Mission Report to Japan 27 July to 10 August, 2014



By

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CRH - IRGM

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Introduction

Within the framework of SATREPS Ny-Mo project, two researchers of Institute for Geological and Mining Research (IRGM), Dr. Festus Aka and M. Djomou Serges, visited Japan for a scientific mission from July 27 to August 10, 2014. During the mission they attended the 11th annual meeting of Asian Oceania Geosciences Society (AOGS 2014) meeting at Sapporo and visited some universities in Japan for training and scientific exchange.

The mission made by M. Djomou Serges had two main goals:

- Participate in AOGS 2014 in Sapporo
- Acquire training on the preparation and cooking data of the syringe method (MK method) and the method to recover the data from Automatic Observation Buoy (AOB) in Ohba Laboratory at Tokai University.

This training is based on:

- One objectively verifiable indicator (Lake observation) and one mean of verification (Hard copy and electric file on web site) of the PDM of the project
- One recommendation (IRGM team members henceforth shall spearhead all future field activities under the supervision of their Japanese counterparts) made in the March 2014 Joint Coordinating Committee (JCC) meeting.

I. Schedule of mission

25th July: TK669 departure from Nsimalen to Istanbul (23:55)

26th July: TK669 Istanbul arrival, TK50 departure to Narita

27th July: TK50 Narita arrival, move to Sapporo by use of a domestic plane

28th July to 1st August: AOGS Sapporo

2nd August: move from Sapporo to Tokyo and Tokai University by use of a domestic plane

3rd to 9th August: Training of MK method and treatment of CTD data at the Laboratory of Tokai University

10th August: TK51 departure at Narita to Istanbul

11th August: TK669 departure at Istanbul to Nsimalen (23:05)

II. 11th Annual Meeting of Asian Oceania Geosciences Society (AOGS2014)

AOGS is an international scientific association to promote geosciences and its application for the benefit of humanity, specifically in Asia and Oceania an overarching approach to global issues. The 2014 meeting took place in Sapporo, Japon at the Royton Sapporo Hotel from 28 July to 01 August 2014.

AOGS 2014 has 8 sections: Atmospheric Sciences, Biogeosciences, Hydrological Sciences, Interdisciplinary Geosciences, Ocean Sciences, Planetary Sciences, Solar & Terrestrial Sciences, Solid Earth Sciences (fig. 1). Twelve participants from SATREPS-NyMo project including 6 Cameroonians (2 researchers from IRGM and 4 Cameroonian students on SATREPS PhD programs in Japanese Universities) participated at this meeting with 6 oral and 3 poster presentations made (Table 1). Five oral presentations were made during the session of "*Prevention of the next liminic eruption at Lake Nyos and Monoun*" chaired by Professor Takeshi Ohba (leader of the project) and Dr. Issa sitting in for Dr Tanyileke Gregory (unavoidably absent). The session was included in the Interdisciplinary Geosciences section. One oral presentation was made during the session of "*Impact, Response and Risk on Water resources Management*" included in the Hydrological Sciences.



Figure1. Attendee Profile by section



Photo 1: Group photo of some member of SATREPS-NyMo project participant to the AOGS 2014 (from left to right: Issa, Chako, Djomou, Ohba, Assobo, Kusakabe, Aka, Ooki and Jude)



Photo 1: (a) poster set-up at the Exhibition Hall, (b) The leader of SATREPS-NyMo project with the two researchers of IRGM and two PhD students in Japan during the presentation of the poster at the Exhibition Hall

