Guest Forum

Lecture by a Jury Member for the 2006 Masao Horiba Awards

Research as Seen from My Experience in a Company, a University and a Public Research Institute

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Some aspects of research in a company, a university and a public research institute vary widely from each other, while there are other aspects that are mostly common. In this lecture, the author presents his experience as a researcher in a company, a university and a public research institute and reflects on problems encountered in each of these settings. In his reflection, basic viewpoints in common with these different settings are presented as three elements of invention or discovery. By taking the invention of ICP as an example, 10 stages involved in the creation of a new analysis method are also discussed.

Introduction

After my tenure that ended in March 2005 in the National Institute for Environmental Studies, I am currently working in University of Tsukuba. In this lecture, I will present my long experience as a researcher in a company, a university and a public research institute, and discuss stages for the creation of a new analysis method. I hope this discussion will be useful for future research activities.

My Study for a Graduation Thesis on the Method of Analyzing the State of Nonmetallic Inclusions in Steel

In a university, I wrote a graduation thesis on the development of a method of analyzing the state of carbon contained in steel. The hardness of steel changes–it is "hardened" when it is quenched from a high temperature, and it is "annealed" when it is cooled down slowly. However, the composition is same in these states. According to the general explanation, this is because carbon state is different. .

When calcium carbide (CaC_2) is dissolved with an acid, acetylene (C_2H_2) is created as a result of the bonding of hydrogen (H) with CC in the crystal. Similarly, hydrocarbon is created when iron is dissolved. Professor Hirano, who was my teacher in university, thought that this reaction might serve for state analysis by exposing the connection of CC in iron, and pursued a study on this theme for several years. I was involved in this study in its last year. Though the study was eventually proved unsuccessful, it gave me an idea of the importance and the difficulty of state analysis.

In selecting a research theme, it is important whether you choose a theme that is easy to get results, or you choose one that is difficult but promises great results if it can be solved.

I tackled a difficult one, thinking that I could manage easy one anytime. In the course of the actual study, however, I found myself in an unhappy situation of having to hang on to an unfruitful theme; in this situation, studies that were producing steady results seemed more appealing. It is important to solve problems, whether they are easy or difficult, but you have to take much thought to determine whether you pick an easy one or difficult one.

Such experience in my fourth-year taught me that research was not so easy.

Mazda Research Laboratory of Toshiba - Analysis of Impurities in Atomic Fuel

I began my career as a researcher in Mazda Research Laboratory of Toshiba. The laboratory was one of the best research institutes among private corporations, with its long history and many illustrious researchers. Toshiba was engaged in a wide range of business from midget lamps to nuclear power. It was also involved in a part of the process to produce UO_2, an atomic fuel. In this business, we needed to analyze impurities in the fuel and I engaged in the establishment of the official analysis method.

The task was developing the analysis methods for all impurities in UO_2; that meant we were supposed to carrying out any kind of elemental measuring methods because UO_2 is inorganic. While I was tackling the task, I acquired a certain level of knowledge on the characteristics and behavior of most elements and related experiences. This would be proved very useful in my career.

Even now, I sometimes remind of the experience that, while doing this research, I found that measurement results differed according to the methods of dissolving UO_2, or uranium dioxide. I obtained four sets of data; two were obtained by the method of using nitric acid, and the other two were obtained by the method of using hydrochloric acid. I found that one set of measurements results were clearly higher than the other set of measurements results. The one set (pair) was higher and the other (pair) is lower. I thought that this difference must have resulted from the difference in dissolution methods, and included this observation in my research report.

However, a later statistical test showed that, assuming a significance level of 1 to 5 %, the difference I had observed was far from statistically significant; my theory could have been justified assuming a significance level of 20 to 30%. In common sense, we tend to assume that there should be a clear difference if we have higher results and lower ones, for two trials each, however, this experience brought home to me that the actual was not always so simple as in common sense.

Oxygen Analysis Using High-Speed Neutrons

Later, a researcher engaged in the study of accelerators began to seek new ways to use an accelerator. Eventually, we decided to analyze oxygen using high-speed neutrons created by the collision of deuterium atoms, which were accelerated to approximately 200 keV, with tritium atoms. In those days, no method had been developed that could analyze oxygen non-destructively and directly. Therefore, our method was unique because it involved having high-speed neutrons collide directly with oxygen atoms and measuring oxygen by measuring radiation resulting from the decay of produced nitrogen. The method enabled us to analyze directly to very low level to an order of 0.001%. As every experimental result of ours was the first, our mood was in high spirits until an analysis yielded a minus value. People around us were so shocked that some advised us never to disclose this finding, on the ground that disclosure would destroy our credibility forever.

