Report of the Inter-laboratory Comparison Project 2005 on Soil

7th Attempt

October 2006

Acid Deposition and Oxidant Research Center

Contents

1.   INTRODUCTION

Since 1998, according to the QA/QC programs in EANET, the National Centers and the (Interim) Network Center ((I) NC) have carried out various QA/QC activities for the EANET monitoring. The Inter-laboratory comparison project on soil sample analysis started in 1999 as one of the activities within the QA/QC programs.

The purposes of this project are, through the evaluation of analytical results by statistical analyses, analytical equipments, operating condition of equipments and other practical problems,

(1)   To recognize the analytical precision and accuracy with equipment analysis and titration methods of each participating laboratory, within-laboratory precision, inter-laboratory precision,

(2)   To give an opportunity to improve the quality of the analysis on soil monitoring of EANET, and

(3)   To improve reliability of analytical data through the assessment of suitable analytical methods and techniques.
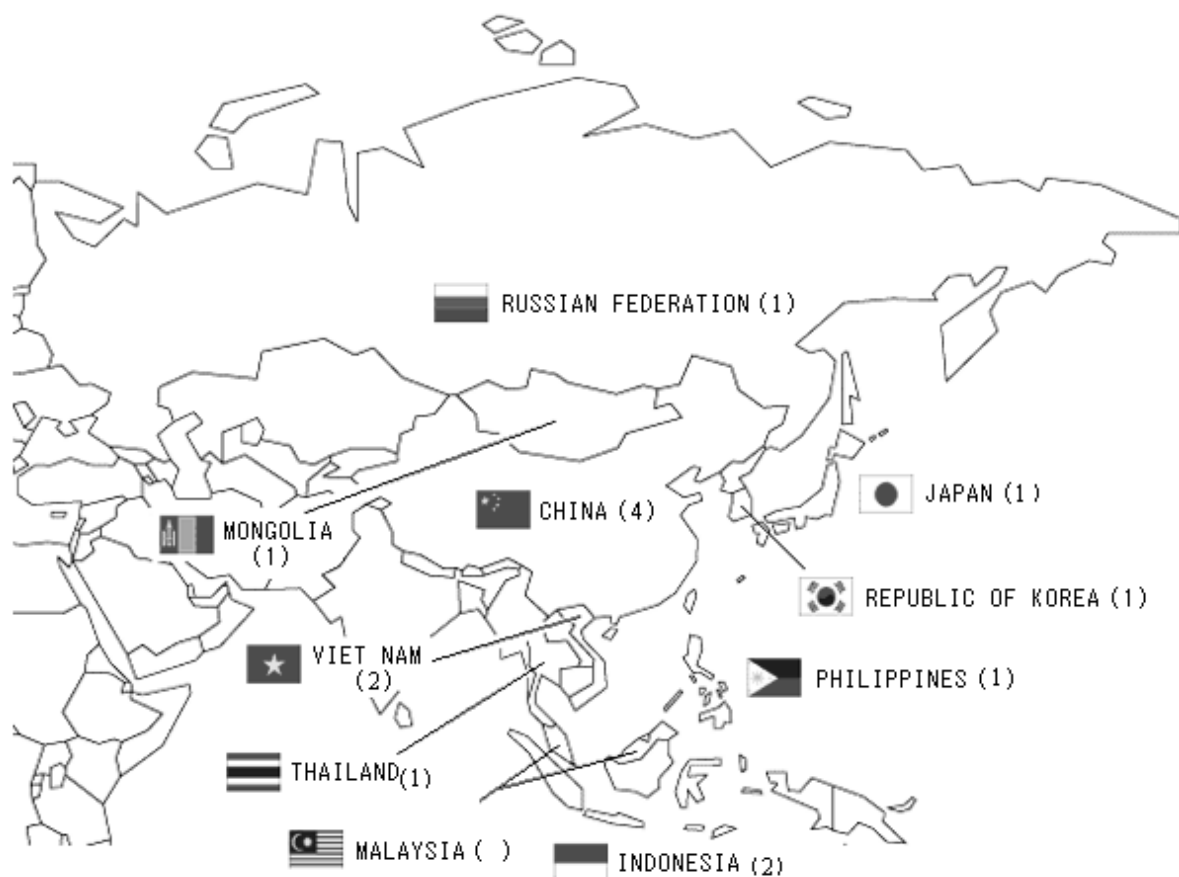
Figure 1. Laboratories participated in Inter-laboratory comparison project 2005 on soil
Number of parenthesis describes the number of laboratories of each EANET country.

1.1 Outlines of the previous projects

Steps in the procedures of soil analysis might 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

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

Moreover, calculation mistakes in the reporting process were suggested in the 4th project, and therefore, NC provided a digital format for calculation/reporting of the data in order to avoid easy mistake. By using digital formats in the 5th project, no obvious calculation mistake was found in the data. The inter-laboratories precisions were still relatively large but clearly improved compared with the previous projects. However, in the 6th project, precisions decreased probably due to level of the concentrations. Further investigation should be considered taking concentrations of the samples into account.

1.2 Objective of the 7th project

In the 7th project, NC provided two soil samples (No.051 and 052) to laboratories to improve the inter-laboratory precision further more by standardization of the methods. Fourteen laboratories of 9 countries participated in the 7th project. Countries and number of laboratories, which participated in the 7th project, are shown in the Figure 1. Names of the participating laboratories are listed in Appendix 1.

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 variability in soil monitoring, and provide useful information to improve precision of soil analysis on EANET.

2. PROCEDURE

2.1. Dispatched Soil Samples

The characteristics of the samples were as follows:

Sample No. 051: collected in the secondary forest of *Quercus serrata*
Sample No. 052: collected in the secondary forest of *Pinus densiflora*

Soil samples were collected in forest area of Morioka City, Iwate Prefecture. Soil types of these samples might be Cambisols (brown forest soil).

Soil was collected from B-horizon in the forest floor. The soils was 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.

2.2. Parameters

All the participating laboratories were expected to measure all the parameters (Table 1).

Table 1. Parameters to be measured

Parameters	Unit	No. 051 and 052
a) Moisture Content	wt %	M
b) pH (H_2O)		M
c) pH (KCl)		M
d) Exchangeable Ca	$cmol(+)\ kg^{-1}$	M
e) Exchangeable Mg	$cmol(+)\ kg^{-1}$	M
f) Exchangeable K	$cmol(+)\ kg^{-1}$	M
g) Exchangeable Na	$cmol(+)\ kg^{-1}$	M
h) Exchangeable Acidity	$cmol(+)\ kg^{-1}$	M
i) Exchangeable Al	$cmol(+)\ kg^{-1}$	M
j) Exchangeable H	$cmol(+)\ kg^{-1}$	M

M: Mandatory items

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

2.3. Procedures for chemical analysis

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 except moisture content 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.

Moisture content was analyzed with three replicates, and the average is used for calculation of all the parameters.

2.3.1. 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 6th 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$_3$COONH$_4$ (pH 7.0)** for analysis of Ex-Ca, Mg, K and Na. Then, 1M CH$_3$COONH$_4$ (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.)

2.3.2. Procedures for Ex-base cations

(1) Extract from air-dry sample with 1M CH$_3$COONH$_4$ (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$_3$COONH$_4$ (pH 7.0). This solution is named " Prepared sample".

(3) Prepare three "prepared samples".

(4) Prepare each standard solution with diluting 1M CH$_3$COONH$_4$ (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(+)/kg soil) = [A * B * V * mcf]/[10 * 20.04 * S]

Ex-Mg (cmol(+)/kg soil) = [A * B * V * mcf]/[10 * 12.15 * S]

Ex-K (cmol(+)/kg soil) = [A * B * V* mcf]/[10 * 39.10 * S]

Ex-Na (cmol(+)/kg 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)

2.3.3.   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:

$$Ex\text{-}Acidity \ (cmol \ (+)/kg) = [(A_{NaOH} - bl_{NaOH}) * M_{NaOH} * c * 100 * mcf] / S$$

$$Ex\text{-}Al \ (cmol \ (+)/kg) = [(A_{HCl} - bl_{HCl}) * M_{HCl} * c * 100 * mcf] / S$$

$$Ex\text{-}H \ (cmol \ (+)/kg) = [(A_{NaOH} - bl_{NaOH}) * M_{NaOH} - (A_{HCl} - bl_{HCl}) * M_{HCl}] * c * 100 * mcf] / S$$

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.)

2.3.4.   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.

2.4. Statistical analysis

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 two decimal places 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.

2) Analysis of variance and estimation of precision

Total variation among laboratories includes within-laboratory and inter-laboratories 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.

3. RESULTS

3.1. Outline of the results

Basic statistics calculated from the laboratory averages are presented in Table 2 for the respective parameters, and especially coefficients of variation (CVs) among laboratories were shown in Figure 2. For both entire (non-verified) data and verified data, the statistics were calculated. Outliers detected by Cochran-Grubbs methods were removed for the verified data.

As for the entire data, the variations (CVs) among the participating laboratories were different in parameter. CVs were very small, 1.4 – 2.4%, in $pH(H_2O)$ and $pH(KCl)$ probably due to their simple analytical procedure and logarithmic values as discussed in the previous reports (ADORC, 2001a; 2000b). However, remarkably large CVs (larger than 100%) were observed in Ex-base cations in No. 051 because of significantly large value (more than ten times larger) by one laboratory. On the other hand, variations in Ex-acidity, Al, and H were remarkably large in No.052 also because of the data of one laboratory.

As for the verified data, the variations (CVs) of $pH(H_2O)$ and $pH(KCl)$ were smaller than 1.5%. CVs for other parameters were improved after removing outliers. CVs of Ex-acidity and Al in No.051 were smaller than 20%, however, the CVs in No.052 were relatively high because of low concentrations. Difference of concentrations between samples may affect the results.

The averages of triplicate analyses (three-time analysis in repeatability condition) and the average of repeat analyses (in within-laboratory-reproducibility condition) in each laboratory were shown in Figure 3.1, 3.2, 3.3 and 3.4. Error bar shows standard deviation of triplicate analyses but it cannot be found in most figures due to its small length. This indicates that triplicate analyses were carried out with high precision under the repeatability condition. Averages of triplicate samples for the respective laboratories were of similar values, and the repeat analyses might also be carried out with high precision under the within-laboratory-reproducibility condition.

Table 2. Basic statistics of the eintire data and the verified data

(Entire data)

Statistics	pH(H$_2$O)	pH(KCl)	Ex-Ca	Ex-Mg	Ex-K	Ex-Na	Ex-acidity	Ex-Al	Ex-H
			cmol(+) kg^{-1}						
No. 051									
Number of Laboratories	14	14	12	12	12	12	14	14	14
Total average	5.1	4.4	0.70	0.43	0.18	0.21	1.68	1.40	0.35
Median	5.1	4.4	0.21	0.15	0.11	0.06	1.57	1.22	0.37
Maximum	5.5	4.5	5.07	2.52	0.66	1.81	3.47	3.34	0.64
Minimum	5.0	4.1	0.10	0.12	0.06	0.04	1.20	0.90	0.12
Standard deviation	0.1	0.1	1.41	0.69	0.20	0.50	0.55	0.60	0.17
CV (%)[*1]	2.4	2.3	202.0	160.0	111	240	32.5	43.1	49
No. 052									
Number of Laboratories	14	14	12	12	12	11	13	14	14
Total average	5.7	5.1	3.85	0.86	0.31	0.31	1.96	1.56	0.33
Median	5.7	5.1	3.45	0.74	0.17	0.08	0.16	0.02	0.16
Maximum	5.8	5.2	5.75	1.62	1.20	2.44	23.00	20.26	2.74
Minimum	5.5	4.9	2.68	0.48	0.13	0.06	0.11	0.00	0.00
Standard deviation	0.1	0.1	1.08	0.37	0.35	0.71	6.32	5.39	0.70
CV (%)[*1]	1.4	1.5	28.1	42.9	112	232	322.2	345.5	212

(Verified data)[*2]

Statistics	pH(H$_2$O)	pH(KCl)	Ex-Ca	Ex-Mg	Ex-K	Ex-Na	Ex-acidity	Ex-Al	Ex-H
			cmol(+) kg^{-1}						
No. 051									
Number of Laboratories	12	11	10	7	10	10	12	13	13
Total average	5.1	4.4	0.21	0.13	0.10	0.06	1.54	1.25	0.34
Median	5.1	4.4	0.20	0.13	0.11	0.05	1.55	1.15	0.30
Maximum	5.2	4.5	0.38	0.15	0.11	0.08	1.89	1.77	0.64
Minimum	5.0	4.3	0.10	0.12	0.06	0.04	1.20	0.90	0.12
Standard deviation	0.1	0.1	0.09	0.01	0.02	0.01	0.19	0.24	0.17
CV (%)[*1]	1.3	1.5	44.1	7.2	21.2	21.0	12.7	19.4	51.3
No. 052									
Number of Laboratories	12	12	11	12	10	10	12	9	12
Total average	5.7	5.1	3.96	0.86	0.16	0.08	0.18	0.01	0.16
Median	5.7	5.1	3.57	0.74	0.16	0.08	0.16	0.00	0.16
Maximum	5.8	5.2	5.75	1.62	0.19	0.10	0.36	0.04	0.23
Minimum	5.6	5.0	2.84	0.48	0.13	0.06	0.11	0.00	0.11
Standard deviation	0.1	0.1	1.07	0.37	0.02	0.02	0.08	0.02	0.04
CV (%)[*1]	0.9	1.0	26.9	42.9	13.5	22.1	44.2	172.9	24.1

