Edible Oils and Fats

- FT-NIR Analyzers for QC in the Lab and Production

Innovation with Integrity

FT-NIR

Bruker Optics Solutions for the Edible Oil Industry

Near Infrared Spectroscopy has been a well-established technique in the agricultural sector for decades and is today an important element of quality control in the food industry. Modern multi-purpose FT-NIR spectrometers can analyze both, liquid and solid samples and are the ideal tool for the non-destructive and rapid analysis of oilseeds and finished oils throughout the entire manufacturing process.

FT-NIR Analyzers for the Edible Oil Industry

FT-NIR Advantage

The FT-NIR technology offers a lot of advantages over classical wet-chemical and chromatographic analyses. It is quick, cost-effective and safe, since no hazardous chemicals are used. It simply measures the absorption of near-infrared light of the sample at different wavelengths. The recorded NIR spectrum is characterized by overtones and combinations of the fundamental molecular vibrations of molecules containing C-H, N-H or O-H groups, making NIR spectroscopy first choice for the analysis of organic materials like oilseeds and edible oils.

The key benefits of FT-NIR spectroscopy are:

- no sample preparation, no waste
- no special skills required
- avoid typical operator errors of classical lab analyses
- analyze multiple component in less than one minute
- suitable for solid and liquid samples

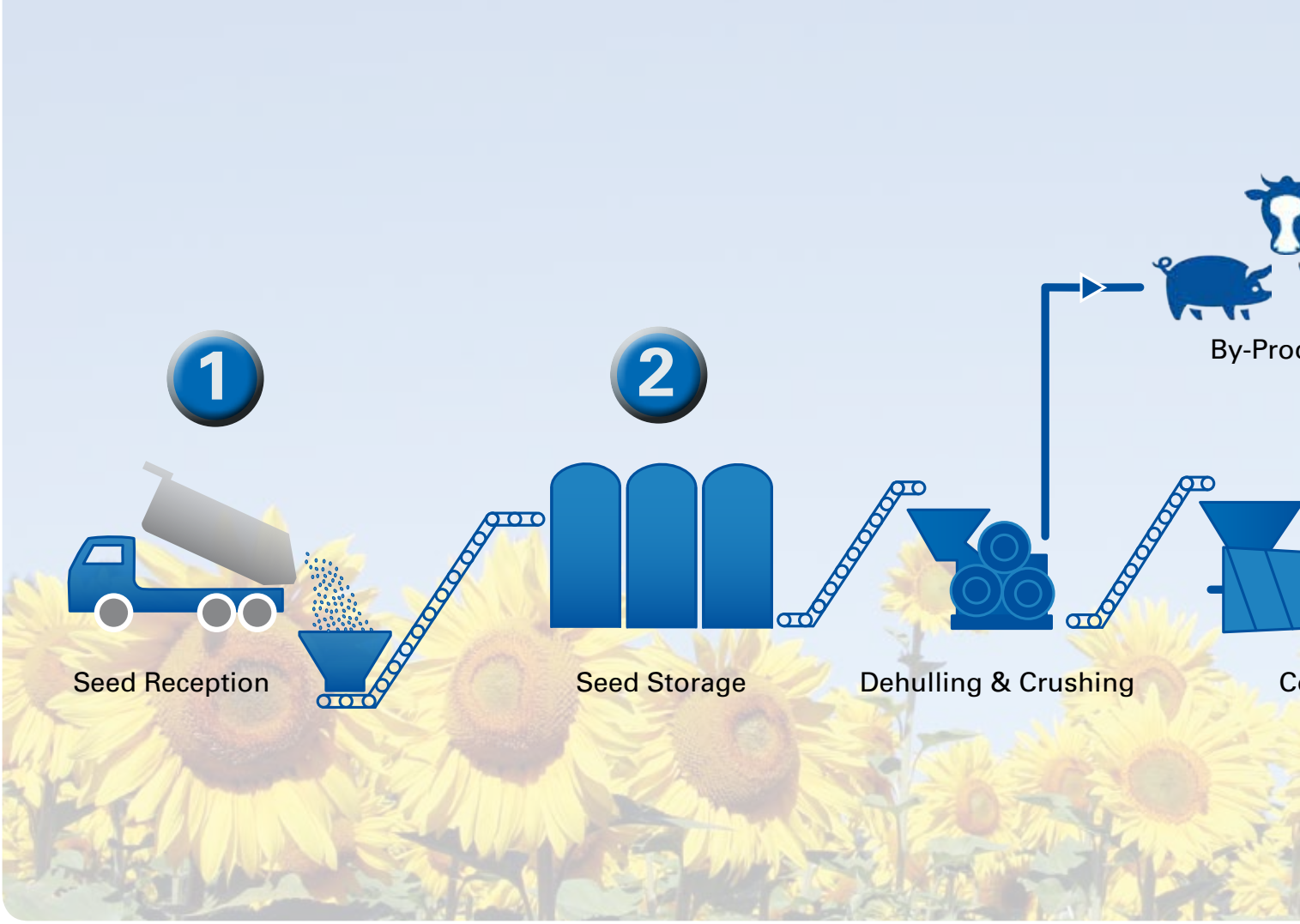
Being able to measure more samples in a shorter time will help the producer to constantly assess the quality of the goods along the production chain - from checking the incoming raw materials up to quality testing the finished product.

