Tips & Tricks
from the Experts

Preface

In order to bring your instruments to full performance, tips and recommendations from practical experience are of great help. These Tips & Tricks can make your daily lab work easier, safer and more sustainable.

Users of this Tips & Tricks can rely on the support of BUCHI's laboratory experts. Some of these experts are presented in this guide. From their own lab experience, they provide useful tips for your benefit. They also explain what drives them forward in their job and share their personal motto with you.

At the end of the guide, you find more information about BUCHI's support materials.

You are welcome to read this guide. We are sure you will find one or the other useful tip helping you to master your laboratory tasks successfully.

BÜCHI Labortechnik AG with headquarters in Flawil, Switzerland, is a leading solution provider in laboratory technology for R & D, quality control and production worldwide.

Founded in 1939, BUCHI serves a wide range of industries such as pharmaceuticals, chemicals, food & beverage, feed, environmental analysis and academia. With a worldwide network of 18 subsidiaries and support centers as well as over 80 qualified distribution partners, BUCHI ensures proximity
and global reach.

Impressum

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The Expert
Chantal Ulmer

My motto

It's not about the problem, it's all about the right method to find the solution.

About my job

After my apprenticeship I studied chemistry at FHNW in Basel. Then I worked in analytical labs and an extraction company. Hence, I understand problems and needs of customers as I used to have their role. At BUCHI, I like the variety of samples. They let me look into many different industries and applications.

Products

As a leader of rotary evaporation BUCHI supplies a complete portfolio of units:
· Rotavapor® R-300 and R-100 units
· Vacuum pumps
· Recirculating coolers
· Interfaces and glassware

Samples

Apricot puree, thyme extract, essential oils, aromatic plants, chromatographic fractions, alcohol with botanicals for gin etc.

Rotary Evaporation

BUCHI Rotavapor® stands for long lasting operation times, long maintenance intervals and high availability.

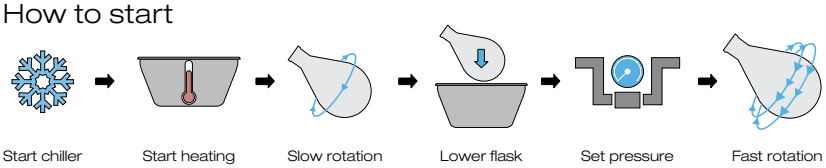
Tip 1: Clean and dry after every use

Cleaning: Rinse the condenser with some acetone or ethanol from the top opening and distill cleaning solvent through the Rotavapor. Then empty receiving and evaporation flasks.
Drying: Set pump to continuous pumping for several minutes.

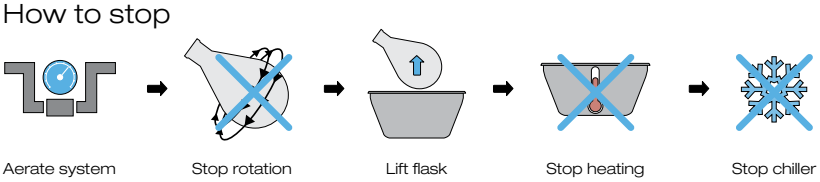
Tip 2: How to start and stop?

The right task sequence ensures safe operation and handling.

How to start

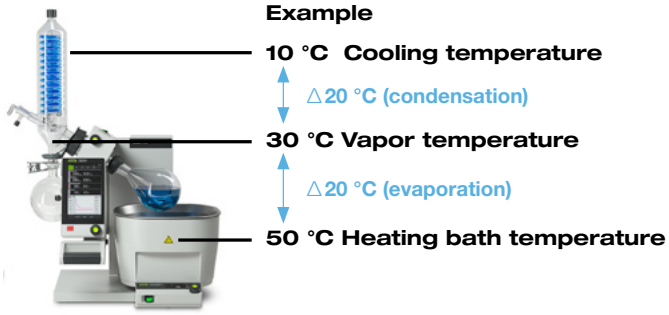


How to stop



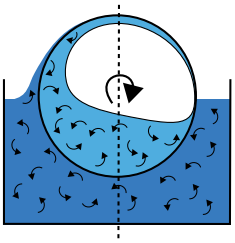
Tip 3: Apply the «Delta 20» rule

Apply the «Delta 20» rule for safe and optimal evaporation performance. The recommended boiling point is about 30 °C to avoid boiling of the collected distillate at ambient temperature.



