Sampling solutions for NIRS Process Analyzers

Find the best solution for your sample

PUSHING
THE
LIMITS
TOGETHER

Metrohm
Process Analytics

Monitoring your process 24/7

The right choice for success

Near-infrared (NIR) spectroscopic techniques are characterized by their ability to gain rapid, accurate information from the high-resolution spectra of solid, gas, and liquid samples without prior sample preparation or chemical reagents. They are economical and facilitate qualitative and quantitative as well as noninvasive and nondestructive analysis. For all these reasons, spectroscopic techniques are ideally suited for industrial quality control and process monitoring.

Maximize profitability, comply with regulations, and stay safer

The determination of multiple parameters in a single measurement, and the possibility of fiber optics implementation for remote analysis has made NIR spectroscopy an invaluable tool for academic research and industrial quality control. Metrohm Process Analytics offers multiple NIR process analyzers that facilitate non-destructive, accurate measurements directly in process streams.

In the industrial world, online and inline analysis are the preferred solutions for «real-time» process monitoring, without interference. Currently, the use of fiber optic probes and flow cells with NIRS systems has opened up new horizons for process monitoring. They are an economical means of performing remote, unattended measurements, reducing operation costs and increasing productivity.

For tight spaces, NIR process analyzers can be placed at a distance from the sampling point with the use of low-dispersion fiber optic cables. Additionally, the selection and implementation of the correct optical probes and flow cells is crucial to obtain successful «real-time» results from a process.

With more than 45 years of experience, Metrohm Process Analytics can design the best solutions for your process. If required, up to 16 sampling points can be multiplexed with each analyzer – providing even more information about your process and its quality. Because sample conditions are dependent on the applications, selecting the right sampling solution to use with a NIR system is crucial for successful process implementation.

This brochure is the perfect guideline for choosing the correct sampling solution for the best analytical performance. Identifying and implementing the right fiber and probes has never been easier. Our sales specialists work with you to ensure that the system you select provides the highest quality and most reliable data. The process of selecting the ideal sampling solution is a team effort requiring the full collaboration of all stakeholders: the user, the integrator (if any), and Metrohm Process Analytics.

The complete solution for your process

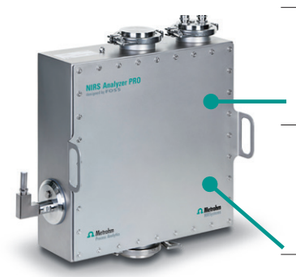
NIRS is a fast, chemical-free, non-destructive analysis technique used in many industries for in-process control of manufacturing operations and measuring product quality. Metrohm Process Analytics offers two versions of process NIRS systems: the NIRS Analyzer Pro (right, bottom image) and the NIRS XDS Process Analyzer (right, top image).

In addition to process analyzers, Metrohm Process Analytics offers complete solution packages for seamless integration: optical fibers, sampling solution (flow cells and/or probes), Industrial PC (IPC), and process communication protocol (digital and/or analog). Implementing online and inline process analyzers requires experience in many technical fields that we have developed over 45 years.

Multiplexing capability of NIR systems – More savings per measurement point and results

No reagents – No chemical waste

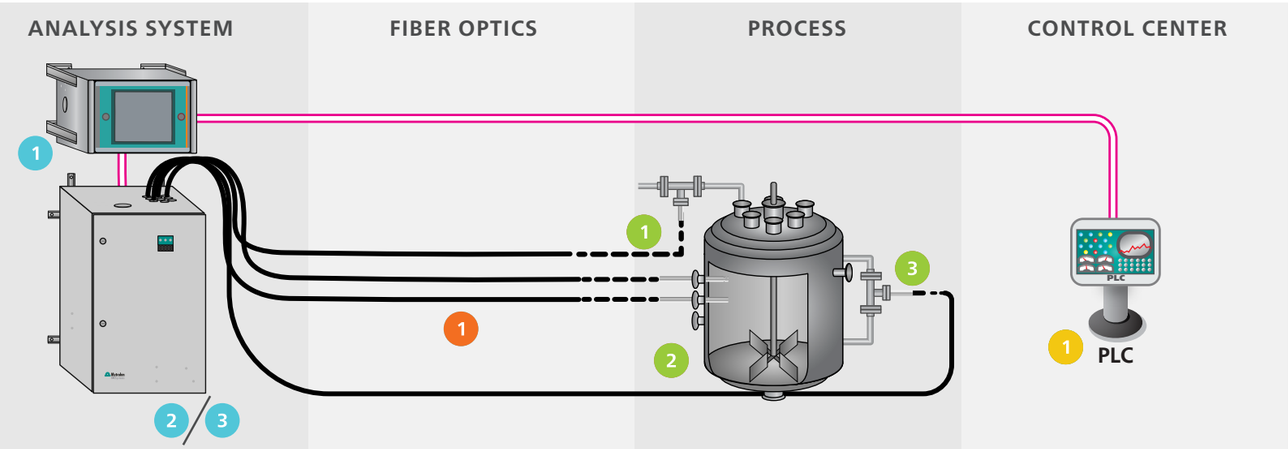
Wide scan range (800 to 2200 nm) that enables a wide range of applications