We reviewed this finding frantically and checked every problematic point and every possibility; e.g. the concentration of impurities in the inert gas used or the trace oxygen on the surface, etc.. However, all our efforts were fruitless and we had no idea on the reason why the analysis yielded a minus value. At long last, we managed to identify the reason. As we were measuring the trace oxygen in organic substances in a liquid, we, of course, measured the blank value of the vessel and subtracted this value from a measurement. After high-speed neutrons had entered the vessel during the measurement of a sample, the back of the vessel wall was radioactivated by neutron and radiation whose intensities had decreased due to scattering, etc. in the sample. This made the measured trace oxygen concentration lower than the value determined at a blank measurement and resulted in a minus value.

This research, which finally resulted in the discovery of a correction method, was really strenuous. The most important lesson I learned in the research was that trace analysis was not an ordinary challenge, but it was required the greatest care.

State Analysis of Fe Using the Mössbauer Effect

One day, I noticed a newspaper article saying that a scientist named Mössbauer had been chosen as that year's Nobel laureate for physics. According to the article, he had found a method of measuring the charge distribution around a nucleus with a very high accuracy, thereby determining the size and change of the nucleus. This article led me to think that I might be able to apply this finding to state analysis and sure enough, the analysis using this approach turned out to be a success.

This experience reminds me that, when searching for new tools or ideas for study, a researcher may get behind if he or she goes to only an academic society's meeting and listens intently to what people are saying. Instead, reading the newspaper can be the quickest way for that purpose. As the source of information, newspaper articles are considered to be inexact, useless or even totally wrong, however, some articles can be useful. The way of reading articles is important on reading about a discovery and you must think of its possible applications, even if there is no direct mention of such applications. You are not likely to learn about useful matters, which has not been considered noteworthy yet, only from some of many academic society's meetings. Therefore, you must wait for 1 to 3 years to get access to important information. By contrast, the newspaper gives you information within a week. This is the reason why you should not ignore the newspaper.

X-Ray Fluorescence Analysis - the Standpoints of People Who Analyze and Those Who Have Something to be Analyzed

After that, I continued conducting X-ray fluorescence analysis as a daily routine. At Toshiba's laboratory, various materials were being developed and a variety of tasks were brought to me. My basic duty was to analyze samples brought in by others without any argument. One day, I had an interesting experience as below.

On that day, I analyzed dozens of samples brought in by one person and found that measurements for some samples were exactly the same. I was sure of the carelessness of the person who requested the analysis and I angrily accused him of having sent the same samples. The person seemed very confused and replied to me that it was a common to mix the same samples occasionally to check the accuracy of analysis. From this, I learned, as a direct experience, that people who analyzed and those who had something to be analyzed had different sensibilities and standpoints, and that these differences made them look at the same task differently.

High-Resolution Double Crystal Spectrometer

While zircaloy (a zirconium alloy) is frequently used as a material in nuclear reactors, the amount of hafnium in zircaloy must be controlled to stay below a certain level because hafnium absorbs a relatively large amount of neutrons. Therefore, the analysis of hafnium in zirconium was an important theme. However, as hafnium is an element that cannot be separated easily, it was difficult to analyze it spectrally; this kind of analysis failed due to the overlapping of the secondary line of zirconium with the analysis line of hafnium. Though a method was proposed that used a diamond-structured crystal to prevent secondary reflection, this method was also failed when applied practically.

Then, I came up with the idea that, if all methods based on a single reflection had failed, the method based on double reflections might be proved successful. When I consulted an expert in X-ray spectroscopy about this idea, he tried to dissuade me from putting it into practice. He said that, since the amount of X-rays was decreased to about 1/1000 after they had passed through a spectrometer, even once, there were no chances of obtaining any signals after the second reflection. I was thus convinced of the hopelessness of my idea but I still had an itch for seeing the actual situation that was after the second reflection in which the interference from zirconium had been eliminated. I, therefore, tried my idea using a totally handmade "barrack" set consisting only of two crystals standing on two axes ; that was a manual scanning device. By using this device, I saw an extremely intense signal emitted, that sent the rate meter to a full swing. I still remember the great excitement I felt at that time. Later detailed studies showed that, once X-rays were Bragg-reflected, they became parallel beams and, in principle, be reflected 100% at an additional reflection. In principle, even if X-rays were additionally reflected several times, it will not result in a decreased intensity. In the first reflection, the intensity sometimes drops to 1/1000, 1/10000 or even less. From the second reflection, however, it does not drop.

