Inline Spectroscopy: From Concept to Function

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Abstract

The field of applied spectroscopy is strongly dominated by publications presenting proof-of-concepts, lab set-ups, and demonstrations. In contrast, the corresponding number of commercial successes of inline spectroscopy is surprisingly lower. This article discusses inline spectroscopy from an instrumentation perspective. It is the authors' firm belief that the success of inline spectroscopy lies in the understanding of how the design and implementation of the optical instrumentation affects the data quality, and how this in turn will limit or enhance the performance of the prediction model. This article emphasizes the need for a strong, multidisciplinary design team, whose design process is rooted in first principles, to bridge the technology "valley of death" and convert research in applied spectroscopy into commercially successful solutions.

Introduction

Equipment for performing spectroscopic measurements, e.g., Fourier transform infrared (FT-IR), grating-based visible (Vis), and near-infrared (NIR) instruments, is available in labs in a wide range of facilities ranging from universities, chemical factories, food processing industry, recycling industry, and more. There are many examples of applied spectroscopy where samples are brought from a process line to the instrument either in the lab or at-line as part of the day-to-day quality control procedures in process lines.[1,2] However, the ultimate goal for operators and quality managers is reliable, nondestructive analysis of samples inline. Although inline spectroscopy exists,[3–9] we believe there is potential for more successful, fully functional inline systems and that instrumentation design plays a large role in this.

Researchers and industrial partners continue to join forces in large-scale investigations to measure, model, and determine if spectroscopy has potential in a wide range of application fields,[10,11] including pharmaceutical,[12–15] food,[16–20] waste sorting,[21–23] bioprocess monitoring,[24] and metallurgy.[25–28] The potential for inline measurements is ever-increasing as new, enabling technology is being developed, e.g., enhanced silicon and InGaAs detectors,[29,30] OEM spectrometers,[31–33] and light-emitting diode (LED) sources.[34]

The objective of this review article is to approach inline spectroscopy from a solution design perspective.

The article aims to explore the overlap between the world of applied spectroscopy–chemometrics and the world of optical measurement system design. Building an inline measurement system requires a profound understanding of the process where it will be installed, having in-depth knowledge of what can be measured spectroscopically, and understanding and respecting the laws of physics that set the limits for any instrumentation design.

A Definition of Terms

For consistency, the following glossary of terms is given:

Component is used for lenses, LEDs, gratings, detectors, light sources, read-out electronics, and amplifier cards. Suppliers of optical components include Hamamatsu, Edmund Optics, Thorlabs, Excelitas, etc.

Instrument can be used for spectrometers (grating-based, diffractive optical element-based, FT-IR, etc.) or hyperspectral imaging cameras. The instrument can sometimes include an integrated source. It can be general-purpose or customized for the application. Suppliers of general-purpose

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spectrometers include Ocean Optics, Ibsen Photonics, Wasatch Photonics, Avantes, Spectral Engines, BW-TEC AG, Bruker, etc.

Optical measurement solution is an application driven design that turns data to information. It includes the instrument, light source, optics, software, data analysis, and sample presentation/handling. Companies that deliver complete measurement solutions include Tomra, Foss, Bruker, RTT, etc.

The Design Process

To have a successful process, the correct resources must be allocated and a road map for the process is required. Figure 1 illustrates the personnel resources that should be available during the design process. Not all of these people are always available, and they do not need to be involved at each stage of the design process, but all have valuable input that the core design team should utilize.

Figure 2 illustrates the suggested methodology for designing an inline measurement solution. The inputs to

the design process are the description of the application challenge and any relevant a priori knowledge that the design team brings to the table. An initial layout for the solution is also needed. The final goal of the design process is a commercial solution. This article will focus on the two middle steps, i.e., controlled characterization measurements and measurements in realistic conditions. However, these two steps are "sandwiched" between two very important control tracks that can be the difference between success and failure, and these are also described in this article.

(a) Entering the design process requires full mapping the end-user requirements and, as the design proceeds, it is beneficial to continue to "check-in" with end-user experts to ensure expectations are still aligned.

(b) The expected performance (accuracy) needs to be analyzed iteratively to optimize the design process. Performance analysis should never be left until the end of the design process.
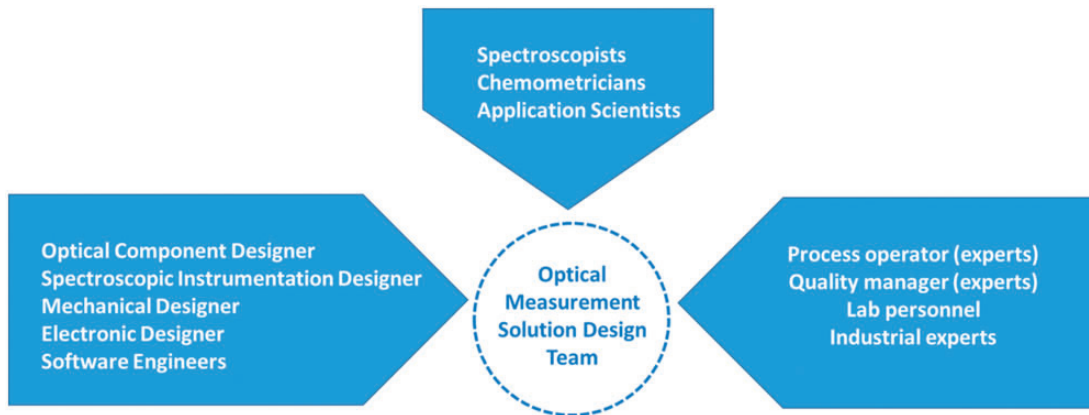