Fast analysis results around the clock (24/7) for any sample (liquids, solids, and gases)

Robust industrial design for operation in harsh and hazardous environments

SAMPLING POSSIBILITIES FOR REACTION MONITORING WITH PROCESS NIR ANALYZERS



ANALYSIS SYSTEM	FIBER OPTICS	PROCESS	CONTROL CENTER
1 2060 Human Interface (HI)	1 Optical fibers (Single or Microbundle)	1 Probe / flow cell (inline analysis)	1 Direct feedback through industrial communication protocols (Programmable logic controller «PLC» or Distributed computer system «DCS»)
2 NIRS XDS Process Analyzer		2 Measuring head / measurement window connection (inline analysis)	
3 NIRS Analyzer PRO		3 Bypass probe / flow cell (online analysis	

SAMPLING SOLUTIONS

Highly precise measurements do not only depend on the analytical process analyzer. In fact, the type and form of sampling is just as important to obtain high quality results. Depending on the application, each sample is subjected to different conditions, such as temperature and pressure. Furthermore, the chemical composition and physical properties are equally important, since they also influence which type of sampling is necessary.

The key factor in sampling determination is to obtain reproducible results. This ultimately requires a representative sample of the same quality in front of the probe at all times which can be challenging. Metrohm Process Analytics has decades of experience in selecting the best sampling solution for a given sample in a specific process. Consult our experts!

BENEFITS OF USING PROBES AND FIBERS WITH NIRS PROCESS ANALYZERS

- **Customizable sampling systems** according to the nature of the sample. Solid, liquid, and gas samples can be measured on the same instrument.
- **Best analytical performance** for your process. Each sampling point has unique process conditions.
- **Measure everywhere, close by or far away.** Sampling points can be hundreds of meters apart (e.g., two sampling points can be 400 m from each other with the instrument in between).

Versatility of fiber optic probes for process monitoring

Chemical analysis in process streams is not always an easy task. The chemical and physical properties such as viscosity and flammability of the sample streams can interfere with the analysis measurements. Some industrial processes are quite delicate – even the slightest changes to the process parameters can lead to significant variability in the properties of the final products. It is therefore essential to continuously measure the properties of the stream and adjust the processing parameters via rapid feedback to assure product quality and consistency.

NIR spectroscopy is well suited to this task, providing «real-time» structural and kinetic process data. It is rapid, requires little or no sample preparation, and minimal technical expertise to operate. Many of these benefits are due in no small part to its most common method of interface – a **fiber optic probe** placed directly into the reactor system. This eliminates the need for sample preconditioning, and consequently reduces time and experimental error associated with it.

ADVANTAGES OF FIBER OPTIC PROBES

The use of fiber optic probes in NIR spectrometer systems has opened up new perspectives for process monitoring. A NIR probe connected to a spectrometer via optical fiber allows direct online and inline monitoring without interfering with the production process. Fiber optic probes can be placed in very harsh working environments, while the spectrometer and analysis computer remain safe and secure in a shelter. In fact, remote monitoring can be achieved at large distances without significant impact on the signal-to-noise ratios.

Nowadays, a wide variety of NIR optical probes are available, from transmission pair probes and immersion probes to reflectance probes suitable for contact and non-contact measurements. This diversity allows NIR spectroscopy to be applied to almost any kind of sample composition – including melts, solutions, emulsions, suspensions, gas, and powders.

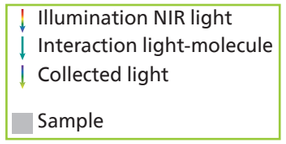
FINDING THE RIGHT MATCH

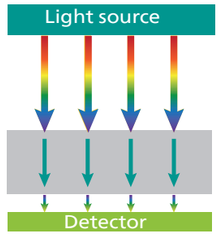
Selecting the right probe, or sample interface, to use with a NIR process analyzer is crucial to successful process implementation for inline or online process monitoring. For the system to perform optimally, the probe must be optically matched with the spectrophotometer and with the optical fiber that transmits the spectral data.

Some of the challenges faced by NIR sampling interfaces for process monitoring include sustained high temperatures, sudden changes in temperature, extreme pressures, turbulent flow issues, and fouling of the probes. In order to succeed and overcome these issues, the probe must be selected based on the application, the measurement method, and environment in which it is to be used. The material of the probe must be inert to the chemicals/materials to which it will be in contact with, and yet it must withstand extreme process conditions.

Measurement principles

NIR spectroscopy can be used to analyze different types of samples. Choosing the right measurement method, sampling module, and accessories is the most important step to developing robust NIR methods.

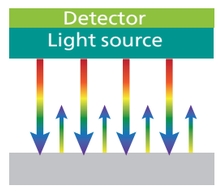




Transmission

– Sample type: clear liquids, suspensions, and solutions

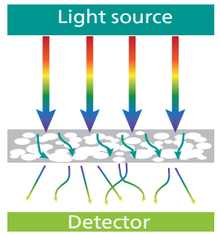
The sample is placed between the NIR light source and detector. NIR light is transmitted through the sample. The non-absorbed NIR energy continues to the detector.