State of the art equipment

Bruker Optics rugged FT-NIR analyzers for quality control in the lab or production area are easy to use, rugged and reliable. 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 operated analyzers to fully automated in-process systems for closed loop control.

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. 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 unrivaled 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.



Bruker's Solutions along the Production Chain

- FT-NIR Analyzers for Quality and Process Control in the Lab and Production

Step 1

Seed Reception

Analyzing the incoming oilseeds directly at the arrival is crucial for quality control and fair payments alike. The quality of the seed also determines the way it needs to be handled or stored. In many companies the samples are still sent to outside laboratories and it can take days to receive the analysis results. During this time, the received goods need to be withheld.

FT-NIR offers a rapid solution for a reliable analysis, e.g. directly at the reception bay, so that a tight quality control of the oilseeds can be carried out before the material is discharged.

Step 2

Storage

Oilseeds that will be stored over a long period need to be kept at a defined moisture content that does not encourage the growth of bacteria, fungi or molds. This will make the seeds and thus the oil inevitably unfit for human consumption, leading to substantial losses in profit.

A regular moisure analysis of the oilseeds by FT-NIR spectroscopy can help to monitor not only the storage conditions, but also drying processes for optimum seed quality at the point of oil processing.

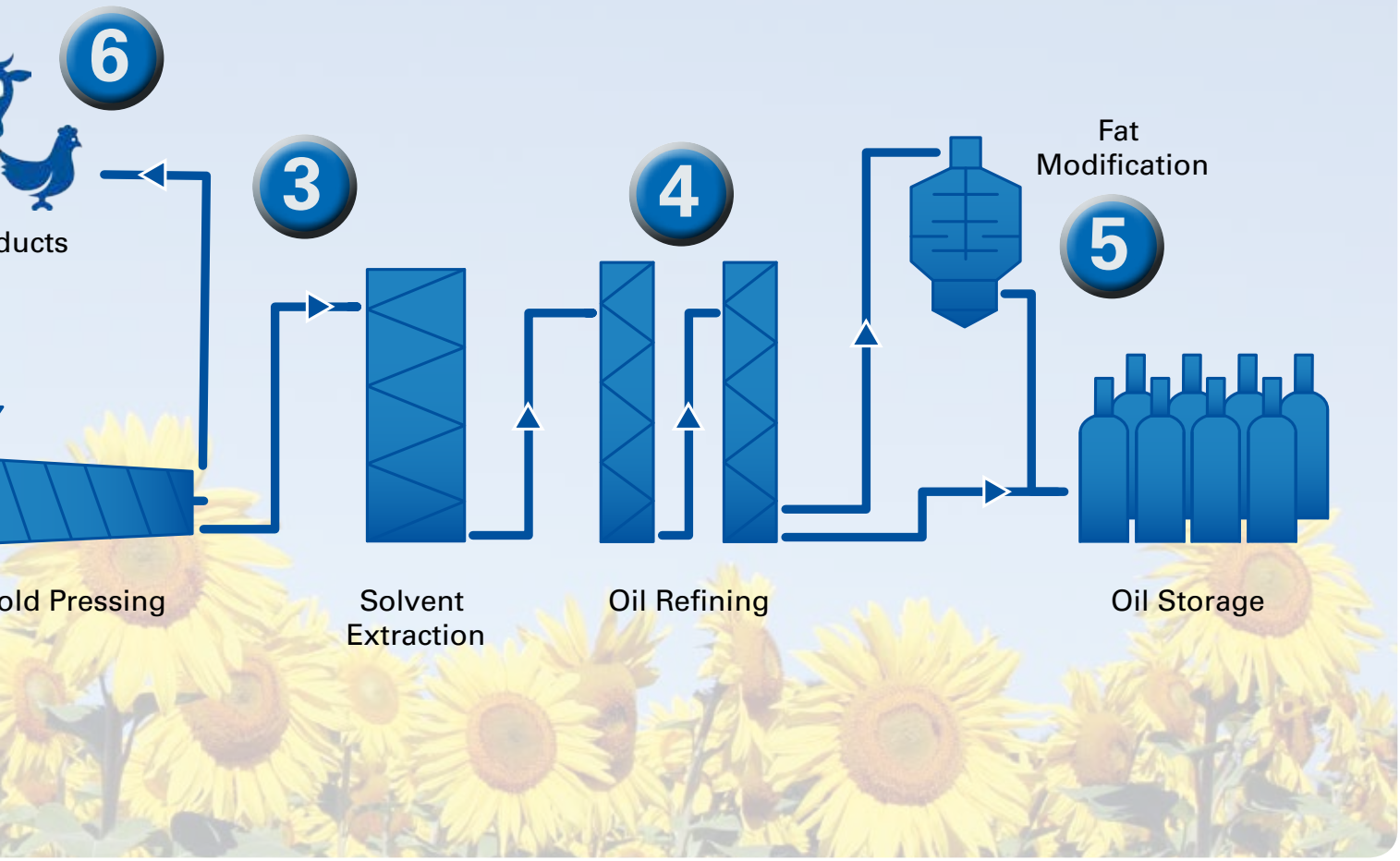
Step 3

Oil Extraction

Cleaning, drying, de-hulling and flaking are essential steps for increasing the oil yield of the seeds. 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 by FT-NIR gives a quick and reliable indication of the efficiency of the process.

The extracted crude oil can be analyzed for parameters like free fatty acids, phospholipids or waxes to find the optimal conditions for the following refining process.



Step 4

Oil Refining

The oil refining process removes undesirable substances from the oil, e.g. free fatty acids or colors, but may also eliminate valuable minor components like antioxidants of vitamins. Therefore a close monitoring of refining as well as testing the finished product process is essential to produce high quality oils.

