WHITE PAPER

OMNIS NIRS: An efficiency boost for your laboratory

Routine lab analysis – simplified with OMNIS NIRS

Near-infrared spectroscopy (NIRS) is an analytical technique used to determine multiple parameters in samples quickly without requiring any chemicals. Determinations can be performed in liquids, pastes, and solids with this non-destructive method.

Many industries benefit from using NIRS in their laboratory workflows. Time and cost savings are two of the most convincing arguments for adopting NIRS in the laboratory. It is also easy to use, even for non-technical operators.

This White Paper discusses the concept and benefits of NIR spectroscopy, supplemented with the use of the Metrohm NIR solution, OMNIS NIRS, in several real-life laboratory application examples. Applications from the **petrochemical**, **food and beverage**, **semiconductor**, and **pharmaceutical industries** are used to demonstrate the unique functionalities of OMNIS NIRS in different situations.

Metrohm

NIR SPECTROSCOPY IN BRIEF

Spectroscopy is the analysis of the interaction between light and matter. Light is a form of electromagnetic radiation which can be categorized by its energy or wavelength/wavenumber. The wavelength range between 780 nm and 2500 nm (12820–4000 cm^{-1}) is referred to as «near-infrared» (**Figure 1**).

This spectral region is used in NIR (near-infrared) spectroscopy, which, like infrared (IR) spectroscopy, triggers molecular vibrations through the absorption of electromagnetic radiation.

While both technologies fall into the category of absorption spectroscopy, induce molecular vibrations, and are fast in analysis, easy to perform, and do not require chemicals (see details in chapter «**Main Benefits of Using NIRS**»), there are distinct differences stemming from variations in their energy levels. Three of the differences between NIR and IR spectroscopy are examined in the following section.

COMPARISON OF NIR VS IR SPECTROSCOPY

1. Deeper sample penetration depth

The higher energy of NIR light compared to IR light (**Figure 1**) results in deeper sample penetration. Therefore, NIRS provides users information about the bulk characteristics of a sample and not just the surface material.

2. Lower band intensity, less saturation

The absorption of NIR light (and therefore the band intensity in the spectra) is typically lower compared to IR. This allows for example the analysis of aqueous solutions of up to 15% water with NIRS.

In general, this eliminates the need for any sample preparation prior to the analysis – something which can be required for IR spectroscopy. It further allows the usage of more convenient sample cells with larger optical path lengths compared to IR spectroscopy.

3. Higher accuracy with quantitative measurements

The lower absorption bands with NIRS allow for a better analysis of complex mixtures such as oils. Furthermore, the sensitivity of NIR bands to physical properties (e.g., particle size, density, or viscosity) can be used to determine these parameters as well, which is typically not possible with IR spectroscopy.

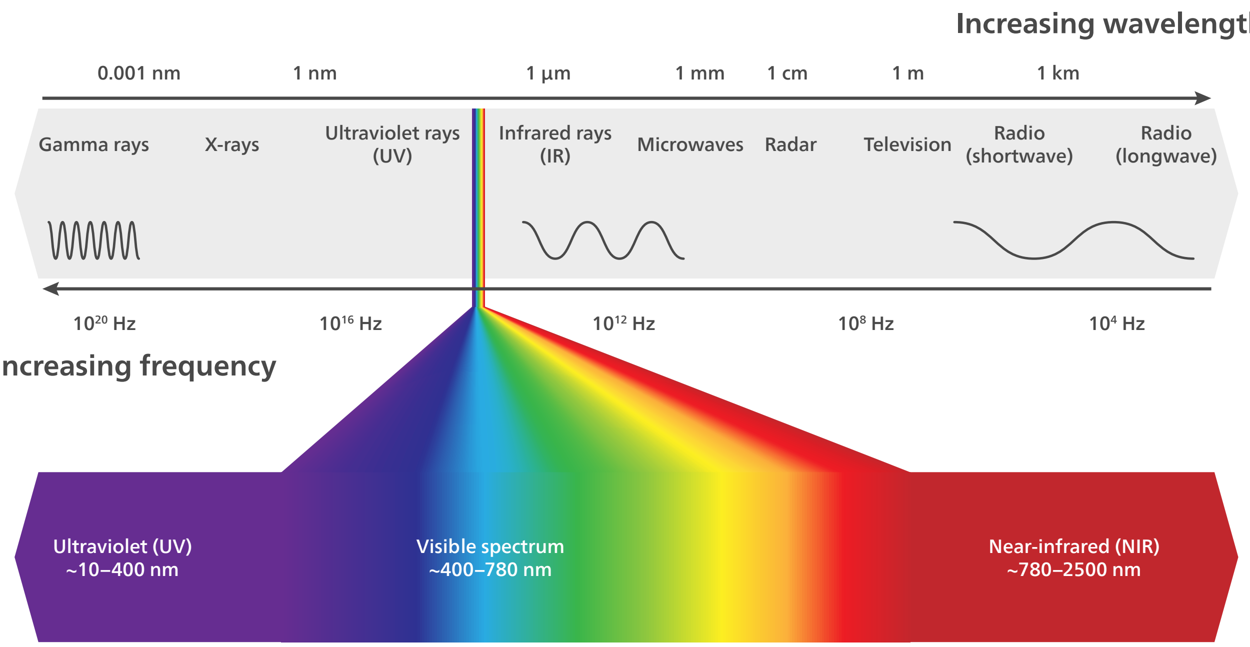


Figure 1. Generalized illustration of the electromagnetic spectrum.