Note: *1. CV, Coefficient of variance (%) = (standard deviation/average)*100　　*2.Outliers judged by Cochran-Grubbs methods and calculation mistakes were removed.
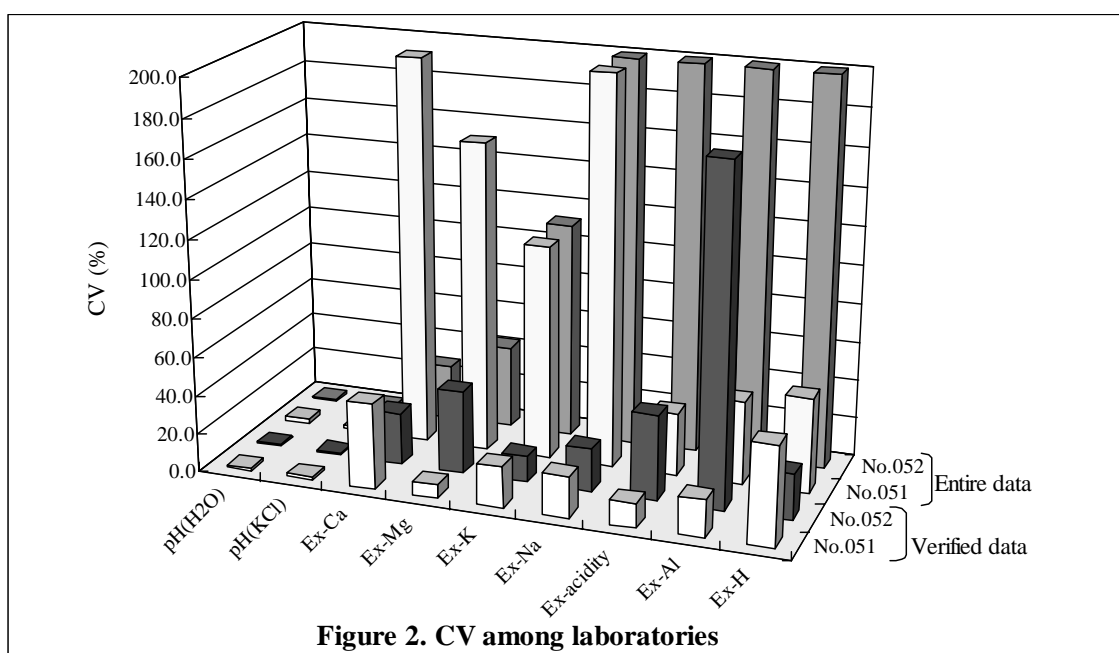
Figure 2. CV among laboratories
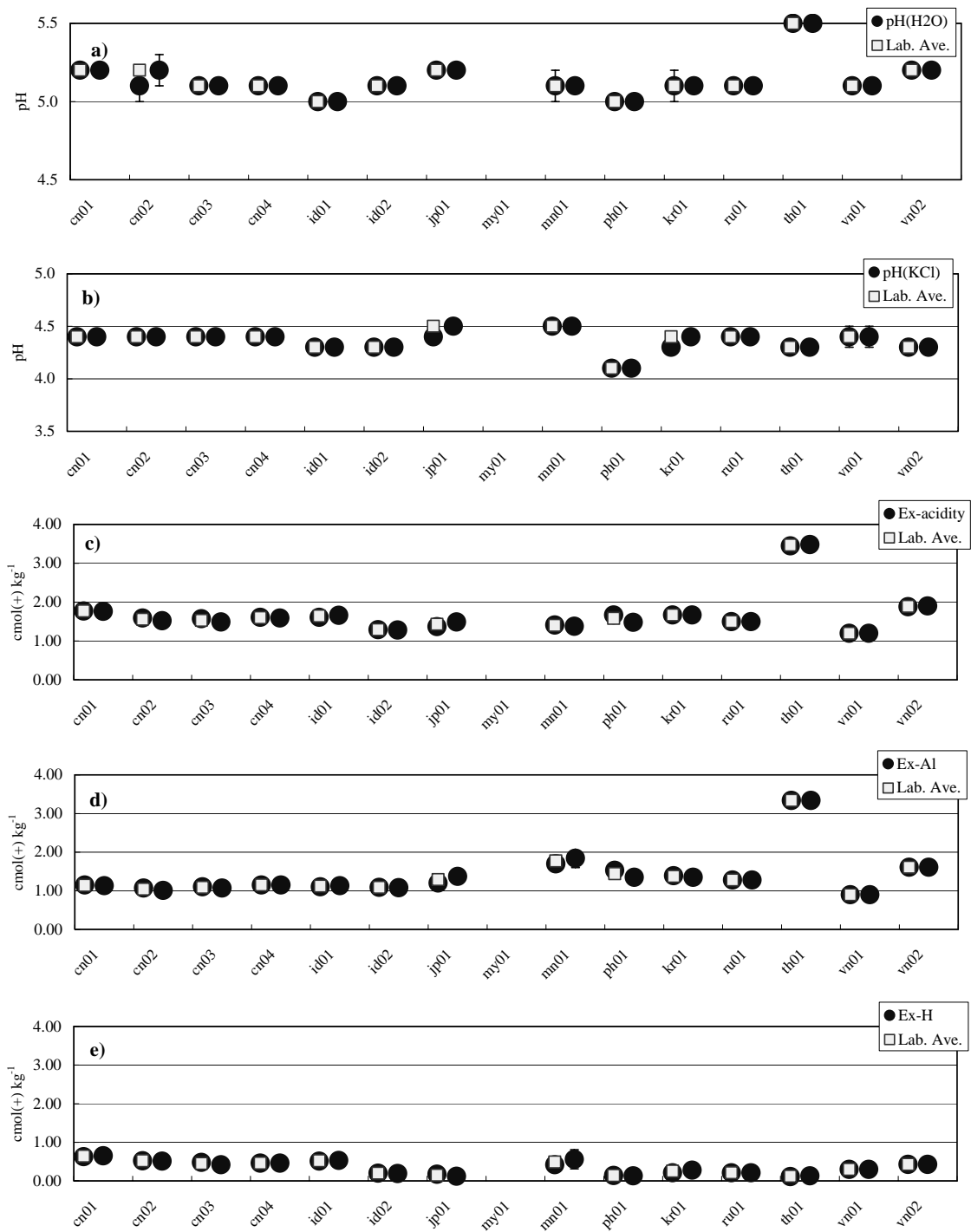
Figure 3.1. Averages of triplicate analysis and the laboratory average for a) pH(H₂O), b) pH(KCl), c) Ex-acidity, d) Ex-Al and e) Ex-H in Sample No. 051. Error bar shows standard deviation of triplicate analysis.
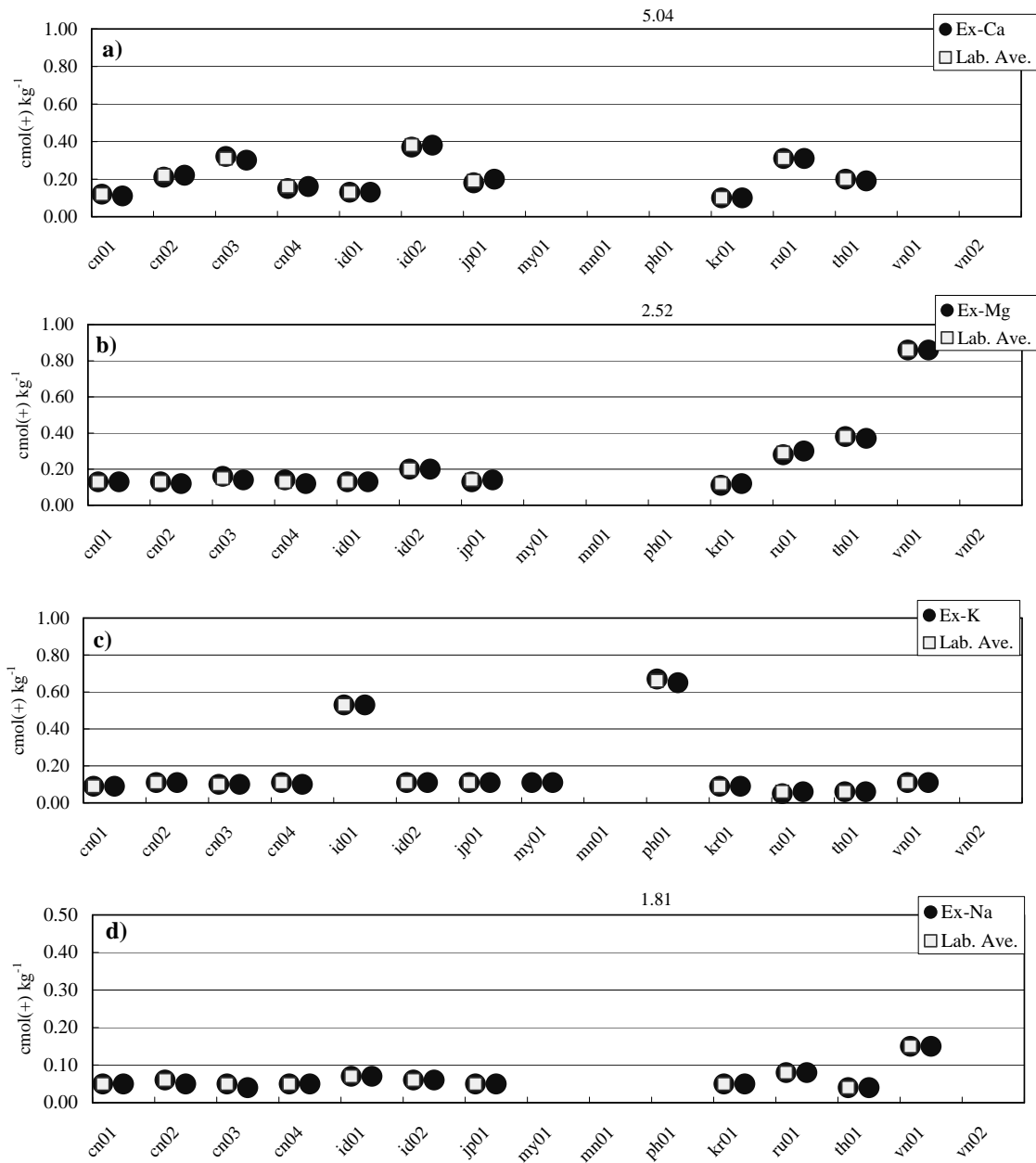
Figure 3.2. Averages of triplicate analysis and the laboratory average for a) Ex-Ca, b) Ex-Mg, c) Ex-K and d) Ex-Na in Sample No. 051. Error bar shows standard deviation of triplicate analysis.
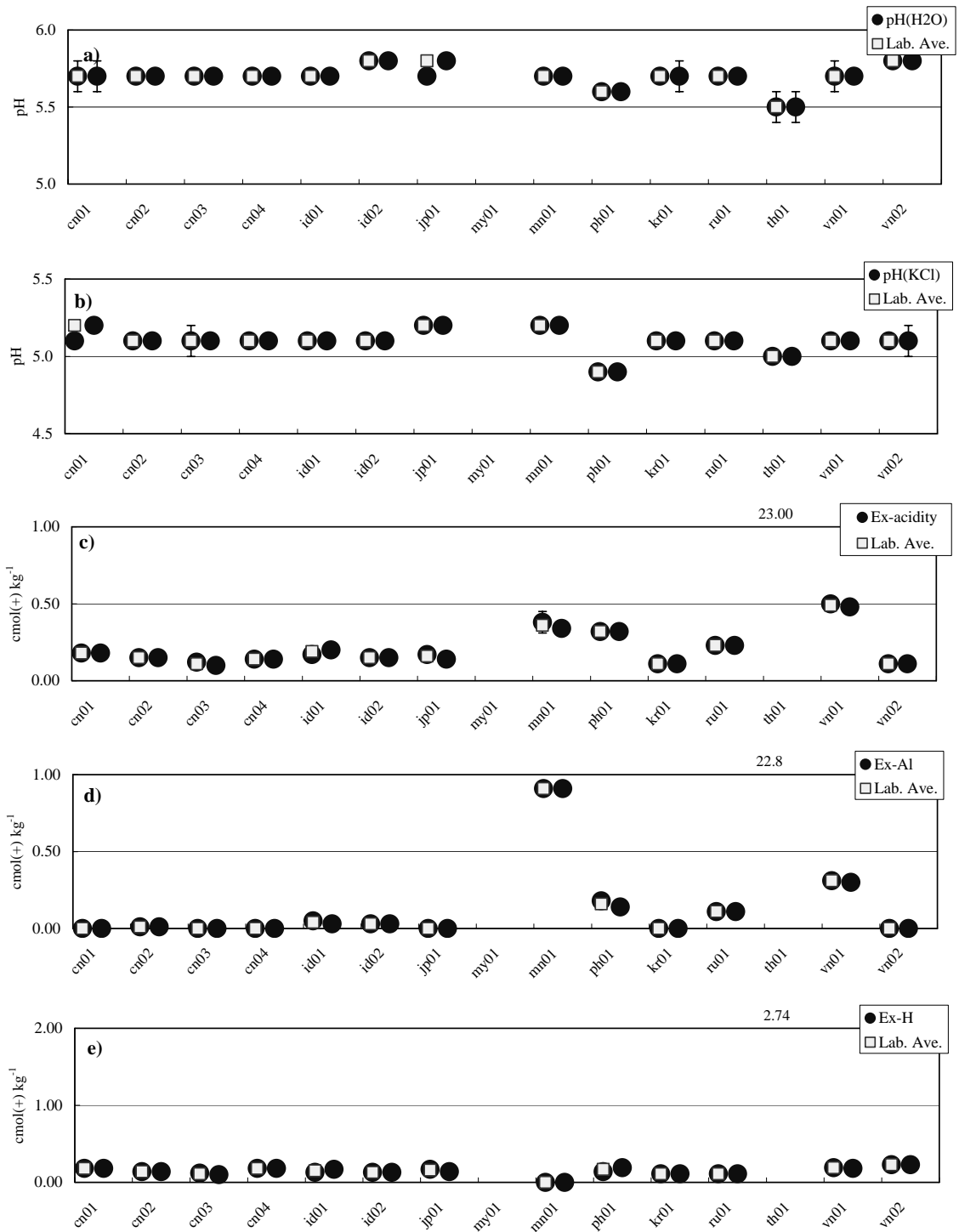
Figure 3.3. Averages of triplicate analysis and the laboratory average for a) pH(H₂O), b) pH(KCl), c) Ex-acidity, d) Ex-Al and e) Ex-H in Sample No. 052. Error bar shows standard deviation of triplicate analysis.
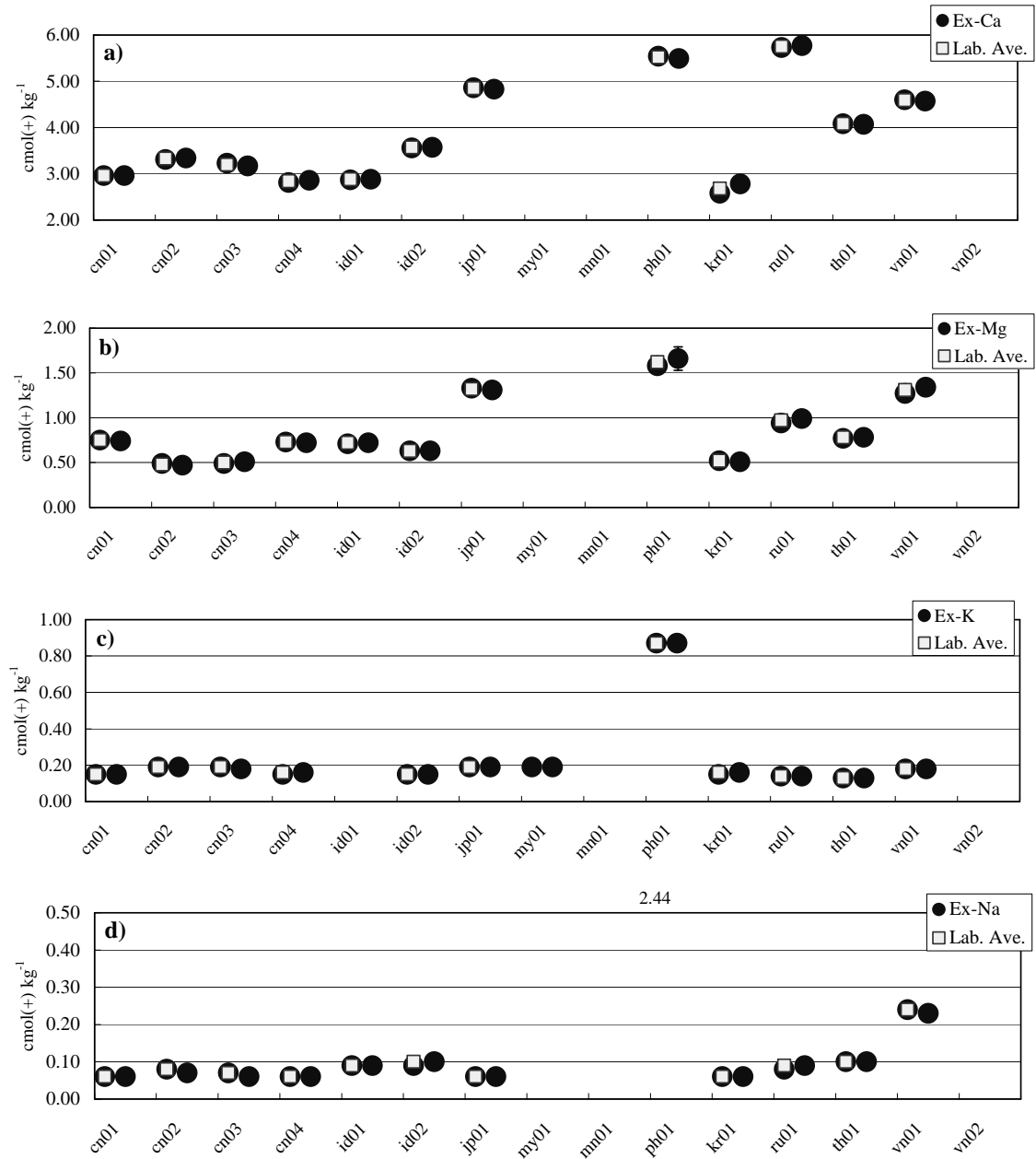
Figure 3.4. Averages of triplicate analysis and the laboratory average for a) Ex-Ca, b) Ex-Mg, c) Ex-K and d) Ex-Na in Sample No. 052. Error bar shows standard deviation of triplicate analysis.

