Food Process Analytics
The Champion's Guidebook

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1: Incoming Goods Inspection

Contents

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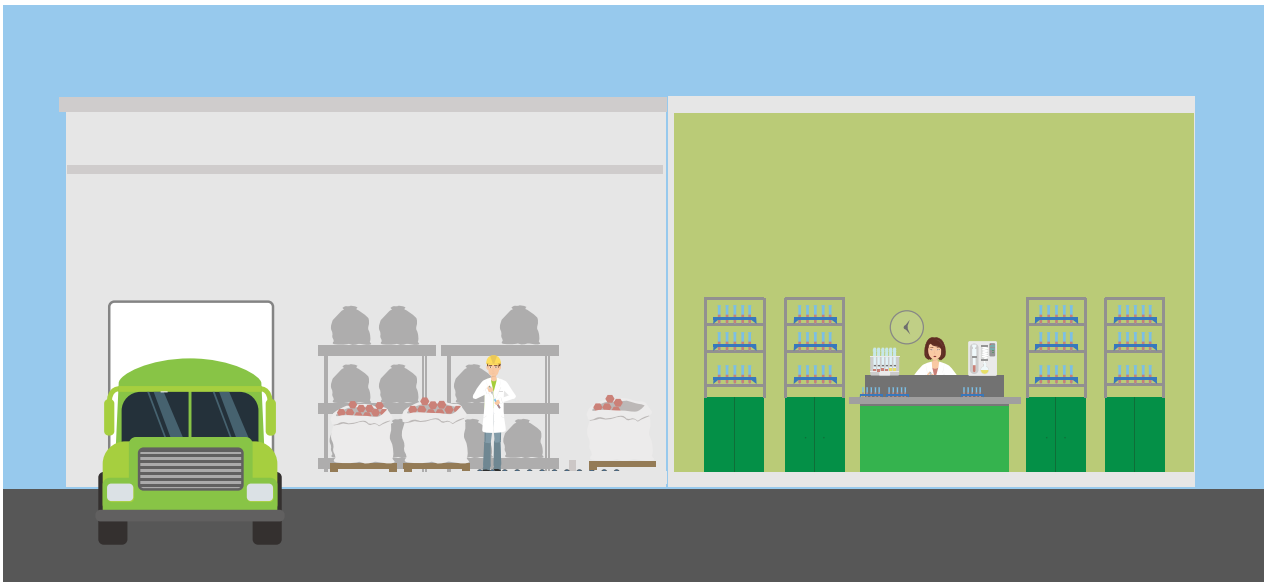
The food manufacturing process chain

The food and beverage manufacturing process chain comprises typically of five steps starting with goods receiving, incoming goods inspection, going over to production, quality control and finishing with logistics and distribution. Along this chain, the quality of raw materials, intermediates and the final products are checked regularly. Samples are taken at specific points throughout incoming goods inspection and further analyzed for relevant quality parameters.



Incoming goods inspection overview

Food companies usually establish inspection procedures
for incoming goods to determine and evaluate the purchased goods. The inspection assures the compliance to the quality levels that are required by legal bodies and producers or are guaranteed by the suppliers. Hence, the inspection of incoming good is the main focus of the first Volume of the food process analysis guide. It puts emphasis on the ambiguous requirements of incoming goods inspection:

· Fast measurements and short time to results
· Reliable and acceptable results
· Fulfilling of prerequisites and requirements

The time factor

Time is increasingly becoming the most relevant factor in incoming goods inspection. The waiting time for the results of the inspection needs to be decreased to a minimum.

One customer states: “Ingredients are delivered just-in-time. There is no time for long analyses.”

The main concerns regarding time in incoming goods inspection are described below:

· Incoming goods need to be quickly processed further or stored properly in order to keep the quality taintless. Maintaining freshness, keeping the cold chain uninterrupted and keeping the goods contamination free are the main interests. Therefore, approval of the delivered materials cannot wait for a long time.
· The supplier expects a timely answer on acceptance or refusal of the delivered goods.
· Just-in-time production procedures are highly sensitive to any delays.
· Logistic chains from transportation, intermediate storage to local delivery are synchronized precisely.

Achieving reliable results for incoming goods inspection

The quality of the incoming goods directly influences manufacturing processes, quality of the final goods and very often the purchase price. However, reliable and safe analytical methods have to be applied to ensure the quality of incoming goods. These analytical methods have to be accepted by all involved stakeholders to avoid re-questioning of results. That is why reference methods are defined and used by various accredited standard bodies (e.g. ISO, AOAC, etc.). The entire inspection has to be documented carefully and completely. Applying these methods exclusively for the incoming goods inspection brings with it certain disadvantages. It requires a lot of labour time and resources and it generates financial costs. There are several actions that can be taken to make incoming goods inspection more efficient and they are described in the next chapters.

How to meet the inspection requirements

For the incoming goods inspection at material receiving, as well as in the laboratory it is important that inspection criteria can be met using the most timely and reliable technologies and processes. Besides an established inspection process and the well-prepared inspection planning, specific measures can be taken to facilitate and speed up the demanding task of incoming goods inspection:

Measure	Time factor	Reliable results
1 Increase the speed of incoming goods inspection and the time to results	Reduce time of measurements carried out in the laboratory Round the clock inspection by applying fast responding techniques (i.e. NIR) Complement standard reference methods with analytical screening methods (i.e. NIR or NIR-Online).	Reduce operator intervention and errors: · Process multiple samples simultaneously · Use an autosampler Use interchangeable sample racks
2 Apply suitable analytical methods	Select optimum methods for fast results (e.g. fast NIR vs. time consuming classical method)	· Apply reference methods for general acceptance of results · Select methods to match criteria of material certificates
3 Choose optimized analytical instruments	Use fast instruments (e.g. short conditioning time, short starting up period) Use easy to handle instruments	Avoid operation errors: · Use easy to operate instruments · Use instruments with user guidance · Use automated instruments · Use instruments with defined Standard Operating Procedures
4 Implement data flow automation from instrument to data storage system (reporting, filing)	Implement automatic reporting Avoid tedious manual data transcriptions to paper or by manual entering into the system Implement automised approval and release of inspected goods.	Achieve safe data status: · Prevent omissions · Avoid result transcription errors and permutation of samples Assure regular back up and archiving of the data

The incoming goods inspection process

Optimum analytical methods, instruments and automated work flows are one important aspect of the incoming goods inspection. However, the entire inspection process may include further circumstances such as the following:

- Government requests: Governments of many countries oblige companies to inspect their incoming goods and follow HACCP principles.
- Collaboration across the supply chain: When inspection plans are shared with the suppliers, they can integrate the plans in their respective inspection scheme already. Applying this process leads to a sharing of risks and efforts regarding incoming goods inspection. In addition, work time for such inspections is retained.
- Real-time analysis: Efficient and immediate analysis of the incoming goods leads to clear conclusions. Proactive measures can then be taken to prevent non-conformance of the produced goods with the required specifications. This avoids an unnecessary work load (i.e. rework of products or storage costs) and analyses, while time saving is considerable.

In addition to the decision of conformity, i.e. being in or out of specification, the basic purpose of incoming goods inspection is to ensure that the raw materials comply with the recipe requirements, that production steps can be undergone as foreseen, that the quality of the end product can be met and that optimal production processes can be carried out. Non-conformity of the incoming goods is detected before production or end customers are affected.

An optimal incoming goods inspection process is critical to manufacturers because it can help to make further production steps more efficient, faster and less wasteful.

Selected methods for incoming goods inspection

The following test methods for incoming goods inspection will be looked at more in detail in for this booklet:

- NIR analysis (simultaneous determination of various parameters)
- Kjeldahl analysis (reference method for protein content)
- Extraction (reference method for fat content)

NIR analysis is the clear example for a rapid and non-destructive analytical method whereas Kjeldahl and Extraction are very typical examples of standard reference methods. These methods are used to calibrate NIR instrumentation and to decide on the acceptance or refusal of incoming goods.

These methods are described in the following chapters. A short introduction to each method is given and selected application examples are presented. The methods are described in the following chapters. A short introduction and selected application examples are presented.

Why is NIR fast?

Near-infrared (NIR) analysis is a spectroscopic method that uses light to measure useful sample properties like fat, protein and moisture. Since NIR is an indirect measurement, it requires calibration using reference data provided by standard wet-chemistry methods. The main benefits of this technology are:

· Short measurement time (seconds)
· Minimal or no sample preparation
· Non-destructive measurements (i.e. sample-sparing)
· Simultaneous determination of various sample parameters
· Broad application base

Due to these qualities, the NIR method is a suitable tool for real-time analysis (RTA). The immediate quality and process control feedback provided by NIR, bypasses the typical QC laboratory hold-times preceding product release. By implementing a rapid measurement technology, out-of-specification starting materials that could result in costly process deviations or degraded finished product quality may be flagged as early as it reaches the loading dock. Moreover, the volume of incoming goods inspected can be greatly increased with fewer resources and less expense than traditional methods, overcoming some of the risks of randomized spot-checking. The application of on-line or in-line NIR sensors provides the added potential for continuous and automated quality measurements at various installation points along the production chain, spanning from raw material intake to finished product release.

A customer from a Swiss chocolate company comments that taking random samples cannot reveal all hidden defects. With the approach of using NIR analysis for continuous inspection of incoming goods it is possible to prevent or minimize the risk of unseen deficiencies.

Calibration is a prerequisite

Near-IR analysis is based on the response of molecular bonds within the sample to NIR radiation (i.e., low-energy light characterized by wavelengths typically ranging from 800 nm to 2500 nm). When NIR light interacts with a sample, the light is either absorbed or scattered. The resulting pattern or spectrum that emerges from these interactions is a direct result of the composition and physical properties of the sample. As mentioned, NIR is an indirect measurement, requiring calibration using reference data from standard wet-chemistry methods. Chemometrics is the mathematical procedure used to transform the paired reference data and complex multivariate NIR spectra into a practical measurement system.

The dataset from which a calibration is generated will greatly impact the measurement system's performance. As such, sample planning is a critical aspect of NIR method development. To achieve acceptable accuracy, precision and robustness, a calibration model must include samples representing all expected sources of sample and environmental variation. For example, the full range of composition (e.g. fat, protein, and moisture) and physical characteristics (e.g. particle size and temperature) that may be encountered during routine sampling should be included in calibration development. Seasonal variations, including temperature and humidity, as well as inter-operator variability, may be accounted for by sampling across the calendar year and using data collected by multiple users, respectively.

The accuracy of the standard reference method must also be considered. The standard error of an NIR method can approach that of the reference method when good sampling practices are employed. The figure below illustrates the general workflow for calibration development. Consider a product for which protein and fat composition are required. The reference data are produced by Kjeldahl and extraction, respectively. Those data are paired with the NIR spectrum of the same sample to generate calibration models. Those calibrations are then configured into to an application which can be employed to predict the composition (i.e., protein and fat) of future samples.

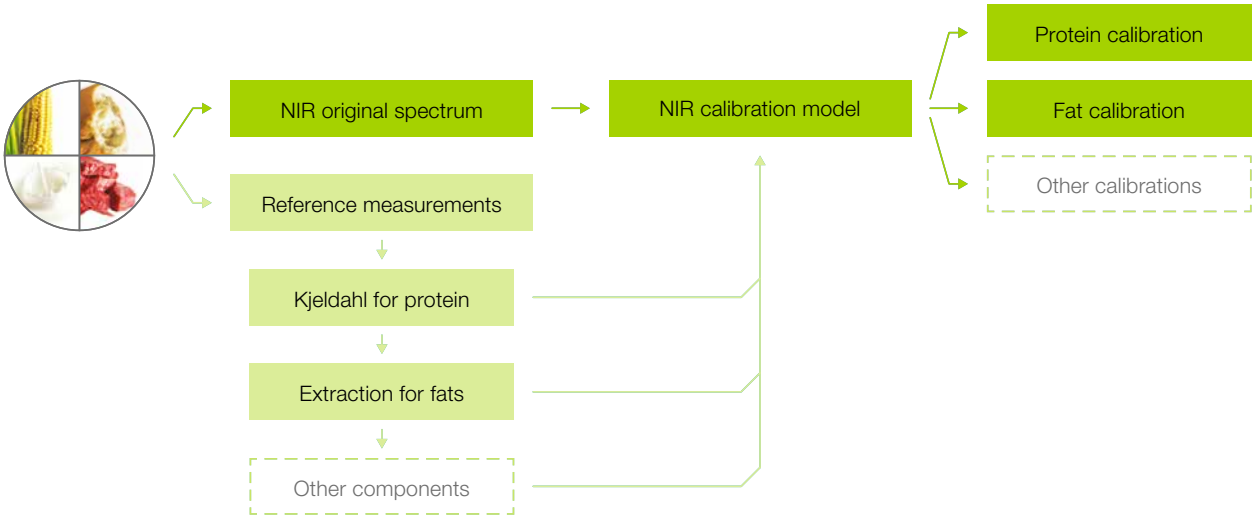


Figure 1: NIR analysis calibration

For typical applications, NIR calibrations may be pre-programmed into the instrument software or available from the instrument manufacturer or third party. Further optimization of these methods can be accomplished by addition of routine samples into existing calibration equations. Calibrations offered by BUCHI are summarized in the Application section of this booklet.

Ensure result quality

There are many calibration outputs which indicate performance. Standard errors or calibration and validation, bias and R2 calculations are among the many performance metrics that are calculated by standard NIR software.

The potential to implement calibrations developed by the instrument manufacturer comes with tremendous time-savings. When those calibrations don't meet expectations of precision or accuracy, improvements may be made by: (1) expanding the calibration dataset to include in-house routine samples, or (2) implementing slope and/or bias corrections to correct for systematic deviations in method performance.

Routine NIR analysis

While calibration itself may be resource-intensive, routine analysis is very fast and efficient. The simplicity of using an NIR method means that operators won't require special skills to achieve good measurement results. Depending on the sample type, measurements may be collected by immersion of a probe into a container, through plastic bags, glass, plastic sample cups or in cuvettes or vials. In the case of an on-line sensor, the NIR measurement will be triggered automatically and acquired as-is. In any case, the NIR measurement will produce a spectrum. That spectrum is then projected into the NIR calibration model(s) associated with the application (e.g. protein and fat), producing an NIR prediction of each property for the current sample, simultaneously. A quantitative or qualitative result is visualized by the operator and recorded by the software. Because NIR is non-destructive, the sample may be easily retained or used for further testing, as necessary.

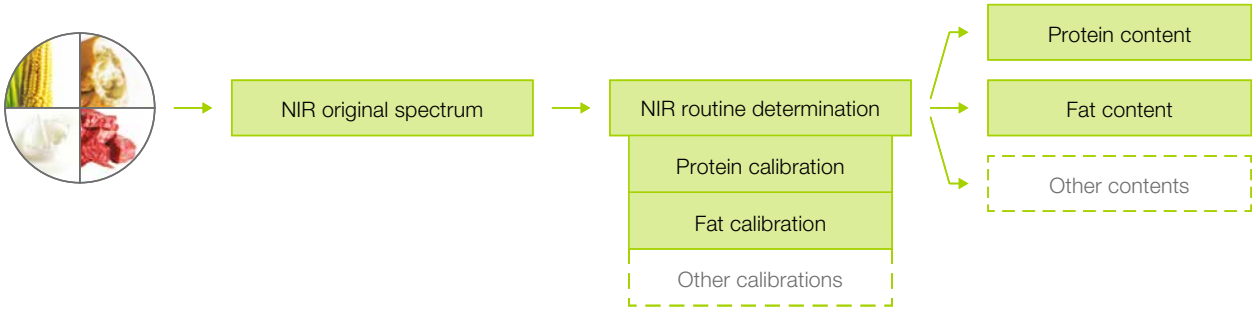


Figure 2: NIR routine analysis

Fit-for-purpose instrumentation

BUCHI NIR instruments cover the entire process line starting with off-line (laboratory), close to the production line (at-line), using the by-pass (in-line) or measuring the sample directly on the production line (online). Choosing the proper measurement method, sampling module and specific is an essential way to develop a robust NIR method for successful applications. Software solutions for chemometric calibration evaluation and electronic data handling complete the offering.

▶ Click on Products and Solutions on www.buchi.com

Reference methods and their usage

Reference methods provide results of accepted reliability such as precision, accuracy and linearity. The decision on conformance of the random samples taken at incoming goods inspection is based on these results. They are also used to calibrate alternative methods such as NIR spectroscopy.

The reference methods are typically performed in the laboratory. The two examples of protein and fat determination are explained in the following chapters.

Protein determination

The Kjeldahl method, an internationally accepted standard for the determination of nitrogen and protein content of food and feed materials, involves 6 steps from sample to result which you can see in Figure 3.

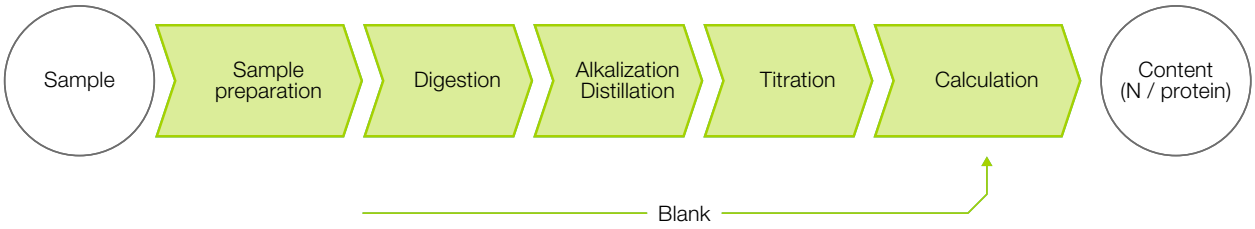


Figure 3: Kjeldahl procedure flow chart

For each step, dedicated instruments and laboratory equipment are available.
Modern instruments support users by providing:
· Optimal operation settings
· High analytical safety standards

Optimizing each step with respect to analysis time leads to a considerable overall time saving.
For example:
· Efficient distillation units can shorten distillation time
· Parallel digestion increases sample throughput
· Fast heating and cooling at the digestion stage saves process time

Reference Kjeldahl methods have been defined for a variety of food and feed samples by AOAC, ISO and other standards.

▶ For more detailed explanations and recommendations see BUCHI handbook "Kjeldahl Knowledge Base"

Kjeldahl instrumentation

Align your laboratory with BUCHI's entire Kjeldahl product line. Powerful and robust instruments for all determination steps are included.

▶ For more information visit our website: www.buchi.com/kjeldahl

Extraction

Extraction is a classical method of separation in the laboratory. It is widely applied to food and feed samples. The prominent example is the total fat extraction according to Weibull Stoldt. The working steps for this analysis are shown in Figure 4.

Figure 4: Extraction procedure flow chart

Extractors

Powerful extractors provide efficient, exhaustive and safe extractions. They are fast and easy to operate and help shortening the extraction duration.

	Classic Soxhlet apparatus	Modern Soxhlet Extractor [1]	Hot Extractor [2]	Economic Continuous Extractor [3]
Extraction cycles	20	20	-	-
Cycle time	10 min	5 min	–	–
Duration	200 min	120 min	45 min	60 min
Time saving	–	80 min	155 min	140 min

Examples of current extractors: (1) BUCHI E-816 SOX. (2) BUCHI E-816 HE. (3) BUCHI E-816 ECE

Table 1: Comparison of extractor instruments and extraction times

Other instruments

Modern instruments are also available for the homogenization and hydrolysis steps. These units help the users to work efficiently and reach reliable results. As an example, mixing the sample improves the homogeneity and increases the result reproducibility.

