Assessment on the operation of analyzers (NC-Analyzer, Isotope ratio mass spectrometer and Inductively coupled plasma atomic emission spectroscopy) in Faculty of Agriculture, Yamagata University

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Research Materials

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Summary

There are several points that have to be taken into consideration concerning the operation of analyzers in universities. These issues are: 1. decision on the ordered schedule of use of analyzers, 2. analytical cost and its collection methods, 3. troubles that arise during the operation of the analyzers, 4. quick restoration of analyzers, 5. self-improvement of operator and other users. Once the precision operation of the analyzer is introduced, the cost for its maintenance has to be established. The operator has to build a system for their smooth management. The efficient management of analyzers in universities has to include negative factors: lower running budgets from universities and higher running costs of analyzers. Despite the problems caused by running costs, the successful and continued operation of the analyzers relies heavily on the acquired skills of the operator and his/her constant up-to-date training.

Key words：operation, analyzers, analytical cost, trouble cases

Ⅰ. INTRODUCTION

The cost of maintenance of analyzers has been mounting every year. On the other hand, the university budget to maintain analyzers hardly increases. Therefore, it is difficult to secure the budget that can be used for repairing unexpected failures. Therefore, it is necessary to consider how the performance of analyzers can be maintained, while the operation expenses are kept low.

This paper shows the present operation of the multi-purpose analyzers used in the Faculty of Agriculture of Yamagata University, including how to use them, setting up the analytical costs and their trouble cases, as well as a discussion of future.

Ⅱ. OUTLINE OF THE ANALYZERS

This paper briefly outlines three types of analyzers installed at the 4th floor of the first Building of the Faculty: NC-Analyzer (SUMIGRAPH NC-220F: Sumika Chemical Analysis Service, Ltd), Isotope Ratio Mass Spectrometer (Flash2000&ConFlo Ⅳ&DELTA V plus: Thermo Fisher Scientific) and Inductively Coupled Plasma Atomic Emission Spectroscopy (iCAP 6500 Duo: Thermo Fisher Scientific).

1. NC-Analyzer (abbreviation: NC-A)

NC-A can concurrently measure the total amount of nitrogen and carbon (Fig.1). The analytical materials include foods, feedstuffs, fats, oils, soil, and plants.

An alumina container on which a powdered sample (weight: 100-800mg) is placed is inserted into a reaction tube at 870°C to decompose the sample to nitrogen and carbon dioxide through an oxidation-reduction process with oxygen being circulated. Then the nitrogen and oxygen are measured with a TCD detector.

Measurement time is about 10 minutes per sample. This method is also suited for simplified measurement of ash content. Since this analysis is done under dry conditions, it does not produce any liquid waste at all.

2. Isotope Ratio Mass Spectrometer (abbreviation: IR-MS)

IR-MS can concurrently measure stable isotopic ratio of nitrogen and carbon (Fig.2). Analytical materials are almost similar to those of NC-A; IR-MS is particularly used for tracer analysis that measures heavy-nitrogen-added samples.

A powdered sample (weight: 0.1-10mg) is enclosed in a tin capsule, which is then introduced into an element analyzer. At this time, the combustion temperature reaches 1800°C with the help of oxidation catalyst, oxidizing organic materials in an instant. Then oxygen is removed with copper for reduction, and the quantity of remaining nitrogen and carbon dioxide is determined with a mass spectroscope.

Measurement time is about 7 minutes per sample for nitrogen only and about 10 minutes for concurrent measurement of nitrogen and carbon.

Since this device is of continuous flow measurement (measurement accuracy: $\delta^{13}C_{CO2} \leqq 0.15‰$, $\delta^{15}N_{N2} \leqq 0.15‰$), its measurement accuracy is lower than that of dual inlet measurement (measurement accuracy: $\delta^{13}C_{CO2} \leqq 0.02‰$, $\delta^{15}N_{N2} \leqq 0.03‰$)(Xiao-Sui *et al.*, 2013).

3. Inductively Coupled Plasma Atomic Emission Spectroscopy (abbreviation: ICP-AES)

ICP-AES can determine the quality and quantity of multiple elements concurrently (Fig.3). It is suited for quantitative analysis of elements in river water and drained water; it is mainly used here for measurements of liquid extracted from plants and soil. Since this method is set up for measurement of solutions, solid samples such as metals and resins have to be dissolved in acid to prepare their water solution, that is, time is required for the pre-treatment of samples.