This finding showed that the spectrometer I came up with could actually be used. Since then, it has been applied to various researches.

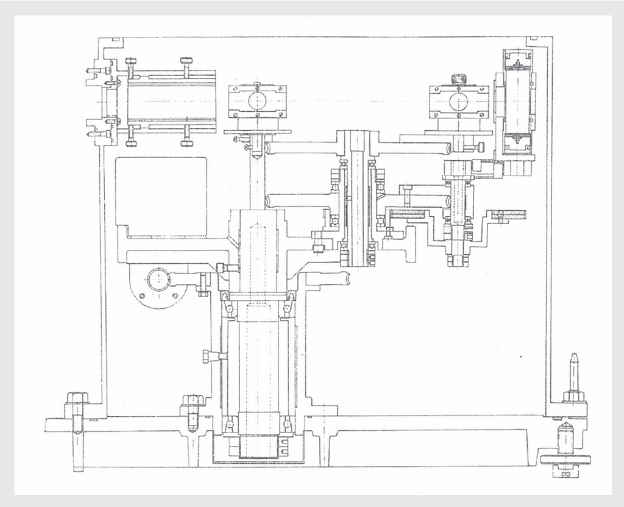

Figure 1 A section of the spectrometer (prototype) [1]

Figure 1 shows the section of the double crystal spectrometer developed at that time. In manufacturing this spectrometer, a designer in the factory for trial production said to me, "You can make modifications only in drawings and I will redraw them as many times as you want." I still reminds of his words. Thanks to the designer, I was able to make fundamental changes five or six times before arriving at the design shown in this figure. The object shown at the upper right, which looks like a square box, is a proportional counter. For the case of a certain weight applied to the position, the parts supporting it must be designed as stout ones to secure the required accuracy. This, in turn, results in that its basis must also be stout and, ultimately, overall design gets bigger. A lot of this kind of problems popped up at the design stage. Even so, it was my duty to be tenacious and ask for the required level of accuracy. For example, I had to perform an analysis to determine what kind of impact would result from a bend of 5 μm and show them whether a deflection to this extent was acceptable or not. Also, this experience turned into lessons on how to design an apparatus in consideration of its machining, assembly, adjustment and maintenance. As shown in Figure 1, the final design features the flat base as a reference surface, which enables us to work on machining, assembly, adjustment and maintenance by only measuring from the base.

After that, I engaged in development related to the reactor chemistry and the safety system of the fast breeder reactor, especially the development of oxygen and hydrogen analyzers. The setting of this job was totally different from the previous one and I felt some unease. However, the tasks were proved beneficial for me in that they taught me good and bad points of a big project.

Later, I took an offer to return to my alma mater as a researcher. In the university, I designed a new double crystal spectrometer to develop high-resolution measurement in the field of X-ray spectrum. This development was pleasant for me, as it resulted in applications to state analysis and led to the finding that the X-ray spectrum had a structure with a very wide non-separated area.

Total Reflection X-Ray Analysis Method

Total reflection X-ray analysis is a method that permits ultra-trace analysis. The way of analysis is putting a small amount of a sample on a very flat substrate and having X-rays entering at an angle of 1/100 to 3/100 degrees. At such a degree, X-rays are totally reflected without scattering and thereby it permits a highly accurate measurement. The potential of this method in trace analysis was recognized promising and it actually achieved a certain level of accuracy, but it was far from the level of ultra-trace analysis.

Under these circumstances, we proposed to use monochromatic beams. Such a step was totally unthinkable in conventional trace analysis that required intense beams, because the intensity of monochromatic beams would be down to from 1/1000 to 1/10000 that of polychromatic ones. In total reflection, however, beams are nearly parallel to each other, forming only very small angles in the order of 1/100 of a degree. Accordingly, we thought that, once beams had become parallel, their intensity would not decrease significantly after reflection, and we could justify the use of monochromatic beams. In fact, no decrease in intensity was observed when we put this idea into practice. This opened the door to ultra-trace analysis by reducing the background dramatically. At present, ISO stipulates that the total reflection X-ray fluorescence spectrometer using monochromatic beams should be used to analyze an ultrasmall volume of pollutants on a silicon wafer, because of the effectiveness of the spectrometer for this application. Though many hardships had to be overcome in developing the spectrometer, I feel pleased that it was developed in Japan and fortunately, with a Japanese manufacturer taking the lead.