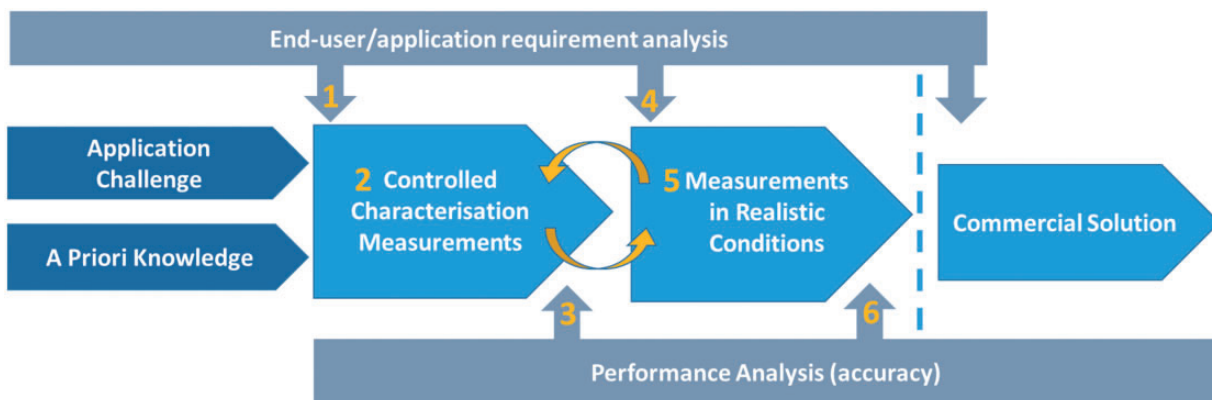**Figure 1.** Design team building.

Figure 2. Time progression and process for inline measurement solution design process.

In Fig. 2, the numbers 1–6 indicate the design progression and these steps will be described in the article. Depending of the complexity of the application, it may be required to repeat some stages. For example, controlled characterization measurements may be required again after the measurements in realistic conditions if the performance analysis reveals some remaining challenges or opportunities. In some instances, the controlled characterization measurements can be quite straightforward and other times more complicated and lengthy.

End User–Application Requirement Analysis Details

Ideally, the design process starts with the end-user requirement (Fig. 2, step 1). Selecting or designing technology for a functioning inline spectroscopic system requires a clear prioritization of what is acceptable to the end user. It gives the designers a framework within which they have design freedom. It is recommended that this discussion takes place at an end-user site so that it is easier to visualize how the final implementation will be.

Examples of end-user requirements that are essential input for the instrument requirement definition are:

- How does the end user currently measure (if at all) and how does it perform or limit them? What are the challenges with the measurements, e.g., are they too subjective, time consuming, or not fully representative?
- What measurement speed is required, e.g., are there belt speeds that must be considered?
- Is a running average sufficient or is it critical to interpret spatially or time-resolved variation?
- What classification/characterization accuracy is necessary? Be clear what definition of accuracy is (e.g., 1% on petrol pump can mean 3-sigma, 1% for a scientist can mean 1-sigma).
- Is it possible to have a contact measurement or is non-contact essential?
- Will the distance from the object to the instrument vary? This is important as it affects the required depth of field of the optical system.
- What is the expected range of sample variation that needs be measured, e.g., minced meat with a potential fat variation of 3–15% or 1–50%.
- What is the expected unwanted or interfering variation in the samples, i.e., what should the measurement be immune to, e.g., seasonal variation, size, and color?
- What do you want to measure and what do you need to distinguish it from, e.g., an end user that wants to sort PET plastic out of a waste stream but also requires that all green PETs should end up in their own pile.
- What is a reasonable development cost and final system price?

- How much does the measurement environment vary, e.g., temperature, humidity, daylight through windows?
- Is it possible to retrofit the new system to an existing line or will a new line be developed?
- What cleaning and validation steps are required to meet industry standards and government regulations?

Each requirement should be discussed in the context of how much leeway there is from the application side and how this affects the solution design. On day one, the end user will often request the best accuracy at the highest speed. However, it is important that the end user understands how this affects the solution design, since it is generally the case that greater accuracy requires lower speed and vice versa. Being inflexible with the solution requirement will limit the design process, whereas understanding the leeway aids the design process. This is a two-way discussion, where the end user needs to understand the consequence of their requirements and the design team learns how best to prioritize these requirements going forward.

Controlled Characterization Measurements

The decision to develop an inline spectroscopic measurement system can either be based on what is found in literature or based on prediction models that have been previously developed from measurements in the lab, using general-purpose instrumentation. When the decision to go inline is made, controlled measurements (Fig. 2, step 2) will need to be performed and analyzed in the online context. For example, if heterogeneity is foreseen as one of the main inline challenges, it is useful to start with homogeneous samples and gradually introduce heterogeneity in a controlled manner when presenting the sample. Another example could be sample height variation and, in this case, it is important to understand how much going from a limited depth of field to a larger depth of field reduces optical throughput.

From an instrumentation perspective, an optimal measurement solution design requires a full understanding of the optical–spectral signal. To start moving from a lab concept to a dedicated inline spectrometer, it is therefore critical to look at your data! How much contrast is in the signal relative to the intensity? How much does your signal vary with sample heterogeneity? How distinguishable is the signal from interfering parameters?

First, a good starting point for defining your instrumentation baseline is answering the following question:

> In a controlled lab measurement, what acquisition time is required to obtain a good enough spectrum?

This measurement time (integration time multiplied by the number of repetitions) is the design baseline. A good

enough spectrum means answering yes to the following:

- Are the changes in the spectral features relative to the physical, target measurements (e.g., concentration), sufficient to achieve the required classification accuracy for the end user?
- Is the signal strength high enough relative to the instrument's statistical noise to distinguish changes in the spectral features?

For inline spectroscopy, it is often essential to maximize the number of photons reaching the detector so that the spectrum will be good enough. Increasing measurement time will give more photons, but if it is necessary to measure the same number of photons at higher speed, design parameters such as resolution, range, amplifier design and illumination design will become more crucial.

Second, the need for dynamics in the measurements should be addressed:

The characterization measurements must span the measurement range.

The spectra from extreme samples can reveal very important features that should be addressed in the final design. For absorption spectroscopy, it will be necessary to

- Measure samples with the highest concentration of the relevant substance so that you see the maximum possible absorption
- Measure samples with the lowest concentration of the relevant substance so that you see the minimum possible absorption

It is important to span range to build a robust prediction model and it is equally important to understand what the physical limits of the potential system in this range. For example, extreme measurements can reveal nonlinearities in absorption that are important to address when designing the dynamic range of the sensors. The dynamic range is closely tied to the electronics design, e.g., what analog-to-digital convertors (ADCs) should be chosen for the spectrometer.

Third, the spectral features should be studied in detail:

Study the spectral features (and the regression vectors, if already available).