3.2. Verification of data

3.2.1. Detection of outliers

The results of verification by Cochran-Grubbs methods were presented in Table 3.1 and 3.2.

Laboratories, which have large difference in repeat analyses, were judged as outliers by Cochran method (examination of the evenness of within-laboratory precision): e.g. "ph01" in Ex-Ca of No.051, "id01" in Ex-K and "kr01" in Ex-Ca of No.052, etc. Then, the rest of data were tested, and laboratories, which have remarkably large or small average, were judged as outliers by Grubbs method (examination of the average value of each laboratory): e.g. "id01" in Ex-K of No.051, "id02", "ph01", "ru01", "th01", and "vn01" in Ex-Ca of No.051, etc. No outlier was detected in Ex-Mg of No. 052. Two to four outliers were detected in most parameters. More than three outliers were observed in three laboratories, namely "ph01", "th01", and "vn01".

3.2.2. Moisture content and moisture correction factor

Measured moisture contents of the samples and measurement conditions were shown in Table 4. Sample No. 052 had approximately two times larger moisture content than No. 051.

Oven-drying period was recommended to be more than 12 hours based on the discussion in the Third Session of Scientific Advisory Committee (SAC3) in 2003. Almost all laboratories followed the recommendation.

3.2.3. Analytical condition

1) Number of analysts and their experience

Number of analysts and years of their experience were shown in Table 5.1. As for the number of analyst, it seemed that different analysts carried out the repeat analyses in "cn04" for all analysis and "vn02" for Ex-acidity. Different analysts operated AAS for Ca/Mg and FEP for K/Na respectively in "cn02", "cn03", "cn04", "id02", and "ph01". No relationship between the number of analyst and the outliers was suggested.

As for years of experience on soil analysis, clear relationship between the experience and the outliers was not 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 5.2. All the laboratories except "vn01" used AAS for measurement of Ex-Ca and Mg, and four laboratories used FEP for Ex-K and Na. Years in use of instruments were varied from 0 to 25 in AAS. All the laboratories except "cn01" and "ru01" applied Sr or La for measurement of Ex-Ca and Mg by AAS. Two laboratories, "mn01" and "vn02" did not analyze Ex-base cations.

As for procedures for extraction of Ex-base cations, six laboratories used percolation tube procedures, three laboratories used centrifuge procedures, two laboratories used Buchner funnel procedures, and

one laboratory used automatic extractor procedures. No clear difference was observed among data by different procedures. As for size of burette for titration of Ex-acidity, capacities were varied from 5 to 50 ml, but minimum graduates were 0.05 or 0.1 ml in most laboratories.

3) Date of analysis

Date of analysis in the respective laboratories and days used for the analysis were shown in Table 5.3. 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 35 days. NC 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. More than half of laboratories followed the recommendation.

Table 3.1. Data verified by Cochran-Grubbs methods: No. 051

Lab.	Repeat analysis	pH(H₂O)	pH(KCl)	Ex-Ca	Ex-Mg	Ex-K	Ex-Na	Ex-acidity	Ex-Al	Ex-H
							cmol(+) kg^{-1}			
cn01	1st	5.2	4.4	0.12	0.13	0.09	0.05	1.77	1.15	0.63
	2nd	5.2	4.4	0.11	0.13	0.09	0.05	1.76	1.13	0.65
cn02	1st	5.1c	4.4	0.21	0.13	0.11	0.06	1.59	1.07	0.52
	2nd	5.2c	4.4	0.22	0.12	0.11	0.05	1.52	1.01	0.51
cn03	1st	5.1	4.4	0.32	0.16	0.10	0.05	1.57	1.10	0.48
	2nd	5.1	4.4	0.30	0.14	0.10	0.04	1.49	1.07	0.42
cn04	1st	5.1	4.4	0.15	0.14	0.11	0.05	1.61	1.15	0.46
	2nd	5.1	4.4	0.16	0.12	0.10	0.05	1.59	1.15	0.46
id01	1st	5.0	4.3	0.13	0.13	0.53g	0.07	1.61	1.10	0.51
	2nd	5.0	4.3	0.13	0.13	0.53g	0.07	1.66	1.13	0.53
id02	1st	5.1	4.3	0.37	0.20g	0.11	0.06	1.29	1.09	0.20
	2nd	5.1	4.3	0.38	0.20g	0.11	0.06	1.28	1.08	0.19
jp01	1st	5.2	4.4c	0.18	0.13	0.11	0.05	1.37	1.20	0.17
	2nd	5.2	4.5c	0.20	0.14	0.11	0.05	1.49	1.37	0.12
my01	1st									
	2nd									
mn01	1st	5.1	4.5					1.41	1.70	0.42c
	2nd	5.1	4.5					1.38	1.84	0.56c
ph01	1st	5.0	4.1g	4.94c	2.52g	0.67c	1.85c	1.67c	1.53	0.14
	2nd	5.0	4.1g	5.20c	2.52g	0.65c	1.77c	1.48c	1.35	0.13
kr01	1st	5.1	4.3c	0.10	0.11	0.09	0.05	1.67	1.39	0.21
	2nd	5.1	4.4c	0.10	0.12	0.09	0.05	1.67	1.35	0.28
ru01	1st	5.1	4.4	0.31	0.28g	0.05	0.08	1.50	1.28	0.21
	2nd	5.1	4.4	0.31	0.30g	0.06	0.08	1.50	1.28	0.21
th01	1st	5.5g	4.3	0.20	0.38g	0.06	0.04	3.45g	3.34g	0.11
	2nd	5.5g	4.3	0.19	0.37g	0.06	0.04	3.48g	3.34g	0.13
vn01	1st	5.1	4.4	1.16g	0.86g	0.11	0.15g	1.20	0.90	0.30
	2nd	5.1	4.4	1.18g	0.86g	0.11	0.15g	1.20	0.90	0.30
vn02	1st	5.2	4.3					1.88	1.61	0.43
	2nd	5.2	4.3					1.90	1.61	0.43

Note: The outliers judged by Cochran and Grubbs methods were marked with asterisk c and g, respectively.