A sample solution (volume: 10-20ml) is introduced into a high-temperature plasma at nearly 10,000°C and the quantitative analysis is performed using the wavelength and luminescence intensity of the atomic emission spectrum obtained by excitation.

The measurement time is about 1 minute per sample. During the measurement high-purity nitrogen gas continues to be fed to the spectroscope and high-purity argon gas to the plasma. Since the gas consumption in this process is large, this method is characterized by frequent exchange of gas cylinders.

Fig.1 NC-Analyzer

Fig.2 Isotope Ratio Mass Spectrometer

Fig.3 Inductively Coupled Plasma Atomic Emission Spectroscopy

Ⅲ. OPERATION OF ANALYZERS

1. Procedure for use of analyzers

The four procedures for the use of analyzers are described as shown below:

- Consultation about analyzer schedule
- Notification of analyzer use date
- Guidance on how to use analyzers
- Evaluation of analysis results

The operator must contact users closely. Depending on convenience of the operator or users' reservation date and usage date may change. The operator obtains information from a user on what is analyzed, the number of samples, the budget, and so on. Basically, the date of use is to be allocated to users coming to the operator for consultation in order. However, when the analytical material is a high-concentration sample, the order of analysis may be changed to prevent the memory effect (Sato and Suzuki, 2010) on the next measurement sample. For example, when a sample with heavy nitrogen isotope (^{15}N) is measured with IR-MS, the sample is always left for last.

2. Charge of analytical cost and its collection

To maintain analyzers properly, it is necessary to construct a system to calculate the analytical cost per sample and surely collect it from the user. In the university, users are undergraduate and graduate students, and the expense is to be charged to their research supervisor (hereinafter referred to as "the payer").

The payer has not only budget for education allocated by the university, but also other funding sources such as scientific research grants and scholarship grants (so-called "external fund"). When analytical expenses cannot be paid only from budget A, it is to be paid from budget B. Therefore, it is necessary to construct a method for dealing with such diversified payment ways. The following 4 methods are used now:

2.1. Transfer of budget

This method simply transfers all the charge from the budget item of the payer to the budget item of the operational cost of analyzers (Fig.4). This method can secure some amount of money and allocate it for particularly expensive repair of an analyzer. This is the most desirable method to be selected.

2.2. Offset by purchasing consumables

This method is to purchase consumables related to analyzers and offsetting the purchased amount from the charge for analysis (Fig.5). This method is for the payer with limited budget to pay collectively. If there are consumables needed immediately, they will be purchased, otherwise, consumables that are to be replaced in the

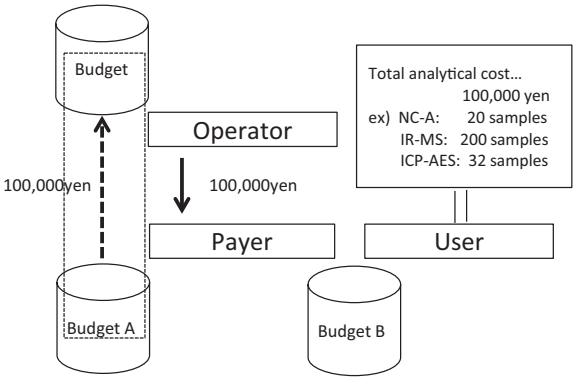


Fig.4 Payment Method of Analytical Cost (2.1).

Dashed arrow: flow of payment to operator, Solid arrow: flow of billing to the payer, Budget: operational cost of analysis, Budget A and B: payer's budget.

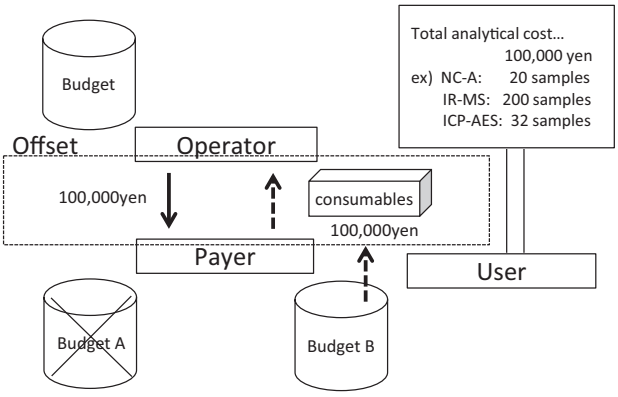


Fig.5 Payment Method of Analytical Cost (2.2).