Pre-studies and lab measurements may have higher spectral resolution than needed in the inline measurement situation. Protein, water and fat have broader spectral features in the NIR and do not necessarily require the resolution found in many general-purpose spectrometers, to be able to make a prediction. Reducing spectral resolution increases optical throughput and thereby allows greater online measurement speed.[19,35] Interpreting Raman shifts, however, requires high spectral resolution in the spectrometer so reducing resolution is not always possible. It is also important to remember that lab spectrometers often have poor sensitivity at the extremes of their spectral range and longer measurement times are often needed to examine features that are present here.

It is common to use the regression vectors to understand the key spectral features when building and validating prediction models.[36] However, it is just as valuable to use the information in the regression vector to optimize the optical measurement solution design process, e.g., when selecting wavelength and range in a customized spectrometer design. The regression vector can also identify the specific parts of the spectrum that hold the most important information, which can simplify the required components/instrumentation in the final design.

Finally, it is also relevant to take note of what additional sample handling or measurements were required to acquire good spectra in the lab:

How is the sample handled and measured in the lab?

The following factors may be crucial in the final inline and understanding them can better guide the design process.

- What was the required level of sample preparation? Was it mixed, flattened, crushed? If the sample is less prepared, at what point does the accuracy of the prediction fall below the desired level. This helps quantify the importance of sample preparation.
- How was the sample presented, i.e., geometry, distance, and angles between illumination, sample, and the detection unit. This also determines whether the optimal measurement geometry is reflection, transmission, or interactance.
- Was it necessary to limit the distance from the spectrometer to the sample to within a certain range to maintain prediction accuracy? If this range is smaller than the expected range for the inline solution, depth-of-field needs to be addressed before moving from the controlled environment.
- How often was ambient light correction performed? If ambient light affects the accuracy, it may need to be corrected for per measurement.
- Was the sample temperature within at a certain temperature for the tests? Does varying sample temperature reduce the prediction accuracy and how likely are inline temperature variations? Does knowing the sample temperature help improve the prediction model?
- Was the sample measured at fixed pressure or under controlled humidity conditions?
- Was the optical measurement path clearer than the intended industrial application, e.g., will there be more dust, steam etc. present in reality?

Performance Analysis (Accuracy)

The accuracy of a spectroscopic measurement solution is the degree to which the measurement output agrees with the correct value (usually measured by a standardized method). To identify the factors that limit the accuracy, it is necessary to build a prediction model using data measured with the system and apply it to new data measured using the same system, i.e., validation. Gaining an understanding of how different factors limit the accuracy of the controlled measurements and relating this to the anticipated accuracy of the measurements in realistic conditions, will ultimately lead to higher potential accuracy in the final design (Fig. 2, step 3).

It is important to identify the main factors that potentially limit the accuracy of the solution, which is equivalent to identifying the main sources of errors in the measurements. These limitations can be within the instrument, e.g., inherent random noise or wavelength drift. They can also be external to the instrument, such as sensitivity to varying sample presentation or unwanted light reflections in the measurement region.

It is also important to identify if the accuracy is limited in a random fluctuating way, or if it is more systematic. The word "random" does not have a consistent usage in literature and some sources of error may be regarded as random or systematic depending on context. We will define an error as random if it can be reduced by averaging repeated measurements of the same sample measurement situation under the same conditions, otherwise it is systematic. If the task is to measure an average value over a long series of measurements, the systematic errors will in general have the highest importance. Alternatively, if individual objects need to be measured accurately at a high speed, the random errors will be increasingly more important.

Based on this discussion, the sources of errors can be classified as random versus systematic, and internal to the instrument versus external or end-user specific. We can then group the errors in a 2×2 matrix, as shown in Fig. 3. In general, random variations can be improved by increasing signal to noise (averaging measurements, improving optical throughput, selecting a detector with higher response, stronger illumination, etc.), while systematic variations will require other design improvements.

Instrument-Specific Sources of Inaccuracy

Random Fluctuations in the Instrument. This is purely random noise that is inherent in any detector and electronics (Fig. 3). It includes thermal noise in the detector, photon shot noise, and noise from electronics and ADCs. The expression signal-to-noise ratio (SNR) of the instrument is defined using this random noise. It should be estimated by measuring the standard deviation of repeated measurements in a controlled, dark environment, such that this instrument-related noise is calculated independently from any variations caused by ambient light. This noise will consume some of the error tolerance in the prediction model and it is helpful to know how much is consumed.

The SNR can be improved by increasing signal (optimizing illumination, improving optical design for optimal optical throughput), reducing noise (choosing best detector, electronics, ADC combination), or by increasing time spent on each measurement.

If it is found that this random noise is insignificant, it might be possible to improve system reliability by reducing power consumption of the illumination, giving less heat, which is preferred for instrumentation in an air-tight housing. Decreasing the voltage of halogen lamps will also increase the lifetime of the bulbs.

	Random Fluctuations	Systematic Variation
Instrument	• Detector related (shot, thermal, electronic noise) • …	• Wavelength shift • Biased outlier detection • … • …
Application/ End-User	• Random sample heterogeneity • Sample size fluctuations • … • …	• Temporal batch variation • Systematic sample heterogeneity (e.g. layering) • … • …

Figure 3. Example of a source-of-inaccuracy matrix.

Electrical interference from motor controllers or switching power supplies can also increase the noise. If a system that works well in the controlled environment is noisier in the industrial environment, this is a likely cause. Improving immunity to such interference must be considered during design process.

Systematic Variations in the Instrument. Long term drift and other instabilities may include uncompensated temperature dependent gain, and changes due to aging, such as wavelength shift (Fig. 3) Detector arrays also have varying degree of fixed pattern noise, which may change slowly over time and temperature. It is *not* statistical noise. It can be improved using robust standardization techniques, e.g., frequent white spectrum calibration, frequent dark correction, and a robust temperature calibration based on temperature cycling and/or including additional cooling.

End User/Application-Specific Sources of Inaccuracy

It is important to identify which of the application driven sources of inaccuracy (Fig. 3) most effect the prediction. It is sometimes less clear if an application specific source of inaccuracy should be classified as random or systematic.

In the lab, it is common to shield the measurement from ambient light. This is not always possible in a real setting, where access is required for inspection and maintenance. Often, there will be fluctuations in ambient light, due to flicker from AC illumination or more slowly varying reflections or shadows from moving objects. Fluorescent lighting in factories may also have spectral features at shorter NIR wavelengths that can disturb some measurements. Correction for these fluctuations requires that the background signal correction occurs at a sufficiently high frequency. This is easier if LED illumination is used in the measurement solution but is more difficult with general broadband sources since some form of mechanical shutter will be needed. The instrument designer should aim to make the system reasonably robust towards varying ambient light; however, the end user may also need to make some adaptions, e.g., allowing the installation of a light-blocking curtain.

