Innovation with Integrity

MATRIX-F/-MF

• Keeping an eye on your process

FT-IR / FT-NIR
Optical spectroscopy is today a highly important technology for online process monitoring and optimization. Fiber-coupled probes allow a direct look into the process without time delay. The award winning MATRIX FT-IR and FT-NIR spectrometers allow the direct measurement in process reactors and pipelines, leading to a better understanding and control of the process. Their innovative technology provides consistent high quality results, less downtime and direct method transfer. All Bruker process spectrometers are characterized by robustness, long-term stability, and low maintenance costs. Thousands of installations in the chemical, petrochemical and polymer industry as well as in pharmaceutical production processes and in the field of food and feed manufacturing prove our experience.

FT-IR / FT-NIR Process Spectrometers

- Check important process parameters immediately for reaction monitoring and control

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FT-NIR Process Monitoring

Today many manufacturers are striving not only to produce the highest quality final product but also to improve manufacturing efficiency by shifting the final quality analysis from the laboratory to the production plants.

By gaining tighter control over the manufacturing process, it is possible to optimize the use of materials and reduce or eliminate the production of off-specification batches, thus saving reprocessing or disposal costs.

The key advantages of using the MATRIX-F FT-NIR spectrometers online are:

- Accurate in-line results in seconds
- Non-destructive, multi-component analysis
- Optional built-in 6-port multiplexer
- Direct method transfer
- Rugged, low maintenance design
- 10 years warranty on moving parts of the interferometer
- Available for Ex hazardous areas
- Ethernet connectivity and industry standard communication protocols

Fiber Optics Advantage

The advantages of real time, on-line FT-NIR analysis have been well established. However, conventional spectrometers can only be installed close to the process that they are monitoring, which often means exposing the analyzer to a hostile environment. This can include drastic temperature changes, high humidity and exposure to dust and dirt. Furthermore, the point of measurement is sometimes positioned in hard-to-access and often Ex hazardous areas.

By utilizing fiber optic technology, the MATRIX-F can be placed several hundred meters away from the actual measurement point, e.g. in an air conditioned room if the local conditions require. This will further optimize the performance of the spectrometer, since extreme temperature changes are eliminated. Moreover, the MATRIX-F is protected from excessive dirt and dust.

Bruker Optics offers complete solutions for various online analysis tasks depending on the customer needs.

- Fiber Optic Probes:
  - Classic diffuse reflectance, transfectance or transmission immersion probes with various path lengths can be adapted as well as process flow cells or pilot plant assemblies. Various probe materials are available, like stainless steel, Hastelloy or ceramics. Moreover, the probe can be customized to different lengths and flange geometries.
  - Optional built-in 6-port multiplexer
  - Direct method transfer
  - Rugged, low maintenance design
  - 10 years warranty on moving parts of the interferometer

- Sensor Heads for Contactless Measurements:
  - The fiber optic NIR illumination and detection head contains tungsten sources which illuminate the sample. The scattered light is collected and guided via a fiber optic cable to the spectrometer. This way, a contactless measurement can be performed remotely, opening a whole array of new applications.

The standard version of the MATRIX-F can adapt a variety of different fiber optic probes and is widely used for the online process monitoring inside reactors, pipes or bypasses. It offers fiber optic connections for the adaption of up to six flow cells or probes for the measurement of liquids and solids in contact.

For contactless measurements, Bruker designed the MATRIX-F emission. It utilizes the fiber-coupled sensor head for measuring solid samples e.g. over a conveyor belt or through a viewing glass inside a process.

The MATRIX-F duplex is a combination of the MATRIX-F and the MATRIX-F emission. Equipped with a second detector the light path can be switched between an internal or external NIR source and therefore offering the customer full flexibility. With these two options the MATRIX-F duplex is the only NIR spectrometer on the market offering the possibility to measure in contact and contactless with only one device.
Immersion Probes

Immersion probes are most widely used for FT-NIR measurements in process control. Bruker will assist to select the appropriate probe with the best materials matching the process conditions. In many cases the plant or reactor will already be equipped standard connection ports for immersion probes.

Immersion probes can be divided into three groups based on the measuring principle:

- Transmission Probes for clear liquids
- Reflection Probes for solid materials
- Transfection Probes for suspensions or emulsions

Different types of fiber optic cables are available. Standard transmission probes use mono fibers, whereas transfection and reflectance probes usually utilize fiber bundles to guide the light from and to the spectrometer.

