Cane, Sugar and Ethanol

FT-NIR Analyzers for QC in the Lab and Production
FT-NIR Advantage
Near infrared spectroscopy provides stable and accurate analysis results of multiple components within seconds. It is a non-destructive method without any sample preparation and does not require the use of any solvents or reagents. Huge time and cost savings can be achieved by implementing FT-NIR:
- in the lab of a sugar mill to speed up and improve efficiency of laboratory operations
- at-line close to the production area where operators can carry out a fast and easy check of various sampling points in the process
- on-line for real time process control to obtain continuous information about changes and variations in composition of product streams.

State of the art equipment
Bruker Optics’ rugged FT-NIR analyzers for quality control in the lab or production area are easy to use, reliable and cost effective. Based on the same FT-NIR platform, users can choose the right analyzer for the job without having to compromise on precision and accuracy, ensuring data integrity and transferability today and in the future. Bruker Optics’ portfolio ranges from small footprint, touch screen operation analyzers to fully automated in-process systems for closed loop control.

FT-NIR Analyzers for the Sugar Industry

Bruker Optics Solutions for the Sugar Industry

A tight quality control is important throughout the complete sugar production chain to ensure efficient processes for optimized yield and quality. FT-NIR offers a fast and effective tool for controlling the critical parameters of raw materials, products and by-products in all production steps: sugar cane, bagasse, juices, molasses, massecuites, raw sugar, refined sugar, syrups and bioethanol.

Bruker - your partner for lab and process analysis
For more than 50 years, Bruker has been driven by the idea to always provide the best technological solution for each analytical task. Being one of the world’s leading analytical instrumentation companies, Bruker systems cover a broad spectrum of applications in all fields of research and development and are used in all industrial production processes for the purpose of ensuring quality and process reliability.

Bruker Optics is the world leading manufacturer of FT-NIR instruments for a wide range of industries including the agricultural sector and food manufacturing. Our analyzers combine an unrivalled flexibility and easy operation with state-of-the-art spectrometer technology. Software controlled optical modules, optimized sampling accessories for numerous applications and user friendly operator interfaces guarantee excellent results from day one.
For the refinery process, the quality of the raw sugar is important. Here the parameters Pol, Moisture, Reducing Sugars, Ash and Color (ICUMSA) are of major interest. During processing the intermediate products like molasses and other cane can be checked for optimizing the refinery process. Final products like crystal sugar and syrups can also be analyzed for quality control.

Sugar Refinery Process

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Bioethanol Process

Fermentation of molasses and other sugar process by-products is a straightforward and widely used production process for bioethanol. With FT-NIR each of the steps can be controlled starting from the raw materials over the fermentation step and distillation/rectification to the final bio-ethanol. Especially for the fermentation and the distillation FT-NIR in-line technology is an option which provides good opportunities for real time process control to optimize yield and processing time.

Bruker’s solutions along the Production Chain

1. Cane Reception
   - Sugar cane analysis for quality and payment purposes can be performed directly in the truck unloading area. The samples of incoming cane taken by a core sampler from the truck are shredded and fed into the automated analysis. Instead of pressing out the cane juice for payment analysis the cane can be directly analyzed with the CPS (Cane Presentation System) on the crucial parameters Pol, Brix, Moisture and Fiber.

2. Cane Milling
   - The control of the various steps in the milling process is vital to optimize yield and extracting efficiency. Like in the reception area, the shredded sugar cane can be analyzed directly with on-line FT-NIR for tracking consignments on variations in recoverable sugar content. Moreover all relevant juice streams can be analyzed to check and optimize the extraction performance.

3. Milling By-Products
   - The power of FT-NIR spectroscopy to analyze by-products rapidly has a huge impact on the overall efficiency of a sugar mill. The residual sugar content in bagasse, mud and the corresponding press juice are important indicators for the extraction yield. In addition, analyzing bagasse on moisture and fiber is of interest to optimize the by-product value for gaining maximum usage of heat, steam and more frequently the generated bioelectricity.

