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

# Report of the Inter-laboratory Comparison Project 2003 on Wet Deposition

**6th Attempt** 

November 2004

Acid Deposition and Oxidant Research Center

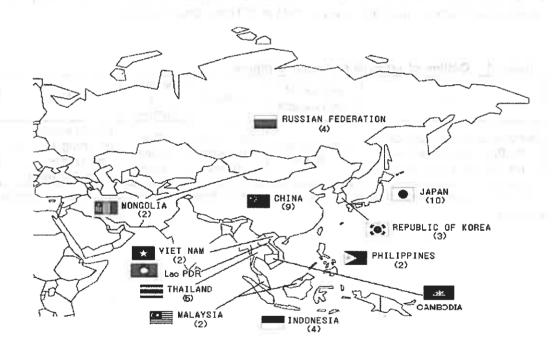
# Contents

INTRODUCTION	
2. PROCEDURE	
2.1 Participating Laboratories	2
2.2 Dispatched Rainwater Samples	
2.3 Analytical Parameters	
	4
3.1 Comparison by Sample	9
Sample No.031(higher concentrations)	9
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The Property of the Control of the C	
	**************************************
Cr	
	18
K <sup>+</sup>	
Ca2+	
(1) (1) (1) (1) (1) (1) (1) (1) (1) (1)	21
NH4	
3.3 Circumstances of Sample Analysis	
Methods Used	
Number of staff in charge of measurement	
() 보고 보고 있는 아들은 이 보고 있습니다. 그런 사람들은 이 사람들은 사람들은 사람들은 사람들은 사람들은 사람들은 사람들은 사람들은	
The number of flagged data in laboratories	28
Water temperature at measurement	29
Water temperature at measurement	
4. COMPARISON OF 1st, 2nd, 3rd, 4th and 5th SU	RVEY
5. FOR IMPROVEMENT OF MEASUREMENT I	
5. FOR IMPROVEMENT OF MEASUREMENT I	-RECISIONS 32
7. REFERENCES	
8. CONTACT INFORMATION	
APPENDIX 1 Contact addresses of participating	ng laboratories i
	iy iv
	alue v

## 1. INTRODUCTION

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

Artificial rainwater samples contained major ions, were prepared and distributed by the Network Center (NC) at the end of 2003. All of the participating laboratories submitted their analytical data to NC. Obtained data for pH, EC and concentrations of  $SO_4^{2^-}$ ,  $NO_3^-$ ,  $CI^-$ ,  $Na^+$ ,  $K^+$ ,  $Ca^{2^+}$ ,  $Mg^{2^+}$  and  $NH_4^+$  were compared with prepared values and statistically treated. List of the participating laboratories, individual analytical data with their laboratory's short name, and various statistical parameters are included in this report.



<sup>\*</sup> Figure in parenthesis shows the number of laboratories of each country (27 laboratories from 12 countries)

Fig.1 Laboratories participated in the Inter-comparison project 2002 of the EANET

## 2. PROCEDURE

#### 2.1 Participating Laboratories

Twenty-seven laboratories in charge of chemical analysis in 12 countries of EANET participated in this survey. The Network Center (NC) shipped the artificial rainwater samples to all of these 24 laboratories, and all of them submitted their analytical data to NC. The names and contact addresses of the participating laboratories are presented in **APPENDIX 1**.

## 2.2 Dispatched Rainwater Samples

Two kinds of artificial rainwater samples (of both higher concentration and lower concentration) were distributed to the laboratories (See Table 1). The information on the analytical precision and accuracy on individual parameters can be obtained through the statistical treatment of submitted analytical data of 100 times diluted samples.

Table 1 Outline of artificial rainwater samples

Name	Amount of each sample	Container	Number of samples	Note
Artificial rainwater samples No.021 (higher concentration) No.022 (lower concentration)	Approximately 100ml	Poly-propyl ene bottle 100ml	One bottle each	Known amount of reagents are dissolved in deionized water

Before the measurement, each laboratory should accurately dilute distributed samples by 100 times under the specified procedure.

## 2.3 Analytical Parameters

All participating laboratories were expected to measure samples and submit the data with the units listed in Table 2 on ten parameters: pH, Electric Conductivity (EC), concentrations of sulfate, nitrate, chloride, sodium-ion, potassium-ion, calcium-ion, magnesium-ion and ammonium. The participating laboratories were informed that concentration of each parameter was within range described in Table 3.

Table 2 Reporting units of analytical parameters

Analyte	Reporting Units	
рН	pH Unites	
EC	milli siemens/meter	mS/m
SO <sub>4</sub> <sup>2</sup> ·	micro mole/liter	$\mu$ mol/L
NO <sub>3</sub>	micro mole/liter	$\mu$ mol/L
Cl	micro mole/liter	$\mu$ mol/L
Na <sup>⁺</sup>	micro mole/liter	$\mu$ mol/L
K <sup>+</sup>	micro mole/liter	$\mu$ mol/L
Ca <sup>2+</sup>	micro mole/liter	$\mu$ mol/L
Mg <sup>2+</sup>	micro mole/liter	$\mu$ mol/L
NH <sub>4</sub> <sup>+</sup>	micro mole/liter	$\mu$ mol/L

Table 3 Concentration range of the artificial rainwater samples\*

Parameter	Range	Parameter	Range
pH	4.0– 5.5	Na <sup>+</sup>	1 - 50 μmol /L
EC	1.0 – 10.0 mS/m	K <sup>+</sup>	1 - 50 μmol /L
SO <sub>4</sub> <sup>2-</sup>	5 – 100μmol/L	Ca <sup>2+</sup>	1 - 50 μmol /L
NO <sub>3</sub>	5 – 100μmol/L	Mg <sup>2+</sup>	1 - 50 μmol /L
Cl	5 – 150μmol/L	NH <sub>4</sub> <sup>+</sup>	3 - 100 μmol /L

<sup>\*</sup> For 100 times diluted samples.

Participating laboratories were expected to use analytical methods and data checking procedures that are specified in the "Technical Manual for Wet Deposition Monitoring in East Asia" and "Quality Assurance/Quality Control (QA/QC) Program for Wet Deposition Monitoring in East Asia". Analytical methods specified in the manual are described in Table 4.

Table 4 Analytical methods specified in the manual

Parameter	Analytical method
Hq	Glass electrode
EC	Conductivity Cell
SO <sub>4</sub> <sup>2</sup> ·	Ion Chromatography
NO <sub>3</sub>	Spectrophotometry
Cl	
Na⁺	Them have to
K <sup>†</sup>	Ion Chromatography
Ca <sup>2+</sup>	Atomic Absorption/Emission Spectrometry
K <sup>†</sup> Ca <sup>2+</sup> Mg <sup>2+</sup>	H. Arie Victoria
NH <sub>4</sub> <sup>+</sup>	Ion Chromatography
INIT4	Spectrophotometry (Indophenol blue)

# 2.5 Data Checking Procedures

- a) Calculation of ion balance (R<sub>1</sub>)
- (1) Total anion (A) equivalent concentration ( $\mu$  eq/ L) is calculated by summing the concentrations of all anions (C:  $\mu$  mol/ L).

**A**  $(\mu \text{ eq L}) = \sum_{i} C_{Ai} (\mu \text{ mol L}) = 2C (SO<sub>4</sub><sup>2</sup>) + C (NO<sub>3</sub>) + C (Cl)$  $C<sub>Ai</sub>: electric charge of ion and concentration <math>(\mu \text{ mol L}^{-1})$  of anion "i".

(2) Total cation (C) equivalent concentration ( $\mu \text{ eq L}^{-1}$ ) is calculated by summing the concentrations of all cations (C:  $\mu \text{ mol L}^{-1}$ ).

**C** 
$$(\mu \text{ eq L}) = \sum_{i} n C_{Ci} (\mu \text{ mol L}^{-1}) = 10^{-(6-pH)} + C (NH_4^+) + C (Na^+) + C (K^+) + 2C (Ca^{2+}) + 2C (Mq^{2+})$$

 $C_{Ci}$ : electric charge of ion and concentration ( $\mu$  mol /L) of cation "i".

(3) Calculation of ion balance (R<sub>1</sub>)  $R_1 = 100 \times (C-A) / (C+A)$ 

(4) R<sub>1</sub>, which is calculated using the above equation, should be compared with standard values in Table 5. If R<sub>1</sub> is out of the range, re-measurement, check with standard solutions, and/or inspection of calibration curves should be undertaken.

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Table 5 Allowable ranges for R<sub>1</sub> in different concentration ranges

C+A (µeq/L)	R <sub>1</sub> (%)
< 50	+ 30 ~ - 30
50 ~ 100	+ 15 ~ - 15
> 100	+8 ~ -8

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

- b) Comparison between calculated and measured values of electrical conductivity (R<sub>2</sub>)
- (1) Total electric conductivity ( $\Lambda$  calc) should be calculated as follows;  $\Lambda$  calc ( $\mu$  S cm<sup>-1</sup>) = {349.7×10 <sup>(3-pH)</sup> + 80.0×2C (SO<sub>4</sub><sup>2-</sup>) + 71.5 C (NO<sub>3</sub><sup>-</sup>) +76.3 C (Cl<sup>-</sup>) + 73.5 C (NH<sub>4</sub><sup>+</sup>) + 50.1 C (Na<sup>+</sup>) + 73.5×C (K<sup>+</sup>) + 59.8×2C (Ca<sup>2+</sup>) +53.3× 2C (Mg<sup>2+</sup>) /10000
- C: Molar concentrations ( $\mu$  mol L<sup>-1</sup>) of ions in the parenthesis; each constant value is ionic equivalent conductance at 25°C.
- (2) Ratio ( $R_2$ ) of calculations ( $\Lambda$  calc) to measurements ( $\Lambda$  meas) in electric conductivity should be calculated as follows;

IN SEC. IN THE C

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

(3)  $R_2$ , which is calculated using the above equation, should be compared with standard values in Table 6. If  $R_1$  is out of the range, re-measurement, check with standard solutions, and/or inspection of calibration curves are necessary.