In addition, current instruments also support safe working procedures.

Modern instruments for extraction

Find all details of instruments, accessories and consumables on the BUCHI homepage.

▶ www.buchi.com/extraction-solutions

Selected applications

To show real application examples of the three methods described above, we have added selected Application Notes. The examples of this chapter have been selected from the food groups milk, nuts and cocoa to represent the incoming goods. For other food products and ingredients such as meat, fish, grains, flour oils, fats, dairy, fruits, etc. respective applications are also available.

The Application Finder gives access to all applications from BUCHI. For Application Notes specific to your industry, please visit www.buchi.com/applications/finder

NIR analysis

With NIR analysis an entire set of parameters can be determined. The selected example (below) from the dairy industry segment proves that parameters as different as moisture (dry matter), fat content or sugar can be determined easily and simultaneously.

Milk

Pre-calibrated application	Milk transflectance N555-509	Milk flow cell**
Dry matter [%]	7.8 – 15.5*	11.5 – 13.2
Fat [%]	0.05 – 9.80*	1.99 – 6.85
Protein [%]	1.1 – 6.5*	2.9 – 3.6
Lactose [%]	0.08 – 5.50**	4.22 – 4.98
Saturated fatty acids [%]	0.03 – 4.68**	
Mono+ Poly unsaturated fatty acids [%]	0.01 – 2.34**	
Casein		2.3 – 3.1
Sample compatibility	Homogenized milk measured with sample cup in transflectance mode	Raw milk measured after homogenization with flow cell in transflectance mode

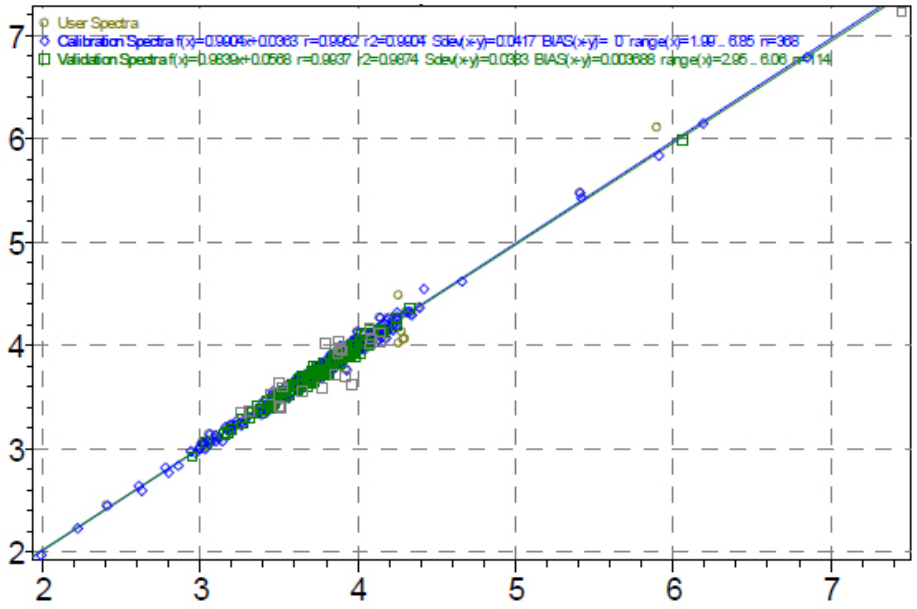
* BUCHI Pre-calibrations
** Pre-calibrations in development

▶ For more details see NIR Application Note Milk and Dairy
www.buchi.com/application-note-milk-dairy

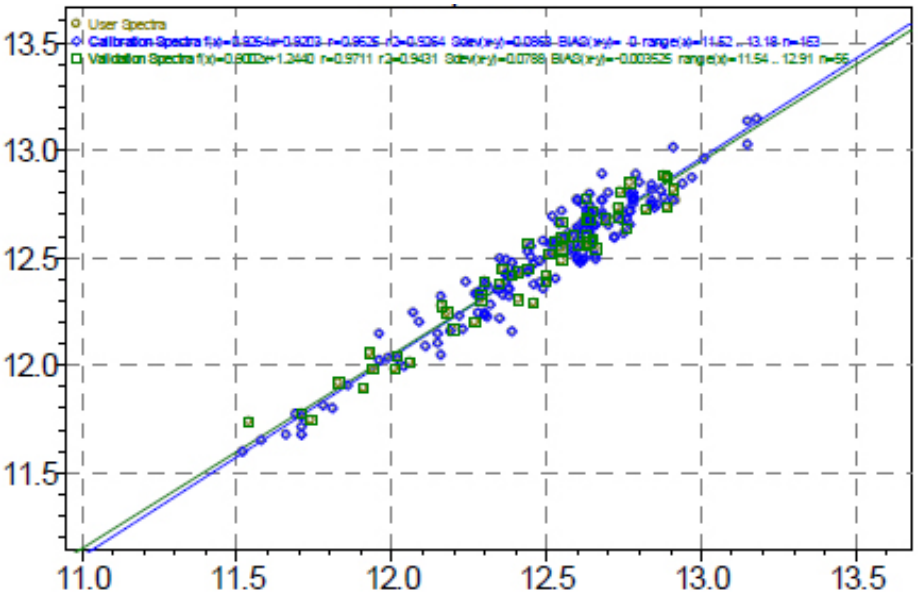
Calibration Examples

Calibrations are preconditions for quantitative results from NIR analysis. Pre-calibrations are available for a vast variety of ingredients. Hence, two examples are shown here. The straight and narrow regression of predicted and reference values indicates the feasibility and high reliability of NIR measurements.

Example 1: Fat content [%] in milk



Example 2: Dry matter [%] in milk



▶ For more details see Application Raw Milk NIRS, 20140606
www.buchi.com/short-note-raw-milk

The following NIR Application Notes for incoming food, feed and beverage ingredients are available from BUCHI.

Application Note	Ingredients
Brewing and Distilling Industry	Barley, hops, malt Fermented grains Alcoholic beverages
Confectionery Industry	Cocoa: beans, mass, butter, powder
Feed and Forage (animal feed)	Cereals, hulls, bran Soybean Animal flour Fish meal Forage, silage
Meat and Meat Products (Short Note Raw Meat, N555-501)	Beef, pork Chicken, turkey Goat, lamb Horse Wild animals
Milk and Dairy Industry Short Note Raw Milk	Milk Yogurt Cheese: fresh, hard, semi-hard, soft, processed Butter Cream Powder Raw milk, cow
Milling and Bakery Industry Wheat Flour	Cereals: whole, ground Hulls, bran Egg: liquid, powder Pasta, noodles Wheat flour
Oils and Fats Industry Application Ligurian Olive Oil	Palm oil and palm oil by-products Crude palm oil Oils and fats mixtures Olive oil and olive oil by-products Oil seeds Ligurian olive oil extra virgin

▶ For more Application Notes please visit www.buchi.com/applications/finder

Kjeldahl determination

There are many different nut varieties. They are important ingredients for many food and feed products providing protein, essential oils, fibres and fat-soluble vitamins. Hence, nuts have been selected for the Kjeldahl application example.

Nitrogen and Protein Determination in Nuts according to the Kjeldahl Method SpeedDigester K-436, K-439 / KjelFlex K 360

The determination of protein in food is a routine procedure for quality assurance and labelling. A simple and fast procedure for protein determination in nuts, as described in the AOAC 950.48, is introduced below. The sample is digested with sulfuric acid using the SpeedDigester K-436 or K 439, followed by distillation and titration with the KjelFlex K 360. The determined protein contents correspond to the labelled values.

Introduction

Protein determination is one of the key analyses performed in the food industry. The samples require digestion with sulfuric acid to convert nitrogen into ammonium sulfate. After conversion to ammonia through the alkalinization with sodium hydroxide, the sample is distilled into a boric acid receiver by steam distillation, followed by a titration with sulfuric acid solution. The nitrogen content is multiplied by a sample-specific factor (5.18 for almonds and 5.30 for other tree nuts) to obtain the protein content.

Experimental

Instrumentation: SpeedDigester K-436, K-439, KjelFlex K-360

Samples: Almonds and hazelnuts, labelled protein contents 22 % and 14 %, respectively.

Figure 1: Almonds (left) and hazelnuts (right)

Determination:

Approx. 0.5 – 0.7 g of the samples (depends on concentration of protein and organic matrix) were added directly into a sample tube. A portion of 20 ml of sulfuric acid and 2 Kjeldahl tablets were added, and the digestion was performed using the “nuts” method (K 439) or the parameters specified in Table 1. After digestion, the ammonia of the sample was distilled into a boric acid solution by steam distillation and titrated with sulfuric acid (Table 2).
The method was verified by using 0.18 g tryptophan as the reference substance.

Table 1: Temperature profile for digestion with the K-436, K-439

	K-349		K-346	
Step	Temp. [°C]	Time [min]	Level	Time [min]
Preheat	480	-	8.5	10
1	480	10	8.5	10
2	550	10	9.5	15
3	490	65	8.5	75
Cooling	-	30	-	30

Table 2: Parameters for distillation with the KjelFlex K-360 and titration

KjelFlex K-360		Metrohm 848 Titrino plus	
Water	80 mL	Titration Solution	H_2SO_4 0.1 mol/l
NaOH	80 mL	Endpoint	pH 4.65
Boric acid 4 %	50 mL	Titration Rate	Optimal
Reaction Time	5 s	Stop Crit.	Drift
Steam Power	100 %	Stop Drift	20 µl/min
Dist. Time	240 s	Stop Volume	40 mL
Titration Start	240 s	Stop Time	Off
Titration Type	Boric Acid	Filling Rate	Max. mL/min
Stirrer Sp. Dist.	5		
Stirrer Sp. Titr.	7		

Results

The tryptophan recoveries were 99.5 %, rsd 0.36 % (K-439) and 99.4 %, rsd 0.41 % (K-436).
The determined protein contents are presented in Table 3.

Table 3: Determined protein contents in nuts (relative standard deviation in brackets, n=4)

	Protein content K 439 [%]	Protein content K-436 [%]
Almonds	20.11 (0.20 %)	20.05 (0.20 %)
Hazelnuts	14.10 (0.43 %)	14.14 (0.37 %)

Conclusion

The determination of protein contents in nuts according to Kjeldahl using SpeedDigester K 436, K-439, and KjelFlex K-360 provides reliable and reproducible results that correspond to the expected values with low relative standard deviations. The total digestion time is approx. 85 min (K-439) or 100 min (K-436).

References

AOAC 950.48

Operation manual SpeedDigester K-425 / K-436
Operation manual SpeedDigester K-439
Operation manual KjelFlex K-360

▶ For more details see Application Note AN 034_2010 Nitrogen Determination in Nuts
www.buchi.com/application-note-nitrogen-in-nuts

The following Kjeldahl Application Notes for incoming food, feed and beverage ingredients are available from BUCHI.

Topic	Title	Number
Milk, dairy	Nitrogen Determination in Milk	AN 020_2010
	Nitrogen Determination in Milk micro Kjeldahl	AN 031_2010
	NPN in Milk	AN 050_2010
	NCN in Milk	AN 051_2010
	Nitrogen Determination in Milk H2O2	AN 054_2010
Grains, flour	Nitrogen Determination in Bran	AN 025_2010
	Nitrogen Determination in Flour	AN 027_2010
	Nitrogen Determination in Whey Protein	AN 028_2010
	Nitrogen Determination in Rice	AN 035_2010
Egg	Nitrogen Determination in Egg	AN 047_2010

▶ For more Application Notes please visit www.buchi.com/applications/finder

Extraction

The extraction procedure for the fat determination can equally be applied to ingredients, mixtures, intermediates and final products. The selected examples are cocoa mixtures. Excerpts of the Application Note are shown below.

BUCHI Hydrolysis Unit B-411
BUCHI Extraction System B-811

Fat Determination in Cocoa Mixes according to the Weibull-Stoldt Method

Summary

A simple and fast procedure for fat determination in Mixes in the range of 24.0-34.0 % fat according to Weibull-Stoldt is introduced. The sample is hydrolysed with the BUCHI Hydrolysis Unit B-411, Soxhlet Extraction is performed with the BUCHI Extraction Unit B-811. The output weight and calculation of the total fat content follows after drying the extract.

Products

· BUCHI Hydrolysis Unit B-411
· BUCHI Extraction Unit B-811
· BUCHI Vac V-503 with secondary condenser and Vacuum Controller V-800
· Analytical balance (tolerance 0.1mg)
· Equipment for sample homogenisation: ball mill, oven
· Vacuum oven for drying

Experimental

With the introduced method absolute fat contents up to a mass of m = 1.5 g can be determined.

Sample: Five different sorts of Cocoa Mix (ca. 24.0 - 34.0 % Fat, Acid Hydrolysis Method, Weibull-Stoldt)

Prior sample preparation

No sample preparation was carried out as the sample composed a fine, homogeneous powder.

Extraction of the fat

The Extraction System B-811 supports a fully automated process following these parameters:

solvent (S)	S: Petroleum ether, fraction 40 - 60 °C
solvent volume (V)	V = 140 mL
extraction mode	Soxhlet Standard
extraction time (t)	t = 140min
heating program lower heating (l), time (t)	Extraction l: 8, t = 120 min Rinsing l: 8, t = 010 min Drying l: 4, t = 010 min
external drying	Vacuum oven

After extraction the sample (residue in the beaker) is dried in a vacuum oven (1h at 100°C/200mbar) to constant weight. Allow the sample to cool down to ambient temperature for 30 minutes in a desiccator, and then record an accurate weight.

Conclusion

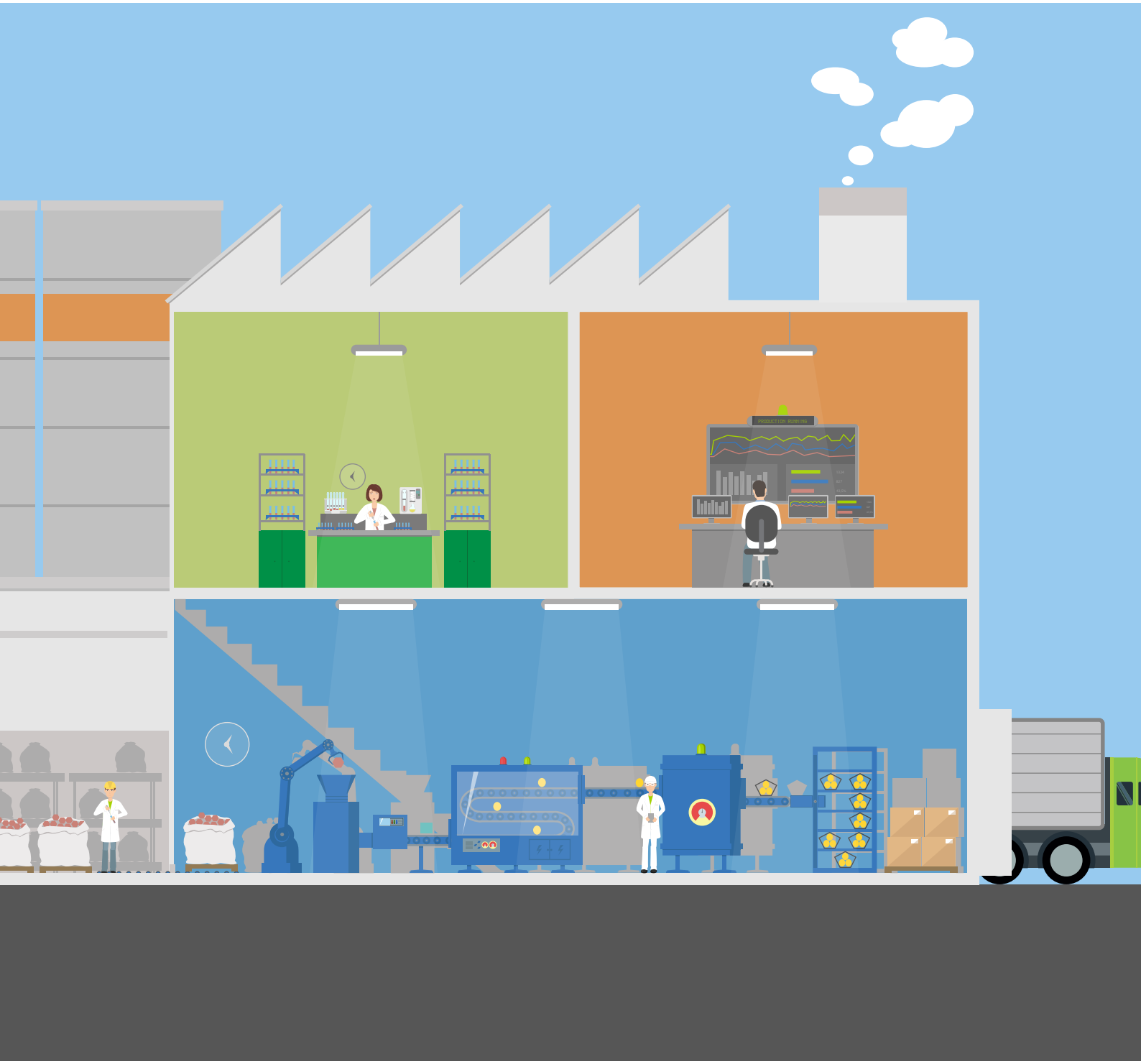
The obtained fat content showed good correlation with the expected value meeting the target range of 24.0 – 34.0 % fat

▶ For more details see Application Note

The following Extraction Application Notes for incoming food, feed and beverage ingredients are available from BUCHI.

Topic	Title	Number
Plant based raw materials	Fat Determination in Green Tea	010-411_811-04A
	Fat Determination in Cocoa Mix	013-411_811-98A
	Fat Determination in Bakery Improver	016-411_811-00B
Meat	Fat Determination in Meat and Meat Products	007-411_811-03B
	Fat Determination in Meat	AN E-416-E-816-HE-002
	Fat Determination in Meat and Meat Products	AN E-416-E-816-Sox-002
Fish	Fat Determination in Fish	AN 005-411_811-03A
Vegetables, herbs	Fat Determination in Grounded Pumpkin	021-411_811-01A
	Extraction of Spices for the Determination of Pesticides	AN 064-2011

▶ For more Application Notes please visit www.buchi.com/applications/finder



2: Process monitoring

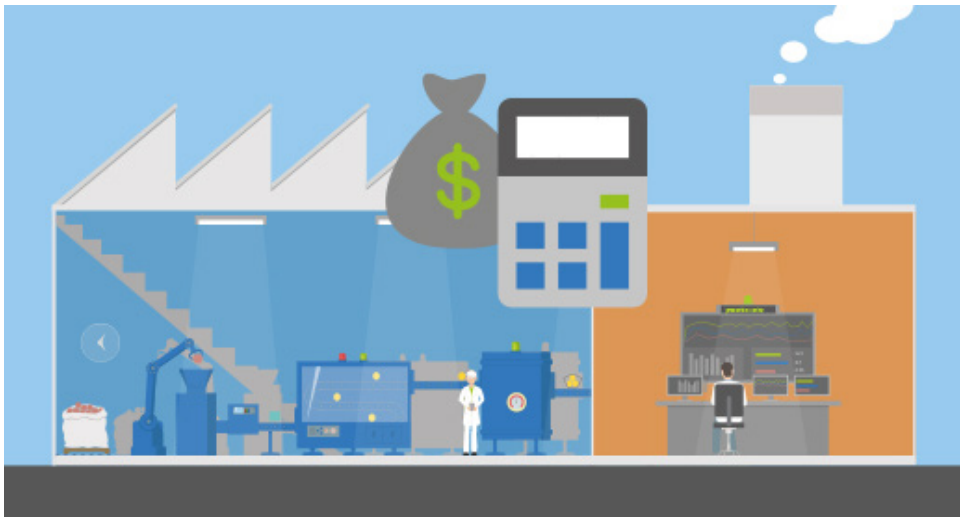
Contents

The food production process

Introduction

The food and beverage production process chain comprises typically of five steps starting with goods receiving, incoming goods inspection, going over to production, quality control, and finishing with logistics and distribution. Along this chain, the quality of raw materials, intermediates, and final products is checked regularly. Samples are taken at specific points throughout the entire process chain and further analyzed for relevant quality parameters.