Reflection

– Sample type: flat surfaces, powder, polymer pellets, fluffy materials

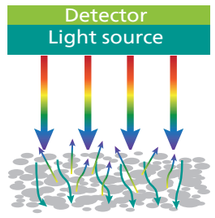
The NIR light is directed on the sample which interacts with the light and reflects the non-absorbed light back to the detector. The sample can either be at a distance from a few millimeters to 30 cm from the light source – refered to as a «non-contact measurement». However, when the sample is in contact with the lens, this is referred to as an «interactance reflectance measurement».



Diffuse transmission

– Sample type: solid dosage forms without sample preparation (e.g. tablets and capsules)

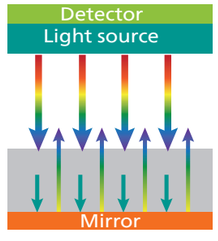
NIR light penetrates into the sample and interacts with the sample. Due to the particles, the light is scattered throughout the sample. The non-absorbed NIR light is transmitted through the sample reaching the detector.



Diffuse reflection

– Sample type: solid samples without sample preparation (e.g. creams, pastes, granulates, coarse, fine powders)

NIR light penetrates into the sample, interacting with it. The NIR energy that is not absorbed is reflected back to the detector.



Transflection

– Sample type: liquids and gels

This measurement method is a combination of transmission and reflection. A reflector is placed behind the sample to reflect the non-absorbed NIR light back to the detector.

Dedicated fibers for optimal performance

FIBER OPTIC INTERFACING

Generally, NIR light from the instrument is transferred to the process probe using fiber optic cables. As the light scattering properties of the process sample increases, the number of fibers used in the fiber optic bundle must be increased in order to maintain analytical performance. All fibers are made of a low-dispersion material to minimize NIR absorption of the fiber itself. Nevertheless, the fibers absorb light strongly above 2200 nm, therefore process NIR instruments with fibers use a maximal wavelength of 2200 nm.

Microbundle fibers are used to monitor slightly scattering liquid media, suspensions, and drying processes. There are two types of microbundle fibers: single and double microbundle.

– **Single microbundles** are composed of 37 fibers with 200 µm diameter silica core. The termination on the instrument is an SMA 905 connector, while the termination on the sampling side is a metal cladding which is inserted within a probe. Therefore the probe length must match with the metal cladding length.

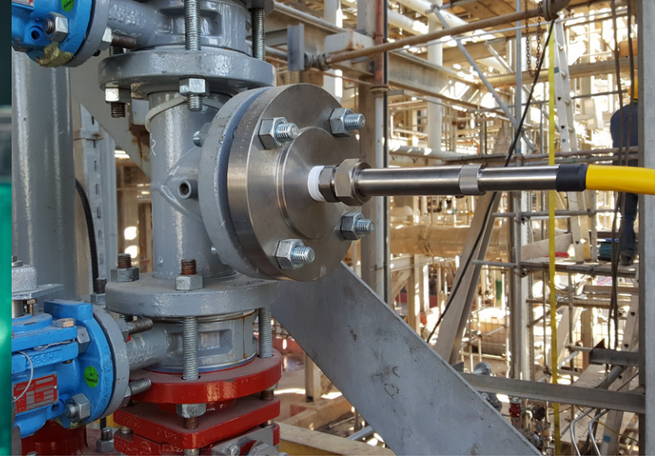
– **Double microbundles** are composed of two bundles of 37 fibers with 200 µm diameter silica core. The termination on the instrument are SMA 905 connectors and the termination on the sampling side is a metal cladding which is inserted within a probe. The illumination bundle forms a circle when the collection fibers are bundled in the center. Therefore the illumination light and the collection light follow the same path.

Single fibers are typically employed to analyze clear liquids. Single fibers have a 600 µm silica core made of single fibers, which are protected with a double layer PVC tubing with Kevlar strain relief. There is a gas-tight gel filling between the layers. Termination on both sides are SMA 905 connectors. Single fibers can have lengths up to 200 m. The use of longer fiber optic lengths can enable a process analyzer to be located outside of electrically classified or safety classified areas, and can keep it isolated from harsh operating conditions such as large temperature variations.

Metrohm Process Analytics designs the best solutions to provide you the best results. In the following pages, you will find the optimal solution that we offer depending of the nature of your sample. The sample properties define the sampling solution (probe/flow cell) which in turn defines the type of fibers to be used.

Liquid Samples

in fast loop, bypass, reactors, and tanks



CLEAR AND TRANSPARENT LIQUIDS

Clear and transparent liquids are easy samples to measure with NIRS because the light passes easily through the sample without any disturbances. For these types of samples, we recommend using either flow through cells for measurements in transmission, or probes for measurements in transflection depending on the various possibilities within your process.

Samples in fast loop, bypass pipelines
Flow through cells can be installed in a bypass line or a sample preconditioning panel for measuring liquid samples. The advantages are clear:

- The flow rate can be controlled to avoid turbulent flow. Laminar flow is preferred for reproducible measurements.
- Bubbles are not an issue, they can be eliminated.
- A preconditioning panel with filters before analysis is possible to implement. This eliminates particles which might interfer with the measurements.
- Easy access to the flow cell (if cleaning is necessary).
- Samples can be collected at the same point as the NIR system, in order to do model building and/or validation.