NIRS MEASURING MODES: FACTORS FOR ACCURACY AND REPRODUCIBILITY

NIR spectroscopy can be used to analyze both liquid and solid samples. Each sample type requires a different NIRS measuring mode for the best results.

CONSIDERATIONS FOR LIQUID SAMPLES

Transparent liquid samples are typically measured with the transmission mode as shown in **Figure 2**.

Some important parameters which influence the feasibility and reproducibility of the measurements for liquid samples are the **optical path length** and the **sample temperature**.

1. Optical path length

The optical path length determines the distance in which the light transmits through the sample, and consequently, how much light it absorbs. When confronted with low concentrations or a low absorbance of the parameter of interest, a longer path length is required. For samples with higher concentrations or a higher absorbance of the measured parameter, a shorter path length is more suitable.

The OMNIS NIR Analyzer (**Figure 2a**) accommodates various sample vessels to fulfill this application requirement. Disposable vials, cuvettes, and flow-through cells with diverse optical path lengths can be utilized. Moreover, the OMNIS NIR Analyzer recognizes and identifies the sample holders designed for each of these vessels, minimizing user errors during routine analysis.

2. Sample temperature

As mentioned in **ASTM 8340** (Standard Practice for Performance-Based Qualification of Spectroscopic Analyzer Systems), sample temperature must be carefully controlled by the analyzer system to obtain reproducible results. The guideline points out that: «Sample temperature is critical to vibrational spectroscopic analyzer performance» and that «Changes in temperature can affect the measured spectral intensities» [**1**].

The OMNIS NIR Analyzer offers sophisticated sample heating and cooling options from 25 °C up to 80 °C, with long-term stability of 0.1 °C at a set temperature. In addition to the conventional method of heating a sample holder to a defined temperature and waiting for an equilibrium to establish between the inserted sample vessel and sample holder, the OMNIS NIR Analyzer employs an elaborate algorithm that evaluates the actual sample temperature and initiates the measurement automatically once the defined value is reached. This not only ensures highly reproducible measurements, but also saves time and eliminates user errors.

CONSIDERATIONS FOR SOLID SAMPLES

When analyzing solids with NIR spectroscopy, a different measuring mode is required. In this case, the reflection measuring mode is used for the best results (**Figure 3**).

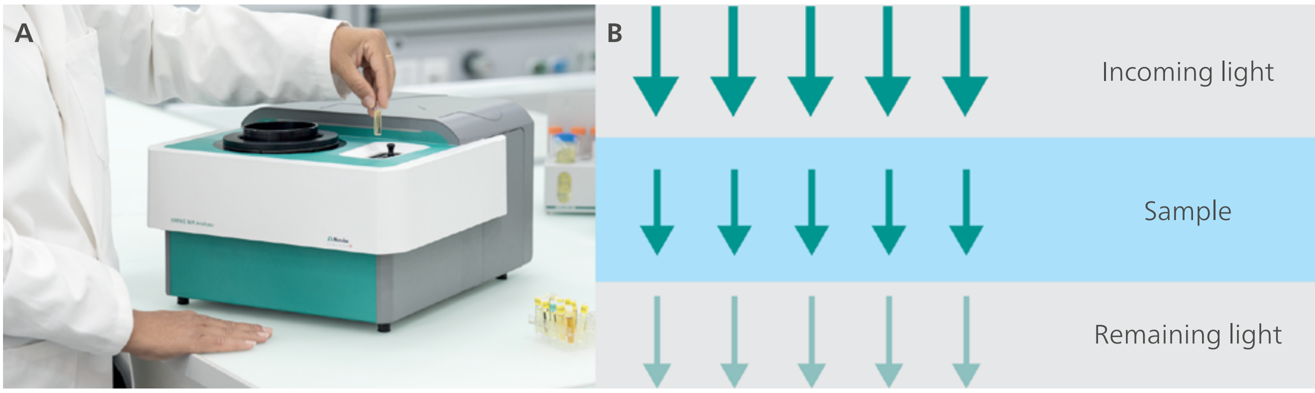


Figure 2. a) Typical transmission measurement performed with a sample in a disposable vial. b) Illustration of the path of light flowing during transmission measurements.