In this second part of the BUCHI Food Process Analytics guide, the focus lies on production process monitoring and how this can bring financial benefits.



Demands of food production processes

In general, the main task of a production department is to manufacture finished goods. Through a series of processes raw materials (input) are turned into final products (output). Examples of such processes may include cooking, fermentation or milling. The output has to match the right quality and quantity requirements at the right time and for the right costs [1, 2]. These terms are mainly defined by customer requirements, such as composition, caloric value, taste, color, shelf life, etc. Despite the occurrence of natural variation in ingredients, customers expect a consistent end-product quality [3, 4]. Regulatory influences further drive product manufacturing standards, particularly as they relate to product composition and labeling or other aspects that may impact consumer health.

Beyond producing high-quality products, manufacturing implies the added responsibilities of machinery and equipment surveillance, hygiene, warehousing and logistics. These and other processes must reach individual targets and be optimized as a whole [5, 6].

Implications for out-of-spec products

Keeping a product within the specification range given by the recipe or formulation, and staying within identified safety margins of certain ingredients, can often be a challenge. Any number of deviations can lead to out-of-specification products that may require reworking, selling product at a reduced price, wasted batches, or even product recall. These are just some of the expensive and unpopular implications of poor process monitoring. Reduced production capacity and throughput are also potential indirect outcomes.

These challenges and implications demand a solution providing real-time surveillance and feedback for process control actions.

Process monitoring

Process monitoring indicates surveillance of process data which is correlated to product quality. Process control occurs when this data is used to adapt the production variables (e.g. drying, mixing or cook time). Seamless and effective production process monitoring followed by consequent process control serve to assure that:

· Specifications are met
· Optimum manufacturing efficiency is attained
· Any waste of labor, materials, machines and tools is prevented [4, 7, 8]

How to meet production challenges

In practice, key performance indicators (KPI) are used to assess, analyze, and track production processes. KPIs evaluate the success of a process in relation to set goals and objectives [9]. Also, the concept of Critical Process Parameters (CPP) can be applied to scrutinize production processes. CPPs affect Critical Quality Attributes (CQA) [10, 11, 12].

The continued surveillance of KPIs and (or) CPPs contributes to mastering the challenges of food production processes.

Fast and immediate monitoring

A powerful monitoring tool is the analysis of product composition. This can include the concentration determination of a lead component or a proximate analysis. The frequent analysis or even continuous determination in real-time of the product composition are prominent measures to avoid out-of-specification products. They allow for the timely identification of processing deviations and errors. Immediate correction measures can be taken.

Best suitable analysis method

As an example, the fast or even continuous read-out of the moisture content is highly valuable because the process can still be adjusted to reach the target moisture content. Other important parameters such as fat or sugar content can also be determined. Even more valuable is the determination of all these content parameters together.

Near-infrared (NIR) analysis is the proven tool for this task. Results are available within seconds. It is also possible to monitor several critical components, like protein and fat, simultaneously. No reagents nor solvents are required for the measurement. Hence, NIR spectroscopy is the method of choice [13].

See the next chapters for what NIR analysis can do for you.

Real-time production monitoring

The measurement technique of choice for prompt or even real-time monitoring is NIR analysis.

Main benefits

NIR analysis is a spectroscopic method that uses light to measure useful sample properties like fat, protein and moisture. The main benefits of NIR analysis technology are:

· Short measurement time (seconds)
· Minimal or no sample preparation
· Suitability for all kinds of sample types (powder, pastes, granules, liquids, etc.)
· Simultaneous determination of various sample parameters
· Non-destructive measurements (i.e. sample-sparing)
· Broad application base

Measurement points

		Discription	Suitability
Batch measurements	Off-line	Instrumentation is located in a controlled environment (e.g. control room, QC laboratory) away from the measurement point Requires sampling and sample transport	Instrument can be used to measure and control multiple production processes Turnaround time is not critical Different sample types can be accommodated
	At-line	The sample is removed and isolated from the process stream. It is analyzed near the process stream. The instrumentation is located in the production area	Non-invasive measurements with faster measurement results than off-line measurements
Continuous measurements	On-line	The sample is diverted from the process stream A probe or sensor is installed in a by-pass loop off the production stream. Measurements are automatic	Near real-time measurements in the by-pass loop Direct process control capabilities Probe or sensor not necessarily exposed to process conditions
	In-line	The sample is not removed nor diverted from the process stream Instantaneous measurements directly in the process stream (in situ) by dedicated probe or sensor Measurements are automatic	Direct real-time measurements Direct process control capabilities Probe or sensor has to withstand process conditions

Table 1: Description of measurement points

Due to these qualities, the NIR method is a suitable tool for real-time analysis (RTA). The immediate quality and process control feedback provided by NIR spectroscopy, bypasses typical QC laboratory hold-times. The application of on-line or in-line NIR sensors provides the added potential for continuous and automated quality measurements at various installation points along the production chain.

Different measurement point options

Typically, there are four types of measurement points throughout a factory. They can be defined by the distance to the process. In-line is the closest point, off-line has the biggest distance. NIR analysis is feasible for all of them. The decision regarding the preferred measurement point also takes into account their opportunities and risks [14, 15].

Opportunity	Risks	Selected instrument examples
Expert knowledge available in the laboratory High flexibility of instrument use Suitable analytical environment	No direct process control Slow due to sample transport Shared ownership of data (laboratory, production department)	BUCHI NIRFlex N-500 
Dedicated analytical instrument Proximity to process enables fast manual sampling Direct ownership of data by production department	Instrument may be underutilized High degree of instrument robustness required	BUCHI ProxiMate™ 
 Fast No manual sampling Direct process control Dedicated analytical instrument Typical fast return on investment	Instrument costs cannot be shared with other users Instrument costs cannot be shared with other users High degree of instrument robustness required	BUCHI NIR-Online® Process Analyzer 

Choosing the right monitoring instrument

When considering the implementation of NIR measurement systems, there are a few guiding principles. First, sample accessibility and stability, safety, and time to result are considered. Then, the impact of the process monitoring on the laboratory or the production plant as well as their needs are estimated. Finally, all points are evaluated to decide on the most feasible monitoring instrument and measurement point.

The following paragraph describes this evaluation process more in detail:

- Accessibility of the sample, e.g. easy access to collect samples
- Sample stability, e.g. segregation, hygroscopicity, evaporation
- Safety: regarding both the sample collector and the sample itself
    - Process equipment extremely cold or hot or under high pressure
    - Need of ladders or scaffolds to collect samples
    - Sterility, contamination
- Time to result, e.g. sample transportation time admissible, end-point determination of a batch operation without any delay

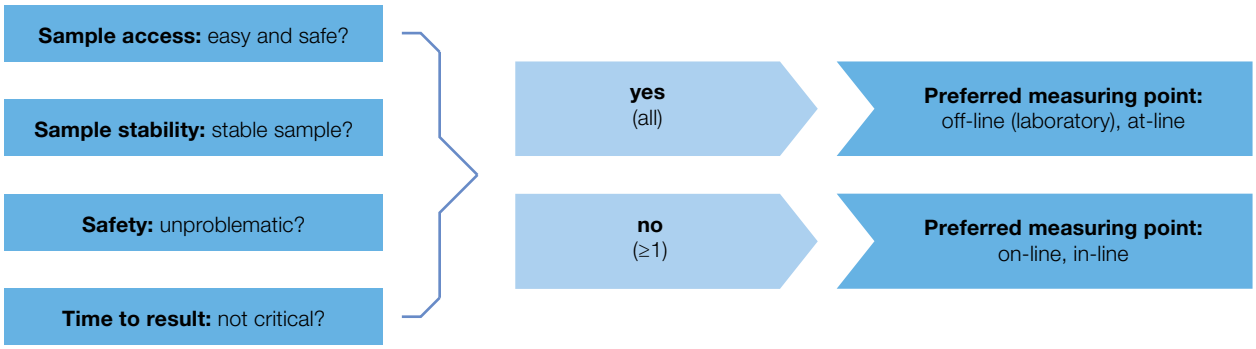


Figure 1: Decision tree to preferred measuring point

The decision tree of Figure 1 is based on the four criteria described above. If all four questions can be answered with a “yes” response, then an off-line or at-line system would be suitable. If any of the four questions are answered with a “no,” then an on-line or in-line system may be better suited; otherwise, the risks associated with off- or at-line sampling need to be carefully addressed by the standard operating procedure employed. In the end, each situation should be addressed according to the unique requirements of the customer.

The following discussion points should be considered when selecting the optimal NIR measurement system(s):

- How many measurement points are required along the production line?
- Does the budget (or estimated return on investment) justify dedicated sensors at each measurement point, or is the flexibility of an at- or off-line unit required?
- Is real-time analysis essential to accomplish direct process control?
- What constraints exist based on the proposed NIR installation points (e.g. required ingress protection and/or ATEX requirements)?
- Requirement to visualize and document the complete production process?

The final decision is based on the evaluation of all concerns. The importance of the concerns depends on the particular case and its conditions.

For more details on choosing the right monitoring instrument and measuring place go to www.buchinir.wordpress.com or contact us.

NIR analysis pays off

NIR analysis offers two basic opportunities for savings:

· Reduce costs of analyses

Many standard reference or other classic analytical methods are rather expensive (costs of labor, reagents, lab equipment, etc.). NIR analytics replace a major number of such analyses. Also, reagent and solvent consumption and disposal is eliminated.

· Set tighter safety margins, get immediate results

To converge closer to set formulation targets and stay easily within the safety margins is one main benefit of on-line and in-line analysis of the sample composition with NIR spectroscopy. Another main benefit is the timely measurement which allows for direct process control.

Profit from batch measurements (off-line, at-line)

NIR analysis instruments designed for off-line use are applied in the laboratory or in the production department (at-line application). In these positions, the NIR instruments complement classical analytical instruments such as Kjeldahl analyzers, fat extractors, chromatographs or titrators and lead to potential analysis cost savings (see Figure 2).

Pay-back example

Saving 10 laboratory samples per day at 20 EUR cost per sample on 200 working days of a year amounts to a total saving of 40.000 EUR. At typical NIR instrument prices of 50.000 EUR a favorable pay-back time of only 1.25 years results.

Additional benefits are achieved. On the one hand, the usage of reagents, solvents and other laboratory consumables is reduced. On the other hand, laboratory productivity increases thanks to the speed of the NIR analysis.

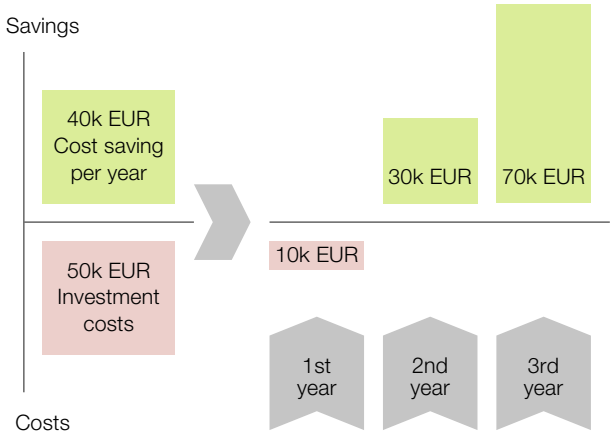


Figure 2: Cumulated annual savings

A customer from a pharmaceutical production company in China explains:
“The NIRFlex N-500 in combination with the disposable sheath makes day-to-day work in the laboratory easier due to its ease of use and efficiency. Since it requires no use of chemical reagent and no sample preparation, the analysis is more economical and environmentally friendly. It is the key for cost-efficient analysis at a high quality level, performed in a short time.”

Profit from continuous measurements (on-line, in-line)

NIR analytics for continuous on-line and in-line compositional analysis is an alternative or extension to other continuous measurements such as temperature or pH value. It is also a supplement or replacement of classical analytical methods.

Because the production process can be adjusted or stopped early in production if specifications are not met, the use of on-line and in-line NIR analysis minimizes expensive rejects and complaints. This benefit is further increased with the possibility of adjusting the relevant contents in real time.

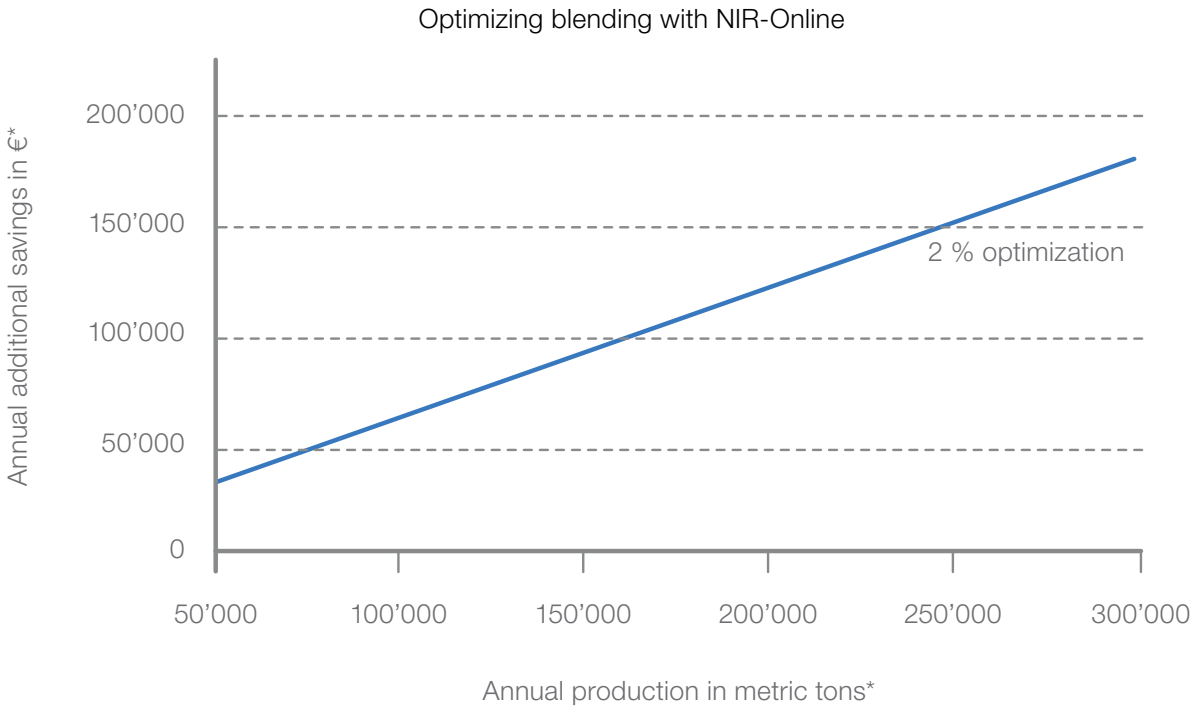
“If we are able to converge within 0.5% of the moisture set point of a batch, we can sell more than 375 tons of additional mixed feed per year – a substantial benefit. The investment in on-line NIR technology will be paid for in a few months”, says a customer from a German feed mill.

Flour blending example

Prior to packaging, flour is often blended to yield uniform color described by ash content (which also correlates to mineral content). Color / ash may be continuously monitored with on-line NIR process analyzers to reach maximum production efficiency. Color monitoring is also applied to achieve constant, reliable baking properties of various flours and to meet legal requirements. Payback depends on:

· The degree of blending optimization
· This means, how much “low price dark flour” can be added replacing “high price white flour” whilst still fulfilling legal and quality requirements. A 1% optimization refers to the addition of 1% dark flour.
· Price difference between “high price white flour” (e.g. Type 405) and “low price dark flour” (e.g. Type 1050)
· Annual production

At a price difference of 30 EUR per ton between the “dark” and “white” flour and 100.000 tons annual production, annual savings of 60.000 EUR are achieved with an optimization level of just 2%. Depending on the exact configuration and installation conditions of the in-line or on-line NIR instrument, the pay-back time at this annual production is between 6 and 14 months.



* ”low price dark flour”: 270 € / metric ton
“high price white flour” 300 € / metric ton

Figure 3: Annual savings of optimized flour blending [16]

For more information please download the NIR -Online Solution Brochure on www.buchi.com/products/nirsolutions/nir-online-process-analyzer.

Continuous process monitoring

In-line and on-line measurement points for NIR analysis

Typical applications of on-line and in-line NIR analysis include moisture control, extraction monitoring, protein addition, etc. Another common example is the survey of mixing processes, where several ingredients are blended. Mixing steps face the difficult task to match set recipe formulations as close as possible. Real-time information about the mix composition e.g. in terms of fat, moisture and protein, enables in-process adjustments. Real-time monitoring of mixing steps helps to:

· Run a production closer to the specified targets
· Avoid or reduce re-work
· Increase energy efficiency
· Ensure consistent final product quality

NIR analysis is an excellent real-time analysis method enabling for immediate process control [17].

On-line instruments master many requirements

Besides being easy to operate, instruments for use in the production area have to meet further requirements.

· Ambient conditions: humidity and temperature in the production environment can be higher and fluctuate more than in a well-kept laboratory. Because air can also be dusty, appropriate dust protection has to be implemented into instruments. Suitable on-line instruments have to withstand such influences.
· Robustness: on-line analyzers need to withstand occurring vibrations. In addition, they should have low maintenance needs, long service intervals and minimum downtime.
· System integration: a basic necessity, see section Process integration.
· Protection: according to IP and ATEX, see section Ingress and ATEX protection.

BUCHI instruments for in-line and on-line use are designed to match these tough requirements and withstand harsh conditions as they occur in the process surroundings.

For more details please access the technical data sheets of the respective NIR analyzer here: www.buchi.com/products/nirsolutions/nir-online-process-analyzer

Some unique features

AutoCal

The patented auto-calibration function eliminates the need to develop extensive in-house calibrations or purchase calibration data bases. If new reference data is available, the calibration is updated and optimized automatically. New data is simply entered into the SX-Suite software and confirmed with one click. No deep knowledge in chemometrics and statistics is required.

NIR + VIS + CCD camera in ONE device

The BUCHI Online® sensors and spectrometers contain detectors to measure both the visible (VIS) and the NIR range of the electromagnetic radiation (refer to Appendix 1). The integrated CCD camera is used to take images of the material, assess its visible properties and to perform image analysis in addition. The combination of these three features is unique and enables comprehensive evaluation of your production process just with one instrument.

Giving the example of the grain milling industry: Work with the NIR range for determining the chemical properties such as moisture and protein. The VIS range enables color (L, a, b) measurements, and provides superior determination of the ash content in the flour. Apply the CCD camera in conjunction with image analysis function of the software to monitor the acceptable level of specks for sifter control.

Multipoint System

Some benefits of the unique Multipoint System are
· Full control along one complete value chain
· Full control of several production lines in parallel at one critical control point
· Patented daisy chain connection

The Multipoint System provides up to 10 measuring points in just one system. Up to nine optional Multipoint Heads (MPH) share one Multipoint Sensor (MPS) and build a budget-friendly control system of entire processes. The need of costly optical fibers is reduced thanks to daisy chain connection of the Multipoint heads (patent pending). Automated report generation and auditing function for instant documentation save time.