Table 3.2. Data verified by Cochran-Grubbs methods: No. 052

Lab.	Repeat analysis	pH(H₂O)	pH(KCl)	Ex-Ca	Ex-Mg	Ex-K	Ex-Na	Ex-acidity	Ex-Al	Ex-H
							cmol(+) kg⁻¹			
cn01	1st	5.7	5.1c	2.96	0.75	0.15	0.06	0.18	0.00	0.18
	2nd	5.7	5.2c	2.96	0.74	0.15	0.06	0.18	0.00	0.18
cn02 xian	1st	5.7	5.1	3.31	0.49	0.19	0.08	0.15	0.01	0.14
	2nd	5.7	5.1	3.34	0.47	0.19	0.07	0.15	0.01	0.14
cn03 xiamen	1st	5.7	5.1	3.23	0.49	0.19	0.07	0.12	0.00	0.12
	2nd	5.7	5.1	3.17	0.51	0.18	0.06	0.10	0.00	0.10
cn04 zhuhai	1st	5.7	5.1	2.81	0.73	0.15	0.06	0.14	0.00	0.18
	2nd	5.7	5.1	2.86	0.72	0.16	0.06	0.14	0.00	0.18
id01 emc	1st	5.7	5.1	2.87	0.71	1.14c	0.09	0.17	0.05	0.13
	2nd	5.7	5.1	2.88	0.72	1.26c	0.09	0.20	0.03	0.17
id02	1st	5.8	5.1	3.56	0.63	0.15	0.09	0.15	0.03	0.13
	2nd	5.8	5.1	3.57	0.63	0.15	0.10	0.15	0.03	0.13
jp01	1st	5.7c	5.2	4.86	1.33	0.19	0.06	0.17	0.00	0.17
	2nd	5.8c	5.2	4.83	1.31	0.19	0.06	0.14	0.00	0.14
my01	1st									
	2nd									
mn01	1st	5.7	5.2					0.38	0.91g	0.00g
	2nd	5.7	5.2					0.34	0.91g	0.00g
ph01	1st	5.6	4.9g	5.54	1.58	0.87g	2.45g	0.32	0.18c	0.14
	2nd	5.6	4.9g	5.49	1.66	0.87g	2.43g	0.32	0.14c	0.19
kr01	1st	5.7	5.1	2.58c	0.52	0.15	0.06	0.11	0.00	0.11
	2nd	5.7	5.1	2.78c	0.51	0.16	0.06	0.11	0.00	0.11
ru01	1st	5.7	5.1	5.73	0.94	0.14	0.08	0.23	0.11g	0.11
	2nd	5.7	5.1	5.77	0.99	0.14	0.09	0.23	0.11g	0.11
th01	1st	5.5g	5.0	4.08	0.77	0.13	0.10	23.00g	20.25g	2.75g
	2nd	5.5g	5.0	4.07	0.78	0.13	0.10	22.99g	20.27g	2.72g
vn01	1st	5.7	5.1	4.60	1.27	0.18	0.24g	0.50g	0.31g	0.19
	2nd	5.7	5.1	4.57	1.34	0.18	0.23g	0.48g	0.30g	0.18
vn02	1st	5.8	5.1					0.11	0.00	0.23
	2nd	5.8	5.1					0.11	0.00	0.23

Note: The outliers judged by Cochran and Grubbs methods were marked with asterisk c and g, respectively.

Table 4. Measured moisture content, moisture correction factor, and measurement condition

Lab.	No. 051			No. 052			Oven temp.	Drying period
	wt%	mcf	Mean mcf	wt%	mcf	Mean mcf	°C	hours
cn01	7.4	1.07	1.08	14.8	1.15	1.15	105	15
	7.6	1.08		14.9	1.15			
	7.5	1.07		15.1	1.15			
cn02	7.7	1.08	1.08	15.0	1.15	1.15	105	15
	7.8	1.08		15.0	1.15			
	7.7	1.08		15.0	1.15			
cn03	7.4	1.07	1.07	14.4	1.14	1.14	105	24
	7.4	1.07		14.3	1.14			
	7.5	1.08		14.4	1.14			
cn04	8.0	1.08	1.08	15.2	1.15	1.15	105	15
	8.1	1.08		15.3	1.15			
	7.9	1.08		15.3	1.15			
id01	5.8	1.06	1.06	12.4	1.12	1.12	105	22
	5.6	1.06		12.2	1.12			
	5.9	1.06		12.4	1.12			
id02	6.1	1.06	1.06	12.1	1.12	1.12	105	16
	5.9	1.06		12.3	1.12			
	6.2	1.06		12.2	1.12			
jp01	6.7	1.07	1.07	13.7	1.14	1.14	105	24
	6.7	1.07		13.7	1.14			
	6.7	1.07		13.7	1.14			
my01								
mn01	6.5	1.07	1.06	14.5	1.15	1.15	105	18
	6.4	1.06		15.0	1.15			
	6.3	1.06		14.8	1.15			
ph01	4.6	1.05	1.05	10.1	1.10	1.10	105	24
	4.6	1.05		10.0	1.10			
	4.6	1.05		10.0	1.10			
kr01	6.7	1.07	1.07	14.1	1.14	1.14	105	17
	6.6	1.07		13.9	1.14			
	6.8	1.07		14.1	1.14			
ru01	7.1	1.07	1.07	13.6	1.14	1.14	105	12
	6.9	1.07		13.8	1.14			
	6.9	1.07		13.5	1.14			
th01	8.4	1.08	1.08	15.2	1.15	1.15	105	12
	8.4	1.08		15.2	1.15			
	8.4	1.08		15.2	1.15			
vn01	6.65	1.07	1.06	12.9	1.13	1.13	105	18
	6.25	1.06		12.8	1.13			
	6.12	1.06		12.7	1.13			
vn02	7.61	1.08	1.08	13.6	1.14	1.14	105	10
	7.66	1.08		13.7	1.14			
	7.50	1.08		13.8	1.14			

Table 5.1. Number and experience of analyst

| Lab. | Ex-base cations | | | Ex-acidity | | | Analyst |
| | Number of analyst | Years of experience | | Number of | Years of experience | | |
		Chemical	Soil		Chemical	Soil	
cn01	1	21	2	1	21	2	s
cn02	1	10	7	1	10	7	d
cn03	1	8	5	1	15	7	d
cn04	2	4	2	2	5	3	d
id01	1	8	3	1	8	3	s
id02	2[*1]	24/26	24/26	1	10	10	d
jp01	1	7	7	1	7	7	s
my01							
mn01	-	-	-	1	12	12	-
ph01	1	14	14	1	29	29	d
kr01	1	13	11	1	13	11	s
ru01	1	9	9	1	9	9	s
th01	1	4	3	1	4	3	s
vn01	1	12	7	1	12	7	s
vn02	-	-	-	2	7	0	-

Note: *1. Ca/Mg and K/Na were analyzed by different analysts; -, Not measured; s, Same analysts; d, Different analysts

Table 5.2. Analytical instruments and condition of the instruments for exchangeable cations

Lab.	Sample	Ex-Ca Instrument	Years[*1]	Ex-Mg Instrument	Years		Ex-K Instrument	Years	Ex-Na Instrument	Years		Procedures for extraction of Ex-base cations	method	Ex-Acidity, Al and H Size of burette (ml) Capacity	Minimum graduate
cn01	No.051	AAS	0	AAS	0	na	AAS	0	AAS	0	na	Percolation tube	Titration	50	0.1
	No.052	AAS	0	AAS	0	na	AAS	0	AAS	0	na				
cn02	No.051	AAS	16	AAS	16	La	AAS	16	AAS	16	Cs	Automatic extractor	Titration	5	0.00125
	No.052	AAS	16	AAS	16	La	AAS	16	AAS	16	Cs				
cn03	No.051	AAS	5	AAS	5	Sr	AAS	5	AAS	5	Sr	Centrifuge	Titration	25	0.1
	No.052	AAS	5	AAS	5	Sr	AAS	5	AAS	5	Sr				
cn04	No.051	AAS	7	AAS	7	Sr	AAS	7	AAS	7	Cs	Centrifuge	Titration	25	0.1
	No.052	AAS	7	AAS	7	Sr	AAS	7	AAS	7	Cs				
id01	No.051	AAS	12	AAS	12	Sr	AAS	12	AAS	12	Sr	Centrifuge	Titration	25	0.05
	No.052	AAS	12	AAS	12	Sr	AAS	12	AAS	12	Sr				
id02	No.051	AAS	19	AAS	19	(Sr)	FEP	36	FEP	36	(Cs)	Percolation tube	Titration	50	0.01
	No.052	AAS	19	AAS	19	(Sr)	FEP	36	FEP	36	(Cs)				
jp01	No.051	AAS	20	AAS	20	Sr	FEP	20	FEP	20	na	Percolation tube	Titration	25/10	0.1/0.05
	No.052	AAS	20	AAS	20	Sr	FEP	20	FEP	20	na			(NaOH/HCl)	(NaOH/HCl)
my01															
mn01	No.051	-	-	-	-	-	-	-	-	-	-	-	Titration	25	0.1
	No.052	-	-	-	-	-	-	-	-	-	-				
ph01	No.051	AAS	15	AAS	15	Sr	AAS	15	AAS	15	na	Buchner funnel	Titration	50	0.01
	No.052	AAS	15	AAS	15	Sr	AAS	15	AAS	15	na				
kr01	No.051	AAS	5	AAS	5	Sr	AAS	5	AAS	5	Sr	Percolation tube	Titration	25	0.05
	No.052	AAS	5	AAS	5	Sr	AAS	5	AAS	5	Sr			0	0.09
ru01	No.051	AAS	25	AAS	25	na	FEP	25	FEP	25	na	Percolation tube	Titration		
	No.052	AAS	25	AAS	25	na	FEP	25	FEP	25	na			-25	0.13
th01	No.051	AAS	9	AAS	9	(Sr)	AAS	9	AAS	9	(Cs)	Percolation tube	Titration	50	0.05
	No.052	AAS	9	AAS	9	(Sr)	AAS	9	AAS	9	(Cs)				
vn01	No.051	Titration		Titration		na	FEP	7	FEP	7	na	Buchner funnel	Titration	10	0.02
	No.052	Titration		Titration		na	FEP	7	FEP	7	na				
vn02	No.051	-	-	-	-	-	-	-	-	-	-	-	Titration	10	0.05
	No.052	-	-	-	-	-	-	-	-	-	-				

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

Table 5.3. Date of analysis

Lab.	Repeat	pH Date[*1]	pH Analysis[*2]	pH Interval[*3] Days	Ex-Ca and Mg Date[*1]	Ex-Ca and Mg Analysis[*2]	Ex-Ca and Mg Interval[*3] Days	Ex-K and Na Date[*1]	Ex-K and Na Analysis[*2]	Ex-K and Na Interval[*3] Days	Ex-acidity, Al and H Date[*1]	Ex-acidity, Al and H Analysis[*2]	Ex-acidity, Al and H Interval[*3] Days
cn01	1st	14-Feb	1	6	20-Feb	6	5	20-Feb	6	5	21-Feb	2	2
	2nd	20-Feb	1		25-Feb	9		25-Feb	9		23-Feb	2	
cn02	1st	2-Dec	2	35	9-Dec	5	32	9-Dec	5	32	17-Dec	6	31
	2nd	6-Jan	2		10-Jan	5		10-Jan	5		17-Jan	5	
cn03	1st	14-Feb	1	8	25-Jan	2	30	25-Jan	2	30	15-Jan	4	35
	2nd	22-Feb	1		24-Feb	2		24-Feb	2		19-Feb	8	
cn04	1st	9-Feb	1	4	9-Feb	2	5	9-Feb	2	5	16-Feb	2	5
	2nd	13-Feb	1		14-Feb	2		14-Feb	2		21-Feb	2	
id01	1st	8-Dec	1	0	21-Dec	7	1	21-Dec	7	1	16-Dec	11	4
	2nd	8-Dec	1		22-Dec	4		22-Dec	4		20-Dec	2	
id02	1st	27-Dec	1	6	29-Dec	1	4	29-Dec	1	4	27-Dec	1	6
	2nd	2-Jan	1		2-Jan	1		2-Jan	1		2-Jan	1	
jp01	1st	31-Jan	1	14	10-Feb	4	5	10-Feb	4	5	10-Feb	4	5
	2nd	14-Feb	1		15-Feb	2		15-Feb	2		15-Feb	2	
my01	1st			0			0			0			0
	2nd												
mn01	1st	11-Jan	1	0	-	-	-	-	-	-	16-Jan	1	0
	2nd	11-Jan	1		-	-		-	-		16-Jan	1	
ph01	1st	15-Feb	1	7	2-Mar	1	7	2-Mar	1	7	10-Mar	1	7
	2nd	22-Feb	1		9-Mar	1		9-Mar	1		17-Mar	1	
kr01	1st	17-Jan	1	15	20-Jan	2	19	20-Jan	4	19	27-Jan	2	14
	2nd	1-Feb	1		8-Feb	2		8-Feb	2		10-Feb	2	
ru01	1st	10-Feb	1	3	20-Feb	1	3	20-Feb	1	3	16-Feb	2	2
	2nd	13-Feb	1		23-Feb	1		23-Feb	1		18-Feb	2	
th01	1st	9-Mar	1	2	15-Mar	5	0	15-Mar	5	0	28-Mar	2	1
	2nd	11-Mar	1		15-Mar	4		15-Mar	4		29-Mar	2	
vn01	1st	5-Feb	1	5	5-Feb	1	5	5-Feb	1	5	5-Feb	1	5
	2nd	10-Feb	1		10-Feb	1		10-Feb	1		10-Feb	1	
vn02	1st	22-Dec	1	1	-	-	-	-	-	-	19-Dec	1	1
	2nd	23-Dec	1		-	-		-	-		20-Dec	1	

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

3.3. Analysis of variance and estimation of precision

Analysis of variance (ANOVA) for the entire data and verified data were shown in Table 6.1 and 6.2, respectively. "Repeatability-precision", "within-laboratory-precision" and "inter-laboratories-precision" were estimated. In the following section, the results of verified data were mainly discussed (see Table 6.2).

