Technology and Product Development of Horiba’s Laboratory Analytical Instrument for R&D and Quality Control

— Focusing on X-ray analytical instruments —

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Abstract

“Ultra Quick Supplier : UQS” is the key word for Horiba’s cooperate activities. We recognize that our most important role as a manufacturer of measuring instruments is to supply products and service quickly to customers based on a clear grasp of the market needs of 21st century which is changing drastically. This paper introduces how is developing products and technologies for analytical instruments for R&D and quality control, focusing on X-ray instruments. It also shows some examples in which alliance is effective to achieve UQS.

Preface

With the advancements being made in electronics, biology, and the energy industry, there is also a growing demand for the development of more advanced analytical technologies and equipment. In particular, high-precision, high-speed analysis is needed in fields dealing with materials, such as metals, semiconductors, and organic materials. Also, in order to instill materials themselves with various types of functions, we must go beyond attempts to detect even smaller volumes of material components -- we must also be able to propose a diverse range of analytical methods such as the microscopic analysis and depth-direction analysis of materials having a micro-structure.

Conversely, when working to develop analytical instruments that will contribute to a customer’s productivity, it is not sufficient to merely work to improve the performance of the instrument itself. As a manufacturer, we must have a true understanding of the details of a customer’s business, offer high-quality products with excellent operability, maintenance, and functions that meet the customer’s objectives, and provide a high level of service.

In order to provide high-level, diverse products that meet a customer’s needs, and to provide these products on a speedy basis, it is extremely important to establish an alliance with multiple corporations and organizations, rather than attempting to cover all bases with just one corporation. Such an alliance must be multi-faceted, taking advantage of the strengths of each of its members, with members complementing each other as they work closely together, and at times, compete against each other.

The Mainstay Technologies of Horiba and the Horiba Alliance

Horiba has developed a vast range of analytical equipment for the physics and chemistry fields, including products related to electrochemistry, such as the pH meter, gas analyzers that utilize nondispersive infrared absorption (NDIR), Fourier transformation infrared spectrophotometers (FTIR), laser-based particle analyzers, and element analyzers that utilize energy dispersive X-ray spectroscopy (EDX).

In 1997, Jobin Yvon, S.A. the world’s leading manufacturer in the field of optical technologies, joined the Horiba Group. This added inductively plasma atomic emission spectrometers (ICP-AES), glow discharge spectrometers (GDS), Raman spectrometers, etc., which utilize ultra-violet and visible spectroscopy technologies, to the Horiba product lineup, greatly expanding the Horiba Group’s range of products related to element analysis and structure analysis.
Likewise, in 2000, Horiba entered into a business partnership with the Analytical Division of Oxford Instruments, plc, a global leader in the field of X-ray analysis. In the past, Horiba and Oxford were competitors in more than a few fields, but by forming an alliance between these two companies, a system was created in which more effective, more productive analytical technologies and products could be supplied to the market.

### The Development of X-ray Analysis Instruments

Analysis instruments that utilize X-rays are used in the compositional analysis and structural analysis of various types of materials ranging from organic and inorganic materials to living organisms. In particular, because these instruments make it possible to perform non-destructive analysis of samples, such instruments are not limited to laboratory applications; they are also being used widely in quality control at factories. Horiba has developed X-ray-related products based on a variety of sensor technologies, such as the Nal (TI) scintillation detector and the semiconductor radiation detector (SSD), which has high energy resolution and a broad range of application. Believing that it is the technologies and products best suited to their applications that will be accepted and survive in the market, Horiba is focusing on fields in which it can fully utilize the characteristics of the technologies in which it specializes, working in these fields to develop truly practical, useful products.

However, as stated at the beginning of this paper, the market is becoming increasingly diverse and undergoing rapid change, so conventional business methods are no longer sufficient, and may even no longer be necessary. Therefore, Horiba is aggressively pursuing alliances with businesses and organizations around the world, working tirelessly on innovations that will allow us to continue to provide products and services that truly satisfy our customers.

In the following sections, I shall introduce a few of these types of projects.

### 4 Energy Dispersive X-ray Analyzers

An energy dispersive X-ray analyzer (EDX) is used in conjunction with a scanning electron microscope (SEM) or a transmission electron microscope (TEM) to perform element analysis of microscopic areas by observing an image displayed on a monitor. With an EDX, quantitative analysis, qualitative analysis, and element distribution imaging can be performed easily for user-specified microscopic areas of a sample. Today, these instruments are indispensable to the research and development of various types of materials and to the defect analysis of electronic parts.

#### 4.1 EMAX Series

Horiba first commercialized an EDX instrument, which included an 8-bit microcomputer, in Japan in 1976. Since that time, Horiba has introduced the EMAX Series to meet the various needs of EDX analysis. The first EDX was designed to be used by specialists in research laboratories, analysis centers, etc., and its main features were a high-level of analysis precision and data analysis. For example, the instrument featured a phase analysis function that used the chemometrics method to count the number of phases in the same type of composition, and to accurately compute the element composition of each phase.