N°	Presenter	Code	Title
1	Mengnjo Jude	HS14-A001	Hydrochemical and isotopic characteristics of
	Wirmvem		groundwater in the Ndop Plain, North West
	(Oral)		Cameroon: Resilience to seasonal climatic
			changes
2	Asobo NE Asaah	IG25-A004	Geochemistry of volcanic rocks in Lakes Nyos
	(Poster)		and Monoun, Including other Lakes of the Oku
			Volcanic Group (OVG) on the Cameroon
			Volcanic Line
3	Kazuto Saiki	IG25-A005	Development of the measuring Method of
	(Poster)		Dissolved CO2 concentration in Cameroonian
			Volcanic Lakes using sound velocity of lakes
			water
4	Djomou B Serges L	IG25-A008	Preliminary results from the lakes Monoun and
	(Poster)		Nyos climate stations (annex I)
5	Minoru Kusakabe	IG25-A001	Measures to prevent the recurrence of limnic
	(Oral)		eruptions at Lakes Nyos and Monoun (Cameroon)
6	Aka Festus	IG25-A002	Preventing limnic eruptions in Lakes Nyos and
	(Oral)		Monoun within the framework Disaster
			Governance, Resilience and Preparedness in
			Cameroon
7	Takeshi Ohba	IG25-A003	Temporal variation of amount of CO2 dissolved in
	(Oral)		Lakes Nyos and Monoun, Cameroon
8	Issa	IG25-A006	Geochemistry of soil gas from Mount Manenguba
	(Oral)		cadera, Cameroon Volcanic Line (CVL)
9	Yosuo MIYABUCHI	IG25-A007	Pyroclastic sequence in and around Lake Nyos,
	(Oral)		Northwestern Cameroon

Table 1: Presentations made at the AOGS 2014 meeting by SATREPS-NyMo members

III. Training in the Tokai University laboratory

The training at the Tokai University laboratory was organized from August 4 to 9, 2014. During this period, we worked with Prof Obha, Seigo Ooki, Issa, Yu Oginuma and T Ohsumi.

III.1. Preparation and cooking data of the syringe method (MK method)

The syringe method is a rapid and cost-effective approach to measure CO_2 concentrations in the lake water (Kusakabe et *al.*, 2000). With this method, the total dissolved carbonate $(H_2CO_3 + HCO_3 - + CO_3^{2-})$ was fixed *in situ* in a plastic syringe containing a concentrated solution of KOH and later determined in the laboratory using micro-diffusion analysis. The $CO_2(aq)$ (or H_2CO_3) concentration was obtained by subtracting HCO_3^- concentration from the total carbonate concentration. Because of limited time, it was not possible for us to train on the sampling procedure. After the preparation of syringe and solution, one sample collected with syringe method at Lake Nyos in March 2014 was used for training on the preparation of the sample and the titration methods at laboratory.

Annex II shows the description of the preparation and cooking data of the syringe method write after this training.

III.2. Recover the data from Automatic Observation Buoy (AOB)

After the training of the MK method, we have trained on the method of recover data from AOB. In March 2014, an AOB was installed in Lake Nyos. It measures the temperature and conductivity of lake water at various depths automatically once per hour. The data measured is transmitted to satellite which relays it in real time through the Gmail account. This data are recovered by a PC through software named "tsBase". The aims of the training are to familiarize ourselves with the "tsBase" program and to learn how to recover the data.

For this training, Professor Ohba was give us a donated PC and USB key with "tsBase" program intended for collection of data from AOB at IRGM. Unfortunately, it has not been possible to use "tsBase" program on this PC to collect data because of the incompatibility of the version of Windows (English) installed on the PC and the "tsBase" program developed in Japanese. Consequently, we used a PC from Ohba laboratory for the training.

The first part of the work was to consult one of the Gmail account (satreps.nymo2@gmail.com) where the data from AOB were sent each hour. From March 8, 2014 to August 7, 2014 (3:33mn, Cameroon time) when we consulted the account, it had received 3786 messages. Each message has 7 parameters:

- Date (year / month / day / hour)
- GPS position of AOB (DMS)
- Conductivity and temperature for each sensor (7 for depths)
- Depth of the deepest sensor
- Temperature of the transmitter
- Battery voltage of AOB

Following consultation of Gmail account, we used the "tsBase" program to download all data sent by AOB of Lake Nyos since it installation. The format of download data is CSV file. The procedure of the downloading is described in Annex 3.

To download data from AOB in Cameroon, Professor Obha recommended that we use the computer of Ms. Inaba which has a Japanese window until the solution of a compatible program is found.

Conclusion

This mission helped us:

- To present the preliminary results from Nyos and Monoun climate stations installed within the framework of the SATREPS-NyMo project in an international meeting
- To improve our knowledge on the MK method and the acquisition of data from AOB. The data transmitted by AOB since it installation (8 March 2014) were downloaded.