4. Sugar Milling Process
   - During the different steps of processing the cleared juice into raw sugar various intermediate products are generated, which need to be analyzed immediately for a close process monitoring and control. With a lab or at-line FT-NIR bench top analyzer all kinds juices, molasses, magma, seed, pan feeds and massecuites can be analyzed on Brix, Pol, Ash, Sucrose, Glucose and Fructose within less than a minute.

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• **Cane Milling: Improve Profitability**

Bruker Optics provides solutions based on FT-NIR for all sample types of interest during the entire cane milling process. The reliable and fast analysis of incoming cane is required for the fair payment of farmers. In the extraction process a continuous control of the different stages is important to optimize yield and extracting efficiency.

**Cane reception**
The samples of incoming cane taken by a core sampler from the trucks are shredded for further analysis. Instead of pressing out the cane juice for payment analysis, the cane can be directly analyzed with the CPS (Cane Presentation System) which allows an analysis of 3 to 15 kg in one single run. The disintegrated cane is automatically settled on a small conveyor belt and passes the NIR sensor head which analyzes the parameters Brix in Juice, POL, Fiber and Moisture simultaneously in less than a minute.

**Online cane analysis**
With FT-NIR process technology the cane can be analyzed on-line directly in the mill for tracking consignments. The fiber optic based sensor head can be installed in harsh environment while the NIR spectrometer with up to six channels is located several tens of meters away. The head can be placed anywhere with good sample access at a distance of 10 cm. An optimal sample interface is obtained using a welded in adapter with a sapphire window towards the sample which is not affected by the soil and sand content of the cane.

*Note:* The on-line analysis of milled cane is subject to a patent applied in a limited number of countries.

**Analysis of juices**
FT-NIR provides different options to analyze different juices (first and last cane juice, clarified juice...) either directly or diluted depending on the requested analysis time and accuracy. The fast and easy analysis on Brix, POL, Dry Solids, Sucrose, Glucose and Fructose can be performed at-line in the milling area or in the laboratory. Moreover an in-line solution with up to six fiber optic probes can be installed for the real time monitoring of various juice streams.

**Cane milling by-products**
The milling by-products, especially Bagasse are an important fuel for the mill boilers to generate steam and electricity. By controlling the bagasse, the mud and the filtrate juice by NIR it is secured that sugar losses are minimized and that the by-products are forwarded with the designated properties:

- Bagasse on Moisture, Pol and Brix
- Mud or filter cake on moisture, Pol and nutrient content
- Mud filtrate juice on Pol and Brix

**Products:**
- Sugar Cane
- Bagasse
- Filter Cake (Mud)
- Cane Juice
- Filter Juice
- Clarified Juice

**Parameters:**
- Brix
- Brix in Juice
- Pol
- Residual Sugars
- Purity
- Fiber
- Moisture/Water
- Ash

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**CPS (Cane Presentation System) for the automated disintegration and FT-NIR analysis of shredded cane**

**Truck with harvested sugarcane waiting to be sampled at a core lab**
**Sugar Milling: Process Control**

The production of raw sugar is a process with multiple steps and many different intermediate sugar factory products. The control of each step is highly important to ensure quality and increase efficiency with regard to optimal yield and reducing power requirements by optimized usage of electricity, heat and steam.

Continuous control at each process step
FT-NIR allows for a fast and easy analysis of any sample in the sugar milling process regardless if it is
- a clear liquid, e.g. juice
- almost solid crystallized sample like massecuites
- the final raw sugar.

All relevant parameters for process control can be derived in seconds: Brix, Pol, Color and content of Ash, Sucrose, Glucose and Fructose.

Depending on suitable procedures for sampling and sample presentations and the requirements for time, location and accuracy of the analysis there are various approaches for implementing FT-NIR as a routine at-line or lab analysis procedure. Moreover on-line analysis can be implemented for a closed loop control.