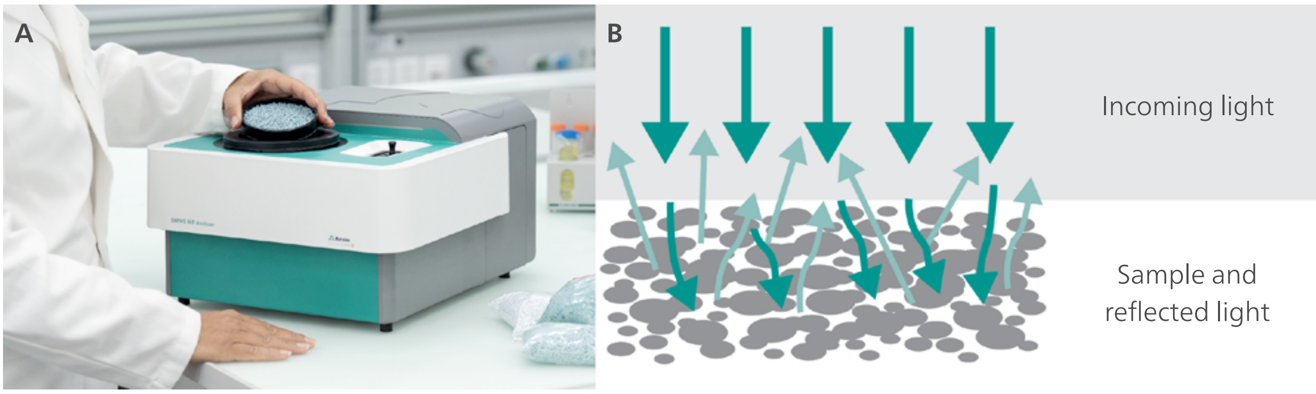


Figure 3. a) Typical reflection measurement performed with a sample in a reusable sample cup. b) Illustration of the path of light flowing during reflection measurements.

In contrast to liquid samples, solid samples often exhibit a higher tendency to be inhomogeneous. This situation can arise from inhomogeneous chemical distribution or physical variations, such as the particle size.

1. Multi-point measurements

To ensure reproducible results despite inhomogeneity, it becomes necessary to analyze the solid sample with a large measurement spot size and/or by analyzing the sample at various locations. Furthermore, the possibility to choose between different sample vessel types to best fit the sample or sample matrix supports users' efforts to produce reliable results.

The OMNIS NIR Analyzer supports different sample vessel types for solids analysis (**Figure 3a**), taking the amount of available sample and its degree of inhomogeneity into consideration. The instrument accommodates small vials (optionally with a lid) suitable for powdered samples, as well as large cups up to 100 mm in diameter. Larger sample vessels such as cups or petri dishes can be measured by the OMNIS NIR Analyzer while rotating automatically (multi-point measurement) to handle larger amounts of inhomogeneous samples.

MAIN BENEFITS OF USING NIRS

CHEMICAL REAGENTS ARE UNNECESSARY

Measurements with near-infrared spectroscopy are based only on the interaction between light and matter. Unlike many other analytical techniques, no chemical reagents are needed during the analysis. As a result, the risk of chemical exposure for operators is eliminated, and no chemical waste needs to be disposed of.

SAMPLE PREPARATION IS NOT REQUIRED

When using NIR spectroscopy, samples are analyzed as-is, regardless of whether they are liquid, viscous, or solid. This eliminates the need for time-consuming sample preparation steps or purchasing consumable items for this purpose.

FAST ANALYSIS SPEED

Measurement of samples with NIRS is fast—spectra are collected in less than one minute. The obtained absorbance spectra include a considerable amount of information. Therefore, measurement with NIRS can be used to determine multiple parameters at once from a single sample.

NIRS APPLICATION EXAMPLES

Near-infrared spectroscopy can be used for myriad applications in many industries.

In the following sections, application examples are given showing how the OMNIS NIR Analyzer is used to boost lab efficiency in the **petrochemical**, **food and beverage**, **semiconductor**, and **pharmaceutical** industries.

– PETROCHEMICAL INDUSTRY: DIESEL ANALYSIS

Sample detection and speed of analysis

NIR spectroscopy is known for its fast analysis speed. Nonetheless, when analyzing large sample batches, the time required to complete the job can still be significant. This application example demonstrates that **even larger sample batches of 20 samples or more can be analyzed in minutes** thanks to the unique features of the OMNIS NIRS solution.

Both hardware and software functionalities must work in synergy to obtain results quickly. From a software point of view, when confronted with many samples, having the option to predefine measurements is considered ideal. Additionally, the ability to start all of the measurements at once, coupled with clear user guidance without the need for further software interactions, is imperative. From a hardware perspective, the device must recognize the insertion and removal of each sample. These functionalities not only enable the operator to concentrate on the measurement process, but also increase sample throughput while minimizing potential user errors.

The OMNIS software from Metrohm meets these requirements with «sample lists» and «series determination» (**Figure 4a**). This is additionally supported by sensors in the OMNIS NIR Analyzer which recognize the sample insertion and removal (**Figure 4b**).

