0.0	0.0	-1.4	1.9
JP06	3.3	-4.2	-1.0	-1.4	-2.1	1.7	3.4	5.4	1.4	2.4
JP07	1.4	-3.6	-3.0	-3.6	-3.5	0.0	0.0	1.1	-2.9	-2.4
LA01	-1.2	-3.6	2.5	1.4	3.5	0.8	-1.7	-5.4	-11.4	3.3
MY01	2.2	-5.4	-5.5	-4.1	0.9	4.2	3.4	3.2	-7.1	3.3
MN01	-0.8	0.0	-3.5	-3.6	-7.9	0.4	-16.9	-5.4	-35.7	-8.5
MM01	8.3	-1.8	3.5	4.5	5.3	-2.5	-5.1	1.1	-1.4	11.3
PH01	-0.2	-0.6	-6.0	-10.5	-8.8	-0.8	10.2	-43.0	5.7	-19.8
PH02	-2.4	-4.2	20.1	6.8	-7.4	35.4	8.5	98.9	22.9	6.1
KR01	-2.4	3.6	-2.0	-1.8	-1.5	-13.8	15.3	-3.2	8.6	20.8
RU01	-1.2	-3.0	-4.0	-0.9	2.1	1.7	-5.1	-5.4	2.9	-7.5
RU02	0.2	0.6	-7.0	-0.5	-5.6	-5.8	1.7	-7.5	11.4	-6.1
TH01	1.2	0.0	-2.0	-2.7	-2.4	2.1	-1.7	19.4	7.1	4.7
TH02	0.6	-9.0	-6.5	-11.8	-10.6	0.8	-1.7	-10.8	-8.6	7.5
TH04	-3.9	1.8	0.0	-2.3	-7.4	14.6	20.3	0.0	-7.1	-17.5
TH05	8.1	-1.2	-5.0	-7.7	-7.4	-0.8	-6.8	40.9	15.7	-1.9
TH06	1.6	-6.0	0.5	-10.9	-3.8	-3.3	-3.4	9.7	12.9	23.6
VN01	0.8	0.0	2.0	-1.8	-9.7	2.9	5.1	-3.2	0.0	-0.9
VN02	-0.2	0.6	-0.5	-2.3	1.2	-5.8	27.1	-4.3	-1.4	-9.0
VN03	4.3	-6.6	3.5	---	---	-11.7	1.7	-26.9	-5.7	29.2
VN04	6.3	-4.8	13.1	-16.8	13.2	-8.8	18.6	50.5	15.7	15.1
VN05	1.0	-1.2	30.7	-15.9	-3.8	-22.5	3.4	33.3	18.6	7.5
Number of data	34	34	34	33	33	34	34	34	34	34
Average	1.0	-2.6	1.2	-3.3	-2.2	-0.9	2.1	7.3	2.8	3.1
Minimum	-11.6	-16.9	-7.0	-16.8	-10.9	-22.5	-16.9	-43.0	-35.7	-19.8
Maximum	8.3	3.6	30.7	6.8	21.2	35.4	27.1	98.9	22.9	29.2

Note: "---", Not measured

Appendix 2.4 Z-score evaluation

The NC applied Z-score for further statistical evaluation of the analytical values in the inter-laboratory comparison on wet deposition.

- **Definition of Z-score**

Z-score is one of the statistical measures that quantify the distance from the mean of a data set. The formula for the calculation of Z-score (Robust method) was shown below:

$$Z = \frac{X - Q_2}{0.7413 \times (Q_3 - Q_1)}$$

where X: Measurement values of samples
Q_1: The 1st quartile value of entire data
Q_2: The 2nd quartile value of entire data (i.e. Median)
Q_3: The 3rd quartile value of entire data

- **Evaluation of calculated Z-score**

Z-score was given to each data submitted by the participating laboratories, and was evaluated as follows:

$|Z| \leq 2$: Satisfactory
$2 < |Z| < 3$: Questionable
$3 \leq |Z|$: Unsatisfactory

The calculated Z-scores were shown in Appendix Table 2.4.1 and 2.4.2, and were also graphed in Appendix Figure 2.4.1 through 2.4.10.

Appendix Table 2.4.1 Results of Z-score evaluation for sample No. 091w

Sample No. 091w (High concentrations)

Lab. ID	pH	EC	SO_4^{2-}	NO_3^-	Cl^-	Na^+	K^+	Ca^{2+}	Mg^{2+}	NH_4^+
KH01	3.15	-1.07	2.03	0.00	0.22	-1.02	-1.43	-0.28	0.54	1.60
CN01	-1.05	-0.39	0.00	0.15	0.13	-1.90	-1.43	0.83	0.54	0.03
CN02	-0.15	0.51	0.79	-1.20	-0.39	-0.58	-0.42	0.72	1.16	0.92
CN03	-0.60	0.51	1.78	-1.05	-0.22	-0.80	-1.43	1.05	-0.39	0.08
CN04	-1.65	1.18	1.34	-1.50	-0.44	-0.44	0.25	0.50	0.69	-0.03
ID01	0.75	-1.74	-0.15	-0.75	0.04	-0.80	-0.25	-0.39	0.54	-4.06
ID02	-0.75	0.51	0.94	5.70	-1.78	-1.39	0.08	0.61	0.85	3.06
ID03	3.15	-9.27	-0.50	-0.15	-0.30	0.07	1.94	-1.27	0.08	-1.13
JP01	0.15	1.18	-0.45	0.60	0.09	0.66	0.42	-0.83	-0.69	-0.03
JP02	0.00	2.08	0.05	0.00	1.17	0.22	0.25	-0.39	-0.08	0.45
JP03	0.30	1.07	-0.05	-0.30	0.17	-0.15	0.42	0.17	-1.46	0.08
JP04	0.30	0.84	-0.40	0.30	0.52	-0.36	-0.76	-0.28	-0.69	-0.24
JP05	0.45	-1.18	-0.40	-0.60	-0.83	-0.51	0.08	0.06	-0.08	0.29
JP06	0.90	0.06	-1.29	0.60	0.52	0.88	0.25	0.61	0.08	-1.75
JP07	0.45	-0.96	0.20	-0.75	1.17	0.95	-0.08	-0.61	-0.08	-0.45
LA01	-1.65	-1.07	0.30	0.00	1.09	0.15	-0.08	-1.49	-1.46	0.29
MY01	0.60	-0.17	-0.35	0.30	0.13	1.68	1.26	1.93	-1.46	-1.70
MN01	-0.90	0.96	-0.45	-0.90	-1.65	0.07	-0.08	-1.05	-2.24	-0.76
MM01	-3.45	-0.84	1.68	3.90	3.39	-0.36	-0.59	-0.28	-0.39	1.02
PH01	0.45	-0.28	3.76	2.55	3.57	3.43	1.43	-1.49	-2.54	-5.11
PH02	-1.80	-0.06	2.33	3.00	-2.44	6.27	1.26	7.65	1.77	0.81
KR01	-4.65	-0.62	-0.35	0.15	-0.09	-2.48	1.60	-0.61	0.54	0.65
RU01	0.15	0.17	-0.84	-0.15	0.57	-0.44	-0.76	-1.82	1.00	-0.81
RU02	-0.15	0.51	-1.98	-0.30	-1.35	0.00	-1.10	-0.06	2.39	-0.34
TH01	0.90	0.06	0.00	-0.45	0.00	0.80	-0.42	2.04	0.08	-0.29
TH02	0.00	0.39	0.05	-2.70	-1.57	1.90	0.08	2.04	-0.23	1.49
TH04	-2.25	0.51	1.83	1.20	-1.39	0.36	-0.25	-1.71	-2.85	-3.96
TH05	-0.90	0.28	-0.99	-3.00	-1.65	-0.80	-0.42	5.67	2.24	-0.29
TH06	0.00	-0.06	-0.40	-5.25	-1.26	1.24	1.10	0.72	3.78	3.27
VN01	-0.30	1.41	0.84	1.80	1.13	0.36	-0.08	-0.28	-0.85	0.24
VN02	-0.90	-0.84	0.15	0.90	-0.13	0.00	-0.25	-0.39	-0.85	-0.97
VN03	2.55	-4.78	-4.65	---	---	0.44	3.96	0.17	1.16	3.06
VN04	2.10	-1.18	-2.77	1.05	6.83	-2.33	5.82	7.76	-0.39	0.18
VN05	0.30	-0.73	2.08	-0.75	-0.04	-7.36	2.28	9.31	0.39	-0.71
Number of data	34	34	34	33	33	34	34	34	34	34
$\|Z\| \leq 2$	27	31	28	26	29	29	31	28	28	28
$2 < \|Z\| < 3$	3	1	4	4	1	2	1	2	5	0
$3 \leq \|Z\|$	4	2	2	3	3	3	2	4	1	6

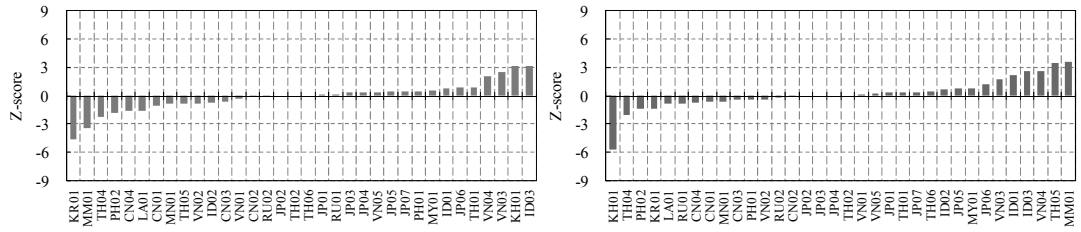
Note: Light mesh, $|Z| > 2$ (Questionable); Dark mesh, $|Z| \geq 3$ (Unsatisfactory); "---", Not measured

Appendix Table 2.4.2 Results of Z-score evaluation for sample No. 092w

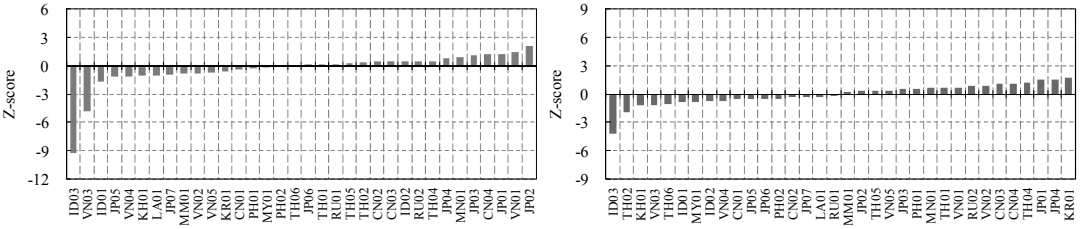
Sample No. 092w (Low concentrations)

Lab. ID	pH	EC	SO_4^{2-}	NO_3^-	Cl^-	Na^+	K^+	Ca^{2+}	Mg^{2+}	NH_4^+
KH01	-5.68	-1.22	0.14	-0.96	-0.79	-0.78	-1.76	0.17	0.90	0.19
CN01	-0.66	-0.52	0.00	-0.77	0.00	-0.69	-1.14	0.94	1.04	0.77
CN02	-0.09	-0.35	0.14	-1.54	-0.48	0.98	-0.10	1.71	1.73	1.28
CN03	-0.47	1.04	1.42	-0.96	-0.08	0.39	-1.56	0.26	-0.07	0.39
CN04	-0.76	1.04	1.56	0.19	0.16	1.37	0.10	1.03	1.04	1.16
ID01	2.18	-0.87	0.28	0.19	0.40	-0.88	1.14	0.17	-0.07	-1.48
ID02	0.66	-0.70	0.57	0.77	-2.14	-2.55	-0.73	2.06	1.04	0.13
ID03	2.56	-4.18	-1.14	0.00	-0.32	1.37	1.76	0.51	0.21	-0.45
JP01	0.28	1.57	-0.14	0.00	0.40	-0.49	0.73	-0.51	-0.48	-0.77
JP02	0.00	0.35	-0.28	0.39	0.56	-0.39	-0.10	-0.34	-0.21	-0.51
JP03	0.00	0.52	-0.14	0.00	0.48	-0.10	0.52	0.43	-0.35	-0.77
JP04	0.00	1.57	0.57	3.08	6.51	-0.39	-0.73	-0.43	-0.21	0.26
JP05	0.76	-0.52	0.00	0.58	0.79	0.29	-0.10	-0.17	-0.21	-0.19
JP06	1.23	-0.52	-0.28	0.19	0.24	0.59	0.31	0.26	0.07	-0.13
JP07	0.38	-0.35	-0.85	-0.77	-0.16	0.20	-0.10	-0.09	-0.35	-0.77
LA01	-0.85	-0.35	0.71	1.35	1.75	0.39	-0.31	-0.60	-1.18	0.00
MY01	0.76	-0.87	-1.56	-0.96	1.03	1.18	0.31	0.09	-0.76	0.00
MN01	-0.66	0.70	-0.99	-0.77	-1.35	0.29	-2.18	-0.60	-3.53	-1.61
MM01	3.60	0.17	0.99	2.70	2.22	-0.39	-0.73	-0.09	-0.21	1.09
PH01	-0.38	0.52	-1.70	-3.66	-1.59	0.00	1.14	-3.60	0.48	-3.15
PH02	-1.42	-0.52	5.68	3.66	-1.19	8.54	0.93	7.71	2.14	0.39
KR01	-1.42	1.74	-0.57	0.00	0.40	-3.04	1.76	-0.43	0.76	2.38
RU01	-0.85	-0.17	-1.14	0.39	1.35	0.59	-0.73	-0.60	0.21	-1.48
RU02	-0.19	0.87	-1.99	0.58	-0.71	-1.18	0.10	-0.77	1.04	-1.28
TH01	0.28	0.70	-0.57	-0.39	0.16	0.69	-0.31	1.37	0.62	0.19
TH02	0.00	-1.91	-1.85	-4.24	-2.06	0.39	-0.31	-1.03	-0.90	0.58
TH04	-2.08	1.22	0.00	-0.19	-1.19	3.63	2.39	-0.17	-0.76	-2.83
TH05	3.50	0.35	-1.42	-2.51	-1.19	0.00	-0.93	3.08	1.45	-0.71
TH06	0.47	-1.04	0.14	-3.85	-0.24	-0.59	-0.52	0.60	1.18	2.76
VN01	0.09	0.70	0.57	0.00	-1.83	0.88	0.52	-0.43	-0.07	-0.58
VN02	-0.38	0.87	-0.14	-0.19	1.11	-1.18	3.22	-0.51	-0.21	-1.67
VN03	1.70	-1.22	0.99	---	---	-2.55	0.10	-2.31	-0.62	3.53
VN04	2.65	-0.70	3.69	-6.36	4.36	-1.86	2.18	3.85	1.45	1.61
VN05	0.19	0.35	8.66	-5.97	-0.24	-5.10	0.31	2.48	1.73	0.58
Number of data	34	34	34	33	33	34	34	34	34	34
$\|Z\| \leq 2$	27	33	31	24	28	28	30	27	32	29
$2 < \|Z\| < 3$	4	0	0	2	3	2	3	3	1	3
$3 \leq \|Z\|$	3	1	3	7	2	4	1	4	1	2

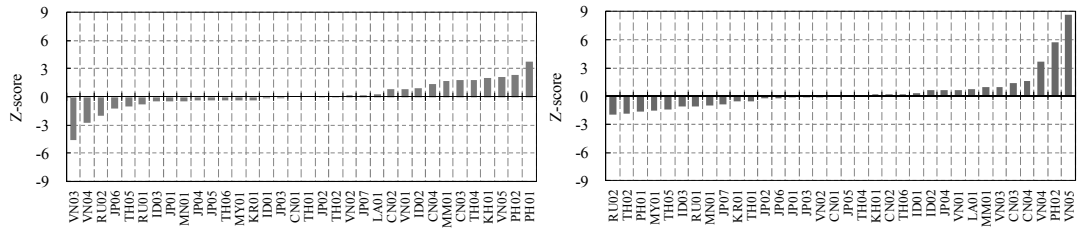
Note: Light mesh, $|Z| > 2$ (Questionable); Dark mesh, $|Z| \geq 3$ (Unsatisfactory); "---", Not measured



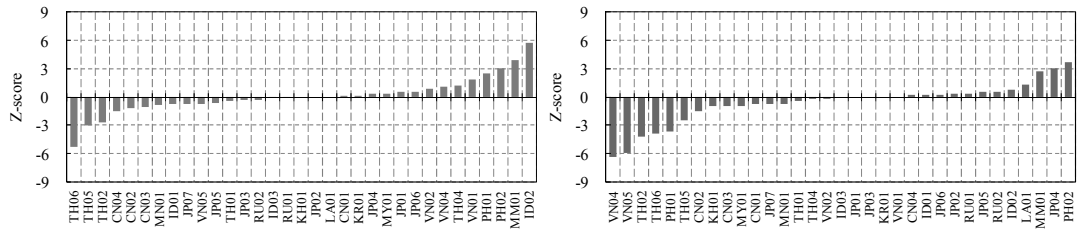
Appendix Figure 2.4.1 Distribution of Z-score for pH (Left: 091w, Right: 092w)



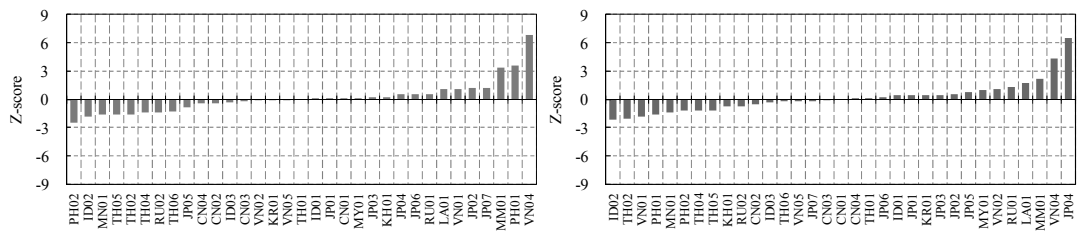
Appendix Figure 2.4.2 Distribution of Z-score for EC (Left: 091w, Right: 092w)



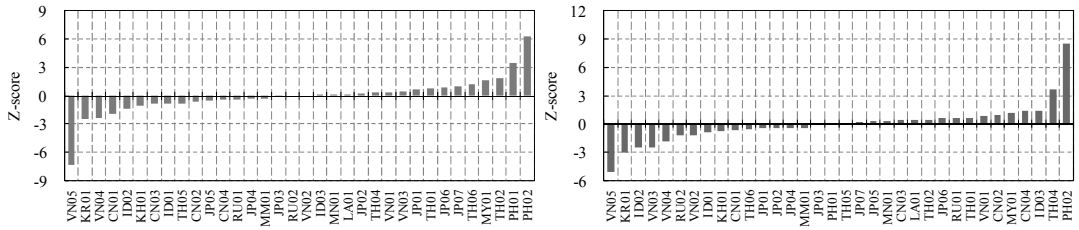
Appendix Figure 2.4.3 Distribution of Z-score for SO_4^{2-} (Left: 091w, Right: 092w)



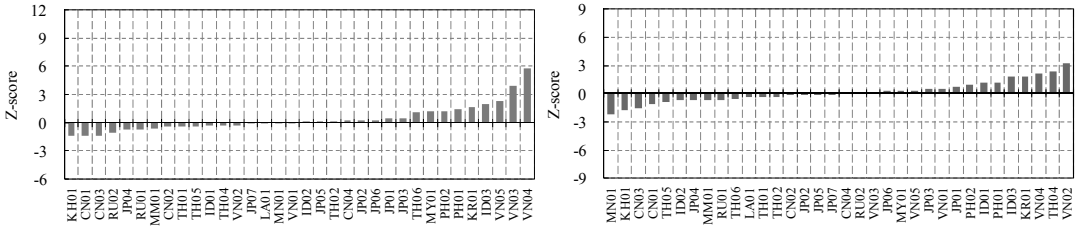
Appendix Figure 2.4.4 Distribution of Z-score for NO_3^- (Left: 091w, Right: 092w)



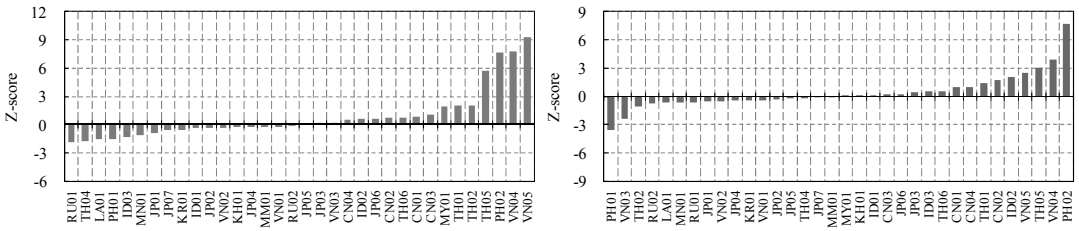
Appendix Figure 2.4.5 Distribution of Z-score for Cl^- (Left: 091w, Right: 092w)



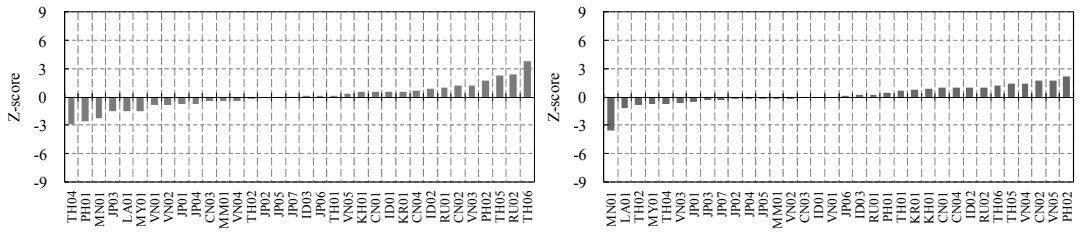
Appendix Figure 2.4.6 Distribution of Z-score for Na^+ (Left: 091w, Right: 092w)



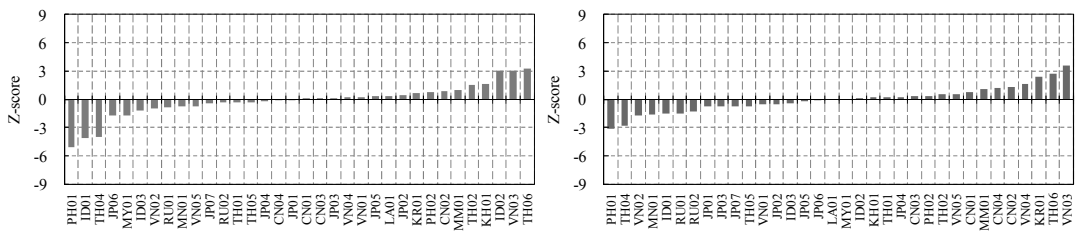
Appendix Figure 2.4.7 Distribution of Z-score for K^+ (Left: 091w, Right: 092w)



Appendix Figure 2.4.8 Distribution of Z-score for Ca^{2+} (Left: 091w, Right: 092w)



Appendix Figure 2.4.9 Distribution of Z-score for Mg^{2+} (Left: 091w, Right: 092w)



Appendix Figure 2.4.10 Distribution of Z-score for NH_4^+ (Left: 091w, Right: 092w)

3. 5th INTER-LABORATORY COMPARISON PROJECT ON DRY DEPOSITION

3.1 Introduction

In the Inter-laboratory Comparison for dry deposition, impregnated filters which contained either $SO_4^{2-}$and Cl^-, or NH_4^+ were prepared and distributed to the participating laboratories by the Network Center (NC) in October 2009. Most of the laboratories participating in EANET joined this activity and submitted their analytical results to the NC. These results for SO_4^{2-}, Cl^-, and NH_4^+ on the filter were compared with the corresponding prepared value and statistically analyzed.

3.2 Procedures

3.2.1 Participating Laboratories

A total of 24 laboratories in charge of EANET monitoring in 11 countries of EANET participated in this fifth activity. All participating laboratories with their abbreviated names and codes are listed in Appendix 3-1.

3.2.2 Description of Samples

Two kinds of sample filters, one contained two ions ($SO_4^{2-}$and Cl^-), the other contained one ion (NH_4^+), were prepared and distributed to the laboratories. The blank filters, which were impregnated with K_2CO_3 or H_3PO_4 but did not contain any SO_4^{2-}, Cl^-, or NH_4^+, were also prepared and distributed. The details of the sample filters were described in Table 3.1. The analytical precision and accuracy on the individual analyte were summarized through statistical calculations of the submitted analytical results from each participating laboratory.

Table 3.1 Outline of filter samples

Name	Details	Container	Number of filters	Note
No.091d-1	Alkali-Impregnated filter (small quantity)	Polyethylene centrifuge tube	3	The K_2CO_3 impregnated filter contains two ions (SO_4^{2-} and Cl^-).
No.091d-2	Acid-Impregnated filter (small quantity)	Polyethylene centrifuge tube	3	The H_3PO_4 impregnated filter contains one ion (NH_4^+).

No.092d-1	Alkali-Impregnated filter (large quantity)	Polyethylene centrifuge tube	3	The K_2CO_3 impregnated filter contains two ions (SO_4^{2-} and Cl^-).
No.092d-2	Acid-Impregnated filter (large quantity)	Polyethylene centrifuge tube	3	The H_3PO_4 impregnated filter contains one ion (NH_4^+).
No.093d-1	Alkali-Impregnated filter (blank)	Polyethylene centrifuge tube	3	The filter impregnated with K_2CO_3.
No.093d-2	Acid-Impregnated filter (blank)	Polyethylene centrifuge tube	3	The filter impregnated with H_3PO_4.

3.2.3 Analytes

All participating laboratories were expected to analyze these sample filters and to submit their values as the net quantity of each ion (SO_4^{2-}, Cl^-, and NH_4^+) in micrograms (μg).

3.2.4 Analytical Methodologies

The recommended procedure for sampling and analyzing by the filter pack method is described in EANET Document, "Technical Document for Filter Pack Method in EAST Asia" (The NC, 2003). Each sample filter was put in a centrifuge tube, a solvent was directly poured into the tube for extraction. The extraction procedure was as follows;

(1) Sample No.091d-1, No.092d-1, No.093d-1

Add 20 mL of H_2O_2 solution (0.05%-v/v) as an extracting solvent into each centrifuge tube, then shake or agitate them for 20 minutes using a shaker or an ultrasonic bath for extraction.

(2) Sample No.091d-2, No.092d-2, No.093d-2

Add 20 mL of deionized water as an extracting solvent into each centrifuge tube, then shake or agitate them for 20 minutes using a shaker or an ultrasonic bath for extraction.

(3) Filtration

Remove insoluble matter from the solution using a membrane filter (pore size 0.45 μm). The membrane filter must be prewashed with pure water (more than 100 mL) before filtration. After filtration, those filtrates are assigned identification numbers and sealed tightly.

Note 1) Carry out the analysis immediately after extraction.

Note 2) In principle, it is strongly recommended that the filtrates be analyzed immediately after extraction, however, in the case that they need to be kept for certain reasons, store them in a refrigerator at 4℃.

The participating laboratories were expected to use the same analytical methods. Analytical methods specified in the Technical Document were shown in Table 3.2.

Table 3.2 Analytical methods specified in the Technical Document

Analyte	Analytical method
SO_4^{2-} Cl^-	Ion Chromatography or Spectrophotometry
NH_4^+	Ion Chromatography Spectrophotometry (Indophenol Blue)

3.2.5 Data Check Procedures

All participating laboratories were requested to report as the net quantity of each ion (SO_4^{2-}, Cl^-, and NH_4^+) in the sample filter.

Each quantity (M_{sol}) was calculated as follows:

$$M_{sol} = C_{sol} \times V_{sol} \quad (1)$$

where M_{sol} : quantity of each component in the filtrate (μg) ;
C_{sol} : concentration of each component in the filtrate (mg/L);
V_{sol} : volume of the solvent (20 mL).

The net quantity of each ion ($netM_{sol}$) was calculated as follows :

$$\text{net } M_{sol} = M_{sol,\,Sample} - M_{sol,\,Blank} \quad (2)$$

where $netM_{sol}$: net quantity of each ion on the filter.
$M_{sol,Sample}$: quantity (μg) of each component in the filtrate from sample No.091d-1,No.091d-2,No.092d-1 and No.092d-2;
$M_{sol,Blank}$: the average quantity (μg) in the filtrate from blank sample No.093d-1 and No.093d-2.

Note 3) $netM_{sol}$ was automatically calculated by entering their values of $M_{sol,sample}$ and $M_{sol,\,blank}$ on Excell file. (File name : 2009Dry_report1_2_format.xls.)

3.3 Results

The NC distributed the sample filters to 24 laboratories in the participating countries of EANET, and received the data on the analytical results. The summary of the results compared to the prepared value was summarized in Table 3.3. The average, minimum (Min.), maximum (Max.), standard deviation (S.D.) and number of data (N) were calculated from each analyzed ion. Analytical results of Sample No.091d and No.092d were shown in *Appendix 3-2.*

Note 4 : Outliers exceeding 3 sigma S.D. were rejected before calculation.

As shown in Table 3.3, most averages were close to their prepared values except for Cl^- and NH_4^+ in the small quantity samples. The deviation for Cl^- in Sample No.091d was -14.4% and for NH_4^+ in Sample No.091d was 6.16%. The other deviations were within 5%.

The Data Quality Objectives (DQOs) of EANET is specified on the QA/QC program of EANET that determined values are expected to fall within ±15% deviation from the prepared value. Each laboratory analyzed each sample 3 times, averaged the values, and these average values were compared with the corresponding prepared value for this report. The flag "E" indicates that the deviation exceeds ±15% but not over ±30%, and the flag "X" indicates that the deviation exceeds more than ±30%.

Deviation (%) = (Determined value - Prepared value) / Prepared value x 100(%) (3)

Flag E: 15% < | Deviation | ≤ 30%
Flag X: 30% < | Deviation |

The evaluation of the results on both Sample No.091d and No.092d was described in “3.3.1 Evaluation of Laboratories’ Performance (by sample)”. The comparison of the results for each analyte was described in “3.3.2 Comparison of Laboratories’ Performance (by analyte)”. The evaluation of their analytical circumstance, such as analytical method, experience of personnel, and other analytical conditions was described in “3.3.3 Information on Laboratories**”.**

Table 3.3 Summary of analytical results of the sample filters

(The results do not include outliers)

Analyte		Prepared* (Vp)	Average (Va)	ΔV/Vp* (%)	S.D.	Number (N)	Min.	Max.
Sample No. 091d (Small)								
SO_4^{2-}	[μg]	35.0	33.5	-4.24	1.94	21	30.4	38.0
Cl^-	[μg]	3.00	2.57	-14.4	0.57	20	1.13	3.31
NH_4^+	[μg]	5.30	5.63	6.16	0.96	23	2.70	7.91
Sample No. 092d (Large)								
SO_4^{2-}	[μg]	120	119	-1.02	6.77	22	103	134
Cl^-	[μg]	38.0	37.0	-2.70	2.08	20	31.5	40.6
NH_4^+	[μg]	40.0	38.0	-4.89	5.30	23	21.6	49.0

* Prepared: Prepared values

* ΔV/Vp: (Average result (Va) - Prepared value (Vp)) / Prepared value (Vp) x 100(%)

3.3.1 Evaluation of Laboratories' Performance (by sample)

Small quantity samples (No. 091d-1, No.091d-2)

For Sample No.091d, 9 analytical data in 67 results exceeded the DQOs by ±15%, but did not exceed DQOs by ±30%, thus, they were flagged "E". Also 8 analytical data exceeded the DQOs by ±30 %, so they were flagged "X". 17 data points were flagged, which is 25.4% of the total for Sample No.091d (Figure 3.2, Table 3.4 and 3.5).

Table 3.4 Number of flagged data for Sample No.091d (Small Quantity)

	SO_4^{2-}	Cl^-	NH_4^+	Total
Flag E *	1	4	4	9
Flag X *	0	4	4	8
Data within DQOs	21	13	16	50
Ratio of Flagged (%)	4.5	38.1	33.3	25.4

* E : Values exceeding the DQOs by 15%, but not over 30%.

* X : Values exceeding the DQOs by 30%.

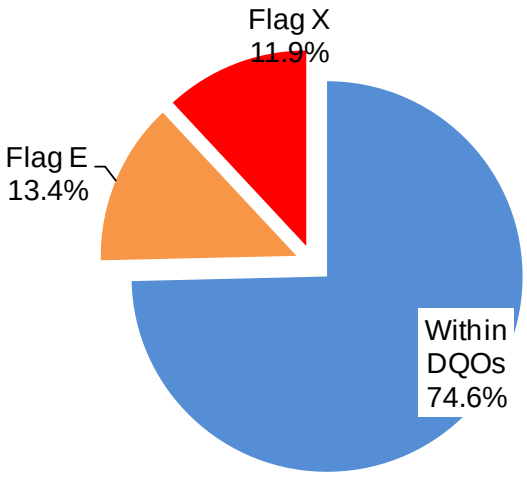


Figure 3.2 Percentage of flagged data for Sample No.091d (Small Quantity)

Table 3.5 Average analytical results of Sample No.091d (Small Quantity)

Lab. Code	SO_4^{2-} (µg)	Cl^- (µg)	NH_4^+ (µg)
KH01	33.5	E 2.29	5.25
CN01	36.2	3.31	X 7.03
ID01	30.7	2.93	X 7.91
ID02	35.3	X 7.31	5.83
ID03	35.3	2.60	E 6.43
JP01	34.0	2.63	5.43
JP02	38.0	3.21	4.93
JP03	34.3	2.75	5.19
JP04	33.9	3.29	5.27
JP05	34.9	2.81	5.56
JP06	33.4	E 2.52	5.19
JP07	33.8	2.83	5.45
KR01	31.7	3.18	E 6.29
MY01	31.7	2.78	5.52
MN01	35.7	2.60	5.60
PH01	-	-	X 2.70
RU01	E 44.8	-	X 1.64
TH01	30.4	E 2.10	5.88
TH02	31.6	X 1.13	E 6.80
TH03	33.0	E 2.48	E 6.11

TH04	31.4		2.59	5.73
VN01	31.3	X	2.05	5.67
VN02	33.7	X	1.31	4.74
VN03	-		-	4.90

* E : Values exceeding the DQOs by 15%, but not over 30%.

* X : Values exceeding the DQOs by 30%.

Large quantity samples (No. 092d-1, No.092d-2)

For Sample No.092d, 5 analytical data in 67 results exceeded the DQOs by ±15%, but did not exceed DQOs by ±30%, thus, they were flagged "E". Also 3 analytical data exceeded the DQOs by ±30 %, so that they were flagged "X". 8 data points were flagged, which was 11.9% of the total for Sample No.092d (Figure 3.3, Table 3.6 and 3.7).

Table 3.6 Number of flagged data for Sample No.092d (Large Quantity)

	SO_4^{2-}	Cl^-	NH_4^+	Total
Flag E *	0	1	4	5
Flag X *	0	1	2	3
Data within DQOs	22	19	18	59
Ratio of Flagged (%)	0.0	9.5	25.0	11.9

* E : Values exceeding the DQOs by 15%, but not over 30%.

* X : Values exceeding the DQOs by 30%.