Direct analysis of factory product samples
For factory products, the sample can simply be filled in a disposable vial. For a juice or low viscous molasses a transflection mirror is inserted to enhance reflectivity. The vial is placed on the instrument and the analysis is finished in 5 seconds. Depending on the setup and accuracy requirements the sample can be temperature stabilized before the measurement.

This straightforward approach for analysing different samples with no sample preparation e.g. at-line shows a reasonable and often sufficient accuracy.

Analysis of factory product samples after dilution
A higher accuracy and precision can be achieved by diluting the samples and performing the analysis on a liquid sample. The liquid is pumped in a flow cell in the sample compartment of a MPA II FT-NIR analyzer. The transmission measurement and the temperature control in the heatable sample holder ensure a higher performance with optimized accuracy and reproducibility.

Analysis of raw sugar
For the analysis, the raw sugar is simply filled into a sample cup with a quartz window on the bottom. The cup is placed on the instrument and rotated during the analysis for averaging.

**Products:**
- Clear Juice
- Filtrate
- Thin Juice
- Molasses A, B, C
- Massecuites A, B, C
- Seed A, B, C
- Magma A, B, C
- Remelt
- Pan Feed
- Syrup
- Raw Sugar

**Parameters:**
- Brix
- Pol
- Dry Solids
- Moisture
- Ash
- Sucrose
- Glucose
- Fructose
- Reducing Sugars
- Purity
- Dextran
- Color
Sugar refining is a multi-step process with large control demands that lead to a huge optimization potential. FT-NIR allows for fast and easy analysis of all materials of interest in the lab, at-line or on-line providing accurate and timely information according to the demands of the production environment.

**Control of (incoming) raw sugar**
Sugar refineries are not necessarily directly linked to a sugar mill and therefore raw sugar is often purchased from third parties as an incoming raw material. The world market for raw sugar is extremely competitive and the quality of raw sugar has a major impact on refining costs. Therefore a tight quality control on Pol, reducing sugars (Glucose and Fructose), Moisture, Ash and Color is important and can be easily realized with FT-NIR spectroscopy. For the analysis, the sugar is filled into a sample cup with a quartz window and placed on the instrument.

**Process control**
Minor adjustments of the refining process can lead to better quality and higher yield of the finished sugars. During the production of white sugar, a fast multi-component analysis of the intermediate products is possible with FT-NIR spectroscopy. This can be achieved not only by taking samples and analyzing at-line, but also directly in the process allowing real time monitoring and control.

**Quality assurance of refinery products**
Some refinery products are liquids and contain not only sucrose but also other types of sugars. By inverting a portion using an enzyme or acid hydrolysis into glucose and fructose, characteristic chemical and physical properties are obtained, i.e. the inverted sugars prevent crystallization of the liquid products. Liquid products can be analyzed in a 22mm vial with transfection mirror on Pol, Brix, reducing sugar and ash:
- Liquid sugars
- Golden Syrup
- Treacle
- Edible Molasses
Finally the fermentation broth, the crude vine, is centrifuged and the vine is going to the distillation step. Both can be analyzed to make sure that an optimal feed is going to the distillation columns. Ethanol distillation and rectification
Distillation columns are monitored already for decades by NIR and the ethanol distillation is an easy task. Nevertheless the ethanol content in the various stages including the dehydration, needs to be analyzed with high accuracy, ideal for the use of FT-NIR spectroscopy. Independent of the analysis being done in the lab, at-line or in-line, the ethanol content in hydrous and anhydrous ethanol can be analyzed with an accuracy of about 0.1%, helping to maximize the yield of the process.

**Bioethanol Plant: Increase Productivity**
Fermentation of biomass is one important source for renewable energy and a tight process control of several industrial, chemical and microbiological parameters is required for optimal ethanol conversion rates. FT-NIR allows a fast and efficient control of the sensitive fermentation process as well as the other steps before and after.