If the objects to be measured vary in temperature or humidity, this can give spectral changes that affect the result of the spectral analysis. This will give a systematic error that can often be reduced by modifying the prediction model, or by including temperature measurements as inputs to the model. Sample heterogeneity and size can give both systematic and random errors, depending on how the "repeated measurements" in our definition is done. Reflections from nearby surfaces, including the conveyer belt, can also give errors.

Another application-specific error is the error in the reference lab method required to build a prediction model, i.e., the target value in the prediction plot. Every reference method has its own inherent error, which decreases with the number of sample repeats. Using spectroscopy for sorting of manufactured materials such as plastic, is straightforward because obtaining an accurate reference method is simply a case of reading the manufacturing marking on the material.[37] However, this is not the case when a quantitative prediction model is required. Quantitative analysis of manufactured products such as pharmaceutical tablets, require preparation of controlled samples spanning the model range for building a model,[38] or offline reference methods measuring stratified (tablet) samples.[39] Accurate reference methods for biological samples are challenging because the methods can have significant systematic and random errors. Drip loss measurements for water holding capacity,[40] for example, is subjective as it is heavily dependent on how the samples are prepared for measurement. Soil analysis is based on sampling a very heterogeneous sample from a large spatial area and therefore representative sampling is challenging.[41,42] Reference methods for prediction of biomass pellet quality, will include several time-consuming steps like drying and weighting.[43] Sometimes lab methods do not exist, and the reference is based on an expert evaluation, e.g., sensory panel,[44] which is very subjective. If the measurement is to be done using a time varying process, it can be challenging to synchronize the spectroscopic measurement with the reference measure to ensure that both measurements have occurred simultaneously.[19,45]

When the sources of inaccuracy are identified, it often requires a collaborative effort between the end user and the design team to improve the performance. The end user may need to compromise, e.g., accepting longer measurement times or providing more controlled sample presentation. However, the design team will need to deal with challenges in the measurement situation that cannot be eliminated.

Sources of Inaccuracy and Solution Performance

In Fig. 4, the blue trace describes the relationship between accuracy and measurement time for a given measurement solution. The blue shaded area of the graph represents the measurement times where the accuracy of the measurement solution is limited by random fluctuations, which can be either instrument or application related. These errors can be reduced by averaging or increasing measurement time. At some point, the limit of the measurement solution will be reached and increasing measurement time will no longer improve accuracy. This is represented by the green shaded area, where the accuracy limit is increasingly dominated by systematic variations. The more dominated the solution is by systematic variation, the less influence measurement time has on the accuracy.

Generating the plot below requires applying a prediction model to either data that is measured at different
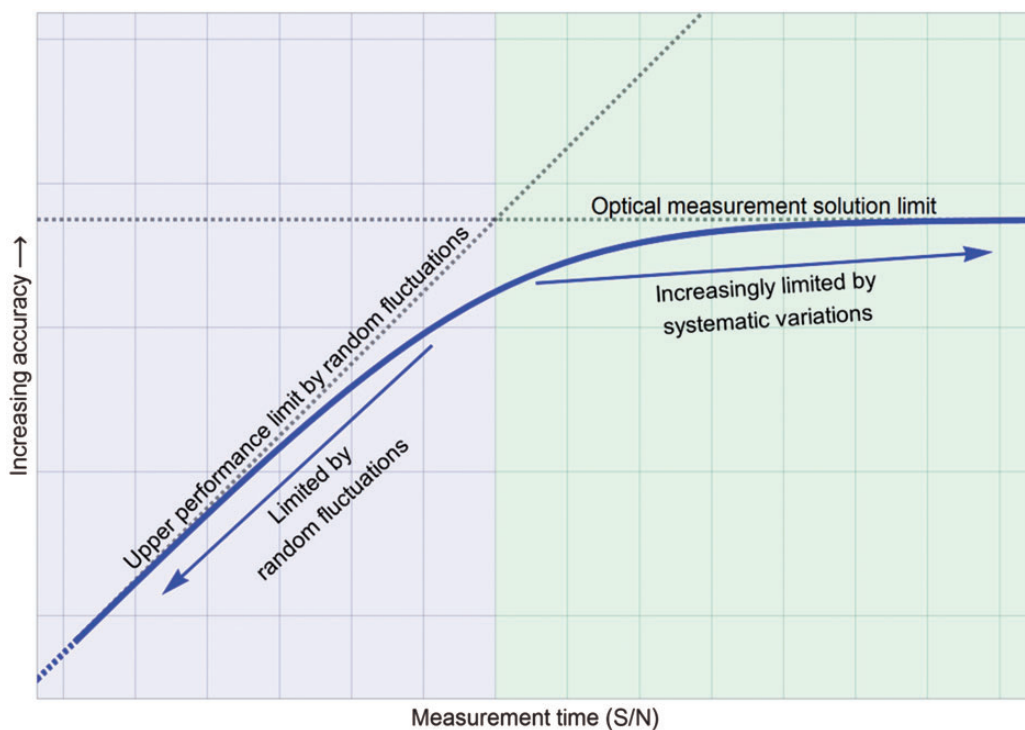
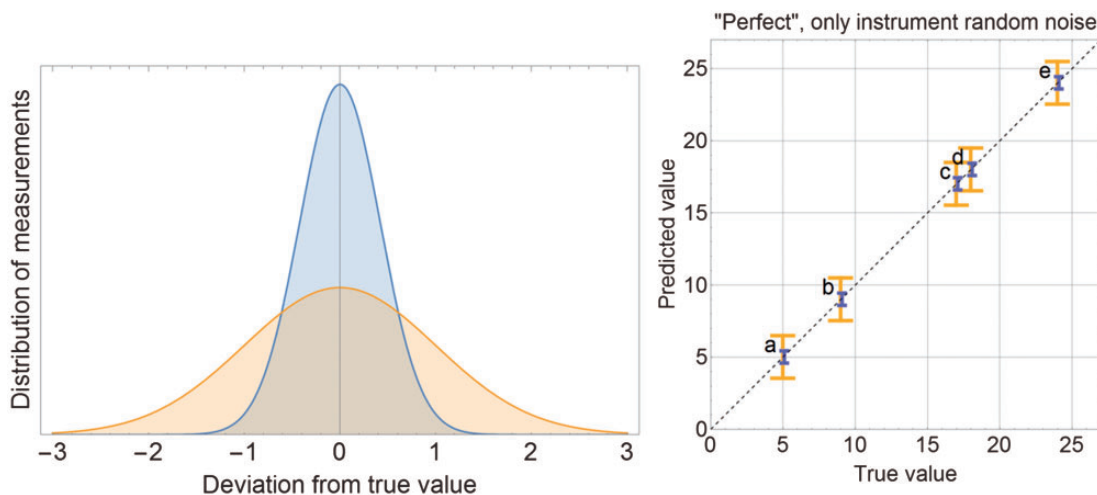**Figure 4.** The relationship between accuracy and measurement time for a given measurement solution.