Dashed arrow: flow of payment to operator, Solid arrow: flow of billing to the payer, Budget: operational cost of analysis, Budget A and B: payer's budget.

future will be purchased. Therefore, the operator needs to surely select consumables.

2.3. Combination of (2.1) and (2.2)

This method is to combine the two methods above mentioned (Fig.6).

2.4. Carryover for the next period (from April 1 to March 31)

A method of carrying over the charge for the next period as it is when it cannot be paid within the charge period (Fig.7). The frequent increase in the carried over charge causes troubles with the operation of analyzers. Therefore, even when analytical cost is low, the operator of analyzers should try to collect it by method (2.1).

3. Calculation of analysis unit cost

Analysis unit cost per sample is calculated by dividing the sum of money of a certain consumable by the number of samples that can be analyzed practically. For example, each analysis unit cost for three analyzers is set as shown in Table 1-3.

Examples of analysis unit cost are: for NC-A, 600 Yen per sample (Tokyo university of agriculture and

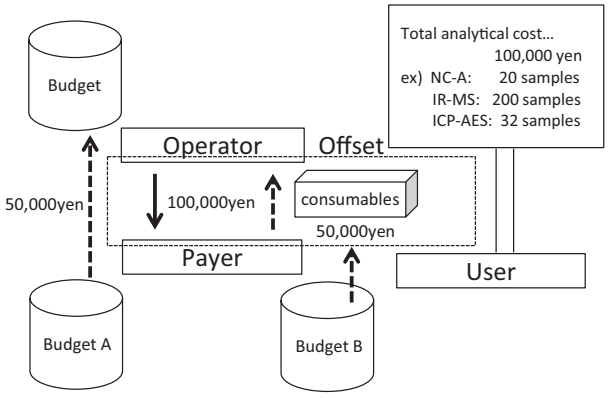


Fig.6 Payment Method of Analytical Cost (2.3).

Dashed arrow: flow of payment to operator, Solid arrow: flow of billing to the payer, Budget: operational cost of analysis, Budget A and B: payer's budget.

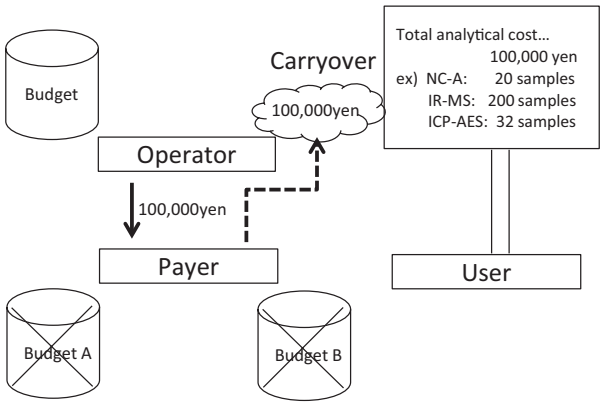


Fig.7 Payment Method of Analytical Cost (2.4).

Dashed arrow: flow of payment to operator, Solid arrow: flow of billing to the payer, Budget: operational cost of analysis, Budget A and B: payer's budget.

Table.1: Calculation Method of Analysis Unit Cost (FY2014, NC-A)

Consumables	Cost (yen)	Life per consumable (cycles)	Cost per sample (yen)
Helium gas	57,000	600	95
Oxygen gas	40,000	500	80
Gas chromatography-column	200,000	20,000	10
Reactor	350,000	15,000	23
Quartz tube (Reactor side)	200,000	4,000	50
Pump for oxygen circulation	55,000	20,000	3
Filter tube	20,000	5,000	4
Boat holder	32,000	3,000	11
Alumina container	7,000	1,500	5
Metal parts	10,000	5,000	2
Water/Carbon monoxide trap tube	10,000	20,000	1
Silicon sealing materials	4,000	5,000	1
Copper wires	7,000	600	12
Quartz wool : SiO_2	4,000	3,000	1
Anhydrus	10,000	10,000	1
O-rings	6,000	3,000	2
Total			300

technology, 2015); for nitrogen isotopic ratio measurement with IR-MS, about 10 pounds or more per sample (Robinson, 2001); and for ICP-AES, 1,600 Yen/30 minutes(Yamagata research institute of technology, 2015).

Analysis unit cost changes depending on various conditions. Particularly, the price revision of consumables and unexpected troubles with analyzers may incur in additional expenses. Therefore, it is difficult to set lower unit costs (Table.4).