Tip 4: Increase rotation speed

Rotation considerably increases the evaporation surface, the agitation of the sample and the turbulence in the heating bath. Set the rotation speed to the maximum to increase distillation rate and reduce bumping and foaming. Keep rotation speed low for high viscosity samples and when drying powders.



The Expert
Urs Hartfelder

My motto

I don't always evaporate. But when I do, it's parallel.

About my job

I studied Chemistry at ETH Zurich and completed a Ph.D. focusing on heterogeneous catalysis and spectroscopy. In my time at BUCHI, I have worked with nearly all our instruments. Currently, I am focusing on parallel evaporation and NIR spectroscopy.

Products

- Syncore® Analyst: Inhibited cross-contamination, maximized recoveries
- Syncore® Polyvap: Up to 96 samples parallel
- Multivapor™ P-6 and p-12: Tailored to maximize efficiency
- Interface I-300 Pro

Samples

Polychlorinated biphenyls in waste water, pesticides in animal food, polyaromatic hydrocarbons in water Ethyl carbamate in port wine etc.

Parallel Evaporation

The BUCHI Syncore® Analyst evaporates multiple samples simultaneously to a defined residual volume. A cooled appendix is used to prevent evaporation of the residual volume and to protect the analytes.

Tip 1: Before evaporation

Make sure that sufficient heat transfer medium is filled into the Syncore® rack.
When working with 1 mL or 0.3 mL appendices, make sure to insert the appropriate Appendix Insulation sleeves to improve efficiency.

Choose the appropriate Syncore® rack and glassware for your working volume – too high a filling level can limit the rotation speed, which will negatively affect evaporation rate and can lead to bumping.

Cool the receiving vessel – e.g. with the BUCHI refrigerated receiving vessel – to avoid re-boiling from the collection vessel. Re-boiling limits the evaporation rate especially with low-boiling dichloromethane and hexane.

Tip 2: Evaporation conditions

Most evaporations can be carried out efficiently with a heating block temperature of 65 °C and a cooling temperature of 10 °C. Sufficiently high rotation settings are necessary to obtain high evaporation rates and to prevent bumping. For most configurations, 280 rpm is a good starting point.

In routine operation, a pre-programmed pressure gradient on an I-300 Pro unit automates pressure control and ensures consistent evaporation conditions.

Tip 3: Transferring methods from a Rotavapor® to the Syncore®

The following pressure gradient is suggested as the starting point for any solvent. X is the pressure used on the Rotavapor®.

The heating block temperature on the Syncore® should be 5 °C higher than the water bath temperature of the Rotavapor®.

Time [min]	Pressure [mbar]
0	X + 500
4	X + 150
9	X + 50
19	X
End of evaporation	X
Evaporation + 2 min	Atmosperic pressure

The Expert
Maren Sander

My motto

Extraction is much more than just preparing tea!

About my job

I love the SpeedExtractor extracting any kind of sample. I have extracted a lot of different samples: Acarian, Barbie, Cosmetics, Dairy, Eggs, Fish, Gelatin, Hazardous waste, Ink, Jelly beans, Kenaf seeds, Leather, Mayonnaise, Nylon, Oil, Polyethylene, Quark, Rock core, Stents, Textiles, Upper wisdom tooth (no, just joking), Vegetables, Wool, Xylene, Yarn, Zurich Lake water.

Products

- SpeedExtractors E-914 and E-916, Extraction System B-811
- Extraction Units E-812 and 816, Hydrolysis Unit E-411 and E-416, Mixer B-400.

Samples

Classical fat extraction in chocolate, müsli, pet food, animal feed, potato chips, cake, biscuits, etc.etc.

Classical Extraction

Extraction methods – Soxhlet, hot and continuous extraction – are widely applied in the food, feed, environment, textile, polymer and waste industries. The fat determination of a chocolate sample includes three steps: homogenization, hydrolysis and extraction.

Tip 1: What to do when recovery is too high?

- Use clean solvents.
- After extraction, the beaker with the fat is dried in a drying oven. Dry it to a constant weight.
- Some fats and oils are very heat sensitive (e.g. sunflower oil). Dry such samples at lower temperature and under reduced pressure in a vacuum oven.

Tip 2: What to do when recovery is too low?

- Loss of sample during hydrolysis: wash the digestion vessel carefully to transfer the entire sample to the sample tubes. For optimum recovery rinse using water at 50 °C. If the water is too warm there will be a resultant loss of fat. If the water is too cold, there will be an incomplete transfer of the sample.
- Incomplete extraction: extraction time is too short. Apply recommended time. Level sensor is set too high or too low. Too much solvent is accumulated on the top of the sample.
- Different beaker temperature: before weighing, allow all beakers to reach the same (ambient) temperature.