Fast NIRS measurements are not only achieved by using the features already mentioned, but also because the OMNIS NIR Analyzer is built based on a modern 1D InGaAs array technology. This eliminates the need for moving parts within the spectrograph which allows the collection of a spectrum within a few milliseconds (**Figure 5**). With a typical average of 100 spectra per measurement, the OMNIS NIR Analyzer can provide **results in less than 10 seconds†**.

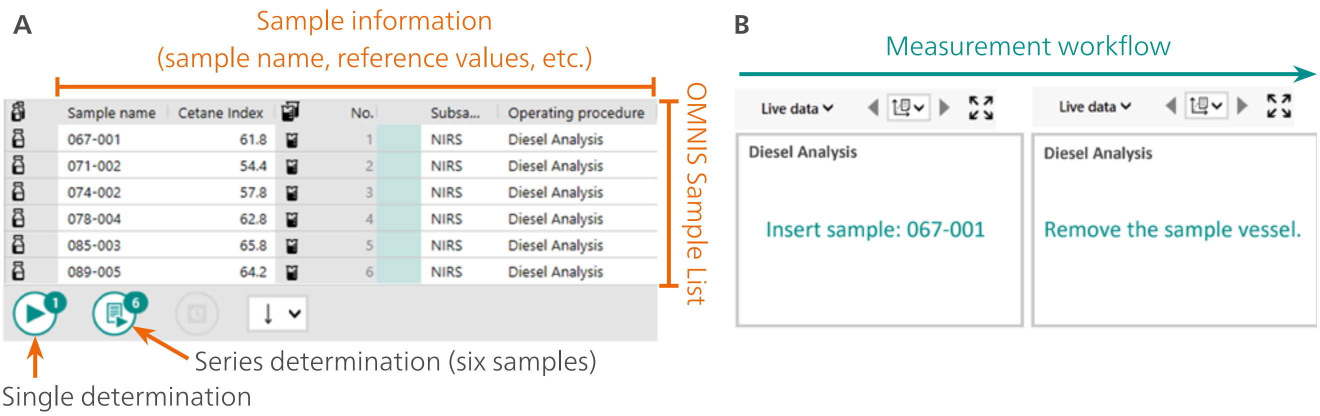


Figure 4. Sample list view in the OMNIS software. All future measurements can be defined in a sample list and started with a single click. Sensors recognize the insertion and removal of the sample.

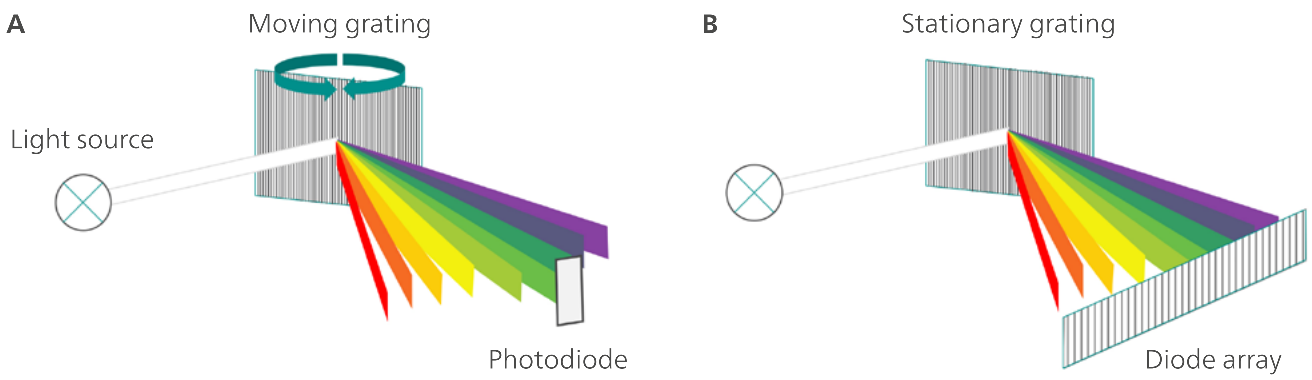


Figure 5. Differences between a photodiode setup vs. diode array setup. a) Photodiode setups require a moving grating to reflect individual light wavelengths to the detector. b) Diode array setups use a stationary grating and all light at individual wavelengths is detected at once.

†For a single-point measurement and without additional heating or cooling of the sample.

As mentioned earlier, it is essential to control the temperature when using NIRS to analyze liquid samples [**1**]. However, the process of heating and cooling samples take time and extends the overall analysis duration.

