

Mission Report

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1. Introduction

In this period from 21st Oct. to 14th Nov., we did maintenance of analytic equipment in Nkolbisson Laboratory. We mainly took a long time on AAS (Atomic Absorbance Spectrometer) and PICARRO (Water Isotope Ratio Laser Analyzer). In this report, I will write the problems we found and how they were solved.

2. About AAS

On 21st Oct., the problems were the following:

1. An improper position of C₂H₂ and N₂O gas cylinders
2. Lamp status error
3. Prism correction error
4. No or negative absorbance of some elements

Then, we tried to solve these problems. In this report, I will mainly describe the past and present situation and solving procedures.

2-1. An improper position of C₂H₂ and N₂O gas cylinders

Before coming to Nkolbisson, C₂H₂ and N₂O gas cylinders were kept on the floor with lying on. This situation is dangerous especially in case of N₂O gas cylinder, which is kept with liquid gas condition. The liquid N₂O may leak from port of cylinder and cause damage to regulator. Then, we changed these gas positions in Fig.1. Furthermore, we could see that N₂O gas pressure was increasing when we opened main gas valve. Before changing gas position, we could not confirm the pressure increasing. This indicates that the misunderstanding of no N₂O gas was caused by an incorrect gas cylinder position.

2-2. Lamp status error

The problem indicates that we cannot ignite Xe-lamp and, in other

words, all analysis is impossible. Normally, Xe-lamp is ignited automatically when we start to setup. But, when we started the setup, we could confirm an error message informing cooling water was too low and Xe-lamp did not ignite. Xe-lamp of AAS can not work under the temperature is over 60°C. Then we checked cooling system (Fig.2) and the cooling water was almost empty. After putting tap water, that error message never appeared (Fig 2) and we could proceed to next maintenance. But after two weeks, cooling water decreased again to half volume. This means we need to constantly check cooling system.

2-3. Prism correction error

This error is also a serious problem and sometimes happens with Na and K whose primary wavelength for analysis is longer than that of other elements. This error may relate to spectrometer conditions and we need to check every time to get good results. To identify if prism error occurred or not, we use “Spectrometer” in the top window of Aspect CS. In the case of Nkolbisson, only K had a prism error. If we can see prism error only elements with long primary wavelength for analysis, spectrometer settings have a little problem. So, we did new parameter setting procedure. After this procedure, spectrometer condition was recovered. Now some LAGE members already learned how to solve prism error.

2-4. No or negative absorbance in some elements

Two reasons may cause this problem. One is we did not use the appropriate gases and another reason is we analyze without making appropriate methods condition in “Methods” menu. Such problems happen especially in Al, Ca and Si, which need a high-temperature flame. In fact, after solving lamp status error and prism error, we tried to make calibration line of these three elements. But we could not get good calibration line and I suspected that flame condition was not optimized under the recommended conditions in Cookbook, which has recommended condition of each element by AnalyticJena. But all these conditions cannot be applied to Nkolbisson’s AAS because optimized condition depends on each machines condition. For

example, spectrum condition and valve condition inside machine. Among the parameter in “Methods”, amounts of fuel gas is the most important factor to find the appropriate condition. In case of Si, however with the recommended fuel rate of 250L/h, all absorbance data were negative. Then, we changed to 265L/h and again tried to make a calibration line. At this experiment, we could get very good calibration line ($R^2 > 0.99$). From this problem, we could understand that we need to check the most optimized flame condition with trying various fuel amounts to get more accurate data.

2-5. Other maintenance procedure

This time, I also showed some maintenance procedure as follows:

- ✧ Remove and install procedure of Xe-lamp
- ✧ Remove and install procedure of Burner system
- ✧ Cleaning procedure of Burner system
- ✧ Solving procedure of siphon and drain system error
- ✧ Maintenance of Burner sensor

3. About PICARRO

On 21st Oct., the problems were the following:

1. An improper position of PICARRO
2. High background H₂O concentration
3. An improper peak shape and an unstable concentration of injected samples
4. Low Laser Frequency Current

3-1. An improper position of PICARRO

PICARRO is recommended to install in place that is not warm because data analysis is often affected by heat. Just before, PICARRO has been set near AAS in same room (Fig.3). Then, we moved PICARRO to a new room locating in opposite side (Fig.4). After moving, we started the normal setup procedure. But, error message informing a wrong valve connection occurred. So, we reconnected all cables and tubes. Then we found we needed

to connect vacuum valve more tightly. After all, error message never appeared and we concluded that the installation mission was a success.