Figure 5. Instrument statistical noise (high = orange, low = blue).

integrations time or data with different levels of SNR, e.g., by adding varying amounts of synthetic noise to the signal.

Sources of Inaccuracy and Prediction

Figure 5 shows the effect of random sources of inaccuracy on prediction. Figure 5a illustrates the distribution of measurements relative to the deviation from the true value for a repeated measurement of the same object. The orange trace represents high sample heterogeneity or high statistical noise and the blue trace represents homogenous samples or low statistical noise. Figure 5b shows the corresponding prediction plot, where the T-bar represents the variation in the estimated value from the true value when a model is applied. The T-bar should be reduced to an acceptable level. Relying on single measurement accuracy in the scenario where
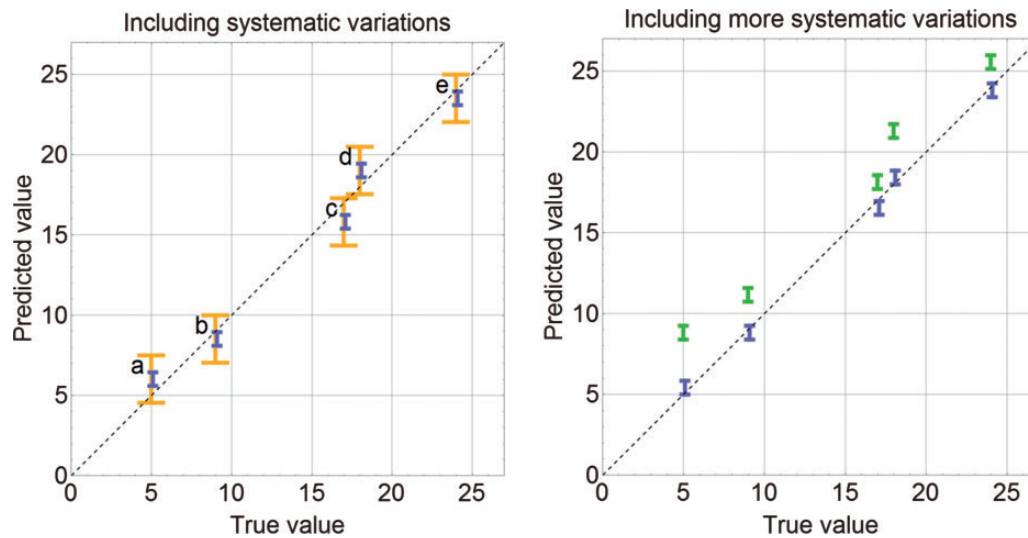
Figure 6. Understanding how sources of systematic variation affect accuracy (undefined units).

random sources of inaccuracy are high will result in a longer T-bar, whereas increasing the integration time or the number of samples averaged will reduce the T-bar.

Figure 6 shows how systematic variations, whether instrument- or application-driven, will affect the prediction model. Figure 6a shows the case where increasing SNR only moderately improves the accuracy when systematic variation is present. This could be that the prediction of, for example, dry matter in a natural object, is systematically dependent on the size of the naturally varying sample object. This error could be that prediction of small sample objects is generally overestimated (Figs. 6a and 6d) and the prediction of big sample objects is underestimated (Figs. 6b, 6c, and 6e).

Figure 6b shows the case where good SNR does not improve the accuracy when systematic variation is present. A simple example of this is a temperature-induced drift that is not corrected for. Unless a temperature calibration is included in the solution, good SNR will not be enough to maintain accuracy.

Measurements in Realistic Conditions

The prototype for testing in the real-world scenario (Fig. 2, step 5) should be based on the performance analysis that was completed on data collected from the controlled measurements. The design team, including end user–application representatives, needs to:

(a) Identify if any of the sources of inaccuracy result in measurement errors that are critical to the required prediction accuracy (Fig. 2, step 3).
(b) Revisit the application requirements in the context of installing the prototype in a realistic measurement

environment, to ensure that everyone in the design team has the same understanding of the inline installation challenges (Fig. 2, step 4).

The prototype may require only slight modifications of the lab setup or it may be a more advanced solution, depending on the demands of the measurement conditions and the duration of the tests. The ideal prototype for real-world testing should prioritize accurate data collection over measurement speed, e.g., by increasing illumination, increasing integration time, using frequent dark correction, averaging several measurements etc. While increasing the speed of the measurement can and will be addressed in the final solution, it is still necessary to have a plan in place at this stage for how this will be addressed.

> The ultimate aim of this prototype is to demonstrate the inline measurement principle and to reveal unforeseen challenges that the real-world conditions introduce.

Unforeseen challenges are unfortunately a part of the design process. If the prototype used for the realistic measurements has effectively minimized foreseen sources of inaccuracy, it is much simpler to identify those that were unforeseen. Examples of pitfalls when going from controlled measurements to an inline prototype demonstration are:

- The ambient light in the measurement location is too strong, has spectral features or is modulated in some way, influencing the quality of the signal.
- Other processes in the measurement location interfere with the instrument, e.g., cleaning of the process line each night requires that the prototype needs to be encased in a sealed housing (IP65).

- The system is not stable enough over time when measuring continuously in the measurement location, e.g., ambient temperature variations.
- The measurement environment contaminates the optical surfaces e.g., dust/dirt on windows/lenses or moisture build-up.
- The measurement distance varies more than anticipated and is not covered by the prototype's depth of field.
- Unforeseen sample variation (natural or process induced) introduces an error in the prediction.
- The measurement environment introduces stray light, e.g., unwanted reflections from stainless steel surfaces or brightly colored conveyor belts.