During this mission, we wrote a manual of MK sample that will be inserted on the website of the SATREPS-NyMo project. We recommend IRGM to develop a download program data from AOB (similar to "tsBase" program) compatible with English version of windows for solved the problem of incompatibility of "tsBase" program.

Annex I – Djomou Poster at AOGS 2014



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Annex II – How to prepare and cook data of the syringe method

The syringe method is a rapid and cost-effective approach to measure CO2 concentrations in the lake water (Kusakabe et *al.*, 2000). With this method, the total dissolved carbonate $(H_2CO_3 + HCO_3 - + CO_3^{2^-})$ was fixed *in situ* in a plastic syringe containing concentrated solution of KOH and later determined in the laboratory using micro-diffusion analysis. The $CO_2(aq)$ (or H_2CO_3) concentration was obtained by subtracting HCO_3^- concentration from the total carbonate concentration.

Step 1: Pre-sampling preparation

1. Preparation of the syringes

Use 50 ml plastic syringe.

- Remove piston plunger and make 4 cuts to its base, spaced 90 degrees,
- Use drilling machine to make 4 holes on the plunger tip,
- Re-assemble and label them properly





Photo1: (a) piston plunger with the 4 cuts (b) plunger tip with the 4 holes

2. Preparation of the Potassium Hydroxide solution (5 M)

The operation must be performed with great precaution and care (Protect yourself).

KOH salts (85% weight) are used for the preparation.

(1) Determination of the quantity (in grams) of KOH granular

Example: Preparation of 500 ml of KOH solution,

With the equation n=C*V=m/M

where, m: quantity (g) of KOH, (g), M: molar mass (g/mol) of KOH, C: concentration (mol/l) of KOH needed and V: volume (l) of KOH solution needed (l)

m = 5 x 0.500 x 56.10 /0.85 = 165 g

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- (2) Use 165 g of KOH
- (3) Put the salt in the flask or a beaker
- (4) Then put DIW up to 500ml
- (5) stir until complete dissolution
- (6) Wait until the solution cools down before storage in a plastic bottle (protect the solution during cooling to avoid the atmospheric contamination).

3. Put 10ml of KOH solution in each syringe

- (1) Connect the syringe to the 2 ways outlet
- (2) Open the valve and pull 10 ml of the solution
- (3) Push the air at the top of the syringe
- (4) Cap the tip of syringe
- (5) Weight the syringes with solution





Photo 2: (a) syringe connect to the 2 way outlet (b) air in process of being push at the top of the syringe

4. Protection of the syringe tip and storage

Tape the cap and store the syringes in aluminum bag



Photo 3: (a) and (b) cap taped (c) aluminum bag

Set 2: Sampling procedure

1. How to set syringe in MK sampling



Figure 1: Schematic presentation of a syringe sampler

During the sampling, the total dissolved carbonate $(H_2CO_3 + HCO_3 - + CO_3^{2-})$ was fixed (see equation 1) in a plastic syringe containing concentrated solution of KOH.

Equation 1: $2KOH + CO_2 \longrightarrow K_2CO_3 + H_2O$

Step 3: Preparation of the sample at laboratory

1. Determination of the weight of the collected sample

- (1) Clean the syringes using kimwipes
- (2) remove the dirts Remove the tape
- (3) Weigh the syringes
- (4) Determine the amount of lake water collected

 $Weight_{(Sy+KOH+sample)} - Weight_{(Sy+KOH)}$

Step 4: Preparation of titration

1. Dilution of the sample

(1) Prepare a empty plastic bottle of 100 ml

- (2) Put the bottle on the balance and Tare
- (3) Empty the syringe content in the bottle
- (4) Add distilled water up to of 100 mg

2. Preparation of a 0.1mol Hydrochloric Acid solution

(1) Determination of the quantity (in liters) of concentrated HCl solution

Example: Preparation of 500 ml of 0.1 mol/l HCl solution using concentrated HCl (1 mol/l),

With the equation n=C*V

Ci*Vi=Cf*Vf Vi = Cf*Vf/Ci Vi = 0.1 x 0.5 x 1 = $5x10^{-2}$ 1

- (2) Use pipette to take 50 ml of 1mol/l HCl solution
- (3) Put the solution in the flask
- (4) Then put DIW up to 500ml
- (5) Storage the solution into a glass bottle

3. Preparation of saturated Barium Hydroxide Octahydrate solution [Ba(OH)₂]

- (1) Determination of the quantity (in grams) of solid of Ba(OH)₂
- (2) Determined de quantity (in grams) of the Thymol Blue and the Cresol Red reagent

For 1 liter of Ba(OH)₂ solution, use 24 mg of the Thymol Blue reagent and 8 mg of the Cresol Red reagent.

