Simplifying quality control using Near-Infrared Spectroscopy

Alexander Kadenkin

Quality control is impacted by multiple challenges, which can have an influence on the functioning of the QC lab. The present White Paper provides approaches, how to simplify the daily quality control using near-infrared spectroscopy combined with a dedicated smart software like Vision Air.

Metrohm International Headquarters; Herisau, Switzerland

Metrohm

Challenges in the daily quality control

Quality control (QC) is indispensable for maintaining a successful long-term customer relationship. Delivery of a product, which does not meet the specifications can damage the relationship with the customer, and have a negative business impact.

However, the operation of a QC lab poses some challenges to a business. Such labs often face high workloads and the company-internal demand for cost savings. Typically, the lab utilizes multiple instruments for the various operating procedures. The workflow for the sample analysis can be complex and the QC manager needs to decide, which parameters to analyze and by which procedure. This is often done under time pressure constraints and can lead to oversights. Running costs for the QC lab are often high, because some techniques require significant expenses for chemicals, sample preparation, and instrument maintenance.

On the other hand, the situation can be significantly improved by using Near-Infrared Spectroscopy (NIRS) as a quality control tool. This technique provides multiple quality parameters simultaneously without sample preparation or the use of hazardous chemicals. This can increase sample throughput, streamline quality control, and reduce running costs and workload of the QC lab. Further simplification can be reached through the use of dedicated smart NIRS software, which can assist the lab manager in decision-making. Some cases in which NIRS can be used to simplify quality control are presented in this white paper.

All-in-one solution

Modern products are often complex or dedicated solutions, which have to meet multiple customers' requirements. As a result, it is often insufficient to verify only one quality parameter. Consequently, the manufacturer has to determine multiple quality parameters, which increases the challenge for the QC lab. For example, five parameters need to be determined during quality control of polyols (**Figure 1**). Analysis of these five parameters requires five instruments with five operating procedures (OP), each of which can take up to one hour. In addition, some of these quality control procedures require sample preparation, the use of chemicals, and skilled and trained lab technicians for execution.

On the other hand, the lab manager of such a QC lab can avoid this complexity by using an NIR analyzer. One NIR analyzer can determine multiple quality parameters in a single operating procedure without any sample preparation or chemical waste within a minute. An operator can be trained to use the system within five minutes. This can significantly simplify the workflow and reduce dramatically the costs of the daily quality control.