The move from controlled to realistic measurements can be put in the context of the performance plot (as seen previously in Fig. 4). An example case is illustrated in Fig. 7, where the solid blue trace represents a controlled measurement; it is more accurate due to the very controlled environment and sample presentation. The dashed blue trace represents the performance of the prototype solution with the same core components but adapted to measure in realistic conditions; it drops in accuracy due to the presence of more sources of inaccuracies. If, after completing the trials, it is found that the accuracy is sufficient, but the speed needs to be increased, the green trace represents a potential increase in measurement speed if the system sensitivity is improved. However, if it is found that the target accuracy in not reached, improved SNR will not

help, and it is necessary to identify what other sources of inaccuracy are dominating, before going further. This may involve conducting further measurements in controlled and realistic environments.

The performance analysis on the data collected should also address robustness and stability over time.

The desired result after realistic trials is that the target accuracy is reached and robust over time. It is acceptable if the measurement speed is slower than the target online speed, since this can be addressed with improved SNR in the final design (Fig. 2, step 6).

Progressing to a Commercial Solution

If it is found that the final design requires improvements e.g., higher SNR, it is important to evaluate if better off-the-shelf instrumentation is available, or if customized components are needed to meet the end-user requirement.

If existing spectrometers/cameras cannot measure fast enough for the application, a spectrometer with optimized range, resolution, and detector material can give significant sensitivity and efficiency improvement and can therefore measure at higher speeds. Customizing a spectrometer can be well worth the initial non-recurring engineering costs and should not be dismissed if the cost–benefit analysis is promising.

If a high repetition rate of reference measurements is required to increase the robustness of the measurement, this will need to be included in the optical and/or
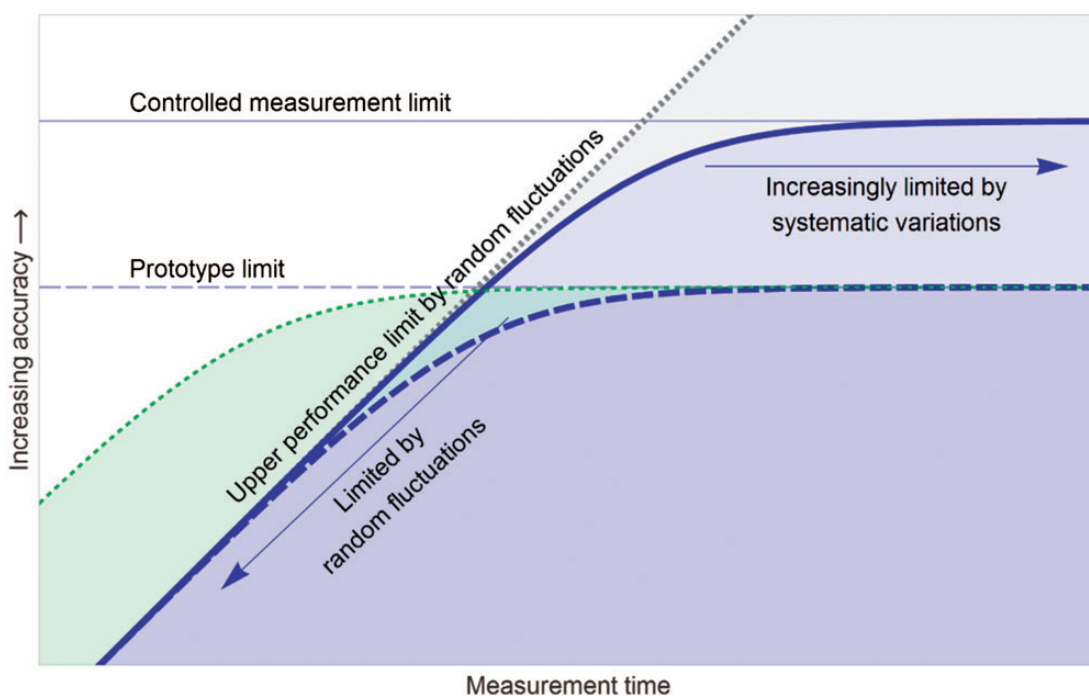

Figure 7. Performance plot of three different measurement solutions.

mechanical design. An example of this is if the instrumentation requires a stable temperature to perform well but the application is such that the measurements need to be performed in both warm and cold environments. This can also be the case for an environment with varying humidity. GasSecure is an example of a measurement that requires a reference measurement for each signal measurement. In this case, the reference measurement is included in the optical design using filter functions.[46]

The cost of the available off-the-shelf spectrometers–cameras can be too high for some applications, especially for high-volume cases. Dedicated, simplified spectrometers can often be lower in cost in volume. In some cases, the use of filter–detector or LED–detector combinations give sufficient spectral information and can even be preferred, e.g., due to the reduced power consumption.

The footprint of the instrument is also a driver for customization. For example, if several spectral ranges need to be covered, this could require several general-purpose spectrometers with different detector materials, making a very cumbersome and expensive instrument. Designing a customized spectrometer can allow combining different spectra ranges.

The sampling method or sample presentation can be critical in reducing random and systematic variations in the measurement. Mechanical presentation combined with an optimized design of the optical geometry using customized lenses, can be just as important as which light source and detectors are used.

As mentioned previously, there are several manufacturers of off-the-shelf spectrometers–cameras (industrial, high-end, consumer) with the potential for different application configurations (e.g., handheld, inline, online, harsh environment, etc.). However, even if an available spectrometer reaches the accuracy and price requirements, it is still necessary to determine how much additional hardware design is required to complete the solution. This usually includes, but is not limited to:

- **Illumination design**: i.e., the size, shape, evenness, and intensity of light spot or field-of-illumination. Illumination solutions include optical fiber sources, LEDs, glow bars, light bulbs, etc.
- **Optical components and geometry**: For example, selecting lenses for the required size and shape of the field-of-view and the relative position of the field-of-illumination to the field-of-view. Filters may be needed to suppress part of the spectral range (especially for Raman or fluorescence measurements).
- **Collection optics**: i.e., the optical design for collecting light from the sample into the spectrometer. Optical fibers, for example, have advantages over free space optics when it comes to light coupling, but they come at a cost to the SNR and have a limited field-of-view.