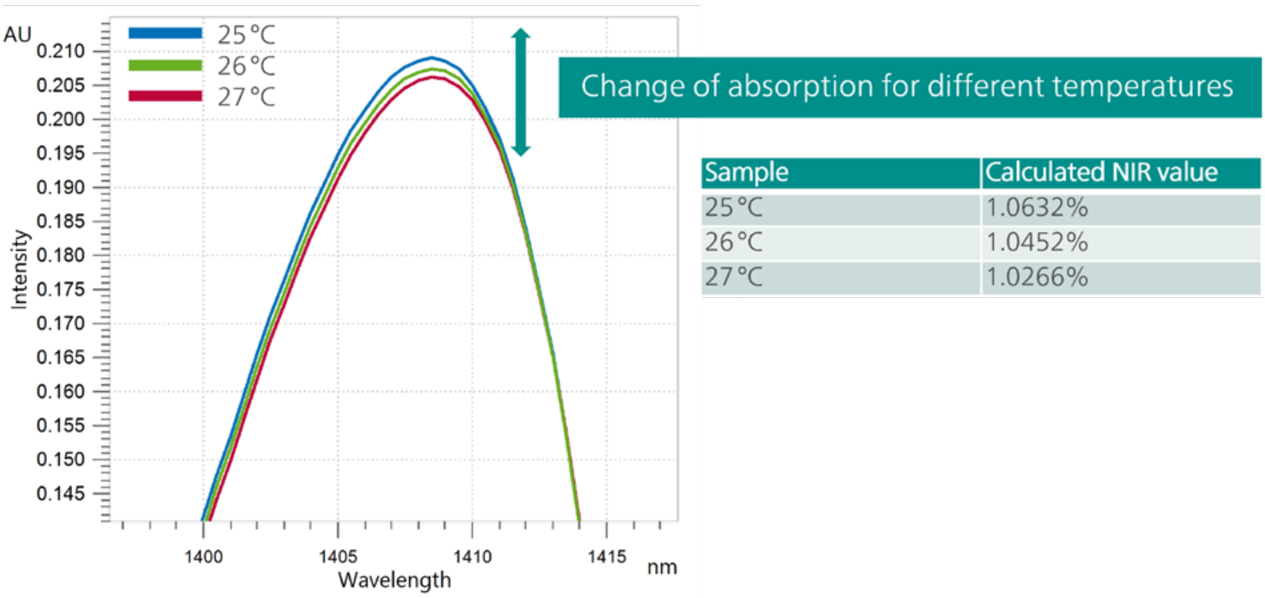
Historically, users have addressed this in two ways. They either accept the delay and wait until the sample reaches the defined temperature in the analyzer (assuming the analyzer provides a reliable heating option), or they preheat the sample first. The latter approach was chosen to leverage the speed advantage of NIR spectroscopy. However, this procedure has the drawback that temperature-related events (sample cooling) may occur during the transfer of the preheated sample to the analyzer. Such temperature variation can introduce spectra changes, causing poor result reproducibility (**Figure 6**).

To overcome this drawback, the OMNIS NIR Analyzer not only measures and regulates the temperature at the sample holder, but also directly at the sample vessel (e.g., vial, cuvette, or flow-through cell). With the use of a sophisticated algorithm, the OMNIS software can evaluate the **direct** sample temperature and initiate the measurement as soon as the target temperature matches the user setting. With preheated samples, temperature control is faster, allowing measurement of large sample batches in a few minutes, with the peace of mind that the crucial parameter of temperature is correct.

The OMNIS functionalities described in this section were used to analyze 50 diesel samples for the parameters of cetane index and cetane number. The analysis of all samples for both parameters took only 19 minutes. Measurement of the same samples under identical conditions took at least 90 minutes with the DS2500 Liquid Analyzer (a Metrohm legacy NIRS instrument).

Samples were poured into 8 mm vials, preheated to 30 °C, and then analyzed with the OMNIS NIR Analyzer using the series determination function and the sample vessel T-control (i.e., temperature control directly at the sample) of OMNIS.

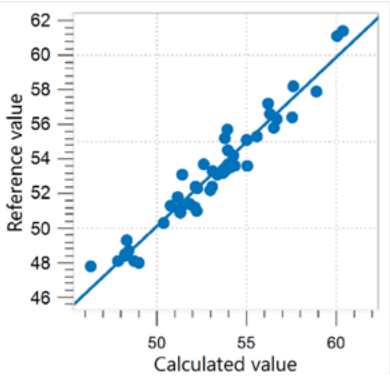
The NIR prediction model was created using the OMNIS Model Developer (OMD) feature which automatically defined all model parameters. The following figures of merit and correlation diagrams (**Figure 7**) were obtained, showing a good correlation between the predicted values by NIRS (labeled «calculated value») and the values estimated according to the ASTM D613 engine test (labeled «reference value»).



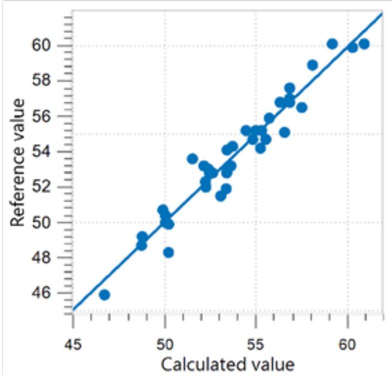
Sample	Calculated NIR value
25 °C	1.0632%
26 °C	1.0452%
27 °C	1.0266%

Figure 6. Effect of small temperature changes on the spectrum. The water band absorbance changes, affecting the NIR prediction results. Such temperature changes can already happen during the transport of a preheated sample prior to the measurement.

