

Near-Infrared Spectroscopic Probe for Diverse Applications

Takeaki Ishii*¹ Risa Hara*¹ Kazuyuki Kakehi*²
Toshiyuki Saruya*¹ Kazuhiko Inamura*¹

Inline measurement technologies for low-transparency process streams of raw materials have potential applications across a wide range of industries. Toward this end, we investigated a near-infrared spectroscopic probe to address key challenges in inline analysis. The probe's first feature is a window-mounted design for pipes or tanks that enables quick installation and removal. This design allows the probe to be removed from the production line without interrupting production. As a result, calibration models can be developed and maintained in the laboratory. The second feature is a periodic reference measurement function that enables correction for changes in the measurement system while the probe remains installed. We experimentally evaluated this function and confirmed that it suppresses absorbance fluctuations under variations in spectrometer temperature.

INTRODUCTION

Yokogawa Electric Corporation aims to contribute to the automation of manufacturing processes that still depend on human operation by enabling inline measurement of the composition and physical properties of raw materials during production.

Among various inline measurement technologies, we focus specifically on those targeting raw materials with low optical transparency. There are two primary reasons for this focus. First, some raw materials cannot be properly measured using the same methods applied to highly transparent materials and therefore require a different approach. Second, the scope of potential applications is wide. Because raw materials with low optical transparency are handled across diverse industries—such as food, cosmetics, pharmaceuticals, and resins—the associated measurement technologies have the potential for extensive cross-sector applicability.

Against this background, this paper first describes the characteristics of raw materials with low optical transparency and provides an overview of inline measurement, which is essential for monitoring the state of these materials during manufacturing processes. Next, the paper discusses the role of inline sensors in Yokogawa's solutions for the food manufacturing industry and presents examples of their application and the value they provide. Finally, the paper introduces a case study on the development of an inline probe for a near-infrared spectrometer designed to measure raw materials with low optical transparency.

CHARACTERISTICS OF RAW MATERIALS WITH LOW OPTICAL TRANSPARENCY (DISPERSED SYSTEMS)

Raw materials with low optical transparency often take the form of dispersed systems or colloids. Depending on the state of the dispersion medium (continuous phase) and the dispersed substance (dispersed phase), these materials are classified into powders, emulsions, gels, suspensions, and similar systems.

Examples of dispersed systems can be found in everyday food products⁽¹⁾. For instance, wheat flour and soybean flour are powders. Milk is an O/W (oil-in-water) emulsion, butter is a W/O (water-in-oil) emulsion, and yogurt is a gel. Thus, even within the same category of dairy products, the physical states as dispersed systems vary considerably.

An important consideration in the measurement of raw materials with low optical transparency is that the dispersion state may change as a result of dilution or the passage of time. The inline measurement method described below is designed to prevent such changes and to monitor the actual state of the raw materials during the manufacturing process.

TYPES OF ANALYTICAL METHODS AND THE POSITIONING OF INLINE MEASUREMENT

In production processes, it is necessary both to assess the state or properties of raw materials and to adjust the process parameters accordingly. Process control is particularly critical in processes where the composition of raw materials varies significantly, or where unstable reactions or products may occur. For example, food manufacturing processes that use agricultural products as raw materials require strict control because the composition of agricultural raw materials varies with the season and region of production. In contrast, in

*1 Innovation Division, Marketing Headquarters

*2 Yokogawa Solution Service Corporation

processes with low raw material variability and high process reproducibility, the need for continuous monitoring of raw materials is relatively low.

Analytical methods for raw materials are classified as inline, online, at-line, or offline, according to the location of the analytical instruments (Figure 1)⁽²⁾⁽³⁾. Methods listed earlier in this sequence offer superior real-time capability, whereas analytical accuracy generally tends to be lower.

The inline method offers the highest level of real-time performance, as the analytical instrument is directly integrated into the manufacturing equipment. All other methods require sampling prior to analysis. In the online method, sampling is performed automatically. The remaining two methods rely on manual sampling. In the at-line method, analysis is conducted near the sampling point, whereas in the offline method, analysis is carried out in a separate laboratory located away from the sampling point.