Table 6 Allowable ranges for R2 in different ranges of EC

Ameas (mS/m)	R <sub>2</sub> (%)
< 0.5	+ 20 ~ - 20
0.5 ~ 3	+ 13 ~ - 13
> 3	+9 ~ -9

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

#### 3. RESULTS

The Network Center shipped artificial rainwater samples to 27 laboratories in the participating countries of EANET, and received the data on analytical results from all laboratories. Obtained data are summarized in Table 7. Statistics were calculated for each constituent of the artificial rainwater samples such as: Average, Minimum (Min.), Maximum (Max.), Standard deviation (S.D.), and Number of data (N). Outlying data, which are apart from the Average greater than a factor of 3 of S.D. were not included for this calculation. As shown in Table 7, average of submitted data were fairly well agreed with the prepared values/concentrations within a range of -4 to +8%. But there are few laboratories that submitted measured values of considerable differences with prepared concentrations.

Table 7 Summary of analytical results of the artificial rainwater samples (Reported data after removing of outliers)

Constituents	Prepared (Vp)	Average (Va)	Va/Vp (%)	S.D.	N	Min.	Max.
[SampleNo.031] pH	4.52	4.57	1.1	0.09	26	4.40	4.83
EC(mS/m)	3.44	3.32	-3.6	0.22	26	2.78	4.07
$SO_4^{2-}\mu \operatorname{mol/L})$	44.7	43.9	-1.9	2.88	24	36.0	51.1
$NO_3$ ( $\mu$ mol/L)	30.9	31.1	0.7	2.83	25	25.1	38.1
CΓ(μmol/L)	66.0	65.5	-0.7	7.99	25	45.2	86.2
Na <sup>†</sup> (μ mol/L)	46.1	46.2	0.3	5.05	25	33.0	59.7
$K^{\dagger}(\mu \text{ mol/L})$	6.9	6.7	-2.6	0.79	25	4.1	8.8
Ca <sup>2+</sup> (μ mol/L)	20.5	19.8	-3.5	2.13	25	14.8	26.3
$Mg^{2+}(\mu mol/L)$	7.0	6.9	-1.2	0.76	25	5.3	9.2
$NH_4^+(\mu \text{ mol/L})$	48.3	47.0	-2.6	6.02	25	27.9	61.8
[SampleNo.032] pH	4.80	4.86	1.2	0.10	25	4.68	5.11
EC(mS/m)	1.48	1.44	-2.7	0.11	26	1.25	1.79
$SO_4^{2-}\mu  mol/L)$	12.0	12.3	2.6	1.42	24	10.1	17.4
$NO_3(\mu \text{ mol/L})$	21.3	21.4	0.2	1.68	24	18.1	26.3
CΓ(μmol/L)	29.6	29.3	-1.1	3.43	25	23.8	38.8
Na <sup>+</sup> (μ mol/L)	25.6	25.1	-2.0	3.09	25	17.5	33.5
$K^{\dagger}(\mu \text{ mol/L})$	2.5	2.7	8.8	0.44	25	2.2	3.9
Ca <sup>2+</sup> (μ mol/L)	4.4	4.6	3.7	0.70	25	3.6	6.3
$Mg^{2+}(\mu \text{ mol/L})$	3.4	3.6	4.8	0.39	25	2.9	4.3
$NH_4^+(\mu \text{ mol/L})$	15.1	14.9	-1.7	2.99	26	7.3	20.6

(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 data obtained during the preparatory-phase activities of EANET was specified for every constituent as  $\pm 15\%$  by the QA/QC program of the EANET. In this report, analytical data on the artificial rainwater samples were compared with the prepared value/concentration and evaluated by the excess of DQOs value: the flag "E" was put to the data that exceed DQOs by a factor of 2 ( $\pm 15\% \sim \pm 30\%$ ), and the flag "X" was put to the data that exceed DQOs more than a factor of 2 (<-30% or >30%). A set of data for each sample was evaluated by the data checking procedures described in chapter 2.5 .

The flag "I" was put to poor ion balance data sets, and the flag "C" was put to poor conductivity agreement data sets.

The results were evaluated from the three aspects: i) comparison of concentration dependence – sample No.031 (higher concentrations) and No.032 (lower concentrations), ii) comparison of individual parameters, and iii) comparison of circumstances of analysis in each participating laboratory. Evaluation of data on both the sample No.031 and No.032 is presented in "3.1 Comparison by Sample", evaluation of data for each constituent is presented in "3.2 Analytical Parameter", and evaluation of data by the circumstances of analysis such as analytical method used, experience of personnel, and other analytical condition is presented in "3.3 Circumstance of Sample Analysis".

# 3.1 Comparison by Sample

# Sample No.031 (higher concentrations)

Table 8 Numbers of flagged data for the Sample No.031 (higher concentrations)

								<u> </u>			
Flag	рН	EC	SO <sub>4</sub> <sup>2</sup> -	NO <sup>3-</sup>	Cľ	Na⁺	K <sup>+</sup>	Ca <sup>2+</sup>	Mg <sup>2+</sup>	NH₄⁺	Total
E	1	2		5	2	3	1	5	3	3	26
×	0	1	1	0	2	0	2	E 11 S	2	2	11
Data within DQOs	26	24	23	20	21	22	23	20	21	21	221
Flagged(%)	3.7	11.1	8.0	20.0	16.0	12.0	11.5	23.1	19.2	19.2	14.3

(Total data=258)

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

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

For sample No.031(higher concentrations), 26 analytical data out of 258 exceeded the DQOs by a factor of 2 and flagged by "E". 11 analytical data out of 258 exceeded the DQOs more than a factor of 2 and flagged by "X" Data flagged by "E" and "X" were 37 out of 258, shared about 14.3 percents of all reported data for sample No.031 (Fig.2).

Especially measured values of Ca<sub>2</sub><sup>+</sup>, NO<sub>3</sub><sup>-</sup>, Mg<sup>2+</sup>,K<sup>+</sup> and NH<sub>4</sub><sup>+</sup> have many results with flags.

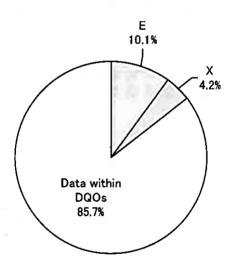


Fig.2 Percentage of flagged data for Sample No.031

Table 9 Analytical Results of Sample No.031

22		9.0	2.3	1.7	80	-0.5	2.5	-2.3	-0.1	0.7	0	9.0-	9.0-	က	4.0	0.	7.1	-0.7	9.0	0.1	4.6	-0.5	6.0	4.0	6	6.5		
쥰	-	0.8	4.8	-0.3	4.0	-7.1	-2.9	-1.6	9.0-	-3.3	1.8	0.3	3.9	9.0-	1.0	-7.2	-7.5	6.0	4. 1-	0.1	4.1	0.0	3.3	4.1	-3.9	8.4		
, ₹	(// mol/L)	45.4	42.6	46.5	48.1	27.9	48.3	46.8	44.1	48.6	47.8	46.3	49.6	48.7	48.9	50.8	61.8	44.3	50.5	48.1	46.4	49.4	56.1	46.3	50.4	80.8	34.7	
Mg <sup>2+</sup>	( / (T mol/√)	7.4	7.5	9.7	6.9	5.3 X	9.2	6.7	7.1	6.1	7.3	6.8	6.8	6.9	7.1	7.5	6.2 E	6.5	6.7	7.1	7.8	6.9	6.9 E	5.6	5.9	7.3 X	22.1 E	N. N.
t, Ca	(π mol/L) (	20.5	21.0	19.9	19.3	14.8 E	20.6 X	18.3	21.3	17.3	19.5	20.1	20.7	19.6	21.8	18.6	26.3	17.4	17.3	18.4	18.7	20.2	20.5	19.6 E	23.0 E	20.1	34.5 X	OK.
	) (π mol/L) (	6.5	9.9	6.5	6.9	6.5 E	7.8	7.2	7.1	6.3 E	6.7	6.5	6.5	4.1	6.7	7.4	8.8 E	6.8 E	6.8 E	7.1	7.6	6.5	6.5	6.3	0.9	6.4	21.2 X	
+ Pa N	(π mol/L) (	47.6	48.7	46.0	48.3	33.0	47.5	46.1	46.3	45.7	49.0	44.4	44.1	44.0 X	46.4	48.7	58.2 E	41.6	39.8	48.2	41.3	45.0	45.9	44.6	45.7	29.7	×	
් ට්	( ποl/L)	63.9	71.8	63.0	63.8	( 45.2 E	75.4	63.9	64.7	68.3	61.3	63.4	56.1	63.2	65.0	80.7	86.2 E	55.5	59.6	69.8	68.9	65.4	64.1	59.1	69.1	70.8 E	1	
SO S	( μ mol/L)	30.6	31.7	31.5	30.5	35.5 X	E 36.6	31.2	30.7	30.2	29.3	29.4	29.3	30.0	30.1	32.5 E	E 38.1 X	27.3 E	31.9	30.5	E 25.1	30.8	29.3	29.6	30.1	E 36.4		
SO4"	(π mol/L)	42.8	48.7	43.6	43.2	E 36.0	44.7	43.2	43.9	45.3	43.5	43.2	41.3	44.5	44.4	45.3	73.5	X 39.4	44.8	42.3	47.4	43.2	42.9	41.9	46.3	51.1 E		-
<u>ျ</u>	(mS/m)	3.26	3.30	3.15	3.15	3.27	3.32	3.37	3.30	3.35	3.34	3.38	3.22	3.47	3.40	3.16	4.07	E 2.99	3.34	3.44	3.17	3.36	3.40	3.60	3.19	3.41	X 11.84	- C
풉		4.56	4.56	4.58	4.58	4.60	4.57	4.59	4.57	4.53	4.57	4.53	4.58	4.52	4.56	4.70	4.40	4.62	4.51	4.51	4.59	4.56	4.55	4.40	4.76	4.54	3.76	00,
Lab. ID		CN01	CN02	CN03	CN04	<u></u>	1002	JP01	JP02	JP03	JP04	JP05	JP06	JP07	JP08	KR01	MY01	MN01	PH01	RU01	RU02	된	1H02		TH05	VN01	KH01 E	700