Cetane Index in diesel:



Cetane Number in diesel:



Product	Parameter	SEC wt%	SECV wt%	R^2
Diesel	Cetane Index	0.67	0.80	0.940
Diesel	Cetane Number	0.74	0.82	0.937

Figure 7. Correlation diagram for the parameters of Cetane Index and Cetane Number in diesel. The NIRS prediction («calculated value») for both parameters correlate well with the reference values obtained by the ASTM D613 engine test.

– FOOD INDUSTRY: COFFEE ANALYSIS

Multi-point measurement for accurate analysis results of inhomogeneous samples

To obtain reproducible and representative results from an inhomogeneous sample, larger amounts of the sample must be analyzed. This is achieved first by using a large spot size illumination, and second by measuring not only at a single sample spot but in multiple places. While multiple spot size measurements can be done manually, it is more convenient to have an automatic procedure in place.

The OMNIS NIR Analyzer has an implemented rotation option which enables **automatic multiple spot size measurements**. This rotation option is suitable for various sample vessels such as sample cups or petri dishes in different sizes.

In this application example, roasted coffee beans (whole and ground) were analyzed for their caffeine content with the OMNIS NIR Analyzer. The caffeine reference values for the NIR prediction model development were determined by ion chromatography following the ISO 20481 guideline. NIR spectra were obtained using the OMNIS NIR Analyzer rotation option (**Figure 8**) for both sample types. To increase sample throughput, the measurement series was defined upfront, allowing users to initiate the entire batch analysis with just one click. The OMNIS software clearly indicates the end of each individual measurement to the user and informs when to position the next sample.

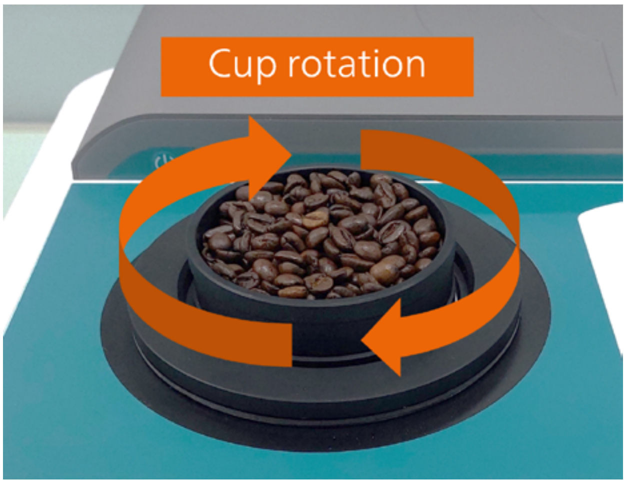
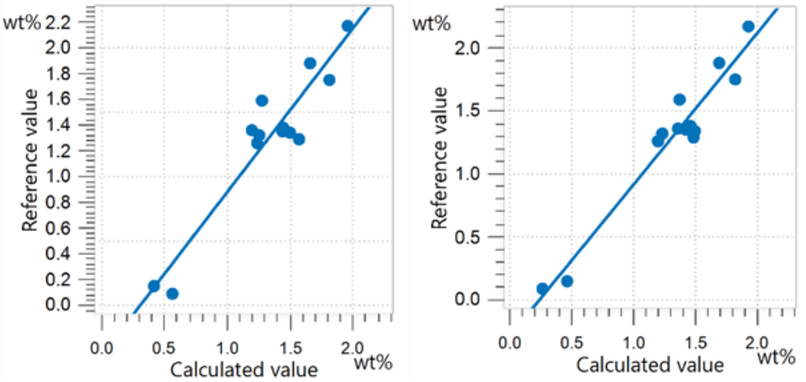


Figure 8. Coffee bean sample in a Large cup OMNIS NIR, 100 mm. The cup rotation option provides reproducible results even for inhomogeneous samples.

Thanks to the rotation option, the quality of the calculated NIR prediction model for caffeine was very similar for both whole and ground coffee beans. The obtained figures of merit and correlation diagrams are given in **Figure 9**.

Caffeine in whole coffee beans: Caffeine in ground coffee beans:



Product	Parameter	SEC wt%	SECV wt%	R^2
Whole	Caffeine	0.07	0.22	0.890
Ground	Caffeine	0.08	0.17	0.942

Figure 9. Correlation diagram of caffeine content in whole and ground roasted coffee beans. The NIRS predictions («calculated value») correlate well with the reference values obtained by ion chromatography.