For measurements intended for continuous monitoring or control, it is necessary to employ either inline or online methods. The advantage of inline measurement lies in its ability to directly observe the actual state of the sample within the manufacturing equipment. In contrast, the advantage of online measurement is that analytical accuracy can be improved by applying sample pretreatment.

EXAMPLE OF A SOLUTION UTILIZING INLINE SENSORS

We next present a solution for the food manufacturing industry as an example of integrating inline sensors with higher-level systems.

Role of Inline Sensors in Solutions for the Food Manufacturing Industry

In Yokogawa's Life Business, the vision is to address

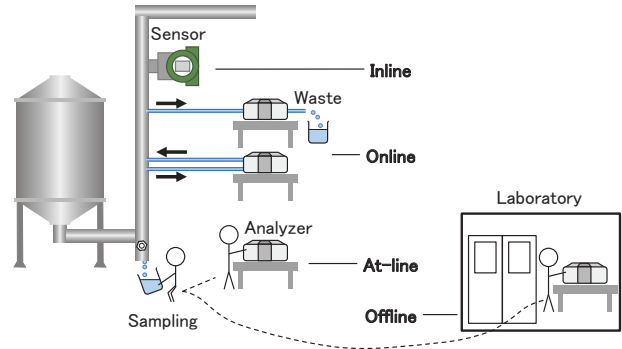


Figure 1 Classification of analytical methods

manufacturing challenges by *measuring* production processes, *visualizing* production instructions and results, and *connecting* the production floor with management. Using the food manufacturing industry as an example, Figure 2⁽⁴⁾ illustrates how this vision can be realized.

The food manufacturing industry faces significant challenges, such as balancing quality with cost efficiency and addressing labor shortages. Factors contributing to these issues include the continued use of paper-based work instructions, handwritten production records, and delays in the automation of weighing, feeding, and sampling operations. Inline sensors are utilized in the *measuring* domain as a solution to the automation-related issues mentioned above. The following section describes a specific example focusing on the automation of sampling inspections and subsequent adjustments based on their results.

Implementation of Automatic Control Using Inline Sensors

If inline concentration measurement is achieved, the processes of taking samples from manufacturing equipment, measuring its component concentration, and adjusting the

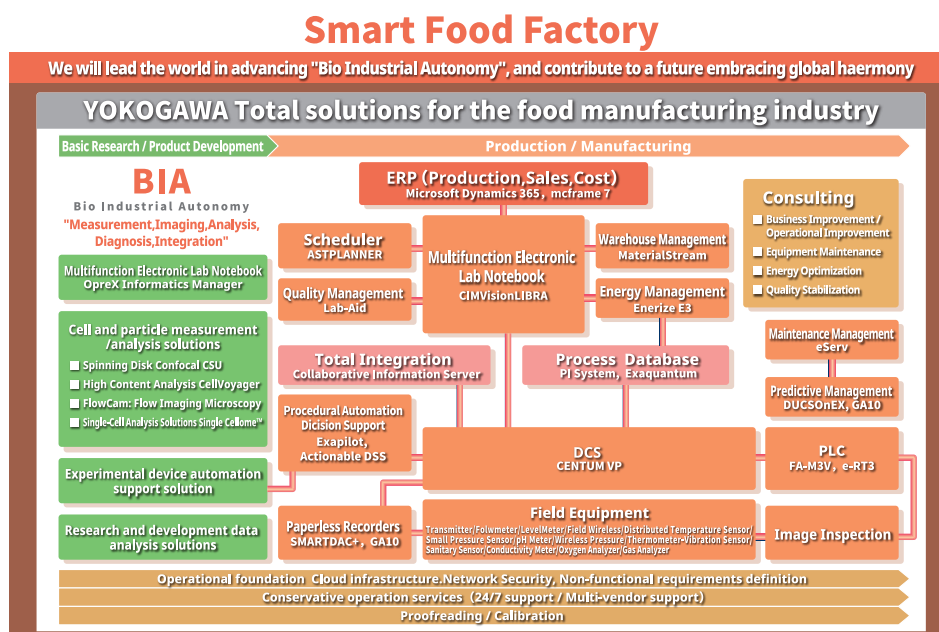


Figure 2 Yokogawa total solution for the food manufacturing industry⁽⁴⁾

concentration based on the measured value can be automated (Figure 3). In this configuration, the manufacturing execution system (MES) provides the target concentration specified for the product to the distributed control system (DCS) or programmable logic controller (PLC). The DCS or PLC compares the component concentration measured by the inline sensor with the target concentration received from the MES and controls the raw-material concentration inside the process equipment in real time.