1) Repeatability-precision
Repeatability standard deviations were relatively small for most of the parameters in the verified data, and CVs of most parameters were smaller than 10%. Especially CVs of pH(H_2O) and pH(KCl) in both samples were smaller than 0.5%, and that of Ex-Ca in No.052 was about 1.7%.

It seems that triplicate analyses were carried out under the same condition. Processes on extraction, dilution of the sample, and stability of the instruments may affect the results. The small CVs suggested that 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 8%. Especially CVs of pH(H_2O) and pH(KCl) in both samples were smaller than 0.2%, and CVs of Ex-Ca, Mg, and K in No. 052 were smaller than 3%.

The values were almost same as repeatability-precision. For most parameters, the CVs 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. It was also suggested that the participating laboratories could analyze the parameters with their own standard procedures.

3) Inter-laboratories precision
Inter-laboratories standard deviation was still large. CVs of a few Ex-base cations were larger than 40%, however, CVs of some parameters, namely, Ex-Mg and Ex-acidity of No.051, and Ex-K of No.052 were smaller than 15%.

4) Calculation of permissible tolerance
As for the repeatability limit and within-laboratory reproducibility limit, values may be enough small, and it can be used as a reference value for the repeat analysis on the instrumental analysis in the respective laboratories.

As for the reproducibility limit, inter-laboratories precision should be improved for Ex-base cations, and then the discussion should be carried out.

Table 6.1. Analysis of variance for the entire data

Statistics	No. 051								
	pH(H$_2$O)	pH(KCl)	Ex-Ca	Ex-Mg	Ex-K	Ex-Na	Ex-acidity	Ex-Al	Ex-H
Number of Laboratories	14	14	12	12	12	12	14	14	14
Total sum of square	186538	133810	2506	962	163	225	19867	13860	851
ST/lmd	2221	1593	35	13	2	3	237	165	10
Number of Data	84	84	72	72	72	72	84	84	84
Total sum	431.9	365.8	50.06	31.02	12.78	15.01	140.95	117.73	29.17
Total average	5.1	4.4	0.70	0.43	0.18	0.21	1.68	1.40	0.35
Sum of square inter-laboratories (S_R)	1.1	0.7	130.59	31.51	2.58	16.81	23.13	28.60	2.33
Sum of square within-laboratory (S_{RW})	0.0	0.0	0.10	0.00	0.00	0.01	0.10	0.13	0.05
Sum of square repeatablility (S_r)	0.0	0.0	0.03	0.06	0.00	0.00	0.06	0.26	0.21
Total sum of square (S_T)	1.2	0.7	130.72	31.57	2.58	16.82	23.30	28.99	2.59
Inter-laboratories degree of freedom (ϕ_R)	13	13	11	11	11	11	13	13	13
Within-laboratory degree of freedom (ϕ_{RW})	14	14	12	12	12	12	14	14	14
Repeatability degree of freedom (ϕ_r)	56	56	48	48	48	48	56	56	56
Total degree of freedom (ϕ_T)	83	83	71	71	71	71	83	83	83
Inter-laboratories variance ($V_R = S_R/\phi_R$)	0.09	0.05	11.872	2.864	0.234	1.528	1.779	2.200	0.179
Within-laboratory variance ($V_{RW} = S_{RW}/\phi_{RW}$)	0.00	0.00	0.009	0.000	0.000	0.001	0.007	0.009	0.004
Repeatability variance ($V_r = S_r/\phi_r$)	0.00	0.00	0.001	0.001	0.000	0.000	0.001	0.005	0.004
Laboratory component of variance ($s_b^2 = (V_R - V_{RW})/(2*3)$)	0.01	0.01	1.977	0.477	0.039	0.255	0.295	0.365	0.029
Within-laboratory component of variance ($s_c^2 = (V_{RW} - V_r)/3$)	0.00	0.00	0.003	0.000	0.000	0.000	0.002	0.002	0.000
Repeatability component of variance ($s_r^2 = V_r$)	0.00	0.00	0.001	0.001	0.000	0.000	0.001	0.005	0.004
Inter-laboratories standard deviation ($s_R = SQRT(s_r^2/(2*3) + s_c^2/2 + s_b^2)$)	0.1	0.1	1.41	0.69	0.20	0.50	0.54	0.61	0.17
Within-laboratory standard deviation ($s_{RW} = SQRT(s_r^2/3 + s_c^2)$)	0.0	0.0	0.05	0.01	0.00	0.02	0.05	0.06	0.03
Repeatability standard deviation ($s_r = SQRT(s_r^2)$)	0.0	0.0	0.02	0.04	0.01	0.01	0.03	0.07	0.06
Inter-laboratories precision CV (%)	2.3	2.2	202.31	160.37	111.30	242.07	32.45	43.20	49.73
Within-laboratory precision CV (%)	0.2	0.6	7.73	1.84	2.82	7.70	2.94	4.00	9.98
Repeatability precision CV (%)	0.4	0.4	3.41	8.26	3.45	3.53	1.98	4.88	17.57
Reproducibility limit ($R = D(2, 0.95)*s_R$)	0.34	0.27	3.939	1.935	0.553	1.413	1.525	1.695	0.484
Within-laboratory-reproducibility limit ($R_W = D(2, 0.95)*s_{Rw}$)	0.03	0.07	0.151	0.022	0.014	0.045	0.138	0.157	0.097
Repeatability limit ($r = D(3, 0.95)*s_r$)	0.07	0.05	0.078	0.117	0.020	0.024	0.110	0.226	0.201

Statistics	No. 052								
	pH(H$_2$O)	pH(KCl)	Ex-Ca	Ex-Mg	Ex-K	Ex-Na	Ex-acidity	Ex-Al	Ex-H
Number of Laboratories	14	14	12	12	12	12	14	14	14
Total sum of square	228962	183441	76845	3812	490	418	23621	17145	751
ST/lmd	2726	2184	1067	53	7	6	281	204	9
Number of Data	84	84	72	72	72	72	84	84	84
Total sum	478.5	428.3	277.21	61.74	22.14	20.45	153.69	130.94	27.41
Total average	5.7	5.1	3.85	0.86	0.31	0.28	1.83	1.56	0.33
Sum of square inter-laboratories (S_R)	0.5	0.4	77.34	9.00	7.94	30.58	2896.06	2265.27	37.61
Sum of square within-laboratory (S_{RW})	0.0	0.0	0.08	0.02	0.02	0.00	0.02	0.00	0.01
Sum of square repeatablility (S_r)	0.0	0.0	0.19	0.05	0.00	0.03	0.04	0.00	0.00
Total sum of square (S_T)	0.6	0.5	77.61	9.07	7.97	30.61	2896.12	2265.28	37.62
Inter-laboratories degree of freedom (ϕ_R)	13	13	11	11	11	11	13	13	13
Within-laboratory degree of freedom (ϕ_{RW})	14	14	12	12	12	12	14	14	14
Repeatability degree of freedom (ϕ_r)	56	56	48	48	48	48	56	56	56
Total degree of freedom (ϕ_T)	83	83	71	71	71	71	83	83	83
Inter-laboratories variance ($V_R = S_R/\phi_R$)	0.04	0.03	7.031	0.818	0.722	2.780	222.774	174.252	2.893
Within-laboratory variance ($V_{RW} = S_{RW}/\phi_{RW}$)	0.00	0.00	0.007	0.002	0.002	0.000	0.002	0.000	0.001
Repeatability variance ($V_r = S_r/\phi_r$)	0.00	0.00	0.004	0.001	0.000	0.001	0.001	0.000	0.000
Laboratory component of variance ($s_b^2 = (V_R - V_{RW})/(2*3)$)	0.01	0.01	1.171	0.136	0.120	0.463	37.129	29.042	0.482
Within-laboratory component of variance ($s_c^2 = (V_{RW} - V_r)/3$)	0.00	0.00	0.001	0.000	0.001	0.000	0.000	0.000	0.000
Repeatability component of variance ($s_r^2 = V_r$)	0.00	0.00	0.004	0.001	0.000	0.001	0.001	0.000	0.000
Inter-laboratories standard deviation ($s_R = SQRT(s_r^2/(2*3) + s_c^2/2 + s_b^2)$)	0.1	0.1	1.08	0.37	0.35	0.68	6.09	5.39	0.69
Within-laboratory standard deviation ($s_{RW} = SQRT(s_r^2/3 + s_c^2)$)	0.0	0.0	0.05	0.02	0.02	0.01	0.02	0.01	0.02
Repeatability standard deviation ($s_r = SQRT(s_r^2)$)	0.0	0.0	0.06	0.03	0.01	0.02	0.03	0.01	0.01
Inter-laboratories precision CV (%)	1.4	1.5	28.12	43.07	112.83	239.66	333.04	345.72	212.79
Within-laboratory precision CV (%)	0.4	0.4	1.23	2.79	8.03	2.02	1.30	0.59	4.65
Repeatability precision CV (%)	0.5	0.3	1.64	3.75	2.54	8.10	1.47	0.54	2.17
Reproducibility limit ($R = D(2, 0.95)*s_R$)	0.23	0.21	3.031	1.034	0.971	1.906	17.061	15.089	1.944
Within-laboratory-reproducibility limit ($R_W = D(2, 0.95)*s_{Rw}$)	0.06	0.06	0.133	0.067	0.069	0.016	0.067	0.026	0.043
Repeatability limit ($r = D(3, 0.95)*s_r$)	0.09	0.05	0.209	0.106	0.026	0.076	0.089	0.028	0.023

Table 6.2. Analysis of variance for the verified data

Statistics	No. 051								
	pH(H$_2$O)	pH(KCl)	Ex-Ca	Ex-Mg	Ex-K	Ex-Na	Ex-acidity	Ex-Al	Ex-H
Number of Laboratories	12	11	10	7	10	10	12	13	13
Total sum of square	135424	83175	160	30	32	11	12259	9541	687
ST/lmd	1881	1260	3	1	1	0	170	122	9
Number of Data	72	66	60	42	60	60	72	78	78
Total sum	368.0	288.4	12.63	5.52	5.65	3.27	110.72	97.68	26.22
Total average	5.1	4.4	0.21	0.13	0.09	0.05	1.54	1.25	0.34
Sum of square inter-laboratories (S_R)	0.3	0.2	0.47	0.00	0.02	0.01	2.53	4.27	2.19
Sum of square within-laboratory (S_{RW})	0.0	0.0	0.00	0.00	0.00	0.00	0.05	0.13	0.02
Sum of square repeatablility (S_r)	0.0	0.0	0.00	0.00	0.00	0.00	0.06	0.26	0.09
Total sum of square (S_T)	0.3	0.3	0.48	0.01	0.03	0.01	2.64	4.67	2.30
Inter-laboratories degree of freedom (ϕ_R)	11	10	9	6	9	9	11	12	12
Within-laboratory degree of freedom (ϕ_{RW})	12	11	10	7	10	10	12	13	13
Repeatability degree of freedom (ϕ_r)	48	44	40	28	40	40	48	52	52
Total degree of freedom (ϕ_T)	71	65	59	41	59	59	71	77	77
Inter-laboratories variance ($V_R = S_R/\phi_R$)	0.03	0.02	0.053	0.001	0.003	0.001	0.230	0.356	0.183
Within-laboratory variance ($V_{RW} = S_{RW}/\phi_{RW}$)	0.00	0.00	0.000	0.000	0.000	0.000	0.004	0.010	0.002
Repeatability variance ($V_r = S_r/\phi_r$)	0.00	0.00	0.000	0.000	0.000	0.000	0.001	0.005	0.002
Laboratory component of variance ($s_b^2 = (V_R-V_{RW})/(2*3)$)	0.00	0.00	0.009	0.000	0.000	0.000	0.038	0.058	0.030
Within-laboratory component of variance ($s_c^2 = (V_{RW}-V_r)/3$)	0.00	0.00	0.000	0.000	0.000	0.000	0.001	0.002	0.000
Repeatability component of variance ($s_r^2 = V_r$)	0.00	0.00	0.000	0.000	0.000	0.000	0.001	0.005	0.002
Inter-laboratories standard deviation ($s_R = SQRT(s_r^2/(2*3) + s_c^2/2 + s_b^2)$)	0.1	0.1	0.09	0.01	0.02	0.01	0.20	0.24	0.17
Within-laboratory standard deviation ($s_{RW} = SQRT(s_r^2/3 + s_c^2)$)	0.0	0.0	0.01	0.01	0.00	0.00	0.04	0.06	0.02
Repeatability standard deviation ($s_r = SQRT(s_r^2)$)	0.0	0.0	0.01	0.01	0.01	0.00	0.04	0.07	0.04
Inter-laboratories precision CV (%)	1.3	1.5	44.49	7.41	22.31	20.84	12.73	19.46	51.90
Within-laboratory precision CV (%)	0.2	0.0	3.70	6.85	3.26	7.11	2.35	4.65	6.67
Repeatability precision CV (%)	0.3	0.4	3.93	4.98	5.65	8.54	2.34	5.67	12.04
Reproducibility limit ($R = D(2, 0.95)*s_R$)	0.19	0.18	0.262	0.027	0.059	0.032	0.548	0.682	0.489
Within-laboratory-reproducibility limit ($R_W = D(2, 0.95)*s_{RW}$)	0.03	0.00	0.022	0.025	0.009	0.011	0.101	0.163	0.063
Repeatability limit ($r = D(3, 0.95)*s_r$)	0.05	0.06	0.027	0.022	0.018	0.015	0.119	0.234	0.134