Industrial flow cell for fast loop and bypass pipelines

Industrial flow cells for harsh industrial environments

Flow through cells work perfectly with single fibers. The **optical path length** (OPL) is fixed, but it can be adjusted on some flow cell models. For protic solvents (e.g., alcohols, amines) an optical path length of 4 mm is recommended, with 5 mm recommended for aprotic solvents (e.g., alkanes, DMF, DMSO).

For aqueous samples with water content above 95 wt%, an OPL of 0.5 mm is advised. Each time the water content decreases by 10 %, 0.05 mm can be added to the OPL(e.g., for a sample with a maximum of 55 % water, we select an OPL of 0.7 mm).

Depending on the requirements, several flow cells are available with different options, such as temperature control or in different materials. Flow cells are usually connected to the process with Swagelok®, but flanges can also be used.

Samples in reactors, pipelines and tanks
Some chemical processes consist of multiple reactor vessels, long pipelines and immense tanks, in which flow cells are difficult to implement.

In such cases, industrial probes are the right solutions to perform the NIR measurement. There are two types of measurement principles that can be used to measure liquid samples: transmission (the same as in flow cells) or transflectance.

Transmission probe, left: fiber connection; right: sampling with a 2 mm OPL

Transmission probes work with single fibers and true transmission measurements. The determination of the OPL is identical to that of flow cells, and is fixed for this type of probes. Transmission probes are also called «single pass» probes.

The light passes twice through the sample with **transflectance probes**[1], thus, the measured gap in the probe is actually half of the OPL (e.g., a 1 mm transflectance probe corresponds to a 2 mm OPL). The liquid sample is contained between a lens and a concave gold-coated mirror. Therefore, the light is focused on the sample, and the signal-to-noise ratio is higher than for transmission measurements (more precise).

Top: Single fiber transflection probe with a set of mirrors for defined OPL (left to right): 0.5 mm, 1 mm, 2 mm, 5 mm, 10mm. Bottom: Microbundle fiber transflection probe with adjustable mirror

For single fibers, mirrors with pre-set OPL are available, but it is not the case for microbundle fibers. A mirror slides along the probe axis to adjust the OPL. When an application requires high precision measurements, microbundle fibers are the preferred selection. Similar to the flow cells, probes can be connected to the process stream via Swagelok®.

Example of process connections: left: Transmission probe with welded flange; right, Transflection probe with welded flange

[1] Note that for the same sample and OPL, the spectrum from a transflectance probe will have **25 %** more absorbance than the spectrum measured in transmission. Transflectance probes work with single fibers or microbundle fibers.

Liquid Samples

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in fast loop, bypass, reactors, and tanks

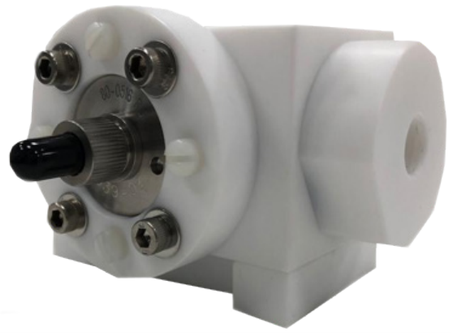
CLEAR AND TRANSPARENT LIQUIDS – HARSH CHEMICALS

Sampling solutions made of metals (e.g., stainless steel) are not always compatible for all applications. In cases where inorganic acids such as sulfuric acid (H_2SO_4) or hydrofluoric acid (HF) are present in the sample stream, other materials are required for the sampling solutions. Perfluorinated polymers are compatible with such substances. Here, we describe some sampling solutions made of polytetrafluoroethylene (PTFE). Be aware that other types of perfluorinated polymers can also be used (e.g., perfluoroalkoxy alkanes, PFA; or polyvinylidene fluoride, PVDF).

Single fiber flow cell (adjustable OPL with spacers)
Transmission measurements can be performed with a flow through cell made of PTFE. The same rules apply for the determination of the OPL as if it was made of stainless steel 316L. This PTFE configuration allows sample contact with a sapphire window, which is inert to most chemicals. Optionally, an extra thin layer of PTFE can be added to the window so the sample is only be in contact with PTFE. For applications with high water content, a path length between 0.5 and 1.0 mm is preferred.

Transparent process pipelines (PTFE Clamp-on flow cell)
When modifications on the process line are not possible (e.g., using a bypass), or the sample stream cannot be in contact with the sampling solution (e.g., flow cell), a PTFE clamp-on flow cell is the right solution. This clamp-on flow cell works with single fibers and is simple to implement. Just clamp it on to a pre-existing tubing at tyour production site. The OPL depends on the size of the sample stream tubing.

Result quality is non-proportional to the diameter of the process tubinge. Rather, as the tube decreases in size, its curvature increases, which interferes with the results.



PTFE Single fiber flow cell on top of a stand

PTFE Single fiber clamp-on flow cell

PTFE transflection probe
Probes are also in contact with the sample stream, and therefore chemical compatibility is important to consider. These probes work perfectly with microbundle fibers and the OPL is adjustable by moving the mirror position.