The implementation of such automated control is expected to provide a broad range of benefits, including addressing labor shortages, stabilizing product quality, preventing out-of-specification products, shortening adjustment times, and reducing contamination risks.

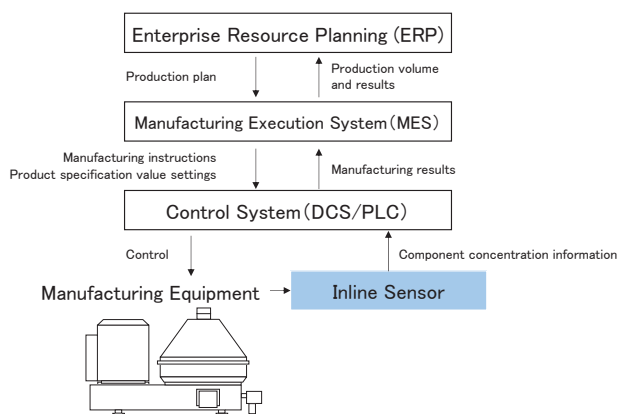


Figure 3 Example of automation using an inline sensor

NEAR-INFRARED SPECTROSCOPY

Near-infrared spectroscopy is often used for offline analysis, but it can also be used for inline and online analysis, and even for process control based on analytical results. For example, Yokogawa's InfraSpec NR800 Fourier Transform Near-Infrared Analyzer⁽⁵⁾ is employed not only for analysis but also for process control⁽⁶⁾.

Characteristics of Near-Infrared Spectroscopy⁽³⁾⁽⁷⁾

Near-infrared spectroscopy is a technique used to obtain information based on changes in the intensity of light in the near-infrared region (wavelength range: 800–2500 nm) arising from the interaction of the light with the sample. Variations in light intensity arise from absorption and scattering by components in the sample, and their wavelength dependence (the spectrum) contains information related to molecular structure and intermolecular interactions. Such information is typically extracted using calibration models developed by multivariate analysis, which are used to predict the composition or physical properties of the sample from its spectrum.

A distinctive aspect of near-infrared spectroscopy is that, unlike conventional measuring instruments such as thermometers and pressure gauges, it requires the development of a calibration model for each measurement item. This characteristic offers several advantages, including applicability

to a wide range of prediction targets and the ability to predict multiple measurement parameters simultaneously, once the calibration models have been developed. On the other hand, disadvantages include the considerable time and effort required to develop calibration models, the fact that the achievable predictive performance cannot always be assessed in advance, and the need for maintenance of the calibration models if predictive performance deteriorates during operation. It should also be noted that the predictive performance of near-infrared spectroscopy depends not only on the measurement method and hardware performance but also on how the calibration models are developed.

Near-infrared spectroscopy encompasses various measurement techniques, and an appropriate method is selected according to the sample to be measured. The transmission method measures the intensity of light transmitted through the sample and is used for materials with high optical transparency. For example, Yokogawa's InfraSpec NR800 Fourier Transform Near-Infrared Analyzer is primarily designed to measure raw materials with high optical transparency using the transmission method. In contrast, the diffuse reflectance method measures light that emerges after undergoing multiple scattering events within the sample, and is used for samples with low transparency. Since the probe examined in this paper is intended for raw materials with low optical transparency, the diffuse reflectance method was employed.

Challenges in Inline Measurement

Several issues may limit the broader application of inline measurement using near-infrared spectroscopy. These include the fact that equipment offering an appropriate balance between cost and performance for the intended application may not always be available, the substantial effort required to develop and maintain calibration models, and the possibility that predictive performance may be insufficient to replace conventional offline analysis. These challenges are discussed below.