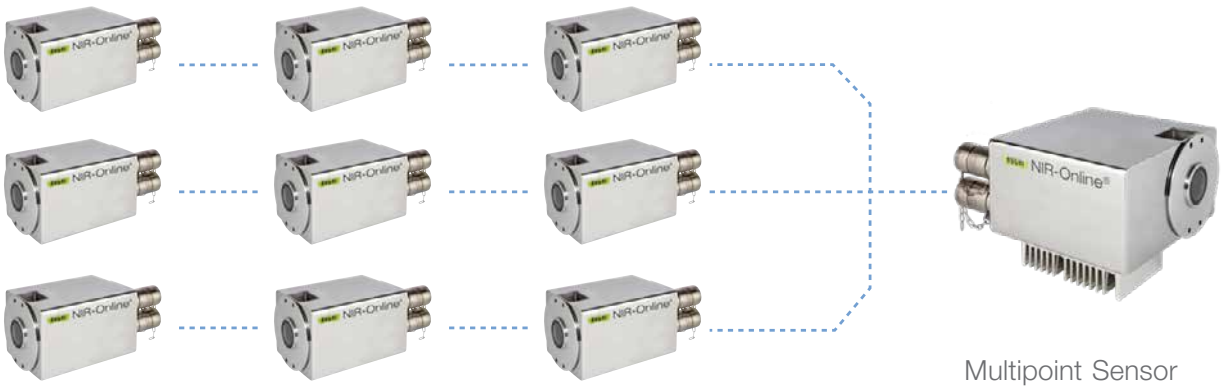


Figure 4: Schematic of nine MPH connected to an MPS as 3x3 array with daisy chain connection

NIR software function to monitor mixing

Monitor mixing processes for the purpose of determining homogeneity and end point control by applying patented software function. The spectrometer continually measures the mixture during the entire mixing process. Variations in the NIR spectrum in the process enable homogeneity and chemical composition to be determined. No calibration is needed for the determination of the homogeneity. In addition to this, the correct end point and chemical composition are verified by means of the current spectrum being compared with a spectrum stored in a library.

Process integration

Smooth process integration is a necessity for on-line analyzers. Only rapid process integration of NIR on-line sensors ensures a fast return of the investment . BUCHI NIR Online® analyzers are easily integrated mechanically and electronically into processes.

Several available integration options offer optimal solutions [18]. Hardware adapters for the mechanical integration are summarized in Figure 5. A series of interface modules enable the electronic integration. The following interfaces for PLC integration are available: Profibus, Modbus, Datalab I/O and OPC (TCP / IP).

Some outstanding characteristics of NIR process integration solutions from BUCHI:

· Comprehensive industry-proven hardware adapter portfolio for optimum installation in existing processes
· Various convenient solutions to directly display all relevant parameters in the database and control room

Figure 5: Hardware adapters for the mechanical integration

Ingress and ATEX protection

Ingress protection (IP) of electrical equipment against solid foreign objects (e.g. dust) and liquids (water) is classified according to ANSI/IEC/EN standard 60529. BUCHI instruments provide high protection levels against dust and water.

BUCHI instrument	Ingress protection
NIR-Online® Process Analyzer	IP65 or IP67 (individual IP classes on request)
NIR-Online® Multipoint	IP65

IP class description
- IP 65: dust tight / protection against water jets (6.3 mm nozzle)
- IP 67: dust tight / protected against immersion (1 meter, 30 seconds)

Table 2: IP classes of some BUCHI NIR instruments

ATEX

The ATEX Directive 2014/34/EU defines requirements and assessment procedures for equipment intended for use in potentially explosive atmospheres which exist when a mixture of air gases, vapors, mists, or dusts combine and can ignite. This applies to flour mills amongst others [19].

BUCHI NIR-Online® instruments offer ATEX protection. The “ExProof” solution gives rise to uncritical operation in potentially explosive environments. The process analyzer is constantly over-pressurized, therefore penetration of flammable gases into the system is excluded at all times. Enjoy full installation flexibility, as additional explosion proof cabinets are not required. Moreover, BUCHI NIR-Online® "ExProof" solution is compatible with the large BUCHI process integration portfolio.

Batch process monitoring

Off-line and at-line measurement points for NIR analysis

NIR based determinations are carried out within several seconds, accelerating sample throughput regardless of installation type. Substantial savings of time and money are the gains. The advantages apply to all laboratories, from R & D to production to quality control.

A NIR analyzer housed in the laboratory can be used for determinations across the entire plant. Environmental conditions in the laboratory are usually well kept and moderate compared to the situation on a production floor. Hence, instruments can be more sensitive and achieve higher precision with more care taken with sample handling.

Typical off-line instrument requirements

Predominant requirements for laboratory instruments are:

· High system performance, including wavelength accuracy and precision
· Flexibility in hardware and/or software to optimize measurement performance for a variety of samples
· The capacity to develop or modify existing calibrations to suit unique requirements
· The ability to integrate data into LIMS and ERP software

Also for off-line use, BUCHI offers a line of highly qualified NIR instruments.

Some unique features

Robust design

The BUCHI ProxiMate is the most robust spectrometer available, designed to be used in harshest of environments. The instrument is completely water and dust proof allowing measurement near or next to a food or feed production line. The instrument is resistant to chemicals used for sanitation in food preparation areas meaning that alkaline, acidic and chlorine-based detergents can be used at high temperature and pressures.

Optimum choice of NIR technology

To help ensure that end users achieve their desired balance between purchase cost, features and measurement performance, BUCHI offer a number of NIR technologies across a range of instruments. In all cases, robust, consistent performance is the key criteria to optimum operation. Both the polarisation interferometer and diode array technologies have been optimised to provide the best possible, most consistent measurement performance. BUCHI ProxiMate offers cost effective operation in the harshest environments, whilst the NIRFlex N-500 offers the highest level of spectroscopic performance with a very wide range of sampling options.

Hot-swappable sampling modules

For laboratories required to do a combination of solids and liquids sampling, different sampling modules may be required to achieve optimal NIR method performance. The BUCHI NIRFlex N-500 offers various measurement cells and sample adapters to accommodate samples ranging from loose powders to transparent liquids. The measurement cells themselves can easily be switched without needing to power-down the system first. This is particularly beneficial when a lower sampling capacity does not justify multiple NIR units in the lab.

Secure data

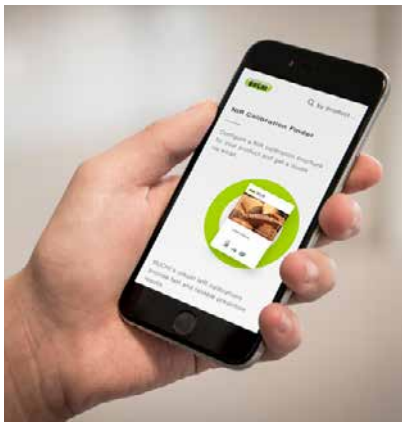
The NIRWare software used to operate the NIRFlex N-500 and NIRMaster products was designed to meet stringent requirements of data recording and security. User groups can be easily created to manage access rights to various modules or functions of the software. Additionally, the database-oriented software ensures that critical data cannot be erased or lost, while also providing an easy way to back-up and restore data on any computer running the NIRWare Software Suite.

For more information on the BUCHI NIR solutions please visit
www.buchi.com/content/nirsolutions

The calibration finder

Reliable NIR analysis results base upon correct calibrations. Calibrations have to include all kind of variation incorporated into the samples. The BUCHI databases consist of thousands of NIR spectra covering geographical and seasonal variation. Hence, these spectra provide the background for
accurate and reliable results.

Get direct access to the NIR calibrations available from BUCHI. Use the on-line calibration finder to find pre-calibration details and expected performance data. The calibration finder is continuously updated by the BUCHI experts to provide most recent information to NIR users.



Selected industries

The following sections of this chapter group segments of the food industry and provide examples of the most often analyzed content parameters. In most cases, the determinations can be carried out as batch or continuous measurements. Some comments and benefits of the NIR method conclude the short chapters.
Besides the parameters mentioned, many more determinations are possible. For many of them, pre-calibrations are available from BUCHI. The latest versions are summarised in the calibration finder online tool.

Common sample types
· Oils (olive, sunflower, palm, rapeseed)
· Tallow, lard, chicken fat

Fats and oils

Free fatty acid content (FFA) of fats, oils and mixtures or total fat content of oil seeds and oil by-products are just two examples of frequently analysed quality parameters. The conventional methods for FFA and total fats are elaborate, require reagents and solvents and take considerable time to complete. However, both parameters can be measured easily within seconds by NIR analysis reducing testing costs considerably.
Many more determinations can be carried out the same favorable way with NIR such as peroxide value, acid number, iodine number, K and ΔK values.

Benefits

· Fast composition analysis of oil seeds before crushing
· Monitoring entire product process

Common sample types
· Milk
· Butter
· Yogurt
· Cheese
· Powders

Milk and dairy

Three major constituents of milk and dairy products are moisture (or dry matter), protein and fat. Classical determinations are tedious, time consuming and require chemical reagents. NIR analysis is an easy alternative method.

More parameters such as lactose, ash, salt, pH value or total sugar can also be determined by NIR. A further advantage of NIR: all the parameters are determined within one measurement run only.

The producers of milk are paid mainly on butterfat and protein content. Therefore, producers and customers depend on fast, accurate and reliable results [22].

Benefits

· Analyze intermediates and finished goods with the same instrument
· Optimize process using NIR analysis
· Check quality of finished goods

Common sample types
· Whole cereals (wheat, barley, maize/corn)
· Ground cereals / wheat flour
· Semolina
· Gluten

Milling and bakery: grains and flours

Moisture, protein, and ash content of flours and grains are frequently measured quality parameters. Results within seconds are delivered by NIR analyzers. An entire variety of additional parameters, e.g. starch, gluten, fat, fibre, specks (bran) can also be measured with NIR-Online®.

Continuously monitoring e.g. protein content gives rise to confidently operating closer to target specifications and therefore, prevents expensive rework. Blending optimization contributes to a rapid payback of the installed system which is on average less than one year.

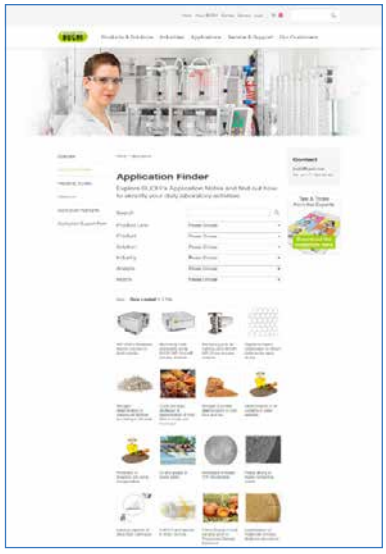
Benefits
· Distinguish grains and cereals
· Decide which flours to blend
· Control ingredients
· Maintain consistent high quality

Common sample types
· Beef
· Pork
· Chicken
· Fish
· Sausage

Meat and fish

Protein, fat, and moisture content are three major quality parameters of meat, meat products, sausages, fish and fish products which are easily determined by NIR analysis. In contrast, the reference methods for protein (Kjeldahl) and fat (extraction) are classical chemical analyses. Modern Kjeldahl and extraction instruments strongly support the user. However, considerably more time is consumed to get the results and well-educated laboratory staff needs to work diligently. Easy NIR analysis also delivers more content determinations such as ash, collagen and salt.

Benefits
· Take rapid decisions in the slaughterhouse
· Optimize formulation during production
· Inspect goods at the packaging stage



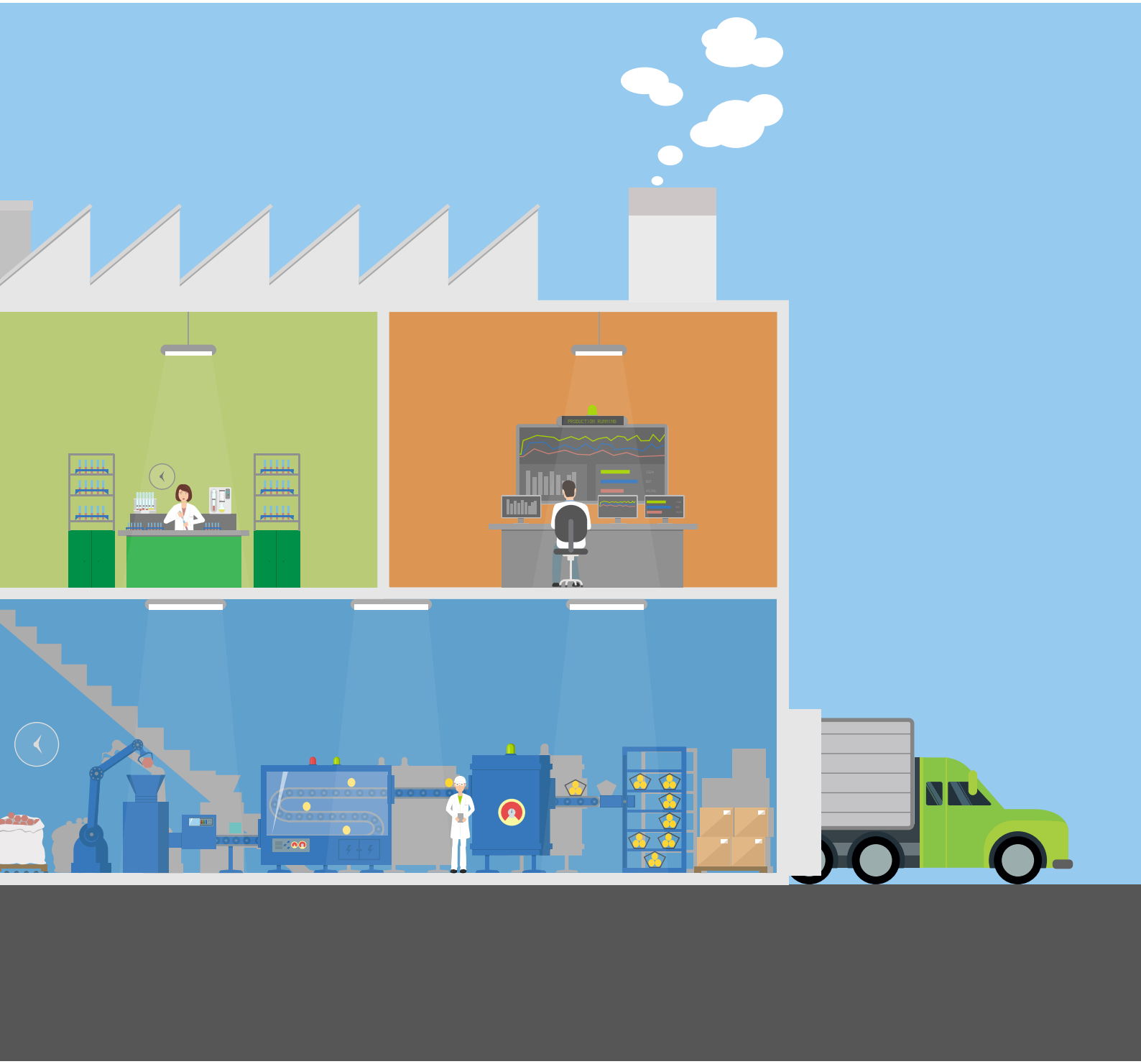
Other industries and other applications

Besides the above-mentioned industries, BUCHI also serves the following industries:
· Beverage
· Feed
· Pharma and Biotech
· Chemical
· Environmental Analysis

Application Notes and Short Notes from BUCHI covering all industries and including other measurement methods than NIR are available in the Application Finder. This site is steadily updated to provide the latest examples and customer support.

Go to www.buchi.com/applications/finder

Food Process Analytics
The Champion's Guidebook

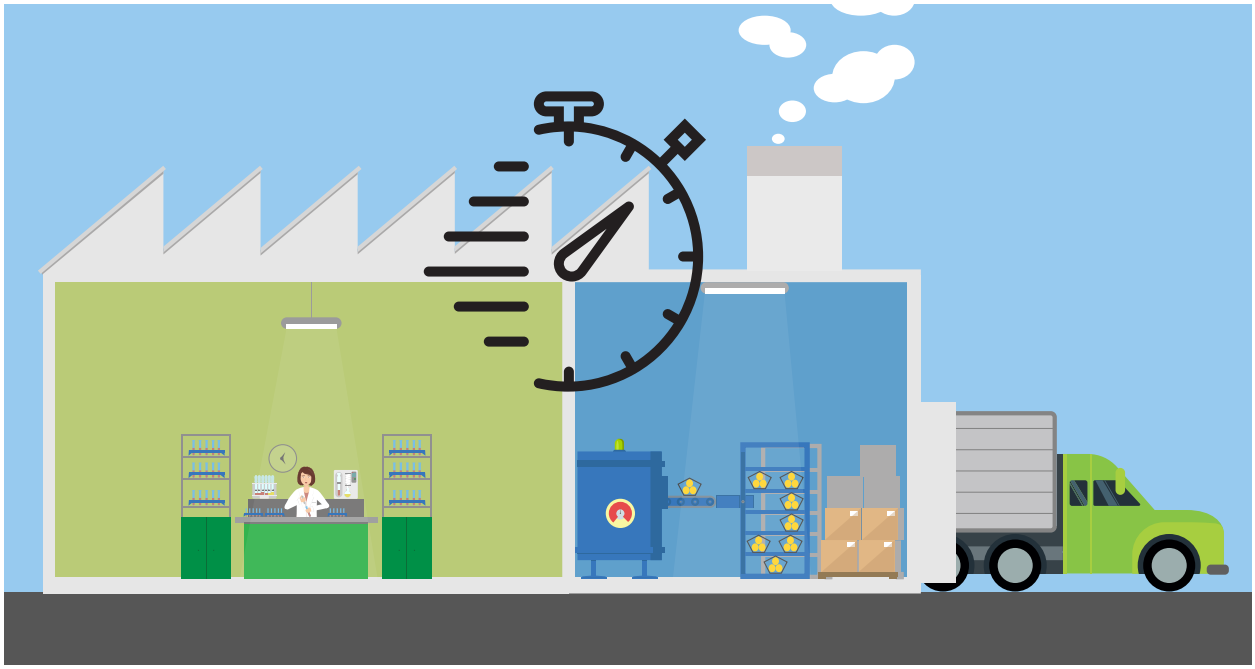


3: Final Goods Inspection

The food manufacturing process chain

The food and beverage manufacturing process chain comprises of five steps – incoming goods receipt and inspection, production, quality control, logistics and retail. Along this chain, the quality of raw materials and final products is checked once, whereas the compliance with the food production recipe is monitored by continuously testing the intermediates. Samples are taken at specific stages throughout the entire process chain and further analyzed for relevant quality parameters.

The third booklet of the BUCHI food analysis guide covers quality control and its function in the overall quality assurance of final goods. The focus lies on final goods inspection, frequently asked questions and trouble-shooting.



Quality management

Quality control and quality assurance

In the ISO 9000 norms, both quality control and quality assurance are mentioned as part of the quality management system. Quality assurance is an overall system to assure quality along a production workflow in order to reduce product defects, whereas in quality control the final good is tested for conformity with legal requirements and its nutritional composition for compliance with food labelling. In quality control, aspects of food safety, such as absence of pathogens and heavy metals, as well as food quality, such as content of nutrients, vitamins and minerals are analysed.