In general, life of consumables is not described in detail by the manufacturers of analyzers. It is known that the life of the product may change by inappropriate of

Table.2: Calculation Method of Analysis Unit Cost (FY2014, IR-MS)

Consumables	Cost (yen)	Life per consumable (cycles)	Cost per sample (yen)
Helium gas	57,000	600	95
Oxygen gas	40,000	5,000	8
Nitrogen gas	5,000	5,000	1
Carbon dioxides gas	100,000	10,000	10
Filament	51,000	2,000	26
Gas chromatography-column	150,000	10,000	15
Oil for pumps	10,000	2,000	5
Cool fan	10,000	5,000	2
Quartz tube (Reactor side)	10,000	250	40
Quartz tube (Reduction side)	10,000	500	20
Water/Carbon monoxide trap tube	65,000	10,000	7
Chromium oxide : Cr_2O_3	11,000	250	44
Sivered cobaltous-cobaltic oxide : Co_3O_4/Ag	21,000	500	42
Copper wires	14,000	500	28
Quartz wool : SiO_2	4,000	500	8
Anhydrus magnesium perchlorate : $Mg(ClO_4)_2$	16,000	5,000	3
O-rings	4,000	2,000	2
Others			75
Total			430

Table.3: Calculation Method of Analysis Unit Cost (FY2014, ICP-AES)

Consumables	Cost (yen)	Life per consumable (cycles)	Cost per sample (yen)
Argon gas	25,000	300	83
Nitrogen gas	5,000	500	10
Spray chamber	65,000	5,000	13
Nebuliser	75,000	5,000	15
Adaptor duo	35,000	5,000	7
Centre tube	36,000	5,000	7
Holder centre tube	45,000	5,000	9
Torch-Duo	52,000	3,000	17
Clamp	7,000	5,000	1
O-rings	13,000	2,000	7
Pump Tubing (aqueous sampling)	2,000	500	4
Pump Tubing (aqueous drain)	2,000	500	4
Sample introduction kit	55,000	3,000	18
Water filter	30,000	5,000	6
Ceramic parts	110,000	5,000	22
Others			26
Total			250

Table.4: Trend of Analysis Unit Cost

(Unit : yen)

Item	FY2011	FY2012	FY2013	FY2014
NC-A	233	233	250	300
IR-MS	342	342	378	430
ICP-AES	200	200	220	250

maintenance and wrong operation by users.

Usually, restoration of an analyzer is discussed after some troubles have actually occurred with the analyzer. As a result, if the charge exceeds the budget, the user's research is delayed. It should surely be determined; therefore, which consumables are stored.

That is, if only specific consumables are stored, the budget to purchase a consumable needed suddenly cannot be secured. Considerable attention in particular should be paid to method (2.2). In reality, this method is the most selected.

4. State of usage of analyzers

Monthly schedule of each analyzer, the number of users and the number of samples, is shown in Fig.8-13. The data collection period is from April 1 to March 31 next year in accordance with the budget fiscal year.

In the period from October to February next year, during which there are many requests to use the analyzers, it is not allowed that the same user is allocated the use of the analyzer in succession. This rule is set so that as many users as possible have equivalent time to use them.

5. Maintenance of analyzers

To reduce the frequency of problems with analyzers, daily maintenance is essential. Users are required to write: the material to be analyzed, use time of the analyzer, remaining amount of gas, presence or absence of any abnormality etc. in the record notebook. The operator has to check the condition of the analyzers every day. The continuation of these procedures enables the smooth operation of the analyzers.

If the analyzers are continuously in operation, it becomes difficult to reserve maintenance time. Depending on the time required to replace parts, the standby time of the analyzer is necessary; for example, the analyzer must be turned off, and the temperature of the reactor must be lowered. Accurate and quick maintenance enables users to use the analyzer without waiting time. Maintenance should be scheduled, focusing on the spare time between analyses operations.

Figure 14 shows trouble cases of each analyzer. Since all of the three analyzers use high pressure gas, a common problem observed is the wrong handling of a regulator. Particularly, a stop valve is closed too tight, often causing the next user not to be able to turn the valve open.

The troubles are often attributed to wrong operation of analyzers, unsatisfactory sample preparation process, or deterioration of parts. If a user operates various places to try to restore the analyzer trouble by her/himself, the situation often becomes worse.