Fiber optic probes are produced with the highest accuracy in terms of reproducible light transfer and exact path length. Calibration models built on a certain type of probe can be transferred without any manipulation of the data to systems using the same type of probes.

Flow Cells

Besides immersion probes, flow cells are widely used in process control. They can be implemented directly into a pipe or a bypass, allowing the sample to flow through the cell. The cells vary in size and can be applied to different pipe diameters. The NIR measurement is similar to the analysis using an immersion probe; a fiber optic cable transfers the light beam from the source to the sample. The light penetrates the sample, is collected by a second fiber and brought back to the detector.

The fiber optic cables are normally kept in a fixed position using brackets. Therefore, it is possible to remount the cell with high precision, after exchanging or cleaning optical components, allowing the calibration transfer from one measurement point to another.

Bruker Optics offers a variety of flow cells specified to the customer needs.

Sensor Heads for non-contact Analysis

The NIR sensor head was designed by Bruker for contactless measurements of moving solid materials in diffuse reflection mode. Compared to conventional reflectance, cubbies the Sensor Head offers some unique advantages:

- Two NIR light sources illuminate a sampling spot of approx. 10 mm in diameter - 20 times more than a conventional diffuse reflection probe.
- Monofibers are used to transmit the light back to the spectrometer, compared to cost-expensive fiber bundles for conventional reflectance probes.
- A reference standard is integrated for automatic background measurements without the need to dismount the sensor during the process, in contrast to most solid probes.

The large sampling area reduces the effect of variable particles sizes making it ideal for the measurement of heterogeneous materials like food and feed constituents, polymer pellets or slurries.

The head can be attached to any viewing window in pipelines or reactors and can also be mounted over conveyor belts to measure moving materials.

With the internal optical multiplexer of the MATRIX-F, up to 6 sensor heads can be controlled by a single spectrometer. This is reducing the investment per measurement position drastically, allowing the precision of FT-NIR spectroscopy at the cost of less advanced grating or filter technology.

Maximum Utility

Bruker developed various accessories, to facilitate an installation of the sensor heads in most demanding environments.

- A vortex cooling device is available for those areas, where elevated temperatures up to 75°C can occur.
- For dust-prone areas, an optional air jet assembly prevents fouling of the measurement window.
- A flexible connection, using a rubber gaiter allows the installation to vibrating machinery, like sifters.

Moreover, customizable adaptions to viewing windows or pipelines are available on request.
Process Analyzer Cabinets

The MATRIX-F and its peripherals like the external fiber optics multiplexer and industrial PC are designed to fit easily into standard 19" rack, making it ideal for the use in process analyzer cabinets.

These cabinets are recommended for the following situations:
- Occurrence of drastic temperature changes
- Access to the spectrometer needs to be restricted
- Exposure to dust
- Environment with high humidity

Air conditioned cabinets maintain a constant temperature and humidity inside hence guaranteeing a stable measurement even if the outside conditions are fluctuating.

Bruker offers cabinets in two different sizes. The large cabinet provides enough room for the MATRIX-F as well as for the external 6-port multiplexer and an industrial PC with TFT-display. The smaller size is designed for confined spaces and houses a single MATRIX-F.

Multiplexer

The MATRIX-F spectrometers are available with 1-, 2- or a 6-channel internal multiplexer. With only one MATRIX-F a maximum of 6 measurement points can be monitored thereby reducing the investment costs. The fiber optic probes are connected to the spectrometer using standard SMA connectors or BQCs (Bruker Quick Connectors).

If more analysis points need to be monitored, the external 6-channel multiplexer provides a convenient way to further expand the multi-point analysis capabilities of the MATRIX-F. It features a sequential, precise optical switching mechanism, similar to the internal multiplexer, for accurate and reliable measurements. The external multiplexer is equipped with connectors for fibers with SMA and BQC type.

Software

CMET Process Software

Especially in process environments a stable and reliable software solution is mandatory. Bruker developed the CMET process software as an independent platform, using the OPUS software only for spectrometer communication and evaluation tasks. An embedded watchdog functionality assures reliability and long-term stability of the software for a trouble-free operation.

CMET consists of a setup interface and a runtime environment. The setup interface with its modular and flexible interface incorporates all the necessary functionalities to setup a scenario for the different applications. This includes the spectrometer setup, product setup, defining the input-output communication protocols as well as the scenario setup.

Inside the scenario setup everything is combined. It allows the user to assign a specific spectrometer, with a specific measurement channel to a specific product. If necessary external triggers can be defined which allow the user to integrate the results of CMET into a process control software.