3-2. High background H₂O concentration

According to previous results, PICARRO condition had been good until last July and some problem had occurred from last September 2014. High background H₂O concentration is one of these problems disturbing analysis data. In September, baseline concentration was over 17,000 ppm while basic background was 200-300 ppm. We checked the condition of drynrite that supplies dry air as carrier gas and it was almost finished. After changing with a new one, background values were decreasing to less than 300 ppm. But we found new two problems; one was improper peak shape and another was unstable peak height on injection samples.

3-3. An improper peak shape and an unstable concentration of injected samples

These problems were sometimes caused from the syringe clogged with or damaged by particles. We changed to new one and achieved good peak shape and stable value of injected water concentration. However, the concentration was very high (> 50,000 ppm). In manual, PICARRO can calculate isotope data if water concentration keep from 6,500 ppm to 25,000 ppm. Also recommended volume of sample injection is described to 1.8 ul. Then, we tried to adjust sample injection volume in Auto Sampler System with changing parameter. This sampler is expected to use 5 ul syringe, however, Nkolbisson has only 10 ul syringe. We set new injection volume (0.9 ul) considered to the difference between two syringes. At last, the water concentration could keep about 23,000 ppm. Moreover, I heard that calculation period was also improper from last September. Normally, calculation system is applied at later part of peak, however, calculation period shifted to the former part. This problem may be related to high water concentration. We also confirmed that calculation system was applied at former part with almost or over 25,000 ppm concentration. The calculation period shift to proper position at 23,000 ppm after adjusting the volume.

3-4. Low Laser Frequency Current

After solving the above problems, we analyzed some standards and unknown samples several time for a week. We did not see any problem in results and we concluded that PICARRO condition was recovered. That is why, I did not do setting procedure of Laser Frequency Current. But, if we had any problem soon, it may be needed to adjust it.

4. Some comments

Firstly, I recommend strongly to have stocks of analytical goods, chemical reagents and high-pressure gases. We ordered N₂O gas at the start of this working terms, however, gas has not arrived until I returned to Japan. This indicates that we may need to wait to get ordered goods for more than one month. I also suggest that the Laboratory should have enough stocks of some precious and fundamental goods for analysis so that it can continue experiment against the late delivery of ordered equipment.

Secondly, we need to make checklists of goods and chemical reagents. This may help you to grasp the remains more easily. During my stay, some lists of analytical goods, such as KimWipe and syringe, were already prepared (Fig.5). This list informs amounts of stock and checking dates.

Also I am eager for IRGM to purchase standard solution of trace elements for AAS analysis. Our AAS system is very powerful equipment for cation analysis. During my stay, I asked for Tokai University students to confirm the possibility of trace elements analysis by making calibration curve and their reply was satisfactroy with our purpose. At the present, we conclude that we can analyze Cd and Zn and maybe Hg and Pb. We need to search the possibilities of other elements. Considering the present AAS condition, we can also analyze other elements. If you need some results, I will try to show analysis data as long as possible.

Finally, I appreciate all of Cameroonian colleagues for their kind cooperation. If any problem occurred and requests, please let me know.

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Fig.1 Present gas position



Fig.2 Xe-lamp cooling system



Fig.3 Previous PICARRO location



Fig.4 Present PICARRO location

Fiche de stock

Produit : Seringue souel		Description : seringue souel cône luer-lock		
Référence :				
Date	Entrée	Sortie	Stock	Remarque
07/11/14	10	—	10	

Fiche de stock

Fig.5 List of laboratory goods

Appendix Data

About AAS, we tried to make calibration curve of Al, Ca, Si, Mg and Mn. We will show results and each calibration curve.

Element: Al with C₂H₂-N₂O

Concentration (ppm)	Absorbance	Standard division
0	-0.00038	0.00016
0.5	0.00348	0.00029
1.0	0.00763	0.00026
3.0	0.02505	0.00059
5.0	0.04339	0.00024

Element: Ca with C₂H₂-N₂O

Concentration (ppm)	Absorbance	Standard division
0	0.00057	0.00075
0.2	0.01131	0.00040
0.4	0.02876	0.00084
0.6	0.03507	0.00080
1.0	0.06411	0.00160

Element: Si with C₂H₂-N₂O

Concentration (ppm)	Absorbance	Standard division
0	0.00029	0.00004
0.4	0.00145	0.00016
0.8	0.00270	0.00004
1.2	0.00363	0.00010
2.0	0.00607	0.00017

Element: Mg with C₂H₂-Air

Concentration (ppm)	Absorbance	Standard division
0	-0.00039	0.00007
0.05	0.03420	0.00059
0.10	0.06812	0.00041
0.15	0.10374	0.00250
0.20	0.12851	0.00190

Element: Mn with C₂H₂-Air

Concentration (ppm)	Absorbance	Standard division
0	-0.00001	0.00003
0.2	0.01901	0.00034
0.4	0.03984	0.00018
0.6	0.06021	0.00064
1.0	0.09815	0.00153