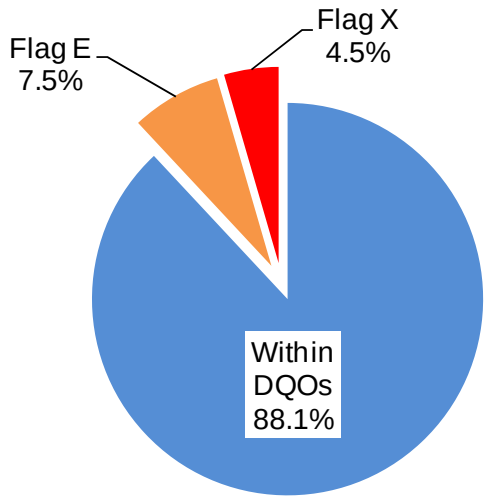


Figure 3.3 Percentage of flagged data for Sample No.092d (Large Quantity)

Table 3.7 Average analytical results of Sample No.092d (large quantity)

Lab. Code	SO_4^{2-} (μg)	Cl^- (μg)	NH_4^+ (μg)
KH01	123	33.7	38.4
CN01	114	39.6	E 49.0
ID01	103	E 31.5	40.1
ID02	107	38.7	36.8
ID03	128	34.5	41.1
JP01	121	38.2	39.0
JP02	127	40.6	37.3
JP03	120	37.7	38.5
JP04	119	37.7	39.6
JP05	119	37.5	41.3
JP06	115	37.7	37.5
JP07	121	35.6	E 32.8
KR01	115	38.5	39.0
MY01	118	38.1	36.5
MN01	129	37.7	37.6
PH01	-	-	X 21.6
RU01	134	-	X 10.4
TH01	120	35.5	39.2
TH02	117	X 26.4	E 28.1
TH03	116	34.8	E 46.3
TH04	118	37.2	40.5
VN01	114	36.9	41.3
VN02	115	37.8	38.5
VN03	-	-	35.0

* E : Values exceeding the DQOs by 15%, but not over 30%.

*X : Values exceeding the DQOs by 30%.

Blank Sample (No.093d)

Each quantity of SO_4^{2-}, Cl^-, and NH_4^+ was determined for blank sample No.093d-1 and No.093d-2. Their obtained values were shown in Table 3.8. Blank values were detected in a wide range, including 0μg. Table 3.9 showed the ratio of the blank value to analytical results by laboratory. Light gray color cell indicated that the sample was flagged E and dark fray color cell indicated that the sample was flagged X. At some laboratories flags did not appear even though some blank values were high.

Table 3.8 Analytical results of Sample No.093d (blank)

Lab. Code	SO_4^{2-} (μg)	Cl^- (μg)	NH_4^+ (μg)
KH01	0.00	2.86	0.09
CN01	0.00	0.31	0.30
ID01	0.00	0.00	0.00
ID02	0.00	0.01	0.64
ID03	0.00	0.00	0.00
JP01	0.16	0.34	0.18
JP02	0.30	0.67	0.34
JP03	0.00	1.14	0.27
JP04	1.23	0.53	0.24
JP05	0.00	0.52	0.29
JP06	0.00	0.50	0.59
JP07	0.00	1.10	1.08
KR01	1.06	3.19	0.62
MY01	0.14	0.57	0.37
MN01	0.60	0.20	0.80
PH01	-	-	0.18
RU01	0.00	-	1.58
TH01	0.32	0.31	0.24
TH02	0.00	1.67	0.00
TH03	0.00	0.80	0.44
TH04	0.60	0.51	0.56
VN01	1.03	0.00	0.83
VN02	0.00	0.00	2.41
VN03	-	-	5.17
Average	0.25	0.74	0.72
Median	0.00	0.51	0.36
Minimum	0.00	0.00	0.00
Maximum	1.23	3.49	5.17
Standard deviation	0.35	0.90	1.07

Table 3.9 Ratio of blank value to analytical result (V_{blank} /V_{result})

	Small Quantity (No.091d)			Large Quantity (No.092d)		
Lab. Code	SO_4^{2-}	Cl^-	NH_4^+	SO_4^{2-}	Cl^-	NH_4^+
KH01	0.00	1.25	0.02	0.00	0.08	0.00
CN01	0.00	0.09	0.04	0.00	0.01	0.01
ID01	0.00	0.00	0.00	0.00	0.00	0.00
ID02	0.00	0.00	0.11	0.00	0.00	0.02
ID03	0.00	0.00	0.00	0.00	0.00	0.00
JP01	0.00	0.13	0.03	0.00	0.01	0.00
JP02	0.01	0.21	0.07	0.00	0.02	0.01
JP03	0.00	0.41	0.05	0.00	0.03	0.01
JP04	0.04	0.16	0.05	0.01	0.01	0.01
JP05	0.00	0.19	0.05	0.00	0.01	0.01
JP06	0.00	0.20	0.11	0.00	0.01	0.02
JP07	0.00	0.39	0.20	0.00	0.03	0.03
KR01	0.03	1.10	0.10	0.01	0.09	0.02
MY01	0.00	0.21	0.07	0.00	0.01	0.01
MN01	0.02	0.08	0.14	0.00	0.01	0.02
PH01	-	-	0.07	-	-	0.01
RU01	0.00	-	0.96	0.00	-	0.15
TH01	0.01	0.15	0.04	0.00	0.01	0.01
TH02	0.00	1.48	0.00	0.00	0.06	0.00
TH03	0.00	0.32	0.07	0.00	0.02	0.01
TH04	0.02	0.20	0.10	0.01	0.01	0.01
VN01	0.03	0.00	0.15	0.01	0.00	0.02
VN02	0.00	0.00	0.51	0.00	0.00	0.06
VN03	-	-	1.06	-	-	0.15

: Data flagged E

: Data flagged X

3.3.2 Comparison of Laboratories' Performance (by Analyte)

The overview of the results was shown in the following Figures and Tables for each analyte (SO_4^{2-}, Cl^- and NH_4^+). The obtained values from each laboratory were evaluated for their deviations. The number of flagged data was shown in Table 3.4 and 3.6 for each analyte.

SO_4^{2-} (Sulfate)

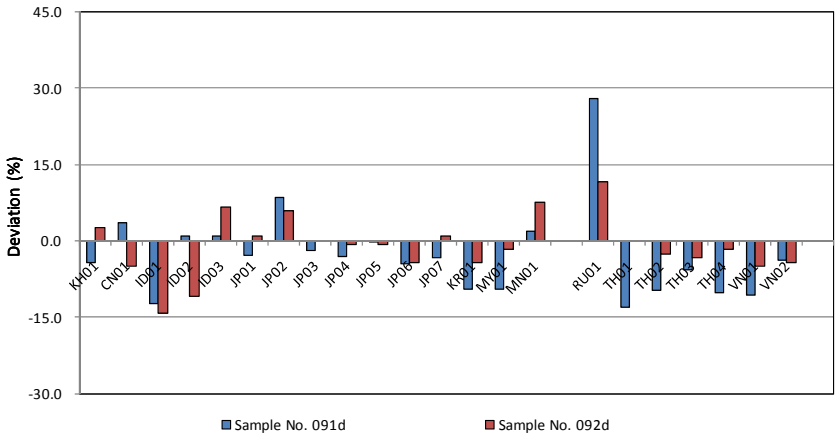


Figure 3.4 Deviation for SO_4^{2-}

Deviation (%) = (Determined value- Prepared value) / Prepared value x 100

Table 3.10.1 Analytical method of SO_4^{2-}

*Analytical Method**		
Ion Chromatography	21/22	

*1 lab : no information

Table 3.10.2 Flagged data of SO_4^{2-}

Flagged Data	Flag E	Flag X	Flagged (%)
Sample No.091d	1	0	4.5
Sample No.092d	0	0	0.0

All participating laboratories used Ion Chromatography for the determination of SO_4^{2-}. Only 1 laboratory was flagged E. DQOs were within 95.0 % of Sulfate sample.

Cl^- (Chloride)

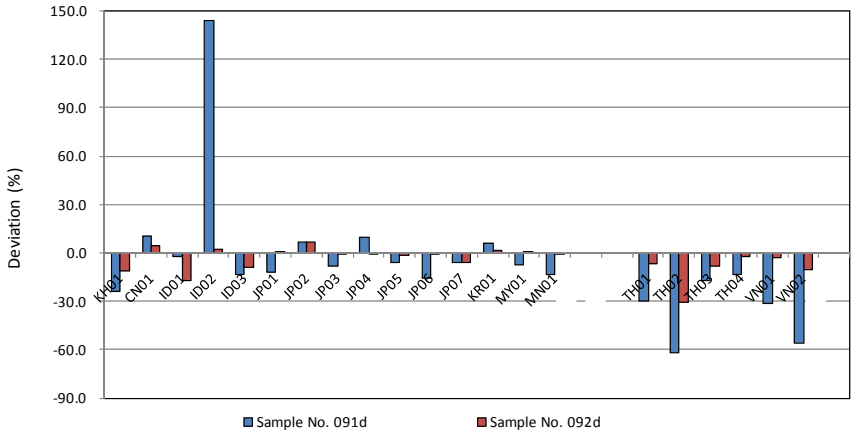


Figure 3.5 Deviation for Cl^-

Table 3.11.1 Analytical method of Cl^-

*Analytical Method**		
Ion Chromatography	19/20	

*1 lab : no information

Table 3.11.2 Flagged data of Cl^-

Flagged Data	Flag E	Flag X	Flagged (%)
Sample No.091d	4	4	40.0
Sample No.092d	1	1	10.0

As with the analysis of SO_4^{2-}, all laboratories used Ion Chromatography for the determination of Cl^-. "E" flags appeared at 4 laboratories for Sample No.091d and "X" flags appeared 4 laboratories. One flag of each ("E" and "X") appeared at 2 laboratories for Sample No. 092d.

NH_4^+ (Ammonium)

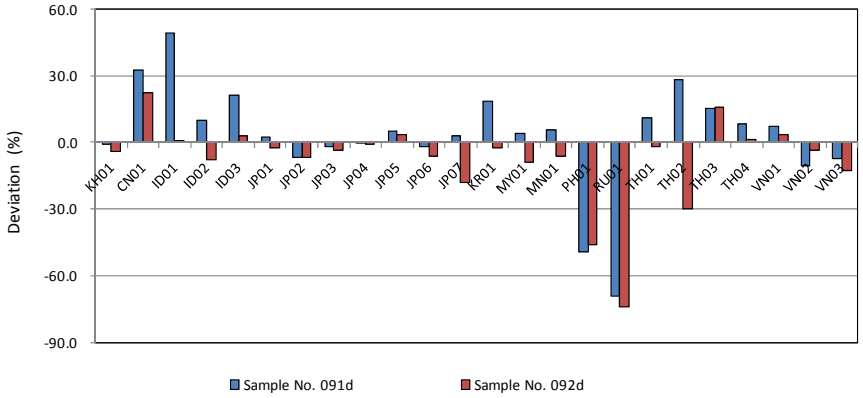


Figure 3.6 Deviation for NH_4^+

Table 3.12.1 Analytical method of NH_4^+

*Analytical Method**	
Ion Chromatography	20/24
Spectrophotometry (Nessler)	1/24
Spectrophotometry (Indophenol lue)	1/24

2 labs : no information

Table 3.12.2 Flagged data of NH_4^+

Flagged Data	Flag E	Flag X	Flagged (%)
Sample No.091d	4	4	33.3
Sample No.092d	4	2	25.0

Twenty laboratories used Ion Chromatography, 1 laboratory used Spectrophotometry (Nessler) and 1 laboratory used Spectropohotometry (Indophenol Blue). There were 4 E flags and 4 X flags for Sample No.091d, and 4 E flags and 2 X flag for Sample No.092d.

3.3.3 Information on Laboratories

Methodologies Used

As shown in Table 3.13, most participating laboratories used recommended methods of EANET and all laboratories used Ion Chromatography for the determination of anions. As for the determination of NH_4^+, 20 of 24 laboratories used Ion Chromatography and 2 laboratories used Spectrophotometry.

Table 3.13 Analytical methods used for sample analysis

Lab. Code	SO_4^{2-},Cl^-	NH_4^+
KH01	Ion Chromatography	
CN01	Ion Chromatography	
ID02	Ion Chromatography	
ID03	Ion Chromatography	
JP01	Ion Chromatography	
JP02	Ion Chromatography	
JP03	Ion Chromatography	
JP04	Ion Chromatography	
JP05	Ion Chromatography	
JP06	Ion Chromatography	
JP07	Ion Chromatography	
KR01	Ion Chromatography	
MY01	Ion Chromatography	
MN01	(no information)	
PH01	-	(no information)
RU01	Ion Chromatography (Only SO_4^{2-})	Spectrophotometry(Nessler)
TH01	Ion Chromatography	
TH02	Ion Chromatography	
TH03	Ion Chromatography	
TH04	Ion Chromatography	
VN01	Ion Chromatography	
VN02	Ion Chromatography	
VN03	-	Spectrophotometry (Indophenol Blue)

Years of staff experience

Years of experience for staff in charge were summarized in Table 3.14. Data in light gray color cell indicated that there was a flag for Sample No.091d or 092d. Data in dark gray color cell indicated flagged data in both Sample No.091d and No.092d.

Table 3.14 Years of experience (unit: year)

Lab. Code	SO_4^{2-}	Cl^-	NH_4^+
KH01	1	1	1
CN01	1	1	1
ID01	8	8	8
ID02	10	10	10
ID03	2	2	2
JP01	25	25	25
JP02	5	5	5
JP03	3	3	3
JP04	2	2	2
JP05	4	4	4
JP06	3	3	3
JP07	2	2	2
KR01	12	12	13
MY01	7	7	4
MN01	*	*	*
PH01	-	-	*
RU01	11	-	11
TH01	2	2	2
TH02	6	6	6
TH03	3	3	3
TH04	7	7	12
VN01	16	16	16
VN02	5	5	5
VN03	-	-	4

*No information

: 1 Datum (ether small or large quantity sample) is flagged.

: 2 Data (both small and large quantity samples) are flagged.

Flagged Data

In the results of Sample No.091d and 092d, the total number of flagged data was 24 (E: 14, X: 11) in the whole values (134). Flagged data in each laboratory was shown in Figure 3.8. The number of laboratories with the results without flagged data was 9 (37.5%). One laboratory had 4 flagged data.

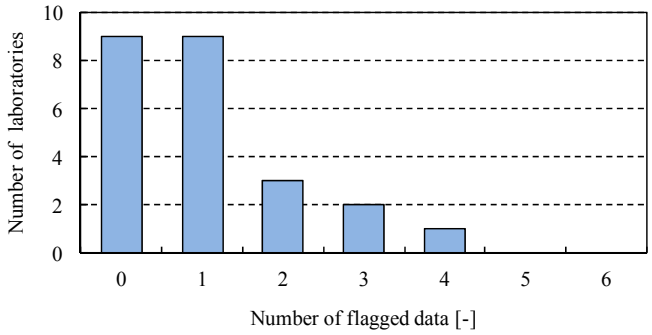


Figure 3.8 Number of flagged data and laboratories

Calibration standard solution

Table 3.15 showed the lowest (except for zero) and highest concentrations of their calibration standard solutions (SO_4^{2-}, Cl^-, NH_4^+) used in each laboratory, and also showed their concentrations of the prepared values in μmol/L. The concentrations of the standard solutions in some laboratories were not in the appropriate range. A boldfaced number in Table 3.15 indicated that the concentration value of standard solution was lower than that of the prepared value or higher than that of the prepared value.

Each concentration of prepared value was expected within the range of both concentrations of lowest and highest standard solutions. However, some laboratories used inappropriate solution ranges. If the concentrations of their obtained values were not in the range of the calibration standard, laboratories should have analyzed again with the appropriate concentration range of standard solution.

Table 3.15 Ranges of the calibration standard solution in each laboratory

	SO_4^{2-}	µmol/L	Cl^-	µmol/L	NH_4^+	µmol/L
Lab Code.	Lowest	Highest	Lowest	Highest	Lowest	Highest
KH01	1.04	52.1	2.82	141	5.54	277
CN01	**20.5**	208	**22.0**	225	**41.2**	361
ID01	2.08	62.5	**5.63**	169	5.56	167
ID02	9.25	148	**5.00**	80.0	8.00	128
ID03	6.25	93.8	**5.63**	84.5	5.54	111
JP01	10.5	210	**28.4**	569	**21.9**	548
JP02	0.52	104	1.41	282	2.77	139
JP03	5.21	156	2.82	84.6	5.54	166
JP04	1.04	104	2.82	141	2.77	277
JP05	1.04	521	1.41	705	2.77	1386
JP06	1.67	167	**4.52**	226	4.41	220
JP07	0.21	104	0.56	282	1.11	277
KR01	1.04	52.1	1.41	70.5	5.54	277
MY01	1.04	62.5	1.41	113	2.77	166
MN01	4.27	125	**6.49**	170	11.1	111
PH01	-	-	-	-	**27.7**	111
RU01	6.24	20.8	-	-	5.56	222
TH01	1.04	41.6	2.82	113	0.55	111
TH02	2.08	62.5	**5.64**	169	4.43	111
TH03	1.04	83.3	2.82	113	5.54	222
TH04	0.10	104	0.28	282	0.55	139
VN01	2.08	104	2.82	141	5.54	277
VN02	1.04	62.5	2.82	169	5.54	139
VN03	-	-	-	-	5.56	**66.7**
Sample No. 091d	18.2		4.23		14.7	
Sample No. 092d		62.5		53.6		111

Boldfaced Value: The concentration of standard solution was lower/higher than that of the prepared value.

Light gray cell : Data Flagged E

Dark gray cell : Data Flagged X

Lowest and Highest: lowest/highest concentrations in the calibration standard solutions.

3.3.5 Comparison with past surveys

This Inter-Laboratory Comparison on dry deposition has been implemented since 2005. The results showing the percentages of flagged data and percentages of data that were satisfied the DQOs were shown in Figure 3.9.

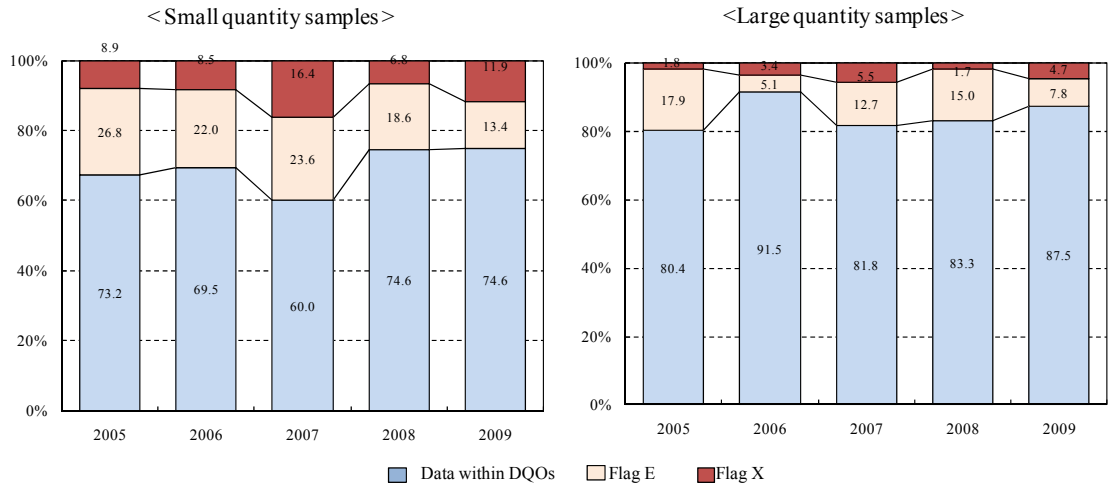


Figure 3.9 Comparison of DQOs' results for the past 5 years

The comparison for each analyte in Inter-Laboratory Comparison on dry deposition year–by-year was shown in Figure 3.10.

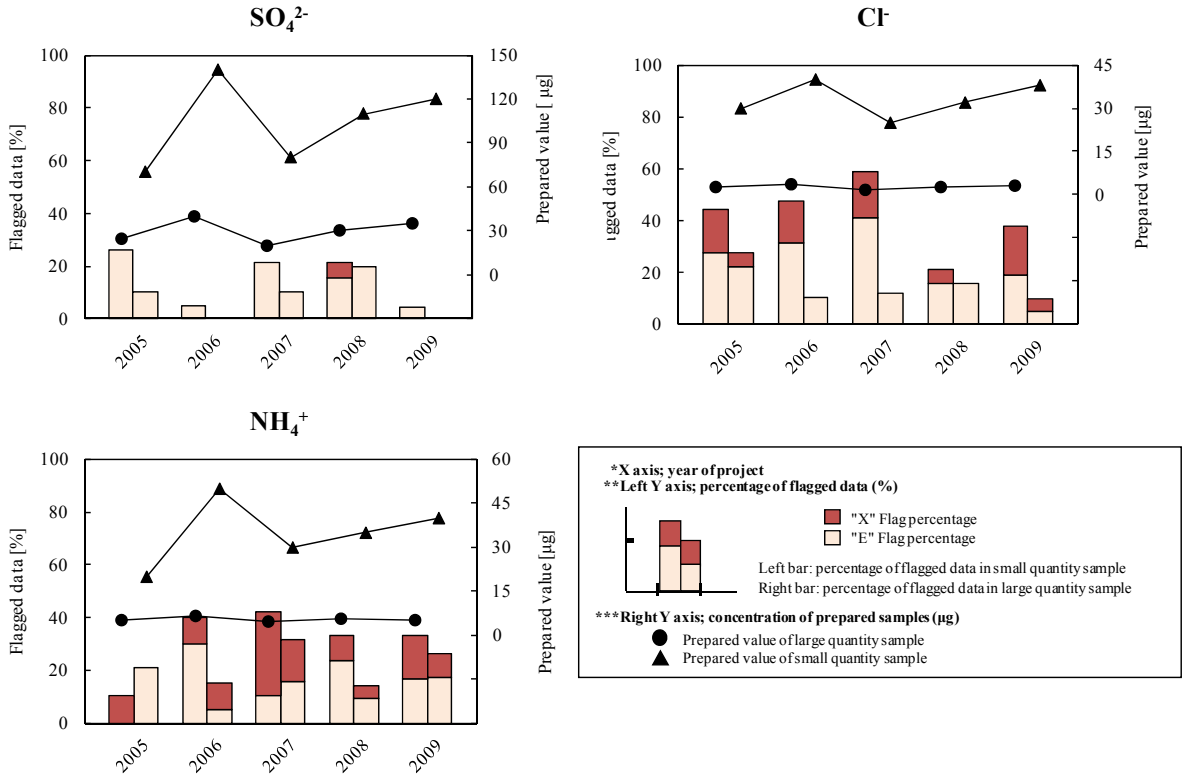


Figure 3.10 Comparison for each parameter in inter-laboratory comparison project

References

1) The Second Interim Scientific Advisory Group Meeting of Acid Deposition Monitoring Network in East Asia. 2000. Guidelines for Acid Deposition Monitoring in East Asia. Network Center for EANET. Acid Deposition and Oxidant Research Center, Niigata, Japan.

2) The Second Interim Scientific Advisory Group Meeting of Acid Deposition Monitoring Network in East Asia. 2000. Technical Documents for Wet Deposition Monitoring in East Asia. Network Center for EANET. Acid Deposition and Oxidant Research Center. Niigata, Japan.

3) The Third Session of the Scientific Advisory Committee (SAC3). 2003. Technical Documents for Filter Pack Method in East Asia. Network Center for EANET. Acid Deposition and Oxidant Research Center, Niigata, Japan.

Appendix 3.1 Participating laboratories

Countries / Laboratories	Code
1. CAMBODIA	
1) Environmental Quality Research and Laboratory Office	**(KH01)**
2. CHINA	
2) Xiamen Environmental Monitoring Central Station	**(CN01)**
3 INDONESIA	
3) Environmental Management Center (PUSARPEDAL) (EMC)	**(ID01)**
4) Meteorological and Geophysical Agency of Indonesia (BMG)	**(ID02)**
5) Indonesian National Institute of Aeronautic and Space (LAPAN)	**(ID03)**
4. JAPAN	
6) Hokkaido Institute of Environmental Sciences	**(JP01)**
7) Niigata Prefectural Institute of Public Health and Environmental Sciences	**(JP02)**
8) Nagano Environmental Conservation Research Institute	**(JP03)**
9) Gifu Prefectural Institute of Health and Environmental Science	**(JP04)**
10) Shimane Prefectural Institute of Public Health and Environmental Science	**(JP05)**
11) Okinawa Prefectural Institute of Health and Environment	**(JP06)**
12) Acid Deposition and Oxidant Research Center (ADORC)	**(JP07)**
5.RRPUBLIC OF KOREA	
13) National Institute of Environmental Research	**(KR01)**
6. MALAYSIA	
14) Department of Chemistry	**(MY01)**
7. MONGOLIA	
15) Central Laboratory of Environment and Metrology	**(MN01)**
8. PHILIPPINES	
16) Environmental Management Bureau (EMB)	**(PH01)**
9. RUSSIA	
17) Limnological Institute, Russian Academy of Sciences/Siberian Branch	**(RU01)**
10. THAILAND	
18) Pollution Control Department (PCD)	**(TH01)**
19) Khon Kaen University (KKU)	**(TH02)**
20) Chiang Mai University (CMU)	**(TH03)**
21) Environmental Research and Training Center (ERTC)	**(TH04)**

11.VIET NAM

22) Center for Environmental Research, Institute of Meteorology and Hydrology **(VN01)**

23) Mid-central Regional Hydro Meteorological Center **(VN02)**

24) Sub-institute of Hydrometeorology and Environment of South Vietnam **(VN03)**

Appendix 3.2 Summary of data

Appendix Table 3.2.1 Analytical results of Sample No. 091d (small quantity)

Lab. ID	SO_4^{2-} [μg]	Cl^- [μg]	NH_4^+ [μg]
KH01	33.5	2.29	5.25
CN01	36.2	3.31	7.03
ID01	30.7	2.93	7.91
ID02	35.3	7.31	5.83
ID03	35.3	2.60	6.43
JP01	34.0	2.63	5.43
JP02	38.0	3.21	4.93
JP03	34.3	2.75	5.19
JP04	33.9	3.29	5.27
JP05	34.9	2.81	5.56
JP06	33.4	2.52	5.19
JP07	33.8	2.83	5.45
KR01	31.7	3.18	6.29
MY01	31.7	2.78	5.52
MN01	35.7	2.60	5.60
PH01	-	-	2.70
RU01	44.8	-	1.64
TH01	30.4	2.10	5.88
TH02	31.6	1.13	6.80
TH03	33.0	2.48	6.11
TH04	31.4	2.59	5.73
VN01	31.3	2.05	5.67
VN02	33.7	1.31	4.74
VN03	-	-	4.90
Prepared value	35.0	3.00	5.30
Number of data	22	21	24
Average	34.0	2.80	5.46
Minimum	30.4	1.13	1.64
Maximum	44.8	7.31	7.91
Standard Deviation	3.02	1.15	1.23

Appendix Table 3.2.2 Analytical results of Sample No. 092d (large quantity)

Lab. ID	SO_4^{2-} [μg]	Cl^- [μg]	NH_4^+ [μg]
KH01	123	33.7	38.4
CN01	114	39.6	49.0
ID01	103	31.5	40.1
ID02	107	38.7	36.8
ID03	128	34.5	41.1
JP01	121	38.2	39.0
JP02	127	40.6	37.3
JP03	120	37.7	38.5
JP04	119	37.7	39.6
JP05	119	37.5	41.3
JP06	115	37.7	37.5
JP07	121	35.6	32.8
KR01	115	38.5	39.0
MY01	118	38.1	36.5
MN01	129	37.7	37.6
PH01	-	-	21.6
RU01	134	-	10.4
TH01	120	35.5	39.2
TH02	117	26.4	28.1
TH03	116	34.8	46.3
TH04	118	37.2	40.5
VN01	114	36.9	41.3
VN02	115	37.8	38.5
VN03	-	-	35.0
Prepared value	120	38.0	40.0
Number of data	22	21	24
Average	119	36.5	36.9
Minimum	103	26.4	10.4
Maximum	134	40.6	49.0
Standard Deviation	6.77	3.03	7.58

Appendix 3.3 Z-score evaluation

The NC applied Z-score for further statistical evaluation of the analytical values in the inter-laboratory comparison on dry deposition.

- **Definition of Z-score**

Z-score is one of the statistical measures that quantify the distance from the mean of a data set. The formula for the calculation of Z-score (Robust method) was shown below:

$$Z = \frac{X - Q_2}{0.7413 \times (Q_3 - Q_1)}$$

where X: Measurement values of samples
Q_1: The 1st quartile value of entire data
Q_2: The 2nd quartile value of entire data (i.e. Median)
Q_3: The 3rd quartile value of entire data

- **Evaluation of calculated Z-score**

Z-score was given to each data submitted by the participating laboratories, and was evaluated as follows:

$|Z| \leq 2$: Satisfactory
$2 < |Z| < 3$: Questionable
$3 \leq |Z|$: Unsatisfactory

The calculated Z-scores were shown in Appendix Table 3.3.1 and were also graphed in Appendix Figure 3.3.1 through 3.3.3.

Appendix Table 3.3.1 Results of Z-score evaluation for sample No. 091d and 092d

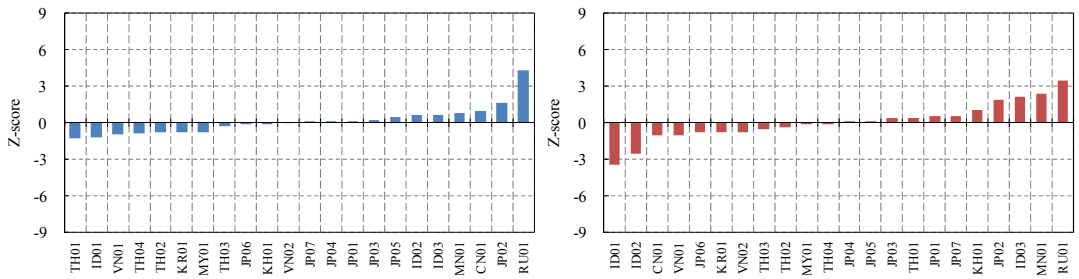
Sample No. 091d (Small quantity)

Lab. ID	SO_4^{2-}	Cl^-	NH_4^+
KH01	-0.10	-1.02	-0.45
CN01	0.94	2.04	2.61
ID01	-1.18	0.90	4.23
ID02	0.60	14.03	0.45
ID03	0.60	-0.09	1.53
JP01	0.10	0.00	-0.27
JP02	1.64	1.74	-1.17
JP03	0.21	0.36	-0.63
JP04	0.06	1.98	-0.45
JP05	0.44	0.54	0.09
JP06	-0.13	-0.33	-0.63
JP07	0.02	0.60	-0.09
KR01	-0.79	1.65	1.35
MY01	-0.79	0.45	-0.09
MN01	0.75	-0.09	0.09
PH01	---	---	-5.13
RU01	4.26	---	-7.10
TH01	-1.29	-1.59	0.63
TH02	-0.83	-4.50	2.25
TH03	-0.29	-0.45	0.99
TH04	-0.91	-0.12	0.27
VN01	-0.94	-1.74	0.27
VN02	-0.02	-3.96	-1.53
VN03	---	---	-1.17
Number of data	22	21	24
$\|Z\| \leq 2$	21	17	19
$2 < \|Z\| < 3$	0	1	2
$3 \leq \|Z\|$	1	3	3

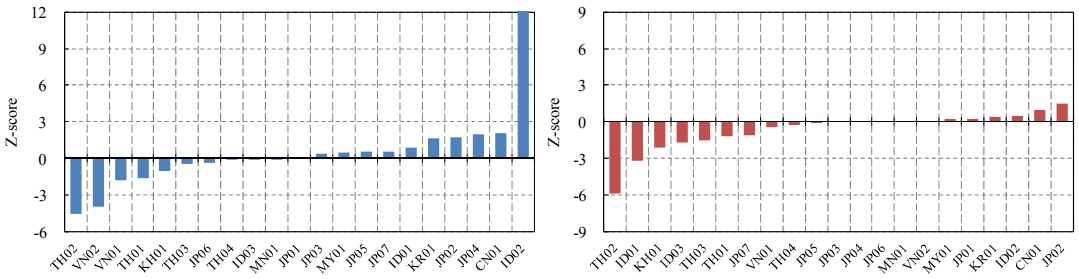
Sample No. 092d (Large quantity)

Lab. ID	SO_4^{2-}	Cl^-	NH_4^+
KH01	1.01	-2.08	-0.04
CN01	-1.01	0.99	4.08
ID01	-3.48	-3.22	0.62
ID02	-2.59	0.52	-0.66
ID03	2.14	-1.66	1.01
JP01	0.56	0.26	0.19
JP02	1.91	1.50	-0.47
JP03	0.34	0.00	0.00
JP04	0.11	0.00	0.43
JP05	0.11	-0.10	1.09
JP06	-0.79	0.00	-0.39
JP07	0.56	-1.09	-2.21
KR01	-0.79	0.42	0.19
MY01	-0.11	0.21	-0.78
MN01	2.36	0.00	-0.35
PH01	---	---	-6.56
RU01	3.48	---	-10.91
TH01	0.34	-1.14	0.27
TH02	-0.34	-5.86	-4.04
TH03	-0.56	-1.50	3.03
TH04	-0.11	-0.26	0.78
VN01	-1.01	-0.42	1.09
VN02	-0.79	0.05	0.00
VN03	---	---	-1.36
Number of data	22	21	24
$\|Z\| \leq 2$	17	18	18
$2 < \|Z\| < 3$	3	1	1
$3 \leq \|Z\|$	2	2	5

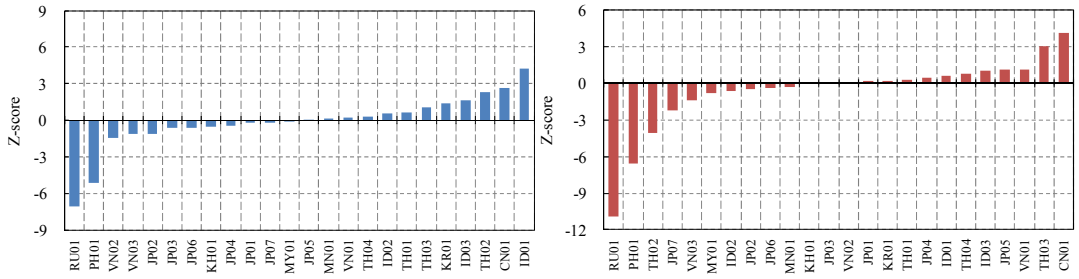
Note: Light mesh, $|Z| > 2$ (Questionable); Dark mesh, $|Z| \geq 3$ (Unsatisfactory); "---", Not measured



Appendix Figure 3.3.1 Distribution of Z-score for SO_4^{2-} (Left: 091d, Right: 092d)



Appendix Figure 3.3.2 Distribution of Z-score for Cl^- (Left: 091d, Right: 092d)



Appendix Figure 3.3.3 Distribution of Z-score for NH_4^+ (Left: 091d, Right: 092d)

4. 11th INTER-LABORATORY COMPARISON PROJECT ON SOIL

4.1 Introduction

The Inter-laboratory comparison project on soil analysis started in 1999 as the activities within the QA/QC program on Soil and Vegetation Monitoring. The inter-laboratory precision should be clarified as well as the within-laboratory and repeatability precisions in the project to improve the analytical quality of the EANET laboratories. Factors of precision have been discussed through the previous projects.

Soil analysis has complicated procedures and steps in comparison with environment water. Steps in the procedures of soil analysis may be related to the variation among laboratories; e.g. extraction, instrumental analysis and/or titration. Results of the first three projects from 1999 to 2001 suggested that instrumental analysis have relatively large effect on the total precision of soil analysis, and the following analytical conditions could affect results:

- Addition of La or Sr solution for AAS analysis of Ex-Ca
- Preparation method of standard solution
- Instrument for Ex-K and Na analysis

The participating laboratories shared the information on these possible factors to improve the precisions.

Other possible factors, such as level of the concentrations, were suggested in the previous projects. Further investigation should be considered taking concentrations of the samples into account.