PTFE PROPERTIES:
- Hydrophobic
- Chemically inert
- Nonreactive to almost all known chemicals
- Excellent thermal resistance
- Nonadhesive

CLEAR AND TRANSPARENT LIQUIDS – DEMANDING PROCESSES

Transmission probe pair
Some applications are quite demanding and require specific solutions for reliable analysis. Sample streams at elevated temperatures and pressures are particularly challenging for regular flow cells. An alternative solution is transmission probe pairs from Metrohm Process Analytics, which can withstand up 250 °C and 5000 psi. Transmission probe pairs work with single fibers or microbundle fibers, the latter giving about 20 % more signal for the same sample and OPL.

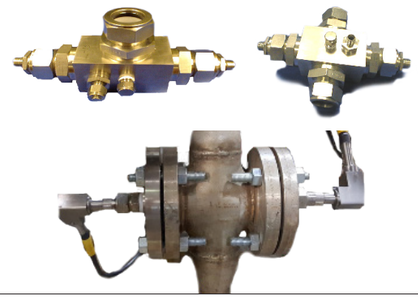
In addition, transmission probe pairs are also available with threads, making them especially suitable for polymer extrusion applications or for applications requiring up to 30,000 psi.

High temperature probes
For sample temperatures above 250 °C, the probe design includes an inlet for cold compressed air to cool the tip of the internal fiber and increase the fiber lifetime. Transmission and transflection microbundle probes are suitable for high temperature processes.

Although it sounds complicated, implementing these probes is extremely simple and straightforward. Transmission probe pairs can be directly inserted in a cross section of a pipeline, connected through a Flange, a Swagelok® or a combination of both. For installation on smaller pipelines, flow cells in which probes can be inserted are available. (Same situations applies for transflectance probes).

Top: Transmission probe pairs with spacer, top center: single fiber, middle center: microbundle fiber. Bottom center: Transmission pair with thread for implementation in extruder.

Low temperature probes
For temperatures from 0 °C down to -110 °C, the probe design includes an inlet for dry nitrogen to prevent condensation and the formation of ice crystals inside the probe. Special O-rings adapted for such temperatures are used. Single fiber single pass transmission probes can be used for low temperature processes.



Top left: flow cell for transflection probes. Top right: flow cell for transmission probe pairs. Bottom: example of a transmission probe pair implementation directly on a process pipeline.

Solid Samples

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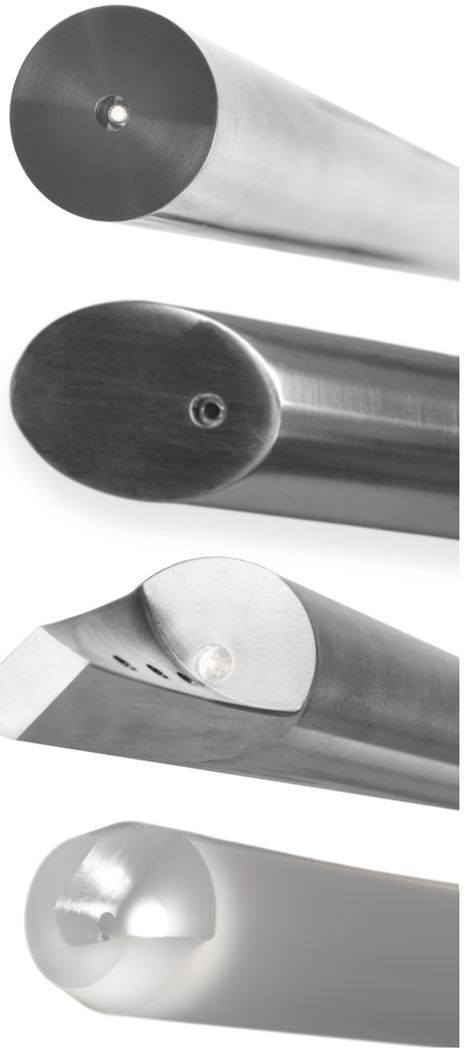
powders, crystals, tablets and more

FINE POWDERS

Reflectance probes for fine powders
For measurements of fine powders, the reflective power of the sample itself is used to our advantage. The particles can act as small mirrors, thus interactance reflectance probes are the best solutions to measure this type of sample. The spectra obtained by the process analyzer is the NIR light which is absorbed by the solid particles. Additionally, the scattered light is also measured, influencing the position of the spectrum baseline. Therefore it is possible to measure particle size by analyzing the baseline position on the spectra. Other physical parameters of solid particles such as melt index or intrinsic viscosity can be measured. The bandwidth of the peaks is influenced by those physical parameters and can be quantified as well.

The **reflectance probe** has a sapphire lens which is directly in contact with the metal (no epoxy or O-ring in contact with the sample). This probe can be used in conditions up to 250 °C and 5000 psi and with additional options which withstand up to 400 °C. The lens focuses the light on the external surface of the sapphire. Therefore, the sample needs to be in contact with the sapphire to obtain precise and reproducible spectra.

Sample color is also important, especially when it comes to black powders. Black powders influence the amount of reflected light and very often these samples cannot be measured. However, it can be that the powder is black but composed of metallic particles, in which case they will act as actual mirrors.



Example of reflectance probes from top to bottom: regular probe, 45° angle probe, SS spoon probe, and PTFE spoon probe.