E:Value exceeded the DQO( $\pm15$ ) by a factor of 2 I:Poor ion balabce(R1) X:Value exceeded the DQO( $\pm15$ ) more than a factor of 2 C: Poor condu

C: Poor conductivity agreement(R2)

## Sample No.032 (lower concentrations)

Table 10 Number of flagged data for the Sample No.032 (lower concentrations)

Flag	рΗ	EC	SO <sub>4</sub> <sup>2</sup>	NO <sup>3-</sup>	Cľ	Na <sup>⁺</sup>	K⁺	Ca <sup>2+</sup>	Mg <sup>2+</sup>	NH₄⁺	Total
Ш	0	2	2	2	1	2	3	4	6	4	26
×	0	-1-	2	1	2	2	5	3	_1	4	21
Data within DQOs	27	24	21	22	22	21	18	19	19	18	211
Flagged(%)	0.0	11.1	16.0	12.0	12.0	16.0	30.8	26.9	26.9	30.8	18.2

(Total data=258)

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

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

For sample No.032 (lower concentrations), 26 analytical data out of 258 exceeded the DQOs by a factor of 2 and flagged by "E". 21 analytical data out of 239 exceeded the DQOs more than a factor of 2 and flagged by "X". Data flagged by "E" and "X" were 47 analytical data out of 258, shared up to 18.2 percents of all reported data for sample No.032 (Fig.3).

Many data on K<sup>+</sup> Ca<sup>2+</sup>, Mg<sup>2+</sup>, NH<sub>4</sub><sup>+</sup> are marked with flags E or flags X.(Table 11)

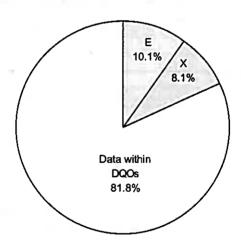


Fig.3 Percentage of flagged data for Sample No.032

#### **Evaluation**

The ratio of flagged data for sample No.031 was 14.3%, and this value was almost the same as the result in the sample No.021(2002). Both of them had almost same concentration for each ion. In the sample with low concentration, the ratio of flagged sate in the sample No.032 was 18.9% and No022(2002) was 29.6%. The concentration of ion in the sample No.032 was higher than the sample No.022 in the range from 1.3 to 5 times.

In general terms, this indicate the difficulty of analysis would depend on the concentration in the sample especially on the trace analysis.

	Mg²+ NH₄+ R1 R2	-   -   (πωσ/L)   -   -	_	-5.9	14.9	16.2 1.2	-31.3	4.3 E 10.7 -2.6 -0.1	3.3 15.2 -2.8 -4.4	3.9 13.2 1.7 -1.5	3.3 16.0 -1.7 0.8	2.2	3.4 16.0 2.8 -0.4	4.3	3.6 E 17.6 1.6 -4.3	3.7 15.8 2.8 -1.4	14.0 -7.9	4.3 X 20.0 -7.1 6.0	13.3 -0.1	3.0 16.9 -1.7 -1.0	3.6 15.9 -1.2 -2.7	4.1 16.5 -1.6 -0.3	3.5 E 11.7 -4.3 -2.0	4.0 E 18.9 6.0 2.3	2.9 14.5 2.4 -1.7	3.1 14.0 -2.3 -1.0	4.0 X 20.6 0.5 -4.9	X 7.3	
12	Ca²⁺	$(\mu \mod L)$	4.2	3.8	4.8	5.0	4.0	6.3	3.6	5.3	4.1	4.8	4.3	4.2	4.0	4.8	6.2	4.9	3.9	3.8	4.0	5.0	4.5	4.0	4.2	5.3	5.1	24.2	
0.03		<u>ځ</u>						×	Ш	Ш							×									Ш	Ш	×	
ample N	±	(# mol/L)	2.6	2.7	2.6	2.7	3.9	3.6	2.7	2.9	2.2	2.5	2.3	2.3	2.4	2.5	3.3	2.9	2.2	2.4	2.5	3.1	2.2	2.4	2.3	2.7	3.3	11.4	
of Sa							×	×		Ш							×	Ш				ш					×	×	
Analytical Results of Sample No.032	Na⁺	( μ mol/L)	26.4	26.6	25.9	26.1	17.5	22.3	25.8	26.1	25.2	26.5	25.0	24.4	26.6	26.2	26.7	33.5	22.0	18.5	26.2	21.7	25.0	24.9	24.0	25.6	29.4	'	
call		_					×											×		Ш		Ш							
Analyti	Ö	(mol/L)	30.3	32.3	30.7	29.8	25.5	28.3	28.9	28.9	30.1	27.3	27.5	23.8	28.5	28.7	38.7	38.8	25.4	26.3	30.5	29.4	28.7	28.2	26.9	29.3	29.9	'	
11		_						_						ш			×	×											
Table 11	NO3.	μ mol/L)	22.0	22.3	22.3	21.9	31.2	18.2	22.0	21.1	22.2	20.7	20.6	20.3	21.0	20.9	23.7	26.3	19.0	21.2	21.5	18.1	22.6	20.3	21.4	20.7	23.0	-	
		_					×											Ш				Ш							
	SO <sub>4</sub> <sup>2-</sup>	(	12.6	13.2	12.8	12.8	17.4	14.4	11.8	11.1	12.6	11.9	11.5	10.6	11.8	11.2	12.9	22.7	10.7	11.2	12.1	12.0	12.9	10.1	12.0	12.0	12.8	٠	
		_	_				×	Ш										×						Ш					L
	EC	(mS/m)	1.41	1.40	1.42	1.44	1.35	1.40	1.48	1.44	1.49	1.43	1.48	1.43	1.53	1.47	1.35	1.79	1.30	1.44	1.53	1.25	1.52	1.37	1.66	1.34	1.43	52.67	100
																		П			-	Ш						×	
	Ηd	1	4.86	4.85	4.85	4.79	5.37	4.86	4.89	4.85	4.78	4.87	4.78	4.87	4.86	4.83	4.94	4.68	4.86	4.79	4.83	5.01	4.80	4.81	4.68	4.94	5.11	4.34	20
									Н												_								_
	Lab		CN01	CN02	CN03	CN04	1001	ID02	JP01	JP02	JP03	JP04	JP05	JP06	JP07	JP08	KR01	MY01	MN01	PH01	RU01	RU02	TH01	TH02	TH04	TH05	VN01	CA01	700

E:Value exceeded the DQO( $\pm$ 15) by a factor of 2 X:Value exceeded the DQO( $\pm$ 15) more than a factor of 2

I:Poor ion balance (R1) C:Poor conductivity agreement (R2)

#### 3.2 Analytical Parameter

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

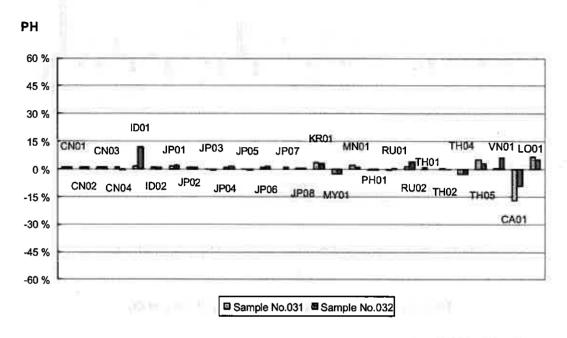


Fig.14 Distribution of pH data normalized by prepared value

Table 12 Analytical method and flagged data of pH

Analytical Method				
pH meter and electrode			27/27	
AUT TO THE PERSONNEL				
Flagged data				
macranical \$15 to	and the second	eta Xanna n	Flagged (%)	
Sample No.031	= 1 1	0	3.7	
Sample No.032	0	0	0	

All participating laboratories used pH meter with glass electrode for measurement of pH. Most of obtained data was agreed with prepared value. Many laboratories submitted slightly higher pH values than prepared value. The relative standard deviations of the pH values for sample No.031 and No.032 were good to be 1.9% and 2.0% correspondently.

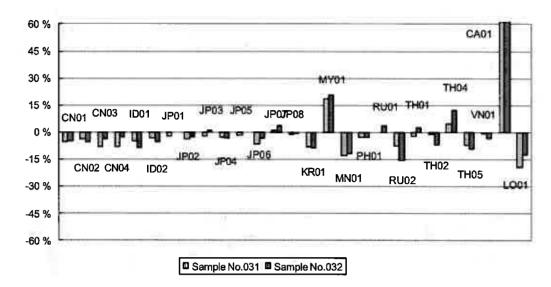


Fig.15 Distribution of EC data normalized by prepared value

Table 13 Analytical method and flagged data of EC

Conductivity meter and cell			27/27	
Sanaad data				
lagged data	E	Х	Flagged (%)	
Sample No.031	E 2	X	Flagged (%)	

All participating laboratories used conductivity cell for the measurement of EC. Obtained data were almost agreed with the prepared values. However, Lab.CA01 submitted results flagged by "X". There would be some problem in measurement. 74% of the Laboratories reported lower data than prepared value for Samples including Sample No.031 and No.032.