National Institute for Environmental Studies

Subsequently, I moved to the National Institute for Environmental Studies, where I exclusively performed managerial tasks. Though I did not pursue research activities directly, I could think much about the direction of the study by discussing topics with many colleagues. Studies for discovering some substances or some phenomena causing the problems on the environment can be accepted more easily in society. However, studies for aiming at showing the absence of problems are also valuable in terms of protecting the environment. There are some behind-the-scenes environmental problems, that are invisible at present, but they will be visible in the future. I point out three important keys to regarding this kind of problems, that is; "Don't overlook", "Don't neglect", and "Don't lose your head." I learned these lessons from past cases of environmental destruction and pollution-related diseases [2].

Three Elements Required for New Development

So far, I have told you about events that left deep impressions on me in my life as a researcher. To rephrase what I felt in performing various tasks in a concise expression, I think that three elements are required for new development: (1) science and technology; (2) thinking ; and (3) information (Figure 2).

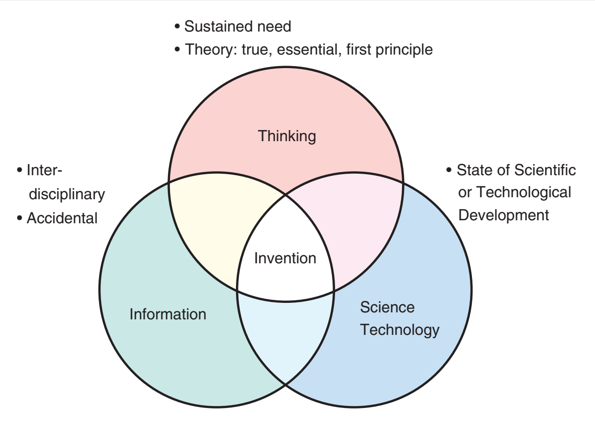

Figure 2 Three elements required for new development

(1) Science and Technology

The most important thing is that the science or the technology should have been developed and the development must be reflected in the activity.

(2) Thinking

Thinking has two aspects. One aspect is a sustained need and you must be clearly conscious of what you have to do or want to do. This is the motive force for thinking.
The other is theory. A theoretical problem needs to be clarified and clarification should be in essence. Imagine a pair of scales that are balanced. Is it correct to say that an object on the right scale has the same mass as that of another object on the left scale? Of course, you can say this is correct in the ordinal situation.

However, Shockley, the famous inventor of transistor, says that it is correct on the condition that the gravitational acceleration at the position of both scales is the same. In other words, you must be clearly conscious that the objects bringing a balance are not masses but forces.

(3) Information

To think of new things, it is highly important how much you can collect information not only from your special field, but also from other fields. You can have a good control over matters already present in your brain. The problem is how to utilize accidents. That is, from another viewpoint, how to create accidents. You can gather information from accidents by attending meetings of various academic societies and meeting and discussing with various people. I think that a freewheeling atmosphere, in which you can create many accidents, is necessary in reality to develop and improve new things.

As I mentioned above, I first worked in a company and then, moved to a university. After moving to the university, I did not feel that the conditions for my research had improved. A major reason why I felt this way was that I found surrounding people who were working only in fields similar to mine in the university. In my previous workplace, the circumstances allowed me to be in contact with people in a variety of fields on a daily basis; there were some people specializing in communication on the floor above, others specializing in electricity on the floor below, and also, specialists in machinery in another building. These circumstances were very useful in my research, however, after moving to the university, I lost the opportunity for this kind of contacts. That was why I joined about 20 academic societies as a conscious effort to make up for what I had lost.

10 Stages Leading to the Creation of a New Analysis Method

Lastly, I would like to talk about Fassell, who invented ICP-OES (IOP Optical Emission Spectroscopy) and ICP-MS.
He said that there were 10 stages that led to the creation of a new analysis method, and that this applied to most cases. From my experiences and also from one of Alan Walsh, who is the inventor of atomic absorption, our ideas roughly agree with Fassell's opinion.