Tip 3: What to do when there is large variation in results?

Inhomogeneous or too small samples can cause variation in results. Homogenize samples using a pestle and mortar or BUCHI Mixer B-400. The recommended sample size depends on the approximate fat content. Increase sample weight of very inhomogeneous samples (e.g. salami).

Fat content [%]	80 – 100	50 – 80	20 – 50	10 – 20	<10
Sample weight [g]	0.7 – 1.0	1 – 1.5	1.5 – 3.5	3.5 – 7	7 – 10

Tip 4: When is hydrolysis needed for fat extraction?

When fat is bound or enclosed in cells, hydrolysis breaks the boundaries and enables subsequent extraction. Also standards can ask for hydrolysis prior to extraction. For processed samples, where fat or oil was added and mixed, hydrolysis is usually not required.

The Expert
David Vinzent

My motto

It's all about the good old standard methods!

About my job

After working in the food industry for several years, I studied food and beverage technology. The experiences I've gained during this time helps me to understand the customer's needs in daily business.

Products

All nitrogen determination units provide high performance for their class and are safe to operate. Here is a short selection of BUCHI's offerings:

- Digestion: KjelDigesters K-446 and K-449, SpeedDigesters K-425, K-436 and K-439
- Distillation: KjelMaster K-375, KjelFlex K-360, KjelSamplers K-376 and K-377, Distillation units K-350 and K-355

Samples

Tetanus toxin, milk powder, salami, pet food, animal feed, waste water, beer, cosmetics, fertilizer, medicine, soil etc.

Nitrogen Determination

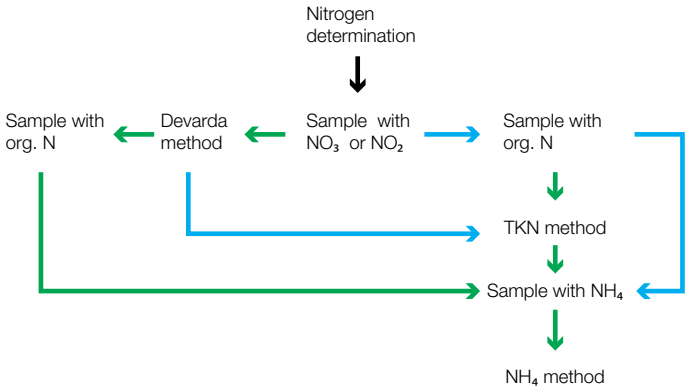
Total Kjeldahl Nitrogen (TKN) and TKN Plus (TKN+) are well-established methods to determine organically bound nitrogen including nitrate and nitrite respectively.

<table>
<tr><td colspan="3">Total nitrogen</td></tr>
<tr><td rowspan="2">Total Kjeldahl Nitrogen (TKN)</td><td colspan="2">Direct distillation (DD)</td><td rowspan="2">Not detectable by TKN and DD</td></tr>
<tr><td>Ammonium distillation</td><td>Devarda distillation (Nitrate, nitrite)</td></tr>
<tr><td colspan="3">Total Kjeldahl Nitrogen Plus (TKN+)</td></tr>
</table>

TKN and TKN+ methods include a digestion step followed by steam distillation and titration.

Tip 1: Select the right determination method

Following the decision tree, different methods are applied to determine the nitrogen content depending on the nitrogen sources of the sample.



Tip 2: Optimal conditions for digestion step

The digestion transfers organically bound nitrogen to ammonium. Hence, optimum conditions are required for complete digestion. The typical sample size is 0.125 – 2 g depending on the N content. Sulfuric acid is added according to the type and size of the sample. Do not forget the catalyst which also contains K^2SO^4 in order to reduce the evaporation of the sulfuric acid.

Temperature 420 °C
Time 290 – 120 min
Sulfuric acid (98 %) fat 9.7 mL per g
2protein 4.9 mL per g
2carbohydrate 4.0 mL per g
Catalyst (Kjeldahl tablet) 1 g per 2 mL sulfuric acid

Tip 3: Optimal distillation conditions

The distillation has to be exhaustive and safe but as short as possible. Recommended conditions:

Deionized water 4 mL per used mL sulfuric acid
NaOH4.5 mL per used mL sulfuric acid
Time 150 – 240 s

The Expert
Aurélie Demont

My motto

Research is a bit like cooking, except that you have no recipe and cannot lick the spoon.