Even when a skilled user uses an analyzer, a problem like this occurs suddenly. With some exceptions, therefore, analysis days are allocated only to the operator's working days.

Since an analyzer contains many parts, of which deterioration cannot be determined, the conditions of all parts cannot be understood exactly. When a problem seems to be attributed to such a part, we have to ask on-site repair. Although the repair incurs in more expenses, it is a good chance to acquire important information.

Therefore, we keep try to obtain as much information about the analyzer as possible from a technician who deals with the problem.

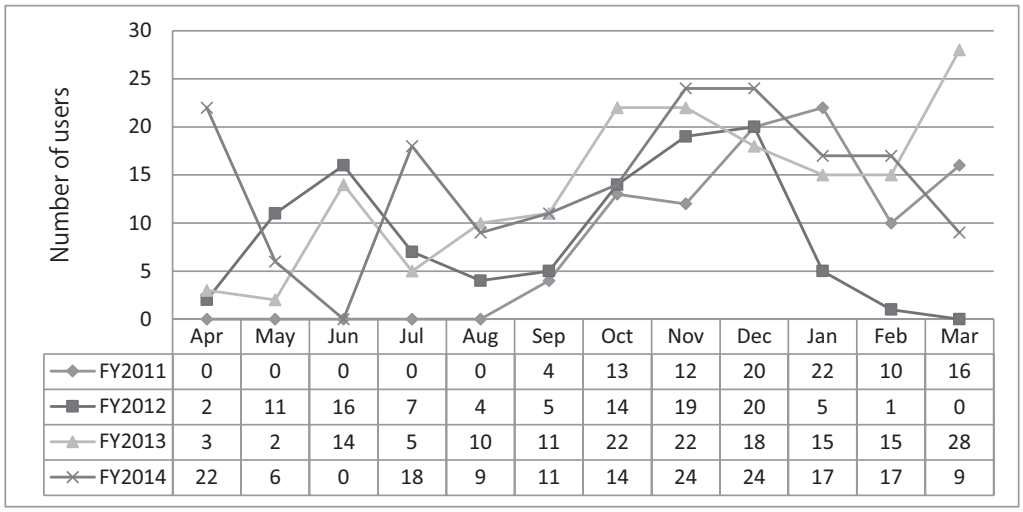
Ⅳ. ISSUES IN THE FUTURE

As described in the section of cost calculation for analysis, a user who uses an analyzer for the first time must be burdened with high analysis unit cost. To make cost burden fair so as not to be dependent on usage periods, the unit charge of analysis can vary depending on the payers. In that case, the operator has to spend more time for data collection, with the workload becoming larger.

Operator's work-load may be reduced by increasing the number of operators. But that requires an organizational reform and therefore not practical.

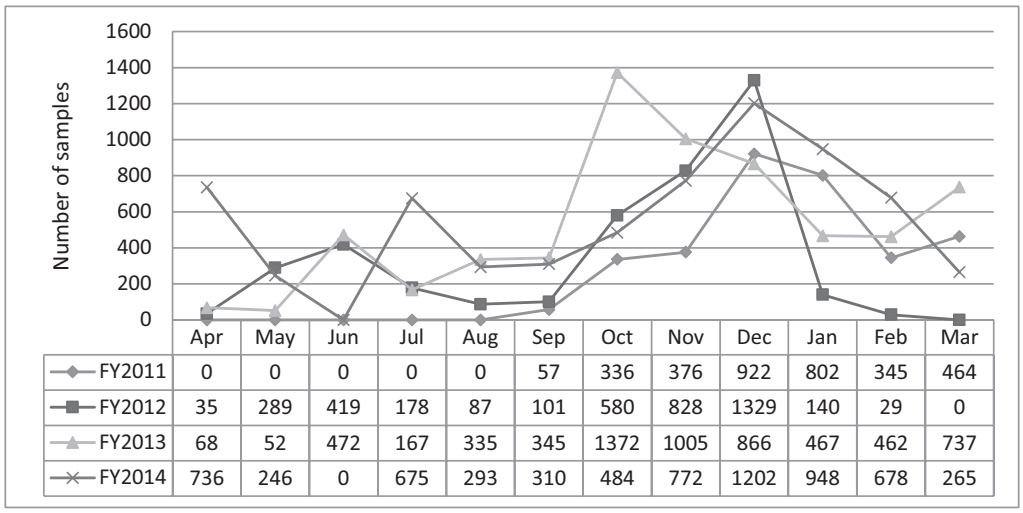
It is necessary to examine the present situation of operating analyzers.