Bruker offers FT-NIR solutions not only for the laboratory, but also for real-time on-line process measurements, which eliminate the delay in receiving analysis results and avoid costly rework of out of spec products.

Step 5

Fat Modification

Being a natural product, only a very limited number of different oils and fats are available on a commercially relevant scale, and those often do not meet the physical or nutritional properties required for use in food production.

Technologies like fractionation, interesterification and hydrogenation are mainly applied to change the fatty acid composition of the lipids in order to adapt them to the demands of the food industry.

FT-NIR allows the monitoring physical and chemical properties of the lipids including fatty acid profile, free fatty acids, trans fatty acids, iodine value or SFC content.

Step 6

By-Products

Many of the by-products which accrue during edible oil production are valuable raw materials for other industries.

The hulls remaining from the de-hulling process can for example be pelletized to add an excellent source of fiber to animal feed. The value of adding expeller cakes to feed products lies in the high nitrogen content.

With FT-NIR, parameters like moisture, oil, protein, fiber and ash content can be analyzed in less than a minute, helping to assess the market price of the material.

• Oil Seeds: Right from the Beginning

The analysis of oilseeds plays a major role in ensuring the best quality of food as well as of agricultural products. Near Infrared spectroscopy offers solutions for oil producers as well as for breeders.

Seed reception

The value of the oilseed harvest is not only determined by the oil and moisture content. Also the amount of free fatty acids and the fatty acid composition, e.g. the amount of oleic, linoleic acid or erucic acid determines the payment of the crops. This makes a close control of the incoming seeds important for oil producers and farmers alike.

With FT-NIR spectroscopy, the composition of ground or unground oilseeds can be analyzed in less than a minute without any sample preparation. The sample is simply filled in a cup with quartz glass bottom. Several lots of the consignment can be analyzed in short time to give an optimal overview of the quality of the intake at the point of delivery.