Contribution of quality assurance to final goods quality

Following recent trends of food quality management systems, waste of final goods has been decreasing due to a higher number of audits of ingredients, manufacturers and suppliers, and an increase in incoming goods inspections and in-process controls. This holistic approach, including HACCP[1], improves both consumer safety and the environmental footprint while maximising profit [1].

The final goods inspection workflow

The inspection of final goods is the last step in the quality control program. Final goods are analysed and released before being shipped to food retailers and consumers. Depending on regulatory requirements, consumer needs, type of food and food product, a sampling plan is established that outlines the sampling scheme, parameters to determine and sample size. The sampling plan defines how random samples are taken [2].

The final goods inspection workflow starts with the sampling and the collection of the samples and ends with the product release or embargo. The flow chart below defines the process of the final goods inspection.

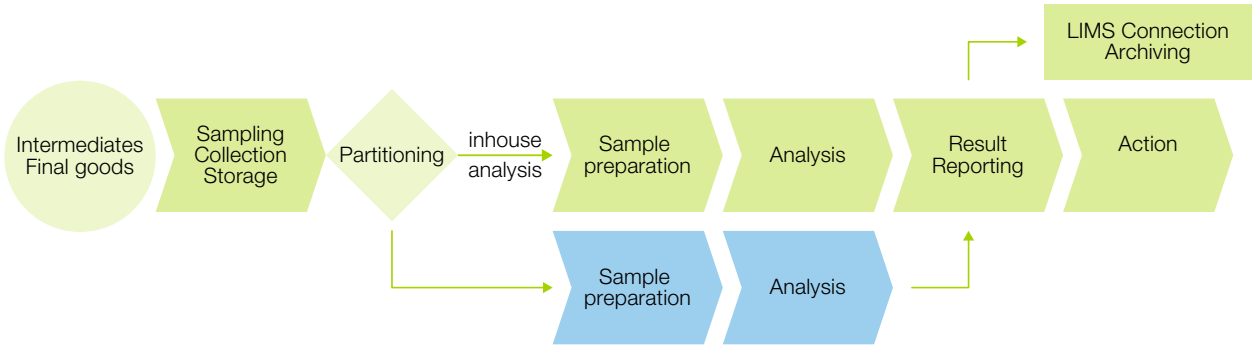


Fig 1: Workflow of final goods inspection

[1] HACCP: Hazard analysis and critical control points

Sampling

The selection of an appropriate fraction from the entire production lot is an important stage of food analysis. If not carried out correctly, substantial errors can occur. Several points need to be considered for the sampling step [3]. These include:

- Which fractions of the product lot are examined?
  - Random sampling
  - Systematic sampling (e.g. every 60 minutes)
- How much sample needs to be taken
  - To be representative of the entire lot (homogeneity)?
  - To suffice for all analyses?
- How often should a material property, such as fat content, be determined?
- How many samples should be measured (single, duplicates, triplicates, etc.)?
- Is a composite sample prepared from different fractions of the lot or are the different fractions examined separately?

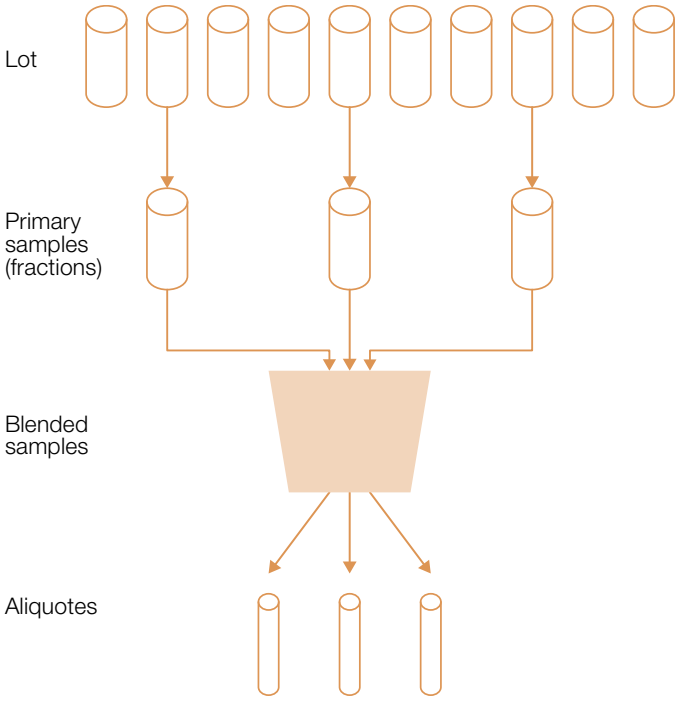


Fig 2: Sampling plan

Figure 2 shows a typical sampling procedure. Immediately after sampling, the sample is correctly labelled and registered. This helps to avoid later confusions.

Collection and storage

During the collection and storage of the samples, any deterioration, alteration or modification of the samples must be avoided by. Such sample changes cannot be corrected during downstream analysis. Depending on the sample, the following measures help to maintain the initial status of the sample [4]:

- Shorten collection time as much as possible
- Consider sample temperature (keep initial temperature, cool, freeze)
- Protect from moisture
- Protect from air and light to avoid oxidation of the sample
- Avoid contamination (use clean glassware, use sterile glassware)

Partitioning

At the partitioning step, samples are sorted and directed to the required analysis and measurement method. Separate aliquots are prepared if different analytes need separate aliquot samples. However, aliquoting requires meticulous work. If an aliquot does not represent the original sample accurately, the results are questionable [5].

Samples are either immediately analysed or placed in central sample storage. A climate-controlled storage room is the place of choice if storage temperature plays an important role in preserving the sample [6].

Samples have to be partitioned for the following disciplines:
· Chemical, e.g. Kjeldahl (protein), extraction (fat)
· Physical, e.g. viscosity, color
· Microbiological, e.g. Salmonella, E. coli, total colony forming units (CFU)
· Sensorial, e.g. mouth-feel, taste and smell

Additionally, the decision on whether to perform in-house analysis or to rely on external analytical services is taken. The decision depends on the capabilities and capacities of the in-house laboratory. The time-to result factor has to be considered as well [7, 8].

External analyses may be outsourced to governmental or private testing labs. Some benefits and disadvantages related to external testing laboratories are listed below.

Benefits	Disadvantages
High analytical competence	Sample shipment efforts
Transparent costs	Time delays
High sample through-put	Confidentiality of results

Sample preparation

Sample preparation describes how a sample is treated prior to analysis. It is a critical and important step because:
· The consecutive analytical measurement is only as good as the preceding sample preparation has been
· The measurement techniques are often not responsive to the analyte in its original form
· Results can be distorted by interferences of the original sample's matrix

During sample preparation, sample loss should be minimized and sample contamination prevented. Sample loss can occur by various ways, including loss as dust or particulates when ashing or grinding, through volatilization or due to reactions between sample and glassware or preparation tools. Contamination sources are numerous. They include reagents and solvents, glassware and labware, cross-contaminations from other samples, as well as laboratory equipment, instruments and instrument accessories [5, 9]. A typical source of contamination are impure solvents used for dilution or dissolution of the samples.

See also chapter *Sample preparation*

Analysis

Many analytical techniques and methods are applied for the analysis of food samples [10]. In this volume, the focus lies on Kjeldahl, extraction and near infrared (NIR) techniques. Methods based on these techniques for the quantification of protein, fat and other ingredients are explained.

Nowadays, the analyst in the laboratory is supported by automated analytical instruments. Instruments have undergone an enormous development in the past decades due to the efforts of instrument manufacturers to continuously increase sample throughput, sensitivity and application scope [11]. For example, BUCHI's latest NIR spectrometer, ProxiMate™, combines extreme ruggedness with high resolution, accuracy and intuitive ease of use.

See also chapters *Proximate protein* and *Proximate fat*

Sample preparation examples

Filtering
Diluting
Grinding
Mixing
Dissolving
Extracting
Precipitating
Ashing

Results and documentation

Evaluation and calculation of the results and the presentation in a report are the concluding steps after the laboratory work is completed. In the past, this was a purely manual and time-consuming procedure. Today, modern analytical instruments provide automated evaluation and calculation functions and connectivity with networks such as Laboratory Information Management System (LIMS).
The report of analytical results should contain the following information:

- The results of the determination with identification of the corresponding samples
- A reference to the applied standard operating procedures (SOP) or eventually a brief outline of the method
- Possible peculiarities observed during the test
- All deviations from the SOP

Moreover, statistical data such as relative standard deviation and specifications of the measurement uncertainty need to be included in a report of analytical results [10, 12].

Connection to system and archiving

Modern analytical instruments are connected to LIMS, a potential part of an enterprise resource planning system (ERP). For laboratory benchtop instruments, the connection is installed once and then continuously used afterwards. Interfaces such as RS232, USB, ethernet or the wireless Bluetooth are typically used [13].

See also chapter *Results documentation*.

Final product release

Based on the results and the completeness of the reports, the corresponding action is taken:

- All the results are within the specifications ▶ Product release
- One or more results are outside of the specifications ▶ Product rejection

The goods can be reworked or must be discarded.

Regulations

Food manufacturing is subject to regulations

Regulatory frameworks escort the food chain for the benefit of food quality, food safety, consumer protection as well as international and national trade. Hence, food producers and manufacturers are under pressure to deliver high quality food and to comply with national laws and global food safety and quality standards. They are supported by quality management systems such as ISO 9001, ISO 22000, GMP or FDA's Food Modernization Act (FSMA) [14]. Furthermore, it is becoming increasingly important for food manufacturers to be certified according to a food-specific, GFSI accepted standard [15]. The Global Food Safety Initiative (GFSI) benchmarks existing food standards against food safety criteria with the goal of standardizing certifications and eliminating multiple audits. The following selected GFSI-accepted standards are often used worldwide:

· BRC – BRC Global Standards
· FSSC 22000 – Food Safety System Certification
· IFS – International Featured Standards
· SQF – Safe Quality Food Institute

Method validation and standard operation procedures

Besides applying an appropriate quality management system, carrying out food analysis is the corner stone of food quality. The final goods inspection mainly confirms product quality.
Analytical methods are preferably based on common standards such as AOAC, ISO, EN, LFGB, etc. These reference methods describe the analytical procedure in detail and are transcribed into SOPs. Of course, reference methods may be adapted or other methods can be applied to a particular sample after validation.

The method validation process (see Figure 3) confirms that the method is suitable for the intended use and reliable results of established accuracy, precision and uncertainty are obtained with it [12, 16]. Validation is a requirement of good manufacturing practice (GMP) and other regulatory requirements. According to the FDA, the goal of method validation is to prove that an analytical method is fit for its purpose [17].

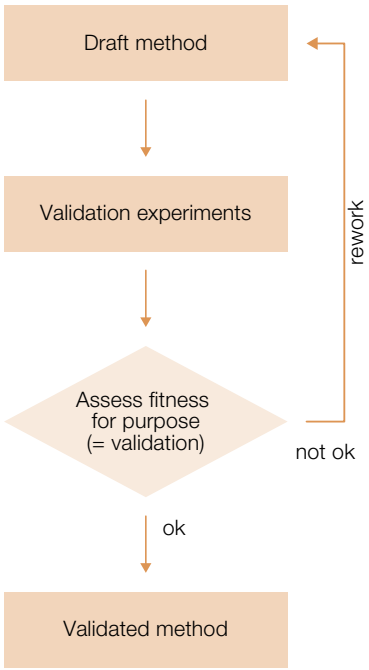


Fig 3: Method validation

Qualification and verification

In laboratories working according to GMP standards or similar regulations, it is mandatory to prove the correct functionality of the instrumentation used to perform an analysis. Instrument qualification (IQ), operation qualification (OQ) and performance qualification (PQ) also require verification of the equipment. The IQ/OQ verifications play a central role to ensure that a specific measuring accuracy and reproducibility is achieved and documented. Verification requires confirmation by examination and provision of objective evidence to prove that specified requirements, such as IQ and OQ, have been fulfilled [17]. Qualification procedures are an important part of a holistic GMP approach.

Equipment Qualification is divided into four steps:

1. Design Qualification (DQ)
2. Installation Qualification (IQ)
3. Operational Qualification (OQ)
4. Performance Qualification (PQ)

DQ	Defines the functional and operational specifications of the instrument.
IQ	Ensures that the instrument is received as designed and specified, that it is properly installed in the selected environment and that this environment is suitable for the operation of the instrument.
OQ	Demonstrates that the instrument will function according to its operational specifications in the selected environment.
PQ	Proves that the instrument consistently performs according to the specifications and is appropriate for routine usage.

BUCHI offers complete IQ/OQ documentation sets including test equipment. The available IQ/OQ test procedure is a complete solution offered by the BUCHI certifying system functionality.

Verification of Kjeldahl instruments

The distillation and titration step of the Kjeldahl method is tested by using a dry ammonium salt of known purity. The ratio of the determined and the expected nitrogen content is expressed as recovery in %. The distillation and titration step is verified when the recovery rate lies within the range of 98 - 102 %.

Thereafter, the performance of the digestion step is tested by digestion of a defined nitrogen containing substance which is known to undergo a complete digestion, e.g. glycine. Subsequently, the digested sample is distilled and titrated using the verified steps mentioned above.

Labelling

Since 1985, labelling of prepacked foods is described for instance by norms such as STAN 1 of Codex Alimentarius [18]. The nutrients declaration as well as other data such as list of ingredients, lot identification, storage instructions, net contents and date of expiry are mandatory requirements.
The nutritional analysis of proximates is based on reference methods that are structured by the type of product.

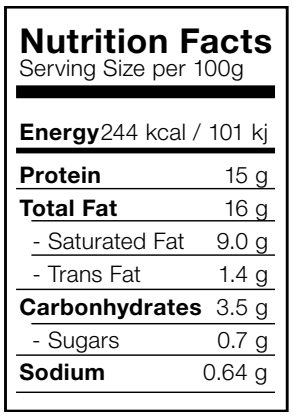


Fig 4: Nutrition labels [19]

Protein content

A main constituent of food is protein. The protein (nitrogen) content is determined by the Kjeldahl method. Table 1 depicts a list of selected methods.

Nitrogen by product group	Official method	BUCHI Application
Nitrogen content of milk	ISO 8968 AOAC 991.20	AN 054/2010 AN 020/2010
Nitrogen content of meat and meat products	ISO 937 AOAC 928.08	AN 023/2010 AN 114/2013
Nitrogen content of beer	AOAC 920.53	AN 024/2010 AN108/2013
Nitrogen content of flour	AOAC 920.87	AN 027/2010
Nitrogen content of whey protein and whey powder	ISO 8968 AOAC 991.20	AN 028/2010

Tab 1: Selected protein methods

Fat content

Fat is the most energy-dense macronutrient. Fats are extracted and determined gravimetrically. Some fat determination methods are listed in Table 2.

Fat by product group	Official method	BUCHI Application
Fat in meat and meat products	AOAC 963.15 EN 98/64/EC	AN E-416-E-816-SOX/2007
Fat in dairy products	AOAC 963.15 EN 98/64/EC	AN E-416-E-816-SOX/2007
Trans-fatty acid in food	JOCS Method 3.1.1-1996	SN 226/2016
Fat of edible oils and fat products	AOAC 938.06	AN E-816-SOX-006/2016
Fat in food and feed samples (manually hydrolysed)	AOAC 963.15	AN 018/2009

Tab 2: Selected fat determination methods

Results documentation

Current quality management systems require the archiving of results, instrument calibration data, operator related information and all other relevant specifications. Automated data transfer assures data integrity. Laboratory staff can work more efficiently leading to reduced labour costs.

Electronically stored data supports high data security, within LIMS for example, and can be tracked and recalled easily. BUCHI instruments can interface to LIMS systems.

- NIRWare, the operator software for NIR instruments, provides a bi-directional LIMS interface for flexible and secure data exchange and full traceability.
- The new ProxiMate™ NIR instrument comes with NIRWise™ software to automatically store all results (see Figure 5). The software can generate reports and with the report configuration tool, reports are customized to individual requirements.
- KjelLink PC software supports the preparation and evaluation of sample data in combination with the KjelMaster K-375. Analytical results can be imported and data statistically evaluated.

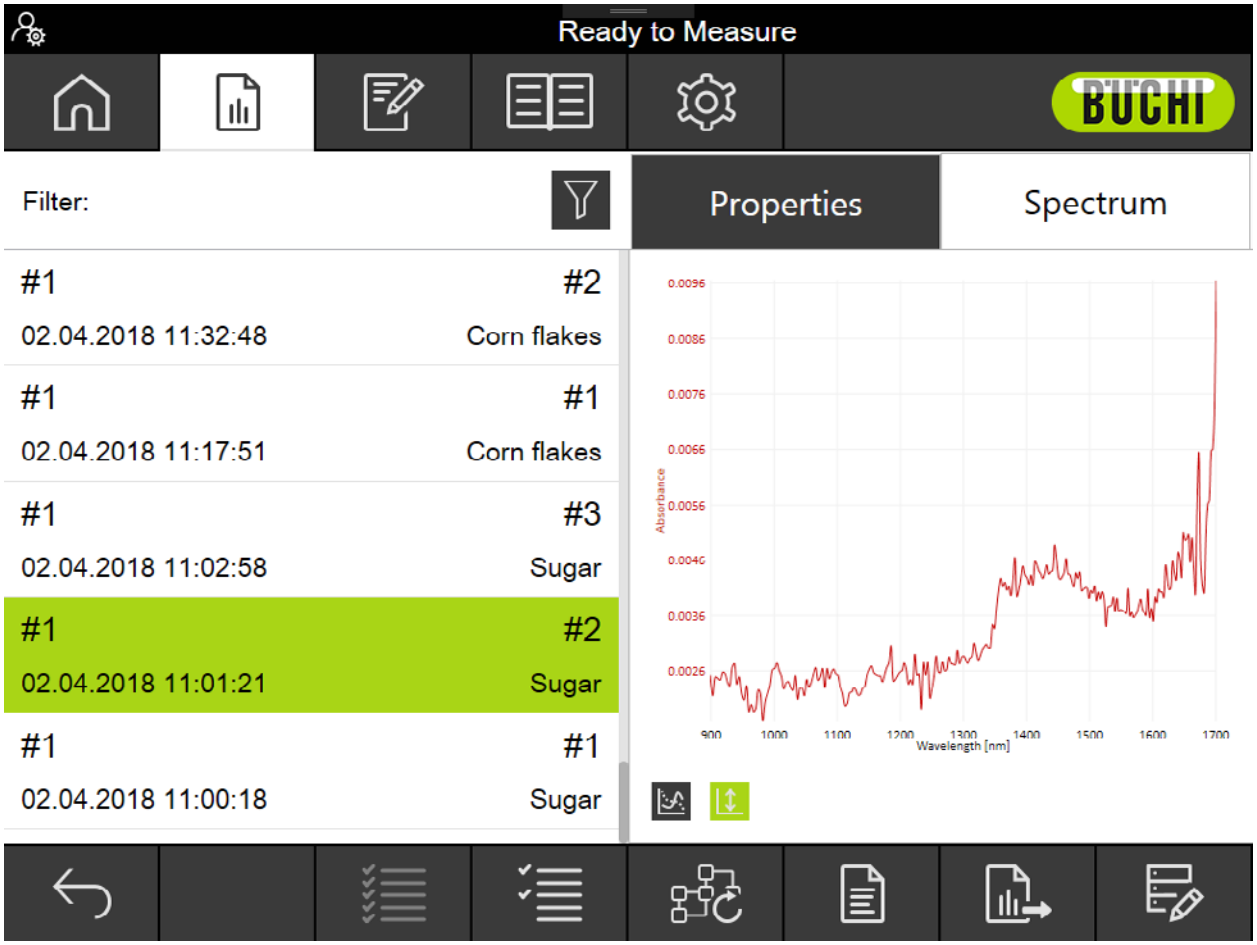
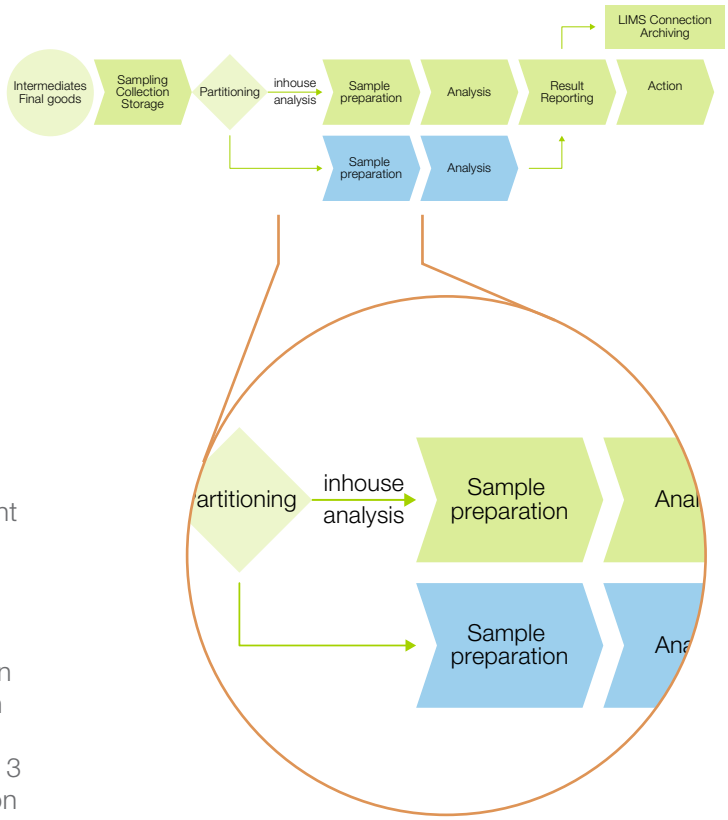


Fig 5: A screenshot of the NIRWise™ software

Sample preparation

Sample preparation in final goods control consists of two main disciplines.
· Sample homogenization
· Preparation for analysis



Sample homogenization

Sample homogenization is one of the inherent elements of sample preparation. The aim is to obtain a sample with a uniform, small particle size and an enlarged surface area leading to improved access of the reactant or solvent to the matrix and reduced variation of results. Thorough sample homogenization decreases the number of required replicates and therefore the overall analysis time. Table 3 depicts a selection of sample homogenization procedures, examples and respective devices.