- 1) Cost-effectiveness: Several factors increase the cost of inline measurement devices. Because each device can take measurements only at its installation point, a separate instrument is required for each measurement point. In addition, the instruments require structures suitable for field environments (e.g., dustproof, waterproof, or explosion-proof designs) as well as additional components (e.g., optical fibers or sampling units). Furthermore, constraints on the system configuration often force the use of spectrometers with higher performance specifications than necessary (e.g., measurement wavelength range, wavelength resolution, signal-to-noise ratio), which in turn lowers cost-effectiveness.
- 2) Development and maintenance of calibration models: Acquiring data for calibration model development while the inline measurement instrument is installed on production piping or tanks requires a large quantity of raw materials. In addition, in order to develop calibration models with

good predictive performance, data covering the full measurement range of the target parameter are necessary. However, collecting a sufficiently wide range of data during production, including compositions that fall outside specification limits, can be challenging.

- 3) Predictive performance: Inline measurement instruments are affected by field environments, including temperature, humidity, and vibration. Consequently, the accuracy of spectra acquired by near-infrared spectroscopy tends to be lower under inline conditions than in laboratory settings. Such adverse effects on the acquired spectra, together with variability in measurement conditions because sample pretreatment cannot be performed in inline settings, ultimately lead to deterioration in the predictive performance of calibration models.

DIFFUSE REFLECTANCE PROBE PROTOTYPE DEVELOPMENT

This paper examined the functions of an inline diffuse reflectance probe designed for measuring raw materials with low optical transparency and attempted to address some of the challenges described above.

- 1) Cost-effectiveness: We aimed to design the system with a structure that allows the spectrometer and optical components to be replaced, to optimize both cost and performance for each application. In addition, in order to maximize the performance of the spectrometer, the probe was equipped with a compensation function (reference measurement function). By assuming periodic reference measurements, a wider range of spectrometers becomes applicable, allowing for cost reduction and simplification of the overall system.
- 2) Development and maintenance of calibration models: Our concept was to develop calibration models by moving the inline probe to the laboratory and measuring samples prepared with various compositions. To accomplish this, we designed a structure that allows the probe to be mounted facing the window of a sight glass installed on piping or tanks, allowing the probe to be installed and removed without interrupting production.
- 3) Predictive performance: To ensure stable predictive performance in a field environment, we designed a structure that enables periodic reference measurements even while the probe remains connected to the piping.

Characteristics of the Probe

The prototype probe has two principal features: ease of installation and removal, and a built-in reference measurement function.

The ease of installation and removal was realized by a structure in which the sample inside the sight glass is measured by a probe mounted using a clamp (Figure 4). Owing to this non-wetted structure, the probe can be attached or detached without interrupting production or disassembling the piping. This facilitates startup operations and maintenance when predictive performance deteriorates.

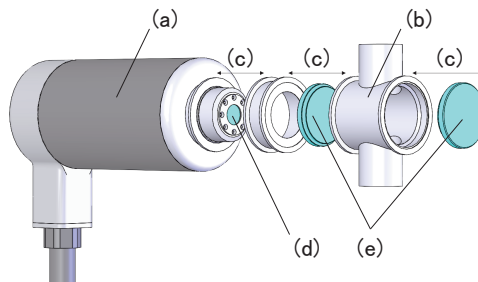


Figure 4 Probe and sight glass connection method. (a) Probe, (b) Sight glass, (c) Clamp mounting location, (d) Probe window, (e) Sight glass window

Figure 5 shows the positional relationship between the illumination unit and the detection unit, which together constitute the optical system inside the probe. The optical mounting component (a) holds both the illumination unit (b) and the detection unit (c) in place. In this optical system, the illumination unit emits light toward the measurement target, and the detection unit collects light scattered from the target.

Scattered light collected by the detection unit is guided to the spectrometer. The spectrometer can be placed either inside or outside the probe. Placing it inside the probe allows for a more compact configuration, but the spectrometer becomes more susceptible to the environmental influences at the probe installation site. Placing it outside the probe avoids such environmental influences. In this case, however, the scattered light must be guided to the spectrometer via an optical fiber, which increases cost. In addition, attenuation in the optical fiber or fiber bending may adversely affect the measurement results.