To do so, it is essential for operators to collect a wide range of information about analyzers. Certainly, the operator must determine replacement timing of



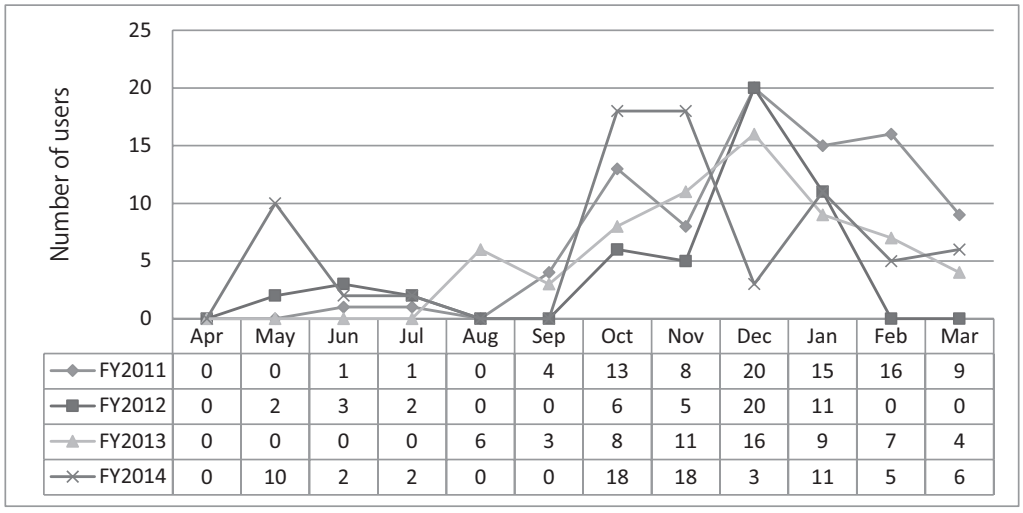
	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar
FY2011	0	0	0	0	0	4	13	12	20	22	10	16
FY2012	2	11	16	7	4	5	14	19	20	5	1	0
FY2013	3	2	14	5	10	11	22	22	18	15	15	28
FY2014	22	6	0	18	9	11	14	24	24	17	17	9

Fig.8 Monthly Usage of Analyzers (NC-A, Number of users)



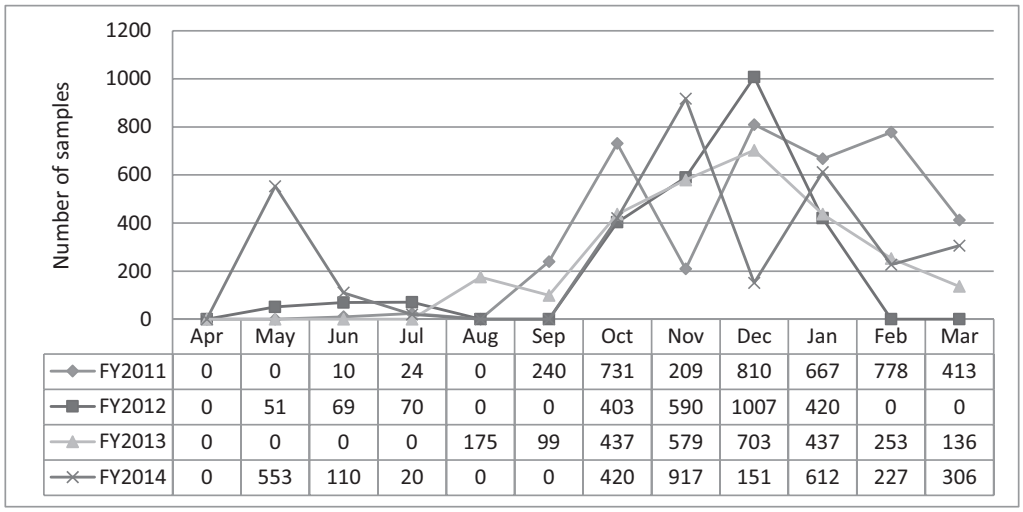
	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar
FY2011	0	0	0	0	0	57	336	376	922	802	345	464
FY2012	35	289	419	178	87	101	580	828	1329	140	29	0
FY2013	68	52	472	167	335	345	1372	1005	866	467	462	737
FY2014	736	246	0	675	293	310	484	772	1202	948	678	265