Example: Preparation of 1000 ml of 0.05 mol/l Ba(OH)₂ at 98%

Determination of mass of Ba(OH)₂ with the equation n=C*V=m/M

m=C*V*M

where, m: quantity (g) of solid Ba(OH)2, (g), M: molar mass (g/mol) of [Ba(OH)2.8H2O],

C: concentration (mol/l) of [Ba(OH)₂.8H₂O], and V: volume (l) of KOH solution needed (l)

m = 0.05 x 1 x 315.46/0.98 = 16.09 g

- (3) Use 16,09 g of solid Ba(OH)₂, 24 mg of Thymol Blue reagent, 8 mg of Cresol Red reagent
- (4) Crush solid Ba(OH)₂ to a fine powder to facilitate its dissolution (photo 4a)

- (5) Put the solid $Ba(OH)_2$ and reagents in the flask or a beaker
- (6) Then put DIW up to 1000ml
- (7) You obtain a violet solution (Photo 4b)



Photo 4: Preparation of satured Ba(OH)

4. Preparation of Sulfiric Acid solution (10% HSO₄)

The preparation of 10%HSO₄ is based on the same calculation use for the preparation of HCl above.

Step 5: Titration procedure

The micro-diffusion cell analysis is used. The micro-diffusion cell is glass vessel separated in two compartments: An outer (b) and inner (a) (fig. 2)



Figure 2: Micro-diffusion cell (a) inner cell, (b) outer cell

Three aliquots of each sample are titrated

The principle is to evolve the carbonate fixed in the syringe by the KOH during the sampling to diffuse and form barium carbonate which will be titrated.

1. Preparation of the micro-diffusion cells

- (1) Rub a thin film of Vaseline on the ground glass of the cell and on the (photo 5a)
- (2) Pipet 5ml of the Ba(OH)₂ solution and put it into the inner cell (Photo 5b)

- (3) Pipet 1 ml of sample and put it into the outer cell [Avoid atmospheric contamination by isolating the cell (cover) immediately after introducing the solutions]
- (4) Incline the cell and introduce 1 ml of Sulfiric Acid into the outer cell to liberate the CO2 and isolate quickly (photo 5c, equation 2) *Equation 2:* $K_2CO_3 + H_2SO_4 \longrightarrow CO_2 + K_2SO_4 + H_2O$
- (5) Gently swirl the cell to mix the sample and the H_2SO_4 solution
- (6) Allow 1 hour for the reaction to go to completion
- (7) During that time, a film of barium carbonate precipitate may form at the surface of the inner cell (equation 3). Shake the cell slowly and gently to brake it.

Equation 3: $Ba(OH)_2 + CO_2 \longrightarrow BaCO_3 + H_2O$ (inner reaction)

(8) Repeat as necessary



Photo 5: Preparation of the micro-diffusion cells

2. Operational titration

- (1) Start the auto-titration kit and run the acid solution to clear air bubble from the capillary
- (2) Place the micro-diffusion cell under the auto-titration kit (photo 6a)
- (3) Slowly titrate the solution in the inner cell using 0.1 HCl solution until you obtain light yellow coloration
- (4) Use the glass rod to mix the solution while adding Hcl.
- (5) Record the HCl volume required to reach the endpoint (photo 6b)
- (6) Repeat for other samples.





Photo 6: (a) at the beginning of the titration (b) endpoint

Step 6: calculation

Total carbonate ($\sum CO_2$) in the sample was computed as follows (Issa, master thesis).