Since the window of the sight glass is in direct contact with the measurement target, sapphire with a thickness of 12 mm was selected in consideration of the risk of fracture. To ensure consistent measurement conditions for reference measurement (B) and sample measurement (C), the optical configuration must be adjusted according to the thickness of the sight glass window. The switching mechanism between these configurations was implemented as part of the second feature, the reference measurement function. The details of

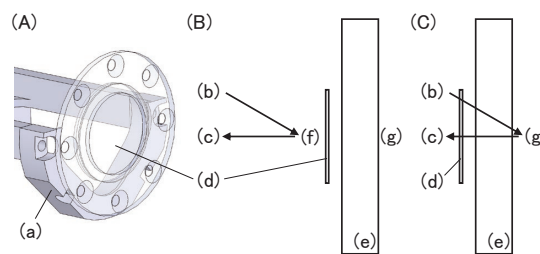


Figure 5 Positional relationship between irradiation and detection (arrows indicate the optical path). (A) Portion of the probe, (B) Configuration during reference measurement, (C) Configuration during sample measurement, (a) Optical mounting component, (b) Illumination unit, (c) Detection unit, (d) Probe window, (e) Sight glass window, (f) Reference, (g) Sample

this feature are described below.

To implement the reference measurement function, a shutter with a reflective plate for reference measurements was integrated into the probe. This shutter enables periodic reference measurements to be performed even while the probe remains installed on the piping, thereby compensating for changes in the instrument's condition. The operation of the shutter is shown in Figure 6. During the transition from the reference-measurement configuration (A) to the sample-measurement configuration (B), a cylindrical cam drives the shutter's rotational motion synchronized with the linear motion of the optical system. In the sample-measurement configuration, the shutter moves to a position that avoids the optical system, allowing the optical system to move close to the probe window. The switching of configurations is actuated by compressed air and controlled by a solenoid valve. In the following evaluation, this shutter mechanism was used to automatically alternate between reference and sample measurements.

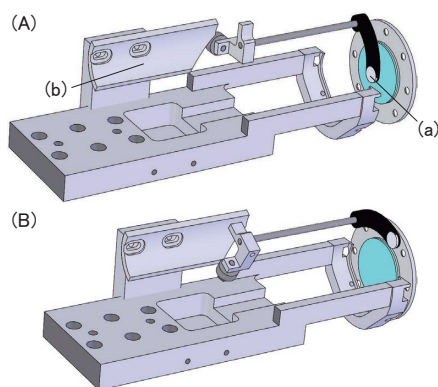


Figure 6 Linear motion of the optical system and rotational motion of the shutter. (A) Configuration during reference measurement, (B) Configuration during sample measurement, (a) Reflective plate for reference measurement, (b) Cylindrical cam

Evaluation of the Effectiveness of the Reference Measurement

The effectiveness of the reference measurement function was evaluated by using the variation in absorbance, as defined by the equation below, during continuous measurements of the same sample.

$$A = -\log_{10} \frac{I}{I_0}$$

Here, I_0 denotes the detected light intensity in the reference measurement, and I denotes the detected light intensity in the sample measurement.

When the same sample is measured continuously, the absorbance should ideally remain constant. However, changes in the instrument's operating conditions may lead to variations in absorbance. These changes include factors common to both reference and sample measurements (e.g., changes in spectrometer temperature or illumination intensity) and

those unique to each measurement. Only the former can be compensated for by the reference measurement. This paper evaluated whether absorbance variations caused by changes in spectrometer temperature—one of the factors common to both measurements—can be compensated for by periodic reference measurements.

The evaluation setup is shown in Figure 7. Two thermostatic chambers were used. In one chamber, the ambient temperature of the spectrometer was varied between 20°C and 30°C, while in the other chamber, the temperature around the probe and sample was maintained at 30°C. To reduce the effects of airflow inside the chambers, the spectrometer and probe were placed in a box. The sample was a diffuse white standard attached to the inner surface of the sight glass window. A tungsten lamp was used as the light source, and a commercially available Fourier transform near-infrared spectrometer was employed. Spectra averaged over 64 scans were alternately acquired for the reference and the sample. In the subsequent data processing, absorbance was calculated approximately every 17 s.