Plant breeding

Looking at the plant breeding process, at each stage the breeder needs to choose the best seeds for propagation to the next generation. In the past the breeder has had to sacrifice some of these valuable seeds in order to test for the traits of interest. NIR has become increasingly popular in the plant breeding-sector, as it is a non-destructive method of analysis, capable of measuring many of the important traits. It allows the breeder to analyze seeds quickly and to cost effectively determine the best and successfully grow them on.

Depending on the use of oilseeds, different parameters are of interest: For sunflower seeds, the amount of oleic acid is an important parameter. Over the last decade, there has been an increased interest in breeding sunflower seeds with high oleic acid content. This type of breed is high in mono-saturates and

MPA II Multi Purpose Analyzer for testing edible oils as well as oilseeds

Oil Seeds

Products:
- Sunflower Seeds
- Rapeseed
- Canola
- Corn/Maize
- Soybeans
- Flax/Linseed
- Sesame Seeds
- and many more...

Parameters:
- Moisture
- Oil
- Protein
- Fiber
- Ash
- Glucosinolates
- Free Fatty Acids
- C18:0
- C18:1
- C18:2
- C18:3
- C22:1

therefore associated with a healthy diet and improved frying properties. FT-NIR can determine the content of oleic and linoleic acid directly inside the seed. Rape seed (canola) breeders can obtain valuable additional information like fatty acids, including erucic acid as well as the glucosinolate content, crucial parameters for the palatability of the finished product.

Single seed analysis

With FT-NIR even single seeds, from an intact soybean down to a single canola seed can be analyzed for various parameters. Customized sample holders ensure reproducible measurements for optimum results.

Animal nutrition

Not only the oilseeds, but also meals and hulls can be analyzed for the content on moisture, oil, protein, fiber ash and starch for the cost optimization of animal feed formulas. Moreover, the amino acid composition of oilseeds and meals can be monitored with FT-NIR spectroscopy to calculate the correct animal feed supplements.

Soybeans are not only valued for its oil, but also for its high protein content.

• Edible Oils: QC of finished Products

Oils and fats are recognized as essential nutrients of our daily diet and contribute significantly to the regulation of different body functions. Numerous parameters are used to assess their quality. Bruker's dedicated FT-NIR solutions enable a rapid analysis of edible oils and fats.

Continuous control at each process step

Edible fats and oils are an integral part of the human diet, being consumed in a variety of forms. Different parameters are used to assess the quality of edible fats and oils, including iodine value (IV), free fatty acids (FFA), trans fatty acids (TFA), anisidine value (AV), and various other parameters.

The traditional analyses are generally carried out using standardized chemical and physical methods approved by the American Oil Chemist Society (AOCS) and/or the German Society for Fat Science (DGF).

However, these methods are normally designed for analysis of only one specific parameter and tend to be time-consuming, e.g. GC analysis. Moreover, they often require hazardous solvents and reagents, which create a potential health risk and add disposal costs.

Edible oil producers and food processors are seeking fast and non-destructive way to analyze fats and oils for process and quality control purposes.

Rapid quality control for edible oils can be achieved by Bruker's dedicated FT-NIR solutions. The analysis is quick, cost-effective and safe to use, even for untrained staff, since no sample preparation is required. The oil is simply filled into an 8mm glass vial and measured in the sample compartment of the spectrometer.

Fatty acid composition

An important quality parameter of edible oils and fats is the fatty acid profile (C16:0, C18:0, C18:1 etc.), since it is a measure of the amounts of individual fatty acids in an oil or fat. Coconut oil has a substantially different fatty acid profile compared to a sunflower oil. The relation of

High quality edible oils are a healthy addition to the human diet.

Edible Fats & Oils

Products:
- Sunflower Oil
- Rapeseed Oil
- Canola Oil
- Corn Oil
- Soybean Oil
- Coconut Oil
- Palm Oil
- Fish Oil
- Tallow
- Lard
- and many more...

Parameters:
- Free Fatty Acids
- Trans Fatty Acids
- Iodine Value
- Peroxide Value
- Anisidine Value
- Fatty Acid Profile
- Triglyceride Profile
- SFC Screening
- Saturation
- Color

the different fatty acids does not only determine type of the oil and its nutritional value, but also influences the physical properties its stability.