In the 11th project, NC provided two soil samples (No.091 and 092) to laboratories to improve the inter-laboratory precision further more by standardization of methods. In this report, the data from participating laboratories were evaluated statistically according to the QA/QC program for soil monitoring, and the results may be utilized for estimation of inter-laboratory variation in soil monitoring, and provide useful information to improve precision of soil analysis on EANET.

4.2 Procedures

4.2.1 Participating Laboratories

Sixteen laboratories of 10 countries participated in the 11th project. Names of the participating laboratories are listed in Appendix 4.1.

4.2.2 Description of Samples

The characteristics of the soil samples were as follows:

Sample No. 091: Brown forest soil (Cambisols)

Sample No. 092: Red soil (Acrisols)

Soils for Sample No.091 and No.092 were collected in the forest floor of Japanese cypress (*Chamaecyparis obtusa*) plantation and the pasture (non-cow-pastured field) in Yamaguchi prefecture.

Both soils were collected from B-horizon composed chiefly of soil minerals. The soils were air-dried, sieved to separate the fine earth fraction (< 2 mm), and mixed well by the following procedures: the bulk sample was divided into two parts, each part was mixed well, the parts were joined and mixed well, and then the sample was divided again. This procedure was repeated 15 times to ensure a completely homogeneous bulk sample. Finally, portions of ca. 500 g were weighed out, packed in 500 ml plastic bottles, and then, sterilized using radioisotope (20kGy) for distributing (exporting) to the participating countries.

4.2.3 Parameters Analyzed

All the participating laboratories were expected to measure all the parameters (Table 4.1).

Table 4.1. Parameters to be measured

Parameters	Unit	No.091 and 092
a) Moisture Content	wt %	M
b) pH (H_2O)		M
c) pH (KCl)		M
d) Exchangeable Ca^{2+}	$cmol_c\ kg^{-1}$	M
e) Exchangeable Mg^{2+}	$cmol_c\ kg^{-1}$	M
f) Exchangeable K^+	$cmol_c\ kg^{-1}$	M
g) Exchangeable Na^+	$cmol_c\ kg^{-1}$	M
h) Exchangeable Acidity	$cmol_c\ kg^{-1}$	M
i) Exchangeable Al^{3+}	$cmol_c\ kg^{-1}$	M
j) Exchangeable H^+	$cmol_c\ kg^{-1}$	M

M: Mandatory items

“Exchangeable” were abbreviated to “Ex-“ in this report; e.g. Ex-Ca, Ex-Mg, etc.

4.2.4 Analytical Methodologies

All the procedures for chemical analysis were carried out basically according to the "Technical Manual for Soil and Vegetation Monitoring in East Asia" (2nd ISAG, 2000).

In the respective laboratories, all the parameters were analyzed three times under the same conditions (repeatability condition: analyst, time, and instrument are the same; three replicates). Then, under within-laboratory-reproducibility condition (part or all of analyst, time, and instrument are different), all the analytical procedures should be repeated twice.

4.2.4.1 Standardization of methods

All the procedures for chemical analysis should be carried out basically according to the "Technical Documents for Soil and Vegetation Monitoring in East Asia (March 2000, Adopted at: The Second Interim Scientific Advisory Group Meeting of Acid Deposition Monitoring Network in East Asia)".

In the 8th project,

(1) **Atomic absorption spectrometry (AAS)** method should be used basically for analysis of Ex-Ca, Mg, K and Na. (If it is impossible to use AAS, Flame (emission) photometry method is allowable for Ex-K and Na).
(2) **Titration method** should be used for analysis of EX-acidity, Al and H.
(3) **Calibration curve method** should be used for determination of Ex-Ca, Mg, K and Na.
(4) The Samples should be extracted and diluted with **1M CH_3COONH_4 (pH 7.0)** for analysis of Ex-Ca, Mg, K and Na. Then, 1M CH_3COONH_4 (pH 7.0) solution should be used to prepare each standard solution as the solvent.
(5) **Sr** should be added to the samples and each standard solution to eliminate the interference of the sample for analysis of Ex-Ca and Mg. These are to be the same concentration Sr. (If Sr can not be obtained, La is allowable.)

4.2.4.2 Procedures for Ex-base cations

(1) Extract from air-dry sample with 1M CH_3COONH_4 (pH 7.0) solution. (According to the "Technical Documents for Soil and Vegetation Monitoring in East Asia")
(2) Pipette an appropriate aliquot of the soil extract into volumetric flask and add 100g-Sr/L solution to be 1000mg-Sr/L as final concentration Sr. ($SrCl_2$ solution eliminates the interference of the sample.) And then make to volume with 1M CH_3COONH_4 (pH 7.0). This solution is named " Prepared sample".
(3) Prepare three "prepared samples".
(4) Prepare each standard solution with diluting 1M CH_3COONH_4 (pH 7.0) solution.

(5) Add 100g-Sr/L solution to each standard solution to be the same concentration $SrCl_2$ as the sample.

(6) Analyze the standard solution and the prepared samples by AAS.

(7) Store the calibration curves certainly and report them together with reporting formats.

(8) **Repeat the procedure 1) - 7) twice.**

(9) Calculation of content in the soil

Content in the soil could be calculated by the following formulas:

Ex-Ca ($cmol_c\ kg^{-1}$ soil) = [A * B * V * mcf]/[10 * 20.04 * S]

Ex-Mg ($cmol_c\ kg^{-1}$ soil) = [A * B * V * mcf]/[10 * 12.15 * S]

Ex-K ($cmol_c\ kg^{-1}$ soil) = [A * B * V* mcf]/[10 * 39.10 * S]

Ex-Na ($cmol_c\ kg^{-1}$ soil) = [A * B * V* mcf]/[10 * 23.00 * S]

Where

A = Measurement values of prepared (diluted) samples (mg/L)

B = Dilution ratio (B = 2, if 25mL sample was diluted to 50 mL for making prepared sample.)

mcf = Moisture correction factor (Measured value)

S = Weight of air-dry sample (g)

V = Volume of extract (mL)

4.2.4.3 Procedures for Ex-acidity

(1) Extraction and titration would be carried out according to the "Technical Documents for Soil and Vegetation Monitoring in East Asia" basically.

(2) Prepare three samples. Analyze each sample and at least one blank.

(3) Repeat the procedure twice

(4) Calculation of content in the soil

Content in the soil could be calculated by the following formulas:

Ex-Acidity ($cmol_c\ kg^{-1}$ soil) = $[(A_{NaOH} - bl_{NaOH}) * M_{NaOH} * c * 100 * mcf] / S$

Ex-Al ($cmol_c\ kg^{-1}$ soil) = $[(A_{HCl} - bl_{HCl}) * M_{HCl} * c * 100 * mcf] / S$

Ex-H ($cmol_c\ kg^{-1}$ soil) = $[(A_{NaOH} - bl_{NaOH}) * M_{NaOH} - (A_{HCl} - bl_{HCl}) * M_{HCl}] * c * 100 * mcf] / S$

Where

A_{NaOH} = Titration volume of 0.025 M NaOH solution needed for percolate (mL)

A_{HCl} = Titration volume of 0.02 M HCl solution needed for percolate (mL)

bl_{NaOH} = Titration volume of 0.025M NaOH solution needed for blank (mL)

bl_{HCl} = Titration volume of 0.02M HCl solution needed for blank (mL)

M_{NaOH} = Molarity of NaOH solution (mol/L)

M_{HCl} = Molarity of HCl solution (mol/L)

S = Weight of air-dry sample (g)

c = Aliquot factor (c = 2, if 50mL percolate of 100mL is used.)

4.2.4.4 Reporting

(1) Preparation of the report

Digital formats (Microsoft Excel) for reporting were provided to the participating laboratories, and the laboratories were requested to fill in the formats. Contents in the soil sample would be calculated automatically by the formula above if the formats were filled in.

(2) Submission of the report

Data reporting formats together with all of the copy of calibration curve were submitted by using digital devices.

4.2.5 Data Checking Procedures

Data were statistically evaluated according to the following procedures described in the "Technical Manual for Soil and Vegetation Monitoring in East Asia" (2nd ISAG, 2000). Data of the soil content with one decimal place for pH and two decimal places for Ex-cations and acidity were used for the analysis.

1) Verification of data

Evenness of within-laboratory precision was verified by Cochran methods, then the laboratory averages was verified by Grubbs methods. We call this verification "Cohchran-Grubbs methods".

2) Analysis of variance and estimation of precision

Total variation among laboratories includes within-laboratory and inter-laboratory variation. As described in the following equation, Total sum of square (S_T) is consisted of Sum of square inter-laboratories (S_R), Sum of square within-laboratory (S_{RW}) and Sum of square repeatability (S_r).

$$S_T = S_R + S_{RW} + S_r$$

Based on the above equation, Inter-laboratories variance, Within-laboratory-reproducibility variance, and Repeatability variance were calculated, and then the precisions were estimated.

3) Calculation of permissible tolerance

Permissible tolerances were calculated based on the above precisions.

4.3 Results

4.3.1 Detection of outliers

Detection of outliers by Cochran-Grubbs methods was shown in Table 4.2. The laboratory which has a large difference in repeat analyses was judged as outlier by Cochran method (examination of the evenness of within-laboratory precision): e.g. “cn01” in pH (H_2O) of No.091, “jp01” in Ex-Ca of No.091, etc. Then, the rest of data were tested by Grubbs method (examination of the average value of each laboratory). The laboratory which has remarkably large or small average was judged as outliers.

Cochran-Grubbs method detected the 2 and 5 outliers for pH (H2O) of No.091 and No.092, respectively. Each outlier was detected in pH (KCl) of No.091 and No.092. In exchangeable base cations (Ex-Ca, Ex-Mg and Ex-K), more outliers were detected in No.091 (3 to 4 outliers) than those in No.092 (1 to 2 outliers). We also found 1 to 3 outliers for the Ex-Acidity, Ex-Al and Ex-H.

We discussed inter-laboratory variation in the following sections using 2 kinds of datasets, that is, entire dataset and verified dataset after removing outlier.

Table 4.2 Data verified by Cochran-Grubbs methods

No. 091

Country	Lab.	Rep.	pH(H_2O)	pH(KCl)	Ex-Ca	Ex-Mg	Ex-K	Ex-Na	Ex-acidity	Ex-Al	Ex-H
								cmol(+)/kg			
China	cn01	1st	4.92 c	4.12	0.72	0.60	0.50	0.07	3.27 c	2.88 c	0.38 c
		2nd	4.83 c	4.03	0.76	0.58	0.51	0.06	3.62 c	3.06 c	0.55 c
	cn02	1st	4.96	4.11	0.48	0.56	0.39	0.06	5.67	4.70	0.96
		2nd	4.97	4.10	0.47	0.57	0.39	0.06	5.59	4.62	0.97
	cn03	1st	4.94	4.15	0.38	0.38	0.28	0.11	6.38	5.46	0.93
		2nd	4.95	4.15	0.38	0.38	0.30	0.13	6.40	5.47	0.93
	cn04	1st	4.73	3.89	0.31	0.42	0.38	0.06	4.20	3.49	0.70
		2nd	4.73	3.90	0.29	0.40	0.40	0.07	4.22	3.50	0.70
Indonesia	id01	1st	4.91	4.03	24.62 c	0.98 c	0.71 c	0.09	4.01	3.62	0.39
		2nd	4.93	4.04	29.61 c	1.05 c	0.77 c	0.09	4.00	3.54	0.46
	id02	1st	4.65	3.76	0.35	1.03	0.14 g	0.09	14.73 g	13.06 g	1.67
		2nd	4.61	3.75	0.35	1.03	0.14 g	0.09	14.72 g	13.06 g	1.66
Japan	jp01	1st	5.11	4.06	1.10 c	0.58	0.45	0.03	4.88	3.16	1.68
		2nd	5.11	NA	0.77 c	0.59	0.45	0.05	4.93	3.17	1.67
Malaysia	my01	1st	4.47	3.92	0.00	0.00	0.00 g	0.00	6.03	6.47 g	0.76
		2nd	4.49	3.88	0.00	0.00	0.00 g	0.00	6.12	6.55 g	0.65
Mongolia	mn01	1st	4.78	4.03	NA	NA	NA	NA	4.75	4.28	0.43
		2nd	4.78	4.06	NA	NA	NA	NA	4.75	4.28	0.43
Phillipin	ph01	1st	4.50	3.90	5.61 g	2.13 g	0.95 c	1.55 c	4.57 c	3.61	0.96
		2nd	4.50	3.90	5.57 g	2.13 g	0.91 c	1.58 c	4.37 c	3.50	0.86
Korea	ko01	1st	4.87	3.87 c	0.60	0.60	0.39	0.03	4.08	3.65	0.44
		2nd	4.90	4.07 c	0.62	0.56	0.39	0.04	4.12	3.68	0.49
Russia	ru01	1st	4.77	4.09	1.24 c	0.70 c	0.41	0.06	4.45	4.06	0.32
		2nd	4.77	4.09	1.37 c	0.79 c	0.41	0.05	4.48	4.11	0.32
Thailand	th01	1st	4.90	4.03	1.09	0.62	0.37	0.12	4.48	4.24	0.24
		2nd	4.89	4.00	1.10	0.63	0.36	0.12	4.47	4.21	0.26
Vietnum	vn01	1st	4.83 c	3.93	0.68	0.55	0.40	0.06	4.04	3.02	1.03
		2nd	4.92 c	4.01	0.70	0.57	0.39	0.06	4.04	3.05	0.99
	vn02	1st	5.17	4.54 g	NA	NA	NA	NA	3.90	3.47	0.39
		2nd	5.16	4.54 g	NA	NA	NA	NA	3.88	3.50	0.36
	vn03	1st	4.76	4.00	0.97	1.06	0.44	0.32 g	4.51	7.97 g	0.41
		2nd	4.76	4.00	0.97	1.05	0.45	0.32 g	4.53	7.97 g	0.39

No. 092

Country	Lab.	Rep.	pH(H_2O)	pH(KCl)	Ex-Ca	Ex-Mg	Ex-K	Ex-Na	Ex-acidity	Ex-Al	Ex-H
								cmol(+)/kg			
China	cn01	1st	5.83	4.75	7.15 c	1.07	0.46	0.14	0.13	0.00	0.13
		2nd	5.83	4.66	6.87 c	1.06	0.48	0.13	0.13	0.00	0.13
	cn02	1st	5.81	4.71	6.00	1.22	0.38	0.11	0.40	0.18	0.22
		2nd	5.81	4.73	6.01	1.21	0.38	0.11	0.40	0.21	0.19
	cn03	1st	5.82	4.71	5.03	0.62	0.13	0.16	0.47	0.30	0.17
		2nd	5.81	4.70	5.03	0.62	0.13	0.16	0.51	0.34	0.17
	cn04	1st	5.60	4.55	5.82	0.87	0.39	0.09	0.17	0.00	0.17
		2nd	5.59	4.56	5.81	0.88	0.40	0.10	0.17	0.00	0.17
Indonesia	id01	1st	5.81	4.67	10.70 c	1.97	0.72 c	0.18	0.15	0.08	0.07
		2nd	5.83	4.68	11.13 c	1.93	0.68 c	0.21	0.15	0.08	0.07
	id02	1st	5.19 g	4.12 g	2.18	0.65	0.07	0.04	2.49 g	2.13 g	0.36
		2nd	5.18 g	4.13 g	2.18	0.65	0.08	0.04	2.49 g	2.13 g	0.36
Japan	jp01	1st	6.17 g	4.66	10.69 c	1.18	0.44	0.10	0.19 c	0.07	0.14 c
		2nd	6.17 g	NA	9.78 c	1.19	0.45	0.13	0.09 c	0.07	0.07 c
Malaysia	my01	1st	4.71 g	3.86 g	0.00	0.00	0.00	0.00	14.44 c	12.93 c	0.31
		2nd	4.69 g	3.88 g	0.00	0.00	0.00	0.00	14.30 c	13.04 c	0.31
Mongolia	mn01	1st	5.71	4.69	NA	NA	NA	NA	0.43	0.00	0.43
		2nd	5.71	4.70	NA	NA	NA	NA	0.43	0.00	0.43
Phillipin	ph01	1st	5.40 c	4.60	22.72	2.13	0.98 c	1.13 g	0.31	0.10	0.21
		2nd	5.50 c	4.60	22.68	2.13	0.93 c	1.16 g	0.27	0.09	0.18
Korea	ko01	1st	5.68	4.58	9.13	1.14	0.37	0.09	0.15	0.11	0.10
		2nd	5.69	4.69	9.12	1.10	0.37	0.08	0.18	0.10	0.08
Russia	ru01	1st	5.67	4.77	11.34	1.53 c	0.41	0.12	0.11	0.00	0.11
		2nd	5.67	4.77	11.31	1.34 c	0.42	0.10	0.11	0.00	0.11
Thailand	th01	1st	5.79	4.68	15.07	1.04	0.28	0.23	0.10	0.06	0.04
		2nd	5.78	4.68	15.05	1.06	0.26	0.21	0.08	0.03	0.05
Vietnum	vn01	1st	5.80 c	4.69	10.69	0.86	0.43	0.13	0.32	0.00	0.32
		2nd	5.76 c	4.76	10.67	0.86	0.43	0.13	0.33	0.00	0.33
	vn02	1st	5.68	5.17 g	NA	NA	NA	NA	0.27	0.00	0.32
		2nd	5.66	5.14 g	NA	NA	NA	NA	0.27	0.00	0.32
	vn03	1st	5.51	4.65	11.95	2.24	0.44	0.44 g	0.19	0.37	0.08
		2nd	5.51	4.65	11.96	2.21	0.44	0.44 g	0.18	0.37	0.08

Note; The outliers judged by Cochran and Grubbs methods were marked with c and g, respectively (lab. code was explained in appendix 4.1)

4.3.2 Outlines of inter-laboratory variation

The statistical summary was calculated for both entire and verified datasets (Table 4.2). Inter-laboratory variation was visually shown in Figure 4.1.1 and 4.1.2 for both datasets by box-and-whisker plot with coefficient of variances (CVs).

The CVs of pH (H_2O) and pH (KCl) were very small (less than 5 %) in entire and verified datasets. Removing outliers by Cochran-Grubbs methods improved CVs of most parameters of exchangeable cations (e.g. from 245% to 58.6% in Ex-Ca of No.091 and from 280% to 53% in Ex-Acidity of No.092). Meanwhile, CVs were still high (around 50%) in almost all parameters of exchangeable cations, although CVs of Ex-K, Ex-Acidity and Ex-Al of No.091 were relatively low (less than 20%).

Box-and-whisker plot provides the five-number summaries: lower quartile, median and upper quartile shown by box and bold lines, and maximum and minimum values drawn by error bars. In addition, the values lower quartile minus 1.5 times the inter-quartile range, or larger than the upper quartile plus 1.5 times the inter-quartile range are shown as outliers, that is, non-parametrical outliers.

The plot of entire data visually showed one or two clear (non-parametrical) outlier in respective parameters (Figure 4.1.1). Although the (non-parametrical) outliers clearly decreased after Cochran-Grubbs test (Figure 4.1.2), inter-laboratory variation in each parameter was still large. Especially, the range from lower quartile to upper quartile was relatively wide in Ex-Ca and Ex-Mg of No.092 and Ex-Acidity, Ex-Al and Ex-H of No.091, in which the average concentrations were high. Extremely high CV in Ex-Al of No.092 (133%) in the verified dataset (Figure 4.1.2) was due to the very low concentration including zero (lower determination limit). In this case, the range of inter-laboratory variation was very small, as shown by thin box in the figure.

Table 4.3 Statistical summary of the entire and verified datasets

(Entire data)									
Statistics	pH(H_2O)	pH(KCl)	Ex-Ca	Ex-Mg	Ex-K	Ex-Na	Ex-acidity	Ex-Al	Ex-H
			cmol(+) kg^{-1}						
	No. 091								
Number of Laboratories	16	16	14	14	14	14	16	16	16
Total average	4.8	4.0	2.90	0.73	0.42	0.19	5.26	4.83	0.73
Median	4.9	4.0	0.72	0.59	0.40	0.07	4.48	3.88	0.59
Maximum	5.2	4.5	27.11	2.13	0.93	1.57	14.73	13.06	1.68
Minimum	4.5	3.8	0.00	0.00	0.00	0.00	3.44	2.97	0.25
Standard deviation	0.2	0.2	7.10	0.49	0.22	0.40	2.65	2.58	0.44
CV (%)*1	3.9	4.1	245.0	66.7	53	209	50.4	53.4	61
	No. 092								
Number of Laboratories	16	16	14	14	14	14	16	16	16
Total average	5.6	4.6	9.15	1.17	0.39	0.21	1.26	1.02	0.20
Median	5.7	4.7	9.69	1.09	0.41	0.12	0.23	0.08	0.17
Maximum	6.2	5.2	22.70	2.22	0.95	1.14	14.37	12.98	0.43
Minimum	4.7	3.9	0.00	0.00	0.00	0.00	0.09	0.00	0.04
Standard deviation	0.3	0.3	5.63	0.61	0.24	0.29	3.54	3.23	0.12
CV (%)*1	5.8	6.1	61.6	52.3	61	135	280.4	315.4	61
(Verified data)*2									
Statistics	pH(H_2O)	pH(KCl)	Ex-Ca	Ex-Mg	Ex-K	Ex-Na	Ex-acidity	Ex-Al	Ex-H
			cmol(+) kg^{-1}						
	No. 091								
Number of Laboratories	14	14	10	11	10	12	13	12	15
Total average	4.8	4.0	0.56	0.58	0.41	0.07	4.73	3.89	0.75
Median	4.8	4.0	0.55	0.58	0.40	0.07	4.48	3.62	0.70
Maximum	5.2	4.2	1.10	1.06	0.51	0.12	6.39	5.46	1.68
Minimum	4.5	3.8	0.00	0.00	0.29	0.00	3.89	3.03	0.25
Standard deviation	0.2	0.1	0.33	0.29	0.06	0.03	0.82	0.69	0.45
CV (%)*1	4.2	2.6	58.6	49.9	14.3	51.8	17.2	17.7	60.5
	No. 092								
Number of Laboratories	12	14	13	13	12	12	13	14	15
Total average	5.8	4.7	9.01	1.15	0.32	0.12	0.25	0.09	0.20
Median	5.8	4.7	9.13	1.06	0.39	0.11	0.19	0.06	0.17
Maximum	6.2	5.2	22.70	2.22	0.47	0.22	0.49	0.37	0.43
Minimum	5.5	4.6	0.00	0.00	0.00	0.00	0.09	0.00	0.04
Standard deviation	0.2	0.1	5.84	0.63	0.16	0.06	0.13	0.12	0.12
CV (%)*1	2.9	3.0	64.8	55.0	50.5	52.6	53.0	133.0	60.1

Note: *1. CV, Coefficient of variance (%) = (standard deviation/average)*100 *2.Outliers judged by Cochran-Grubbs

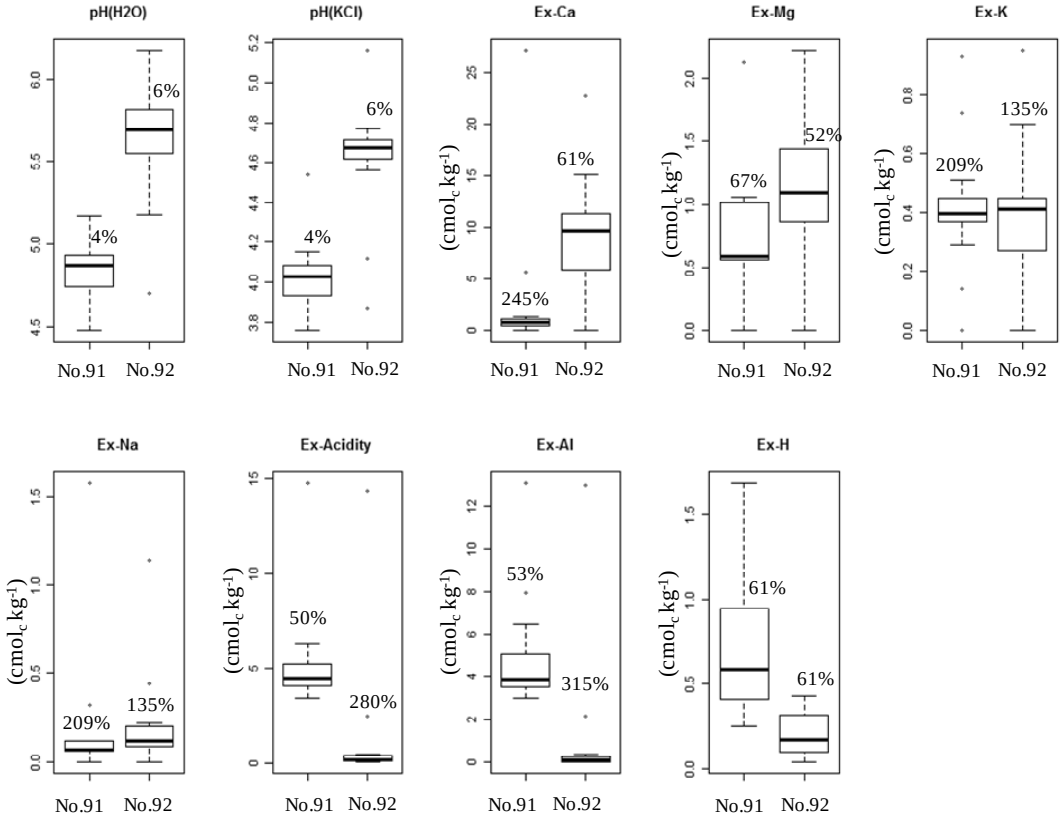


Figure 4.1.1 Box-and-whisker plot and CVs for entire dataset of No.091 and No.092.

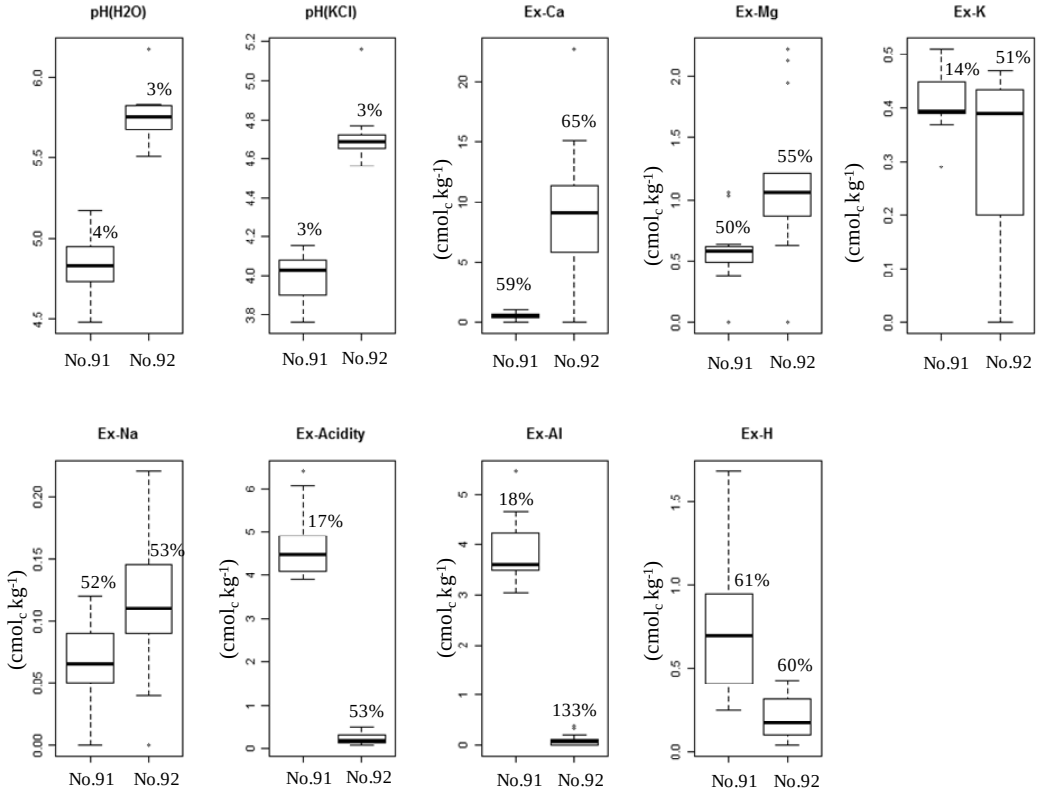


Figure 4.1.2 Box-and-whisker plot and CVs for verified dataset of No.091 and No.092.

4.3.3 Analysis of variance for entire and verified datasets

"Repeatability-precision", "within-laboratory-precision" and "inter-laboratories-precision" were discussed using analysis of variance model (ANOVA) (Table 4.4).

1) Repeatability-precision

Repeatability standard deviations were small for most parameters in the verified data. The CVs were less than 10% except Ex-Na of both samples and Ex-Al of No.092. CVs of pH (H_2O), pH(KCl), Ex-Ca, Mg and K were smaller than 5%. Very low average including zero (less than determination limit) was observed in the parameters which provide high CVs (e.g. Ex-Na and Ex-Al).

The result suggests that triplicate analyses were carried out under the same condition. The participating laboratories could analyze the parameters with their own standard procedures and stable instruments.

2) Within-laboratory precision

Within-laboratory standard deviations were also relatively small for most of the parameters in the verified data, and CVs of most parameters were smaller than 10%.

CVs of within-laboratory precision in pH (H_2O) and basic cations were smaller than those of repeatability precision. It was suggested that the average of triplicate analyses under the repeatability condition could be representative value for the analysis in a laboratory. We assumed that participating laboratories could analyze the parameters with their own standard procedures.

Meanwhile, CVs of within-laboratory prevision in pH (KCl) and Ex-acidity, which is extracted by KCl solution, were relatively large and higher than those of repeatability precision. The CVs might be derived from the quality of the KCl solution. The concentration is more effective for extracted volume of Ex-Al^{3+} from the soil-mineral surfaces with variable negative charge than those with permanent negative charge. In the soil samples of No.091 and No.092, a variable negative charge may be high because this soil includes high 1:1 minerals and organic matter which could be source of variable charge. We assumed that KCl solution concentration was different between 1^{st} and 2^{nd} analysis in the laboratory.

3) Inter-laboratories precision

CVs of the inter-laboratories precision were smaller than 6% and 14% in pH (H_2O) and pH (KCl), respectively. However, CVs of exchangeable cations were large. We discussed the possible factor of the large CVs of Inter-laboratory precisions, in the following section.

4) Calculation of permissible tolerance

The repeatability limit and within-laboratory reproducibility limit might be enough small to use as a

reference value for the repeat analysis on the instrumental analysis in the respective laboratory. The reproducibility limit and inter-laboratories precision should be improved for exchangeable cations. We discussed the factor of inter-laboratory variation in each parameter, in the following section.