The window of the probe has a small diameter. Some tricks can be used to ensure the best contact:

- When the sample stream is vertical (e.g., the sample is falling down a pipeline or in a dryer), spoon probes are the best solution. The measurement is performed when sufficient sample falls in the spoon. After the measurement is completed, compressed air is blown to remove the sample, so a fresh sample can be collected next. This spoon probe is available in stainless steel 316 L or alternatively in PTFE. The contact surface with the sample is exclusively PTFE. A small layer of PTFE covers the lens, and the light passes through this layer.

This is particularly useful if the sample has etching properties or if glass, lenses, or windows are prohibited in a process.

- 45° probes are used for powder flowing in a horizontal pipeline or in a blender with a mechanical stirrer. Since the sample can be flowing in different directions, the 45° angle on the probe nozzle ensures good contact between the sample and the lens.

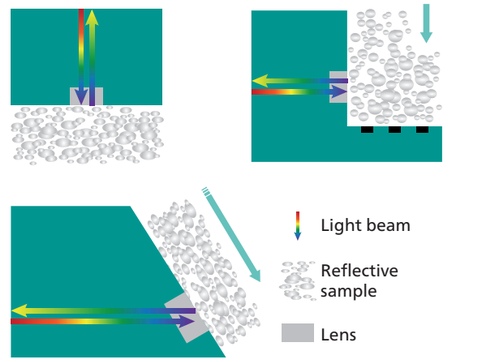


Illustration of different type of reflectance probes

Spoon probe implemented in a dryer

OPAQUE SAMPLES

Diffuse reflectance and diffuse transmission measurements for tablets
Some samples such as tablets or polymer films are opaque but the light can still pass. They appear opaque, milky, or white due to the scattering of light within the sample. For these types of samples, a diffuse type of measurement is used. We recommend using the microbundle transmission probe pair for polymer films and opaque samples, and diffuse reflectance probe for tablets and porous samples.

Diffuse reflectance probe

Solid Samples

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pellets, crystals, ores, and more

MACROSCOPIC PARTICLES

Non-contact probes for large particles
Because of their bigger shape and non-uniformity, it is almost impossible to use contact probes to measure macroscopic solid particles such as polymer pellets, wood chips, and crystals, among others.

For such samples, a non-contact probe is preferably used for inline analysis. This probe is unique, as the fibers are permanently attached to it. Microbundles consist of 6 x 15 fibers for illumination, and 1 x 91 fibers for collection.

The illumination fibers form a focal plane approximately 8 mm away from the surface of the probe. All particles present between the focal plane and the probe will act as a mirror and reflect the light back to the detector.

Non-contact probe used for the analysis of large non-uniform particles

For very soft samples such as wool, a spoon sleeve can be to collect the sample and then remove it with compressed air as it is done with fine powders.

Even if the principle is called non-contact, the probe is still in contact with the sample. It can be used in non-contact mode through a window if the sample is in permanent contact with the window.

Example of non-contact probe inserted in an outer sleeve for fluffy samples

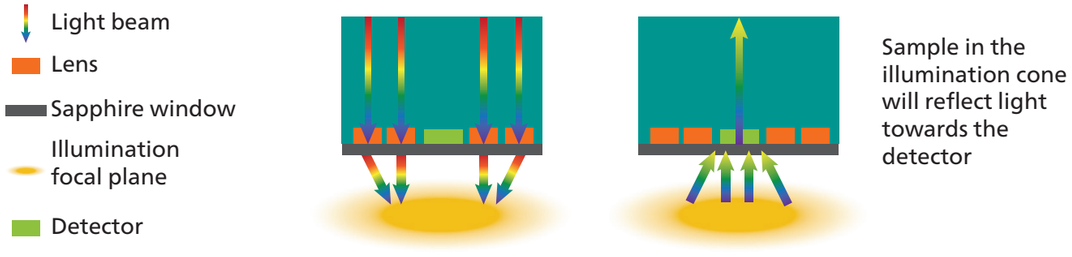

Illustration of the non-contact probe measurement principle

PLAIN SOLID SAMPLES

With the NIRS Analyzer PRO, it is possible to measure samples from a distance. This is ideal when samples are:

- Moving on a conveyor belt
- Rolls of polymers films
- Plain solid substrate (e.g., paper)

The distance between the NIRS Analyzer PRO and the sample will define the measuring spot size. For reproducible measurements, it is important that this distance is kept as constant as possible. With heterogeneous samples, a bigger collection spot size is advised to be able to average the sample. Alternatively, the NIRS Analyzer PRO can be put in motion itself to cover as much surface as possible.

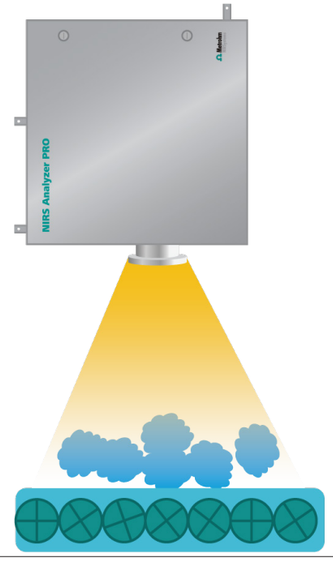

Illustration of a NIRS Analyzer PRO above a conveyor belt

DISTANCE SAMPLE-INSTRUMENT GUIDELINES

Height above sample [mm]	Collection spot size [mm]
50	25
100	50
150	70
200	90
250	100
300	120

Non-contact probes for plain solids
Non-contact probes can also be used to measure from a short distance (10 mm) with a small spot size (15 mm) when the NIRS Analyzer PRO provides too much averaging, such as in the example below.