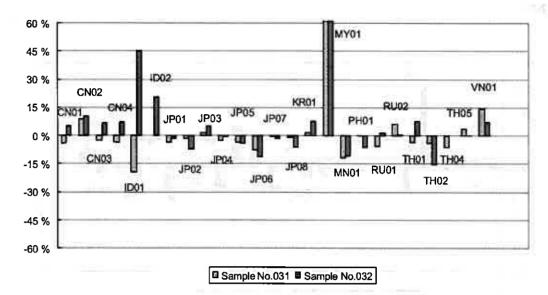


Fig.16 Distribution of SO<sub>4</sub><sup>2-</sup> data normalized by prepared concentration

Table 14 Analytical method and flagged data of SO<sub>4</sub><sup>2</sup>·

Ion chromatography		•	24/25
Other method (Nephelo	ometry) =		1/24
Flagged data			277
	E	X	Flagged (%)
Sample No.031	1	1	8.0
Sample No.032	2	2	16.0

All of the participating laboratories used ion chromatography for the determination of SO<sub>4</sub><sup>2-</sup> except for one laboratory (RU02), which used Nephelometry.

In the Lab.ID01, the straight line was applied as a calibration curve, even though the plot points for the calibration draw a curve. As the result the data of sampleNo.031 and No.032 would exceed the range of DQOs value. This would be improved by using quadratic curve for the calibration curve.

Lab.MY01 reported the values approximately twice as much as the prepared value for both Samples. It seemed to be a problem in the calculations.

Market sealing and a property of the sealing of the

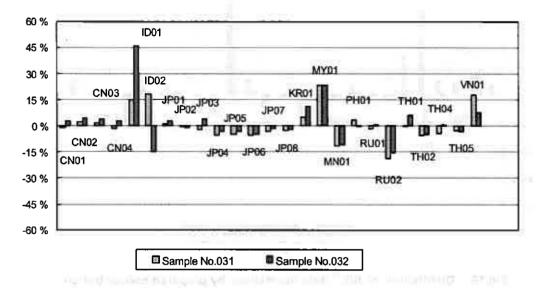


Fig.17 Distribution of NO<sub>3</sub> data normalized by prepared concentration

Table 15 Analytical method and flagged data of NO<sub>3</sub>

Ion chromatography	24/25
Other method (Colorimetry)	1/25

# Flagged data

	E	X	Flagged (%)
Sample No.031	5	0	20.0
Sample No.032	2	1	12.0

The data flagged by "E" were increased approximately twice as much as the project 2002 for the sample with high concentration.

The data of the Lab.ID01 (obtained by ion chromatography) were flagged in Sample No.031 and No.032. The reason would be the same as the data of sulfate. It seemed to be the calibration error.

The results of the Lab.MY01 exceed  $\pm 15\%$  than the prepared value for the both samples No.031 and No.032. The plot point for the calibration is relatively on the calibration curve. So there is a possibility to have some problems in pretreatment of the standard solutions or in the dilution of the artificial rain samples.

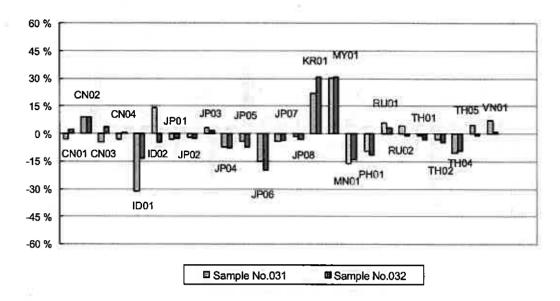


Fig.18 Distribution of Cl data normalized by prepared concentration

Table 16 Analytical method and flagged data of Cl

Ion chromatography	24/25
Titration	1/24

Flagged data

	E	X	Flagged (%)
Sample No.031	2	2	16.0
Sample No.032	1	2	12.0

Same as SO<sub>4</sub><sup>2-</sup> and NO<sub>3</sub>, 24 laboratories used ion chromatography for the determination of Cl. The Lab.RU02 used titration method for the determination of Cl.

As for the sample No.031, the data of four Lab. (ID01,ID02, KR01,MY01) exceeded 15% or 30%. The reason why the data of the Lab ID01 was flagged would be the same as the data of sulfate and Nitrate ion. In the Lab ID02, KR01 and MY01, plot points for the calibration were not on the line. There would be some problems in the preparation of standard solution.

As for the sample No.032, the data of three Lab. (JP06, KR01, MY01) exceeded 15% or 30%. The flagged data for KR01 and MY01 would be caused by the preparation of standard solution.

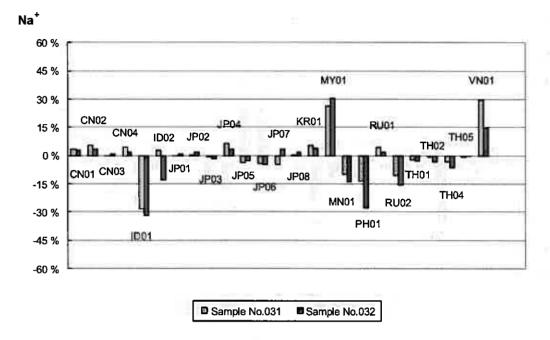


Fig.19 Distribution of Na<sup>+</sup> data normalized by prepared concentration

Table 17 Analytical method and flagged data of Na<sup>+</sup>

Analytical Method	
Ion chromatography	21/25
Atomic absorption spectrometry	3/25
Flame (emission) spectrometry	1/25
Classed data	1 TO TO TO THE LICE

i lagged data			
Tell Have III	E	X	Flagged (%)
Sample No.031	3	0	12.0
Sample No.032	2	2	16.0

21 laboratories used ion chromatography, 3 laboratories used atomic absorption photometry (Lab.KR01, PH01, RU01), and 1 Laboratories used flame (emission) photometry (Lab.RU02) for the determination of Na<sup>+</sup>.

As for the sample No.031, 3 laboratories (Lab. ID01, MY01, VN01) have the date flagged "E" . The plot points not on the calibration curve were observed in the data of ID01 and MY01. Regarding the ID01, quadratic curve for the calibration curve would give good results.

And it is observed in the date from VN01 that peaks were broadened and the separation of the peaks  $Na^{\dagger}$  and  $NH_4^{\dagger}$  were not good enough for the determination.

It seemed to be the same reason explained in the item NO<sub>3</sub>\* that the results of ID01and MY01 exceeded below -30% or above +30% of the prepared value for the sample No.032.

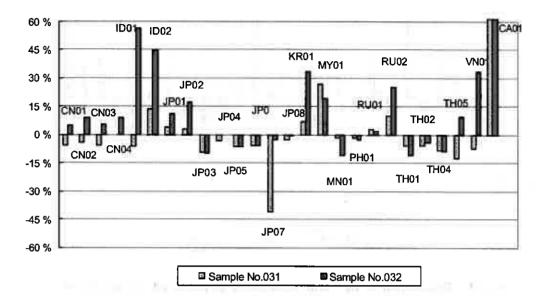


Fig.20 Distribution of K<sup>+</sup> data normalized by prepared concentration

Table 18 Analytical method and flagged data of K<sup>+</sup>

Ion chromatography	22/26
Atomic absorption spectrometry	3/26
Flame (emission) spectrometry	1/26

Flagged data

	Ш	X	Flagged (%)
Sample No.031	1	2	11.5
Sample No.032	3	5	30.8

22 laboratories used ion chromatography, 3 laboratories used atomic absorption and one laboratory used flame (emission) photometry, for the determination of  $K^{+}$ .

There were many flagged data than the result of Na<sup>+</sup>. The concentrations of the sample No.031 and No.032 were almost the same as that of the sample No.021 and No.022 respectively. But the ratio of the flagged data in this project decreased one half for both samples. Obviously there were many flagged data in the sample No.032 of which concentration is low than the sample No.031.

It seemed that the results depend on whether the calibration curve was made precisely or not as explained in parameters SO<sub>4</sub><sup>2-</sup>, NO<sub>3</sub><sup>-</sup>, Cl<sup>-</sup>, and Na<sup>+</sup>.



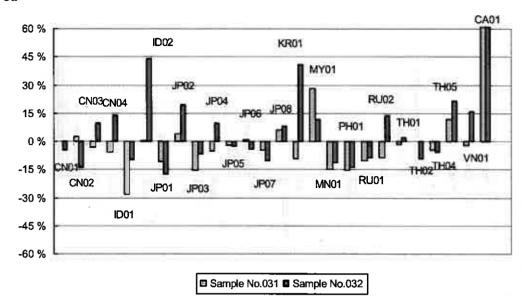


Fig.21 Distribution of Ca<sup>2+</sup> data normalized by prepared concentration

Table 19 Analytical method and flagged data of Ca<sup>2+</sup>

lon chromatography	22/26
Atomic absorption photometry	4/26

Flagged data

	E	X	Flagged (%)
Sample No.031	5	1	23.1
Sample No.032	4	3	26.9

22 laboratories used ion chromatography, 4 laboratories used atomic absorption for the determination of Ca<sup>2+</sup>.

One fourth of the data were flagged in both samples. Though the concentration of  $Ca^{2+}$  (4.4  $\mu$  mol/L) in the sample No.032 is lower than that of the project 2002, ratio flagged date was improved from 41.7%(2002) to 26.9%.