Oleic acid is for example is very desirable from a nutritional point of view. Palmitic acid, being completely saturated is less preferred. Linolenic acid, containing three double bonds, is the most chemically reactive and therefore inferior from the view of stability. But not only the degree of saturation, but also differences in the positional distribution of the fatty acid on the glycerol backbone influences the characteristics of the oil.

FT-NIR spectroscopy can not only quantify the major fatty acids, but also assess the triacylglycerol (TAG) structure of the oils. Moreover the total content of saturated, mono-unsaturated and polyunsaturated fats can be determined with one quick measurement.

Compositional analysis of rendering products

Approximately 40-50% of the weight of any slaughtered animal is not fit for human consumption. This material is mostly transformed by the rendering industry into highly nutritional ingredients, e.g. for the animal feed production. These include sources of energy like tallow or lard as well as protein rich material like meat and bone meal or poultry meal. FT-NIR is well established for the analysis of edible fats of animal origin where parameters like the total fatty acid content as well as free fatty acids and iodine value can be analyzed simultaneously. For animal meals the classical constituents like moisture, fat, protein, fiber and ash can be determined as well as more specialized parameters like energy values or amino acid profiles.

Measurement of edible oils in an 8mm vial in the sample compartment of the TANGO FT-NIR spectrometer.

• Olive Oil: Liquid Gold

The quality of extra virgin olive oil vastly depends on the olive itself, as it is the only cooking oil that is made by cold-pressing the fruit without the use of chemicals and industrial refining. This makes it expensive and thus prone for adulteration. FT-NIR spectroscopy offers a valuable tool for monitoring the complete production process and detect low-quality oils.

Quality control of incoming olives

The vast majority of olives grown all over the world are used for the manufacture of olive oil. The value of an olive crop is mostly determined by the oil content. Depending on the time of the harvest and the olive variety, the olive oil content may vary between 10 – 30%. To determine the exact oil content is essential for the farmers and the industry alike to estimate the value of a harvest.

Traditional wet chemical methods, e.g. Soxhlet analysis become increasingly unacceptable by the industry since they require large amount of solvents, creating health and safety risks as well as environmental issues. Furthermore, the results are operator dependent and the procedure is slow compared to FT-NIR spectroscopy, which delivers results in less than a minute. With the same measurement, the acidity inside the olives can be assessed at the point of delivery, a criterion for good manufacturing practice during the harvest and the storage of the olives.

Production process control

In addition to the fat content, the amount of water is also an important parameter for the optimization of the yield during the extraction process.

After the extraction steps, the remaining pomace can be analyzed to assess the oil content, which should be 2% or less. A substantially higher level of oil content indicates problems with the pressing process and thus loss of profit.

FT-NIR allows an extensive screening of the process since it is fast, allows screening of large sample numbers in real-time and is environmentally sustainable.

A careful harvest of the olives is the prerequisite for a good quality oil.

Olives & Olive Oil

Products:
- Oilve Oil
- Olives
- Olive Paste
- Pomace

Parameters:
- Oil
- Moisture
- Acidity
- K232
- K270
- 1,2-Diglyceride
- Pyropheophytin
- Peroxide Value
- Fatty Acid Profile

Olive oil analysis

An acidity value below 0.8% is the main criterion for the classification of the olive oil as“extra virgin”. Other quality parameters include the peroxide value, an indication for the rancidity of the oil as well as as well as the K-values (UV absorption) and many others.

Unlike wine, the quality of olive oil does not improve with age and sooner or later it will become rancid. The amount of 1,2-diglycerides as well as the pyro-pheophytin content in the oil reveals if a an olive oil was stored for too long or even adulterated with refined (olive) oils to obtain lower acidity values.

All these critical parameters can be tested with a 30 second FT-NIR measurement, enabling a thorough quality control along the production chain of the oil.

Testing on adulteration

A common problem not only for the olive oil industry is the adulteration of high priced olive oil with cheaper seed oils such as sunflower or hazelnut oil. Today’s public awareness of the health benefit of olive oil makes the adulteration economically attractive.

Chemically, most oils are very similar and a blend is difficult to identify with common quick tests like measuring refractive index. However the different oils vary in their fatty acid profile and FT-NIR spectroscopy can offer a valuable tool for determining other types of oil in olive oil down to a low percentage range.