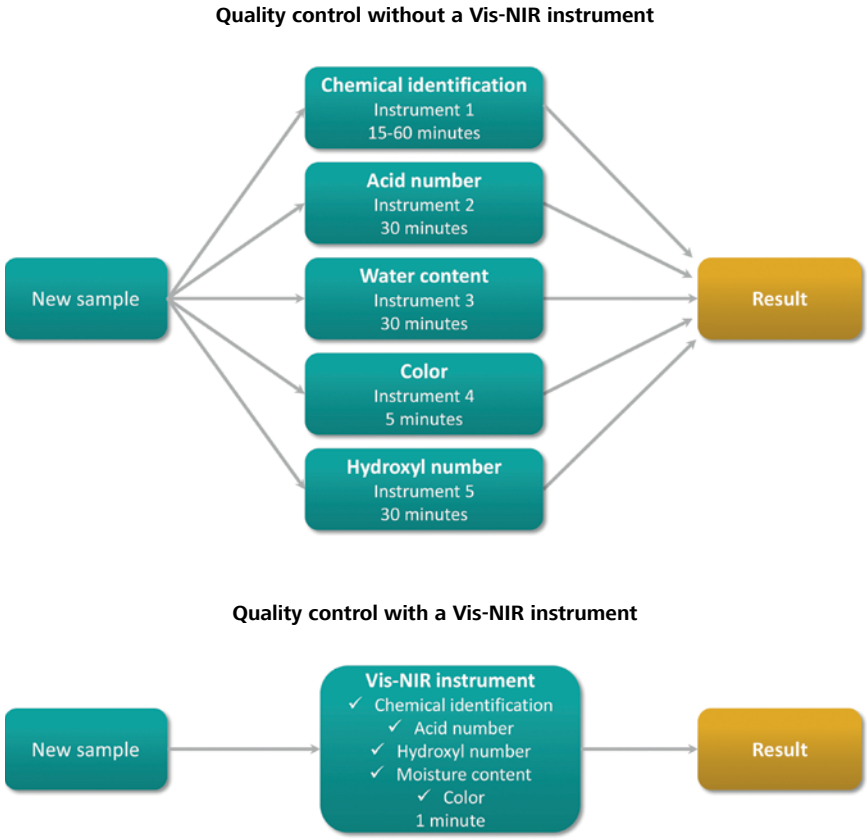

Figure 1. Quality control of polyols with and without Vis-NIR.

Fast screening and verification

The capability to measure multiple quality parameters simultaneously enables NIRS to be used as a fast screening tool in quality control (**Figure 2**). Instead of measuring multiple quality parameters for each sample using time-consuming and cost-intensive wet chemical methods, the lab manager can immediately obtain multiple quality parameters within a minute with NIRS. If the results are inside specifications, the product can be released or the operator can continue with post-processing. Otherwise, the process can be interrupted and the reason for deviation can be investigated.

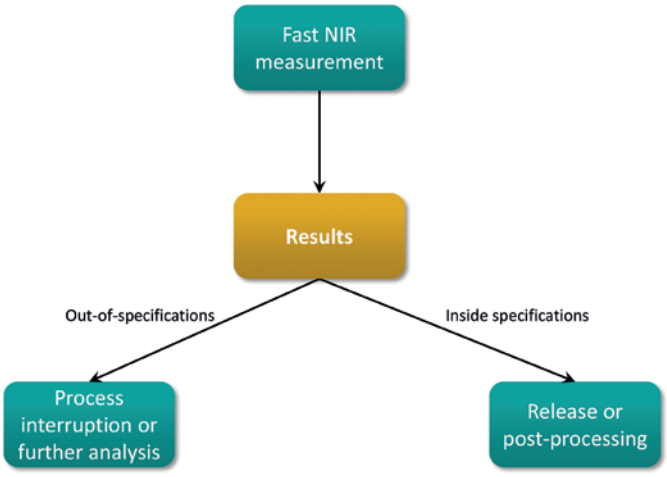

Figure 2. Screening using NIRS.

NIRS can be the solution in various situations:

Atline quality control

The complexity and duration of the sample analysis limits the determination of the product quality in the production process. Because of the long analysis time, the operator avoids the determination of key quality indicators in the production and runs the plant based on empirical data. Sometimes the production is finished before the product reaches the target specification. Such out-of-spec products can cause significant monetary losses. In contrast, the operator can immediately obtain multiple quality parameters atline or online, when using NIRS. This information can be used identify the endpoint of the reaction and improve the production process.

Mobile screening

Counterfeits or misbranded products occur in various industries. For example, the value of the annual earnings from substandard and counterfeit drugs was estimated to be over $32 billion in 2003 **[1]**. Local low quality lubricating oil is sometimes mixed with water and filled in bottles of a premium-branded high quality product. The operator of a gas station could potentially fill low quality gasoline in the petrol tank of a high quality product. One of the problems in the identification of such counterfeits is the complexity of the analysis. A governmental body or manufacturer performs random sampling at the point-of-sales and sends these samples to a QC lab for inspection and determination of multiple quality parameters. This procedure is costly and inefficient for fast and large scale anticounterfeit detection. The use of NIRS as a mobile screening tool can simplify the process and improve the fight against crime. When operated in a van, the analyzer can immediately identify whether, e.g., the octane rating of the gasoline sample is correct or, for tablets, whether it is an authentic sample or a counterfeit. In case of inconclusive or doubtful results, the samples can be sent to the QC lab for further investigations. This has the advantage that the control body only focuses on problematic samples instead of analyzing all samples. This reduces the workload of the QC lab significantly and improves the efficiency of counterfeit detection.