As for the result of ID02 by ion chromatograph in the sample No.032, though the calibration curve were made well and the blank value in the deionized water also was low enough, the data was flagged by "X". There is a possibility to have the problems for preparing the standard solutions.

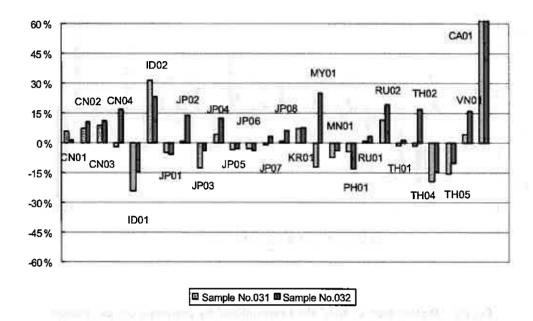


Fig.22 Distribution of Mg<sup>2+</sup> data normalized by prepared concentration

Table 20 Analytical method and flagged data of Mg<sup>2+</sup>

lon chromatography	22/26
Atomic absorption spectrometry	4/26

Flagged data

	Е	X	Flagged (%)
Sample No.031	3	2	19.2
Sample No.032	6	1	26.9

lon chromatography, atomic absorption were used in the analysis of  ${\rm Mg}^{2^+}$  same as for  ${\rm Ca}^{2^+}$ .

One fifth of the data was flagged in the sample No.031 and one fourth of the data was flagged in the sample No.032.

Due to the low concentration of  $Mg^{2+}$  (1.75  $\mu$  mol/L) in the sample No.022 (project 2002), there were many fagged data with the ratio of 54.2%. In this project 2003, the ratio of flagged data decreased to 26.9% by being affected higher concentration with 3.4  $\mu$  mol/L.

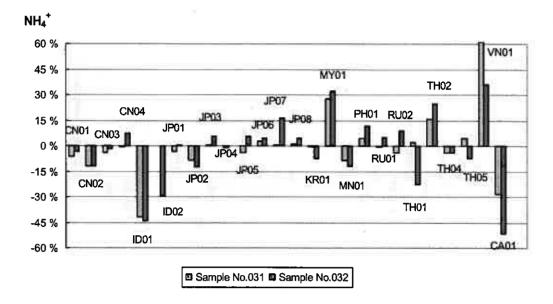


Fig.23 Distribution of NH<sub>4</sub><sup>+</sup> data normalized by prepared concentration

Table 21 Analytical method and flagged data of NH<sub>4</sub><sup>+</sup>

lon chromatography	21/26
Spectrometry (Indophenol blue)	3/26
Other method (Spectrometry)	1/26
Other method (Colorimetry)	1/26

Flagged data

	Е	Х	Flagged (%)
Sample No.031	3	2	19.2
Sample No.032	4	4	30.8

24 laboratories used recommended analytical method of EANET for the determination of NH<sub>4</sub><sup>+</sup>: 21 laboratories used ion chromatography; 3 laboratories used spectrometry (Indophenol blue). one laboratory used spectrometry without using indophenol blue method; one laboratory used Colorimetry.

Both the ratios of flagged data sample No.031 and No.032 were almost the same as the project 2002, even though the concentration of sample No.032(3.4 $\mu$  mol/L) was 3 times as high as the sample No.022 (project 2002).

In the data of ID01, both results were below the -30% of prepared values. The calibration curve was drowned fairly good. This would lead a estimation that injection volume of the sample wasn't exact.

Regarding the data of VN01, the peak of NH<sub>4</sub><sup>+</sup> wasn't separated well from that of Na<sup>+</sup>.

#### **Overall Evaluation**

The concentrations of anions and cations in the samples No.031 were approximately as same as the sample of project 2002 except CI<sup>-</sup> and Na<sup>+</sup>. On the other hand, in regarding to the sample with low concentration, the concentration of ions in the sample No.032 was higher than the project 2002 except Ca<sup>2+</sup> in the range of 1.3 times to 5 times.

The relative standard deviation (R.S.D) of the sample No.031 and No.032 are shown in the figure 14. The R.S.D of each parameter in the sample No.032 with low concentration diminished comparing to the project 2002.

Comparing the ratio of the Flagged data, the ratios of the sample No.021(2002) and the sample No.031, with high concentration were 13.8% and 14.2% respectively. As for the ratio of the sample no.022(2002) and No.032, with low concentration were 29.6% and 18.2% respectively.

As reported in "the Report of the Inter-laboratory Comparison Project 2002 on Wet deposition" ratio of flagged data was affected by the concentration of the ions.

In this project, as mentioned in the chapter 3.2, there are some laboratories having problems in the determination of the ions and the measurement of pH and EC.

Principal cause of the flagged data depends on the calibration curve. The person in charge of analysis should confirm the calibration curve drown on the chart. And before the analysis of the rain samples, the reliability of the calibration should be examined by using the working standard. This practice would avoid the acquisition of low-trust data.

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

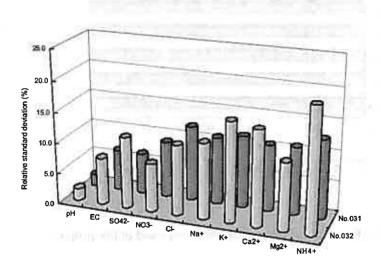


Fig.24 Relative standard deviation of each constituent data

#### 3.3 Circumstance of Sample Analysis

#### **Methods Used**

Colorymetry.

As shown in Fig.25, most of participating laboratories made use of recommended methods of EANET. Regarding the determination of anions, 24/25 laboratories used a ion chromatography.

Nephelometry, Colorymetry, and Titrometration were used in the determination of SO<sub>4</sub><sup>2-</sup>, NO<sub>3</sub><sup>-</sup> and Cl<sup>-</sup> respectively.

As for determination of the catios, 21/25(Na<sup>+</sup>), 22/26(K<sup>+</sup>, Ca<sub>2</sub><sup>+</sup>, Mg<sup>2+</sup>), and 21/26(NH<sub>4</sub><sup>+</sup>) laboratories used an ion chromatography. 3 laboratories used atomic absorption spectrometry (Na<sup>+</sup>, K<sup>+</sup>), 4 laboratories used atomic absorption spectrometry Ca<sub>2</sub><sup>+</sup>, Mg<sup>2+</sup>), Regarding the NH<sub>4</sub><sup>+</sup>, 3 laboratories used the Indophenol Spectrophotometry, and one laboratory used the

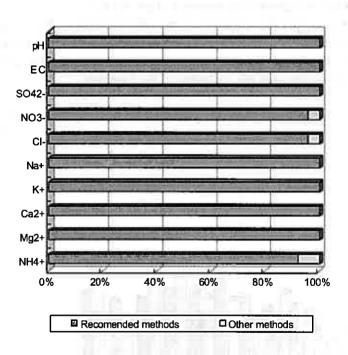


Fig.25 Ratio of recommended method used in the project

Table 22 List of methods

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

Table 23 Number of laboratories used different analytical method

Samo	.I.A.N.I	_	വ വ	
320 M	ne iv	11 1	ו ר.וו	

Method	pН	EC	SO <sub>4</sub> <sup>2</sup>	NO <sub>3</sub> *	Cl <sup>-</sup>	Na⁺	K <sup>†</sup>	Ca <sup>2+</sup>	Mg <sup>2+</sup>	NH₄⁺
0	27(1)	341			E			- "	+ 1	
1		27(3)		_		A				
2			- 6	Ī	_1_					
3						4	3(1)	4(1)	4	
4						1	71		1	
5			24(2)	24(4)	24(4)	21(3)	22(3)	22(5)	22(5)	21(4)
6				7			-	_		
7			1			C		4	1	1(1)
8	<u> </u>					T		1	4-01	3(1)
9	5 4			41=17		71-	-55			J
10		-	100			1		27.0	1.2	Э
Χ			1-11	1(1)			- 7	2.9		1
Flagged E	1	2	_1_	5	2	3	1	5	3	3
Flagged X	0	31	1	0	2	0	2	1	2	2

Sample No.032

Sample INC			1 2.1					74	7.	
Method	pН	EC	SO <sub>4</sub> <sup>2</sup>	NO <sup>3</sup> .	CI.	Na <sup>⁺</sup>	_K <sup>†</sup> _	Ca <sup>2+</sup>	Mg <sup>2+</sup>	$NH_4^{\dagger}$
0	27			#					T-	F
1/2-1	1	27(3)		į						
2		7	71	7	1					
3		4	1-0	-1		3(1)	4(1)	4(1)	4(1)	ì
4	10			Ţ		1(1)	1(1)			
5			24(4)	24(2)	24(3)	22(2)	22(6)	22(6)	22(6)	21(7)
6									<u>.                                    </u>	
7		1.3.3	1							1(1)
8		D= 4	10.00		COMP	الاستان				3(1)
9	·						100			
10										
Х				1(1)						1
Flagged E	0	2	2	2	1	2	3	4	6	4
Flagged X	0	1	2	1	2	2	5	3	1	4

## Number of staff in charge of measurement

The number of staff in charge of measurement on rainwater samples is described in Table 24. In 17 laboratories only one person carried out measurement of rainwater samples. In 6 laboratories two persons carried it. In one laboratory three persons carried it. And in 3 laboratories, three persons carried it.

In the laboratories that more than 2 persons carried out measurement, usually their responsibilities were separated according to the methods used for analysis such as pH and EC-ions(MN01), pH-EC and ions(TH05,VN01).