– SEMICONDUCTOR INDUSTRY: HYDROCHLORIC ACID ANALYSIS

Unattended, safe liquid analysis with an automated NIR system

Laboratories strive to reduce the handling of toxic or corrosive chemicals. One approach is to use automated systems which can both safely conduct measurements with minimal user interaction and increase the lab efficiency by freeing up human resources. However, the implementation of an automatic system is often expensive and requires significant effort. Solutions from a single supplier simplify these challenges and may be available, although single-supplier solutions become harder to find if several analytical technologies must be combined.

This is unfortunate because such technological combinations can add significant value—e.g., when a titrator and a NIR analyzer are integrated into an automated system. Application possibilities include using NIR spectroscopy for fast and chemical-free screening of different parameters, thereby reducing costs and increasing sample throughput, and titration as a complementary method to evaluate additional parameters. Another possibility is to use titration for specific events, for example to validate NIRS results, thereby minimizing reagent consumption while giving higher confidence in the obtained results.

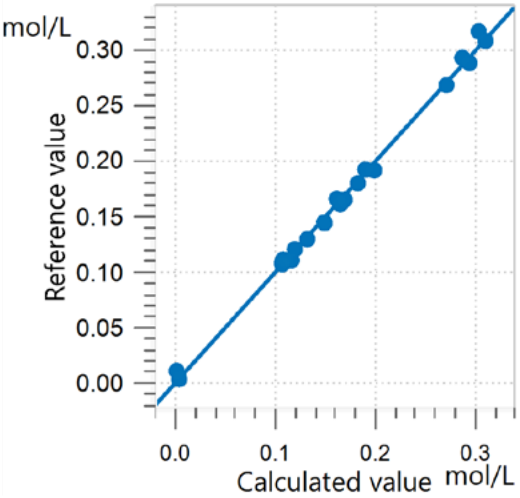
Needless to say, creating such a customized solution with devices from different suppliers, not designed for such an integration, is extremely difficult to realize. With the modular approach of OMNIS, which is designed as an analytical platform, **multiple technologies can be combined easily**.

The following application example demonstrates how OMNIS can be used to individually configure automated setups. In this case, 43 hydrochloric acid solutions were analyzed using an OMNIS NIR Analyzer together with an OMNIS Sample Robot and an OMNIS Titrator (**Figure 10**).

Figure 10. The modular OMNIS platform from Metrohm (shown here with an OMNIS NIR Analyzer, OMNIS Sample Robot, and OMNIS Titrators) combines and controls multiple analytical technologies.

Twenty-two samples were used to create the necessary data to develop a prediction model for hydrochloric acid content. The samples were aspirated from beakers into a NIR flow-through cuvette and then into the titration chamber to obtain **both NIR spectra and titration values** (acid concentration of HCl). A good correlation with low errors was obtained, as shown in **Figure 11**.

Acid concentration in HCl solution:



Product	Parameter	SEC mol/L	SECV mol/L	R^2
HCl solution	Acid concentration	0.0042	0.0051	0.996

Figure 11. Correlation diagram of the acid concentration in a solution of HCl. The NIRS prediction («calculated value») correlates well with the reference values obtained by titration.

For the routine analysis, the OMNIS measurement workflow was programmed in such a way that NIR spectroscopy was chosen to analyze each sample and a titration measurement was performed only if the NIRS results indicated a limit violation. This type of setup has two main advantages:

1. **faster time to result**
2. **less rinsing and waste**

The NIR measurement, including all sample handling activities (e.g., aspiration of sample, rinsing of sample tube), required less than four minutes to complete. When performed in combination with titration, a total of 11 minutes was needed. **Figure 12** displays the OMNIS UI (user interface) which indicates the measurements and the individual results.