Table 4.4.1 Analysis of variance for the entire data

Statistics	No. 091								
	pH(H_2O)	pH(KCl)	Ex-Ca	Ex-Mg	Ex-K	Ex-Na	Ex-acidity	Ex-Al	Ex-H
Number of Laboratories	16	16	14	14	14	14	16	16	16
Total sum of square	215064	140538	59212	3802	1231	259	254615	214621	4917
ST/lmd	2240	1464	705	45	15	3	2652	2236	51
Number of Data	96	93	84	84	84	84	96	96	96
Total sum	463.8	374.9	243.34	61.66	35.09	16.10	504.59	463.27	70.12
Total average	4.8	4.0	2.90	0.73	0.42	0.19	5.26	4.83	0.73
Sum of square inter-laboratories (S_R)	3.2	25.1	3934.00	18.71	3.90	12.61	631.08	598.25	17.58
Sum of square within-laboratory (S_{RW})	0.0	24.8	37.55	0.03	0.01	0.00	0.27	0.11	0.09
Sum of square repeatablility (S_r)	0.0	0.0	0.47	0.05	0.04	0.04	0.28	0.16	0.19
Total sum of square (S_T)	3.3	49.9	3972.02	18.78	3.95	12.66	631.63	598.51	17.86
Inter-laboratories degree of freedom (ϕ_R)	15	15	13	13	13	13	15	15	15
Within-laboratory degree of freedom (ϕ_{RW})	16	16	14	14	14	14	16	16	16
Repeatability degree of freedom (ϕ_r)	64	64	56	56	56	56	64	64	64
Total degree of freedom (ϕ_T)	95	95	83	83	83	83	95	95	95
Inter-laboratories variance ($V_R = S_R/\phi_R$)	0.21	1.67	302.615	1.439	0.300	0.970	42.072	39.883	1.172
Within-laboratory variance ($V_{RW} = S_{RW}/\phi_{RW}$)	0.00	1.55	2.682	0.002	0.001	0.000	0.017	0.007	0.006
Repeatability variance ($V_r = S_r/\phi_r$)	0.00	0.00	0.008	0.001	0.001	0.001	0.004	0.002	0.003
Laboratory component of variance ($s_b^2 = (V_R-V_{RW})/(2*3)$)	0.04	0.02	49.989	0.240	0.050	0.162	7.009	6.646	0.194
Within-laboratory component of variance ($s_c^2 = (V_{RW}-V_r)/3$)	0.00	0.52	0.891	0.000	0.000	0.000	0.004	0.001	0.001
Repeatability component of variance ($s_r^2 = V_r$)	0.00	0.00	0.008	0.001	0.001	0.001	0.004	0.002	0.003
Inter-laboratories standard deviation ($s_R = SQRT(s_r^2/(2*3) + s_c^2/2 + s_b^2)$)	0.2	0.5	7.10	0.49	0.22	0.40	2.65	2.58	0.44
Within-laboratory standard deviation ($s_{RW} = SQRT(s_r^2/3 + s_c^2)$)	0.0	0.7	0.95	0.02	0.02	0.01	0.08	0.05	0.04
Repeatability standard deviation ($s_r = SQRT(s_r^2)$)	0.0	0.0	0.09	0.03	0.03	0.03	0.07	0.05	0.05
Inter-laboratories precision CV (%)	3.9	13.1	245.16	66.72	53.53	209.78	50.38	53.43	60.51
Within-laboratory precision CV (%)	0.5	17.8	32.64	3.34	3.85	5.13	1.43	0.97	5.89
Repeatability precision CV (%)	0.5	0.4	3.16	3.95	6.09	13.80	1.25	1.03	7.50
Reproducibility limit ($R = D(2, 0.95)*s_R$)	0.53	1.48	19.885	1.371	0.626	1.126	7.414	7.219	1.238
Within-laboratory-reproducibility limit ($R_W = D(2, 0.95)*s_{Rw}$)	0.07	2.01	2.648	0.069	0.045	0.028	0.211	0.131	0.121
Repeatability limit ($r = D(3, 0.95)*s_r$)	0.07	0.06	0.302	0.096	0.084	0.087	0.217	0.164	0.181

Statistics	No. 092								
	pH(H_2O)	pH(KCl)	Ex-Ca	Ex-Mg	Ex-K	Ex-Na	Ex-acidity	Ex-Al	Ex-H
Number of Laboratories	16	16	14	14	14	14	16	16	16
Total sum of square	292761	184574	590149	9657	1078	322	14700	9671	352
ST/lmd	3050	1923	7026	115	13	4	153	101	4
Number of Data	96	93	84	84	84	84	96	96	96
Total sum	541.1	429.6	768.21	98.27	32.83	17.95	121.24	98.34	18.77
Total average	5.6	4.6	9.15	1.17	0.39	0.21	1.26	1.02	0.20
Sum of square inter-laboratories (S_R)	9.6	36.4	2475.21	29.24	4.57	6.40	1128.59	939.63	1.26
Sum of square within-laboratory (S_{RW})	0.0	32.7	1.64	0.06	0.01	0.00	0.05	0.02	0.01
Sum of square repeatablility (S_r)	0.0	0.0	4.44	0.10	0.07	0.05	0.02	0.09	0.08
Total sum of square (S_T)	9.7	69.1	2481.29	29.40	4.65	6.46	1128.66	939.74	1.35
Inter-laboratories degree of freedom (ϕ_R)	15	15	13	13	13	13	15	15	15
Within-laboratory degree of freedom (ϕ_{RW})	16	16	14	14	14	14	16	16	16
Repeatability degree of freedom (ϕ_r)	64	64	56	56	56	56	64	64	64
Total degree of freedom (ϕ_T)	95	95	83	83	83	83	95	95	95
Inter-laboratories variance ($V_R = S_R/\phi_R$)	0.64	2.43	190.401	2.249	0.352	0.493	75.240	62.642	0.084
Within-laboratory variance ($V_{RW} = S_{RW}/\phi_{RW}$)	0.00	2.04	0.117	0.004	0.001	0.000	0.003	0.001	0.001
Repeatability variance ($V_r = S_r/\phi_r$)	0.00	0.00	0.079	0.002	0.001	0.001	0.000	0.001	0.001
Laboratory component of variance ($s_b^2 = (V_R-V_{RW})/(2*3)$)	0.11	0.06	31.714	0.374	0.059	0.082	12.539	10.440	0.014
Within-laboratory component of variance ($s_c^2 = (V_{RW}-V_r)/3$)	0.00	0.68	0.013	0.001	0.000	0.000	0.001	0.000	0.000
Repeatability component of variance ($s_r^2 = V_r$)	0.00	0.00	0.079	0.002	0.001	0.001	0.000	0.001	0.001
Inter-laboratories standard deviation ($s_R = SQRT(s_r^2/(2*3) + s_c^2/2 + s_b^2)$)	0.3	0.6	5.63	0.61	0.24	0.29	3.54	3.23	0.12
Within-laboratory standard deviation ($s_{RW} = SQRT(s_r^2/3 + s_c^2)$)	0.0	0.8	0.20	0.04	0.01	0.01	0.03	0.02	0.01
Repeatability standard deviation ($s_r = SQRT(s_r^2)$)	0.0	0.0	0.28	0.04	0.04	0.03	0.02	0.04	0.04
Inter-laboratories precision CV (%)	5.8	13.8	61.60	52.34	61.93	134.10	280.39	315.42	60.50
Within-laboratory precision CV (%)	0.4	17.9	2.16	3.22	3.45	4.83	2.59	2.17	7.66
Repeatability precision CV (%)	0.4	0.3	3.08	3.59	9.16	13.86	1.39	3.59	18.08
Reproducibility limit ($R = D(2, 0.95)*s_R$)	0.92	1.78	15.773	1.714	0.678	0.802	9.915	9.047	0.331
Within-laboratory-reproducibility limit ($R_W = D(2, 0.95)*s_{Rw}$)	0.06	2.31	0.554	0.106	0.038	0.029	0.092	0.062	0.042
Repeatability limit ($r = D(3, 0.95)*s_r$)	0.07	0.05	0.929	0.138	0.118	0.098	0.058	0.121	0.117

Table 4.4.2 Analysis of variance for the verified data

Statistics	No. 091								
	pH(H_2O)	pH(KCl)	Ex-Ca	Ex-Mg	Ex-K	Ex-Na	Ex-acidity	Ex-Al	Ex-H
Number of Laboratories	14	14	10	11	11	12	15	14	16
Total sum of square	164228	104875	1131	1470	588	23	144117	83731	4757
ST/lmd	1955	1249	19	22	9	0	1601	997	50
Number of Data	84	81	60	66	60	72	81	75	93
Total sum	405.3	323.8	33.63	38.34	24.24	4.81	379.63	289.36	68.97
Total average	4.8	4.0	0.56	0.58	0.40	0.07	4.69	3.86	0.74
Sum of square inter-laboratories (S_R)	3.2	22.4	5.83	5.01	1.06	0.08	209.64	138.81	18.39
Sum of square within-laboratory (S_{RW})	0.0	24.7	0.00	0.00	0.00	0.00	19.64	14.12	0.50
Sum of square repeatablility (S_r)	0.0	0.0	0.02	0.02	0.02	0.00	0.26	0.11	0.17
Total sum of square (S_T)	3.2	47.2	5.86	5.03	1.08	0.08	229.53	153.03	19.07
Inter-laboratories degree of freedom (ϕ_R)	13	13	9	10	10	11	14	13	15
Within-laboratory degree of freedom (ϕ_{RW})	14	14	10	11	11	12	15	14	16
Repeatability degree of freedom (ϕ_r)	56	56	40	44	44	48	60	56	64
Total degree of freedom (ϕ_T)	83	83	59	65	65	71	89	83	95
Inter-laboratories variance ($V_R = S_R/\phi_R$)	0.24	1.72	0.648	0.501	0.106	0.007	14.974	10.677	1.226
Within-laboratory variance ($V_{RW} = S_{RW}/\phi_{RW}$)	0.00	1.77	0.000	0.000	0.000	0.000	1.309	1.009	0.031
Repeatability variance ($V_r = S_r/\phi_r$)	0.00	0.00	0.001	0.001	0.000	0.000	0.004	0.002	0.003
Laboratory component of variance ($s_b^2 = (V_R-V_{RW})/(2*3)$)	0.04	-0.01	0.108	0.083	0.018	0.001	2.277	1.611	0.199
Within-laboratory component of variance ($s_c^2 = (V_{RW}-V_r)/3$)	0.00	0.59	0.000	0.000	0.000	0.000	0.435	0.336	0.010
Repeatability component of variance ($s_r^2 = V_r$)	0.00	0.00	0.001	0.001	0.000	0.000	0.004	0.002	0.003
Inter-laboratories standard deviation ($s_R = SQRT(s_r^2/(2*3) + s_c^2/2 + s_b^2)$)	0.2	0.5	0.33	0.29	0.13	0.03	1.58	1.33	0.45
Within-laboratory standard deviation ($s_{RW} = SQRT(s_r^2/3 + s_c^2)$)	0.0	0.8	0.01	0.01	0.01	0.01	0.66	0.58	0.10
Repeatability standard deviation ($s_r = SQRT(s_r^2)$)	0.0	0.0	0.02	0.02	0.02	0.01	0.07	0.04	0.05
Inter-laboratories precision CV (%)	4.2	13.4	58.63	49.73	32.95	51.55	33.71	34.58	60.96
Within-laboratory precision CV (%)	0.2	19.2	2.09	2.05	2.09	10.76	14.09	15.03	13.80
Repeatability precision CV (%)	0.4	0.4	4.07	3.95	4.90	13.31	1.39	1.14	6.96
Reproducibility limit ($R = D(2, 0.95)*s_R$)	0.57	1.50	0.920	0.809	0.373	0.096	4.423	3.735	1.266
Within-laboratory-reproducibility limit ($R_W = D(2, 0.95)*s_{Rw}$)	0.03	2.15	0.033	0.033	0.024	0.020	1.850	1.623	0.287
Repeatability limit ($r = D(3, 0.95)*s_r$)	0.07	0.06	0.075	0.076	0.065	0.029	0.215	0.146	0.170
Statistics	No. 092								
	pH(H_2O)	pH(KCl)	Ex-Ca	Ex-Mg	Ex-K	Ex-Na	Ex-acidity	Ex-Al	Ex-H
Number of Laboratories	12	15	13	13	12	12	14	9	15
Total sum of square	142381	145657	493823	8037	525	72	370	59	329
ST/lmd	1978	1618	6331	103	7	1	4	1	4
Number of Data	66	81	78	78	72	72	78	84	90
Total sum	377.3	381.7	702.73	89.65	22.92	8.46	19.24	7.67	18.13
Total average	5.7	4.7	9.01	1.15	0.32	0.12	0.25	0.09	0.20
Sum of square inter-laboratories (S_R)	180.4	148.7	2454.99	28.78	1.72	0.25	1.59	0.76	1.21
Sum of square within-laboratory (S_{RW})	0.0	32.7	1.37	0.01	0.00	0.00	0.01	0.00	0.00
Sum of square repeatablility (S_r)	0.0	0.0	3.74	0.05	0.01	0.01	0.01	0.08	0.02
Total sum of square (S_T)	180.4	181.4	2460.10	28.84	1.73	0.26	1.60	0.84	1.23
Inter-laboratories degree of freedom (ϕ_R)	11	14	12	12	11	11	13	8	14
Within-laboratory degree of freedom (ϕ_{RW})	12	15	13	13	12	12	14	9	15
Repeatability degree of freedom (ϕ_r)	48	60	52	52	48	48	56	36	60
Total degree of freedom (ϕ_T)	71	89	77	77	71	71	83	53	89
Inter-laboratories variance ($V_R = S_R/\phi_R$)	16.40	10.62	204.582	2.398	0.157	0.022	0.122	0.095	0.086
Within-laboratory variance ($V_{RW} = S_{RW}/\phi_{RW}$)	0.00	2.18	0.106	0.000	0.000	0.000	0.000	0.000	0.000
Repeatability variance ($V_r = S_r/\phi_r$)	0.00	0.00	0.072	0.001	0.000	0.000	0.000	0.002	0.000
Laboratory component of variance ($s_b^2 = (V_R-V_{RW})/(2*3)$)	2.73	1.41	34.079	0.400	0.026	0.004	0.020	0.016	0.014
Within-laboratory component of variance ($s_c^2 = (V_{RW}-V_r)/3$)	0.00	0.73	0.011	0.000	0.000	0.000	0.000	-0.001	0.000
Repeatability component of variance ($s_r^2 = V_r$)	0.00	0.00	0.072	0.001	0.000	0.000	0.000	0.002	0.000
Inter-laboratories standard deviation ($s_R = SQRT(s_r^2/(2*3) + s_c^2/2 + s_b^2)$)	1.7	1.3	5.84	0.63	0.16	0.06	0.14	0.13	0.12
Within-laboratory standard deviation ($s_{RW} = SQRT(s_r^2/3 + s_c^2)$)	0.0	0.9	0.19	0.01	0.01	0.01	0.01	0.01	0.01
Repeatability standard deviation ($s_r = SQRT(s_r^2)$)	0.0	0.0	0.27	0.03	0.01	0.01	0.01	0.05	0.02
Inter-laboratories precision CV (%)	28.9	28.2	64.81	55.01	50.75	51.90	57.86	137.91	59.57
Within-laboratory precision CV (%)	0.2	18.1	2.08	1.11	1.79	8.48	4.83	13.69	4.14
Repeatability precision CV (%)	0.4	0.3	2.98	2.76	3.64	11.16	4.63	50.94	8.78
Reproducibility limit ($R = D(2, 0.95)*s_R$)	4.63	3.73	16.350	1.770	0.452	0.171	0.400	0.352	0.336
Within-laboratory-reproducibility limit ($R_W = D(2, 0.95)*s_{Rw}$)	0.03	2.39	0.525	0.036	0.016	0.028	0.033	0.035	0.023
Repeatability limit ($r = D(3, 0.95)*s_r$)	0.07	0.04	0.886	0.105	0.038	0.043	0.038	0.153	0.058

4.3.4 Inter-laboratory variation in each parameter

We showed the data of each laboratory by scatter plot between No.091 and No.092. Each median of verified dataset was shown by solid line in the plot. As a guide for comparison between each parameter, 20% of median was also shown by dashed lines while 0.2 pH unit were used for pH (H_2O) and pH (KCl). Outliers selected by Cohcran-Grubbs method were denoted by ¶ for No.091 and § for No.092. The plot did not include extreme outliers in the plot for eye-friendly.

1) pH

Linear correlation between No.091 and No.092 indicated the systematic errors of the inter-laboratory variation in pH (Fig. 4.2.1 and Fig.4.2.2). Most laboratories were included within the range of 0.2 pH unit for No.091 and No.092.

The systematic error might be caused by the condition of pure water, standard solution or glass electrode. In addition, measuring time to the stabilization of value may lead to the variation because a carbon dioxide pressure, linkage of KCl solution from the electrode or settling the clay particles in the sample tube changes the ion balance in soil suspension.

2) Base cations

The plot of exchangeable cations (Fig. 4.2.3 to 4.2.6) also suggested systematic error of inter-laboratory variations with a few exceptions. The data was much uniformly distributed in Ex-Ca in comparison with Ex-Mg and Ex-K which were normally distributed. Almost all laboratories were not included in the range of 20% of median value for Ex-Ca, while the prepared value in half laboratory were included in the range in Ex-Mg and Ex-K.

By a chemical interaction on electrical double layer of soil colloids composed of clay mineral and organic matter, pH and concentration of ammonium acetate (extraction liquid) directly regulate the concentration of exchangeable cations. In case of acidic soil like a No.091 and No.092, high concentration and high pH of extraction liquid increases exchangeable base cations by the interaction. In addition, Ex-Ca might be affected by AAS analysis because extracted soil solution includes much aluminum and iron which are interfering substance for Ca^{2+}.

3) Acidity

The plots of Ex-Acidity, Ex-Al and Ex-H indicated random error of inter-laboratory variation (Fig. 4.2.7 to 4.2.9). Some laboratories were outside 20% range of median in No.092, while almost all laboratories were included in the range in No.091.

The random error was derived from very low concentration of No.092. We assumed that titration in low concentration are easily affected by factor of volumetric solution or end-point detection.

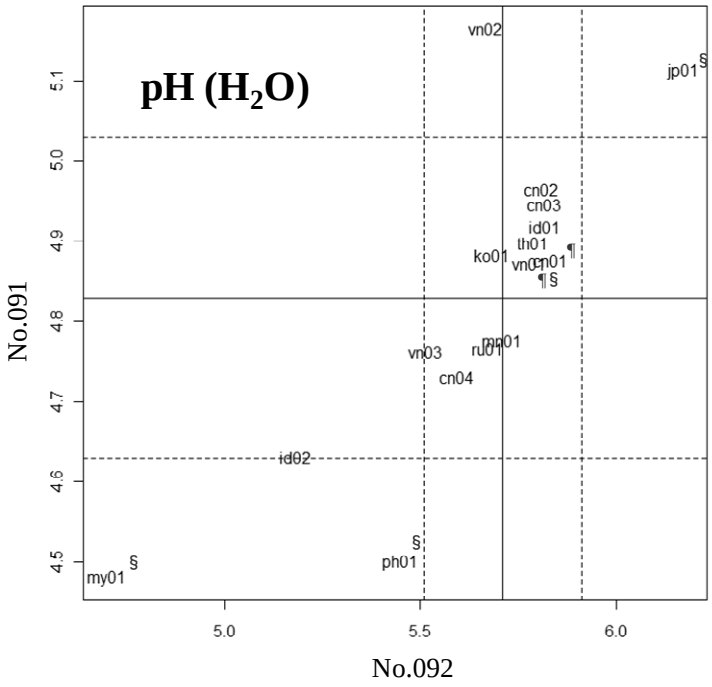


Figure 4.2.1 Scatter diagram of pH (H_2O) between No.091 and No.092 (Solid lines show median of verified datasets and dashed lines show 0.2 pH units from the median, ¶ ; outlier in No.091, § ; outlier in No.092, lab. codes were explained in appendix 4.1)

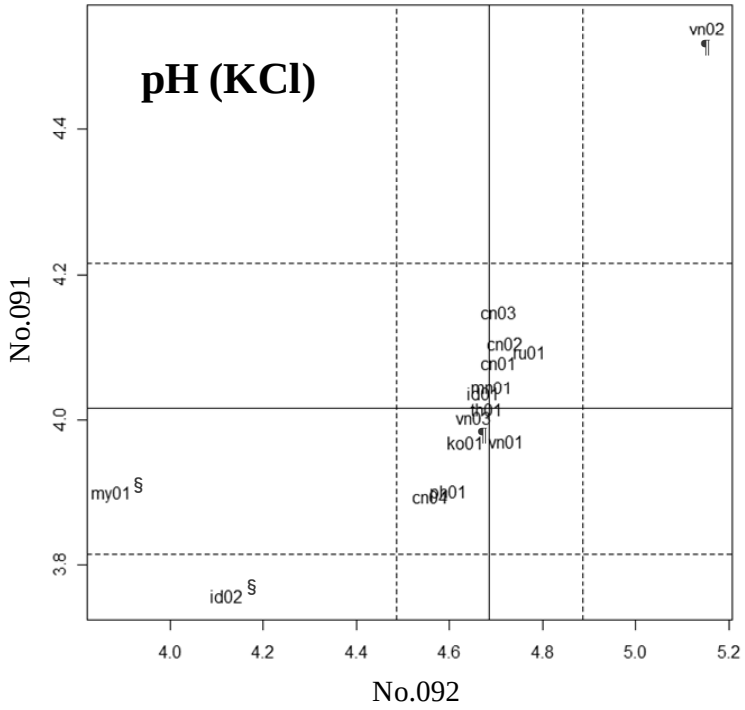


Figure 4.2.2 Scatter diagram of pH (KCl) between No.091 and No.092

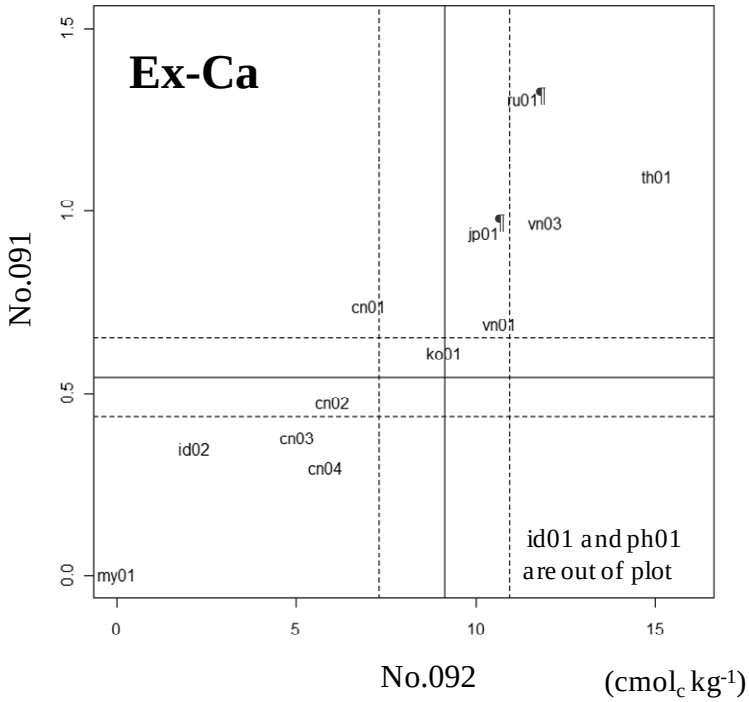


Figure 4.2.3 Scatter plot of Ex-Ca between No.091 and No.092 (Solid lines show median of verified datasets and dashed lines show 20% from the median, ¶ ; outlier in No.091, § ; outlier in No.092, lab. codes were explained in appendix 4.1)

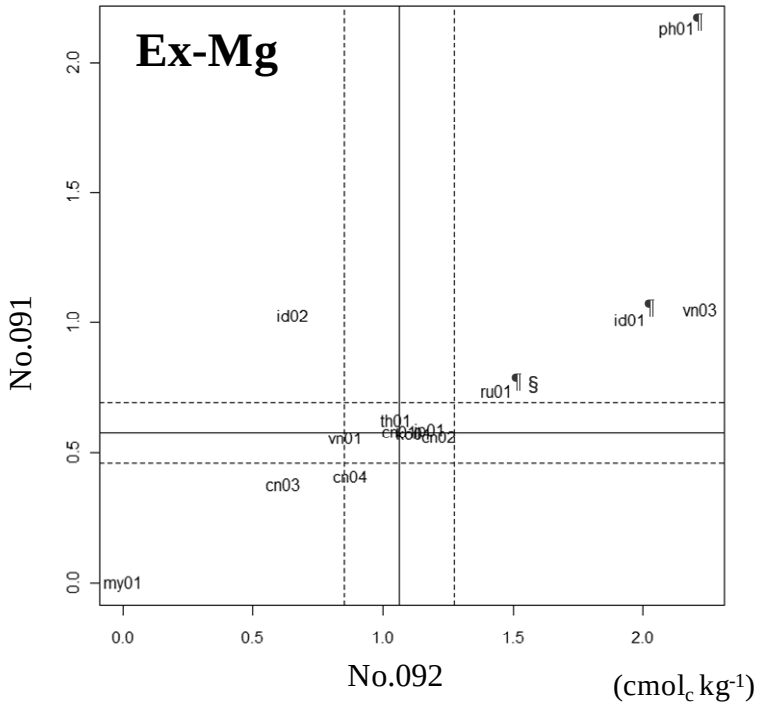


Figure 4.2.4 Scatter plot of Ex-Mg between No.091 and No.092

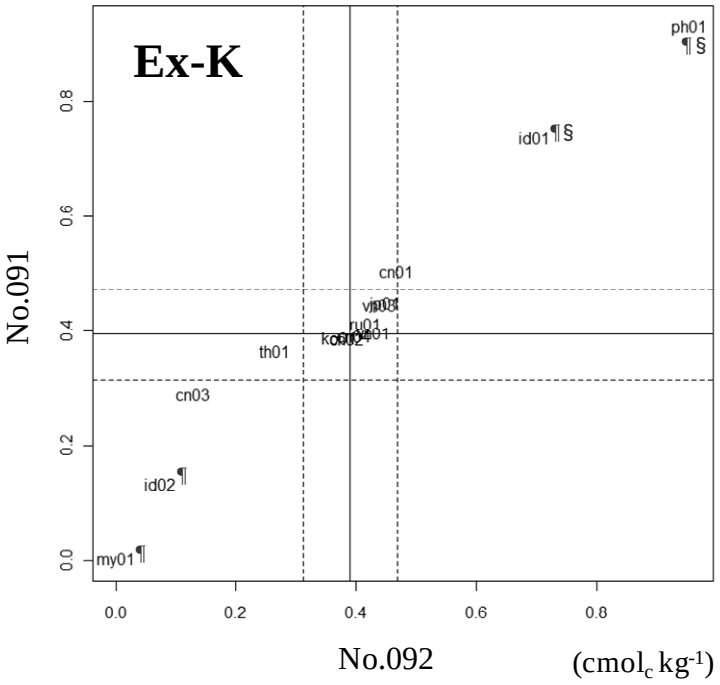


Figure 4.2.5 Scatter plot of Ex-K between No.091 and No.092

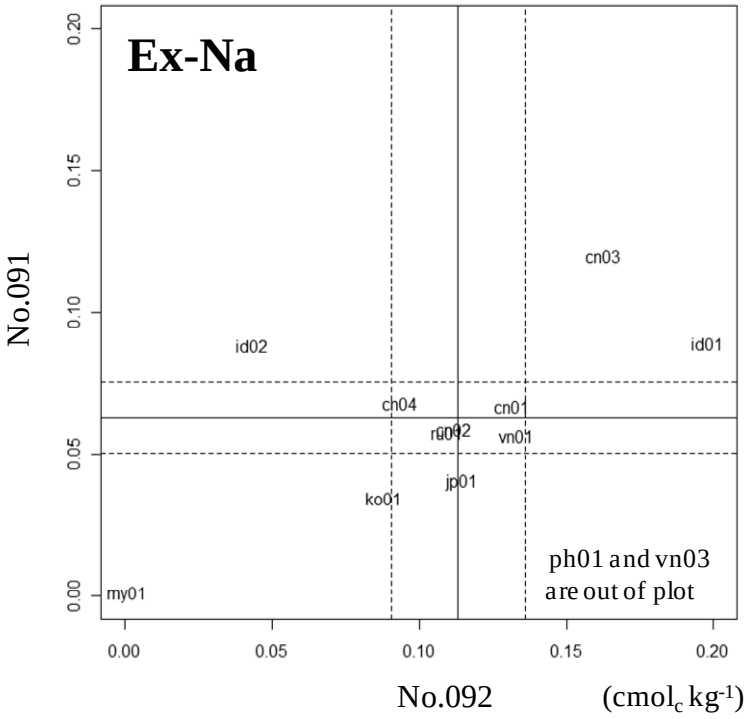


Figure 4.2.6 Scatter plot of Ex-Na between No.091 and No.092

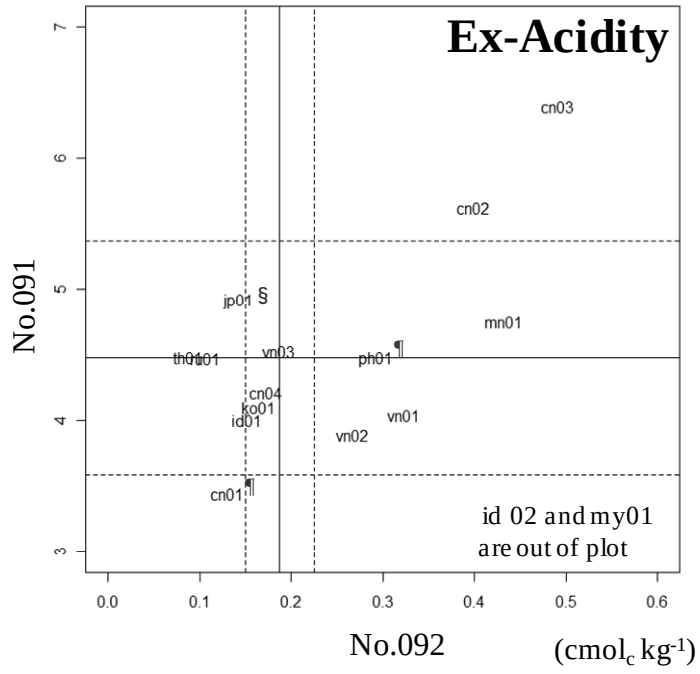


Figure 4.2.7 Scatter plot of Ex-Acidity between No.091 and No.092

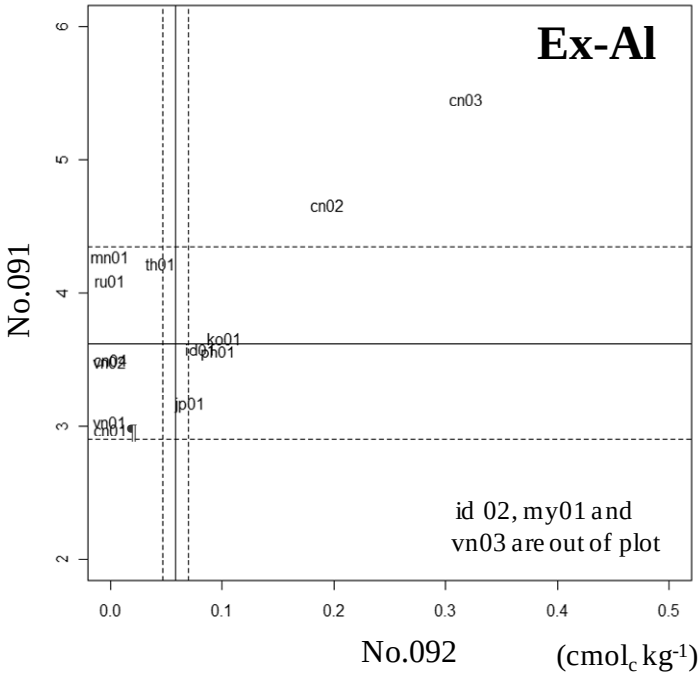


Figure 4.2.8 Scatter plot of Ex-Al between No.091 and No.092

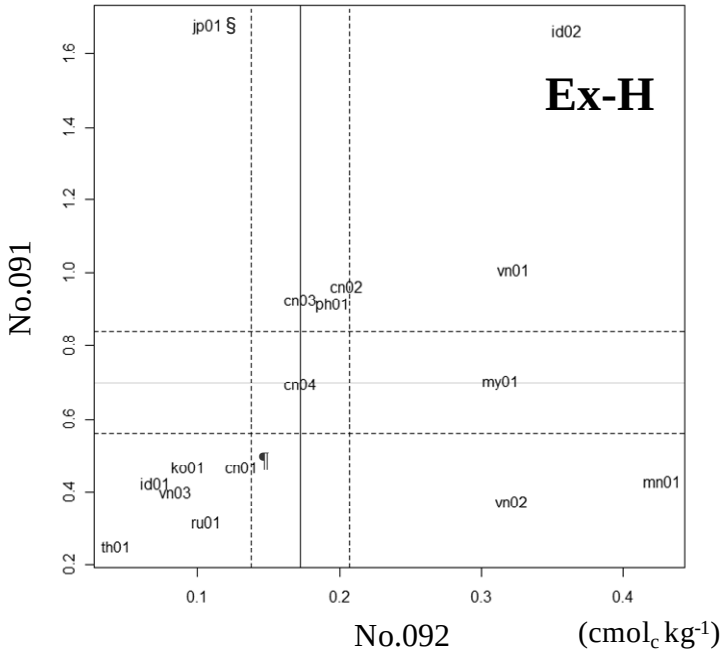


Figure 4.2.9 Scatter plot of Ex-H between No.091 and No.092

4.3.5 Comparison with information on Laboratories

1) Number of analysts and their experience

Number of analysts and years of their experience were shown in Table 4.5. The same analyst carried out the repeat analyses in most laboratories for all parameters. No relationship between the number of analyst, years of experience and the outliers was suggested.

2) Analytical instruments and condition of instruments

Analytical instruments used for the measurement, procedures for extraction of Ex-base cations, and size of burette used for the titration method in Ex-acidity were shown in Table 4.6. All the laboratories used AAS for measurement of Ex-Ca and Mg, and 6 laboratories used FEP for Ex-K and Na. Years in use of instruments were varied from 1 to 21 for AAS and from 2 to 6 for FEP. All the laboratories except “vn01” and “vn03” applied Sr or La for measurement of Ex-Ca and Mg by AAS, although “id01” and “id02” have not reported it.

Eight laboratories used percolation tube procedures for extraction of exchangeable base cations, while Buchner funnel procedures, centrifuge procedures and automatic extractor procedures were used in 3, 1 and 2 laboratories, respectively. No clear difference was observed among data by different procedures. As for size of burette for titration of Ex-acidity, capacities were varied from 3 to 50 ml while minimum graduates were 0.00125 to 0.1.

3) Date of analysis

Date of analysis in the respective laboratories and days used for the analysis were shown in Table 4.7. Most laboratories carried out the analysis by February. There were no significant implication between date of analysis and the data. Days used for the analysis were only one or two days in most laboratories. Interval between the first and second analyses of the repeat analyses was varied from 0 (in a same day) to 71 days. It was suggested that repeat analyses would be carried out with several-day interval (three days or more) in order to estimate actual within-laboratory reproducibility, as a supplementary instruction for the project, based on the discussion at SAC3. Mostly half of the laboratories followed the recommendation, although a few laboratories might conduct the instrumental analysis of both samples in a same day.

Table 4.5. Number and experience of analyst

Lab.	Ex-base cations			Ex-acidity			Analyst
	Number of analyst	Years of experience Chemical	Soil	Number of	Years of experience Chemical	Soil	
cn01	1	24	6	1	24	5	s
cn02	1	14	11	1	14	11	d
cn03	1	11	11	1	19	19	d
cn04	3	7	5	2	7	5	d
id01	1	7	4	1	7	4	s
id02	2	n	n	1	11	9	d
jp01	1	1	1	1	1	1	s
my01	2	3	3	1	3	3	d
mn01	-	-	-	1	16	16	-
ph01	1	21	2	1	33	33	d
kr01	1	11	11	1	11	11	s
ru01	1	5	5	1	14	14	d
th01	2	5/1	5/1	2	5/1	5/1	s
vn01	1	17	12	1	17	12	s
vn02	-	-	-	1	7	4	-
vn03	1	6	6	1	4	4	d

Note: -, Not measured; n, no information; s, Same analysts; d, Different analysts

Table 4.6 Analytical instruments and their conditions for exchangeable cations

Lab.	Sample	Ex-Ca		Ex-Mg			Ex-K		Ex-Na		
		Instrument	Years*1	Instrument	Years		Instrument	Years	Instrument	Years	
cn01	No.091	AAS	4	AAS	4	Sr	FEP	4	FEP	4	Sr
	No.092	AAS	4	AAS	4	Sr	FEP	4	FEP	4	Sr
cn02	No.091	AAS	2	AAS	2	Sr	AAS	2	AAS	2	Sr
	No.092	AAS	2	AAS	2	Sr	AAS	2	AAS	2	Sr
cn03	No.091	AAS	11	AAS	11	Sr	AAS	11	AAS	11	Sr
	No.092	AAS	11	AAS	11	Sr	AAS	11	AAS	11	Sr
cn04	No.091	AAS	5	AAS	5	Sr	FEP	5	FEP	5	+
	No.092	AAS	5	AAS	5	Sr	FEP	5	FEP	5	+
id01	No.091	AAS	4	AAS	4	+	AAS	4	AAS	4	+
	No.092	AAS	4	AAS	4	+	AAS	4	AAS	4	+
id02	No.091	AAS	21	AAS	21	+	AAS	21	AAS	21	+
	No.092	AAS	21	AAS	21	+	AAS	21	AAS	21	+
jp01	No.091	AAS	6	AAS	6	Sr	FEP	6	FEP	6	na
	No.092	AAS	6	AAS	6	Sr	FEP	6	FEP	6	na
my01	No.091	AAS	2.5	AAS	2.5	na/Sr	FEP	2.5	FEP	2.5	Sr
	No.092	AAS	2.5	AAS	2.5	na/Sr	FEP	2.5	FEP	2.5	Sr
mn01	No.091	-	-	-	-	-	-	-	-	-	-
	No.092	-	-	-	-	-	-	-	-	-	-
ph01	No.091	AAS	0	AAS	0	Sr	AAS	0	AAS	0	na
	No.092	AAS	0	AAS	0	Sr	AAS	0	AAS	0	na
kr01	No.091	AAS	1	AAS	1	Sr	AAS	1	AAS	1	na
	No.092	AAS	1	AAS	1	Sr	AAS	1	AAS	1	na
ru01	No.091	AAS	+	AAS	+	Sr/na	FEP	+	FEP	+	na
	No.092	AAS	+	AAS	+	Sr/na	FEP	+	FEP	+	na
th01	No.091	AAS	2	AAS	2	Sr	FEP	2	FEP	2	La
	No.092	AAS	2	AAS	2	Sr	FEP	2	FEP	2	La
vn01	No.091	AAS	3	AAS	3	na	AAS	3	AAS	3	na
	No.092	AAS	3	AAS	3	na	AAS	3	AAS	3	na
vn02	No.091	-	-	-	-	-	-	-	-	-	-
	No.092	-	-	-	-	-	-	-	-	-	-
vn03	No.091	AAS	4	AAS	4	na	AAS	4	AAS	4	na
	No.092	AAS	4	AAS	4	na	AAS	4	AAS	4	na

Lab.	Sample	Procedures for extraction of Ex-base cations	Ex-Acidity, Al and H		
			method	Size of burette (ml)	
				Capacity	Minimum graduate
cn01	No.081 No.082	Percolation tube	Titration	25	0.1
cn02	No.081 No.082	Automatic extractor	Titration	5	0.00125
cn03	No.081 No.082	Percolation tube	Titration	50	0.1
cn04	No.081 No.082	Automatic extractor	Titration	3	0.02
id01	No.081 No.082	Centrifuge	Titration	50	0.05
id02	No.081 No.082	Percolation tube	Titration	50	0.02
jp01	No.081 No.082	Percolation tube	Titration	25	0.1
my01	No.081 No.082	Percolation tube	Titration	25	0.05
mn01	No.081 No.082	-	Titration	25	0.1
ph01	No.081 No.082	Buchner funnel	Titration	50	0.01
kr01	No.081 No.082	Percolation tube	Titration	10	0.02
ru01	No.081 No.082	Percolation tube	Titration	50	0.05
th01	No.081 No.082	Percolation tube	Titration	25	0.05
vn01	No.081 No.082	Buchner funnel	Titration	10	0.02
vn02	No.081 No.082	-	Titration	10	0.05
vn03	No.081 No.082	Percolation tube	Titration	10	0.02

Note: AAS, Atomic absorption spectrometry; FEP, Flame (emission) photometry; na, Not added; -, Not measured; +, No information. *1. Years in use of instrument.