Fig.9 Monthly Usage of Analyzers (NC-A, Number of samples)



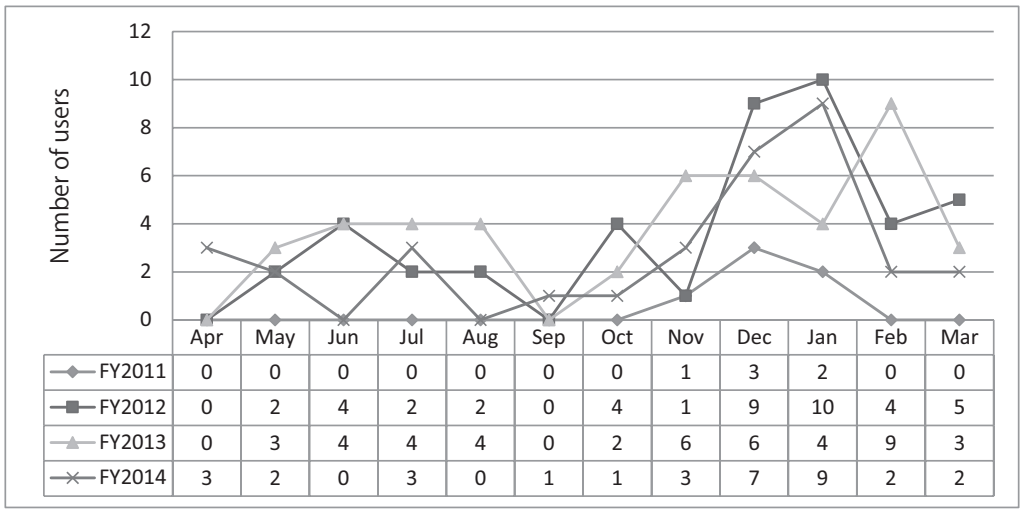
	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar
FY2011	0	0	1	1	0	4	13	8	20	15	16	9
FY2012	0	2	3	2	0	0	6	5	20	11	0	0
FY2013	0	0	0	0	6	3	8	11	16	9	7	4
FY2014	0	10	2	2	0	0	18	18	3	11	5	6

Fig.10 Monthly Usage of Analyzers (IR-MS, Number of users)



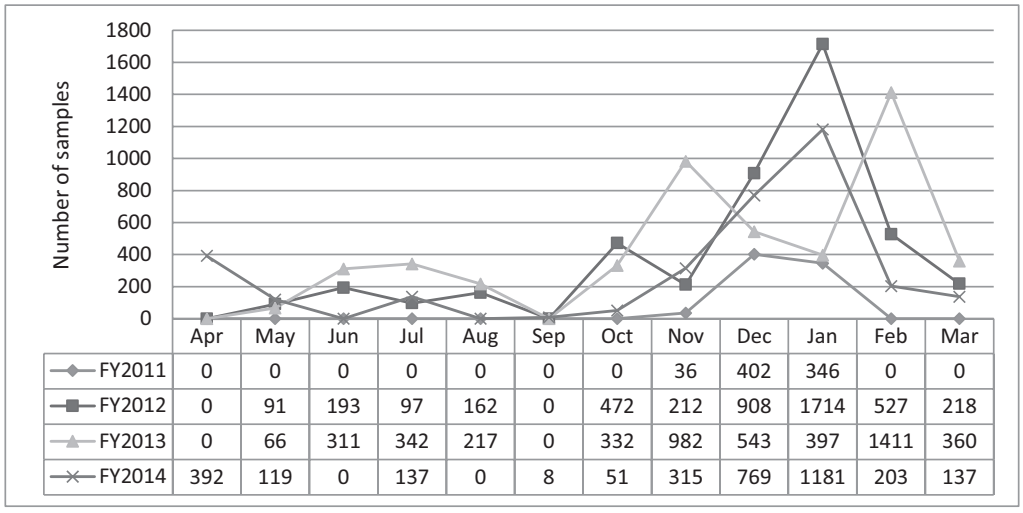
	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar
FY2011	0	0	10	24	0	240	731	209	810	667	778	413
FY2012	0	51	69	70	0	0	403	590	1007	420	0	0
FY2013	0	0	0	0	175	99	437	579	703	437	253	136
FY2014	0	553	110	20	0	0	420	917	151	612	227	306

Fig.11 Monthly Usage of Analyzers (IR-MS, Number of samples)