Table 24 Staff in charge of measurement

			ı a	DIE 24	Stall in	Citatye	OI Intas	uremer			
Lab.ID	Total	pН	EC	SO4 <sup>2</sup>	NO3 <sup>-</sup>	Cl <sup>-</sup>	Na <sup>⁺</sup>	K <sup>+</sup>	Ca <sup>2+</sup>	Mg <sup>2+</sup>	NH4 <sup>†</sup>
CN01		Α	Α	Α	Α	Α	Α	Α	Α	Α	Α
CN02	4	Α	Α	В	В	В	С	C	С	С	C
CN03	1.13	Α	Α	Α	A	A	Α	Α	Α	Α	Α
CN04	1 5	A	Α	Α	Α	Α	Α	Α	Α	Α	Α
ID01	4	Α	В	В	В	С	С	D	D	D	Ä
ID02	2	Α	Α	Α	A	Α	В	В	В	В	В
JP01		Α	Α	Α	A	Α	Α	Α	A	Α	Α
JP02		Α	Α	Α	A	Α	Α	Α	Α	Α	Α
JP03	1	Α	Α	Α	A	Α	A	Α	Α	Α	Α
JP04	C-1-3	Α	Α	Α	A	Α	Α	Α	Α	Α	Α
JP05		Α	Α	Α	l A	Α	Α	Α	Α	Α	Α
JP06	11	Α	Α	Α	A	Α	Α	Α	Α	Α	Α
JP07	1	Α	Α	Α	A	Α	Α	A_	Α	Α	Α
JP08	1	Α	Α	Α	A	Α	Α	Α	Α	Α	Α
KR01	1	Α	Α	Α	A	Α	Α	Α	Α	Α	Α
MY01	2	Α	A	В	В	В	A	A	A	Α	Α
MN01	2	Α	В	В	В	В	В	В	В	В	В
PH01	4	Α	Α	В	В	В	C	C	С	С	D
RU01	3	Α	Α	В	В	В	С	С	С	С	Α
RU02	1	Α	Α	Α	A	Α	Α	Α	Α	Α	Α
TH01	2	Α	В	В	В	В	Α	Α	Α	Α	Α
TH02	_1	Α	Α	Α	l A	Α	Α	Α	A	Α	Α
TH04	1	Α	Α	Α	A	Α	Α	Α	Α	Α	Α
T1H05	2	Α	Α	В	В	В	В	В	В	В	В
VN01	2	Α	Α	В	В	В	В	В	В	В	В
CA01	1	Α	A		WEET 4-			Α	A	A	Α
LA01	1	Α	Α	W.,							

"A", "B", "C", and "D" represent individuals of staff in each laboratory who are in charge of measurement. Reverse mesh: Flagged data of "E" or "X" in sample No.031 and/or sample No.032.

Reverse mesh with dark are flagged data of both sample No.031 and No.032

# Years of experience (Acid rain)

According to information obtained through this project, clear evidence of data quality improvement was not found in terms of "years of experience of the staff", same as previous surveys except CA01 and LA01.

Regarding the Lab.ID CA01 and LA01, the project in this time was the first experience for them. There would be some problems in measurement of pH, EC (and ions).

Table 25 Years of experience

Lab.ID	pН	EC	SO4 <sup>2-</sup>	NO3 <sup>™</sup>	Cl	Na <sup>⁺</sup>	K <sup>+</sup>	Ca <sup>2+</sup>	Mg <sup>2+</sup>	NH4 <sup>†</sup>
CN01	12	12	12	12	12	12	12	12	12	12
CN02	5	5	13	13	13	2	2	2	2	2
CN03	5	5	5	5	5	5	5	5	5	5
CN04	8	8	8	8	8	8	8	8	8	8
ID01	1	4	4	4	4	43	4	4	4	4
ID02	6	6	6	6	6	-3	3	3	3	3
JP01	19	19	19	19	19	19	19	19	19	19
JP02	2	2	2	2	2	2	2	2	2	2
JP03	6	6	6	6	6	6	6	6	6	6
JP04	1	1	1	1	_1_	1	1.	1	1	_ 1_
JP05	_ 1	_1_	1_1_	_1_	1	1	1	1_	1_1_	1_
JP06	_1_1_	_1_	1	_1_	1	1	_1_	1	1_1_	1_
JP07	1-1	1	1	1	1	1	10	1	1	1
JP08	2	2	2	2	2	2	2	2	2	2
KR01	3	3	3	3	3	3	3	3	3	3
MY01	5	5	1	1		5	5	5	5	5
MN01	6	6	6	6	6	6	6	6	6	6
PH01	4	4	2.5	2.5	2.5	7	7	7	7	2
RU01	1	1	6	6	6	4	4	4	4	1
RU02	31	31	31	31	31	31	31	31	31	31
TH01	6	1	1	1	1	6	6	6	6	6
TH02	7	8	2	2	2	2	2	2	2	2
TH04	2	2	2	2	2	2	2	2	2	2
TH05	1	1	1	1	1	1	1		[E1]	-1
VN01	1	1	18	18	18	18	18	18	18	18
CA01	1	1			<u> </u>		1	11	4	1
LO01	1 1	-1								_

Unit: year

Reverse mesh: Flagged data of "E" or "X" in sample No.031 and/or sample No.032 Reverse mesh with dark are flagged date of both sample No.031 and No.032

<sup>1</sup> year means experience with one year or less

# The number of flagged data in laboratories.

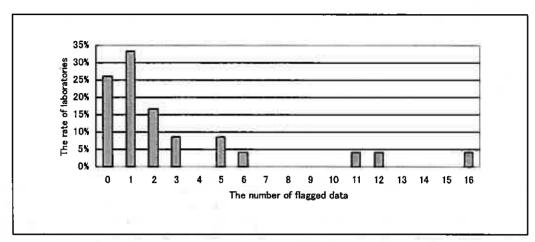


Fig.26 The distribution of laboratories with the number of flagged data

Number of flagged data	Number of laboratories	Share
0	7	26%
1	8	33%
-2	4 - 4	<b>-17%</b>
3	2	8%
4	0	0%
5	2	8%
6	1	4%
7	0	0%
8	0	0%
9	0	0%
10	0	0%
11	1	4%
12	1	4%
13	0	0%
14	0	0%
15	0	0%
16	1	4%

Table 26 Number of flagged data in each laboratory.

In this project, the total number of flagged data was 84(E52, X32) among the whole set of 516 data. The attribution of flagged data in each laboratory was presented in Table 26.

The rumber of excellent laboratories without flagged data was 7, which was equivalent to about 26% of the all-participating laboratories. The number of laboratories that submitted less than 2 flagged date were 6(24%) during the comparison test carried out in 2002, but there were 19 (76%) laboratories this time.

There were three laboratories that produced more than 11 flagged data. These laboratories should make more efforts for preparing standard solutions and also for the operation of the equipment.

# Water temperature at measurement (pH and EC)

As described in Table 27, 19(70%) laboratories measured pH and EC at temperature around 25 °C as recommended condition by EANET. One laboratory was found with the data flagged by "E" on pH measurement. As for the EC measurement, 4 laboratories had the data with flagged by "E" or "X". Unfortunately, even though measure temperature was around 25 °C , 2 laboratories had the flagged data in EC measurement.

Table 27 Water temperature at measurement (pH and EC)

Lab.ID	pH No.031	pH No.032	EC No.031	EC No.032
CN01	25	25	25	25
CN02	21	21	21	21
CN03	15.0	15.0	15.0	15.0
CN04	23.0	23.2	23.1	23.2
ID01	25	25	25	25
ID02	25.0	25.0	25.0	25.0
JP01	23.6	24.1	23.4	23.9
JP02	25.0	25.0	25.0	25.0
JP03	24.8	24.8	24.5	24.5
JP04	24.6	24.6	24.6	24.6
JP05	23.7	23.7	23.7	23.7
JP06	25.0	25.0	25.0	25.0
JP07	24.6-25.0	24.6-25.0	24.6-25.0	24.6-25.0
JP08	25.0	25.0	25.0	25.0
KR01	25	25	25	25
MY01	23.1	23.2	24.8	24.8
MN01	25	25	25	25
PH01	24.9	24.9	24.7	25.2
RU01	25	25	25	25
RU02	25	25	25	25
TH01	25	25	25	25
TH02	25	25	25	25
TH04	25	25	25	25
TH05	25	25	25	25
VN01	25	25	25	25
CA01			i	
LO01			THE WAY WE TH	

Unit: degrees centigrade

Reverse mesh with light are flagged data of "E" Reverse mesh with dark are flagged date of "X"

comment: The measurement temperatures of CA01 and LO01 are now asking for them.

# 4. COMPARISON OF 1st, 2nd, 3rd, 4th AND 5th SURVEY

The inter-comparison surveys were carried out 6 times, so far their results of the number of flagged data are shown in Fig. 27. The rate of data that satisfied the required data quality objectives (DQOs) increased from 75-78% to 84-93% until the  $4^{th}$  (2001) survey. The data quality seemed to be improved by accumulating experiences. But on the  $5^{th}$  project (2002), both DQOs on the sample No.01 (High concentration) and the sample No.02(Low concentration) decreased because the ion concentrations were a half of their content in the samples of previous projects.

In the sample No.01 (correspond to the sample NO.031 on 6<sup>th</sup> project), DQOs of 6<sup>th</sup> project was almost the same as the project 5<sup>th</sup> which the concentration is almost the same. On the other hand, in the sample No.02 (correspond to the sample NO.032 on 6<sup>th</sup> project), the DQOs increased, it seems that it would be the reason for the sample No.032 that the concentration of ions were higher than the sample No.022 already mentioned in "Overall Evaluation".

Considering that the concentrations of the ions in the sample No.032 were still lower than the sample on 4<sup>th</sup> project, even though the DQOs was almost the same, it seems that the level for the analysis of ions was improved in many laboratories.