The recently marketed EMAX Energy features not only high sensitivity and high speed, but also a high operability such as built-in report creation functions greatly increase the efficiency of analysis work. This instrument has been extremely well received by our users.

The advantages offered by the high purity silicon detector (HP-Si), an original technology developed by Horiba, are put to full use in our liquid nitrogen-free detectors.

HP-Si is highly stable and can be stored at room temperature, making this instrument extremely good operability.
4.2 SEMEDX Series

The SEM, on the other hand, is being used for on-site quality control, etc., by semiconductor and material manufacturers. One of the primary objectives of SEM design is to make the equipment easy for anyone to operate. While there is indeed an increasing number of SEMs that incorporate EDX, the software for the two instruments is developed separately, making it a bit difficult to efficiently operate the instruments as one system.

Therefore, Horiba has formed an alliance with Hitachi Ltd./Hitachi Science Systems Ltd., a leading electron microscope manufacturer, and Oxford Instruments, plc, a leader in application software. Through this alliance, we have commercialized the SEMEDX Series analysis system that combines SEM and EDX into one unit designed with the goals of being easy for anyone to operate, providing fast, high-resolution SEM observation capabilities and accurate EDX analysis.

In the development of the SEMEDX, our three companies examined the question of what was needed to make the instrument easy for our customers to use, and each company worked in its fields of expertise to create the SEMEDX. This instrument is a system upgrade that provides consistent, unified operation for both SEM and EDX, and is targeted at engineers who do not need to use analytical equipment frequently.

5 X-ray Fluorescence Element Analyzer

X-ray fluorescence Element Analyzer (XRF) performs element analysis by irradiating a sample with x-rays and then measuring the material-specific x-rays that are generated. XRF allows non-destructive analysis, and measurements can be performed under standard atmospheric conditions, so XRF can be used in a wide range of applications, from the measurement of solid samples to liquids and powders. The XRF is marketed in a diverse range of formats, including instruments for general measurements, specialized instruments with a specific range of application, high-performance instruments designed for maximum sensitivity, and cost-effective general-purpose instruments.

5.1 MESA-500 Series

Horiba’s entry into the XRF-related product market consists of the MESA Series of energy dispersive x-ray fluorescence analyzers. The X-Ray Fluorescence Element Analyzer uses FPM standardless quantification software, allowing easy, high-precision quantitative analysis of samples for which the structure is unknown, and analysis even when no suitable standard sample is available. The x-ray detector uses an HP-Si detector, so liquid nitrogen only needs to be supplied while measurements are actually being made, making maintenance easy as well.

5.2 X-ray Analytical Microscope

Conventional wisdom states that, while XRF instruments offer the advantage of being able to analyze a sample as is, they cannot be used to analyze microscopic areas. This problem has been solved with the XGT Series x-ray analytical microscopes. The XGT-2700W can perform element analysis and imaging for a wide range of sample area sizes, from a minimum area diameter of 10mm to a maximum area of 100mm×100mm.

The basis for the x-ray guide tube (XGT) was developed by Hiroki Nakazawa of the National Institute for Research in Inorganic Materials. With support from the Japan Science and Technology Corporation, Horiba further developed this technology and combined it with Horiba’s EDX technologies and micromechanics to create the world’s first desktop x-ray analysis microscope products. This is an excellent example of the results that can be achieved through an alliance of advanced research and manufacturer know-how.

5.3 MDX Series

MDX Series units are element analyzers that combine the functions of both EDX and WDX in one compact unit. The MDX Series is manufactured by Oxford Instruments, plc and sold by Horiba.

Used in instruments like the MESA an XGT, the EDX is easy to operate and is used in a wide-range of applications, from specialized instruments to general-purpose instruments. However, the EDX has lower sensitivity than the WDX with respect to light elements, and is not suitable for use in the element analysis of ultra-microscopic amounts.

The MDX combines WDX and EDX in one unit, making it an extremely flexible general-purpose element analyzer with which either or both of the WDX and EDX can be used depending on sample properties, analysis objectives, etc.
In Conclusion

This concludes my introduction of the trends in Horiba's products and technologies for laboratory-use analysis instruments, particularly those related to x-ray analysis.

With the start of the 21st century, we can expect to see increasing demand for the following functions in laboratory-use analysis instruments.

1. High-performance (analysis of trace elements and transient phenomena).
2. Speedy analysis (high throughput).
3. High-level data analysis and visual display of analysis results.
4. A basis of standard scales (global standards).
5. Easy operation by any user -- the most important consideration of all.

All of these needs are extremely important to analytical instrument manufacturers, and none may be overlooked. As with the examples introduced in this paper, Horiba shall continue to provide speedy solutions to our customers needs by forming effective partnerships and timely alliances based on the needs of the market and on Horiba's core technologies.

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