**Fast Analysis of molasses and fermentation feed**
For fermentation the sucrose content in molasses and other sugar products is the most important parameter which is easily available by FT-NIR. The actual quality and composition of raw materials which may vary during a fermentation batch or continuous process setup is most important for a smooth and continuous productive fermentation. Here a fast analysis of material in delivering trucks, storage tanks or even in-line in feeding pipes has a direct impact in yield, securing of a smooth fermentation development and time efficiency.

**Direct in-line control of fermentation processes**
Bruker Optics offers comprehensive solutions for in-line analysis of fermentation processes. The MATRIX process analyzer series consists of FT-IR and FT-NIR instruments allowing the connection of various fiber optic coupled probes for the installation directly into the fermenters. Several measurement points can be covered by one instrument with an optical multiplexer. The unique FT-NIR transflection probe allows the analysis at any stage of the fermentation from the fairly transparent broth in the beginning to high biomass containing broth at the end of the process.
Fed-batch or continuous processes can be monitored by analyzing the most relevant parameters like Brix, individual sugars, ethanol, biomass and glycerol every minute. This allows the optimization of process by adjusting the Sucrose/Yeast ratio and controlling the ethanol content to not exceed a certain limit. This tight control increases productivity at reduced laboratory cost.

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**State-of-the-Art Technology with easy handling**

The Bruker Optics FT-NIR technology incorporates state-of-the-art optics for outstanding performance and stability. The heart of the instrument is Bruker’s permanently aligned RockSolid interferometer with cube corner mirrors, ensuring exceptional stability and reliability even in harsh environments, making it ideal for the laboratory as well as the factory floor.

Moreover, the instrument maintains the wavelength accuracy over time - a precondition for a successful calibration transfer.

All analyzers are designed to be easily maintained by the user, and to minimize downtime and maintenance costs. Consumables such as the light source are pre-aligned modules which can be easily and quickly changed by the user.

**Technology**

**Software**

**OPUS - Optics User Software**

Bruker Optics’ OPUS is an easy-to-use and powerful all-in-one spectroscopy software package. It includes the most comprehensive collection of data acquisition, processing, and evaluation functions and can be completely configured to meet your needs including extended user management and access features.

For method setup there are three main functions for:

- Calibration development for quantification of components and properties
- Library setup for identification of raw materials
- Conformity test for quality control

**Multi Evaluation**

Using the unique OPUS Multi Evaluation (ME) function, users can set up hierarchical methods to automate different evaluation and decision steps or to perform additional calculations. With ME, an identification step can be followed by a quantification step and a conformity test; or a quantification step can be followed by an additional quantification step depending on the results of the first step. The results are displayed, and customizable reports are stored and printed.

**In the Laboratory**

The OPUS/LAB package is an intuitive and easy-to-use software interface for routine analysis tasks. It can be used by routine operators who can quickly be trained to perform analyses. The operator just selects the product to be analyzed and enters the sample ID and optional sample information. The results are displayed on screen and stored in PDF and log files readable by LIMS.

**In the Process**

OPUS/PROCESS is a software package used to easily set up scenarios for automated process control and visual display of results. The scenarios can be configured with many optional settings for cyclic measurements or analysis triggered by process control systems. Triggers and results can be exchanged with PCS using Fieldbus, Modbus, 4-20mA connections or OPC.

**Data Security**

OPUS ensures the safety and integrity of your data.

- No loss of data or overwriting of raw data
- Fully GMP/GLP compatible, 21 CFR Part 11 conform
- Automatically generated data history (audit trail)
- All relevant data (measurement parameters, manipulations, evaluation results, reports, etc.) are stored in one data file

**Spectrometer Diagnostics**

Only a permanently monitored spectrometer can ensure the acquisition of reliable data. OPUS includes:

- Permanent online diagnostics
- Real-time display of instrument status
- Instrument status reports
- Integrated automatic instrument tests (OQ, PQ)

Example scheme of a Multi Evaluation method with Hierarchical Identification followed by Conformity Testing or Quantitative Evaluations.
Service and Support

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

Bruker Optics spectrometers are designed to provide years of trouble-free operation, but should a problem occur, a large 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.