After the configuration is complete the runtime software is executed. The runtime software gives the user a complete overview of the running scenario including current task as well as trend charts regarding all the specified products.

CMET offers the most common industry standard interfaces which allow it to be integrated in any process control environment, using a wide range of standard communication protocols, including:
- 4-20 mA
- Modbus
- Profibus DP
- OPC
- ...
The standard configuration of the MATRIX-MF includes 1 fiber port for ATR probes. Optional available is a 6-port multiplexing unit offering a higher flexibility. ATR probes are mostly equipped with silver halide fibers. Due to a higher self-absorbance by the silver halide fibers the maximum distance between spectrometer and measurement point is limited to 5 m.

The MATRIX-MF offers the same benefits as the MATRIX-F such as long term stability and high accuracy. In case of a hostile environment the FT-IR can be housed inside a cabinet under controlled temperature and humidity. In the mid-IR region water vapor and carbon dioxide can cause interfering IR-absorption. Bruker offers an optional accessory for purging the spectrometer with dry air or N₂ for optimized spectral performance.

The application fields of the MATRIX-MF include:
- General R&D and pilot plants tasks
- Polymer chemistry
- Organic synthesis
- Fermentation
- Catalytic reactions

FT-IR Process Monitoring

The MATRIX-MF is the mid-infrared (FT-IR) version of Bruker’s MATRIX process spectrometer series. It has the same rugged and compact design as the MATRIX-F as well as the robustness and stability. Samples are measured by ATR (attenuated total reflection) probes without the need of sample preparation.

The MATRIX-MF utilizes the information rich mid-IR-region also known as the “fingerprint”. The mid-IR region is characterized by less overlapping of the absorption bands and functional groups can be easily identified. This is important for R&D, application development or process optimization since complex changes can be easily followed simply by peak height or peak integration.

The MATRIX-MF is available with two different detector types, DTGS and MCT. The DTGS detector is used at ambient temperature. However, it is less sensitive and normally requires a longer measurement time.

For more demanding applications, Bruker recommends the MCT detector. It is more sensitive and has faster response time, but it has to be cooled down to low temperatures during operation. Depending on the application, Bruker offers detectors which can be cooled with liquid nitrogen (LN₂), or permanently cooled detectors:
- LN₂: Detectors with 12 or 24 hours holding time
- permanently cooled: Cyrocooler or thermoelectrically (TE)-cooled detector

For more complex processes or R&D purposes, different pretreatment methods as well as multivariate methods can be applied in real time. This helps the user to setup the best possible way to follow the process trend. A 3D report is also available for process monitoring and end point determination. Bruker’s software enables several options to setup and define the end point of a process.

With the integrated event log, all actions (e.g. stirring or adding chemicals) can be written down precisely in relation to the currently measured spectra. This way, spectral changes can be related to either external influences or chemical reactions.

You can choose what specific kinds of information are important. The software can be adapted to monitor simple chemical reactions where e.g. a peak integration or peak absorbance can be used to monitor a process.

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- LN₂: Detectors with 12 or 24 hours holding time
- permanently cooled: Cyrocooler or thermoelectrically (TE)-cooled detector

Depending on the application Bruker offers different ATR probes made from different materials. In most cases an ATR probe with a diamond tip is the best solution due to its hardness and chemical resistance. For some chemicals, such as cyanates or isocyanates it could be necessary to use a Silicon ATR probe. The process probes are available in different diameters, length, materials and also as an ATEX version to accommodate process dependent needs.

FT-IR Fiberoptic Probes

Silicon ATR fiber probe (Ø 6 mm), ideal for measuring isocyanates.

Diamond ATR fiber probe (Ø 3.17 mm) for use in small reactors.

Diamond ATR fiber probe (Ø 12 mm), also for use in ATEX areas.
Bruker’s process spectrometers, the MATRIX-F and -MF, as well as the sensor head are all certified for the use in areas exposed to explosion hazards. The standard spectrometers and the sensor head can be retrofitted to comply with the high safety standards for Ex hazardous areas. Depending which part of the entire system has to be placed in an ex-rated area, the retrofitting can include the following changes:

- Data transfer via optical conduits in ex-rated areas
- Internal compartments of the spectrometers are pressurized and purged with dry air or N₂
- All surface materials (light fibers, power cable etc.) have to comply with the ATEX requirements regarding electrostatic discharges

MATRIX-F ex
The MATRIX-F is also available as an ex-proof, ATEX rated version, complying to the following standards:

- II 2G Ex px II T6 Gb
- II (1) G (Ex op is T4 Ga) II C

MATRIX-MF ex
The MATRIX-MF is also available as an ex-proof, ATEX rated version, complying to the following standard:

- II 2G Ex px II T6 Gb
- II (1) G (Ex op is T4 Ga) II C

Sensor heads for non-contact analysis in ATEX zones
In combination with a MATRIX-F the Q412 sensor head is also available as an ex-proof, ATEX certified version. It complies with the following standards for ATEX zones:

- II 2D Ex d A21 IP 6X T85°C
- II 21H G Ex d (op is T6) II CT78

With our ATEX certified systems Bruker offers you a customer-tailored solution for your specific needs in hazardous environments.

Process software solutions
All Bruker Optics’ software product for the process industry, including the OPUS (OPtical User Software) as well as CMET and the Reaction Monitoring software are fully validated to reliably meet the requirements for their intended use.

Certification
Bruker Optics’ products and services meet all quality standards, such as the ISO 9001 and ISO 13485, successfully audited by several pharmaceutical corporations and regarded as a fully approved hardware and software supplier. Each customer receives a full set of certificates for the instruments and accessories.
Typical application areas for FT-NIR analysis are:

- Monitoring and control from the end product to the production has been recognized by the chemical, petrochemical and pharmaceutical industry alike. This is achieved by real-time analysis of raw materials, intermediates and final products by FT-NIR spectroscopy.

Example: Chemical Industry

FT-NIR technology is widely used in a variety of chemical industries. The high information content in NIR spectra, measured in only a few seconds, allows for the simultaneous analysis of many different components and system parameters with high precision. Typical examples are monitoring the synthesis of basic chemicals, distillation and rectification processes as well as the end-point determination of chemical reactions.

Example: Oil Seed Processing

Depending on the oil type, the seeds/fruits are either cold-pressed or heat-treated before applying mechanical or solvent extraction. Monitoring moisture and oil levels of the material going into extraction as well as of the expeller cakes after pressing by online FT-NIR enables a quick and reliable indication of the efficiency of the process.

The extracted crude oil can then directly be analyzed for parameters like free fatty acids, phospholipids or waxes to find the optimal conditions for the following refining process. This will avoid costly rework of out-of-spec production batches.

Example: Monitoring of Butter Production

From an economic point of view, it is crucial to keep the water content as closely as possible to the statutory limit of 16 %, as water is of course much cheaper than the butter fat. To monitor the moisture as well as salt content with FT-NIR spectroscopy, a reflection probe or sensor head can be built into the butter stream using a standard flange.

Taking the high throughput and 24/7 operation into account, the return on investment of FT-NIR technology is normally reached within months.

Example: Petroleum Refining Processes

Refining gasoline from petroleum is another complex process where FT-NIR can be applied to optimize and control the different processing steps. The first key stage is the fractional distillation of the crude oil yielding the raw products light and heavy naphtha and diesel which are analyzed by FT-NIR. The final gasoline contains several additives for optimal performance.

Typical quality parameters monitored by FT-NIR are the density, Melt Flow Index, OH-number or free monomer content. With FT-NIR you can monitor the production of polymers such as polyethylene at the crucial processing steps. The current status of the polymerization can be determined as well as the product quality before and after the extrusion.

Another typical example is the online analysis of rubber for vinyl- and styrene content.

Example: Pharmaceutical Tablet Production

The production of tablets in the pharmaceutical industry involves mixing, granulation, drying, compression and coating. These different processing steps can be monitored online with a MATRIX-F in combination with a diffuse reflectance probe or a sensor head to ensure the product safety and minimize the risk of faulty batches.

Moreover, Bruker offers solutions for the automated online analysis of tablets for content uniformity.
Service and Support

Bruker Optics is staffed by expert scientists and engineers that have an in-depth knowledge of instrumentation and applications. Our product specialists are available to assist you with method development either remotely or in your lab. Our FT-IR and FT-NIR application scientists will assist you in the selection and use of sampling accessories, choice of optical components and software operation, as well as supporting you with method development. We offer customized instruction and support packages to fit your needs.

Bruker spectrometers are designed to provide years of dependable trouble-free operation, but should a problem occur a network of Bruker companies and representatives throughout the world are ready to promptly respond to your needs. Professional installations, comprehensive applications support as well as high standard of post-delivery service are commitments Bruker Optics makes to each of its customers.