Smart selection of the operating procedure

When a new sample arrives, the lab manager needs to decide which parameters need to be analyzed via which operating procedure and assign the task to a lab technician. Such a workflow is a potential source for errors. It is not unusual that one selects or uses a wrong operating procedure or forgets to measure an important quality parameter. Such mistakes are usually identified at the end of the quality control. Often this means that the whole work for this sample needs to be repeated, which costs time and money.

However, it is possible to simplify this workflow and at the same time eliminate this source of error by using a single instrument for qualitative and quantitative information combined with dedicated software. With Vision Air software for Metrohm NIR analyzers, the manager can merge all operating procedures into a single smart operating procedure. Such an all-in-one operating procedure is shown in **Figure 3**, exemplifying the quality control of creams in the personal care industry. The company produces various creams, some of them with and others without sunscreen substances. For the quality control of the last product category, only moisture and additive content need to be determined. Creams with sunscreen substances are different in their physical and chemical properties and require therefore another QC procedure. For those, it is also necessary to determine an additional quality parameter: the sun protection factor (SPF).

Instead of using multiple instruments with different operating procedures for both samples, the operator can acquire a single NIR spectrum. In the next step, the software uses this spectrum and does the same as we humans do, when we decide: the spectrum moves through a decision tree. In a qualitative

analysis, the software identifies the sample. If it is a product with SPF, then the software automatically selects quantitative methods for creams with sun protection. Otherwise, it uses methods for creams without SPF. The operator can even input target quality specifications for each product. The software will verify if the product meets the quality specifications. This means that the smart software simplifies the work of the lab manager. It identifies a sample, selects the correct OP for this sample type, performs analysis of all relevant parameters, and verifies the specifications. When the result is inside the specification, the operator only needs to review the report and release the product. The complete process spanning sample preparation until the review of the results takes less than five minutes, which is much faster than the classical way.

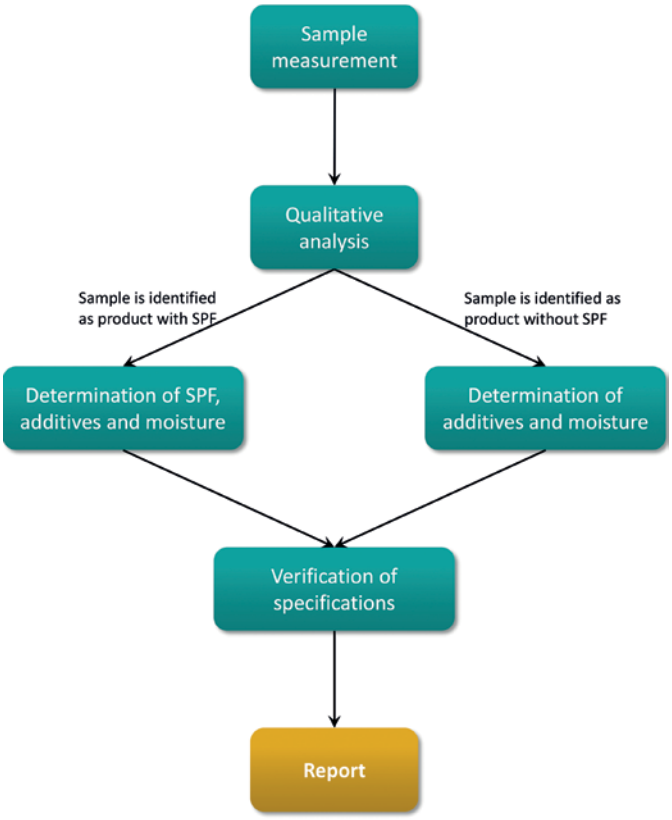

Figure 3. Smart operating procedure for quality control of multiple products enabled by Vision Air software.