(1) Focus on Goals to be Achieved and Problems to be Solved Clearly

First, it is necessary to "focus on goals to be achieved and problems to be solved clearly." It is difficult to find a new analysis method if you are just searching for some interesting ideas. In Fassell's case, he set out to devise a method for analyzing multiple elements simultaneously. He was very interested in emission and ICP-MS, but never in atomic absorption. In short, you must clearly focus on the goal to be achieved or the task to be fulfilled, and they must remain the back of your mind regardless of what you are seeing.

(2) Preceding Basic or Applied Studies on Scientific Principles

As science is a process of accumulation, progress in R&D presupposes a preceding technological basis.

(3) Flash of Inspiration

Then it comes to a "flash of inspiration." Most people regard it as the start of a process toward invention, but Fassell says that you cannot get flash of inspiration without having passed the previous stages (1) and (2) described above.

(4) Design and Manufacture of an Experimental Apparatus to Corroborate Principle

Once you have an inspiration, you move to the next step of testing. In Fassell's case, the challenge was to devise a stable ion source using a high-frequency charge. In my case, I manually adjusted the barrack set when having X-rays reflect twice. In retrospect, this means that I could achieve an accuracy of one second through manual operation. Such a feat could not have been possible without complete absorption.

(5) Design and Manufacture of an Apparatus to Confirm the Usefulness and Effect of an Invention under Ideal Conditions

After confirming that an invention is feasible in principle, you must also confirm that it works satisfactorily under ideal conditions.

(6) Studies on Whether the Method Exhibit Satisfactory Functions on Various Samples on Site or in a Laboratory

Normally, once academicians have arrived at the stage (5), they think that they have achieved great results and therefore regard it as been completed. According to Fassell, however, they still have to test various samples to confirm whether the method exhibit satisfactory functions on site or in a laboratory or not. In other words, they must check whether the method can be put to practical use.
Academicians tend to become overconfident when they do reach this stage and think, "we have developed something so good and it is unbelievable that it will not be used."

(7) Gaining Acceptance in the World of Analysts

In fact, however, all the developed methods are not accepted in the world of analysts. It still has to "impress" people in the same business and this process is actually difficult. Both Fassell and Walsh say that the process takes full 10 years in some cases. In other words, the world is reluctant to recognize what you have done. It is difficult to "impress" people.

(8) Design and Manufacture of a Prototype for Practical Use

(9) Design and Manufacture of a Model for Commercial Use

Next, you must go through the stages of design and manufacture of a prototype for practical use and that of a model for commercial use. Researchers are hardly accustomed to this stage, however, they must take perfect care when handling samples, etc..

(10) Market Exploration and Sales

The next stage is market exploration and sales. At this stage, the developed method must face keen competition from rival methods. It will develop into an established method if it survives this competition and thus avoids being screened out.

Conclusion

Research consists of various elements and has wide differences according to setting. In a university, the focus is on conducting interesting research. In a company, it is naturally on meeting a certain goal. In a public research institute, you are required to achieve a certain level of research results to show to society, whether the research is interesting or not. Each of these approaches is important.

A future task is to sufficiently discuss what kind of research is going to be important. You cannot produce something real out of nothing. If you lack adequate information, discussion and analysis, you cannot achieve any significant results in industrial, public or academic research projects, however well they are funded. Though the theme of such discussion is vague as you cannot hope for reaching any clear conclusions, I think that the science of measurement will change in such a manner that we will see a new focus on measurement which is relevant to human activities. More "research for aiming at maintaining human activities" should be conducted. I hope that active discussions will be conducted on this topic.

<An extract from a lecture given at the Lecture Meeting of Members of the Jury Committee for the Masao Horiba Awards (on September 26, 2006)>
(Publication members have responsibility for the translation)

Reference

[1] Yohichi Gohshi, Kohei Hori, Yoshio Fukao: Double Crystal X-Ray Spectrometer and X-Ray Fluorescence Analysis, *Progress of X-Ray Analysis* II (edited by the Japan Society for Analytical Chemistry and the Council for Studies on the X-Ray Industrial Analysis Method (Tokyo, Science Press, 57 [1971])

[2] Yohichi Gohshi: *How to Think of Behind-the-Scene Environmental Problems* (Report by Gakushikai, 841, 35 [2003])