Procedure	Aggregation	Example	Device
Mixing	Solids with pieces in varying size Dispersions	Cereals, grain	BUCHI Mixer B-400 ULTRA-TURRAX®
Grinding	Solids with pieces in varying size	Grain	Grinder
Sieving	Solids with varying in particle size	After grinding, mixing	Sieves with defined particles size
Stirring	Liquids with particles or tendency to separate	Yoghurt	With spatula On magnetic plate
Grating	Solids with varying surface	Chocolate	Grater
Melting	Solids with varying surface	Chocolate	Heating plate or water bath
Shaking	Homogeneous liquids Emulsion	Milk, shakes, dairy based drinks, dressings	Manual shaking Shaker

Tab 3: Procedure of sample homogenization

FAQs about sample homogenization

How to assure the sample is evenly homogenized?
· The sample can be evenly homogenized with the Mixer B-400.
· By operating the mixer in pulses, the particle size and the homogeneity can be assessed visually.
· Sieving the sample with a defined mesh size can assure only particles of a specific size used for the subsequent analysis.

How to avoid overheating of the sample and subsequent analyte decomposition or loss?
· The sample can be frozen, mixed with dry ice or liquid nitrogen prior to mixing.
· Overheating of the sample and separation of fat during comminution can be avoided by mixing in pulses.

How to avoid cross contamination during comminution?
· The beaker can be cleaned in a dishwasher.
· The blades can be disassembled for thorough cleaning.

In what way can a sample be prepared for heavy metal analysis?
· For the Mixer B-400, ceramic blades (accessory) can used for homogenization of samples. This eliminates contact with metal sources in the Mixer B-400.

Proximate protein

The Kjeldahl workflow

The entire Kjeldahl workflow consists of five steps as depicted in Figure 6.
For sample homogenization see previous chapter.

Fig 6: Kjeldahl workflow

Protein determination

Protein or more precisely nitrogen is determined by the Kjeldahl method, a three-step procedure including digestion, distillation and titration.

Work step	Action	Explanation
Digestion	· Weighing sample · Choosing catalyst · Digesting sample by IR or Block digester · Digesting sample by IR/block · Cooling of digested sample	The organically bonded nitrogen is converted into ammonium ions with concentrated sulfuric acid. Kjeldahl catalyst tablets raise the boiling point of the acid by sulfate salts and accelerate the process.
Distillation	· Diluting with water · Adding sodium hydroxide · Steam distillation into boric acid	The digestion mixture is alkalized with sodium hydroxide prior to distillation to free up the ammonia. The ammonia is steam distilled into an acidic receiver solution.
Titration	· Titrating with acid or base · Applying either potentiometric (direct or back) or colorimetric titration	The pH in the acidic receiver solution rises upon addition to ammonia. The nitrogen and protein contents are then determined by titration of the borate complex.

The determination is concluded by result calculation and reporting.

Results and reporting	· Applying the Kjeldahl Reports App · Connecting to LIMS · Using the PC software KjelLink	The nitrogen content is then calculated. To calculate the protein content, the nitrogen is multiplied by a sample specific protein factor.

FAQs about the Kjeldahl method

Background information on specific work steps or actions supports best practice methods and eliminates errors. The following FAQ section outlines a selection of the most important aspects of the Kjeldahl method.

Digestion

How to calculate the correct sample weight?

· The optimal nitrogen content ranges from 1 to 200 mg of nitrogen by glass sample tube. The limit of determination is 0.02 mg of nitrogen per sample tube.
· Use the weighing table supports to calculate the optimal sample weight. Start with step [1]. See Figure 7.

KjelOptimizer
To optimize your individual Kjeldahl method.
www.buchi.com/kjeloptimizer

Example for the usage of the weighing table

1	Expected %N of the sample must be selected (here 2 %)
2	Selection of the titrant concentration used (e.g. 0.05 mol/L)
3	Determination of the expected titrant consumption in mL ► here 3.6 and 14.3 mL
4	Result: For samples containing 2 % N and with titrant concentration of 0.05 mol/L, the expected consumption should be in a range of 3 – 17 mL. Therefore the weight must be between 0.125 and 0.5 g.

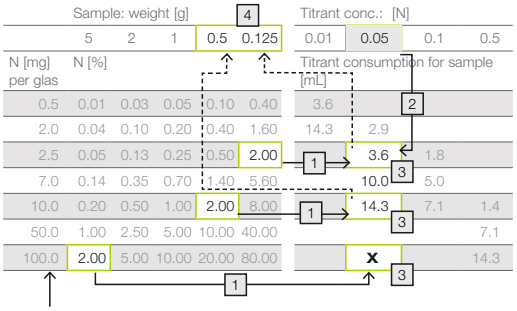


Fig 7: Weighing table

What are the optimal conditions for perfect digestions?

· Optimal ratio of sulfuric acid and catalyst is 2 mL of H_2SO_4 per 1 g of catalyst or 2 tablets per sample tube for easy handling.
· Boiling at 370°C.
· Tight system considering condensation zone (5 cm below the constriction, safety zone)*.
· Minimal time needs.

Get your Kjeldahl Practice Guide here:
www.buchi.com/applications/literature/request-form

Follow BUCHI's guidelines for digestion parameters (IR / Block). Observe 5 cm safety zone (see Figure 8).

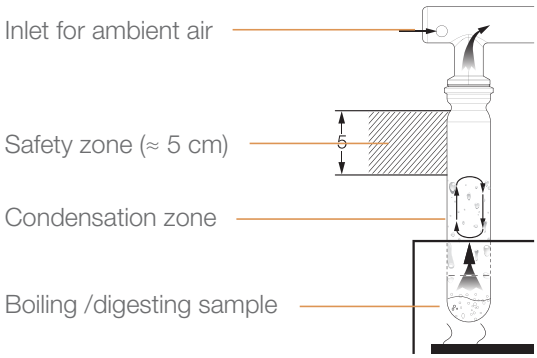


Fig 8: Intersection of a digester

Complete digestion yields to light green, translucent, uniform samples (see Figure 9).

Fig 9: Light green, translucent samples

How to determine the necessary amount of sulfuric acid?	Example: salami
Conversion of K_2SO_4 to $KHSO_4$. (All Kjeldahl catalyst tablets contain K_2SO_4)	2-3 mL
Consumption by organic matter (refer to Table 5).	+3.97 mL +1.51 mL +0.00 mL
Loss rate of acid due to evaporation (tight system considered): approx. 1 mL / h.	2 mL
Recommended remaining volume of acid to avoid losses of nitrogen.	> 10 mL
Total amount of sulfuric acid	**19 mL**

Organic matter	H_2SO_4 / g [mL]	Example: Salami	e.g. for 1.5 g weight (weight · org. matter):
Fat	9.7	27.3 %	$1.5 \cdot 9.7 \cdot \frac{27.3}{100} = 3.97$ mL
Protein	4.9	20.6 %	$1.5 \cdot 4.9 \cdot \frac{20.6}{100} = 1.51$ mL
Carbohydrates	4.0	0.0 %	$1.5 \cdot 4.0 \cdot \frac{0.0}{100} = 0.0$ mL

Tab 5: Sulfuric acid consumption

Article	Composition	Weight
Titanium # 11057980	3.5 g K_2SO_4 / 0.105 g $CuSO_4 \cdot 5\ H_2O$ 0.105 g TiO_2	3.71 g
Benefit: Recommendation:	Time saving Optimal compromise between environmental and performance priorities.	
Titanium Micro # 11057981	1.5 g K_2SO_4 / 0.045 g $CuSO_4 \cdot 5\ H_2O$ 0.045 g TiO_2	1.59 g
Benefit: Recommendation:	Reduced chemical amount Same as Titanium (11057980) but for semi-micro & micro-Kjeldahl applications.	
Missouri # 11057982	4.98 g K_2SO_4 0.02 g $CuSO_4 \cdot 5\ H_2O$	5 g

Tab 6: Kjeldahl catalysts

What is the best choice of Kjeldahl catalyst?

The choice of the Kjeldahl catalyst should be based on the following aspects:
· Applicative fit
· Minimal time needs
· Environmental-friendly
· Amount of sample
· Reduction of foaming

Kjeldahl Tablet Configurator
Use the Configurator to select the Kjeldahl Tablet for digestion that suits your needs best.
www.buchi.com/tablet-configurator

best@buchi No. 65
Decode the mysteries of the Kjeldahl Tablets
www.buchi.com/media-center

See table 5 in the Kjeldahl Practice Guide:
www.buchi.com/applications/literature/request-form

How is foaming minimized?

· Add one Kjeldahl tablet “antifoam” or stearic acid.
· Slow increase of the digestion temperature (heating ramp)

Distillation

Why is the acidic solution diluted with water after digestion?

· Prior to distillation the sample must be cooled to 50 – 100°C.
· The dilution with water attenuates violent reactions during alkalization with sodium hydroxide.

What is the mode “IntelliDist” used for?

The potentiometric distillation enables the distillation mode “IntelliDist” (KjelMaster), which assures ideal operating temperature for the distillation step. Therefore, the countdown of the set distillation time only starts after the operating temperature is attained. With single samples or sample list measurements, this mode guarantees result accuracy from previous runs as any errors deriving from an intermediate cool-down of the device are eliminated.

Titration

What concentration to choose of boric acid as titrant receiver?

The amount of sample and the concentration of the titrant should be optimized to attain a titrant volume in the range of 3 to 17 mL (burette volume: 20 mL). For very low nitrogen content, choose 2% boric acid with potassium chloride (3 g/L) as a receiving solution to achieve lower detection limits.

Nitrogen content absolute	Nitrogen content relative	Protein content relative (Protein factor 6.25)	Sample size	Boric acid concen-tration	Titrant concen-tration	Titrant volume
0.02 mg	20 ppm		1.0 g	2 % (+3 g KCl/L)	0.005 N	2 mL
0.1 mg	100 ppm		1.0 g	2 %	0.005 N	3 mL
1 mg	0.2 %	1 %	0.2 g	2 %	0.01 N	8 mL
5 mg	1 %	6 %	0.5 g	2 %	0.1 N	4 mL
10 mg	1 %	6 %	1.0 g	4 %	0.1 N	8 mL
20 mg	2 %	13 %	1.0 g	4 %	0.1 N	14 mL
50 mg	5 %	31 %	0.4 g	4 %	0.1 N	14 mL
100 mg	10 %	63 %	1.0 g	4 %	0.5 N	14 mL
100 mg	20 %		0.5 g	4 %	0.5 N	14 mL
200 mg	20 %		1.0 g	4 %	0.5 N	28 mL

Tab 7: Boric acid and titrant concentration, source OM K-375

Why does adding potassium chloride (KCl) to the receiving solution help to reach lower LOD / LOQ of nitrogen?

- The addition of KCl minimizes the effect of stirring, which is particularly relevant at the end of the titration when the amount of distilled water is high and the stirring becomes even more important to detect changes in pH.
- The addition of KCl lowers the measured pH close to the set point (pH: 4.65)
  ▶ Less titrant is needed (also resulting in lower blank values).
- The addition of KCl effectively creates smaller pH intervals prior to reaching the set point (pH: 4.65)
  ▶ Allowing faster titration rate.

See also best@buchi “How to Achieve Low Detection and Quantification Limits for the Nitrogen Determination with Kjeldahl”, 58/2010
www.buchi.com/sites/default/files/downloads/Low_detection_and_quantification_limits.pdf

What are the best conditions to determine low nitrogen concentrations?

2% boric acid with a concentration of 40 mM KCl.
Although the detection and quantification limits are slightly lower with 20 mM KCl and the blank values are comparable, the mean values of the recoveries as well as the pH shift are optimal with 40 mM KCl.

See also best@buchi “How to Achieve Low Detection and Quantification Limits for the Nitrogen Determination with Kjeldahl”, 58/2010
www.buchi.com/sites/default/files/downloads/Low_detection_and_quantification_limits.pdf

Troubleshooting the Kjeldahl method

Fast troubleshooting procedures are needed to eliminate systematic errors in the measurement workflow and to shorten any down-times impacting the immediate release or rejection of the final good. The following outline shows the most relevant causes and corrective measures identified for the Kjeldahl method.

In case the nitrogen / protein content is too high…

Location / work step	Cause	Corrective measure
Air in the titration system (burette, tubes)	Instead of titrant, “air” is dosed which impacts the final titrant volume needed to reach the SET point.	· Refill the burette and release any air · Check screw connections · Observe refilling and check for complete filling of the tube · Verify recovery with standard substance
Carry over of sodium hydroxide during distillation	Transfer of sodium hydroxide into the receiving solution immediately increases the pH.	· Observe reaction and set reaction time · Dilute digested sample with more water · Optimize volume of sulfuric acid
Wrong titrant concentration	Lower concentrated titrant was effectively used while higher concentrated one was entered in the calculation.	· Compare titrant concentrations in use and in calculation · Adjust concentrations · If titrant was changed, refill burette to ensure the labelled titrant concentration · Check titrant certificate

In case the nitrogen / protein content is too low…

Location / work step	Cause	Corrective measure
Incomplete digestion - no color change of digested sample to translucent and light green	· Digestion time too short · H_2SO_4 insufficient · Incorrect ratio H_2SO_4 / catalyst	· Increase digestion time · Adjust volume of H_2SO_4 (refer to FAQs) · Adjust ratio (refer to FAQs)
Digestion: dried, partially crystallized sample	· Remaining H_2SO_4 too low (incomplete dissolution) · Scrubber suction capacity too high	· Should be > 10 mL H_2SO_4 · Adjust scrubber suction capacity (refer to operation manual BUCHI Scrubber K-415)
Incorrect addition of sodium hydroxide solution prior to distillation	· Concentration of sodium hydroxide incorrect · Volume of sodium hydroxide too low	· Verify correct concentration of sodium hydroxide (32%) · Adjust volume of sodium hydroxide until color change is visible
Sample amount (content nitrogen) too high	· Nitrogen content out of specification range (0.02 – 200 mg per sample tube)	· Adjust sample amount (refer to FAQs)
Leakage during digestion	· Too high temperature (high condensation zone near the neck of the digestion tube) and splashing or foaming sample	· Assure correct boiling temperature by adjusting the amount of catalyst tablets and avoid splashing or foaming by using anti-foam tablets
Leakage during distillation	· Loss of nitrogen during distillation	· Check and tighten connection between splash protector and condenser · Replace seal if worn
Wrong titrant concentration	· Higher concentrated titrant was effectively used while lower concentrated titrant was entered in the calculation	· Compare titrant concentrations in use and in calculation · Adjust concentrations · If titrant was changed, refill burette to ensure the labelled titrant concentration is filled

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In case the reproducibility is deficient…

Location / work step	Cause	Corrective measure
Improper sample homogeneity	Inhomogeneous sample	· Homogenize sample thoroughly · Refer to chapter *Sample homogenization*
Sample weighing	Ensure proper weighing conditions	· Calibrate and check balance · Use weighing boats
Air in the titration system (burette, tubes)	Instead of titrant, “air” is dosed which impacts the final titrant volume needed to reach the SET point.	· Refill the burette and release any air · Check screw connections · Observe refilling and check for complete filling of the tube · Verify recovery with standard substance
Stirrer not working	Stirrer changes revolutions per minute	· Clean stirrer · Replace stirrer
Titrant dosing fluctuating	Incorrect positioning of the titration dosing tip	· Check position and eliminate block · Correct position for non-constraining dosing
Sample transfer autosampler – distillation unit	Dip tube autosampler · Blocked · Loose · Too short · Defective	· Check the autosampler dip tube and correct deficiencies

Proximate fat

Workflow fat extraction

Two gravimetrical methods for fat determination are known: total fat and crude fat. Total fat determination consists of two steps, hydrolysis and subsequent extraction. Direct extraction is applied for crude fat determination. The workflow is shown in Figure 10. Sample homogenization is explained in a previous chapter.