Table 4.7 Date of analysis

Lab.	Repeat	pH			Ex-Ca and Mg			Ex-K and Na			Ex-acidity, Al and H		
		Date[*1]	Analysis[*2]	Interval[*3]	Date[*1]	Analysis[*2]	Interval[*3]	Date[*1]	Analysis[*2]	Interval[*3]	Date[*1]	Analysis[*2]	Interval[*3]
			Days			Days			Days			Days	
cn01	1st	11-Dec	2	5	5-Feb	5	4	5-Feb	5	4	19-Jan	3	2
	2nd	16-Dec	2		9-Feb	5		9-Feb	5		21-Jan	3	
cn02	1st	8-Dec	2	29	8-Jan	3	7	8-Jan	3	7	8-Jan	3	7
	2nd	6-Jan	2		15-Jan	3		15-Jan	3		15-Jan	3	
cn03	1st	7-Feb	1	1	11-Dec	2	7	11-Dec	2	7	20-Jan	3	14
	2nd	8-Feb	1		18-Dec	2		18-Dec	2		3-Feb	3	
cn04	1st	5-Dec	1	1	9-Dec	3	2	9-Dec	3	2	17-Dec	2	0
	2nd	6-Dec	1		11-Dec	4		11-Dec	4		17-Dec	2	
id01	1st	31-Dec	2	6	11-Jan	8	0	11-Jan	8	0	4-Jan	1	2
	2nd	6-Jan	2		11-Jan	5		11-Jan	5		6-Jan	1	
id02	1st	3-Jan	19	0	3-Jan	19	0	3-Jan	19	0	3-Jan	19	0
	2nd	3-Jan	19		3-Jan	19		3-Jan	19		3-Jan	19	
jp01	1st	25-Nov	1	71	17-Dec	7	67	17-Dec	7	67	17-Feb	14	6
	2nd	4-Feb	1		22-Feb	11		22-Feb	11		23-Feb	20	
my01	1st	17-Jun	1	1	21-Jun	1	1	21-Jun	1	1	17-Jun	1	1
	2nd	18-Jun	1		22-Jun	1		22-Jun	1		18-Jun	1	
mn01	1st	3-Feb	1	0		-	-		-	-	4-Feb	1	0
	2nd	3-Feb	1			-			-		4-Feb	1	
ph01	1st	15-Apr	1	7	23-Apr	1	7	23-Apr	1	7	19-Apr	1	7
	2nd	22-Apr	1		30-Apr	1		30-Apr	1		26-Apr	1	
kr01	1st	2-Apr	1	7	16-Apr	3	7	12-Apr	11	4	7-Apr	3	10
	2nd	9-Apr	1		23-Apr	3		16-Apr	3		17-Apr	6	
ru01	1st	25-Feb	1	1	18-Feb	3	4	18-Feb	3	4	23-Feb	1	2
	2nd	26-Feb	1		22-Feb	4		22-Feb	4		25-Feb	1	
th01	1st	27-Jan	1	5	29-Jan	3	7	29-Jan	3	7	26-Jan	1	7
	2nd	1-Feb	1		5-Feb	3		5-Feb	3		2-Feb	1	
vn01	1st	8-Jan	3	14	8-Jan	3	14	8-Jan	3	14	8-Jan	3	14
	2nd	22-Jan	3		22-Jan	3		22-Jan	3		22-Jan	3	
vn02	1st	23-Dec	1	0		-	-		-	-	24-Dec	1	1
	2nd	23-Dec	1			-			-		25-Dec	1	
vn03	1st	29-Jan	3	0	24-Feb	1	0	23-Feb	1	0	29-Jan	3	0
	2nd	29-Jan	3		24-Feb	1		23-Feb	1		29-Jan	3	

Note: *1. Finish date of 1st and 2nd analyses. *2. Days used for analysis. *3. Interval between the repeat analyses. +, not reported.

4.4 Comparison with past surveys

Inter-laboratory variations in pH (H_2O) and pH (KCl) were very small during 2002 to 2009, as shown by CVs less than 10%. The ranges from upper to lower quartile, which are indicated by box, are kept within 0.2 pH units in almost all samples (Figure 4.4). We speculate from the figure that participating countries can detect the difference of around 0.5 pH unit. Meanwhile, there were a few extreme outliers in each sample. This outlier might be critical for the temporal variation within the same plot.

Inter-laboratory variation in Ex-Ca and Ex-Acidity were shown on the scales of high concentration and low concentration (Figure 4.4). Large variation in Ex-Ca has been observed for 8 years, as shown by CVs more than 50%. On a large scale (0 to 15 $cmol_c\ kg^{-1}$) we can detect the difference between the sample more than 2 or 3 $cmol_c\ kg^{-1}$ (high concentration sample) and the others (low concentration sample). However, at the area of low concentration, inter-laboratory variation might be well over the difference between the samples. Because exchangeable base cations do not drastically change in time at the same site, accuracy of analysis should be improved in the participating country covered with poor-nutrient soils (e.g. tropical soil or undeveloped soil like a desert). In addition, the minimum values of Ex-Ca were very low in almost all samples, even if the median value were very high. We propose that the laboratory providing the minimum value has a fatal fault for measuring the exchangeable base cations (e.g. background of extraction liquid, maintenance of AAS and so on). Inter-laboratory variations in Ex-Acidity were relatively small in comparison with Ex-Ca, except the samples of very low concentration (e.g. No.052 and No.092). From each range as shown by boxes, we assume that participating country can enough detect the temporal variation in soil acidity of not only strong acidic soil but also moderately or weakly acidic soil.

4.5 Recommendations for Improvement

Inter-laboratory variation is enough small to detect the change of general soil acidity (pH and Ex-Acidity) in participating countries. Meanwhile, a few outliers may affect the detection of temporal variation in the same site. Therefore, reducing the outliers of pH and Ex-Acidity should be considered firstly. The condition of glass electrode, standard solution, KCl solution should be checked.

In this time, we found the clear systematic error of inter-laboratory variations in exchangeable cations (Figure 4.2.3 to 4.2.5), that is, the variation is not derived from specific error of laboratories or analyst. The result suggests that a careful preparing the extraction liquid (ammonium acetate) or appropriate AAS analysis in each laboratory improve the large variation in exchangeable cations.

Not only analytical procedures but also reporting procedures should be checked carefully in the respective laboratories.

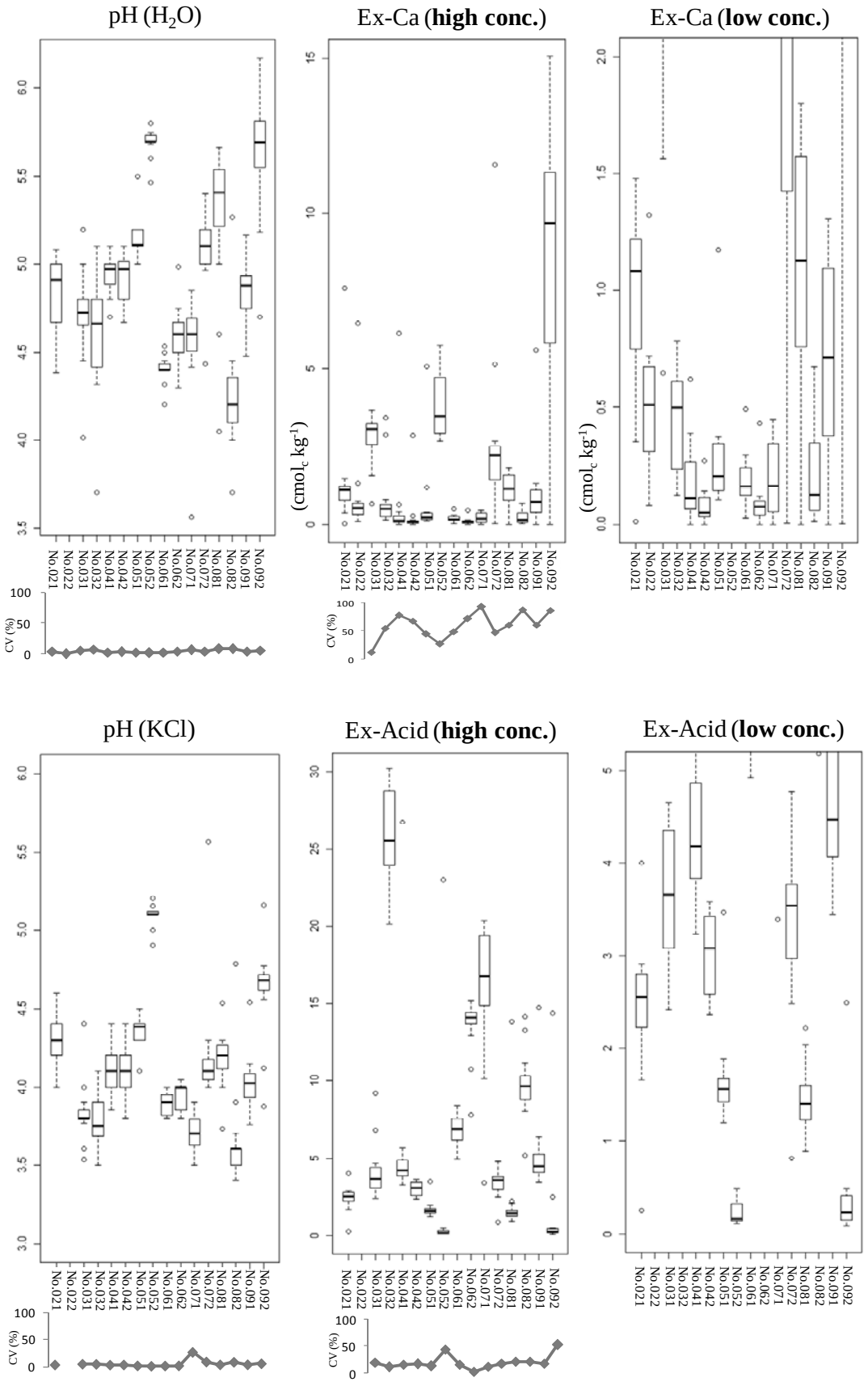


Figure 4.4 Box-and-whisker plots of inter-laboratory variations in pH, Ex-Ca and Ex-Acidity of 16 soil samples from 2003 to 2009, and the variation in CVs (entire dataset).

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2) EANET, 2000. The Second Interim Scientific Advisory Group Meeting of Acid Deposition Monitoring Network in East Asia (2000): Technical Manuals for Soil and Vegetation Monitoring in East Asia.

3) Japanese Standard Association, 1991. General rules for permissible tolerance of chemical analyses and physical tests (JIS Z-8402-$_{1991}$), Japanese Industrial Standard.

4) Acid Deposition and Oxidant Research Center, 2001a. Report of the inter-laboratory comparison project 1999 on soil.

5) Acid Deposition and Oxidant Research Center, 2001b. Report of the inter-laboratory comparison project 2000 on soil.

6) Acid Deposition and Oxidant Research Center, 2002. Report of the inter-laboratory comparison project 2001 on soil.

Appendix 4.1 Participating laboratories

1. CHINA

1) Chongqing Institute of Environmental Science **cn01**
2) Xi'an Environmental Monitoring Station **cn02**
3) Xiamen Environmental Monitoring Central Station **cn03**
4) Zhuhai Environmental Protected Monitoring Station **cn04**

2. INDONESIA

5) Air Quality Laboratory, Environmental Management Center **id01**
6) Soil Research Institute **id02**

3. JAPAN

7) Shimane Agricultural Technology Center **jp01**

4. Malaysia

8) Universiti Putra Malaysia **my01**

5. MONGOLIA

9) Central Laboratory of Environmental Monitoring **mn01**

6. PHILIPPINES

10) University of the Philippines, Los Baños **ph01**

7. Republic of KOREA

11) Soil and Groundwater Research Team, NIER **ko01**

8. RUSSIA

12) Limnological Institute Russian Academy of Science/Siberian Branch **ru01**

9. THAILAND

13) King Mongkut's University of Technology Thonburi **th01**

10. VIET NAM

14) Center for Environmental Research, Institute of Meteorology and Hydrology, MoNRE **vn01**
15) Environment Analysis Laboratory II area, Middle of Central regional Hydro-Meteorological Observatory, National Hydro-Meteorological Center, MoNRE **vn02**
16) Environmental Research Division, National Hydro-Meteorological Center, MoNRE **vn03**

Appendix Table 4.2.1 Entire data of pH in Sample No.091

Lab.	pH(H2O)			pH(KCl)									
	Lab. Ave.	Average	repeat	Lab. Ave.	Average	repeat							
cn01	4.9	4.9 (0.0)	4.9	4.1	4.1 (0.0)	4.1	mn01	4.8	4.8 (0.1)	4.8	4.1	4.0 (0.1)	3.9
			4.9			4.1				4.7			4.1
			5.0			4.1				4.8			4.1
		4.8 (0.0)	4.8		4.0 (0.0)	4.0			4.8 (0.0)	4.8		4.1 (0.0)	4.1
			4.8			4.0				4.8			4.1
			4.8			4.0				4.8			4.1
cn02	5.0	5.0 (0.0)	5.0	4.1	4.1 (0.0)	4.1	ph01	4.5	4.5 (0.0)	4.5	3.9	3.9 (0.0)	3.9
			5.0			4.1				4.5			3.9
			5.0			4.1				4.5			3.9
		5.0 (0.0)	5.0		4.1 (0.0)	4.1			4.5 (0.0)	4.5		3.9 (0.0)	3.9
			5.0			4.1				4.5			3.9
			5.0			4.1				4.5			3.9
cn03	5.0	4.9 (0.0)	4.9	4.2	4.2 (0.0)	4.2	kr01	4.9	4.9 (0.0)	4.9	4.0	3.9 (0.0)	3.8
			4.9			4.2				4.9			3.9
			5.0			4.2				4.9			3.9
		5.0 (0.0)	5.0		4.1 (0.0)	4.2			4.9 (0.0)	4.9		4.1 (0.0)	4.1
			5.0			4.2				4.9			4.1
			5.0			4.1				4.9			4.1
cn04	4.7	4.7 (0.0)	4.7	3.9	3.9 (0.0)	3.9	ru01	4.8	4.8 (0.0)	4.8	4.1	4.1 (0.0)	4.1
			4.7			3.9				4.8			4.1
			4.7			3.9				4.8			4.1
		4.7 (0.0)	4.7		3.9 (0.0)	3.9			4.8 (0.0)	4.8		4.1 (0.0)	4.1
			4.8			3.9				4.8			4.1
			4.7			3.9				4.8			4.1
id01	4.9	4.9 (0.0)	4.9	4.0	4.0 (0.0)	4.0	th01	4.9	4.9 (0.0)	4.9	4.0	4.0 (0.0)	4.0
			4.9			4.0				4.9			4.0
			4.9			4.0				4.9			4.0
		4.9 (0.0)	4.9		4.0 (0.0)	4.0			4.9 (0.0)	4.9		4.0 (0.0)	4.0
			4.9			4.1				4.9			4.0
			4.9			4.0				4.9			4.0
id02	4.7	4.7 (0.1)	4.7	3.8	3.8 (0.0)	3.8	vn01	4.9	4.8 (0.0)	4.8	4.0	3.9 (0.0)	3.9
			4.7			3.8				4.9			3.9
			4.6			3.8				4.8			3.9
		4.6 (0.0)	4.6		3.8 (0.0)	3.8			4.9 (0.0)	4.9		4.0 (0.0)	4.0
			4.6			3.7				4.9			4.0
			4.6			3.8				4.9			4.0
jp01	5.1	5.1 (0.0)	5.1	#DIV/0!	4.1 (0.0)	4.1	vn02	5.2	5.2 (0.0)	5.2	4.5	4.5 (0.0)	4.53
			5.1			4.1				5.2			4.54
			5.1			4.1				5.2			4.54
		5.1 (0.0)	5.1						5.2 (0.0)	5.2		4.5 (0.0)	4.53
			5.1							5.2			4.54
			5.1							5.2			4.55
my01	4.5	4.5 (0.1)	4.5	3.9	3.9 (0.0)	3.9	vn03	4.8	4.8 (0.0)	4.8	4.0	4.0 (0.0)	4
			4.4			3.9				4.8			4
			4.5			3.9				4.8			4.01
		4.5 (0.0)	4.5		3.9 (0.0)	3.9			4.8 (0.0)	4.8		4.0 (0.0)	4
			4.5			3.9				4.8			4
			4.5			3.9				4.8			4.01

Note: Value in parenthesis showed standard deviation of triplicate

Appendix Table 4.2.2 Entire data of exchangeable base cations in Sample No.091

Lab.	Ex-Ca			Ex-Mg			Ex-K			Ex-Na		
	cmol(+) kg-1											
	Lab. Ave.	Average	repeat	Lab. Ave.	Average	repeat	Lab. Ave.	Average	repeat	Lab. Ave.	Average	repeat
cn01	0.74	0.72 (0.04)	0.68	0.59	0.60 (0.02)	0.62	0.51	0.50 (0.05)	0.44	0.07	0.07 (0.00)	0.07
			0.76			0.60			0.50			0.07
			0.72			0.58			0.55			0.07
		0.76 (0.03)	0.78		0.58 (0.02)	0.60		0.51 (0.06)	0.56		0.06 (0.01)	0.06
			0.73			0.56			0.54			0.07
			0.77			0.56			0.45			0.06
cn02	0.48	0.48 (0.01)	0.49	0.57	0.56 (0.01)	0.55	0.39	0.39 (0.01)	0.39	0.06	0.06 (0.01)	0.06
			0.48			0.58			0.39			0.05
			0.47			0.55			0.38			0.06
		0.47 (0.03)	0.46		0.57 (0.02)	0.55		0.39 (0.01)	0.39		0.06 (0.01)	0.06
			0.51			0.57			0.39			0.06
			0.45			0.59			0.38			0.07
cn03	0.38	0.38 (0.02)	0.38	0.38	0.38 (0.05)	0.40	0.29	0.28 (0.01)	0.27	0.12	0.11 (0.02)	0.10
			0.36			0.42			0.29			0.10
			0.40			0.32			0.28			0.13
		0.38 (0.02)	0.38		0.38 (0.05)	0.40		0.30 (0.02)	0.28		0.13 (0.00)	0.13
			0.36			0.42			0.33			0.13
			0.40			0.32			0.30			0.13
cn04	0.30	0.31 (0.04)	0.32	0.41	0.42 (0.03)	0.40	0.39	0.38 (0.01)	0.37	0.07	0.06 (0.03)	0.10
			0.34			0.43			0.39			0.05
			0.26			0.45			0.39			0.05
		0.29 (0.04)	0.30		0.40 (0.03)	0.36		0.40 (0.03)	0.41		0.07 (0.02)	0.07
			0.24			0.42			0.37			0.09
			0.32			0.42			0.43			0.06
id01	27.12	24.62 (0.41)	25.08	1.02	0.98 (0.01)	0.99	0.74	0.71 (0.00)	0.71	0.09	0.09 (0.00)	0.09
			24.50			0.96			0.71			0.09
			24.28			0.99			0.71			0.09
		29.61 (0.05)	29.62		1.05 (0.07)	1.08		0.77 (0.00)	0.77		0.09 (0.00)	0.09
			29.66			1.09			0.77			0.09
			29.56			0.97			0.77			0.09
id02	0.35	0.35 (0.01)	0.34	1.03	1.03 (0.01)	1.04	0.14	0.14 (0.00)	0.14	0.09	0.09 (0.01)	0.10
			0.35			1.02			0.14			0.09
			0.34			1.04			0.14			0.08
		0.35 (0.01)	0.35		1.03 (0.01)	1.02		0.14 (0.00)	0.14		0.09 (0.01)	0.08
			0.35			1.04			0.14			0.10
			0.34			1.02			0.14			0.10
jp01	0.94	1.10 (0.07)	1.17	0.59	0.58 (0.02)	0.57	0.45	0.45 (0.01)	0.44	0.04	0.03 (0.00)	0.03
			1.03			0.60			0.46			0.03
			1.09			0.58			0.43			0.03
		0.77 (0.05)	0.74		0.59 (0.03)	0.59		0.45 (0.00)	0.45		0.05 (0.00)	0.05
			0.83			0.62			0.45			0.05
			0.74			0.58			0.45			0.06
my01	0.00	0.00 (0.00)	0.00	0.00	0.00 (0.00)	0.00	0.00	0.00 (0.00)	0.00	0.00	0.00 (0.00)	0.00
			0.00			0.00			0.00			0.00
			0.00			0.00			0.00			0.00
		0.00 (0.00)	0.00		0.00 (0.00)	0.00		0.00 (0.00)	0.00		0.00 (0.00)	0.00
			0.00			0.00			0.00			0.00
			0.00			0.00			0.00			0.00
mn01												
ph01	5.59	5.61 (0.12)	5.70	2.13	2.13 (0.02)	2.12	0.93	0.95 (0.07)	0.88	1.57	1.55 (0.11)	1.50
			5.66			2.15			1.00			1.67
			5.48			2.11			0.98			1.47
		5.57 (0.07)	5.52		2.13 (0.02)	2.15		0.91 (0.07)	0.97		1.58 (0.08)	1.67
			5.55			2.11			0.93			1.53
			5.65			2.13			0.83			1.56
kr01	0.61	0.60 (0.02)	0.61	0.58	0.60 (0.02)	0.59	0.39	0.39 (0.01)	0.38	0.04	0.03 (0.00)	0.03
			0.62			0.62			0.40			0.04
			0.58			0.58			0.39			0.03
		0.62 (0.04)	0.62		0.56 (0.01)	0.55		0.39 (0.01)	0.37		0.04 (0.00)	0.03
			0.66			0.56			0.40			0.04
			0.59			0.57			0.39			0.04
ru01	1.31	1.24 (0.02)	1.26	0.75	0.70 (0.01)	0.70	0.41	0.41 (0.00)	0.41	0.06	0.06 (0.00)	0.06
			1.25			0.70			0.41			0.06
			1.22			0.69			0.41			0.06
		1.37 (0.16)	1.26		0.79 (0.08)	0.74		0.41 (0.00)	0.41		0.05 (0.00)	0.05
			1.30			0.75			0.41			0.05
			1.55			0.88			0.42			0.05
th01	1.10	1.09 (0.01)	1.11	0.63	0.62 (0.01)	0.63	0.37	0.37 (0.02)	0.36	0.12	0.12 (0.00)	0.12
			1.08			0.61			0.36			0.12
			1.09			0.64			0.40			0.12
		1.10 (0.03)	1.11		0.63 (0.01)	0.62		0.36 (0.01)	0.36		0.12 (0.01)	0.13
			1.06			0.64			0.35			0.12
			1.12			0.63			0.37			0.11
vn01	0.69	0.68 (0.01)	0.68	0.56	0.55 (0.02)	0.57	0.40	0.40 (0.00)	0.40	0.06	0.06 (0.00)	0.05
			0.68			0.54			0.40			0.06
			0.69			0.54			0.40			0.06
		0.70 (0.01)	0.69		0.57 (0.01)	0.56		0.39 (0.00)	0.39		0.06 (0.00)	0.06
			0.70			0.57			0.39			0.06
			0.70			0.58			0.40			0.06
vn02												
vn03	0.97	0.97 (0.00)	0.97	1.06	1.06 (0.00)	1.06	0.45	0.44 (0.00)	0.44	0.32	0.32 (0.00)	0.32
			0.96			1.06			0.44			0.32
			0.97			1.06			0.44			0.32
		0.97 (0.01)	0.97		1.05 (0.02)	1.03		0.45 (0.00)	0.45		0.32 (0.00)	0.32
			0.98			1.06			0.45			0.32
			0.96			1.06			0.45			0.32

Note: Value in parenthesis showed standard deviation of triplicate analyses.

Appendix Table 4.2.3 Entire data of exchangeable acidity and acid cations in Sample No.091

Lab.	Ex-acidity			Ex-Al			Ex-H		
	cmol(+) kg-1								
	Lab. Ave.	Average	repeat	Lab. Ave.	Average	repeat	Lab. Ave.	Average	repeat
cn01	3.45	3.27 (0.02)	3.29	2.97	2.88 (0.12)	3.02	0.47	0.38 (0.10)	0.27
			3.26			2.80			0.46
			3.25			2.83			0.43
		3.62 (0.07)	3.65		3.06 (0.03)	3.03		0.55 (0.09)	0.62
			3.66			3.08			0.58
			3.54			3.09			0.45
cn02	5.63	5.67 (0.09)	5.59	4.66	4.70 (0.04)	4.69	0.97	0.96 (0.06)	0.90
			5.76			4.74			1.02
			5.65			4.67			0.98
		5.59 (0.02)	5.57		4.62 (0.08)	4.54		0.97 (0.06)	1.03
			5.61			4.69			0.92
			5.59			4.64			0.95
cn03	6.39	6.38 (0.00)	6.38	5.47	5.46 (0.02)	5.46	0.93	0.93 (0.02)	0.93
			6.38			5.44			0.94
			6.38			5.47			0.91
		6.40 (0.00)	6.40		5.47 (0.01)	5.46		0.93 (0.01)	0.94
			6.40			5.47			0.93
			6.40			5.47			0.93
cn04	4.21	4.20 (0.16)	4.02	3.50	3.49 (0.10)	3.41	0.70	0.70 (0.12)	0.60
			4.27			3.60			0.66
			4.32			3.47			0.83
		4.22 (0.06)	4.19		3.50 (0.05)	3.50		0.70 (0.02)	0.68
			4.17			3.45			0.71
			4.29			3.55			0.72
id01	4.01	4.01 (0.01)	4.00	3.58	3.62 (0.00)	3.62	0.43	0.39 (0.01)	0.38
			4.00			3.62			0.38
			4.02			3.62			0.40
		4.00 (0.00)	4.00		3.54 (0.00)	3.54		0.46 (0.00)	0.46
			4.00			3.54			0.46
			4.00			3.54			0.46
id02	14.73	14.73 (0.02)	14.75	13.06	13.06 (0.02)	13.04	1.67	1.67 (0.04)	1.71
			14.71			13.06			1.65
			14.73			13.08			1.65
		14.72 (0.03)	14.75		13.06 (0.02)	13.04		1.66 (0.06)	1.71
			14.73			13.06			1.67
			14.69			13.08			1.60
jp01	4.91	4.88 (0.15)	4.83	3.17	3.16 (0.09)	3.20	1.68	1.68 (0.03)	1.71
			4.77			3.22			1.68
			5.05			3.05			1.65
		4.93 (0.11)	4.81		3.17 (0.07)	3.09		1.67 (0.04)	1.63
			5.02			3.20			1.71
			4.97			3.22			1.68
my01	6.08	6.03 (0.03)	6.01	6.51	6.47 (0.04)	6.47	0.71	0.76 (0.05)	0.78
			6.02			6.42			0.70
			6.06			6.51			0.78
		6.12 (0.06)	6.19		6.55 (0.04)	6.50		0.65 (0.16)	0.47
			6.10			6.58			0.74
			6.06			6.56			0.74
mn01	4.75	4.75 (0.00)	4.75	4.28	4.28 (0.00)	4.28	0.43	0.43 (0.00)	0.43
			4.75			4.28			0.43
			4.75			4.28			0.43
		4.75 (0.00)	4.75		4.28 (0.00)	4.28		0.43 (0.00)	0.43
			4.75			4.28			0.43
			4.75			4.28			0.43
ph01	4.47	4.57 (0.09)	4.47	3.56	3.61 (0.05)	3.56	0.91	0.96 (0.05)	0.92
			4.66			3.65			1.01
			4.58			3.63			0.95
		4.37 (0.00)	4.37		3.50 (0.02)	3.49		0.86 (0.02)	0.87
			4.37			3.52			0.85
			4.37			3.49			0.87
kr01	4.10	4.08 (0.16)	3.90	3.67	3.65 (0.06)	3.69	0.47	0.44 (0.06)	0.48
			4.18			3.58			0.48
			4.18			3.69			0.37
		4.12 (0.02)	4.10		3.68 (0.03)	3.68		0.49 (0.07)	0.41
			4.13			3.70			0.50
			4.13			3.64			0.56
ru01	4.47	4.45 (0.08)	4.55	4.09	4.06 (0.05)	4.12	0.32	0.32 (0.00)	0.32
			4.41			4.03			0.32
			4.41			4.03			0.32
		4.48 (0.08)	4.57		4.11 (0.06)	4.15		0.32 (0.00)	0.32
			4.41			4.14			0.32
			4.47			4.04			0.32
th01	4.48	4.48 (0.03)	4.45	4.23	4.24 (0.04)	4.20	0.25	0.24 (0.03)	0.26
			4.50			4.24			0.26
			4.50			4.28			0.22
		4.47 (0.03)	4.45		4.21 (0.02)	4.24		0.26 (0.05)	0.21
			4.45			4.20			0.26
			4.50			4.20			0.30
vn01	4.04	4.04 (0.00)	4.04	3.04	3.02 (0.02)	3.01	1.01	1.03 (0.02)	1.04
			4.04			3.01			1.03
			4.04			3.03			1.01
		4.04 (0.00)	4.04		3.05 (0.02)	3.06		0.99 (0.01)	0.98
			4.04			3.03			1.01
			4.04			3.06			0.98
vn02	3.89	3.90 (0.03)	3.92	3.49	3.47 (0.06)	3.50	0.38	0.39 (0.06)	0.36
			3.92			3.40			0.46
			3.86			3.50			0.36
		3.88 (0.03)	3.86		3.50 (0.00)	3.50		0.36 (0.00)	0.36
			3.92			3.50			0.36
			3.86			3.50			0.36
vn03	4.52	4.51 (0.03)	4.55	7.97	7.97 (0.05)	8.00	0.40	0.41 (0.02)	0.42
			4.51			7.92			0.42
			4.49			8.00			0.38
		4.53 (0.02)	4.55		7.97 (0.05)	7.92		0.39 (0.02)	0.38
			4.51			8.00			0.42
			4.53			8.00			0.38

Note: Value in parenthesis showed standard deviation of triplicate analyses.

Appendix Table 4.3.1 Entire data of pH in Sample No.092

Lab.	pH(H2O)			pH(KCl)									
	Lab. Ave.	Average	repeat	Lab. Ave.	Average	repeat							
cn01	5.8	5.8 (0.0)	5.8	4.8	4.8 (0.0)	4.8	mn01	5.7	5.7 (0.0)	5.7	4.7	4.7 (0.0)	4.7
			5.9			4.7				5.7			4.7
			5.8			4.8				5.8			4.7
		5.8 (0.0)	5.9		4.7 (0.0)	4.7			5.7 (0.0)	5.7		4.7 (0.0)	4.7
			5.8			4.7				5.7			4.7
			5.8			4.7				5.7			4.7
cn02	5.8	5.8 (0.0)	5.8	4.7	4.7 (0.0)	4.7	ph01	5.5	5.4 (0.0)	5.4	4.6	4.6 (0.0)	4.6
			5.8			4.7				5.4			4.6
			5.8			4.7				5.4			4.6
		5.8 (0.0)	5.8		4.7 (0.0)	4.7			5.5 (0.0)	5.5		4.6 (0.0)	4.6
			5.8			4.7				5.5			4.6
			5.8			4.7				5.5			4.6
cn03	5.8	5.8 (0.0)	5.8	4.7	4.7 (0.0)	4.7	kr01	5.7	5.7 (0.0)	5.7	4.7	4.6 (0.0)	4.6
			5.8			4.7				5.7			4.6
			5.8			4.7				5.6			4.6
		5.8 (0.0)	5.8		4.7 (0.0)	4.7			5.7 (0.0)	5.7		4.7 (0.0)	4.7
			5.8			4.7				5.7			4.7
			5.8			4.7				5.7			4.7
cn04	5.6	5.6 (0.0)	5.6	4.6	4.6 (0.0)	4.6	ru01	5.7	5.7 (0.0)	5.7	4.8	4.8 (0.0)	4.8
			5.6			4.6				5.7			4.8
			5.6			4.6				5.7			4.8
		5.6 (0.0)	5.6		4.6 (0.0)	4.6			5.7 (0.0)	5.7		4.8 (0.0)	4.8
			5.6			4.6				5.7			4.8
			5.6			4.6				5.7			4.8
id01	5.8	5.8 (0.0)	5.8	4.7	4.7 (0.0)	4.7	th01	5.8	5.8 (0.0)	5.8	4.7	4.7 (0.0)	4.7
			5.8			4.7				5.8			4.7
			5.8			4.7				5.8			4.7
		5.8 (0.0)	5.8		4.7 (0.0)	4.7			5.8 (0.0)	5.8		4.7 (0.0)	4.7
			5.8			4.7				5.8			4.7
			5.8			4.7				5.8			4.7
id02	5.2	5.2 (0.0)	5.2	4.1	4.1 (0.0)	4.1	vn01	5.8	5.8 (0.1)	5.8	4.8	4.7 (0.0)	4.7
			5.2			4.1				5.7			4.7
			5.2			4.1				5.8			4.7
		5.2 (0.0)	5.2		4.1 (0.0)	4.1			5.8 (0.0)	5.7		4.8 (0.0)	4.7
			5.2			4.1				5.7			4.8
			5.2			4.1				5.8			4.8
jp01	6.2	6.2 (0.0)	6.2			4.6	vn02	5.7	5.7 (0.0)	5.7	5.2	5.2 (0.0)	5.18
			6.1			4.7				5.7			5.17
			6.2			4.7				5.7			5.16
		6.2 (0.0)	6.2						5.7 (0.0)	5.7		5.1 (0.0)	5.14
			6.2							5.7			5.15
			6.2							5.7			5.14
my01	4.7	4.7 (0.0)	4.7	3.9	3.9 (0.0)	3.9	vn03	5.5	5.5 (0.0)	5.5	4.7	4.7 (0.0)	4.65
			4.8			3.9				5.5			4.65
			4.7			3.8				5.5			4.66
		4.7 (0.0)	4.7		3.9 (0.0)	3.9			5.5 (0.0)	5.5		4.7 (0.0)	4.65
			4.7			3.9				5.5			4.65
			4.7			3.9				5.5			4.66

Note: Value in parenthesis showed standard deviation of triplicate analyses.