- **Sample presentation**: i.e., the mechanical design for sample presentation or sample access. The end user–application team must be involved here to give input.

It is worth noting that some commercial vendors of general-purpose spectrometers deliver customized versions of their core product and support adapting the spectrometer to the application, based on the required spectral response and resolution. Some vendors provide complete system integration and system development and can add software interfaces adapted for lab, field, and quality control use.[47]

If the final design reaches the performance and price requirements, at the expected production volume, it is very important to plan for the remaining work in instrumentation standardization, software development and integration, supplier reliability, system-to-system variation analysis, calibration model transfer, and adaption to different process lines. This additional work is beyond the scope of this article.

Conclusion

This paper approaches online spectroscopy from an instrumentation perspective and aims to aid researchers, scientists and engineers reach the goal of truly functional inline spectroscopy. It is very common to underestimate the effort and time required to take applied spectroscopy from a proven concept to a functioning inline solution. This article provides a tool for tracking the development progress and identifying sources of inaccuracy in the solution and can be summarized as follows:

- Build a multidisciplinary design team.
- Know your spectral data!
- Gain as much understanding of the application as possible, including restrictions on sample presentation.
- Use first principles: if you do not understand how the data behave in a controlled measurement, you will never understand it in an inline environment.
- Use a structured performance analysis methodology that enables identifying and categorizing sources of inaccuracy at an instrument level and at an application level.
- Be aware of the relationship between measurement speed and accuracy, i.e., the performance of the solution.
- Know when to use off-the-shelf components and when to customize. Do not assume choosing off-the-shelf is the simplest route in the long term.

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References

1. Foss Analytics. "WineScan". https://www.fossanalytics.com/en/products/winescan [accessed Jun 21 2018].

2. Bruker Corporation. "Alpha II FTIR". https://www.bruker.com/prod-ucts/infrared-near-infrared-and-raman-spectroscopy/ft-ir-routine-spectrometers/alpha/overview.html [accessed Jun 21 2018].

3. Metrohm. "NIRS XDS Process Analyzer". https://www.metrohm.com/en-gb/products-overview/process-analyzers/applikon-nirs-xds/ [accessed Jun 21 2018].

4. Tomra Systems ASA. "Autosort". https://www.tomra.com/en/sorting/recycling/products/autosort/ [accessed Jun 21 2018].

5. Foss Analytics. "MilkoStream FT". https://www.fossanalytics.com/en/products/milkostream-ft [accessed Jun 21 2018].

6. Steinert GmbH. "Near-Infrared Sorting". http://www.steinertglobal.com/de/en/products/unisort/ [accessed Jun 21 2018].

7. Foss Analytics. "ProFoss Dairy". https://www.fossanalytics.com/en/products/profoss-dairy [accessed Jun 21 2018].

8. Bruker Corporation. "TANDEM On-Line Tablet Testing". https://www.bruker.com/products/infrared-near-infrared-and-raman-spectro-scopy/ft-nir/tandem/overview.html [accessed Jun 21 2018].

9. Drägerwerk AG and Co. KGaA. "GS01 GasSecure—Fixed Wireless Gas Detection". https://www.draeger.com/en-us_us/Oil-Gas-Industry/GasSecure-GS01-Gas-Detection#GS01-Wireless-Gas-Detector [accessed Jun 21 2018].

10. A.S. Gilbert, R.W. Lancaster. "IR and Raman Spectroscopy. Industrial Applications". In: J.C. Lindon, G.E. Tranter, D.W. Koppenaal, editors. Encyclopedia of Spectroscopy and Spectrometry, 3rd ed. Oxford, UK: Academic Press, 2017. pp.394–407.

11. R. Noll, C. Fricke-Begemann, M. Brunk, S. Connemann, et al. "Laser-Induced Breakdown Spectroscopy Expands Into Industrial Applications". Spectrochim. Acta, Part B. 2014. 93: 41–51.

12. M. Boiret, F. Chauchard. "Use of Near-Infrared Spectroscopy and Multipoint Measurements for Quality Control of Pharmaceutical Drug Products". Anal. Bioanal. Chem. 2017. 409(3): 683–691.

13. M. Anik Alam, Z Shi, J.K. Drennen, C.A. Anderson. "In-Line Monitoring and Optimization of Powder Flow in a Simulated Continuous Process Using Transmission Near Infrared Spectroscopy". Int. J. Pharm. 2017. 526(1–2): 199–208.

14. S. Laske, A. Paudel, O. Scheibelhofer. "A Review of PAT Strategies in Secondary Solid Oral Dosage Manufacturing of Small Molecules". J. Pharm. Sci. 2017. 106(3): 667–712.

15. K. Knop, P. Kleinebudde. "PAT-tools for Process Control in Pharmaceutical Film Coating Applications". Int. J. Pharm. 2013. 457(2): 527–536.

16. S. Grassi, C. Alamprese. "Advances in NIR Spectroscopy Applied to Process Analytical Technology in Food Industries". Curr. Opin. Food Sci. 2018. 22: 17–21.

17. M.T. Munir, W. Yu, B.R. Young, D.I. Wilson. "The Current Status of Process Analytical Technologies in the Dairy Industry". Trends Food Sci. Technol. 2015. 43(2): 205–218.

18. I. Måge, J.P. Wold, F. Bjerke, V. Segtnan. "On-Line Sorting of Meat Trimmings into Targeted Fat Categories". J. Food. Eng. 2013. 115(3): 306–313.

19. M. O'Farrell, J.P. Wold, M. Høy, J. Tschudi, et al. "On-Line Fat Content Classification of Inhomogeneous Pork Trimmings Using Multispectral Near Infrared Interactance Imaging". J. Near Infrared Spectrosc. 2010. 18(2): 135–145.

20. L. Salguero-Chaparro, V. Baeten, J.A. Fernández-Pierna, F. Peña-Rodríguez. "Near Infrared Spectroscopy (NIRS) for On-Line Determination of Quality Parameters in Intact Olives". Food Chem. 2013. 139(1–4): 1121–1126.

21. S.P. Gundupalli, S. Hait, A. Thakur. "A Review on Automated Sorting of Source-Separated Municipal Solid Waste for Recycling". Waste Manage. 2017. 60: 56–74.