Total amount carbonate in the plastic bottle (A)

A=0.5*d*f*(b-a)*(e/c) in millimoles

Total carbonate concentration in the lake (C)

C=A*(1000/s) in millimoles/kg

Where:

- ✓ f= concentration factor for 0,1M HCl solution (1,002)
- \checkmark a= Titrated volume of HCl for sample solution (ml)
- ✓ b= Titrated volume of HCl for blank solution (ml)
- \checkmark c= Volume of sample solution introduced into the micro-cell (ml).
- \checkmark d= concentration of standard HCl solution (N)
- \checkmark e= total weight of sample solution in the plastic bottle (g)
- \checkmark s=weight of lake water in the syringe sampled by the MK-sampler (g)

Annex III – How to collect the data from Automatic Observation Buy (AOB)

1. Start "tsBase" program

Open "tsBase" folder. Double click tsBase.exe (state tsBase.exe) to start "tsBase" program.

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<u>Main menu</u>

We use following menus

 $[\mbox{File}\ (\mbox{F}):\mbox{ Open parameter }(\mbox{O})]$ – Open the saved parameter file except for a defaults parameters file

[File (F): Save parameter (S)] – Save the present parameter settings

[File (F): Exit (X)] – Close main window

[Setting (S): Parameter setting (P)] – Set defaults parameter file, network log and saving folder

[Setting (S): Network setting (I)] - Set parameter about data and transceiver

[Tools (T): Receive (R)] – Receive observed data by e-mail

[Tools (T): Drawing figures (P)] – Draw time series graph

[Help (H): Information (A)] – Confirm this system's version

[Tools (T): Manual (Z)] – Open manual file (pdf file)

To open above menus, press key inside the bracket with "Alt" key

For example: if you want to close main windows, firstly press "Alt" and "F" then "X"

Parameter File

Select "parameter file" when you want to use saved one for dealing with observed data.

If you set defaults parameter file, that parameter file will be opened automatically on opening Main Menu.

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Parameter Setting

Set folders to save each type files: Parameter, Network, Received data and customized data. Normally, we set following: D drive – Program file – tsBase – each folders then click ok.

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- 2. Network setting
 - (1) Buoy settings

To set mail information, you can move to setting dialog of buoy from [Setting (S): Network setting (I)].

In the dialog, enter the following information: E-mail address of sender, Character string, Boy name and Parameter file

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(2) Connecting setting

Select connection type

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(3) Mail receiving settings

- Fill the information: Server address, User name, Password and port number

- Select the method to delete received data

- Set the information of automatic receiving setting: Interval time (min), Starting time (min) and retry times when the network connection is not good.

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(4) Mail sending settings

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- 3. Data classification
- $(\ensuremath{\underline{1}})$ choose the month for which you want to display the data
- ① automatically receive button (ON/OFF): data base will update after receiving data
- (2) to change the time is possible to Greenwich time
- (3) to choose the month for which you want to display the data
- 4 to save data in CSV file

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375	2012/01/03 14:20:00	36 54.1485	137 2.6542	11.57	18.7	9.38	8.88	3.01	10.9	
376	2012/01/03 14:30:00	36 54.1459	137 2.6609	11.57	11.6	9.77	9.57	1.96	10.9	
377	2012/01/03 14:40:00	36 54.1485	137 2.6631	11.55	15.3	8.42	8.12	2.22	10.9	
378	2012/01/03 14:50:00	36 54,1438	137 2.6597	11.55	19.0	8.70	8.23	2,83	10.9	
379	2012/01/03 15:00:00	36 54.1408	137 2.6571	11.54	21.6	8.80	8.18	3.24	10.9	
380	2012/01/03 15:10:00	36 54.1372	137 2.6811	11.56	19.3	8.56	8.08	2.83	10.9	
381	2012/01/03 15:20:00	36 54.1427	137 2.6625	11.55	33.8	8.10	6.73	4.51	10.9	1
382	2012/01/03 15:30:00	36 54.1377	137 2.6638	11.57	68.0	3.24	1.21	3.00	10.9	
383	2012/01/03 15:40:00	36 54.0867	137 2.6645	11.49	8.9	1.80	1.78	0.28	10.9	
384	2012/01/03 15:50:00	36 54.1448	137 2.6613	11.53	35.4	2.25	1.83	1.30	10.9	
385	2012/01/03 16:00:00	36 54.1409	137 2.6640	11.56	5.2	2.48	2.47	0.22	10.9	

Receiving of the e-mail data

[Tools (T): Receive (R)], and then select the speed button on the main menu. The following window will be displayed and while the email data will be receive [reception at regular intervals if it is the automatic reception (ON)].