Statistics	No. 052								
	pH(H$_2$O)	pH(KCl)	Ex-Ca	Ex-Mg	Ex-K	Ex-Na	Ex-acidity	Ex-Al	Ex-H
Number of Laboratories	12	12	11	12	10	10	12	9	12
Total sum of square	169085	135424	68189	3812	95	20	173	0	121
ST/lmd	2348	1881	1033	53	2	0	2	0	2
Number of Data	72	72	66	72	60	60	72	54	72
Total sum	411.2	368.0	261.13	61.74	9.74	4.45	13.16	0.45	11.02
Total average	5.7	5.1	3.96	0.86	0.16	0.07	0.18	0.01	0.15
Sum of square inter-laboratories (S_R)	0.2	0.2	68.37	9.00	0.03	0.02	0.44	0.01	0.09
Sum of square within-laboratory (S_{RW})	0.0	0.0	0.02	0.02	0.00	0.00	0.01	0.00	0.01
Sum of square repeatablility (S_r)	0.0	0.0	0.19	0.05	0.00	0.00	0.02	0.00	0.00
Total sum of square (S_T)	0.2	0.2	68.58	9.07	0.03	0.02	0.46	0.01	0.10
Inter-laboratories degree of freedom (ϕ_R)	11	11	10	11	9	9	11	8	11
Within-laboratory degree of freedom (ϕ_{RW})	12	12	11	12	10	10	12	9	12
Repeatability degree of freedom (ϕ_r)	48	48	44	48	40	40	48	36	48
Total degree of freedom (ϕ_T)	71	71	65	71	59	59	71	53	71
Inter-laboratories variance ($V_R = S_R/\phi_R$)	0.02	0.02	6.837	0.818	0.003	0.002	0.040	0.001	0.008
Within-laboratory variance ($V_{RW} = S_{RW}/\phi_{RW}$)	0.00	0.00	0.002	0.002	0.000	0.000	0.000	0.000	0.001
Repeatability variance ($V_r = S_r/\phi_r$)	0.00	0.00	0.004	0.001	0.000	0.000	0.000	0.000	0.000
Laboratory component of variance ($s_b^2 = (V_R-V_{RW})/(2*3)$)	0.00	0.00	1.139	0.136	0.000	0.000	0.007	0.000	0.001
Within-laboratory component of variance ($s_c^2 = (V_{RW}-V_r)/3$)	0.00	0.00	-0.001	0.000	0.000	0.000	0.000	0.000	0.000
Repeatability component of variance ($s_r^2 = V_r$)	0.00	0.00	0.004	0.001	0.000	0.000	0.000	0.000	0.000
Inter-laboratories standard deviation ($s_R = SQRT(s_r^2/(2*3) + s_c^2/2 + s_b^2)$)	0.1	0.1	1.07	0.37	0.02	0.02	0.08	0.01	0.04
Within-laboratory standard deviation ($s_{RW} = SQRT(s_r^2/3 + s_c^2)$)	0.0	0.0	0.03	0.02	0.00	0.00	0.01	0.00	0.02
Repeatability standard deviation ($s_r = SQRT(s_r^2)$)	0.0	0.0	0.07	0.03	0.00	0.00	0.02	0.00	0.01
Inter-laboratories precision CV (%)	0.9	1.0	26.98	43.07	13.29	22.63	44.72	172.63	24.25
Within-laboratory precision CV (%)	0.2	0.2	0.64	2.79	2.05	5.22	6.64	47.14	9.94
Repeatability precision CV (%)	0.4	0.3	1.65	3.75	2.75	5.22	10.17	56.57	4.87
Reproducibility limit ($R = D(2, 0.95)*s_R$)	0.15	0.14	2.989	1.034	0.060	0.047	0.229	0.040	0.104
Within-laboratory-reproducibility limit ($R_W = D(2, 0.95)*s_{RW}$)	0.03	0.03	0.071	0.067	0.009	0.011	0.034	0.011	0.043
Repeatability limit ($r = D(3, 0.95)*s_r$)	0.08	0.05	0.216	0.106	0.015	0.013	0.061	0.016	0.025

4. DISCUSSION

By using digital formats, no obvious calculation mistake was found in the data. However, some outliers have ten times larger than averages and more than three outliers were detected in three laboratories. It was suggested that quality control within the laboratories and standard operating procedures and reporting system should be elaborated in such laboratories. The laboratories have discussed possible causes of the outliers with NC. Efforts have been made for improvement of the data quality.

Repeatability precisions and within-laboratory-reproducibility precisions were relatively large in Ex-base cations. However, inter-laboratories precisions were improved compared to the results of the 6th attempt. Efforts should be made continuously to improve the precision furthermore.

5. ACKNOWLEDGMENT

ADORC wishes to thank Iwate Prefecture for their cooperation on collecting soil samples.

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7. CONTACT INFORMATION

Please address all inquiries, comments and suggestions about this report to:

Acid Deposition and Oxidant Research Center (ADORC)

1182 Sowa, Niigata city 950-2144 Japan

Tel:	+81-25-263-0550
Fax:	+81-25-263-0566
E-mail:	eanetdata@adorc.gr.jp
URL:	http://www.adorc.gr.jp

Contact persons:

Name	Department & E-mail address
Dr. Hiroyuki SASE	Ecological Impact Research Department/ Data Management Department E-mail: sase@adorc.gr.jp
Mr. Masato KAMISAKO	Ecological Impact Research Department/ Data Management Department E-mail: kamisako@adorc.gr.jp
Mr. Shinji NAKAYAMA (EANET QA/QC Manager)	Dept. Head, Data Management Department E-mail: nakayama@adorc.gr.jp

APPENDIX 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
Air Quality Laboratory, Environmental Management Center   **id01**
Soil Research Institute   **id02**

3. JAPAN
Shimane Agricultural Experimental Station   **jp01**

4. MALAYSIA

                                                                             Not participated

5. MONGOLIA
Central Laboratory of Environmental Monitoring   **mn01**

6. PHILIPPINES
University of the Philippines, Los Baños   **ph01**

7. Republic of KOREA
Soil Environmental Division, National Institute of Environmental Research   **kr01**

8. RUSSIA
Limnological Institute Russian Academy of Science/Siberian Branch   **ru01**

9. THAILAND
King Mongkut's University of Technology Thonburi   **th01**

10. VIET NAM
Center for Environmental Research, Institute of Meteorology and Hydrology,   **vn01**
MoNRE
Highland and Mid-Central Environment Analysis Laboratory, Middle of   **vn02**
Central   regional   Hydro-Meteorological   Observatory,   National
Hydro-Meteorological Center, MoNRE

Appendix 2.1. Entire data of pH in Sample No. 051

Lab.	pH(H₂O)			pH(KCl)		
	Lab. Ave.	Average	repeat	Lab. Ave.	Average	repeat
cn01	5.2	5.2 (0.0)	5.2	4.4	4.4 (0.0)	4.4
			5.2			4.4
			5.2			4.4
		5.2 (0.0)	5.2		4.4 (0.0)	4.4
			5.2			4.4
			5.2			4.4
cn02	5.2	5.1 (0.1)	5.2	4.4	4.4 (0.0)	4.4
			5.1			4.4
			5.1			4.4
		5.2 (0.1)	5.2		4.4 (0.0)	4.4
			5.2			4.4
			5.1			4.4
cn03	5.1	5.1 (0.0)	5.1	4.4	4.4 (0.0)	4.4
			5.1			4.4
			5.1			4.4
		5.1 (0.0)	5.1		4.4 (0.0)	4.4
			5.1			4.4
			5.1			4.4
cn04	5.1	5.1 (0.0)	5.1	4.4	4.4 (0.0)	4.4
			5.1			4.4
			5.1			4.4
		5.1 (0.0)	5.1		4.4 (0.0)	4.4
			5.1			4.4
			5.1			4.4
id01	5.0	5.0 (0.0)	5.0	4.3	4.3 (0.0)	4.3
			5.0			4.3
			5.0			4.3
		5.0 (0.0)	5.0		4.3 (0.0)	4.3
			5.0			4.3
			5.0			4.3
id02	5.1	5.1 (0.0)	5.1	4.3	4.3 (0.0)	4.3
			5.1			4.3
			5.1			4.3
		5.1 (0.0)	5.1		4.3 (0.0)	4.3
			5.1			4.3
			5.1			4.3
jp01	5.2	5.2 (0.0)	5.2	4.5	4.4 (0.0)	4.4
			5.2			4.4
			5.2			4.4
		5.2 (0.0)	5.2		4.5 (0.0)	4.5
			5.2			4.5
			5.2			4.5
my01						
mn01	5.1	5.1 (0.1)	5.2	4.5	4.5 (0.0)	4.5
			5.1			4.5
			5.1			4.5
		5.1 (0.0)	5.1		4.5 (0.0)	4.5
			5.1			4.5
			5.1			4.5
ph01	5.0	5.0 (0.0)	5.0	4.1	4.1 (0.0)	4.1
			5.0			4.1
			5.0			4.1
		5.0 (0.0)	5.0		4.1 (0.0)	4.1
			5.0			4.1
			5.0			4.1
kr01	5.1	5.1 (0.1)	5.1	4.4	4.3 (0.0)	4.3
			5.2			4.3
			5.1			4.3
		5.1 (0.0)	5.1		4.4 (0.0)	4.4
			5.1			4.4
			5.1			4.4
ru01	5.1	5.1 (0.0)	5.1	4.4	4.4 (0.0)	4.4
			5.1			4.4
			5.1			4.4
		5.1 (0.0)	5.1		4.4 (0.0)	4.4
			5.1			4.4
			5.1			4.4
th01	5.5	5.5 (0.0)	5.5	4.3	4.3 (0.0)	4.3
			5.5			4.3
			5.5			4.3
		5.5 (0.0)	5.5		4.3 (0.0)	4.3
			5.5			4.3
			5.5			4.3
vn01	5.1	5.1 (0.0)	5.1	4.4	4.4 (0.1)	4.4
			5.1			4.3
			5.1			4.4
		5.1 (0.0)	5.1		4.4 (0.1)	4.3
			5.1			4.4
			5.1			4.4
vn02	5.2	5.2 (0.0)	5.2	4.3	4.3 (0.0)	4.3
			5.2			4.3
			5.2			4.3
		5.2 (0.0)	5.2		4.3 (0.0)	4.3
			5.2			4.3
			5.2			4.3