22. M. Verheyen, W. Beckers, E. Claesen, G. Moonen, et al. "Vision-Based Sorting of Medium Density Fibreboard and Grade A Wood Waste". In: Proceedings of the 2016 IEEE 21st International Conference on Emerging Technologies and Factory Automation (ETFA). Berlin, Germany: September 6–7, 2016. DOI: 10.1109/ETFA.2016.7733546.

23. E.M. Carstea, J. Bridgeman, A. Baker, D.M. Reynolds. "Fluorescence Spectroscopy for Wastewater Monitoring: A Review". Water Res. 2016. 95: 205–219.

24. C. Dietzsch, O. Spadiut, C. Herwig. "On-Line Multiple Component Analysis for Efficient Quantitative Bioprocess Development". J. Biotechnol. 2013. 163(4): 362–370.

25. R. Noll, V. Sturm, Ü. Aydin, D. Eilers, et al. "Laser-Induced Breakdown Spectroscopy—From Research to Industry, New Frontiers for Process Control". Spectrochim. Acta, Part B. 2008. 63(10): 1159–1166.

26. V. Sturm, R. Fleige, M. de Kanter, R. Leitner, et al. "Laser-Induced Breakdown Spectroscopy for 24/7 Automatic Liquid Slag Analysis at a Steel Works". Anal. Chem. 2014. 86(19): 9687–9692.

27. V. Sturm, C. Meinhardt, R. Fleige, C. Fricke-Begemann, et al. "Fast Identification of Steel Bloom Composition at a Rolling Mill by Laser-Induced Breakdown Spectroscopy". Spectrochim. Acta, Part B. 2017. 136: 66–72.

28. A. Weisberg, R.E. Lakis, M.F. Simpson, L. Horowitz, et al. "Measuring Lanthanide Concentrations in Molten Salt Using Laser-Induced Breakdown Spectroscopy (LIBS)". Appl. Spectrosc. 2014. 68(9): 937–948.

29. Hamamatsu Photonics K.K. "S11510 Series: IR-Enhanced CCD Image Sensors". http://www.hamamatsu.com/resources/pdf/ssd/s11510_series_kmpd1126e.pdf [accessed Jun 21 2018].

30. Hamamatsu Photonics K.K. "G10899 Series: InGaAs PIN Photodiodes". https://www.hamamatsu.com/resources/pdf/ssd/g10899_series_kird1109e.pdf [accessed Jun 21 2018].

31. Ocean Optics, Inc. "New Ocean Optics OEM Brochure". April 2015. https://oceanoptics.com/ocean-optics-oem-brochure/ [accessed Jun 21 2018].

32. Avantes. "Versatile OEM Solutions: Creating Results that Perfectly Align with Your Needs". https://www.avantes.com/support/documen-tation/brochures-miscellaneous/233-oem-brochure/file [accessed Jun 21 2018].

33. Spectra Engines Oy. "NIROne Sensors". https://www.spectralengines.com/products/modules/ [accessed Jun 21 2018].

34. Excelitas Technologies. "ACULED Design Your Own Guide". http://www.excelitas.com/downloads/DTS_ACULEDDYO.pdf [accessed Jun 21 2018].

35. M. O'Farrell, K.A. Hestnes Bakke, J. Tschudi, J. Petter Wold. "Near-Infrared (NIR) Interactance System for Non-Contact Monitoring of the Temperature Profile of Baked Liver Pâté". J. Appl. Spectrosc. 2011. 65(12): 1372–1379.

36. T. Naes, T. Isakson, T. Fearn, T. Davies. "A User-Friendly Guide to Multivariate Calibration and Classification". Chichester, UK: NIR Publications, 2002.

37. D.A. Wahab, A. Hussain, E. Scavino, M.M. Mustafa, et al. "Development of a Prototype Automated Sorting System for Plastic Recycling". Am. J. Appl. Sci. 2006. 3(7): 1924–1928.

38. A.U. Vanarase, M. Alcala, J.I.J. Rozo, F.J. Muzzio, et al. "Real-Time Monitoring of Drug Concentration in a Continuous Powder Mixing Process Using NIR Spectroscopy". Chem. Eng. Sci. 2010. 65(21): 5728–5733.

39. H.W. Ward, D.O. Blackwood, M. Polizzi, H. Clarke. "Monitoring Blend Potency in a Tablet Press Feed Frame Using Near Infrared Spectroscopy". J. Pharm. Biomed. Anal. 2013. 80: 18–23.

40. H. Zhu, M. O'Farrell, G. Bouquet, K. Lunde, et al. "Evaluating Nuclear Magnetic Resonance (NMR) as a Robust Reference Method for Online Spectroscopic Measurement of Water Holding Capacity (WHC)". J. Food. Eng. 2016. 175: 51–57.

41. J.W. Bo Stenberg, R.A. Viscarra Rossel, A. Mounem Mouazen. "Visible and Near Infrared Spectroscopy in Soil Science". Adv. Agron. 107: 163–215.

42. S.J. Parikh, K.W. Goyne, A.J. Margenot, F.N.D. Mukome, et al. "Soil Chemical Insights Provided Through Vibrational Spectroscopy". Adv. Agron. 126: 1–148.

43. G.D. Gillespie, C.D. Everard, K.P. McDonnell. "Prediction of Biomass Pellet Quality Indices Using Near Infrared Spectroscopy". Energy. 2015. 80: 582–588.

44. M.T. Munir, D.I. Wilson, N. Depree, I. Boiarkina, et al. "Real-Time Product Release and Process Control Challenges in the Dairy Milk Powder Industry". Curr. Opin. Food Sci. 2017. 17: 25–29.

45. U. Böcker, S.G. Wubshet, D. Lindberg, N.K. Afseth. "Fourier-Transform Infrared Spectroscopy for Characterization of Protein Chain Reductions in Enzymatic Reactions". Analyst. 2017. 142(15): 2812–2818.

46. Drägerwerk AG and Co. KGaA. "GS01 Wireless Gas Detector: Reliable Infrared Gas Detection with No Need for Field Calibration". https://www.gassecure.com/novus/upload/article/GasSecure_IR_gas_detection_paper_R_01_Web.pdf [accessed Jun 21 2018].

47. Kaiser Optics. "Raman Spectroscopy: Applications/Solutions". http://www.kosi.com/na_en/applications/raman-applications-solutions-by-industry.php [accessed Jun 21 2018].