Appendix Table 4.3.2 Entire data of exchangeable base cations in Sample No.092

Lab.	Ex-Ca			Ex-Mg			Ex-K			Ex-Na		
	cmol(+) kg-1											
	Lab. Ave.	Average	repeat	Lab. Ave.	Average	repeat	Lab. Ave.	Average	repeat	Lab. Ave.	Average	repeat
cn01	7.01	7.15 (0.66)	6.44	1.07	1.07 (0.01)	1.06	0.47	0.46 (0.01)	0.45	0.14	0.14 (0.00)	0.14
			7.30			1.08			0.48			0.14
			7.72			1.06			0.45			0.13
		6.87 (0.43)	6.44		1.06 (0.03)	1.06		0.48 (0.03)	0.48		0.13 (0.00)	0.13
			6.87			1.03			0.44			0.12
			7.30			1.08			0.51			0.13
cn02	6.01	6.00 (0.31)	6.04	1.22	1.22 (0.04)	1.20	0.38	0.38 (0.01)	0.39	0.11	0.11 (0.01)	0.11
			6.29			1.26			0.39			0.11
			5.67			1.19			0.37			0.12
		6.01 (0.08)	5.97		1.21 (0.01)	1.20		0.38 (0.00)	0.39		0.11 (0.00)	0.11
			5.96			1.21			0.38			0.11
			6.10			1.22			0.38			0.10
cn03	5.03	5.03 (0.02)	5.04	0.62	0.62 (0.06)	0.54	0.13	0.13 (0.01)	0.14	0.16	0.16 (0.01)	0.16
			5.01			0.66			0.12			0.16
			5.04			0.65			0.13			0.17
		5.03 (0.02)	5.04		0.62 (0.06)	0.54		0.13 (0.01)	0.13		0.16 (0.00)	0.16
			5.01			0.66			0.14			0.16
			5.04			0.65			0.12			0.16
cn04	5.82	5.82 (0.00)	5.82	0.88	0.87 (0.01)	0.86	0.40	0.39 (0.01)	0.40	0.10	0.09 (0.02)	0.08
			5.82			0.88			0.39			0.11
			5.82			0.86			0.39			0.08
		5.81 (0.02)	5.84		0.88 (0.02)	0.86		0.40 (0.02)	0.41		0.10 (0.01)	0.09
			5.81			0.88			0.37			0.10
			5.79			0.90			0.40			0.09
id01	10.92	10.70 (0.17)	10.56	1.95	1.97 (0.02)	1.95	0.70	0.72 (0.01)	0.72	0.20	0.18 (0.01)	0.19
			10.89			1.99			0.71			0.18
			10.65			1.97			0.73			0.17
		11.13 (0.57)	10.53		1.93 (0.06)	1.99		0.68 (0.04)	0.66		0.21 (0.02)	0.18
			11.65			1.93			0.65			0.23
			11.20			1.88			0.72			0.23
id02	2.18	2.18 (0.01)	2.18	0.65	0.65 (0.01)	0.65	0.08	0.07 (0.00)	0.08	0.04	0.04 (0.01)	0.05
			2.18			0.67			0.07			0.05
			2.17			0.65			0.08			0.04
		2.18 (0.01)	2.19		0.65 (0.01)	0.67		0.08 (0.00)	0.08		0.04 (0.01)	0.04
			2.18			0.65			0.08			0.05
			2.18			0.65			0.08			0.05
jp01	10.24	10.69 (0.31)	11.05	1.19	1.18 (0.04)	1.21	0.45	0.44 (0.01)	0.45	0.12	0.10 (0.01)	0.11
			10.50			1.19			0.45			0.09
			10.53			1.13			0.44			0.10
		9.78 (0.35)	10.18		1.19 (0.02)	1.21		0.45 (0.01)	0.44		0.13 (0.02)	0.12
			9.61			1.19			0.46			0.15
			9.55			1.17			0.45			0.12
my01	0.00	0.00 (0.00)	0.00	0.00	0.00 (0.00)	0.00	0.00	0.00 (0.00)	0.00	0.00	0.00 (0.00)	0.00
			0.00			0.00			0.00			0.00
			0.00			0.00			0.00			0.00
		0.00 (0.00)	0.00		0.00 (0.00)	0.00		0.00 (0.00)	0.00		0.00 (0.00)	0.00
			0.00			0.00			0.00			0.00
			0.00			0.00			0.00			0.00
mn01												
ph01	22.70	22.72 (0.19)	22.55	2.13	2.13 (0.05)	2.15	0.96	0.98 (0.15)	1.13	1.15	1.13 (0.08)	1.08
			22.68			2.07			0.98			1.08
			22.92			2.16			0.83			1.23
		22.68 (0.20)	22.74		2.13 (0.06)	2.07		0.93 (0.10)	0.95		1.16 (0.12)	1.09
			22.45			2.17			0.82			1.09
			22.83			2.16			1.01			1.29
kr01	9.13	9.13 (0.26)	8.96	1.12	1.14 (0.01)	1.14	0.37	0.37 (0.00)	0.37	0.09	0.09 (0.00)	0.09
			9.00			1.13			0.37			0.09
			9.43			1.14			0.37			0.10
		9.12 (0.14)	8.99		1.10 (0.02)	1.08		0.37 (0.01)	0.37		0.08 (0.00)	0.08
			9.27			1.10			0.37			0.08
			9.10			1.13			0.38			0.09
ru01	11.33	11.34 (0.72)	11.60	1.44	1.53 (0.12)	1.62	0.42	0.41 (0.02)	0.41	0.11	0.12 (0.01)	0.12
			11.89			1.58			0.43			0.12
			10.52			1.39			0.40			0.11
		11.31 (0.50)	11.44		1.34 (0.09)	1.37		0.42 (0.02)	0.42		0.10 (0.01)	0.11
			11.73			1.42			0.44			0.11
			10.76			1.24			0.40			0.09
th01	15.06	15.07 (0.03)	15.03	1.05	1.04 (0.03)	1.04	0.27	0.28 (0.01)	0.29	0.22	0.23 (0.04)	0.19
			15.09			1.08			0.26			0.27
			15.08			1.01			0.28			0.24
		15.05 (0.02)	15.06		1.06 (0.03)	1.04		0.26 (0.01)	0.24		0.21 (0.02)	0.20
			15.06			1.09			0.27			0.21
			15.02			1.04			0.26			0.23
vn01	10.68	10.69 (0.04)	10.71	0.86	0.86 (0.00)	0.87	0.43	0.43 (0.01)	0.43	0.13	0.13 (0.01)	0.14
			10.72			0.86			0.42			0.13
			10.64			0.86			0.43			0.14
		10.67 (0.04)	10.68		0.86 (0.01)	0.86		0.43 (0.00)	0.43		0.13 (0.00)	0.13
			10.64			0.86			0.43			0.13
			10.71			0.85			0.43			0.13
vn02												
vn03	11.96	11.95 (0.01)	11.93	2.23	2.24 (0.01)	2.25	0.44	0.44 (0.00)	0.44	0.44	0.44 (0.00)	0.44
			11.95			2.23			0.44			0.44
			11.96			2.23			0.44			0.44
		11.96 (0.01)	11.96		2.21 (0.01)	2.21		0.44 (0.00)	0.44		0.44 (0.00)	0.44
			11.95			2.22			0.44			0.44
			11.97			2.20			0.44			0.44

Note: Value in parenthesis showed standard deviation of triplicate analyses.

Appendix Table 4.3.3 Entire data of exchangeable acidity and acid cations in Sample No.092

Lab.	Ex-acidity			Ex-Al			Ex-H		
	cmol(+) kg-1								
	Lab. Ave.	Average	repeat	Lab. Ave.	Average	repeat	Lab. Ave.	Average	repeat
cn01	0.13	0.13 (0.00)	0.12	0.00	0.00 (0.00)	0.00	0.13	0.13 (0.00)	0.12
			0.13			0.00			0.13
			0.12			0.00			0.12
		0.13 (0.00)	0.13		0.00 (0.00)	0.00		0.13 (0.00)	0.13
			0.14			0.00			0.14
			0.13			0.00			0.13
cn02	0.40	0.40 (0.03)	0.41	0.20	0.18 (0.02)	0.17	0.21	0.22 (0.02)	0.23
			0.36			0.17			0.19
			0.43			0.21			0.22
		0.40 (0.03)	0.43		0.21 (0.03)	0.24		0.19 (0.01)	0.19
			0.41			0.21			0.20
			0.36			0.17			0.19
cn03	0.49	0.47 (0.00)	0.47	0.32	0.30 (0.00)	0.30	0.17	0.17 (0.00)	0.17
			0.47			0.30			0.17
			0.47			0.30			0.17
		0.51 (0.00)	0.51		0.34 (0.00)	0.34		0.17 (0.00)	0.17
			0.51			0.34			0.17
			0.51			0.34			0.17
cn04	0.17	0.17 (0.00)	0.17	0.00	0.00 (0.00)	0.00	0.17	0.17 (0.00)	0.17
			0.17			0.00			0.17
			0.17			0.00			0.17
		0.17 (0.01)	0.17		0.00 (0.00)	0.00		0.17 (0.01)	0.17
			0.17			0.00			0.17
			0.16			0.00			0.16
id01	0.15	0.15 (0.00)	0.15	0.08	0.08 (0.00)	0.08	0.07	0.07 (0.00)	0.07
			0.15			0.08			0.07
			0.15			0.08			0.07
		0.15 (0.00)	0.15		0.08 (0.00)	0.08		0.07 (0.00)	0.07
			0.15			0.08			0.07
			0.15			0.08			0.07
id02	2.49	2.49 (0.02)	2.50	2.13	2.13 (0.02)	2.15	0.36	0.36 (0.03)	0.35
			2.50			2.11			0.40
			2.46			2.13			0.33
		2.49 (0.02)	2.50		2.13 (0.02)	2.11		0.36 (0.04)	0.40
			2.46			2.15			0.31
			2.50			2.13			0.37
jp01	0.14	0.19 (0.05)	0.22	0.07	0.07 (0.12)	0.00	0.07	0.10 (0.22)	0.11
			0.21			0.00			0.32
			0.14			0.21			-0.13
		0.09 (0.03)	0.06		0.07 (0.12)	0.21		0.04 (0.12)	-0.11
			0.12			0.00			0.11
			0.10			0.00			0.11
my01	14.37	14.44 (0.04)	14.46	12.99	12.93 (0.04)	12.95	0.31	0.31 (0.03)	0.34
			14.47			12.88			0.32
			14.39			12.95			0.28
		14.30 (0.02)	14.27		13.04 (0.04)	13.05		0.31 (0.05)	0.36
			14.32			13.08			0.26
			14.30			12.99			0.32
mn01	0.43	0.43 (0.00)	0.43	0.00	0.00 (0.00)	0.00	0.43	0.43 (0.00)	0.43
			0.43			0.00			0.43
			0.43			0.00			0.43
		0.43 (0.00)	0.43		0.00 (0.00)	0.00		0.43 (0.00)	0.43
			0.43			0.00			0.43
			0.43			0.00			0.43
ph01	0.29	0.31 (0.01)	0.31	0.10	0.10 (0.01)	0.11	0.20	0.21 (0.02)	0.20
			0.33			0.09			0.24
			0.31			0.11			0.20
		0.27 (0.02)	0.27		0.09 (0.02)	0.11		0.18 (0.02)	0.16
			0.26			0.07			0.19
			0.29			0.09			0.20
kr01	0.17	0.15 (0.00)	0.15	0.11	0.11 (0.00)	0.11	0.09	0.10 (0.00)	0.10
			0.15			0.11			0.10
			0.15			0.11			0.10
		0.18 (0.00)	0.18		0.10 (0.01)	0.08		0.08 (0.00)	0.08
			0.17			0.11			0.08
			0.18			0.11			0.08
ru01	0.11	0.11 (0.00)	0.11	0.00	0.00 (0.00)	0.00	0.11	0.11 (0.00)	0.11
			0.11			0.00			0.11
			0.11			0.00			0.11
		0.11 (0.00)	0.11		0.00 (0.00)	0.00		0.11 (0.00)	0.11
			0.11			0.00			0.11
			0.11			0.00			0.11
th01	0.09	0.10 (0.00)	0.10	0.05	0.06 (0.03)	0.05	0.05	0.04 (0.03)	0.05
			0.10			0.05			0.05
			0.10			0.09			0.01
		0.08 (0.03)	0.10		0.03 (0.03)	0.05		0.05 (0.00)	0.05
			0.10			0.05			0.05
			0.05			0.00			0.05
vn01	0.33	0.32 (0.01)	0.31	0.00	0.00 (0.00)	0.00	0.33	0.32 (0.01)	0.31
			0.33			0.00			0.33
			0.31			0.00			0.31
		0.33 (0.00)	0.33		0.00 (0.00)	0.00		0.33 (0.00)	0.33
			0.33			0.00			0.33
			0.33			0.00			0.33
vn02	0.27	0.27 (0.00)	0.27	0.00	0.00 (0.00)	0.00	0.32	0.32 (0.00)	0.32
			0.27			0.00			0.32
			0.27			0.00			0.32
		0.27 (0.00)	0.27		0.00 (0.00)	0.00		0.32 (0.00)	0.32
			0.27			0.00			0.32
			0.27			0.00			0.32
vn03	0.19	0.19 (0.00)	0.19	0.37	0.37 (0.05)	0.34	0.08	0.08 (0.00)	0.08
			0.19			0.34			0.08
			0.19			0.42			0.08
		0.18 (0.01)	0.19		0.37 (0.05)	0.34		0.08 (0.00)	0.08
			0.19			0.42			0.08
			0.17			0.34			0.08

Note: Value in parenthesis showed standard deviation of triplicate analyses.

Appendix 4.4 Z-score evaluation

The NC applied Z-score for further statistical evaluation of the analytical values in the inter-laboratory comparison on soil.

- **Definition of Z-score**

Z-score is one of the statistical measures that quantify the distance from the mean of a data set. The formula for the calculation of Z-score (Robust method) was shown below:

$$Z = \frac{X - Q_2}{0.7413 \times (Q_3 - Q_1)}$$

where X: Measurement values of samples
Q_1: The 1st quartile value of entire data
Q_2: The 2nd quartile value of entire data (i.e. Median)
Q_3: The 3rd quartile value of entire data

- **Evaluation of calculated Z-score**

Z-score was given to each data submitted by the participating laboratories, and was evaluated as follows:

$|Z| \leq 2$: Satisfactory
$2 < |Z| < 3$: Questionable
$3 \leq |Z|$: Unsatisfactory

The calculated Z-scores were shown in Appendix Table 4.4.1 and 4.4.2, and were also graphed in Appendix Figure 4.4.1 through 4.4.9.

Appendix Table 4.4.1 Results of Z-score evaluation for sample No. 091

Sample No. 091 (Cambisols)

Lab. ID	pH (H_2O)	pH(KCl)	Ex-Ca	Ex-Mg	Ex-K	Ex-Na	Ex-Acidity	Ex-Al	Ex-H
cn01	0.00	1.08	0.05	0.00	2.07	0.00	-1.38	-0.69	-0.30
cn02	0.90	1.08	-0.48	-0.07	-0.09	-0.26	1.55	0.98	1.02
cn03	0.90	2.16	-0.68	-0.73	-1.89	1.28	2.57	1.78	0.91
cn04	-1.80	-1.08	-0.85	-0.62	-0.09	0.00	-0.36	-0.17	0.30
id01	0.00	0.00	53.77	1.49	6.21	0.51	-0.62	-0.09	-0.41
id02	-1.80	-2.16	-0.74	1.52	-4.59	0.51	13.76	9.28	2.87
jp01	1.80	1.08	0.46	0.00	0.99	-0.77	0.58	-0.49	2.90
my01	-3.60	-1.08	-1.46	-2.04	-7.10	-1.80	2.15	2.81	0.33
mn01	-0.90	1.08	---	---	---	---	0.37	0.60	-0.41
ph01	-3.60	-1.08	9.93	5.33	9.62	38.54	-0.01	-0.11	0.86
kr01	0.00	0.00	-0.21	-0.03	-0.09	-0.77	-0.50	0.00	-0.30
ru01	-0.90	1.08	1.21	0.55	0.27	-0.26	-0.50	0.00	-0.30
th01	0.00	0.00	0.78	0.14	-0.45	1.28	0.01	0.55	-0.89
vn01	0.00	0.00	-0.05	-0.10	0.09	-0.26	-0.58	-0.62	1.12
vn02	2.70	5.40	---	---	---	---	-0.79	-0.18	-0.54
vn03	-0.90	0.00	0.52	1.63	0.99	6.42	0.06	4.25	-0.49
Number of data	16	16	14	14	14	14	16	16	16
$\|Z\| \leq 2$	13	13	12	12	9	12	13	13	14
$2 < \|Z\| < 3$	1	2	0	1	1	0	2	1	2
$3 \leq \|Z\|$	2	1	2	1	4	2	1	2	0

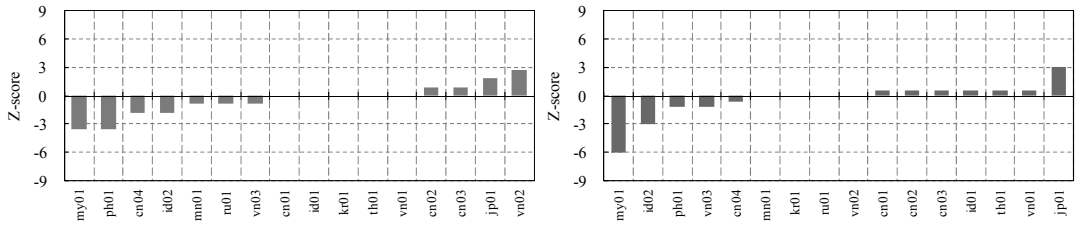
Note: Light mesh, $|Z| > 2$ (Questionable); Dark mesh, $|Z| \geq 3$ (Unsatisfactory); "---", Not measured

Appendix Table 4.4.2 Results of Z-score evaluation for sample No. 092

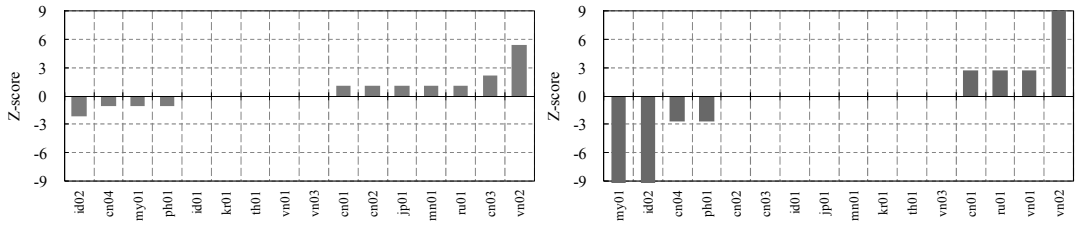
Sample No. 092 (Acrisols)

Lab. ID	pH (H_2O)	pH(KCl)	Ex-Ca	Ex-Mg	Ex-K	Ex-Na	Ex-Acidity	Ex-Al	Ex-H
cn01	0.60	2.70	-0.67	-0.06	0.53	0.23	-0.52	-0.44	-0.24
cn02	0.60	0.00	-0.92	0.32	-0.27	-0.23	0.88	0.73	0.24
cn03	0.60	0.00	-1.17	-1.23	-2.48	0.54	1.35	1.44	0.00
cn04	-0.60	-2.70	-0.97	-0.56	-0.09	-0.39	-0.31	-0.44	0.00
id01	0.60	0.00	0.31	2.22	2.57	1.16	-0.42	0.03	-0.60
id02	-3.00	-16.19	-1.89	-1.15	-2.92	-1.31	11.73	12.05	1.14
jp01	3.00	0.00	0.14	0.25	0.35	-0.08	-0.47	-0.03	-0.60
my01	-6.00	-21.58	-2.44	-2.84	-3.63	-1.93	73.36	75.75	0.84
mn01	0.00	0.00	---	---	---	---	1.04	-0.44	1.56
ph01	-1.20	-2.70	3.28	2.68	4.87	15.80	0.31	0.15	0.18
kr01	0.00	0.00	-0.14	0.06	-0.35	-0.54	-0.31	0.21	-0.48
ru01	0.00	2.70	0.41	0.89	0.09	-0.23	-0.62	-0.44	-0.36
th01	0.60	0.00	1.35	-0.12	-1.24	1.46	-0.73	-0.15	-0.72
vn01	0.60	2.70	0.25	-0.61	0.18	0.08	0.52	-0.44	0.96
vn02	0.00	13.49	---	---	---	---	0.21	-0.44	0.90
vn03	-1.20	0.00	0.57	2.94	0.27	4.86	-0.21	1.73	-0.54
Number of data	16	16	14	14	14	14	16	16	16
$\|Z\| \leq 2$	13	8	12	10	9	12	14	14	16
$2 < \|Z\| < 3$	2	5	1	4	3	0	0	0	0
$3 \leq \|Z\|$	1	3	1	0	2	2	2	2	0

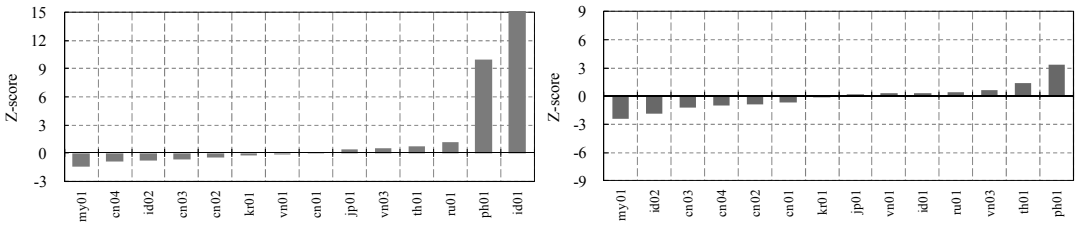
Note: Light mesh, $|Z| > 2$ (Questionable); Dark mesh, $|Z| \geq 3$ (Unsatisfactory); "---", Not measured



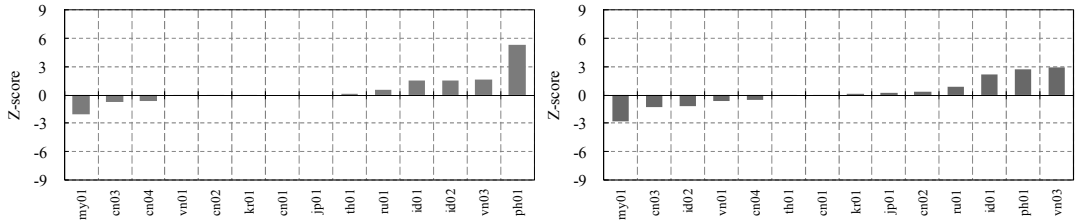
Appendix Figure 4.4.1 Distribution of Z-score for pH(H_2O) (Left: 091, Right: 092)



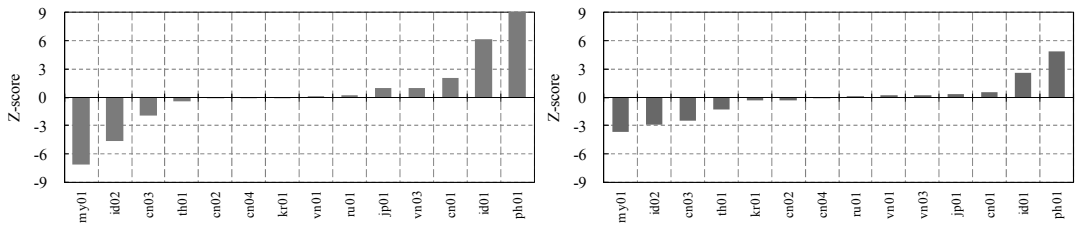
Appendix Figure 4.4.2 Distribution of Z-score for pH(KCl) (Left: 091, Right: 092)



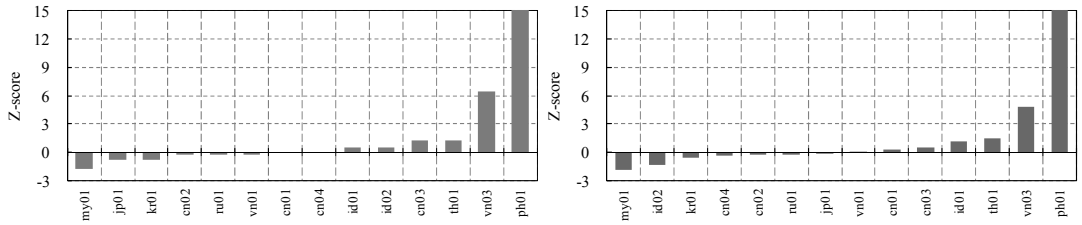
Appendix Figure 4.4.3 Distribution of Z-score for Ex-Ca (Left: 091, Right: 092)



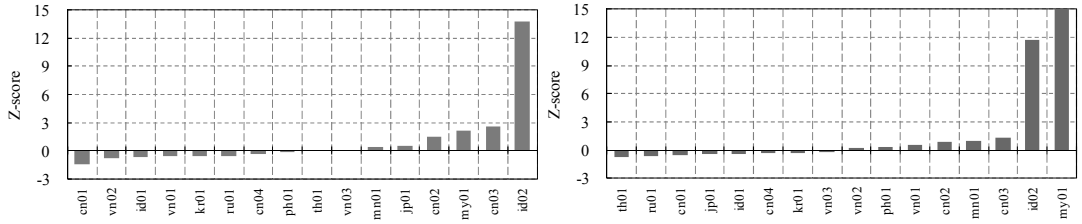
Appendix Figure 4.4.4 Distribution of Z-score for Ex-Mg (Left: 091, Right: 092)



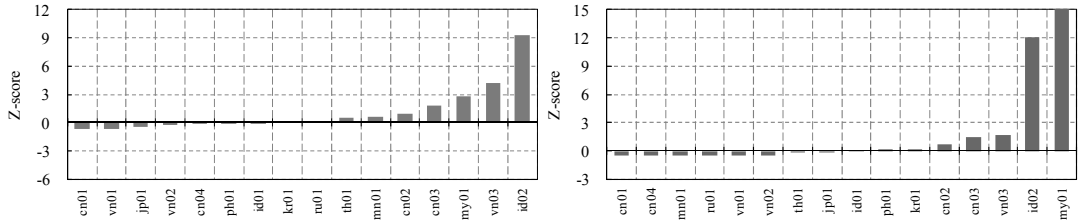
Appendix Figure 4.4.5 Distribution of Z-score for Ex-K (Left: 091, Right: 092)



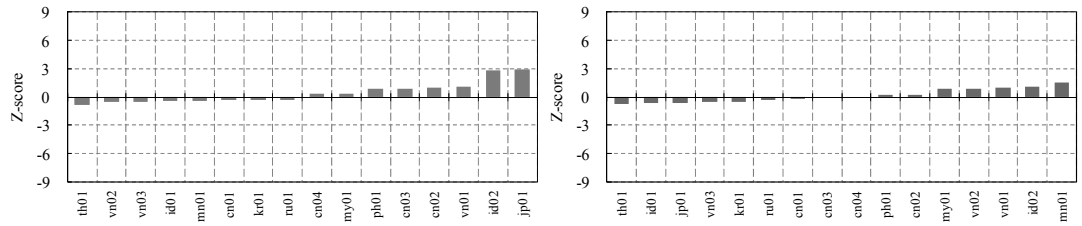
Appendix Figure 4.4.6 Distribution of Z-score for Ex-Na (Left: 091, Right: 092)



Appendix Figure 4.4.7 Distribution of Z-score for Ex-Acidity (Left: 091, Right: 092)



Appendix Figure 4.4.8 Distribution of Z-score for Ex-Al (Left: 091, Right: 092)



Appendix Figure 4.4.9 Distribution of Z-score for Ex-H (Left: 091, Right: 092)

5. 10th INTER-LABORATORY COMPARISON PROJECT ON INLAND AQUATIC ENVIRONMENT

5.1 Introduction

In the 10th inter-laboratory comparison of inland aquatic environment, artificial inland aquatic environment samples containing known concentrations of major ions, were prepared and sent to the participating countries of EANET by the Network Center (NC) in October 2009. All the participating laboratories submitted their analytical data to NC. The measurement of pH, EC, Alkalinity and concentrations of SO_4^{2-}, NO_3^-, Cl^-, Na^+, K^+, Ca^{2+}, Mg^{2+} and NH_4^+ from the participating countries were compared with prepared values and the results were statistically treated.

5.2 Procedure

5.2.1 Participating Laboratories

The Network Center (NC) shipped artificial inland aquatic environment samples to all 22 laboratories on October 1st in 2009, and almost laboratories submitted their analytical data to NC by February 28 in 2010. A list of the participating laboratories with their abbreviated name, and the code are given in Appendix 5-1. For this attempt, the laboratory my02 submitted the data of 3 parameters, pH, EC and Alkalinity. And the laboratory vn03 submitted the all data except SO_4^{2-} and NO_3^-.

5.2.2 Description Samples

A description of the samples is given in Table 5.1.

Table 5.1 Description of the artificial inland aquatic environment sample

Name	Amount of the sample	Container	Number of samples	Note
Artificial inland aquatic environment sample	Approximately 1L	Poly-propylene bottle 1L	One bottle	To analyze directly

The analytical parameters are shown in Table 5.2.

Table 5.2 Analytical parameters

Analytical Parameter	Reporting Units	
pH	pH units	-
EC	milli siemens/meter	mS/m
Alkalinity	milli equivalent/liter	meq/L
SO_4^{2-}	milli gram/liter	mg/L
NO_3^-	milli gram/liter	mg/L
Cl^-	milli gram/liter	mg/L
Na^+	milli gram/liter	mg/L
K^+	milli gram/liter	mg/L
Ca^{2+}	milli gram/liter	mg/L
Mg^{2+}	milli gram/liter	mg/L
NH_4^+	milli gram/liter	mg/L

The participating laboratories were informed that concentration of each parameter was prepared within the range described in Table 5.3.

Table 5.3 Concentration range of artificial inland aquatic environment sample

Parameter	Range	Parameter	Range
pH	5.0 – 8.0	Na^+	2 – 20 mg/L
EC	1.5 – 15 mS/m	K^+	0.2 – 2.0 mg/L
Alkalinity	0.05 – 0.5 meq/L	Ca^{2+}	1 – 10 mg/L
SO_4^{2-}	2 – 20 mg/L	Mg^{2+}	0.2 – 2.0 mg/L
NO_3^-	0.5 – 5 mg/L	NH_4^+	0.05 – 0.5 mg/L
Cl^-	1 – 10 mg/L		

5.2.3 Parameters analyzed

Participating laboratories are required to apply the analytical methods and data checking procedures that are specified in the "Technical Manual for Monitoring on Inland Aquatic Environment in East Asia (2000)" and the "QA/QC Program for Monitoring on Inland Aquatic Environment in East Asia (2000)". Analytical methods specified in the manual are described in Table 5.4.

Table 5.4 Analytical methods specified in the manual

Parameter	Analytical method
pH	Glass electrode
EC	Conductivity cell
Alkalinity	Titration by Burette or Digital Burette with pH Meter (end-point pH4.8)
SO_4^{2-} NO_3^-	Ion Chromatography or Spectrophotometry
Cl^-	Ion Chromatography or Titration
Na^+ K^+ Ca^{2+} Mg^{2+}	Ion Chromatography or Atomic Absorption / Flame (emission) photometry
NH_4^+	Ion Chromatography or Spectrophotometry (Indophenol blue)

5.2.4 Data Checking Procedures

a) Calculation of ion balance (R_1)

(1) Total anion (A) equivalent concentration (μeq/L) is calculated by sum up the concentration of anions (C: μmol/L) and Alkalinity (ALK: μeq/L). Alkalinity considered to be corresponded to bicarbonate ions (HCO_3^-).

$\boldsymbol{A}$ (μeq/L) =Σn C_{Ai} (μmol/L) = C (SO_4^{2-}) + C (NO_3^-) + C (Cl^-) + (ALK)

C_{Ai}: electric charge of ion and concentration (μmol/L) of anion "i".

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

$\boldsymbol{C}$ (μeq/L) = Σn C_{Ci} (μmol/L) = $10^{(6-pH)}$ + C (NH_4^+) + C (Na^+) + C (K^+) + C (Ca^{2+}) + C (Mg^{2+})

C_{Ci}: electric charge of ion and concentration (μmol/L) of cation "i".

(3) Calculation of ion balance (R_1)

$R_1 = 100 \times (\boldsymbol{C}-\boldsymbol{A}) / (\boldsymbol{C}+\boldsymbol{A})$ [%]

(4) R_1, which is calculated using the above equation, should be compared with standard values in Table 5.5. Re-measurement, check with standard solutions, and/or inspection of calibration curves should be undertaken, when R_1 is not within the range.

Table 5.5 Allowable ranges for R_1 in different concentration ranges

(C+A) [μeq/L]	R_1 [%]
< 50	+30 ~ -30
50 ~ 100	+15 ~ -15
>100	+8 ~ - 8

Reference: "Technical Manual for Monitoring on Inland Aquatic Environment in East Asia (2000)"

b) Comparison between calculated and measured electrical conductivity (R_2)

(1) Total electric conductivity (Λcalc) is calculated as follows;

$$\Lambda calc\ (mS/m) = \{349.7 \times 10^{(3-pH)} + 80.0 \times C(SO_4^{2-}) + 71.5 \times C(NO_3^-) + 76.3 \times C(Cl^-) + 73.5 \times C(NH_4^+) + 50.1 \times C(Na^+) + 73.5 \times C(K^+) + 59.8 \times C(Ca^{2+}) + 53.3 \times C(Mg^{2+}) + 44.5 \times (ALK)\}/10000$$

C: Molar concentrations (μmol/L) of ions in the parenthesis; each constant value is ionic equivalent conductance at 25°C. Alkalinity considered to be corresponded to bicarbonate ions (HCO_3^-).

(2) Ratio (R_2) of calculations (Λcalc) to measurements (Λcalc) in electric conductivity is calculated as follows;

$$R_2 = 100 \times (\Lambda calc - \Lambda meas)/(\Lambda calc + \Lambda meas)\ [\%]$$

(3) R_2, which is calculated using the above equation, is compared with standard values in Table 5.6. Re-measurement, check with standard solutions, and/or inspection of calibration curves are necessary, when R_2 is not within the range.

Table 5.6 Allowable ranges for R_2 in different concentration ranges

Λmeas[mS/m]	R_2 [%]
< 0.5	+ 20 ~ -20
0.5 ~ 3	+13 ~ -13
> 3	+9 ~ -9

Reference: "Technical Manual for Monitoring on Inland Aquatic Environment in East Asia (2000)"

5.3 Results

5.3.1 Outline of Results

Original data from the laboratories are shown in APPENDIX5-2 and APPENDIX5-3. Table 5.7 shows the summary of analytical result. Statistics calculated for each constituent of the artificial inland aquatic environment samples were: Average, Standard deviation (S.D.), Number of data (N), Minimum (Min.) and Maximum (Max.). Outlying data that apart from the average greater than a factor of 3 of S.D. is not included for the calculation in Table 5.7. As shown in Table 5.7, average of submitted data agreed fairly well with the prepared value/concentration within a range of ±10%.

Table 5.7 Summary of analytical results of the artificial inland aquatic environment sample

(Reported data after outliers were removed)

Constituents		Prepared	Average	S.D.	N	Min.	Max.
pH		7.00	7.05	0.21	21	6.56	7.46
EC	(mS/m)	3.75	3.68	0.08	20	3.53	3.83
Alkalinity	(meq/L)	0.149	0.154	0.01	22	0.137	0.190
SO_4^{2-}	(mg/L)	2.99	3.02	0.26	20	2.68	3.76
NO_3^-	(mg/L)	0.54	0.53	0.06	19	0.40	0.67
Cl^-	(mg/L)	3.62	3.61	0.31	21	3.18	4.38
Na^+	(mg/L)	3.47	3.45	0.12	19	3.25	3.70
K^+	(mg/L)	1.00	0.98	0.04	19	0.90	1.04
Ca^{2+}	(mg/L)	1.02	1.10	0.14	20	0.75	1.30
Mg^{2+}	(mg/L)	1.01	1.02	0.16	21	0.66	1.42
NH_4^+	(mg/L)	0.21	0.23	0.06	21	0.14	0.37

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

The Data Quality Objectives (DQOs) of EANET is specified for every constituent as ± 15% by the QA/QC program of the EANET. In this report, analytical data on artificial inland aquatic environmental samples is compared with the prepared value/concentration and evaluated by the DQO criteria: the flag "E" is put to the data that exceed DQO within a factor of 2 (±15% - ±30%) and the flag "X" is put to the data that exceed DQO more than a factor of 2 (<-30% or >30%). Data set for each sample was evaluated by the data checking procedures described in chapter 5.2.4 of this report. The results were evaluated following the two aspects: i) comparison of individual parameters, and ii) comparison of circumstance of analysis in each participating laboratory. Evaluation of data for each constituent is presented in “5.3.2 Evaluation of laboratories’ performance (by analytical parameters)”, and evaluation of data by circumstances of analysis such as analytical method used, experience of personnel, and other analytical

condition is described in “5.3.4 Information on laboratories”.

Table 5.8 shows the number of flagged data for each parameters and Figure 5.1 shows the percentage of flagged data.