Measurements with the best accuracy

Quite often companies produce similar products but in varying quality with, for example, different concentration or chemical indices. This means that the range of such quality parameters can be wide. In this case, it is important to keep in mind that the error of the measurement depends on the reference value. For example, as mentioned in various norms such as ASTM D664 «Acid Number of Petroleum Products by Potentiometric Titration», repeatability and reproducibility depend on the concentration **[2]**. The error observed at high concentration is high and vice versa.

This limits the accuracy of the calibration for the whole range, because the calibration error is strongly affected by high errors observed at high concentration values. The normal way to solve this problem is to split the calibration and develop a separate calibration for each of the ranges: low, medium, and high. For example, the error of calibration for the determination of the Kappa number in wood pulp over the full range between 0 and 175 is 3.5 (**Table 1**). This is a very low error for pulp samples with high Kappa numbers. On the other hand, for samples in the low range, this error is too high to be acceptable. Splitting of the global calibration leads to a significant improvement of the accuracy, especially in the low range. A dedicated calibration model for Kappa number between 0 and 36 is characterized by a very low error of 0.77.

Range	R^2	SEC
Full, 0–175	0.996	3.5
0–36	0.994	0.77
36–90	0.986	3.4
90–180	0.977	3.6

Table 1. Coefficient of determination (R^2) as well as standard error of calibration (SEC) for the determination of Kappa number in wood pulp.

This improvement of analytical performance results in an increase of complexity, because the global operating procedure was replaced by three dedicated OPs. The way to reduce this complexity is the use of a smart software like Vision Air for Metrohm NIR analyzers. This software enables a combination of multiple prediction models for the same parameter in order to measure samples with best accessible accuracy and decides which quantitative model needs to be used for a particular sample (**Figure 4**). In the first iteration, the software roughly estimates Kappa number using a global calibration with relatively high error. Using these values, the sample is assigned to a class with low, medium, or high values, which were previously defined by the lab manager. Finally, in the second iteration the software uses the dedicated quantitative model for the identified class. Therefore, it provides a result with the best accessible accuracy without the need to switch manually between different OPs.

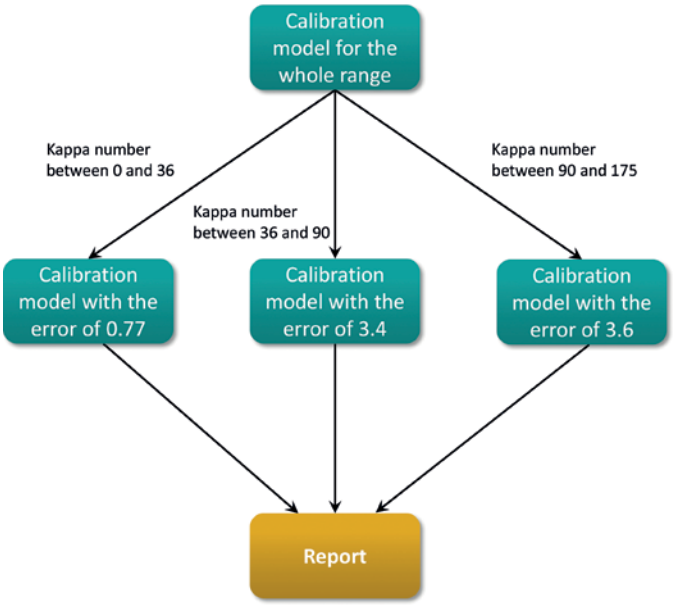

Figure 4. Smart operating procedure for measurements with the best accuracy.

Summary

The present White Paper provides an overview about the benefits of using non-destructive chemical free analysis technology in combination with a dedicated smart software in a daily quality control setting. The implementation of these techniques in the specific quality control process as well as method development can be supported by your local Metrohm representative.

References

[1] WHO, Substandard and counterfeit medicines. 2004.
[2] ASTM D664 - 17a (2017), Standard Test Method for Acid Number of Petroleum Products by Potentiometric Titration, 2017.

WP-037 QC by NIRS, published August, 2018