Appendix 2.2. Entire data of exchangeable base cations in Sample No. 051

Lab.	Ex-Ca			Ex-Mg			Ex-K			Ex-Na		
	Lab. Ave.	Average	repeat	Lab. Ave.	Average	repeat	Lab. Ave.	Average	repeat	Lab. Ave.	Average	repeat
			cmol(+) kg^{-1}									
cn01	0.12	0.12 (0.01)	0.11	0.13	0.13 (0.00)	0.13	0.09	0.09 (0.00)	0.09	0.05	0.05 (0.00)	0.05
			0.12			0.13			0.09			0.05
			0.12			0.13			0.09			0.05
		0.11 (0.01)	0.10		0.13 (0.01)	0.14		0.09 (0.00)	0.09		0.05 (0.00)	0.05
			0.11			0.13			0.09			0.05
			0.12			0.13			0.09			0.05
cn02	0.22	0.21 (0.01)	0.21	0.13	0.13 (0.01)	0.12	0.11	0.11 (0.01)	0.12	0.06	0.06 (0.01)	0.05
			0.21			0.13			0.10			0.06
			0.22			0.13			0.11			0.07
		0.22 (0.01)	0.22		0.12 (0.00)	0.12		0.11 (0.01)	0.10		0.05 (0.01)	0.04
			0.22			0.12			0.11			0.05
			0.21			0.12			0.11			0.06
cn03	0.31	0.32 (0.01)	0.31	0.15	0.16 (0.01)	0.15	0.10	0.10 (0.01)	0.11	0.05	0.05 (0.01)	0.05
			0.33			0.16			0.10			0.06
			0.33			0.16			0.10			0.05
		0.30 (0.01)	0.29		0.14 (0.01)	0.15		0.10 (0.01)	0.11		0.04 (0.00)	0.04
			0.31			0.13			0.09			0.04
			0.31			0.13			0.10			0.04
cn04	0.16	0.15 (0.01)	0.15	0.13	0.14 (0.02)	0.16	0.11	0.11 (0.01)	0.10	0.05	0.05 (0.01)	0.05
			0.14			0.14			0.11			0.04
			0.15			0.13			0.12			0.05
		0.16 (0.02)	0.15		0.12 (0.01)	0.12		0.10 (0.01)	0.11		0.05 (0.01)	0.05
			0.16			0.13			0.10			0.04
			0.18			0.12			0.10			0.05
id01	0.13	0.13 (0.00)	0.13	0.13	0.13 (0.00)	0.13	0.53	0.53 (0.00)	0.53	0.07	0.07 (0.01)	0.07
			0.13			0.13			0.53			0.07
			0.13			0.13			0.53			0.06
		0.13 (0.00)	0.13		0.13 (0.00)	0.13		0.53 (0.01)	0.52		0.07 (0.01)	0.07
			0.13			0.13			0.53			0.07
			0.13			0.13			0.53			0.06
id02	0.38	0.37 (0.01)	0.38	0.20	0.20 (0.01)	0.21	0.11	0.11 (0.00)	0.11	0.06	0.06 (0.01)	0.07
			0.37			0.19			0.11			0.06
			0.37			0.21			0.11			0.06
		0.38 (0.01)	0.38		0.20 (0.01)	0.21		0.11 (0.00)	0.11		0.06 (0.00)	0.06
			0.38			0.21			0.11			0.06
			0.37			0.19			0.11			0.06
jp01	0.19	0.18 (0.01)	0.19	0.14	0.13 (0.01)	0.13	0.11	0.11 (0.01)	0.10	0.05	0.05 (0.00)	0.05
			0.18			0.13			0.12			0.05
			0.18			0.14			0.12			0.05
		0.20 (0.01)	0.21		0.14 (0.01)	0.14		0.11 (0.00)	0.11		0.05 (0.00)	0.05
			0.19			0.14			0.11			0.05
			0.20			0.15			0.11			0.05
my01												
mn01												
ph01	5.07	4.94 (0.08)	4.85	2.52	2.52 (0.12)	2.38	0.66	0.67 (0.00)	0.67	1.81	1.85 (0.03)	1.83
			4.98			2.59			0.67			1.83
			4.98			2.59			0.67			1.88
		5.20 (0.08)	5.24		2.52 (0.12)	2.59		0.65 (0.02)	0.67		1.77 (0.00)	1.77
			5.11			2.38			0.64			1.77
			5.24			2.59			0.64			1.77
kr01	0.10	0.10 (0.01)	0.10	0.12	0.11 (0.00)	0.11	0.09	0.09 (0.00)	0.09	0.05	0.05 (0.00)	0.05
			0.10			0.11			0.09			0.05
			0.11			0.11			0.09			0.05
		0.10 (0.01)	0.10		0.12 (0.01)	0.12		0.09 (0.01)	0.08		0.05 (0.00)	0.05
			0.10			0.12			0.09			0.05
			0.11			0.13			0.09			0.05
ru01	0.31	0.31 (0.02)	0.33	0.29	0.28 (0.01)	0.29	0.06	0.05 (0.00)	0.05	0.08	0.08 (0.01)	0.08
			0.31			0.28			0.05			0.08
			0.30			0.28			0.05			0.07
		0.31 (0.01)	0.31		0.30 (0.00)	0.30		0.06 (0.00)	0.06		0.08 (0.00)	0.08
			0.32			0.30			0.06			0.08
			0.30			0.30			0.06			0.08
th01	0.20	0.20 (0.01)	0.21	0.38	0.38 (0.00)	0.38	0.06	0.06 (0.00)	0.06	0.04	0.04 (0.00)	0.04
			0.20			0.38			0.06			0.04
			0.20			0.38			0.06			0.04
		0.19 (0.00)	0.19		0.37 (0.01)	0.37		0.06 (0.00)	0.06		0.04 (0.00)	0.04
			0.19			0.36			0.06			0.04
			0.19			0.38			0.06			0.04
vn01	1.17	1.16 (0.02)	1.14	0.86	0.86 (0.00)	0.86	0.11	0.11 (0.00)	0.11	0.15	0.15 (0.01)	0.15
			1.17			0.86			0.11			0.15
			1.17			0.86			0.11			0.14
		1.18 (0.02)	1.17		0.86 (0.00)	0.86		0.11 (0.00)	0.11		0.15 (0.00)	0.15
			1.17			0.86			0.11			0.15
			1.21			0.86			0.11			0.15
vn02												

Appendix 2.3. Entire data of exchangeable acidity and acid cations in Sample No. 051

Lab.	Ex-acidity			Ex-Al			Ex-H		
	cmol(+) kg^{-1}								
	Lab. Ave.	Average	repeat	Lab. Ave.	Average	repeat	Lab. Ave.	Average	repeat
cn01	1.77	1.77 (0.04)	1.80	1.14	1.15 (0.05)	1.21	0.64	0.63 (0.03)	0.61
			1.72			1.12			0.62
			1.78			1.13			0.67
		1.76 (0.04)	1.73		1.13 (0.03)	1.11		0.65 (0.02)	0.64
			1.75			1.12			0.65
			1.80			1.16			0.67
cn02	1.56	1.59 (0.07)	1.65	1.04	1.07 (0.06)	1.13	0.52	0.52 (0.01)	0.52
			1.59			1.07			0.52
			1.52			1.01			0.51
		1.52 (0.07)	1.59		1.01 (0.07)	1.07		0.51 (0.01)	0.52
			1.52			1.02			0.50
			1.45			0.94			0.51
cn03	1.53	1.57 (0.05)	1.63	1.09	1.10 (0.07)	1.14	0.45	0.48 (0.06)	0.49
			1.54			1.02			0.53
			1.55			1.14			0.42
		1.49 (0.03)	1.52		1.07 (0.02)	1.10		0.42 (0.01)	0.43
			1.48			1.06			0.42
			1.47			1.06			0.41
cn04	1.60	1.61 (0.05)	1.61	1.15	1.15 (0.13)	1.08	0.46	0.46 (0.12)	0.39
			1.56			1.30			0.60
			1.66			1.08			0.39
		1.59 (0.03)	1.61		1.15 (0.13)	1.30		0.46 (0.12)	0.39
			1.56			1.08			0.39
			1.61			1.08			0.60
id01	1.64	1.61 (0.00)	1.61	1.12	1.10 (0.02)	1.09	0.52	0.51 (0.03)	0.53
			1.61			1.09			0.53
			1.61			1.13			0.48
		1.66 (0.03)	1.63		1.13 (0.08)	1.04		0.53 (0.05)	0.59
			1.68			1.18			0.50
			1.68			1.18			0.50
id02	1.29	1.29 (0.01)	1.30	1.09	1.09 (0.01)	1.10	0.20	0.20 (0.00)	0.20
			1.28			1.08			0.20
			1.28			1.08			0.20
		1.28 (0.00)	1.28		1.08 (0.00)	1.08		0.19 (0.01)	0.20
			1.28			1.08			0.18
			1.28			1.08			0.20
jp01	1.43	1.37 (0.02)	1.38	1.29	1.20 (0.02)	1.21	0.15	0.17 (0.01)	0.18
			1.34			1.18			0.16
			1.38			1.21			0.18
		1.49 (0.02)	1.47		1.37 (0.01)	1.36		0.12 (0.01)	0.11
			1.51			1.38			0.13
			1.49			1.37			0.12
my01									
mn01	1.40	1.41 (0.06)	1.48	1.77	1.70 (0.00)	1.70	0.49	0.42 (0.00)	0.42
			1.38			1.70			0.42
			1.38			1.70			0.42
		1.38 (0.00)	1.38		1.84 (0.24)	2.12		0.56 (0.25)	0.85
			1.38			1.70			0.42
			1.38			1.70			0.42
ph01	1.58	1.67 (0.00)	1.67	1.44	1.53 (0.00)	1.53	0.14	0.14 (0.00)	0.14
			1.67			1.53			0.14
			1.67			1.53			0.14
		1.48 (0.00)	1.48		1.35 (0.00)	1.35		0.13 (0.00)	0.13
			1.48			1.35			0.13
			1.48			1.35			0.13
kr01	1.67	1.67 (0.06)	1.71	1.37	1.39 (0.11)	1.39	0.25	0.21 (0.00)	0.21
			1.71			1.50			0.21
			1.60			1.28			0.21
		1.67 (0.03)	1.71		1.35 (0.06)	1.28		0.28 (0.06)	0.32
			1.65			1.39			0.21
			1.65			1.39			0.32
ru01	1.50	1.50 (0.00)	1.50	1.28	1.28 (0.00)	1.28	0.21	0.21 (0.00)	0.21
			1.50			1.28			0.21
			1.50			1.28			0.21
		1.50 (0.00)	1.50		1.28 (0.00)	1.28		0.21 (0.00)	0.21
			1.50			1.28			0.21
			1.50			1.28			0.21
th01	3.47	3.45 (0.00)	3.45	3.34	3.34 (0.00)	3.34	0.12	0.11 (0.00)	0.11
			3.45			3.34			0.11
			3.45			3.34			0.11
		3.48 (0.01)	3.48		3.34 (0.01)	3.34		0.13 (0.00)	0.13
			3.48			3.35			0.13
			3.47			3.34			0.13
vn01	1.20	1.20 (0.01)	1.19	0.90	0.90 (0.00)	0.90	0.30	0.30 (0.01)	0.29
			1.19			0.90			0.29
			1.21			0.90			0.31
		1.20 (0.02)	1.21		0.90 (0.00)	0.90		0.30 (0.02)	0.31
			1.21			0.90			0.31
			1.17			0.90			0.27
vn02	1.89	1.88 (0.00)	1.88	1.61	1.61 (0.00)	1.61	0.43	0.43 (0.00)	0.43
			1.88			1.61			0.43
			1.88			1.61			0.43
		1.90 (0.03)	1.94		1.61 (0.00)	1.61		0.43 (0.00)	0.43
			1.88			1.61			0.43
			1.88			1.61			0.43

Appendix 3.1. Entire data of pH in Sample No. 052

Lab.	pH(H₂O)			pH(KCl)		
	Lab. Ave.	Average	repeat	Lab. Ave.	Average	repeat
cn01 chongqing	5.7	5.7 (0.1)	5.7	5.2	5.1 (0.0)	5.1
			5.8			5.1
			5.7			5.1
		5.7 (0.1)	5.7		5.2 (0.0)	5.2
			5.7			5.2
			5.8			5.2
cn02 xian	5.7	5.7 (0.0)	5.7	5.1	5.1 (0.0)	5.1
			5.7			5.1
			5.7			5.1
		5.7 (0.0)	5.7		5.1 (0.0)	5.1
			5.7			5.1
			5.7			5.1
cn03 xiamen	5.7	5.7 (0.0)	5.7	5.1	5.1 (0.1)	5.1
			5.7			5.2
			5.7			5.2
		5.7 (0.0)	5.7		5.1 (0.0)	5.1
			5.7			5.1
			5.7			5.1
cn04 zhuhai	5.7	5.7 (0.0)	5.7	5.1	5.1 (0.0)	5.1
			5.7			5.1
			5.7			5.1
		5.7 (0.0)	5.7		5.1 (0.0)	5.1
			5.7			5.1
			5.7			5.1
id01 emc	5.7	5.7 (0.0)	5.7	5.1	5.1 (0.0)	5.1
			5.7			5.1
			5.7			5.1
		5.7 (0.0)	5.7		5.1 (0.0)	5.1
			5.7			5.1
			5.7			5.1
id02 csar	5.8	5.8 (0.0)	5.8	5.1	5.1 (0.0)	5.1
			5.8			5.1
			5.8			5.1
		5.8 (0.0)	5.8		5.1 (0.0)	5.1
			5.8			5.1
			5.8			5.1
jp01	5.8	5.7 (0.0)	5.7	5.2	5.2 (0.0)	5.2
			5.7			5.2
			5.7			5.2
		5.8 (0.0)	5.8		5.2 (0.0)	5.2
			5.8			5.2
			5.8			5.2
my01						
mn01	5.7	5.7 (0.0)	5.7	5.2	5.2 (0.0)	5.2
			5.7			5.2
			5.7			5.2
		5.7 (0.0)	5.7		5.2 (0.0)	5.2
			5.7			5.2
			5.7			5.2
ph01	5.6	5.6 (0.0)	5.6	4.9	4.9 (0.0)	4.9
			5.6			4.9
			5.6			4.9
		5.6 (0.0)	5.6		4.9 (0.0)	4.9
			5.6			4.9
			5.6			4.9
kr01	5.7	5.7 (0.0)	5.7	5.1	5.1 (0.0)	5.1
			5.7			5.1
			5.7			5.1
		5.7 (0.1)	5.8		5.1 (0.0)	5.1
			5.7			5.1
			5.7			5.1
ru01	5.7	5.7 (0.0)	5.7	5.1	5.1 (0.0)	5.1
			5.7			5.1
			5.7			5.1
		5.7 (0.0)	5.7		5.1 (0.0)	5.1
			5.7			5.1
			5.7			5.1
th01	5.5	5.5 (0.1)	5.4	5.0	5.0 (0.0)	5.0
			5.5			5.0
			5.5			5.0
		5.5 (0.1)	5.4		5.0 (0.0)	5.0
			5.5			5.0
			5.5			5.0
vn01	5.7	5.7 (0.1)	5.7	5.1	5.1 (0.0)	5.1
			5.7			5.1
			5.6			5.1
		5.7 (0.0)	5.7		5.1 (0.0)	5.1
			5.7			5.1
			5.7			5.1
vn02	5.8	5.8 (0.0)	5.8	5.1	5.1 (0.0)	5.1
			5.8			5.1
			5.8			5.1
		5.8 (0.0)	5.8		5.1 (0.1)	5.2
			5.8			5.1
			5.8			5.1