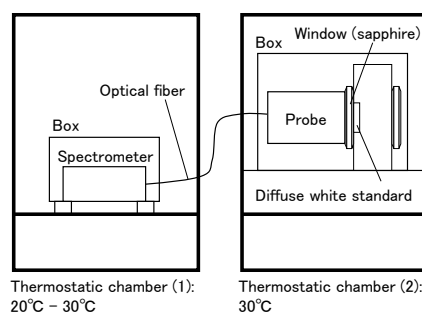


Figure 7 Evaluation setup. The spectrometer temperature was varied in thermostatic chamber (1), while the probe and the sample were maintained at a constant temperature in thermostatic chamber (2).

The temperature profile of the thermostatic chamber containing the spectrometer was as follows. One cycle consisted of heating the thermostatic chamber from 20°C to 30°C at a rate of 10°C per 60 min, holding it at 30°C for 30 min, cooling it back to 20°C at a rate of 10°C per 60 min, and then holding it at 20°C for 30 min. This cycle was repeated twice. The measured ambient temperature within the thermostatic chamber containing the spectrometer and the internal temperature of the spectrometer are shown in Figure 8.

Figure 9 compares the changes in absorbance when the reference was measured only once at the beginning and when it was measured before each sample measurement. After the measurements began, the absorbance remained stable regardless of the frequency of reference measurement, as long as the internal temperature of the spectrometer remained constant. Once the internal temperature of the spectrometer began to change, the absorbance varied significantly when the reference was measured only once. In contrast, when the reference was measured before each sample measurement, the variation in absorbance was suppressed to approximately $\pm 10^{-4}$, which is well below the target range of $\pm 10^{-3}$.

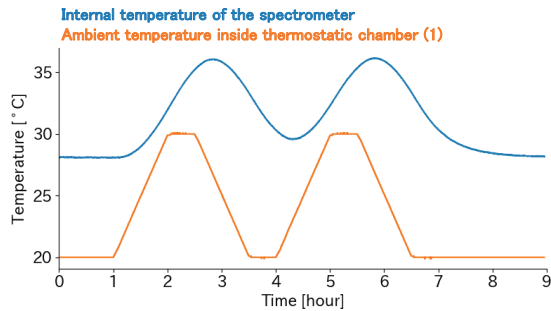


Figure 8 Measured ambient temperature inside the thermostatic chamber containing the spectrometer, and the internal temperature of the spectrometer

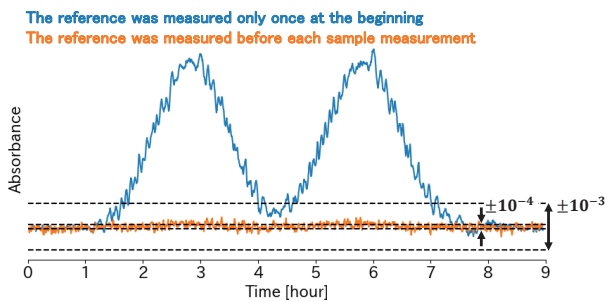


Figure 9 Comparison of absorbance changes at 5000 cm^{-1} depending on the frequency of reference measurements. The absorbance data were smoothed using a 1-min moving-average window.

These results demonstrate that the probe's reference measurement function effectively compensates for variation in absorbance caused by temperature fluctuations inside the spectrometer.

CONCLUSION

In this paper, we address the challenges associated with inline measurements by focusing on the functional design of a near-infrared spectroscopic probe intended for use in diverse manufacturing processes that handle raw materials with low optical transparency. The prototype probe measures through the observation window of piping or tanks, providing a non-wetted structure that allows easy installation and removal. It also enables periodic reference measurements even while remaining installed on the piping. We present evaluation results demonstrating that the probe's reference measurement function can suppress absorbance variations to a sufficiently

small range relative to the target value, even under fluctuating spectrometer temperatures.

Finally, we summarize several items that require further investigation for practical application of the probe discussed in this paper. First, to improve performance under actual field conditions, it is necessary to establish a probe structure that minimizes the configuration-specific variations induced by external environmental changes in both the reference and sample measurement configurations. Second, to achieve an appropriate balance between cost and performance for a given application at the system level, a systematic method is required for estimating the spectrometer performance necessary to satisfy the specified requirements. Finally, from an operational perspective, the capability to accommodate diverse raw materials and prediction targets in manufacturing environments must be improved. To this end, a practical method for the easy development and management of calibration models is required.

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