Another issue regarding adulteration is to determine the geographic origin of the oils for certification purposes (RDO labeling). Combined with other techniques, FT-NIR can deliver valuable supplementary information.

Optimal storage conditions and frequent testing are essential for the quality of the olive oil.

• Good Oils for Great Taste: Frying Fats

In the recent years, frying food has been stigmatized due to health concerns. However, the quality of the frying fat heavily influences the quality of the finished product. This makes the importance of a thorough quality control very obvious – in fast food restaurants and huge frying operations alike.

Monitoring frying oil degradation with FT-NIR

Frying is today a well-established, cost effective and fast method of food preparation. However, frying fats and oils, used continuously and repeatedly at high temperatures, are subject to a series of degradation processes.

Foods produced in large scale frying operations are heavily impacted by the condition of the frying oil. More than 90% of the oil contained in the food before frying is exchanged by the frying medium. Color, texture and flavor of fried foods are influenced by the frying oil, and moreover, the oil itself represents a significant portion of the production cost of the fried food.

Laboratory methods used to assess quality factors in frying oils include determination of free fatty acids (FFA), anisidine value, total polar compounds and polymerized triglycerides. Each of these tests are performed as individual analyses and employ reagents that require proper disposal, training of laboratory personnel, and time to perform each of the tests.

Accuracy, precision and speed of the FT-NIR method make it an ideal choice to monitor the condition of frying oils and enable plant personnel to make informed decisions to control costs as well as sensory qualities for the products they produce.

This was acknowledged by the German Society for Fat Science who issued the Standard Method „*FT-NIR Spectroscopy: Screening analysis of used frying fats and oils for rapid determination of polar compounds, polymerized triacylglycerols, acid value and Anisidine value [DGF C VI 21a (13)]*" in September 2013.

Frying is today a well-established, cost effective and fast method of food preparation.

Frying Fats

Products:
- Frying Oil
- Frying Fat

Parameters:
- Acid Value
- Anisidine Value
- Total Polar Components
- Polymerized Triacylglycerols

Other parameters to check quality and nutrition value, e.g. iodine value, content of saturated, mono--and poly-unsaturated fatty acids, as well as the trans fatty acid (TFA) content, can be evaluated from the same FT-NIR measurement.

Online process control

FT-NIR spectroscopy is however not restricted to the laboratory. There is a strong trend to take spectroscopy on the line rather than taking the sample to the lab. Not only quality and safety issues, but also economic considerations are the motivation to develop methods for the real time process analysis.

Online measurements are suitable for solid and liquid samples alike. There are different contact and non-contact sensors available which can be implemented into tanks, bypasses and even over conveyor belts. Up to six sensors can be multiplexed by a single MATRIX-F spectrometer.

One key application is the constant analysis of the oil during industrial deep-fat frying operations. With FT-NIR not only the frying oil quality can be directly assessed, but it is also possible to gain insight into those variables that can influence the frying process. Fiber optic transmission probes withstand heat up to 260°C making them ideal for batch frying or continuous frying operations alike.

MATRIX-F spectrometer for the in-process analysis of liquid and solid samples.

Technology

State-of-the-art technology for RockSolid results

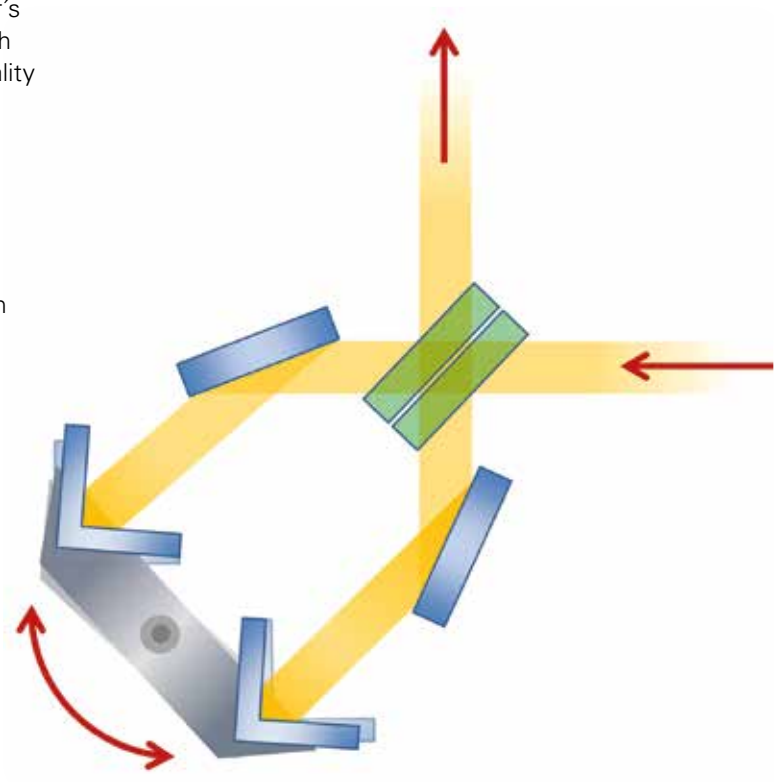
The Bruker Optics FT-NIR technology incorporates state-of-the-art optics for outstanding performance and stability. The heart of the instrument is Bruker´s permanently aligned RockSolid interferometer with cube corner mirrors, providing consistent high quality results, less downtime and highest stability.