Appendix 3.2. Entire data of exchangeable base cations in Sample No. 052

Lab.	Ex-Ca Lab. Ave.	Ex-Ca Average	Ex-Ca repeat	Ex-Mg Lab. Ave.	Ex-Mg Average	Ex-Mg repeat	Ex-K Lab. Ave.	Ex-K Average	Ex-K repeat	Ex-Na Lab. Ave.	Ex-Na Average	Ex-Na repeat
						cmol(+) kg^{-1}						
cn01	2.96	2.96 (0.09)	2.98	0.75	0.75 (0.01)	0.75	0.15	0.15 (0.01)	0.15	0.06	0.06 (0.01)	0.06
			3.03			0.76			0.15			0.06
			2.86			0.74			0.14			0.05
		2.96 (0.09)	2.86		0.74 (0.01)	0.73		0.15 (0.01)	0.14		0.06 (0.00)	0.06
			2.98			0.73			0.15			0.06
			3.03			0.75			0.15			0.06
cn02	3.33	3.31 (0.02)	3.33	0.48	0.49 (0.01)	0.49	0.19	0.19 (0.00)	0.19	0.08	0.08 (0.01)	0.08
			3.29			0.48			0.19			0.07
			3.30			0.49			0.19			0.08
		3.34 (0.01)	3.33		0.47 (0.01)	0.46		0.19 (0.01)	0.19		0.07 (0.01)	0.07
			3.35			0.48			0.19			0.06
			3.35			0.48			0.20			0.07
cn03	3.20	3.23 (0.06)	3.28	0.50	0.49 (0.04)	0.52	0.19	0.19 (0.01)	0.19	0.07	0.07 (0.01)	0.07
			3.23			0.45			0.19			0.07
			3.17			0.49			0.18			0.06
		3.17 (0.02)	3.15		0.51 (0.02)	0.51		0.18 (0.01)	0.17		0.06 (0.00)	0.06
			3.18			0.52			0.18			0.06
			3.17			0.49			0.18			0.06
cn04	2.84	2.81 (0.04)	2.78	0.73	0.73 (0.02)	0.73	0.16	0.15 (0.01)	0.15	0.06	0.06 (0.01)	0.06
			2.86			0.71			0.15			0.06
			2.79			0.75			0.16			0.05
		2.86 (0.03)	2.86		0.72 (0.01)	0.71		0.16 (0.01)	0.17		0.06 (0.01)	0.06
			2.84			0.72			0.15			0.07
			2.89			0.73			0.16			0.06
id01	2.88	2.87 (0.01)	2.86	0.72	0.71 (0.01)	0.72	1.20	1.14 (0.00)	1.14	0.09	0.09 (0.00)	0.09
			2.86			0.70			1.14			0.09
			2.88			0.70			1.14			0.09
		2.88 (0.03)	2.86		0.72 (0.00)	0.72		1.26 (0.00)	1.26		0.09 (0.00)	0.09
			2.88			0.72			1.26			0.09
			2.91			0.72			1.26			0.09
id02	3.57	3.56 (0.01)	3.56	0.63	0.63 (0.01)	0.64	0.15	0.15 (0.01)	0.15	0.10	0.09 (0.01)	0.09
			3.55			0.62			0.16			0.10
			3.56			0.64			0.15			0.09
		3.57 (0.01)	3.58		0.63 (0.01)	0.62		0.15 (0.00)	0.15		0.10 (0.00)	0.10
			3.57			0.64			0.15			0.10
			3.57			0.64			0.15			0.10
jp01	4.85	4.86 (0.14)	4.99	1.32	1.33 (0.01)	1.33	0.19	0.19 (0.01)	0.19	0.06	0.06 (0.01)	0.06
			4.71			1.33			0.18			0.05
			4.87			1.32			0.19			0.06
		4.83 (0.05)	4.83		1.31 (0.02)	1.30		0.19 (0.00)	0.19		0.06 (0.00)	0.06
			4.87			1.33			0.19			0.06
			4.78			1.31			0.19			0.06
my01												
mn01												
ph01	5.52	5.54 (0.16)	5.63	1.62	1.58 (0.00)	1.58	0.87	0.87 (0.02)	0.84	2.44	2.45 (0.06)	2.51
			5.35			1.58			0.88			2.45
			5.63			1.58			0.88			2.39
		5.49 (0.14)	5.35		1.66 (0.13)	1.58		0.87 (0.02)	0.84		2.43 (0.09)	2.33
			5.49			1.81			0.88			2.51
			5.63			1.58			0.88			2.45
kr01	2.68	2.58 (0.02)	2.58	0.52	0.52 (0.01)	0.51	0.16	0.15 (0.01)	0.15	0.06	0.06 (0.00)	0.06
			2.60			0.53			0.16			0.06
			2.56			0.52			0.15			0.06
		2.78 (0.04)	2.82		0.51 (0.02)	0.53		0.16 (0.00)	0.16		0.06 (0.00)	0.06
			2.78			0.51			0.16			0.06
			2.74			0.50			0.16			0.06
ru01	5.75	5.73 (0.03)	5.73	0.97	0.94 (0.01)	0.95	0.14	0.14 (0.00)	0.14	0.09	0.08 (0.00)	0.08
			5.76			0.93			0.14			0.08
			5.71			0.95			0.14			0.08
		5.77 (0.02)	5.77		0.99 (0.02)	1.00		0.14 (0.00)	0.14		0.09 (0.01)	0.09
			5.76			0.97			0.14			0.09
			5.79			0.99			0.14			0.08
th01	4.08	4.08 (0.01)	4.08	0.78	0.77 (0.01)	0.76	0.13	0.13 (0.00)	0.13	0.10	0.10 (0.00)	0.10
			4.08			0.77			0.13			0.10
			4.07			0.77			0.13			0.10
		4.07 (0.01)	4.07		0.78 (0.01)	0.78		0.13 (0.00)	0.13		0.10 (0.00)	0.10
			4.08			0.78			0.13			0.10
			4.07			0.79			0.13			0.10
vn01	4.59	4.60 (0.00)	4.60	1.31	1.27 (0.00)	1.27	0.18	0.18 (0.00)	0.18	0.24	0.24 (0.01)	0.24
			4.60			1.27			0.18			0.23
			4.60			1.27			0.18			0.24
		4.57 (0.06)	4.60		1.34 (0.06)	1.27		0.18 (0.00)	0.18		0.23 (0.01)	0.23
			4.60			1.37			0.18			0.24
			4.50			1.37			0.18			0.23
vn02												

Appendix 3.3. Entire data of exchangeable acidity and acid cations in Sample No. 052

Lab.	Ex-acidity			Ex-Al			Ex-H		
				cmol(+) kg^{-1}					
	Lab. Ave.	Average	repeat	Lab. Ave.	Average	repeat	Lab. Ave.	Average	repeat
cn01	0.18	0.18 (0.02)	0.16	0.00	0.00 (0.00)	0.00	0.18	0.18 (0.01)	0.17
			0.18			0.00			0.18
			0.19			0.00			0.19
		0.18 (0.01)	0.18		0.00 (0.00)	0.00		0.18 (0.01)	0.18
			0.18			0.00			0.18
			0.17			0.00			0.17
cn02	0.15	0.15 (0.00)	0.15	0.01	0.01 (0.00)	0.01	0.14	0.14 (0.00)	0.14
			0.15			0.01			0.14
			0.15			0.01			0.14
		0.15 (0.00)	0.15		0.01 (0.00)	0.01		0.14 (0.00)	0.14
			0.15			0.01			0.14
			0.15			0.01			0.14
cn03	0.11	0.12 (0.02)	0.12	0.00	0.00 (0.00)	0.00	0.11	0.12 (0.02)	0.12
			0.10			0.00			0.10
			0.13			0.00			0.13
		0.10 (0.01)	0.11		0.00 (0.00)	0.00		0.10 (0.01)	0.11
			0.10			0.00			0.10
			0.09			0.00			0.09
cn04	0.14	0.14 (0.03)	0.16	0.00	0.00 (0.00)	0.00	0.18	0.18 (0.00)	0.18
			0.16			0.00			0.18
			0.10			0.00			0.18
		0.14 (0.03)	0.10		0.00 (0.00)	0.00		0.18 (0.00)	0.18
			0.16			0.00			0.18
			0.16			0.00			0.18
id01	0.19	0.17 (0.01)	0.16	0.04	0.05 (0.01)	0.04	0.15	0.13 (0.01)	0.12
			0.17			0.04			0.13
			0.18			0.06			0.13
		0.20 (0.00)	0.20		0.03 (0.00)	0.03		0.17 (0.00)	0.17
			0.20			0.03			0.17
			0.20			0.03			0.17
id02	0.15	0.15 (0.01)	0.16	0.03	0.03 (0.01)	0.02	0.13	0.13 (0.01)	0.14
			0.14			0.02			0.12
			0.16			0.04			0.12
		0.15 (0.01)	0.14		0.03 (0.01)	0.04		0.13 (0.01)	0.12
			0.14			0.02			0.12
			0.16			0.02			0.14
jp01	0.16	0.17 (0.01)	0.17	0.00	0.00 (0.00)	0.00	0.16	0.17 (0.01)	0.17
			0.16			0.00			0.16
			0.17			0.00			0.17
		0.14 (0.01)	0.14		0.00 (0.00)	0.00		0.14 (0.01)	0.14
			0.15			0.00			0.15
			0.13			0.00			0.13
my01									
mn01	0.36	0.38 (0.07)	0.34	0.91	0.91 (0.00)	0.91	0.00	0.00 (0.00)	0.00
			0.46			0.91			0.00
			0.34			0.91			0.00
		0.34 (0.00)	0.34		0.91 (0.00)	0.91		0.00 (0.00)	0.00
			0.34			0.91			0.00
			0.34			0.91			0.00
ph01	0.32	0.32 (0.00)	0.32	0.16	0.18 (0.00)	0.18	0.17	0.14 (0.00)	0.14
			0.32			0.18			0.14
			0.32			0.18			0.14
		0.32 (0.00)	0.32		0.14 (0.00)	0.14		0.19 (0.00)	0.19
			0.32			0.14			0.19
			0.32			0.14			0.19
kr01	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
ru01	0.23	0.23 (0.00)	0.23	0.11	0.11 (0.00)	0.11	0.11	0.11 (0.00)	0.11
			0.23			0.11			0.11
			0.23			0.11			0.11
		0.23 (0.00)	0.23		0.11 (0.00)	0.11		0.11 (0.00)	0.11
			0.23			0.11			0.11
			0.23			0.11			0.11
th01	23.00	23.00 (0.03)	22.97	20.26	20.25 (0.02)	20.23	2.74	2.75 (0.01)	2.74
			23.01			20.26			2.75
			23.02			20.27			2.75
		22.99 (0.03)	22.96		20.27 (0.03)	20.24		2.72 (0.01)	2.71
			23.01			20.29			2.72
			23.01			20.29			2.72
vn01	0.49	0.50 (0.00)	0.50	0.31	0.31 (0.02)	0.33	0.19	0.19 (0.01)	0.18
			0.50			0.30			0.20
			0.50			0.30			0.20
		0.48 (0.02)	0.50		0.30 (0.00)	0.30		0.18 (0.02)	0.20
			0.47			0.30			0.17
			0.47			0.30			0.17
vn02	0.11	0.11 (0.00)	0.11	0.00	0.00 (0.00)	0	0.23	0.23 (0.00)	0.23
			0.11			0			0.23
			0.11			0			0.23
		0.11 (0.00)	0.11		0.00 (0.00)	0		0.23 (0.00)	0.23
			0.11			0			0.23
			0.11			0			0.23