Table 5.8 Number of flagged data

Flag*	pH	EC	Alkalinity	SO_4^{2-}	NO_3^-	Cl^-	Na^+	K^+	Ca^{2+}	Mg^{2+}	NH_4^+	Total	Ratio
E	1	0	1	2	3	2	1	2	9	0	5	26	11.2%
X	0	1	0	0	1	0	1	0	1	3	5	12	5.2%
Data within DQOs	21	21	21	18	16	19	19	19	11	18	11	194	83.6%
Flagged(%)	4.5	4.5	4.5	10.0	20.0	9.5	9.5	9.5	47.6	14.3	47.6	16.4	
*E : Value exceeded the DQO by a factor of 2 of the DQO (±15% - ±30%)													
*X : Value exceeded the DQO more than a factor of 2 of the DQO (<-30% or >30%)													

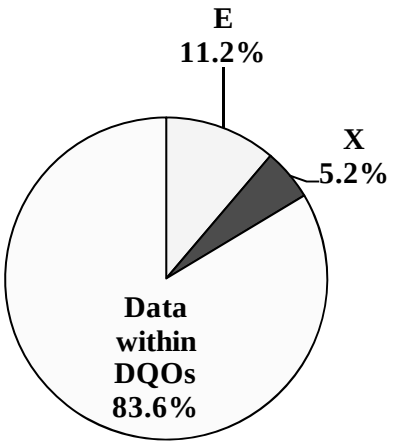


Figure 5.1 Percentage of flagged data

The data flagged by "E", which exceeded the DQOs within a factor of 2, shared 11.2% of all reported data of samples. And the data flagged by "X", which exceeded the DQOs more than a factor of 2, shared 5.2% of all reported data of samples. And the percentage of flagged cations was larger than that of anions. Especially the percentage of flagged Ca^{2+} and NH_4^+ exceeded more than 40%.

The distribution of flagged data in each laboratory is shown in Table 5.9 and Figure 5.2.

Table 5.9 Number of flagged data in each laboratory

Number of flagged data	Number of laboratories	Share
0	5	23%
1	8	36%
2	1	5%
3	6	27%
4	0	0%
5	2	9%
6	0	0%
7	0	0%
8	0	0%
9	0	0%
Total	22	100%

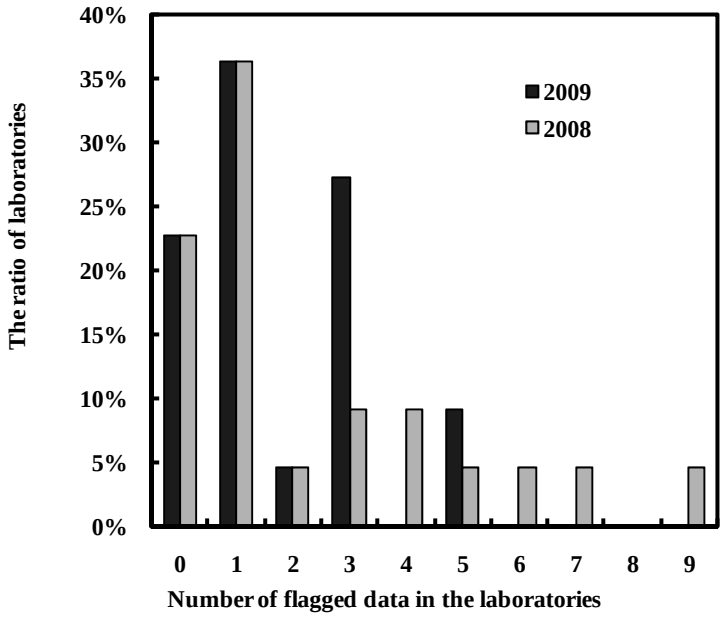


Figure 5.2 Distribution of laboratories with the number of flagged data

The percentage of the laboratories without flagged data was 23% in last attempt (2008), and that of this year was 23%. There was a laboratory that has five flagged data in this attempt.

Table 5.10 Analytical Results of Sample No.091i (artificial inland aquatic environment sample : EANET in 2009)

Lab. ID	pH	EC	Alkalinity	SO_4^{2-}	NO_3^-	Cl^-	Na^+	K^+	Ca^{2+}	Mg^{2+}	NH_4^+	R1	R2
	-	(mS/m)	(meq/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	-	-
cn01	7.10	3.69	0.15	3.03	0.52	3.58	3.42	0.99	E 1.19	1.09	0.23	2.05	2.30
cn02	7.14	3.63	0.14	2.90	0.53	3.52	3.46	1.01	E 1.23	1.12	0.21	4.96	2.58
cn03	7.12	3.69	0.15	3.01	0.55	3.54	3.47	1.03	1.15	1.10	0.22	2.41	2.34
cn04	6.93	3.69	0.14	2.91	0.57	3.53	3.42	0.97	E 1.21	1.13	0.21	4.04	1.73
id01	7.11	3.83	0.16	3.17	0.59	4.12	3.38	0.97	1.16	1.08	0.23	-2.84	2.49
id02	7.10	3.81	0.15	2.89	0.53	4.03	3.49	0.90	1.17	1.01	0.21	-0.67	1.00
id03	E 5.55	3.81	0.16	E 3.76	E 0.40	E 4.23	E 2.79	1.02	1.06	1.15	0.19	I -8.57	3.75
jp01	7.09	3.71	0.16	2.92	0.51	3.52	3.41	0.97	0.97	0.95	0.22	-2.71	0.44
jp02	7.29	3.53	0.15	2.85	0.52	3.55	3.41	0.92	1.01	1.00	X 0.33	0.49	3.19
la01	6.68	3.26	0.17	2.93	0.62	3.36	X 4.67	E 1.26	X 3.31	X 1.42	X 0.28	I 22.99	C 21.22
mn01	6.93	3.64	0.14	2.82	0.52	3.42	3.25	0.93	1.03	1.00	E 0.25	1.49	-0.23
my01	6.56	3.69	0.16	2.85	E 0.44	3.49	3.50	0.99	0.94	0.94	E 0.17	-2.24	0.39
my02	6.73	X 7.67	0.17									-	-
ph01	7.04	3.70	0.14	3.30	X 0.24	3.59	3.36	1.01	E 0.82	0.94	X 0.14	-2.83	-1.13
ph02	7.46	3.67	E 0.19	3.24	E 0.67	3.28	3.38	0.99	1.07	1.00	E 0.26	-5.62	3.72
ru01	7.11	3.65	0.14	2.90	0.53	3.63	3.40	0.98	1.04	1.00	0.20	1.60	0.63
ru02	7.14	3.80	0.15	E 3.50	0.55	3.65	3.70	1.04	E 1.26	0.90	E 0.26	0.92	2.20
th01	6.95	3.64	0.15	3.00	0.50	3.57	3.60	1.00	E 1.20	1.10	0.20	2.97	3.44
th02	7.34	3.64	0.16	2.84	0.49	3.18	3.41	0.92	E 1.30	1.13	0.19	2.95	2.44
vn01	7.05	3.53	0.15	2.68	0.51	3.38	3.70	0.98	E 1.18	X 0.66	E 0.15	-0.33	0.86
vn02	7.07	3.59	0.15	2.95	0.50	3.37	3.31	E 0.72	1.17	X 0.66	X 0.37	-2.32	-0.35
vn03	7.01	3.64	0.16			E 4.38	3.47	0.99	E 0.75	0.96	X 0.37	-	-
Expected value	7.00	3.75	0.15	2.99	0.54	3.62	3.47	# 1.00	1.02	1.01	0.21	-	-

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

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

I:Poor ion balance (R1)

C:Rich Conductivity agreement (R2)

5.3.2 Evaluation of laboratories' performance (by analytical parameters)

The general overviews of data are presented below in Figures 5.3 to 5.13. for each analytical parameter. The results received from each laboratory are normalized by prepared values to evaluate deviation from prepared values.

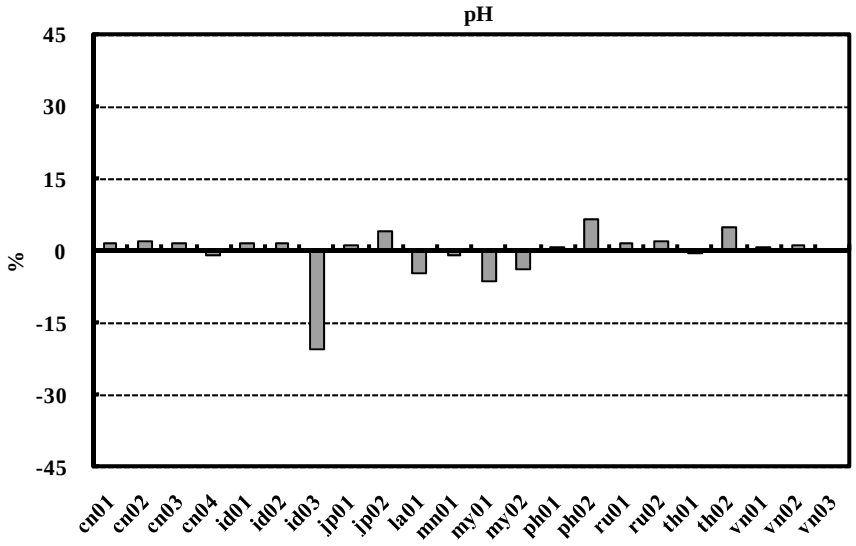


Figure 5.3 Distribution of results for pH (normalized by prepared value)

One data were flagged. All the rest data of pH were within DQOs.

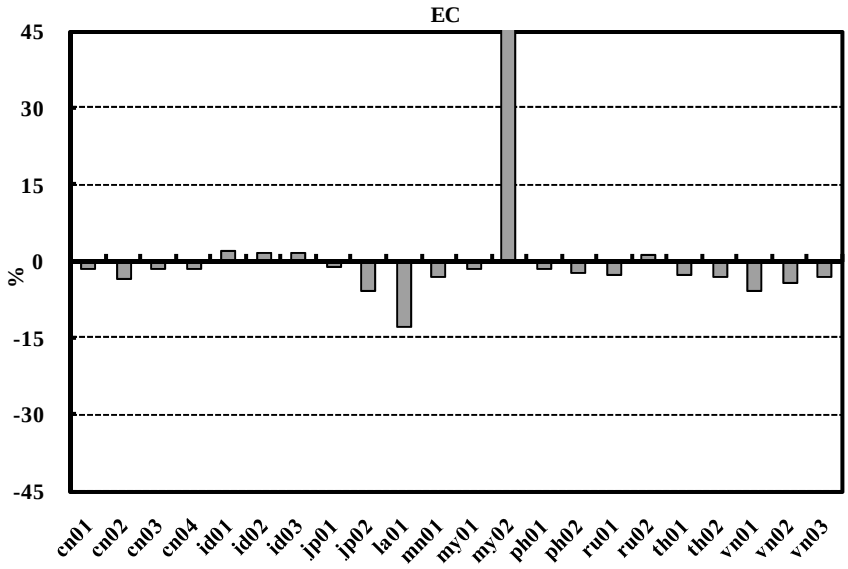


Figure 5.4 Distribution of results for EC (normalized by prepared value)

One data were flagged. It was significantly deviated from prepared value. All the rest data of EC were within DQOs. Most of data are seen a little below from the expected value.

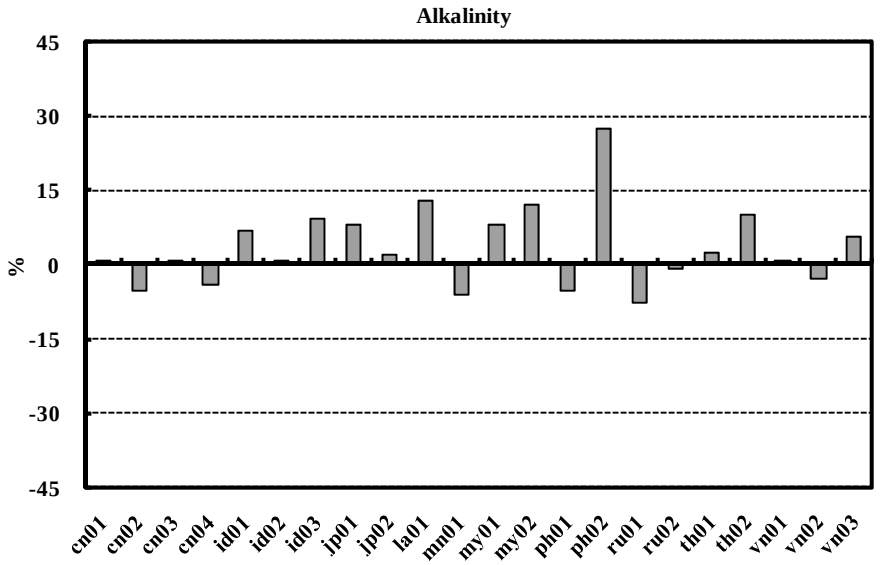


Figure 5.5 Distribution of results for Alkalinity (normalized by prepared concentration)

One data were flagged. All the rest data of Alkalinity were within DQOs..

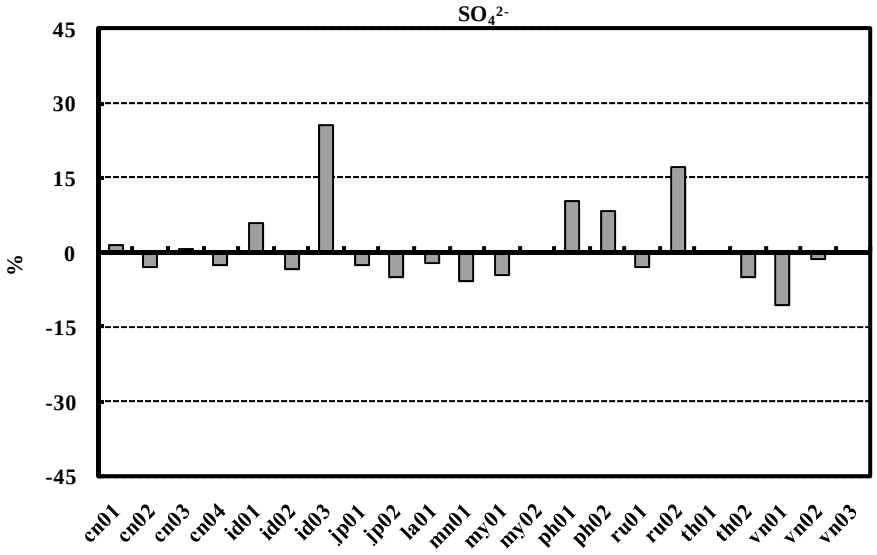


Figure 5.6 Distribution of results for SO_4^{2-} (normalized by prepared concentration)

Data from two laboratories were flagged. Most of participating laboratories used ion chromatography for the determination of SO_4^{2-} and there was no flagged data analyzed with it. While three laboratories used spectrophotometer and there were two flagged data analyzed with it, too.

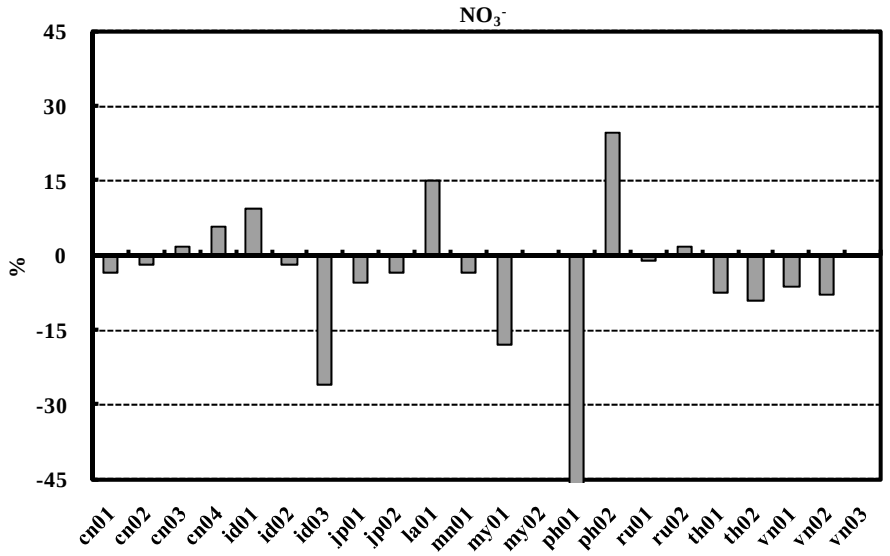


Figure 5.7 Distribution of results for NO_3^- (normalized by prepared concentration)

Most of the participating laboratories used ion chromatography for the determination of NO_3^-, while three laboratories used spectrophotometer. Data from four laboratories were flagged. Especially one of them was significantly deviated from prepared value. Three of the samples which had flagged data were analyzed with ion chromatography method and other one was analyzed with spectrophotometer.

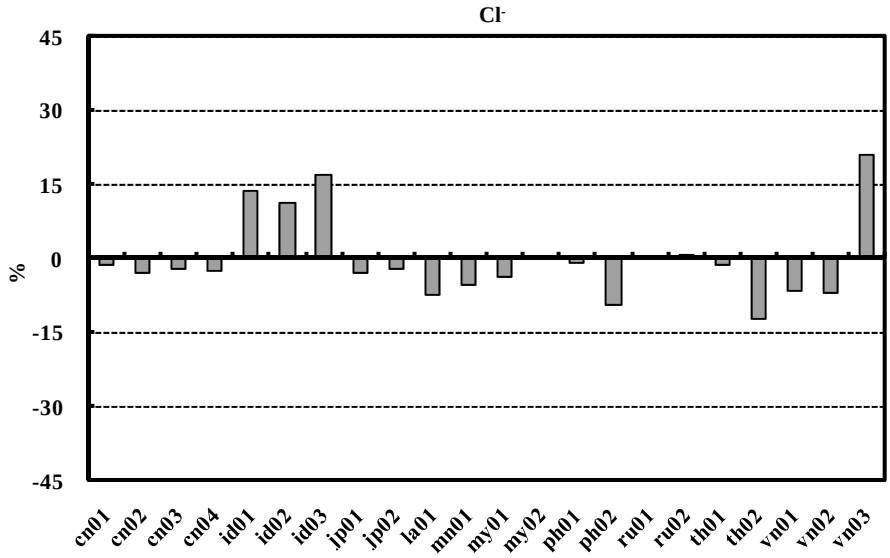


Figure 5.8 Distribution of results for Cl^- (normalized by prepared concentration)

Most of participating laboratories used ion chromatography, four laboratories used titration method and one laboratory used spectrophotometer (other method) for the determination of Cl^-. Data from two laboratories were flagged analyzed by titration method.

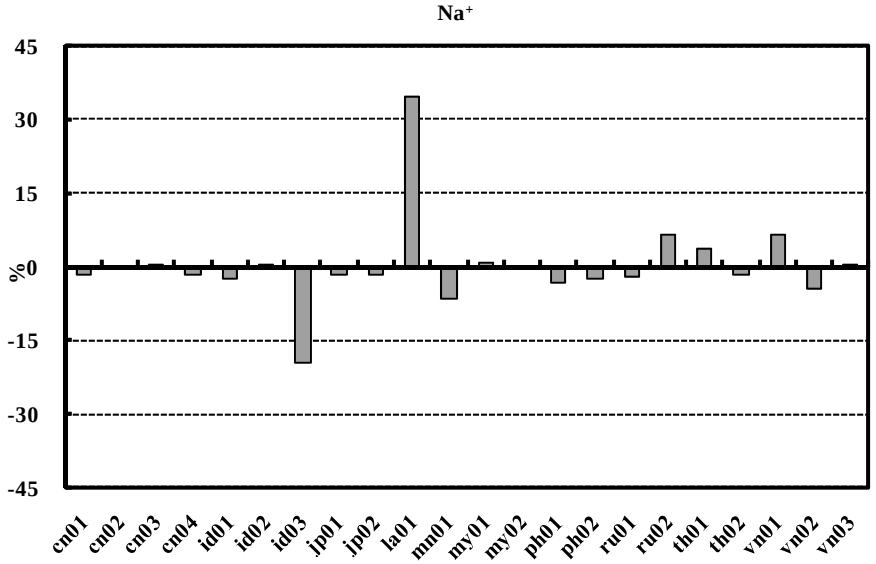


Figure 5.9 Distribution of results for Na^+ (normalized by prepared concentration)

Most of participating laboratories used ion chromatography, while six laboratories used atomic absorption/flame (emission) photometry for the determination of Na^+. Data from two laboratories were flagged, and one data was obtained from the use of atomic absorption/flame (emission) photometry method, another was from use of ion chromatography method.

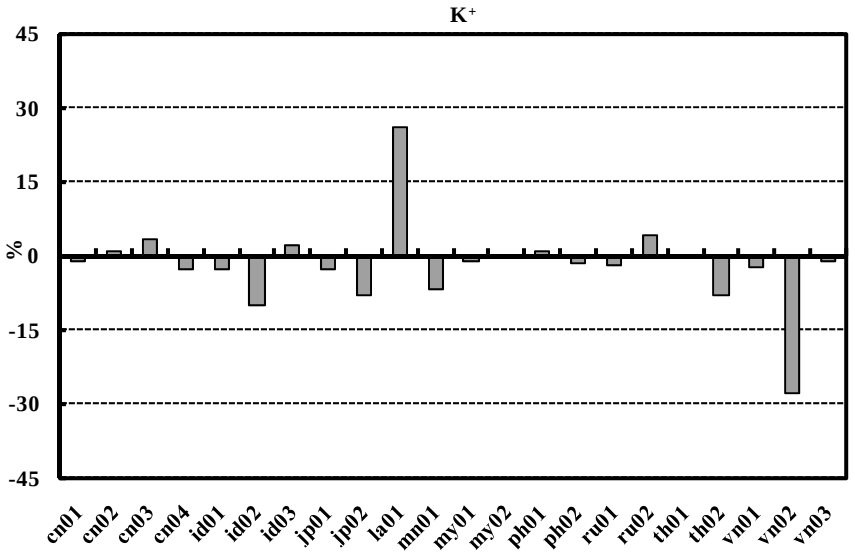


Figure 5.10 Distribution of results for K^+ (normalized by prepared concentration)

Most of participating laboratories used ion chromatography, and six laboratories used atomic absorption/flame (emission) photometry for the determination of K^+.

Data from two laboratories were flagged, and the data were obtained from the use of ion chromatography method.

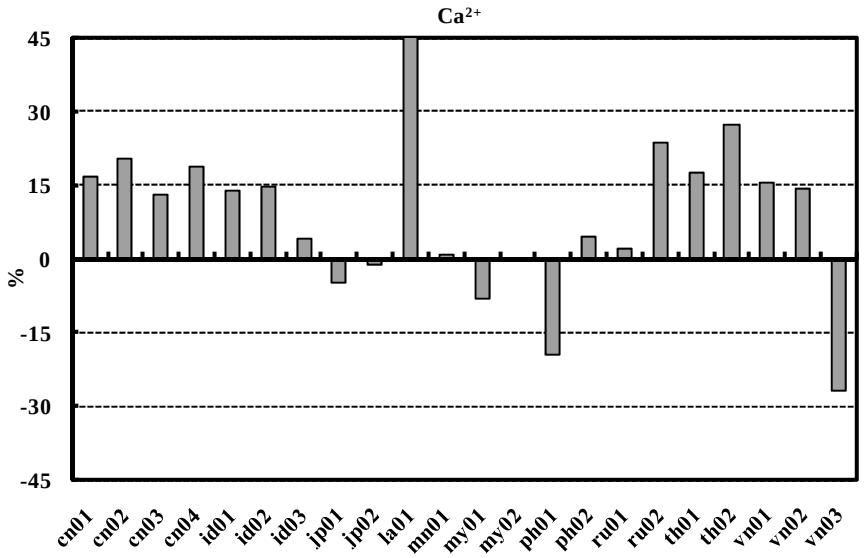


Figure 5.11 Distribution of results for Ca^{2+} (normalized by prepared concentration)

Most of participating laboratories used ion chromatography, six laboratories used atomic absorption/flame (emission) photometry for the determination of Ca^{2+}. Data from ten laboratories were flagged. Seven flagged data were obtained from the use of ion chromatography method, and three data were obtained from the use of atomic absorption/flame (emission) photometry method. And most of flagged data and near flagged data are over the expected value. It may be necessary to pay more attention to the accuracy of Ca^{2+} analyze in the inland water sample in each laboratories. For example, it should be kept clean in the sample flow line of ion chromatography to prevent the contamination.

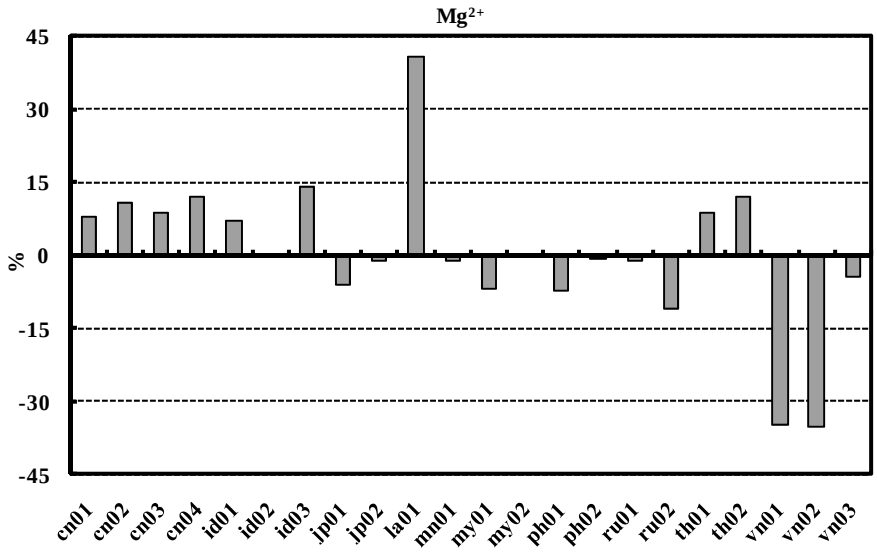


Figure 5.12 Distribution of results for Mg^{2+} (normalized by prepared concentration)

Most of participating laboratories used ion chromatography and six laboratories used atomic absorption/flame (emission) photometry for the determination of Mg^{2+}. Data from three laboratories were flagged, and their data were obtained from the use of ion chromatography method.

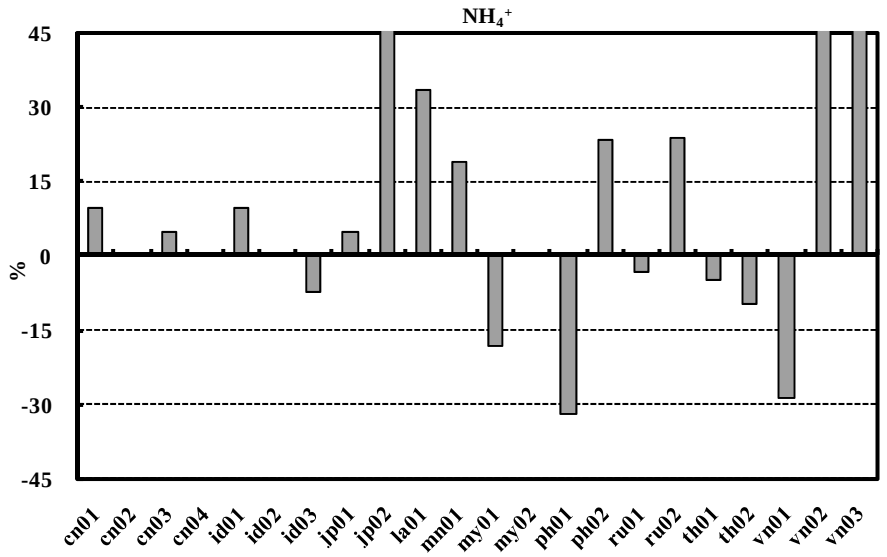


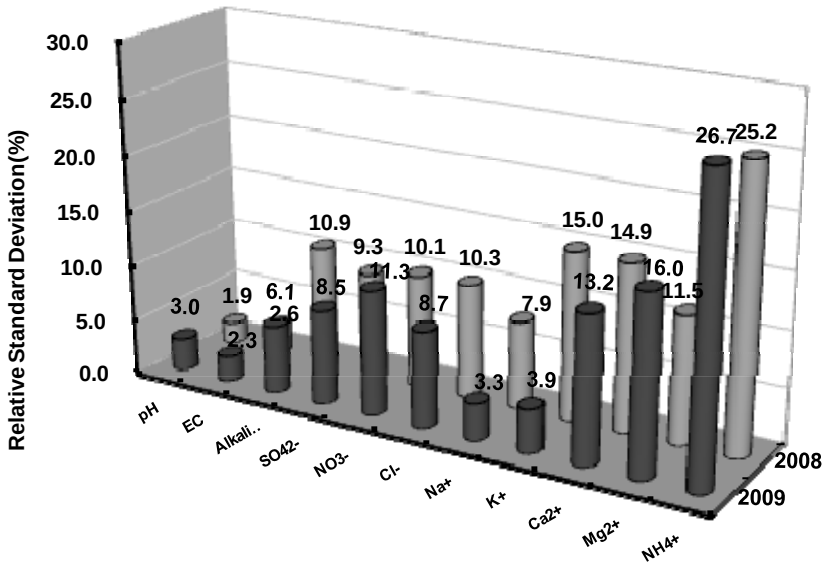
Figure 5.13 Distribution of results for NH_4^+ (normalized by prepared concentration)

Among 21 participating laboratories, 15 laboratories used ion chromatography, 5 laboratories used spectrophotometer (Indophenol) and 1 laboratories used spectrophotometer (other method) for the determination of NH_4^+. Data from nine laboratories were flagged. Especially six data were significantly deviated from prepared value. Seven flagged data were obtained by ion chromatography method, two data were obtained by indophenol method, and one data was obtained by spectrophotometry except indophenol method.

NH_4^+ was the parameter that has the highest flagged percentage in this attempt, too. It had also the highest flagged percentage in the attempt in 2003-2009. It may be necessary to pay more attention to the accuracy of NH_4^+ analyze in the inland water sample in each laboratories.

5.3.3 Overall Evaluation

Calculated relative standard deviation of the whole sets of analytical data is presented in Figure 5.14 with comparison to last attempt (2008).



(Relative standard deviation (%) = Standard deviation / Average x 100, Reported data after outliers were removed)

Figure 5.14 Relative standard deviation of each constituent

The relative standard deviations of three cations (Ca^{2+}, Mg^{2+} and NH_4^+) results from laboratories were larger than other parameters. Na^+, K^+ of this attempt were small than last year. The relative standard of NO_3^- results from laboratories was over 10 %. It may be necessary to pay more attention to the variation among the laboratories of NO_3^-, Ca^{2+}, Mg^{2+} and NH_4^+ analysis in the inland water than other analytical parameters.

5.3.4 Information on laboratories

Methodologies used

The percentages of laboratories using the recommended methods are shown in Fig. 5.15, and the codes used for the various analytical methods are shown in Table 5.11 and 5.12.

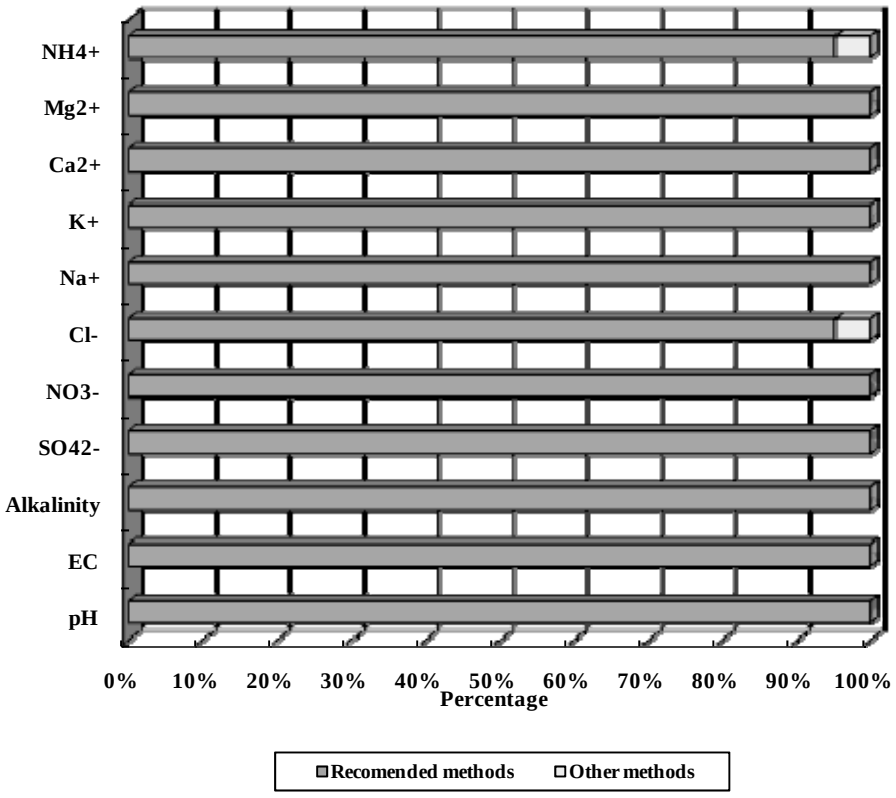


Figure 5.15 Percentage of laboratories using the recommended methods

Table 5.11 List of methods

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

Table 5.12 Analytical methods

Code	pH	EC	Alkalinity	SO_4^{2-}	NO_3^-	Cl^-	Na^+	K^+	Ca^{2+}	Mg^{2+}	NH_4^+
0	22(1)										
1		22(1)									
2			22(1)			4(2)					
3							6(1)	6	6(3)	6	
4				17	17(3)	16	15(1)	15(2)	15(7)	15(3)	15(7)
5											
6											
7				3(2)	3(1)	1					1(1)
8											5(2)
9											
10											
11											
Flagged E	1	0	1	2	3	2	1	2	9	0	5
Flagged X	0	1	0	0	1	0	1	0	1	3	5
	Reverse mesh is a recommended method of EANET										
	( ) : Number of data, which flagged by "E" or "X"										

The participating laboratories used recommended methods of EANET except measurement of Cl^- and NH_4^+. One laboratories used spectrophotometer instead of indophenol blue for NH_4^+ analysis, and its data was flagged. One laboratory used spectrophotometer for Cl^-.

The percentages of flagged cations were larger than those of anions. For the determination of cations, most of participating laboratories used ion chromatography, and some of them used atomic absorption/flame (emission) photometry. There is a tendency the laboratories without ion chromatography has many flagged data more than laboratories with IC, however 2 of 6 the laboratories without ion chromatography has no flagged data in this attempt.

Staff (numbers and years of experience)

Number of staff in charge of measurement in each laboratory is shown in Table 5.13.

Table 5.13 Staff in charge of measurement

Lab.ID	Total	pH	EC	Alkalinity	SO_4^{2-}	$NO3^-$	Cl^-	Na^+	K^+	Ca^{2+}	Mg^{2+}	$NH4^+$
cn01	1	A	A	A	A	A	A	A	A	A	A	A
cn02	3	A	A	B	C	C	C	C	C	C	C	C
cn03	1	A	A	A	A	A	A	A	A	A	A	A
cn04	2	A	A	A	B	B	B	B	B	B	B	B
id01	1	A	A	A	A	A	A	A	A	A	A	A
id02	3	A	A	A	A	B	B	C	C	C	C	A
id03	2	A	A	A	A	A	B	B	B	B	B	A
jp01	1	A	A	A	A	A	A	A	A	A	A	A
jp02	2	A	A	B	B	B	B	B	B	B	B	B
la01	1	A	A	A	A	A	A	A	A	A	A	A
mn01	2	A	A	A	B	B	B	A	A	A	A	A
my01	3	A	A	A	B	B	B	C	C	C	C	C
my02	1	A	A	A								
ph01	3	A	A	A	B	B	B	C	C	C	C	A
ph02	3	A	A	B	C	C	C	C	C	C	C	C
ru01	4	A	B	A	C	C	C	D	D	D	D	B
ru02	3	A	A	A	B	B	A	C	C	C	C	B
th01	2	A	B	A	B	B	B	A	A	A	A	A
th02	1	A	A	A	A	A	A	A	A	A	A	A
vn01	3	A	A	B	C	C	C	B	B	B	B	B
vn02	2	A	A	A	B	B	B	B	B	B	B	B
vn03	4	A	B	B			B	C	B	C	C	B

"-" : No information, "A", "B", "C" and "D" represent individuals of staff in each laboratory who are in charge of measurement.