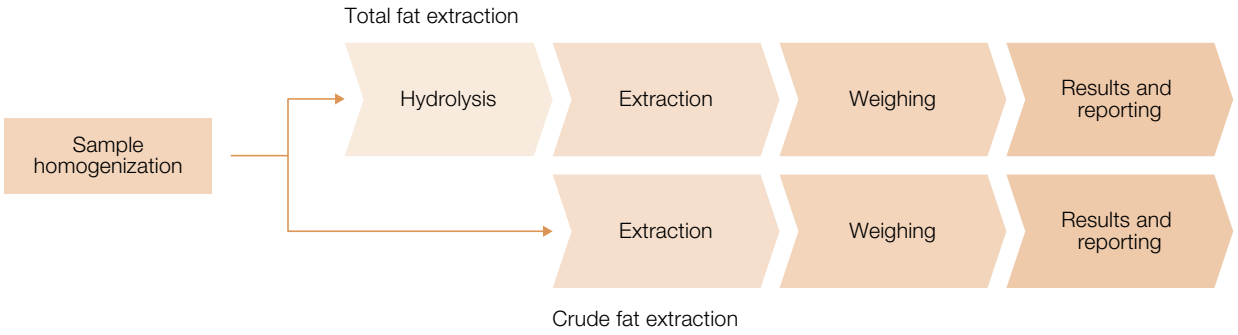


Fig 10: Workflow of total and crude fat determination

Total fat

Steps	Actions	Explanations
Hydrolysis	Mixing sample with Celite® · Adding hydrochloric acid · Boiling · Filtering through Celite® · Mixing Celite® layers · Drying	Proteins are hydrolyzed by boiling with hydrochloric acid. Cell walls are destroyed and physically encased or chemically bound fats are released and become accessible for extraction.
Extraction	Weighing dried beakers · Adding solvent into beakers · Placing sample into chamber · Programming extraction · Extraction · Drying beakers	The boiling solvent dissolves fat from the sample. After the extraction, the solvent is evaporated and the beakers dried to constant weight.
Weighing	Weighing dried beakers	The weight of the beaker is determined and the fat content is calculated.

Crude fat

Prior to extraction, the sample must be mixed with sand and dried in a drying cabinet at 103°C. Optionally, sodium sulfate can be added to the sample as a drying aid. The dried sample can be directly extracted as described in Table 8.

Extraction methods

BUCHI offers a broad variety of extraction methods including automated Soxhlet, hot extraction (Randall or submersion method) and economic continuous extraction (Twisselmann). The parameters are summarized in Table 8.

	Soxhlet extraction [SOX]	Hot extraction [HE] (Randall)	Economic continuous extraction [ECE] (Twisselmann)
Solvent volume	120 mL	80 mL	70 mL
Extraction	120 min	5 min	50 min
Rinse	5 min	30 min	-
Drain Intervall	-	15 min	-
Dry	25 min	5 min	10 min
Total time	150 min	40 min	60 min

Tab 8: Parameters of extraction methods

The principle of the methods is demonstrated in the following video
https://youtu.be/cF4_RRNIGAc

FAQs about fat determination

Hydrolysis

What does the hydrolysis do?

- Proteins are hydrolyzed and cell walls (plant material) broken apart.
- Physically encased and chemically bound fats are released and made accessible to the solvent during extraction.

Why is it important to adjust the temperature of the rinse water?

- Warm water (40-60°C) dissolves sugars and efficiently removes these along with remaining acids.

What is the Celite® used for?

- Celite®, also known as diatomaceous earth, disperses the sample and adsorbs fat during hydrolysis.
- After the hydrolysis, specifically filtration of the hydrolysate, the fat is extracted from the Celite® layer (hydrolysis residue).

What is important to consider for the drying of the hydrolyzed sample?

- Water must be thoroughly removed as it would repel lipophilic solvents and therefore hinders the extraction.
- Loosen the pulp carefully with a spatula while leaving the sand bed intact.
- Follow the heating guidelines for the drying cabinet and microwave to avoid oxidation of the fat.

How to minimize foaming?

- Foaming can be minimized by adding a few droplets of hydrochloric acid to the boiling hydrolysate.
- Make sure the heating level is set between 2 to 3

What to do when the sample is not aspirated properly?

- Thoroughly check the tightness of the connections on the water jet pump / vacuum pump and the hose connections
- Check for proper vacuum, e.g. Brand® 159600 ≤10 mbar
- Finished positions can be closed with lids to increase the vacuum of individual positions.

Extraction

What parameters influence the result?

- The type of solvent, e.g. diethyl ether, petroleum ether, hexane and chloroform, has to be chosen for the specific application.
- The number of cycles is essential for Soxhlet, whereas the extraction time matters for hot and economic continuous extraction.

How does the cooling to ambient temperature of the extraction beaker influence the results?

It is of importance to dry and cool down the extraction beakers for the same amount of time, for instance 30 minutes for drying and 60 minutes for cooling, respectively, prior to and after extraction to avoid temperature-related weight differences.

What is the difference between the three extraction methods?

- During Soxhlet extraction, the sample is placed in a thimble or glass sample tube with frit into the Soxhlet chamber. The fat is extracted by the condensed solvent which percolates through the sample.
- During hot extraction, the sample sits in the boiling solvent under reflux.
- For the economic continuous extraction, the sample is located in the ECE chamber and is heated by the hot solvent vapor while the condensed solvent trickles through the sample.

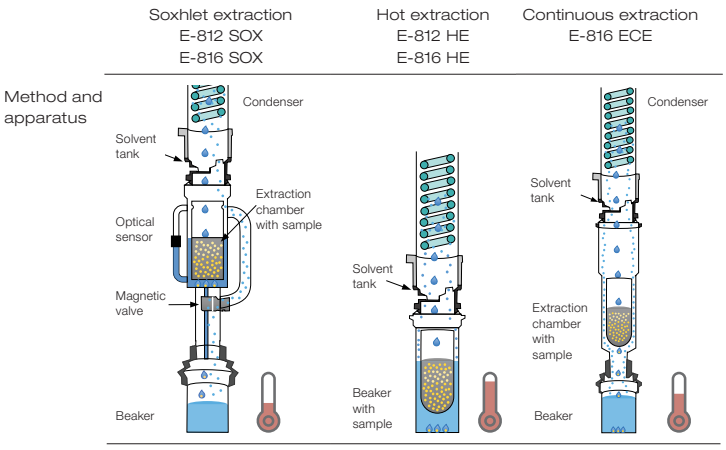


Fig 11: Comparison of the three extraction methods

How do I calculate the proper sample weight?

- Ideally the extracted fat should be in the range of 0.7 g to 1.0 g. However, for filtration of sensitive samples, the extractable fat can be as low as 100 mg. The sample weight should not exceed 10 g.
- As a rough guideline the sample weight can be taken from the following table:

Fat content (%)	Sample weight (g)
<10	7-10
10-20	3.5-7
20-50	1.5-3.5
50-80	1-1.5
80-100	0.7-1

Weighing

Why is it important to maintain the reproducibility of the conditioning procedure?

- The weight of the beaker depends on the temperature.
- Maintaining the same temperature (oven / ambient) and time for drying and cooling is crucial for reducing the variability of the results, both for improved repeatability and reproducibility.

Troubleshooting fat determination

Too high fat content

Cause	Corrective Measure
Drying of extract was not sufficient	· Dry to a constant weight
Oxidation of fat due to too high temperature during the drying step	· Reduce the duration of the drying step or lower the heater setting · Check temperature setting of the oven (103°C)
Some fats and oils are very heat sensitive (e.g. sunflower oil)	· Lower the heater setting and dry the extract at lower temperatures and reduced pressure in a vacuum oven
Celite® was washed out during extraction	· Loose the pulp carefully before drying the hydrolyzed sample · An upper sand bed on top of the Celite® layer avoids washing out Celite® and sample.
Impurities in solvent	· Use clean solvent, either new solvent or freshly distilled-solvent · Avoid the outer tube of the solvent draining hose that comes into contact with the solvent (plasticizers could be dissolved)

Too low fat content

Cause	Corrective Measure
Loss of sample during hydrolysis step	· Wash the hydrolysis tube with several aliquots of equal volumes of water so that the sample is transferred quantitatively into the glass sample tube · Adjust the temperature of the water (40-60°C). Too hot water can result in a loss of fat, too cold water cannot dissolve remaining sample sufficiently for a complete sample transfer
Incomplete extraction	· Set the level sensor to the top end of the sample to assure complete immersion in solvent · Choose extraction time and cycles according to setting specifcations (Table 8) · Avoid accumulation of solvent on top of the sample (see accumulation of solvent)
Differences in beaker temperature	· Make sure to use the same settings for drying (time and temperature) and cooling (time) the beaker when weighing prior to and following the extraction

Too large variation

Cause	Corrective Measure
Too small sample weight	· Use the recommended sample weights (see chapter FAQs) · If a sample is very inhomogeneous, increase the sample weight
Inhomogeneous sample	· Use a professional mixer, e.g. Mixer B-400 or mortar and pestle
Incomplete extraction	· Please refer to the section “incomplete extraction”
Low fat content	· The sample weight can be increased up to 10 g

Boiling retardation

Cause	Corrective Measure
Vapor building accumulating inside hot solvent due to fast heating and/ or surface tension	· Always use boiling aid, e.g. boiling stones, pearls etc.

Accumulation of solvent on top of the sample

Cause	Corrective Measure
The sample was dried insufficiently	· Assure the glass sample tube with the sample is dried sufficiently either in a microwave (17 minutes at 640 W and 13 minutes at 400 W) or a drying cabinet set to 103°C for at least 8 hours
Too fast evaporation of solvent	· Assure that the heater is set to 100% and that the right solvent from the library is selected · Depending on the altitude, the heater setting needs to be adjusted
Compacted Celite® layers	· Mix the Celite® layers carefully and thoroughly using a spatula prior to drying
Frit is blocked	· The frit must be rinsed thoroughly to remove any remaining sand and Celite® prior to cleaning in a dishwasher. Please refer to the glass sample tube cleaning guide. · If the frit cannot be unblocked, the glass sample tube must be replaced.

Contaminates and residues

To assure food safety of food and beverages, the level of additives, contaminants and residues must be monitored. Contaminants and residues could penetrate into our food from various sources. The analyses of contaminates and residues demand for flexible classical or pressurized extraction solutions. Food additives are substances added to food for chemical, physical or physiological effects, such as microbiological preservation, color stability or flavor enhancement. Their steam volatile characteristics make them separable from the food matrix by direct steam distillation.

Contaminants are chemical substances that have not been intentionally added to food. These substances may be present in food because of the various stages of its production, processing, or transport. They also might result from environmental contamination. Contaminants could pose a risk to animal and human health.

Residues of some chemicals are unintentionally present in some foods because of food production processes such as spraying of crops with pesticides, use of veterinary medicines for food-producing animals, as well as food packaging.

The following provides a summary of relevant contaminants and residues being monitored on a regular basis to assure food safety.

Image	Analyte	Matrix	Worksteps	Norm / Publication
	· Sulfur dioxide (Sulfite)	· Seafood (shrimps, canned clamps) · Potato, cherry tomato, hot pepper powder · Wine, Beer · Juice · Jams, jellies and fruit toppings	· Sample homogenization · Steam distillation, SO_2 absorption vessels · Back-titration	· European Regulation 2003/89/EC · AOAC 990.28 · DIN 32645:2008
	· Total Volatile · Basic Nitrogen (TVB-N)	· Seafood · Fish	· Sample homogenization by cutting · Steam distillation / steam regulation · Titration	· European Regulation 95/149/EC
	· Pesticides	· Chili and pepper spices	· Sample preparation · Pressurized solvent extraction · GPC Clean-up · GC-MS/MS	· US Environmental (July 24, 2007), What is a pesticide? EPA Gov. retrieved on September 15, 2007

Image	Analyte	Matrix	Worksteps	Norm / Publication
	· Veterinary drugs	· Pork Muscle	· Sample pre-treatment · Sample homogenization · Pressurized solvent extraction · UPLC-MS/MS	· COMMISSION REGULATION (EU) No 37/2010 · Shen et al.: Journal of AOAC International Vol. 86, No. 3, 2003
	· Mineral oil hydro-carbons · (MOSH, MOAH	· Pasta	· Pre-cleaning · Sample homogenization · Pressurized solvent extraction · LC-GC	· Scientific Opinion on Mineral Oil Hydrocarbons in Food, EFSA Journal 2012, 10(6): 2704 · Removal of mineral oil migrated from paperboard packing during cooking of foods in boiling water – S. Biedermann-Brem, K, Grob, Eur. Food Res. Technol (2011) 232: 1035-1041 · Rapid and sensitive solid phase extraction-large volume injection-gas chromatography for the analysis of mineral oil saturated and aromatic hydrocarbons in cardboard and dried food - S. Moret, L. Barp, G. Purcaro, L. Conte, Journal of Chromatography A, 1243 (2012) 1-5 · Is recycled newspaper suitable for food contact materials? Technical grade mineral oils from printing inks - M. Biedermann, K. Grob, Eur. Food Res. Technol. 230 (2010) 785 – 796

Image	Analyte	Matrix	Worksteps	Norm / Publication
	· Bisphenol A	· Canned vegetables	· Sample homogenization · Lyophilization · Pressurized solvent extraction · LC-MS	· Directive 2004/19/EC of 1 March 2004 amending Directive 2002/72/EC · European Commission: European Union Risk Assessment Report, 4,4'-isopropylidenediphenol (bisphenol-A), 2003 · EFSA: Scientific Opinion on bisphenol A: evaluation of a study investigating its neurodevelopmental toxicity, review of recent scientific
	· Brominated flame retardants (BFRs), polybrominated · diphenyl ethers (PBDEs)	· Seafood · Fish	· Sample homogenization · Sample preparation · Pressurized solvent extraction · Parallel evaporation · GPC Clean-up · GC-MS/MS	
	· Fat · Dioxins · Furans · PCBs	· Eggs · Chicken and pork meat · Fish · Dairy	· Sample homogenization · Lyophilization · Pressurized solvent extraction · Parallel evaporation · GC-HRMS	· FDA Guideline for the Determination of Residual Moisture in Dried Biological Products, 89D-0140. US Food and Drug Administration · EU Commission regulation 1259/2011 regards maximum levels for dioxins, dioxin-like PCBs and non-dioxin-like PCBs in foodstuffs · Chinese National Food Safety Standard, GB 5009.3-2016 · United States Department of Agriculture, Food Composition Databases. https://ndb.nal.usda.gov/ndb (accessed Apr 12, 2017)

NIR analysis

Advantages of NIR analysis

A brief summary

Within a brief measuring time of a few seconds, NIR analysis can determine several food constituents simultaneously. The technique is non-destructive and suitable for different sample types from powder to pastes, granules, liquids and more.
NIR analysis has been diligently explained in volumes 1 and 2 of this Food Process Analytics guidebook.

The following topics have been covered already:

- Principles
- Techniques
- Applications
- Workflow

Sample preparation and analysis step

Thanks to the advantages of NIR analysis, the workflow steps of sample preparation and analysis become easy and fast to carry out. Often, almost no sample preparation is required especially in cases where the occurring sample variations are covered by the calibration procedure and samples are not subjected to surface treatment (such as the application of palatability agents) prior to analysis.

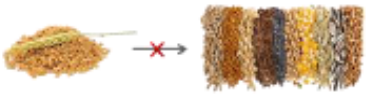
FAQs and typical issues with NIR

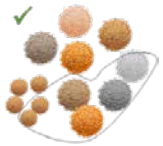
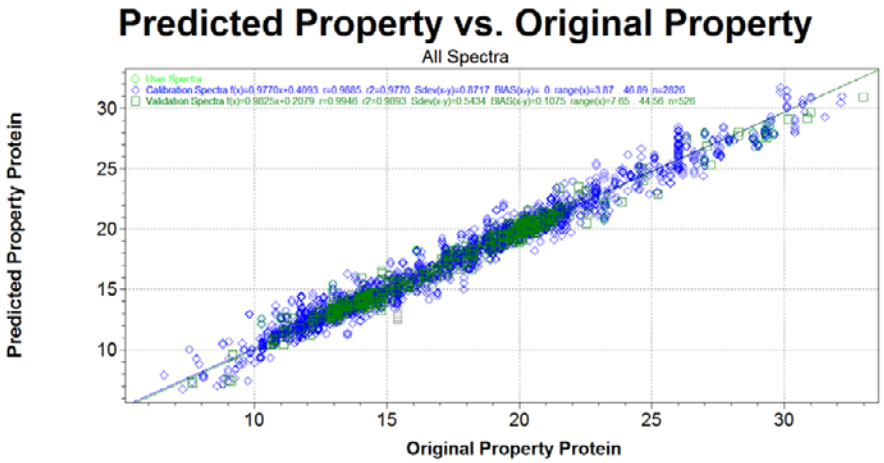
To complete the NIR analysis theme, a chapter of typical issues and “how to …” questions around the NIR analysis is included here [20].

<table>
<tr><th>Issue</th><th colspan="4">Explanation</th></tr>
<tr><td>Why consider NIR spectroscopy for analysis?</td><td colspan="4">· NIR spectroscopy (NIRS) is a fast, non-destructive analytical method. Almost any kind of sample can be scanned within seconds with minimal or even no preparation at all. NIRS can be used to verify the identity of a sample and to quantify multiple chemical and physical properties simultaneously. NIRS is suitable for off-, at-, on- and in-line use.

<table>
<tr><th colspan="2">Batch measurements</th><th colspan="2">Contiuous measurements</th></tr>
<tr><td>Off-line</td><td>At-line</td><td>On-line</td><td>In-line</td></tr>
</table></td></tr>
<tr><td>Do BUCHI FT-NIR spectrometers need to be calibrated?</td><td colspan="4">· In NIR spectroscopy, a calibration model is generally required to interpret sample data. This model needs to be developed during setup of the application. BUCHI’s NIRCal® Software makes it straight forward to develop robust and reliable calibrations by correlating NIR data with reference analytics.</td></tr>
<tr><td>Are BUCHI NIR spectrometer available for work in any environment?</td><td colspan="4">· Yes.
· The NIRFlex® N-500 is a benchtop spectrometer designed for laboratory use.
· The NIRMaster™ and NIRMaster™ Pro provide high ingress protection and are ideal for production sites.
· The ProxiMate™ is a robust, compact and easy to use at-line NIR instrument for the food and feed industry.

  </td></tr>
<tr><td>How many parameters can be analyzed simultaneously?</td><td colspan="4">· The number of parameters measurable within a calibration model is only limited by computing power and complexity of the analysis. It is not unusual to analyze 15-30 properties at the same time.

  </td></tr>
</table>

Issue	Explanation
What are the right samples?	· Samples to be analyzed should be of the same nature as the calibration samples. · Explanation: a calibration developed to predict protein content in wheat is not suitable to predict protein in other types of grains.  Calibration set too narrow  Good calibration set
How to select good calibration samples?	· It is important that calibration samples are representative and evenly distributed over the entire range of expected samples. · For instance, a small set of similar samples does not provide accurate calibration for samples with a wider variation of characteristics.
How to validate and monitor calibrations?	· Validate your calibration by comparing NIR predicted results with the reference values of a set of random samples. · Ideally, the predicted results do not deviate much from the reference values. This way you can monitor your calibration performance regularly and apply corrections if necessary. 

Applications and selected industries

The applications
Dwell on the application galore of BUCHI.
Find out how to simplify daily laboratory activities.
Learn about the optimal use of BUCHI's instruments.

More than 200 applications from food, beverage, feed, biotech and pharmaceuticals are currently available on the Application Finder.

The Application Finder is constantly updated and expanded.

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www.buchi.com/applications/finder

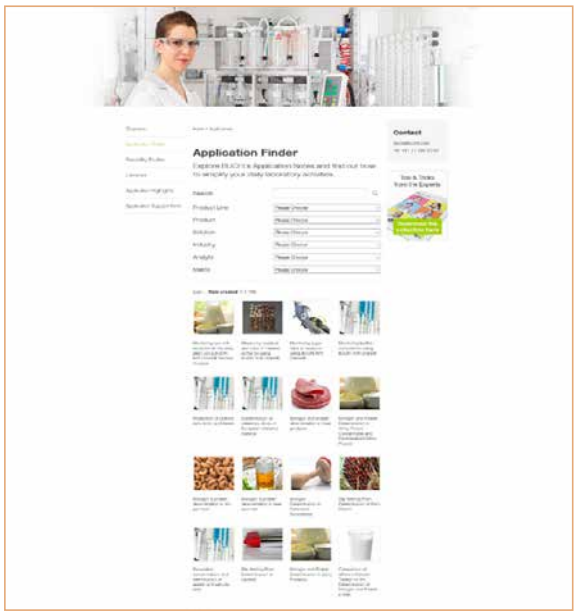


Fig 12: Application Finder start page

How to navigate the Application Finder
The navigation is easy.