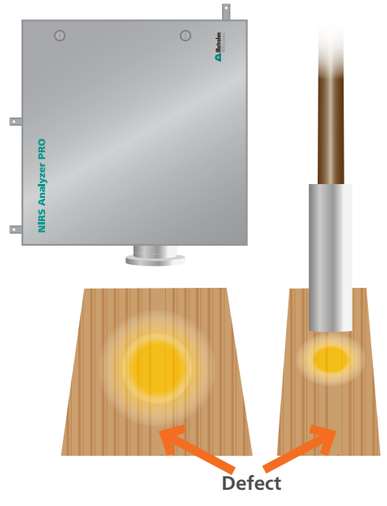

Illustration of the spot size difference between a NIRS Analyzer PRO and a non-contact probe

Suspensions and Slurries – Solid or liquid?

SUSPENSION MEASUREMENTS

> 30 wt% solid content
Sample streams with a relatively large amount of solids can be a challenge to quantify. The measurements have to be done in reflectance mode and the solid part can be measured without a problem. For the liquid part of the sample stream, measurement depends on its properties. If the sample is water-based, the solid particles need to be removed before analysis. If the sample is non-aqueous, the liquid content can be quantified concurrently with the solid content.

< 30 wt% solid content
Sample streams with low solid content can be measured using a transmission probe. Since the solid affects only the baseline and peak bandwidth, the solid part can be quantified, along with the liquid solution. Aqueous solutions can be challenging due the small OPL used and the risk of probe fouling.

Metallic particles
Sample streams which have metallic particles are good reflectors and are therefore easy to quantify. However, because of their reflective power, the flow rate has a large influence on the NIR spectra. For the most reproducible measurements, flow rate should be kept constant.

Gas Samples

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Continuous and batch-wise analysis

There are different ways to treat gas samples for analysis. If they can be liquefied, they are treated as liquid samples. If they remain gaseous, then there are two approaches which can be used depending on the process setup:

- For **continuous** analysis, the gas sample stream is run through a flow cell at a constant flow rate and temperature. Like clear transparent liquids, the measurement is carried out in transmission mode and thus absolute concentration values can be obtained.
- **Batch-wise** analysis is also a possibility to measure gas streams. First, the exit valve of the flow cell is closed so the sample pressure builds up within the flow cell. When the pre-defined pressure is reached, the valve at the entry of the flow cell is closed as well to proceed with the analysis. The measurement is made in transmission mode at constant pressure and temperature, so relative concentrations are obtained. This strategy is used to measure low concentrations.

It is important to note that gas samples follow the ideal gas law: the concentration is directly proportional to the **pressure** and inversely proportional to the **temperature**. Therefore, these two parameters need to be precisely controlled for accurate and safe analysis.

Molecular density is an important parameter in gas samples. The density of the molecules in a gas sample is lower than in liquid samples, therefore longer OPL are used, ranging from a few centimeters to meters. The OPL depends on the precision required and concentration levels.

Near-infrared spectroscopy is a secondary technique, meaning it requires a prediction model (obtained by a primary/reference method such as titration or HLPC) if quantification of samples is desired. For gas samples, gas chromatography is the preferred reference method. A single fiber transmission probe pair or a microbundle transmission probe pair can be used for such gas applications. The microbundle version gives better accuracy due to higher signal-to-noise ratio.

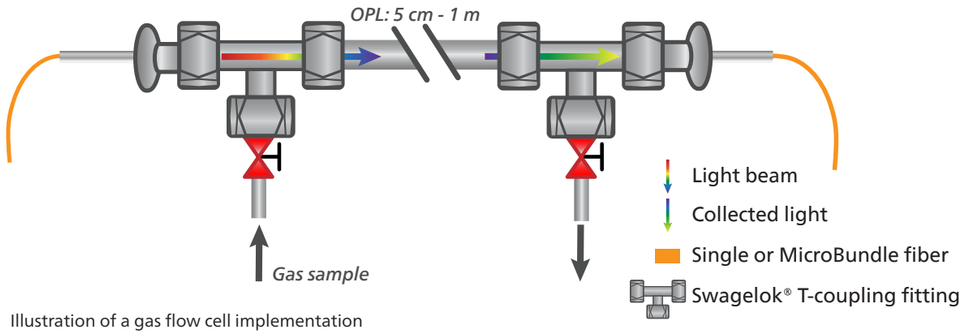


Illustration of a gas flow cell implementation

Selection of sampling solutions

Because every production plant, process, and sample is unique, there is no universal rule for the selection of the ideal sampling solution. The sampling solution is as important as the technology itself. Appropriate sampling will result in good reproducibility and precision. Here are some guidelines on how to select the best sampling solution:

1

MEASUREMENT

First, consider the phase of the sample which will determine which measurement principle to select:

- Transmission (Transflection) for liquids, gas, and some suspensions
- (Diffuse) Reflectance for powders and some suspensions
- Non-contact measurement for plain solids and coarse grains

MEASUREMENT PRINCIPLE REFINING

2

Second, consider the constituents to be measured and the precision needed. These parameters give information about the optical path length to select for transmission/transflection measurements or about the measurement distance for non-contact measurements.