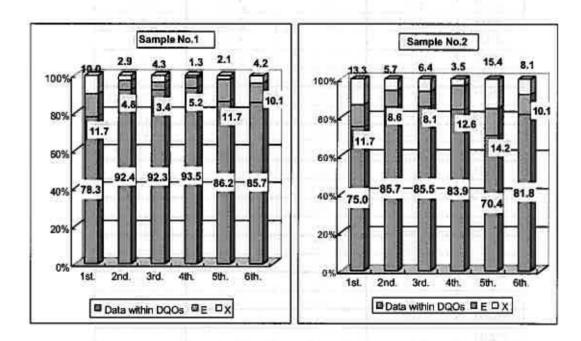


Fig. 27 Comparison of 1st, 2nd, 3rd, 4th and 5th Inter-laboratory comparison project

Table 28 The prepared values of each parameter in artificial rainwater of inter

-laboratory comparison projects of EANET

		рН	EC	SO42-	NO3-	CI-	Na+	K+	Ca2+	Mg2+	NH4+
		(-)	(mS/m)	( <u>  (µmol/l</u> )	(μmol/l)	(µmol/l)	(µmol/l)	(µmol/l)	(µmol/l)	(µmol/l)	(µmol/l)
1000	No.981	4.05	7.94	83.5	93.3	129	95.8	11.1	41.1	13.1	84.8
1998	No.982	4.51	2.82	29.1	36.1	45.1	33.5	7.42	14.3	4.6	29.5
1999	No.991	4.14	6.38	67	75.0	104	77.0	8.9	33.0	11.0	68.0
1999	No.992	4.59	2.30	24.0	27.0	38.0	28.0	3.2	12.0	3.8	25.0
2000	No.001	4.10	6.23	59.7	63.3	101.3	51.3	9.9	29.4	11.7	60.5
2000	No.002	4.85	1.55	20.1	27.5	15.5	8.7	4.9	11.0	7.8	18.2
2001	No.011	4.10	7.45	85.0	93.3	108.4	68.4	15.8	41.1	18.7	87.8
2001	No.012	4.82	1.76	21.5	19.4	34.4	27.4	4.00	13.2	3.7	16.7
2002	No.021	4.30	3.75	40.3	51.0	33.7	13.7	6.92	19.1	7.02	42.4
2002	No.022	5.15	0.69	8.88	8.49	9.13	5.13	1.98	6.6	1.75	4.54
2003	No.031	4.52	3.44	44.7	30.9	66.0	46.1	6.9	20.5	7.0	48.3
2003	No.032	4.80	1.48	12.0	21.3	29.6	25.6	2.5	4.4	3.4	15.1

# 5. FOR IMPROVEMENT OF MEASUREMENT PRECISIONS

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

## 5.1 Fundamental measurement and analysis matters

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

#### 1) Deionized water

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

#### 2) Certified materials and certified samples

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

#### 3) Pretreatment of samples at analytical laboratory

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

#### 4) Adjustment of analytical instruments

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

#### 5.2 Evaluation of reliability

#### 1) Sensitivity fluctuation of analytical instruments

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

### a) For example, Ion chromatography

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

short time, the reasons should be found and removed, then the reference material must be measured again.

#### 5.3 Data control

#### 1) Data check in analysis organizations

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

#### 6. REFERENCES

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   The Second Interim Scientific Advisory Group Meeting of Acid Deposition Monitoring
   Network in East Asia.
- 2) Technical Documents for Wet Deposition Monitoring in East Asia
  - · Technical Manual for Wet Deposition Monitoring in East Asia
  - Quality Assurance / Quality Control (QA/QC) Program for the Preparatory-Phase Wet Deposition Monitoring in East Asia, March 2000 adopted at:
     The Second Interim Scientific Advisory Group Meeting of Acid Deposition Monitoring Network in East Asia.
- Report of the Inter-laboratory Comparison Project 1998 (Round robin analysis survey 1<sup>st</sup>.Attempt) November 1999.
- Report of the Inter-laboratory Comparison Project 1999 (Round robin analysis survey 2<sup>nd</sup>. Attempt) October 2000
- Report of the Inter-laboratory Comparison Project 2000 (Round robin analysis survey 3<sup>rd</sup>. Attempt) October 2001
- Report of the Inter-laboratory Comparison Project 2001 (Round robin analysis survey 4<sup>th</sup>. Attempt) November 2002
- Report of the Inter-laboratory Comparison Project 2002 (Round robin analysis survey 4<sup>th</sup>. Attempt) November 2003
- 8) ICP-WATERS REPORT, 74/2003 (Interconparison 0317) International Cooperative Programme on Assessment and Monitoring of Acidification of Rivers and Lakes, Norwegian Institute for Water Research September 2003

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# APPENDIX 2 Original data

Sample No.031 (higher concentrations)

	рН	EC	SO <sub>4</sub> 2-	NO <sub>3</sub>	Ct	Na⁺	K⁺	Ca <sup>2+</sup>	Mg <sup>2</sup> *	NH₄⁺
	-	mS/m	µmol/L	µmol/L	µmol/L	µmol/L	µmol/L	µmol/L	µmol/L	µmol/L
CN01	4.56	3.26	42.8	30.6	63.9	47.6	6.5	20.5	7.4	45.4
CN02	4.56	3.30	48.7	31.7	71.8	48.7	6.6	21.0	7.5	42.6
CN03	4.58	3.15	43.6	31.5	63.0	46.0	6.5	19.9	7.6	46.5
CN04	4.58	3.15	43.2	30.5	63.8	48.3	6.9	19.3	6.9	48.1
ID01	4.60	3.27	36.0	35.5	45.2	33.0	6.5	14.8	5.3	27.9
ID02	4.57	3.32	44.7	36.6	75.4	47.5	7.8	20.6	9.2	48.3
JP01	4.59	3.37	43.2	31.2	63.9	46.1	7.2	18.3	6.7	46.8
JP02	4.57	3.30	43.9	30.7	64.7	46.3	7.1	21.3	7.1	44.1
JP03	4.53	3.35	45.3	30.2	68.3	45.7	6.3	17.3	6.1	48.6
JP04	4.57	3.34	43.5	29.3	61.3	49.0	6.7	19.5	7.3	47.8
JP05	4.53	3.38	43.2	29.4	63.4	44.4	6.5	20.1	6.8	46.3
JP06	4.58	3.22	41.3	29.3	56.1	44.1	6.5	20.7	6.8	49.6
JP07	4.52	3.47	44.5	30.0	63.2	44.0	4.1	19.6	6.9	48.7
JP08	4.56	3.40	44.4	30.1	65.0	46.4	6.7	21.8	7.1	48.9
KR01	4.70	3.16	45.3	32.5	80.7	48.7	7.4	18.6	7.5	48.1
MY01	4.40	4.07	73.5	38.1	86.2	58.2	8.8	26.3	6.2	61.8
MN01	4.62	2.99	39.4	27.3	55.5	41.6	6.8	17.4	6.5	44.3
PH01	4.51	3.34	44.8	31.9	59.6	39.8	6.8	17.3	6.7	50.5
RU01	4.51	3.44	42.3	30.5	69.8	48.2	7.1	18.4	7.1	48.1
RU02	4.59	3.17	47.4	25.1	68.9	41.3	7.6	18.7	7.8	46.4
TH01	4.56	3.36	43.2	30.8	65.4	45.0	6.5	20.2	6.9	49.4
TH02	4.55	3.40	42.9	29.3	64.1	45.9	6.5	20.5	6.9	56.1
TH04	4.40	3.60	41.9	29.6	59.1	44.6	6.3	19.6	5.6	46.3
TH05	4.76	3.19	46.3	30.1	69.1	45.7	6.0	23.0	5.9	50.4
VN01	4.54	3.41	51.1	36.4	70.8	59.7	6.4	20.1	7.3	80.8
CA01	3.76	118.37					21.2	34.5	22.1	34.7
LO01	4.83	2.78						THE RESERVE	7715	
Prepared va	4.52	3.44	44.7	30.9	66.0	46.1	6.9	20.5	7.0	48 3
Data count	27	27	25	25	25	25	26	26	26	26
Average	4.54	7.58	45.05	31.13	65.53	46.23	7.28	20.36	7.50	48.32
Minimum	3.76	2.78	36.00	25.10	45.23	32.97	4.07	14.76	5.31	27.95
Maximum	4.83	118.37	73.50	38.10	86.20	59.70	21.20	34.50	22.10	80.80
Standard de	0.18	21.73	6.46	2.83	7.99	5.05	2.89	3.52	3.01	8.78

Sample No.032 (lower concentrations)