· Enter your keyword in the Search field
or
· Choose from the selection fields

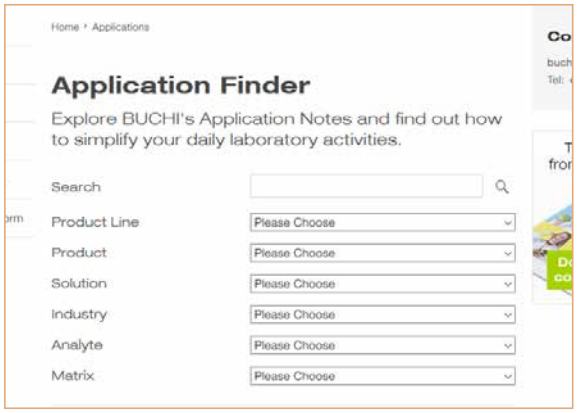


Fig 13: Application Finder fields

More to find there

The Application Finder page contains more information to assist users in the laboratory.

Application highlights
The application of the month is featured since 2013.
Prominent solutions are presented for all kinds of fields of application.

Literature
Request a specific guide or select from the existing list.

Application support form
Request your specific application. Specialists will provide you with competent application support.

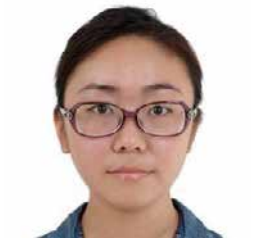
Feasibility studies
Get in direct contact with a team of qualified experts for a customized feasibility study.

David Vinzent, Kjeldahl

Maren Sander, Extraction

Dr. Urs Hartfelder, NIR

Li Lan, Kjeldahl



Yang Yi Chen, Extraction

Selected food industries

BUCHI Kjeldahl, extraction and NIR instruments are used for the analysis of all kind of food products and ingredients from various industry segments. The following list of segments of the food industry are the most prominent ones.

Fats and oils
The reference method for fat content determination is the Weibull-Stoldt method.

Milk and dairy
The three major constituents of milk and dairy products are moisture (or dry matter), protein and fat.

Milling and bakery: grains and flours
Protein, ash and moisture content of flours and grains are frequently measured quality parameters.

Meat and fish
Protein, fat, and moisture content are three major quality parameters of meat, meat products, sausages, fish and fish products.

BUCHI products & solutions

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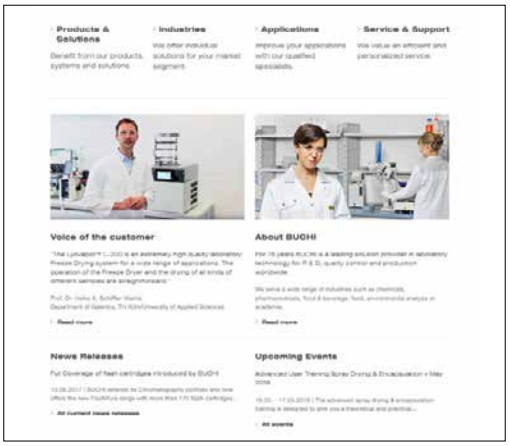

Further guides and handbooks

Kjeldahl Knowledge Base
Decode the mysteries of nitrogen and protein determination according to Kjeldahl,
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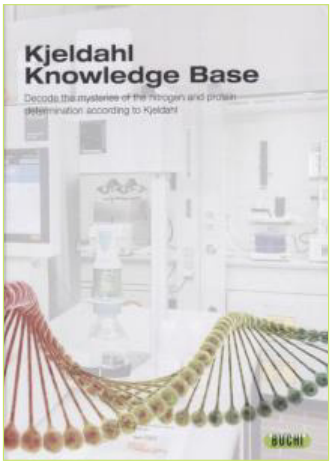
www.buchi.com/kjeldahl_knowledge_base



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www.buchi.com/guidebook-to-proximate-analysis-by-buchi

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To Make More Money in Feed Production
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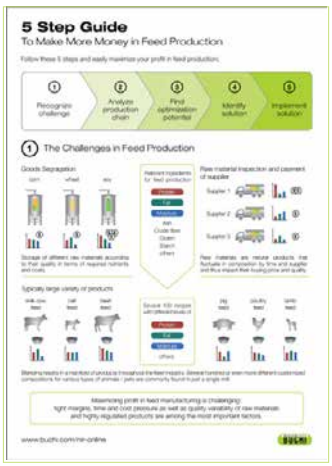

Tips & Tricks from the Experts
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Appendix 1 – NIR spectroscopy and how it works

Spectroscopy in general is a well-accepted and fast measuring technique. It is used for quantitative as well as qualitative analyses. Chemometrics are applied to allow for easy and cost efficient NIR analysis.

A glimpse of theory

Spectroscopy uses light, i.e. electromagnetic radiation, to analyze materials. Light interacts with matter because it can absorb and reflect light. The energy transfer between light and matter (= sample) is described in the spectrum.

The energy of light is defined as $E = h \bullet \nu$

where	h	Planck constant [J s]
	ν	frequency of the light [s-1]

The entire electromagnetic radiation is divided into several sections. Every section is covered by different specific spectroscopic techniques. For example, X-rays have wavelengths of only a few nm and visible radiation (VIS) covers the range between 350 nm and 780 nm. The range between 780 nm and 2500 nm is referred to as near-infrared radiation.

Absorbed infrared radiation causes the bonds between the atoms of the sample molecules to vibrate: bonds between two atoms start to stretch and bend. Bonds with hydrogen (e.g. C-H, O-H) show the largest vibrations, because hydrogen is the lightest atom. Most molecules have several bonds which all can vibrate. Many vibrations are not independent but are coupled in fact.

In addition, chemical and physical properties of all other compounds present in the sample influence the NIR spectra. Small sample-to-sample differences within a sample series can cause spectral differences as well. Therefore, the NIR spectrum depends on more than one variable simultaneously. This is called multivariate.

Exploit of NIR for food analysis

Bonds of carbon (C), nitrogen (N), oxygen (O) and sulfur (S) atoms with hydrogen (H) occur with great numbers in molecules of foodstuff. Oleic acid (a fatty acid), lactose (a sugar) and casein (a protein) are just 3 example molecules.

Because NIR spectroscopy is sensitive to these bonds, it is an advantageous method for food analysis.

Chemometrics

In practice, NIR absorbance is measured over a range of wavelengths. This spectrum is correlated with sample content parameters. Since the spectrum covers several single wavelengths – in fact hundreds of wavelengths – the data is multivariate.
Multivariate data requires multivariate calibration. The relationship between the independent and dependent variables is described by an appropriate multivariate calibration model. Independent variables are the NIR absorbances at selected single wavelengths. Dependent variables are the concentrations of the sample components, e.g. fat content, protein content or lactose content.

Chemometrics filter spectral information correlating to a certain property and separate them from unwanted information such as noise. In the end, the relation between the NIR absorption and the concentration of the analyte (e.g. fat, protein, lactose) is established and can be applied to routine determinations.

Calibration software packages, e.g. BUCHI NIRCal, allow the development of qualitative and quantitative calibrations. The software offers numerous chemometric algorithms, data pre-treatment and visualization tools to analyze spectroscopic data. Graphical visualization is interactive and includes spectral data, chemometric parameters and results in different plot types [23].

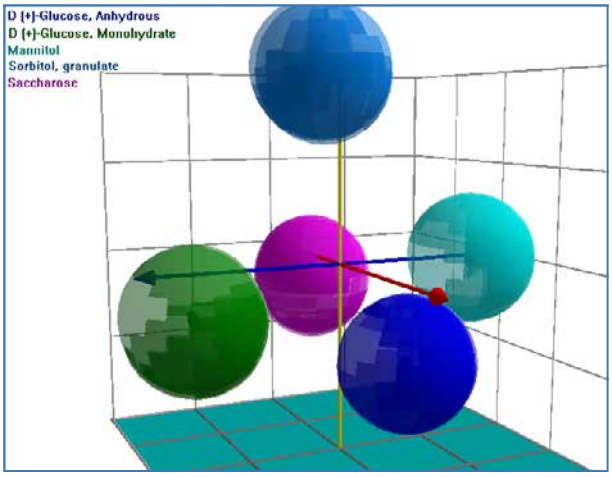

Figure 6: Interactive 3D view of score plot to calibrate cluster models

Calibration Wizard

The patented Calibration Wizard for automated calibration design is based on BUCHI's long term application expertise. It automates the creation and optimization of calibration models. The Wizard requires just a few basic settings. Optimized results through automated outlier detection, selection of suitable algorithms and wavelengths are just some benefits for the NIR users.

An analytical engineer from an Inspection Bureau states:
"The powerful NIRCal chemometrics software makes the huge and complicated data process more efficient, and the rich graphic display improves the visualization and colorfulness of the job."

Sample planning for quantitative NIR methods

A systematic approach helps to collect the right calibration samples to achieve an accurate and robust NIR analysis method. All data including targets and limits are included in the sample plan [24]. The sample plan includes 6 steps.

The sample plan includes 6 steps.

1. Select determination parameter and define target values
2. Define primary method
3. Estimate calibration range
4. Other influence to consider
5. Estimate working range
6. Sampling and sample presentation

A pattern of a calibration sample plan is presented in Figure 7. It provides a general guidance to setting up individual plans.

Formulation property	[Fat]	
Formulation target (TV	[% Fat]	e.g. label claim
Primary method	[Fat, Weibull-Stoldt]	Preferably a standart method Keep contant during calibration
Reference method precision (SE)	[± % Fat]	e.g. standart deviation
Working range (WR)	[% Fat, min] [% Fat, max]	Typical process variation
Calibration range with lower and upper limits (CR, CRLL, CRUL)	[% Fat, CRLL] [% Fat, CRUL]	CR = 20 x SE CR > WR
Sample	[Type]	e.b. finished product, blender sample, oven sample
Sampling / sample presentation	[Method]	e.g. cool to room temperature, finely shred or grind
Sample presentation	[Description]	e.g. use weighted press in unbreakable sample cup
Known sources of variation	[Description]	e.g. protein, moisture, salt, sasonal equipment (production line), termperature, technicians

Table 3: Calibration sample plan for fat content determination

Advantages of pre-calibrations

NIR users can rely on pre-calibrations. They are available for many components and samples. NIR analyzers have them built-in already or they can be downloaded from the NIR instrument manufacturer.

Pre-calibrations provide a smooth start into NIR analysis, build the base for reliable, accurate and robust NIR determinations and reduce initial calibration labor work and costs considerably.

Reference methods

Reference methods such as Kjeldahl protein determination or Weibull-Stoldt fat determination contribute the underlying results for the calibration of NIR methods. Such methods should be kept constant throughout the NIR method development cycle, as different laboratory methods have different accuracy and precision relative to one another. Hence, record your current reference method for that property under consideration. Carefully establish the standard error of such methods. It is a decisive term for the calibrations [24].

Please also refer to volume 1 of this Food Process Analytics Champion's Guidebook which you can download here: www.buchi.com/food-process-analytics-guidebook

Diversity of sample types

A whole variety of sample types can undergo NIR analysis for batch and continuous monitoring. Sample types can be as diverse as a clear liquid, a paste or a solid powder. Additional measure modes have opened the NIR's scope of practice and operation considerably compared for example to the common VIS spectroscopy transmission mode.

NIR instruments from BUCHI can accommodate all kinds of sample types. All viable sample presentation tools such as petri dishes, vials and cuvettes are also available.

Sample types	Measure modes	Principle
· Liquids · Sludges	Transflectance The NIR radiation passes through the liquid, is reflected and passes the sample a second time	
· Solids: powder, granulates, pellets · Gels, pastes	Diffuse reflectance NIR radiation penetrates the sample and is diffracted and/or reflected.	
· Clear liquids (sample in cuvette or vial)	Transmission NIR radiation is sent through a liquid sample on a defined pathlength.	
· Crystal powders, some tablets/pills	Diffuse transmission NIR radiation penetrates the sample and is diffracted and/or reflected. (This measure mode is very rarely used in food analysis.)	

Table 4: Sample types and suitable NIR measure modes

Appendix 2 – References

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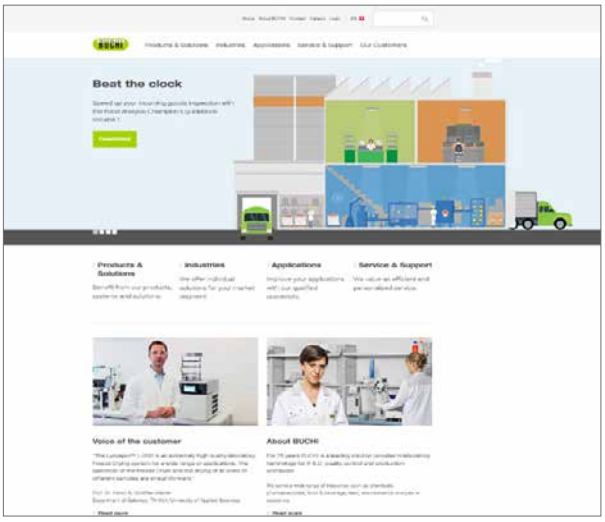

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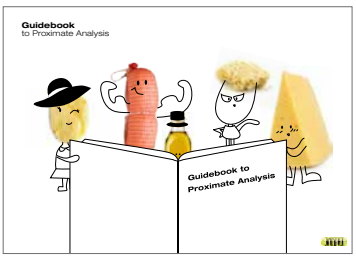

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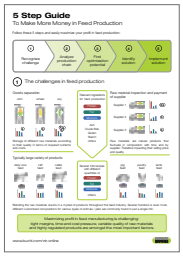

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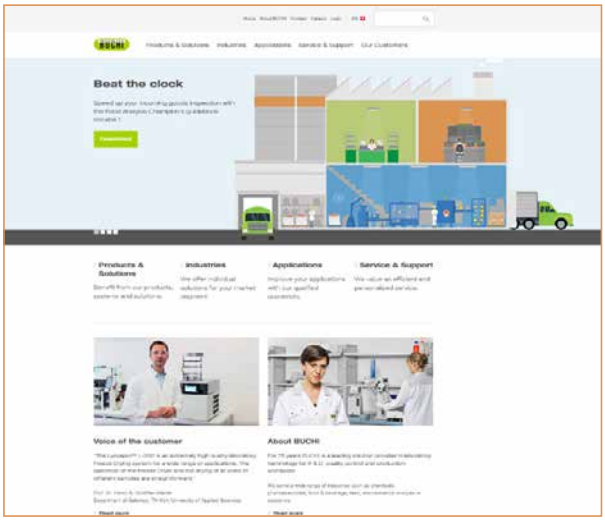

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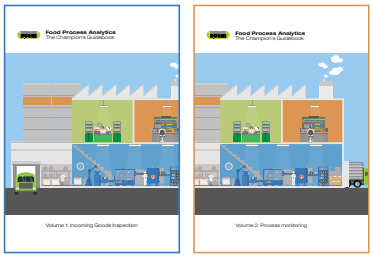

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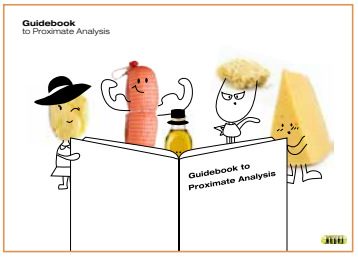

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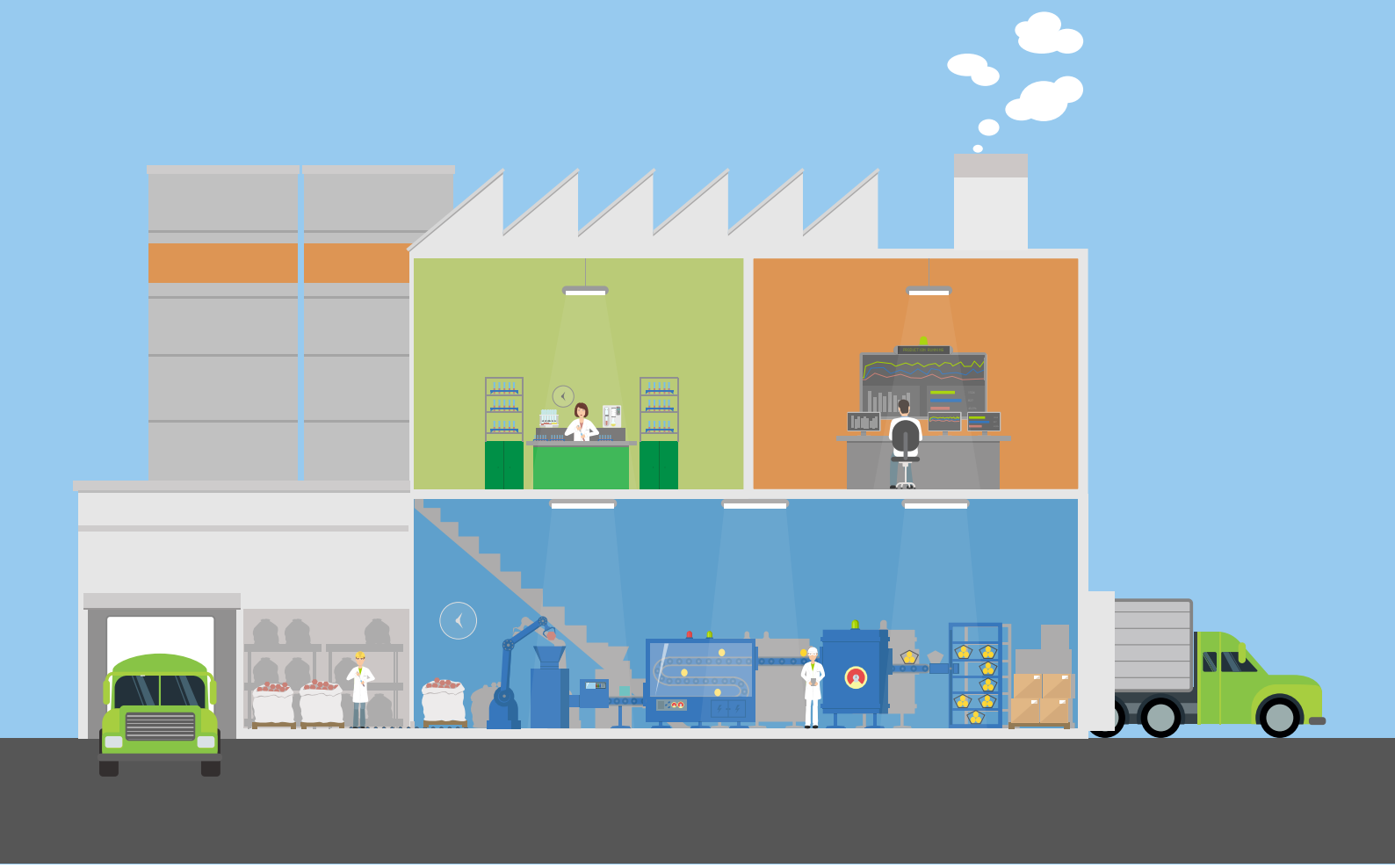

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