Reverse mesh : "E" or "X" in sample flagged Data.

There were no clear relationship between data quality and the number of staff in charge of measurement.

Years of experience of each laboratory are shown in Table 5.14.

Table 5.14 Years of experience

Unit : year

Lab.ID	pH	EC	Alkalinity	SO_4^{2-}	NO_3^-	Cl^-	Na^+	K^+	Ca^{2+}	Mg^{2+}	NH_4^+
cn01	18	18	18	18	18	18	18	18	18	18	18
cn02	19	19	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5
cn03	14	14	14	14	14	14	14	14	14	14	14
cn04	3	3	3	24	24	24	24	24	24	24	24
id01	12	12	12	12	12	12	12	12	12	12	12
id02	27	27	27	27	32	32	5	5	5	5	27
id03	5	5	5	5	5	1	1	1	1	1	5
jp01	1	1	1	1	1	1	1	1	1	1	1
jp02	4	4	7	7	7	7	7	7	7	7	7
la01	3	3	3	3	3	3	3	3	3	3	3
mn01	10	10	10	15	15	15	10	10	10	10	10
my01	3	3	3	7	7	7	7	7	7	7	7
my02	2	2	2								
ph01	1.5	1.5	1.5	1.5	1.5	1.5	20	20	20	20	1.5
ph02	18	18	5	18	18	18	18	18	18	18	18
ru01	7	16	7	14	14	14	24	24	24	24	16
ru02	49	49	49	15	15	49	18	18	18	18	15
th01	12	7	12	7	7	7	12	12	12	12	12
th02	12	12	12	12	12	12	12	12	12	12	12
vn01	1	1	17	27	27	27	17	17	17	17	17
vn02	7	7	7	17	17	17	17	17	17	17	17
vn03	3	4	4			4	6	4	6	6	4

Reverse mesh:Data were Flagged by "E" or "X" in sample

1 year means experienced with one year or less.

There were no clear relationship between data quality and years of experience.

5.4. Comparison with past surveys

The inter-laboratory comparison projects of EANET have been carried out 10 times, and the results showing the percentage of flagged data and the percentage of data that satisfied the DQOs are shown in Figure.5.16.

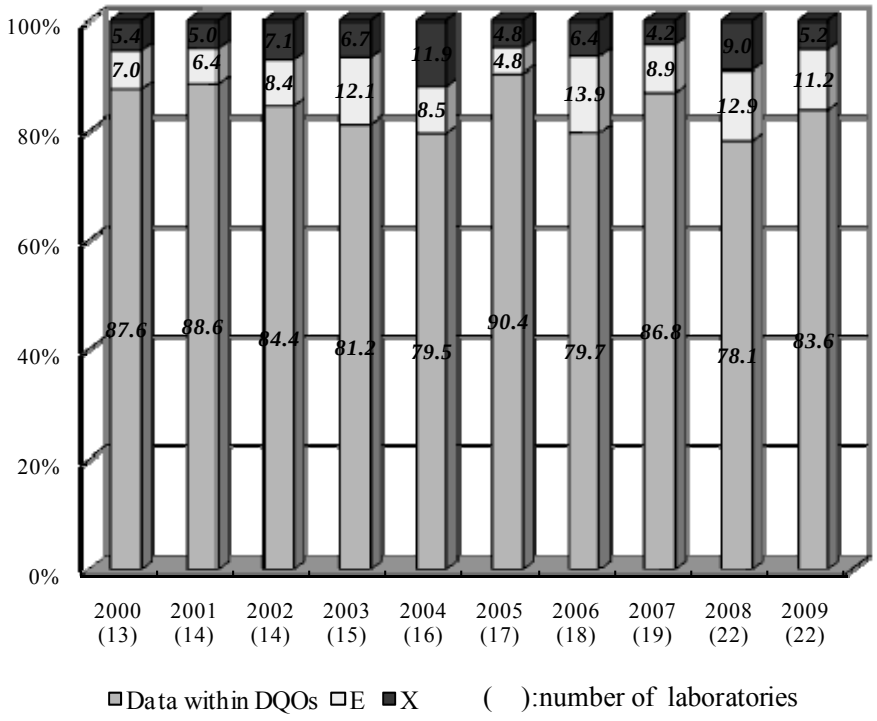


Figure 5. 16 Comparison of the results from the inter-laboratory comparison projects

The graph shows the number of laboratories increase and data within DQOs decrease. But it was relatively good results in this attempt.

The comparison for each parameter from 1st to 10th project with the percentage of flagged data is shown in Figure 5.17.

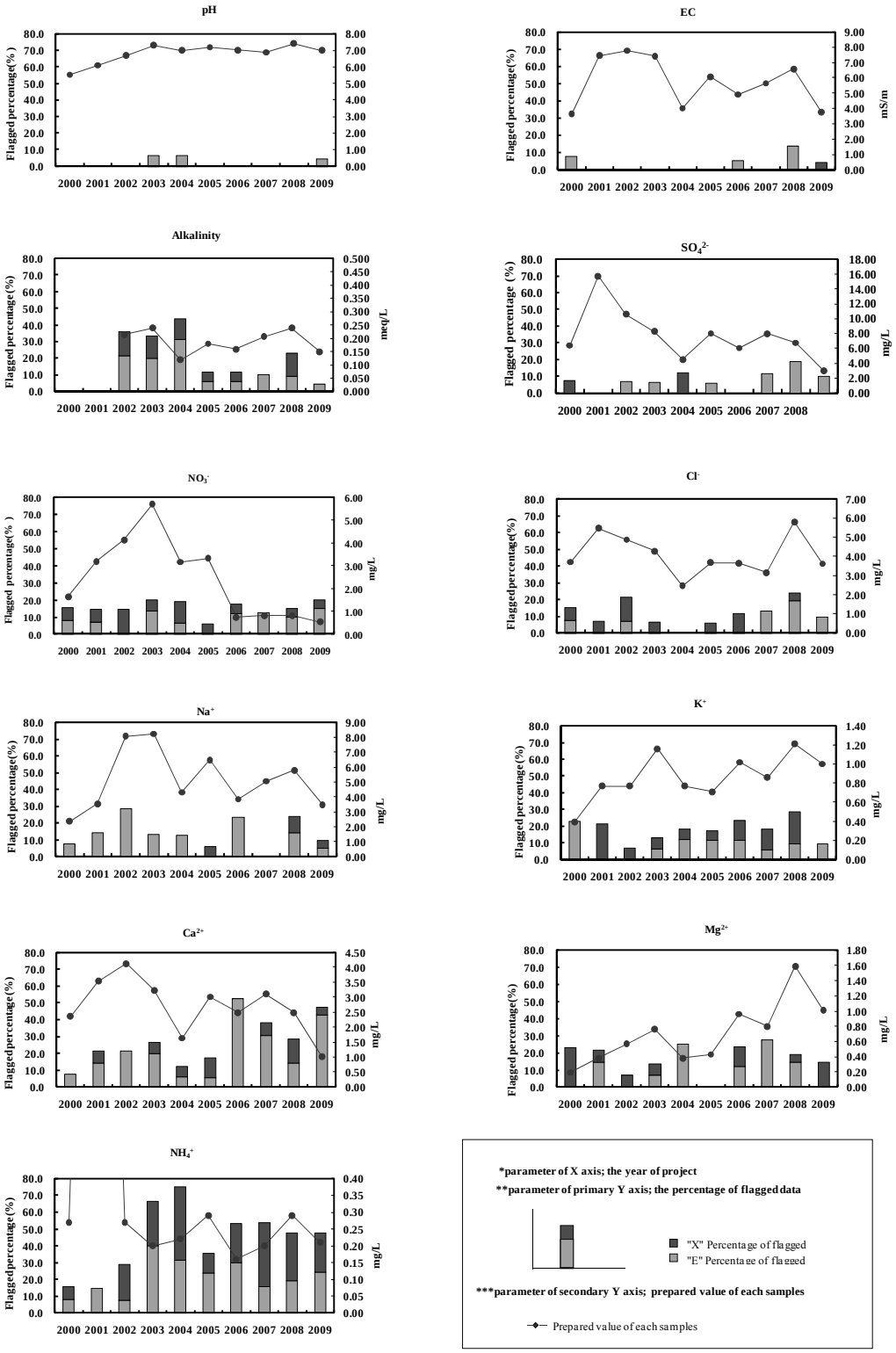


Figure 5.17 Comparison of the percentage of flagged data for each parameter in inter-laboratory comparison projects

The percentage of flagged data of NH_4^+ increased when the concentration of NH_4^+ was low.

Concerning other parameters, there were no clear relationship between the concentration and the percentage of flagged data.

The percentages of flagged cations were larger than those of anions in this project. Especially the percentages of flagged Ca^{2+} and NH_4^+ were more than 25%. The percentage of flagged NH_4^+ was larger than other parameters in every survey except 1st- 3rd project. The percentage of flagged Ca^{2+} of 7th - 10th project was relatively high. It was not clear why the number of flagged data for Ca^{2+} increased. In this project the percentage flagged K^+ was lower than last attempt. But it seems that there is necessity to pay attention to the analysis of Ca^{2+} and NH_4^+ in inland aquatic water.

5.5. Recommendations for improvement

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

The analytical method for analyzing an inland aquatic water sample is almost the same as that for analyzing wet deposition samples. Therefore the recommendations for improvements listed below are similar.

5.5.1 Measurement and Analysis

1) General

- ►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.

2) Deionized water

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

3) 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.

4) 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**.**

5) Calibration of analytical instruments

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

5.5.2 Evaluation of reliability

1) Sensitivity fluctuation of analytical instruments

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

For example, in Ion chromatography

- ►A new calibration should be performed before the measurements are reached to over 30 samples.
- ►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 should be made, and reference solution should be measured again.
- ►If the retention time changes slowly while the separator column is deteriorating, then adequate actions should 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.

5.5.3 Data control

1) Data checks by the analytical laboratories

- ►When the sensitivity of instruments is not stable, when the results of duplicate analyses or re-measurements are significantly different, or when the percentage 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 questionable quality. When abnormal or unrecorded data is detected, the process should be carefully reviewed to prevent the occurrence of the same problem in the future.

References

1) EANET, March 2000. Technical Manual for Monitoring on Inland Aquatic Environment in East Asia: Adopted at The Second Interim Scientific Advisory Group Meeting of Acid Deposition Monitoring Network in East Asia

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3) Acid Deposition and Oxidant Research Center, November 2001. Report of the Inter-laboratory Comparison Project 2000 on Inland Aquatic Environment, 1St attempt,

4) Acid Deposition and Oxidant Research Center, November 2002. Report of the Inter-laboratory Comparison Project 2001 on Inland Aquatic Environment, 2nd attempt

5) Acid Deposition and Oxidant Research Center, November 2003. Report of the Inter-laboratory Comparison Project 2002 on Inland Aquatic Environment, 3rd attempt

6) Acid Deposition and Oxidant Research Center, November 2004. Report of the Inter-laboratory Comparison Project 2003 on Inland Aquatic Environment, 4th attempt

7) Acid Deposition and Oxidant Research Center, November 2005. Report of the Inter-laboratory Comparison Project 2004 on Inland Aquatic Environment,5th attempt

8) Acid Deposition and Oxidant Research Center, November 2006. Report of the Inter-laboratory Comparison Project 2005 on Inland Aquatic Environment, 6th attempt

9) Network Center for EANET. Report of the Inter-laboratory Comparison Project 2006

10) Network Center for EANET. Report of the Inter-laboratory Comparison Project 2007

11) Network Center for EANET. Report of the Inter-laboratory Comparison Project 2008

Appendix 5.1 Participating laboratories

CHINA

1) Zhuhai Environmental Monitoring Station	(cn01)
2) Xiamen Environmental Monitoring Central Station	(cn02)
3) Xi'an Environmental Monitoring Station	(cn03)
4) Chongqing Institute of Environmental Science	(cn04)

INDONESIA

5) Environmental Management Center (EMC)	(id01)
6)Research Center for Water Resources(RCWR), Agency for Research and Development, Ministry of Public Works	(id02)
7))Research Center for Limnology(RCL), Indonesian Institute of Sciences(LIPI) Cibinong Sciences Center	(id03)

JAPAN

8) Gifu Prefectural Institute of Health and Environmental Science	(jp01)
9) Shimane Prefectural Institute of Public Health and Environmental Science	(jp02)

Lao PDR

10)Environment Quality Monitoring Centre, Environment Research Institute, Water Resources and Environment Administration	(la01)

MALAYSIA

11) Division of Environmental Health, Department of Chemistry(DOC)	(my01)
12) Faculty of Applied Science, University Technology Mara (UiTM)	(my02)

MONGOLIA

13)Central Laboratory of Environmental Monitoring(CLEM)	(mn01)

PHILIPPINES

14)Environmental Management Bureau (EMB),	(ph01)
15)Environmental Management Bureau Cordillera Administrative Region (EMB-CAR),	(ph02)

RUSSIA

16)Limnologcal Institute of Russian Academy of Science/Siberian Branch (RAS/SB)	(ru01)
17)Primorskii Environmental Monitoring Center of Roshydromet (Laboratory for Monitoring of inland water Pollution)	(ru02)

THAILAND

18)Environmental Research and Training Center (ERTC)	(th01)
19)Air Quality and Noise Management Division, Pollution Control Department(PCD) Ministry of Science Technology and Environment(MSTE)	(th02)

VIET NAM

20) Environmental Laboratory - Center for Environmental Research - Vietnam Institute of Meteorology, Hydrology and Environment (IMHEN)	(vn01)
21) Middle of Central regional Hydro-Meteorological Observatory National	(vn02)

Hydro -Meteorological Center (NHMS)

22)Environmental research Division, Sub-Institute of HydroMeteorology and Environment of South Vietnam (SIHYMETE) (vn03)

Appendix Table 5.2 Results submitted by the laboratories

Lab. ID	pH -	EC (mS/m)	Alkalinity (meq/L)	SO_4^{2-} (mg/L)	NO_3^- (mg/L)	Cl^- (mg/L)	Na^+ (mg/L)	K^+ (mg/L)	Ca^{2+} (mg/L)	Mg^{2+} (mg/L)	NH_4^+ (mg/L)
cn01	7.10	3.69	0.15	3.03	0.52	3.58	3.42	0.99	1.19	1.09	0.23
cn02	7.14	3.63	0.141	2.90	0.53	3.52	3.46	1.01	1.23	1.12	0.21
cn03	7.12	3.69	0.150	3.01	0.55	3.54	3.47	1.03	1.15	1.10	0.22
cn04	6.93	3.69	0.143	2.91	0.57	3.53	3.42	0.97	1.21	1.13	0.21
id01	7.11	3.83	0.159	3.17	0.59	4.12	3.38	0.97	1.16	1.08	0.23
id02	7.10	3.81	0.150	2.89	0.53	4.03	3.49	0.90	1.17	1.01	0.21
id03	5.55	3.81	0.163	3.76	0.40	4.23	2.79	1.02	1.06	1.15	0.19
jp01	7.09	3.71	0.161	2.92	0.51	3.52	3.41	0.97	0.97	0.95	0.22
jp02	7.29	3.53	0.152	2.85	0.52	3.55	3.41	0.92	1.01	1.00	0.33
la01	6.68	3.26	0.168	2.93	0.62	3.36	4.67	1.26	3.31	1.42	0.28
mn01	6.93	3.64	0.140	2.82	0.52	3.42	3.25	0.93	1.03	1.00	0.25
my01	6.56	3.69	0.161	2.85	0.44	3.49	3.50	0.99	0.94	0.94	0.17
my02	6.73	7.67	0.167								
ph01	7.04	3.70	0.141	3.30	0.24	3.59	3.36	1.01	0.82	0.94	0.14
ph02	7.46	3.67	0.190	3.24	0.67	3.28	3.38	0.99	1.07	1.00	0.26
ru01	7.11	3.65	0.137	2.90	0.53	3.63	3.40	0.98	1.04	1.00	0.20
ru02	7.14	3.80	0.148	3.50	0.55	3.65	3.70	1.04	1.26	0.90	0.26
th01	6.95	3.64	0.153	3.00	0.50	3.57	3.60	1.00	1.20	1.10	0.20
th02	7.34	3.64	0.164	2.84	0.49	3.18	3.41	0.92	1.30	1.13	0.19
vn01	7.05	3.53	0.150	2.68	0.51	3.38	3.70	0.98	1.18	0.66	0.15
vn02	7.07	3.59	0.145	2.95	0.50	3.37	3.31	0.72	1.17	0.66	0.37
vn03	7.01	3.64	0.157			4.38	3.47	0.99	0.75	0.96	0.37
Expected value	7.00	3.75	0.149	2.99	0.54	3.62	3.47	1.00	1.02	1.01	0.21
Number of data	22	22	22	20	20	21	21	21	21	21	21
Average	6.98	3.84	0.154	3.02	0.51	3.61	3.48	0.98	1.20	1.02	0.23
Minimum	5.55	3.26	0.137	2.68	0.24	3.18	2.79	0.72	0.75	0.66	0.14
Maximum	7.46	7.67	0.190	3.76	0.67	4.38	4.67	1.26	3.31	1.42	0.37

blank : not analyzed

Appendix Table 5.3 Data normalized by prepared value

(Original data / Expected Value - 1) * 100 (%)

Lab. ID	pH (%)	EC (%)	Alkalinity (%)	SO_4^{2-} (%)	NO_3^- (%)	Cl^- (%)	Na^+ (%)	K^+ (%)	Ca^{2+} (%)	Mg^{2+} (%)	NH_4^+ (%)
cn01	1.4	-1.6	0.7	1.3	-3.7	-1.1	-1.4	-1.0	16.7	7.9	9.5
cn02	2.0	-3.3	-5.4	-3.0	-1.9	-2.8	-0.3	1.0	20.6	10.9	0.0
cn03	1.7	-1.7	0.9	0.7	1.9	-2.3	0.1	3.3	13.1	8.6	4.8
cn04	-1.0	-1.6	-4.0	-2.7	5.6	-2.5	-1.4	-3.0	18.6	11.9	0.0
id01	1.6	2.1	6.7	6.0	9.3	13.8	-2.6	-3.0	13.7	6.9	9.5
id02	1.4	1.6	0.7	-3.3	-1.9	11.3	0.6	-10.0	14.7	0.0	0.0
id03	-20.7	1.5	9.1	25.7	-26.2	16.8	-19.7	2.2	4.0	14.0	-7.2
jp01	1.3	-1.1	8.1	-2.3	-5.6	-2.8	-1.7	-3.0	-4.9	-5.9	4.8
jp02	4.1	-5.8	2.0	-4.8	-3.7	-2.0	-1.7	-8.0	-1.0	-1.0	57.1
la01	-4.5	-13.1	12.8	-2.0	14.8	-7.2	34.6	26.0	224.5	40.6	33.3
mn01	-1.0	-2.9	-6.0	-5.7	-3.7	-5.5	-6.3	-7.0	1.0	-1.0	19.0
my01	-6.3	-1.7	8.1	-4.6	-17.8	-3.6	1.0	-1.3	-8.1	-6.7	-18.1
my02	-3.9	104.5	12.1								
ph01	0.5	-1.3	-5.1	10.5	-54.9	-0.9	-3.2	0.7	-19.6	-7.3	-31.7
ph02	6.6	-2.1	27.5	8.3	24.6	-9.5	-2.6	-1.5	4.4	-0.7	23.5
ru01	1.6	-2.7	-7.8	-3.1	-1.2	0.2	-2.0	-2.0	2.3	-1.0	-3.2
ru02	2.0	1.3	-0.7	17.1	1.9	0.8	6.6	4.0	23.5	-10.9	23.8
th01	-0.7	-2.8	2.5	0.3	-7.4	-1.4	3.7	0.0	17.6	8.9	-4.8
th02	4.9	-2.9	10.1	-5.0	-9.3	-12.2	-1.7	-8.0	27.5	11.9	-9.5
vn01	0.7	-5.8	0.7	-10.5	-6.2	-6.5	6.6	-2.3	15.4	-34.7	-28.6
vn02	1.0	-4.2	-2.7	-1.3	-8.0	-6.8	-4.5	-28.0	14.4	-35.0	77.8
vn03	0.2	-2.9	5.6			21.0	0.0	-1.2	-26.7	-4.6	76.2
Minimum	-20.7	-13.1	-7.8	-10.5	-54.9	-12.2	-19.7	-28.0	-26.7	-35.0	-31.7
Maximum	6.6	104.5	27.5	25.7	24.6	21.0	34.6	26.0	224.5	40.6	77.8
Average	-0.3	2.4	3.4	1.1	-4.7	-0.1	0.2	-2.0	17.7	0.6	11.2

blank : not analyzed

Appendix 5.4 Z-score evaluation

The NC applied Z-score for further statistical evaluation of the analytical values in the inter-laboratory comparison on inland aquatic environment.

- **Definition of Z-score**

Z-score is one of the statistical measures that quantify the distance from the mean of a data set. The formula for the calculation of Z-score (Robust method) was shown below:

$$Z = \frac{X - Q_2}{0.7413 \times (Q_3 - Q_1)}$$

where X: Measurement values of samples
Q_1: The 1st quartile value of entire data
Q_2: The 2nd quartile value of entire data (i.e. Median)
Q_3: The 3rd quartile value of entire data

- **Evaluation of calculated Z-score**

Z-score was given to each data submitted by the participating laboratories, and was evaluated as follows:

$|Z| \leq 2$: Satisfactory
$2 < |Z| < 3$: Questionable
$3 \leq |Z|$: Unsatisfactory

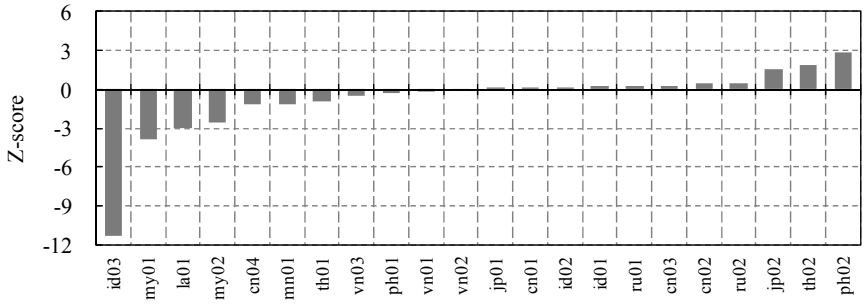
The calculated Z-scores were shown in Appendix Table 5.4.1 and 5.4.2, and were also graphed in Appendix Figure 5.4.1 through 5.4.11.

Appendix Table 5.4.1 Results of Z-score evaluation for sample No. 091i

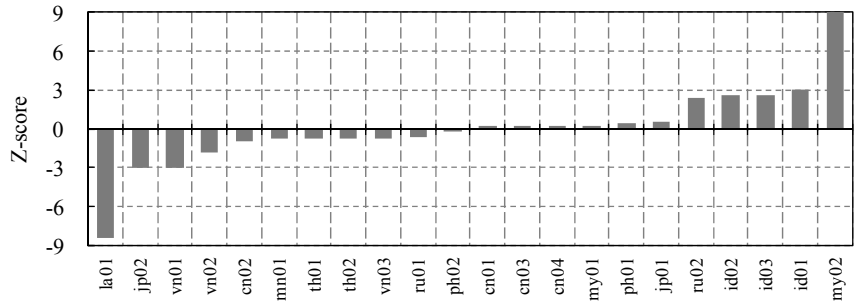
Sample No. 091i

Lab. ID	pH	EC	Alkalinity	SO_4^{2-}	NO_3^-	Cl^-	Na^+	K^+	Ca^{2+}	Mg^{2+}	NH_4^+
cn01	0.15	0.20	0.00	0.77	0.00	0.26	0.00	0.00	0.24	0.81	0.22
cn02	0.44	-1.00	-1.35	-0.18	0.27	-0.13	0.49	0.67	0.56	1.08	-0.22
cn03	0.30	0.20	0.00	0.62	0.81	0.00	0.61	1.35	-0.08	0.90	0.00
cn04	-1.11	0.20	-1.35	-0.11	1.35	-0.06	0.00	-0.67	0.40	1.17	-0.22
id01	0.22	3.00	1.35	1.79	1.89	3.73	-0.49	-0.67	0.00	0.72	0.22
id02	0.15	2.60	0.00	-0.26	0.27	3.15	0.86	-3.04	0.08	0.09	-0.22
id03	-11.31	2.60	1.35	6.09	-3.24	4.43	-7.73	1.01	-0.79	1.35	-0.67
jp01	0.07	0.60	1.35	-0.04	-0.27	-0.13	-0.12	-0.67	-1.51	-0.45	0.00
jp02	1.55	-3.00	0.00	-0.55	0.00	0.06	-0.12	-2.36	-1.19	0.00	2.47
la01	-2.96	-8.39	2.70	0.04	2.70	-1.16	15.33	9.11	17.06	3.78	1.35
mn01	-1.11	-0.80	-1.35	-0.77	0.00	-0.77	-2.08	-2.02	-1.03	0.00	0.67
my01	-3.84	0.20	1.35	-0.55	-2.16	-0.32	0.98	0.00	-1.75	-0.54	-1.12
my02	-2.59	79.74	2.70	---	---	---	---	---	---	---	---
ph01	-0.30	0.40	-1.35	2.73	-7.55	0.32	-0.74	0.67	-2.70	-0.54	-1.80
ph02	2.81	-0.20	5.40	2.30	4.05	-1.67	-0.49	0.00	-0.71	0.00	0.90
ru01	0.22	-0.60	-1.35	-0.18	0.27	0.58	-0.25	-0.34	-0.95	0.00	-0.45
ru02	0.44	2.40	0.00	4.19	0.81	0.71	3.43	1.69	0.79	-0.90	0.90
th01	-0.96	-0.80	0.00	0.55	-0.54	0.19	2.21	0.34	0.32	0.90	-0.45
th02	1.92	-0.80	1.35	-0.62	-0.81	-2.31	-0.12	-2.36	1.11	1.17	-0.67
vn01	-0.22	-3.00	0.00	-1.79	-0.27	-1.03	3.43	-0.34	0.16	-3.06	-1.57
vn02	-0.07	-1.80	0.00	0.18	-0.54	-1.09	-1.35	-9.11	0.08	-3.06	3.37
vn03	-0.52	-0.80	1.35	---	---	5.40	0.61	0.00	-3.25	-0.36	3.37
Number of data	22	22	22	20	20	21	21	21	21	21	21
$\|Z\| \leq 2$	17	14	19	16	15	16	15	15	18	18	18
$2 < \|Z\| < 3$	3	6	2	2	2	1	2	3	1	0	1
$3 \leq \|Z\|$	2	2	1	2	3	4	4	3	2	3	2

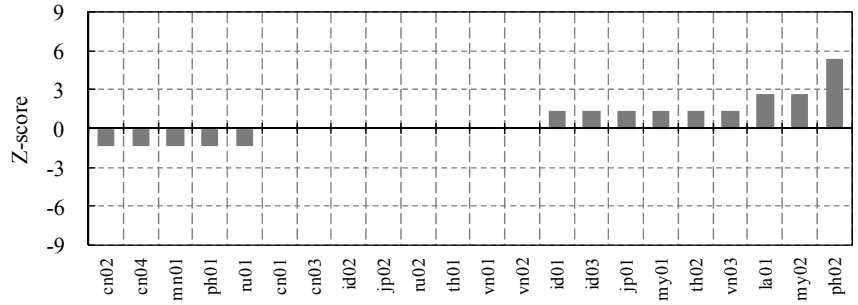
Note: Light mesh, $|Z| > 2$ (Questionable); Dark mesh, $|Z| \geq 3$ (Unsatisfactory); "---", Not measured



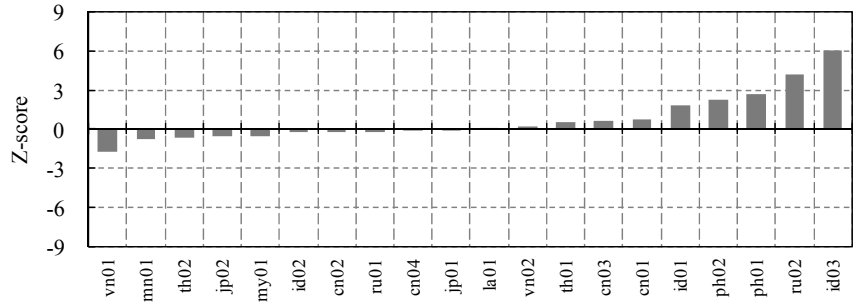
Appendix Figure 5.4.1 Distribution of Z-score for pH



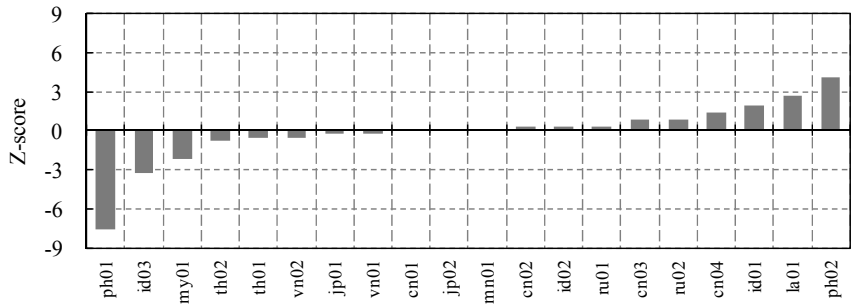
Appendix Figure 5.4.2 Distribution of Z-score for EC



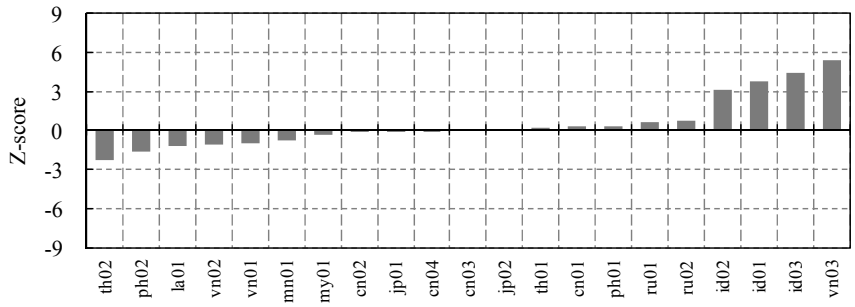
Appendix Figure 5.4.3 Distribution of Z-score for Alkalinity



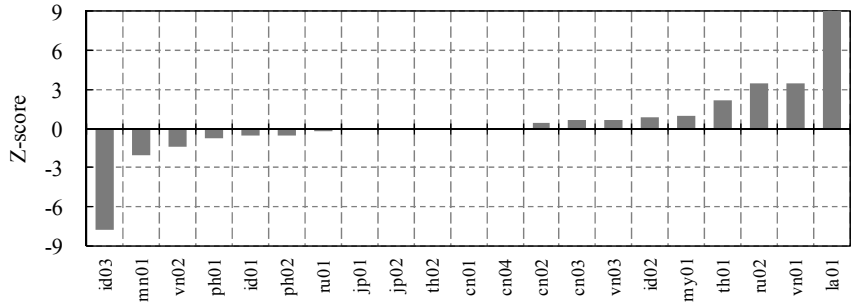
Appendix Figure 5.4.4 Distribution of Z-score for SO_4^{2-}



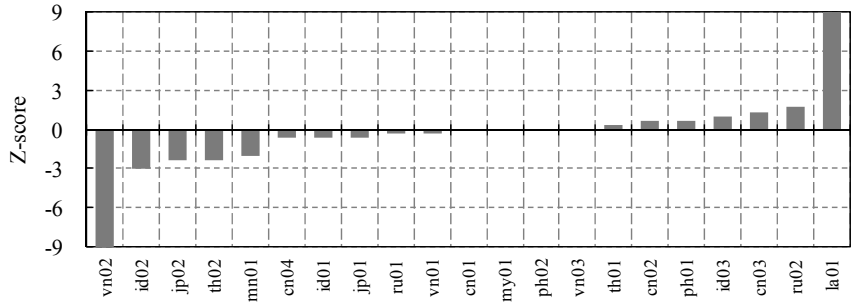
Appendix Figure 5.4.5 Distribution of Z-score for NO_3^-



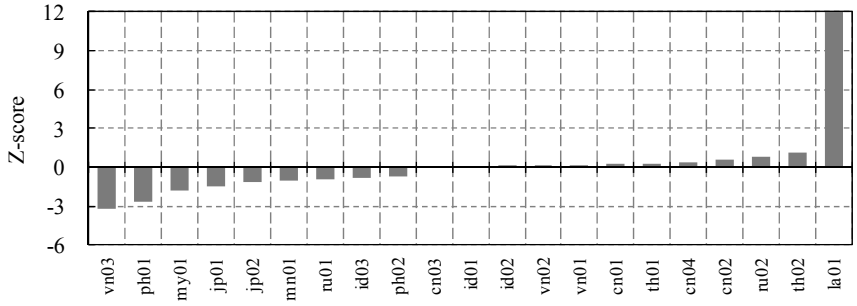
Appendix Figure 5.4.6 Distribution of Z-score for Cl^-



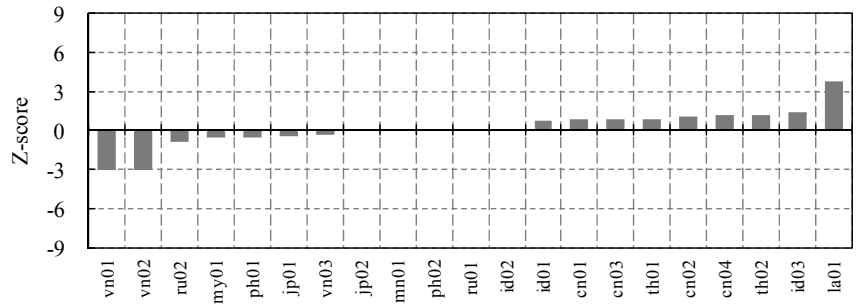
Appendix Figure 5.4.7 Distribution of Z-score for Na^+



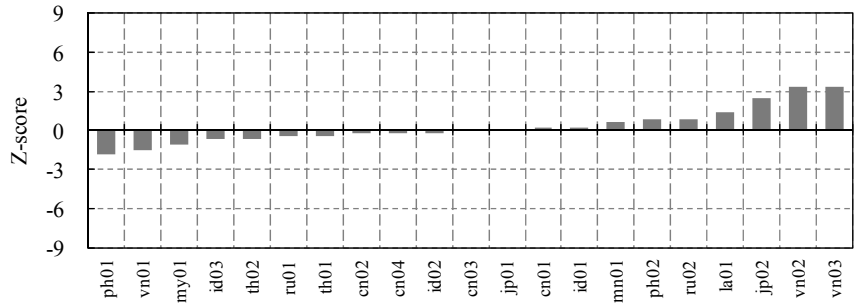
Appendix Figure 5.4.8 Distribution of Z-score for K^+



Appendix Figure 5.4.9 Distribution of Z-score for Ca^{2+}



Appendix Figure 5.4.10 Distribution of Z-score for Mg^{2+}



Appendix Figure 5.4.11 Distribution of Z-score for NH_4^+

6. ACKKNOWLEDGEMENT

ACAP wishes to thank Yamaguchi Prefecture for their cooperation in the collection of soil samples used for the soil inter-laboratory comparison project.

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