	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar
FY2011	0	0	0	0	0	0	0	1	3	2	0	0
FY2012	0	2	4	2	2	0	4	1	9	10	4	5
FY2013	0	3	4	4	4	0	2	6	6	4	9	3
FY2014	3	2	0	3	0	1	1	3	7	9	2	2

Fig.12 Monthly Usage of Analyzers (ICP-AES, Number of users)



	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar
FY2011	0	0	0	0	0	0	0	36	402	346	0	0
FY2012	0	91	193	97	162	0	472	212	908	1714	527	218
FY2013	0	66	311	342	217	0	332	982	543	397	1411	360
FY2014	392	119	0	137	0	8	51	315	769	1181	203	137

Fig.13 Monthly Usage of Analyzers (ICP-AES, Number of samples)

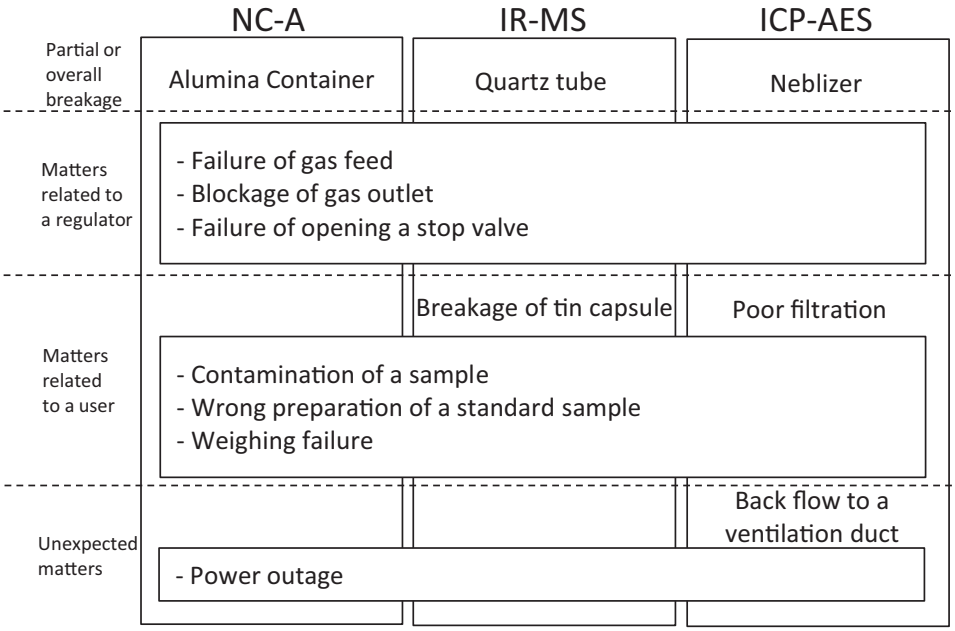


Fig.14 Trouble cases

consumables based on the experienced problem cases of analyzers and continue updating the technological knowledge concerning their operation. To extend the life of analyzers at a low cost and continue operation, the accumulation of such daily work is the most important task.

Ⅴ. ACKNOWLEDGMENT

I thank Dr. H.Fujii, Faculty of Agriculture, Yamagata University, for advice about operation of analyzers.

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山形大学農学部における分析機器（全窒素・全炭素分析装置、安定同位体比質量分析装置、ICP 発光分光分析装置）の保守管理に関する資料

高　橋　拓　也
山形大学農学部技術室
（平成 27 年 9 月 11 日受付・平成 27 年 11 月 10 日受理）

摘　　要

大学における分析機器の保守管理には配慮すべき項目がいくつかある。それは，1. 機器利用日時の決定，2. 分析費用とその徴収方法の設定，3. 機器の不具合の事例分析，4. 機器の復旧に向けた速やかな対応，5. 機器管理者および使用者の自己研鑽，などである。分析機器の正確な保守管理を目指すには，管理にかかるコストを計算しておく必要があり，保守管理の担当者にはこれらの運用を円滑にするためのシステムづくりが求められている。しかし，分析機器の十分な保守管理には負の要因がある。それが，大学から配分される予算の低減や分析機器のランニングコストの増加によることである。コストだけの問題ではなく，分析機器を円滑に管理し続けるには，保守管理者自身のスキルを向上させることはもちろん，継続的に最新の訓練を受けることが必要である。

キーワード：保守管理，分析機器，分析費用，不具合事例