Lab. ID	pН	EC	SO <sub>4</sub> 2-	NO <sub>3</sub> .	Cr	Na⁺	K*	Ca <sup>II+</sup>	Mg <sup>2+</sup>	NH₄⁺
		mS/m	µmol/L	µmol/L	µmol/L	µmol/L	μmol/L	µmol/L	µmol/L	µmol/L
CN01	4.86	1.41	12.6	22.0	30.3	26.4	2.6	4.2	3.5	14.6
CN02	4.85	1.40	13.2	22.3	32.3	26.6	2.7	3.8	3.8	13.3
CN03	4.85	1.42	12.8	22.3	30.7	25.9	2.6	4.8	3.8	14.9
CN04	4.79	1.44	12.8	21.9	29.8	26.1	2.7	5.0	4.0	16.2
ID01	5.37	1.35	17.4	31.2	25.5	17.5	3.9	4.0	2.9	8.4
ID02	4.86	1.40	14.4	18.2	28.3	22.3	3.6	6.3	4.3	10.7
JP01	4.89	1.48	11.8	22.0	28.9	25.8	2.7	3.6	3.3	15.2
JP02	4.85	1.44	11.1	21.1	28.9	26.1	2.9	5.3	3.9	13.2
JP03	4.78	1.49	12.6	22.2	30.1	25.2	2.2	4.1	3.3	16.0
JP04	4.87	1.43	11.9	20.7	27.3	26.5	2.5	4.8	3.9	15.1
JP05	4.78	1.48	11.5	20.6	27.5	25.0	2.3	4.3	3.4	16.0
JP06	4.87	1.43	10.6	20.3	23.8	24.4	2.3	4.2	3.3	15.8
JP07	4.86	1.53	11.8	21.0	28.5	26.6	2.4	4.0	3.6	17.6
JP08	4.83	1.47	11.2	20.9	28.7	26.2	2.5	4.8	3.7	15.8
KR01	4.94	1.35	12.9	23.7	38.7	26.7	3.3	6.2	3.7	14.0
MY01	4.68	1.79	22.7	26.3	38.8	33.5	2.9	4.9	4.3	20.0
MN01	4.86	1.30	10.7	19.0	25.4	22.0	2.2	3.9	3.3	13.3
PH01	4.79	1.44	11.2	21.2	26.3	18.5	2.4	3.8	3.0	16.9
RU01	4.83	1.53	12.1	21.5	30.5	26.2	2.5	4.0	3.6	15.9
RU02	5.01	1.25	12.0	18.1	29.4	21.7	3.1	5.0	4.1	16.5
TH01	4.80	1.52	12.9	22.6	28.7	25.0	2.2	4.5	3.5	11.7
TH02	4.81	1.37	10.1	20.3	28.2	24.9	2.4	4.0	4.0	18.9
TH04	4.68	1.66	12.0	21.4	26.9	24.0	2.3	4.2	2.9	14.5
TH05	4.94	1.34	12.0	20.7	29.3	25.6	2.7	5.3	3.1	14.0
VN01	5.11	1.43	12.8	23.0	29.9	29.4	3.3	5.1	4.0	20.6
CA01	4.34	52.67					11.4	24.2	16.4	7.3
LO01	5.06	1.30								9 , 20
repared va	4.80	1.48	12.0	21.3	29.6	25.6	2.5	4.4	3.4	15.1
Data count	27	27	25	25	25	25	26	26	26	26
Average	4.86	3.34	12.7	21.8	29.3	25.1	3.0	5.3	4.1	14.9
/linimum	4.34	1.25	10.1	18.1	23.8	17.5	2.2	3.6	2.9	7.3
Maximum	5.37	52.67	22.7	31.2	38.8	33.5	11.4	24.2	16.4	20.6
Standard de	0.17	9.68	2.47	2.53	3.34	3.09	1.73	3.84	2.49	2.99

# APPENDIX3 Normalized values by prepared value

Normalized by prepared value Sample No.031

Lab. ID	рН	EC	SO <sub>4</sub>	NO <sub>3</sub>	Cr	Na⁺	-K⁺	Ca <sup>2</sup>	Mg²⁺	NH₄⁺
CN01	100.9	94.8	95.7	99.0	96.8	103.3	94.2	100.0	105.7	94.0
CN02	100.9	95.9	108.9	102.6	108.8	105.6	95.7	102.4	107.1	88.2
CN03	101.3	91.6	97.4	101.8	95.5	99.8	94.5	97.1	108.7	96.2
CN04	101.3	91.6	96.6	98.6	96.7	104.7	100.0	94.3	98.0	99.6
ID01	101.8	95.1	80.5	115.0	68.5	71.5	93.9	72.0	75.8	57.9
ID02	101.1	96.5	100.0	118.4	114.2	103.0	113.5	100.5	131.4	100.0
JP01	101.5	98.0	96.6	100.9	96.9	100.1	104.1	89.3	95.0	96.9
JP02	101.1	95.9	98.2	99.4	98.0	100.4	102.8	103.9	100.9	91.3
JP03	100.2	97.4	101.3	97.7	103.5	99.1	90.9	84.4	87.4	100.6
JP04	101.1	97.1	97.3	94.8	92.9	106.3	97.1	95.1	104.3	99.0
JP05	100.2	98.3	96.6	95.1	96.1	96.3	93.6	98.0	96.9	95.9
JP06	101.3	93.6	92.4	94.8	85.0	95.7	94.5	100.8	97.1	102.7
JP07	100.0	100.9	99.6	97.1	95.8	95.4	59.0	95.6	99.0	100.8
JP08	100.8	98.8	99.3	97.4	98.4	100.6	97.4	106.2	101.2	101.1
KR01	104.0	91.9	101.4	105.3	122.2	105.6	107.2	90.7	107.1	99.5
MY01	97.3	118.3	164.4	123.3	130.6	126.2	127.1	128.3	87.9	128.0
MN01	102.2	86.9	88.1	88.3	84.1	90.2	98.6	84.9	92.9	91.7
PH01	99.8	97.1	100.2	103.2	90.3	86.3	98.6	84.4	95.7	104.6
RU01	99.8	100.0	94.5	98.6	105.7	104.4	102.8	89.8	101.0	99.6
RU02	101.5	92.2	106.0	81.2	104.4	89.6	110.1	91.2	111.4	96.1
TH01	100.9	97.7	96.6	99.7	99.1	97.6	94.2	98.5	98.6	102.3
TH02	100.7	98.8	96.1	94.8	97.1	99.6	94.3	100.1	98.6	116.0
TH04	97.3	104.7	93.8	95.8	89.5	96.7	91.9	95.6	80.6	95.8
TH05	105.3	92.7	103.6	97.5	104.7	99.1	87.4	112.0	84.6	104.3
VN01	100.4	99.1	114.3	117.8	107.3	129.5	92.8	98.0	104.3	167.3
CA01	83.2	3441.0			1		307.2	168.3	315.7	71.8
LO01	106.9	80.8							Section A	100
Minimum	83.2	80.8	80.5	81.2	68.5	71.5	59.0	72.0	75.8	57.9
Maximum	106.9	3441.0	164.4	123.3	130.6	129.5	307.2	168.3	315.7	167.3
Average	100.5	220.2	100.8	100.7	99.3	100.3	105.5	99.3	107.2	100.0

Normalized by prepared value Sample No.032

Lab. ID	pН	EC	SO <sub>4</sub> 2-	NO <sub>3</sub>	Cr	Na*	K,	Ca <sup>2+</sup>	Mg <sup>2+</sup>	NH₄*
CN01	101.3	95.3	105.4	103.1	102.3	103.0	105.3	95.5	101.7	96.6
CN02	101.0	94.6	110.4	104.5	109.0	103.7	109.3	86.4	110.5	88.0
CN03	101.0	95.9	106.8	104.3	103.6	100.9	105.7	109.5	111.3	98.3
CN04	99.8	97.3	107.3	102.7	100.7	101.7	109.3	113.6	116.9	107.3
ID01	111.9	91.2	145.1	146.1	86.1	68.1	156.6	90.1	85.7	55.6
ID02	101.3	94.5	120.4	85.3	95.5	87.0	144.5	143.9	123.5	70.8
JP01	101.9	100.0	98.8	102.9	97.5	100.7	110.9	82.5	94.5	100.5
JP02	101.0	97.3	92.8	98.9	97.5	101.8	117.4	119.3	114.2	87.3
JP03	99.6	100.7	105.4	104.0	101.6	98.3	90.3	93.2	96.2	105.8
JP04	101.5	96.6	99.5	97.0	92.1	103.4	100.0	109.8	112.5	99.9
JP05	99.6	100.0	96.2	96.5	92.8	97.5	93.9	97.3	97.4	105.8
JP06	101.5	96.6	88.6	95.1	80.3	95.2	94.3	96.1	96.5	104.5
JP07	101.3	103.4	98.7	98.4	96.2	103.7	97.6	90.0	103.5	116.4
JP08	100.6	99.3	93.9	97.7	96.7	102.1	99.6	108.2	106.5	104.4
KR01	102.9	91.2	107.9	110.9	130.7	104.1	133.6	140.9	107.6	92.6
MY01	97.5	120.9	189.8	123.2	130.9	130.7	119.0	111.6	125.0	132.3
MN01	101.3	87.8	89.5	89.0	85.7	85.8	89.1	88.6	95.9	88.0
PH01	99.8	97.3	93.6	99.3	88.8	72.2	97.2	86.4	87.2	111.8
RU01	100.6	103.4	101.3	100.7	103.0	102.1	102.0	91.4	103.2	105.0
RU02	104.4	84.5	100.3	84.8	99.2	84.6	125.5	113.6	119.2	109.1
TH01	100.0	102.7	107.9	105.9	96.9	97.5	89.1	102.3	101.7	77.4
TH02	100.2	92.6	84.5	95.2	95.3	97.0	96.0	90.7	116.9	124.7
TH04	97.5	112.2	99.9	100.5	90.8	93.5	91.1	94.3	85.5	95.8
TH05	102.9	90.5	100.2	97.0	99.0	99.7	109.7	121.4	90.1	92.6
VN01	106.5	96.6	107.0	107.8	100.9	114.7	133.6	115.9	116.3	136.2
CA01	90.4	3558.8					461.5	550.0	476.7	48.3
LO01	105.4	87.5					constant			
Minimum	90.4	84.5	84.5	84.8	80.3	68.1	89.1	82.5	85.5	48.3
Maximum	111.9	3558.8	189.8	146.1	130.9	130.7	461.5	550.0	476.7	136.2
Average	101.20	225.51	106.04	102.04	98.93	97.96	122.39	120.86	119 09	98.26