3

ROBUSTNESS/RESISTANCE

After the measurement principle is defined, the type of probe or flow cell must be selected. The sample properties (corrosive, abrasive) and conditions (temperature/pressure/viscosity/flow rate) need to be considered to select the correct sampling solution and construction material. Standard metals used include SS316, Hastelloy C, Titanium 6, Monel, and nickel.

FIBERS

4

The type of fiber to be used will be deduced from the steps above. It is important to consider a protection for the fibers with a PVC conduit, for instance, to prevent mechanical and/or chemical damage. Usually, microbundle fibers are used for increased signal-to-noise and precision.

Implementation of sampling solutions

The placement of a probe or a flow cell is not trivial. Very often, the existing industrial process has limitations such as space contraints or safety regulations to comply to. Here are some guidelines on how to implement sampling solutions:

PROBES

Probes that measure in-situ are connected directly to the process via flanges and/or Swagelok®. Such immersion probes are permanently installed which make them more difficult to maintain. However, they are are typically cleaned by circulating or pulverizing a cleaning solution on the probe surface or with the use of steam. Care must be exercised when using steam due to the extreme temperature. When the only option for sufficient probe cleaning is the probe from the process itself, implementing a flow cell is much more practical. In doing so, the flow cell is isolated from the main process stream avoiding any disturbance to normal process operations while cleaning the probe. Solutions for probe extraction are available if needed.

The immersion depth of the probes depends on the type of flow within the process. The ideal immersion depth should be where the sample is the most representative and is therefore process dependent.

FLOW CELL

Flow cells are usually installed on fast loops or on bypass streams. Differences in flow rates and pipeline size have to be considered to avoid turbulent flows or the presence of bubbles. Flow cells are preferably installed in a box isolated from external conditions. Flow cells offer the possibility to use preconditioning panels prior to the flow cell to prepare the sample (e.g., filtration, extraction) and to achieve good sample reproducibility. Another advantage to integrate flow cells within sampling panels is that validation can be easily implemented in two different ways: known sample concentrations can be injected directly, while manual samples can be collected at the same point for laboratory measurements. Temperature control is also easier with flow cells. It is recommended to have the sample flowing upward to avoid gravity effects or settling effects when measuring suspensions.

Timeline for sampling solution deliveries

Some probes are standard items but in more than 90% of the cases, the sampling solution is customized to the process requirements. The selection and ordering of the sampling solution is a multi-step process involving many parties. This process should not be overlooked, but rather addressed from the beginning of a project.

SAMPLING:

- Laminar flow – reproducible measurements
- Filtration options possible (particulate/water) to avoid interferences
- Temperature control for better accuracy
- Easy accessibility to probe (for cleaning or running calibration standards)
- Ability to collect the sample at the same point where the NIR instrument collects spectra (for calibration, validation, or updating of models)

1

APPLICATION QUESTIONNAIRE

Metrohm Process Analytics is a solution provider and will help you to select the best technology for your application.

The NIRS technology is selected based on the application questionnaire filled in by the user.

2

PROBE QUESTIONNAIRE

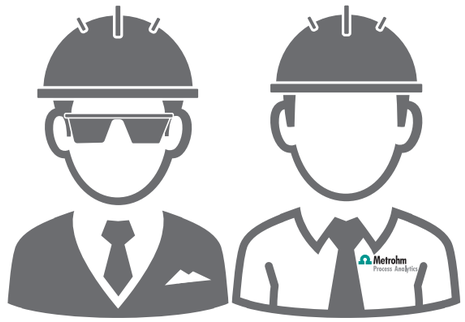
The requirements for the sampling solution are defined to get the best fit.

The sampling solutions are selected and presented to the user with a budgetary offer.

3

TECHNICAL DISCUSSION

The solution is discussed between Metrohm and the user, including all stakeholders from the process engineers to IT personnel.

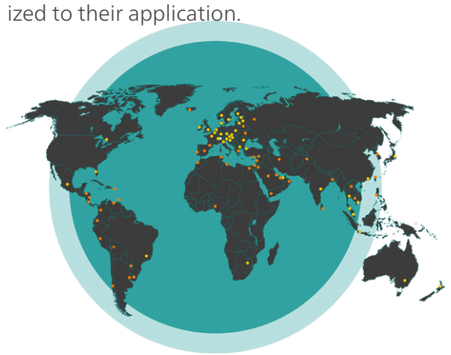


The decision is not only based on the best sampling but also on their implementation. Drawings are approved.

4

PRICING - DELIVERY TIMES

A complete project estimate (including commissioning, trainings and sample preconditioning panels) is provided to the user customized to their application.



Metrohm Process Analytics is locally present worldwide, with more than 10,000 industrial process solutions installed and commissioned.

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