Unlike flat mirrors, cube corners are practically immune to mirror tilt (i.e. angular movement of the mirror). This is an important consideration in FT technology since for the modulation the light returning to the beam splitter must be precisely recombined for interference to avoid a reduction in stability, resolution, and spectral quality.

The RockSolid interferometer design therefore leads to a superior resistance to vibration and thermal effects, ensuring exceptional robustness 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.



Bruker's well-proven RockSolid interferometer with Cube Corner Mirrors.

Software

OPUS - Optics User Software

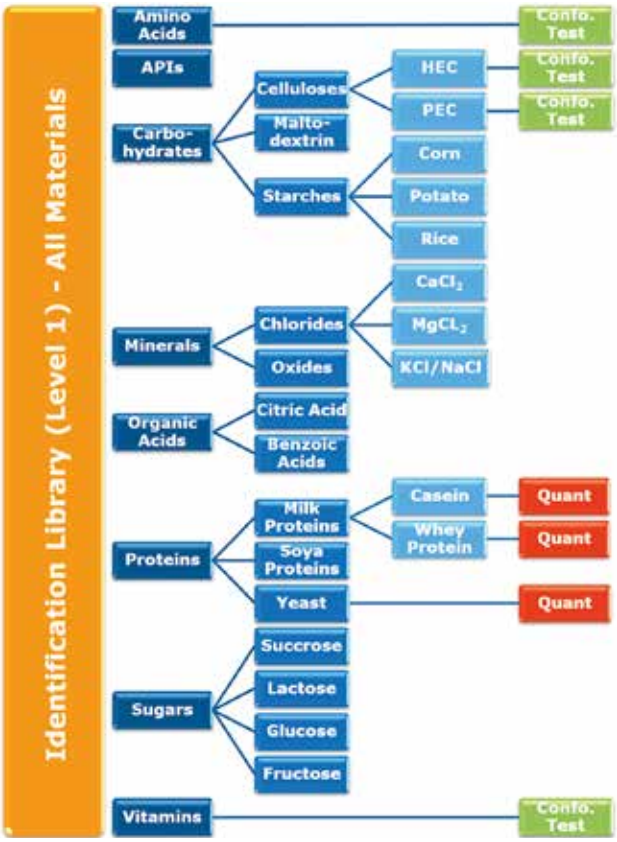
Bruker Optics' OPUS is an easy-to-use and a powerful all-in-one spectroscopy software package. It includes the most comprehensive collection of data acquisition, processing, and evaluation functions and can be completely configured to meet your needs including extended user management and access features.

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.



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

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 visualized 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 Profibus DP, 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)

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.

Technologies used are protected by one or more of the following patents:
US 7034944. Additional patents pending.

Bruker Optics is ISO 9001 and ISO 13485 certified.

Laser class 1 product.

www.bruker.com/optics

Bruker Optics Inc.
Billerica, MA · USA
Phone +1 (978) 439-9899
Fax +1 (978) 663-9177
info.bopt.us@bruker.com

Bruker Optik GmbH
Ettlingen · Germany
Phone +49 (7243) 504-2000
Fax +49 (7243) 504-2050
info.bopt.de@bruker.com

Bruker Shanghai Ltd.
Shanghai · China
Phone +86 21 51720-800
Fax +86 21 51720-899
info.bopt.cn@bruker.com