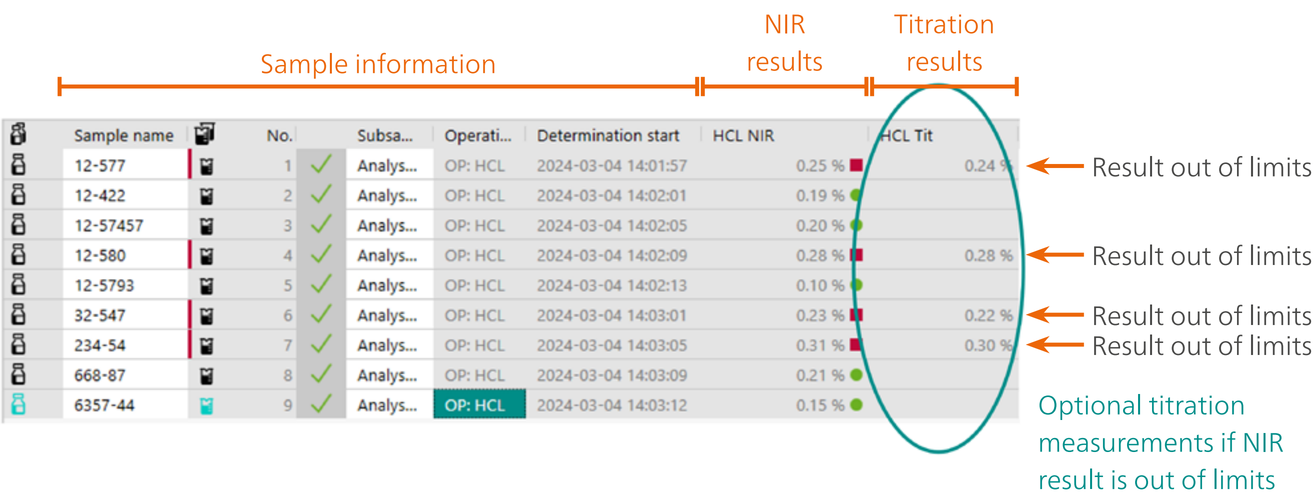


Figure 12. OMNIS sample list for a combined automated setup of NIRS and titration. Titration measurements are only triggered when NIRS results indicate samples to be out of specification. This concept reduces both waste and the analysis time.

– PHARMACEUTICAL INDUSTRY: LYOPHILIZED PRODUCT ANALYSIS

Unattended analysis of solid samples with an automated system

The analysis of lyophilized products is an ideal application for NIR spectroscopy. These products can be expensive and any analysis method causing sample loss is a significant cost driver. Also, once sealed in a vial, it is not very easy to conduct an analysis without a risk of sample contamination. NIRS is an ideal analytical technique to avoid sample contamination because it can perform the analysis directly through glass vials and, thus, also does not consume the sample. Therefore it is suitable to detect the remaining water content and to validate the conformity of lyophilized samples.

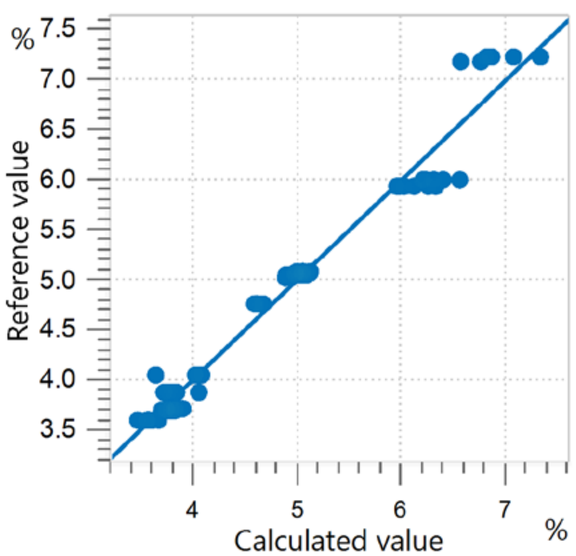
For such applications, the general requirements for a NIR analyzer to produce accurate and reproducible results are often not sufficient since the high number of samples also requires the use of an autosampler. Metrohm offers the OMNIS Sample Robot – NIR combined with an OMNIS NIR Analyzer for applications which require the analysis of many vials. The analysis system can be equipped with **more than 200 samples** as shown in **Figure 13**.

The usage of OMNIS NIRS is not only ideal from a workflow perspective, but also from a regulatory point of view, where guidelines such as USP 856 and FDA 21 CFR Part 11 need to be fulfilled. The OMNIS software can be used in a compliant mode which includes functionalities such as audit trails, signatures, and user management levels as stipulated by FDA 21 CFR Part 11. Furthermore, different system tests (e.g., validation of wavelength accuracy, signal-noise tests, photometric linearity tests) for a full validation of the OMNIS NIR Analyzer are present.

Figure 13. OMNIS Sample Robot – NIR with different racks for different vial sizes. One sample vial is positioned for measurement on the OMNIS NIR Analyzer and is automatically exchanged by the sample robot arm once analyzed.

The usage of such a system is shown in this application, where 72 lactose samples with different levels of water content were analyzed. The samples were stored in sealed vials, and analysis occurred directly through the glass. The following figures of merit and correlation diagram were obtained (**Figure 14**) which confirm the applicative feasibility to determine quality control parameters of samples in closed vials with NIRS.

Water content in lactose samples:



Product	Parameter	SEC wt%	SECV wt%	R^2
Lactose	Water	0.18	0.19	0.970

Figure 14. Correlation diagram and figures of merit for the determination of water content in lactose samples. The reference water content was determined using a Karl Fischer oven method.

SUMMARY

With OMNIS NIRS, users get a flexible, modular analytical solution which is suitable for many industries. Unique functionalities include:

- automatic model development (OMD)
- a high measurement speed
- the possibility to recognize sample insertion and removal
- sophisticated temperature control
- the ability to combine different analytical technologies from a single manufacturer—even in an automated system

Boost the efficiency of your laboratory with OMNIS NIRS.

Reference

[1] ASTM International. *Standard Practice for Performance-Based Qualification of Spectroscopic Analyzer Systems*; ASTM D8340-21; ASTM International: West Conshohocken, PA, USA, 2022.

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