About my job

After a PhD in biotechnology, I met my new challenge at BUCHI. The combination of feasibility studies and development of new applications is always exciting. We work with samples coming from everywhere around the world. From micro-organisms to diamond powder – the more provocative the sample, the more I'm interested in it …

Products

BUCHI's freeze drying offering includes:
· Lyovapor™ L-300
· Lyovapor™ L-200
· Lyovapor™ Software

Samples

Chicken, other meat samples, bananas, truffle, vaccines, microorganisms for storage or before extraction, soil for extraction etc.

Freeze Drying

Applications of BUCHI laboratory freeze drying units range from research and development to quality control and they cover a broad spectrum of market segments. The units stand out by their efficiency and practical capability.

Tip 1: Why is heat required during freeze drying?

The principle of freeze drying is based on the direct transition of a substance from the solid to the gaseous state, called sublimation. Sublimation is an endothermic process requiring energy in the form of heat. The product experiencing sublimation gives off heat and is therefore cooled down. The required sublimation energy then needs to be supplied to the product. Higher product temperatures lead to larger pressure differences and hence to a more efficient sublimation process. The additional input of heat boosts the sublimation and represents the real driver of freeze drying.

Tip 2: Use shielding

Three different sources of sublimation energy can be distinguished: (1) conduction from heated shelf, (2) convection by moving gas molecules in the vacuum and (3) infrared radiation. However, conduction is the heat flow that can be controlled best. Therefore, install a shielding for best controlled and reproducible results:

· Use ferrule around the vials.
· Leave a ring of outer vials empty.
· Install empty shelf over the vials.

Tip 3: Multiple thermocouples in samples

If empty shelves are installed for shielding reasons, these can be used to plug in additional thermocouples to monitor sample temperature.

Tip 4: Which samples cannot be freeze dried?

The following sample types cannot be freeze dried:

· Oil rich products
· Sugar-rich materials
  Products forming impervious skin
· High-salt-containing products

Tip 5: Keep distance between freezer and Lyovapor™ short

Place the freezer and the Lyovapor™ freeze dryer in the same room to reduce the time of sample transfer and to keep the frozen sample at a low temperature. Perform a leak test on the Lyovapor™ to detect misconfigurations and defects.

The Expert
Chantal Ulmer

My motto

It's not about the problem, it's all about the right method to find the solution.

About my job

After my apprenticeship I studied chemistry at FHNW in Basel. Then I worked in analytical labs and an extraction company. Hence, I understand problems and needs of customers as I used to have their role. At BUCHI, I like the variety of samples. They let me look into many different industries and applications.

Products

Melting and Boiling Point models BUCHI
- M-560: manual operation, quick and easy.
- BUCHI M-565: automatic operation, Pharmacopeia compliant.
- Sample Loader M-569 for fast and efficient capillary filling.

Samples

Palm oil, cocao butter, lipsticks, vaping liquid, synthesis products, base chemicals etc.

Melting Point & Boiling Point

Melting and boiling point are characteristic values of solid and liquid materials respectively. Both values are applied for material identification and characterization purposes. Contaminants lead to melting point depression and changes of the boiling point. Hence, melting and boiling point can be used for purity checks.

Tip 1: Grind sample well

A fine and uniform powder is crucial for accurate melting point determinations. Therefore, mix and grind the samples well for at least 5 minutes using pestle and agate mortar.

Tip 2: Melting point capillary filling level

The filling level of the capillaries significantly affects accuracy and reproducibility of the melting point determination. A uniform filling level of 4 mm and compact sample filling are recommended. There is a 4 mm filling level mark on the sample holder.

Tip 3: Boiling capillary filling level

For boiling point determination, a filling level of 10 mm is suitable. It is important that all capillaries have the same filling level.

Tip 4: Boiling point determination requires an ambient air pressure value

In order to correctly correlate the measured boiling point of a liquid, the current ambient air pressure must be entered in the M-565. If no external pressure sensor is available, many Rotavapors with vacuum control can be used instead to determine the ambient pressure.

The Expert
Maren Sander

My motto

Extraction is much more than just preparing tea!

About my job

I love the SpeedExtractor extracting any kind of sample. I have extracted a lot of different samples: Acarian, Barbie, Cosmetics, Dairy, Eggs, Fish, Gelatin, Hazardous waste, Ink, Jelly beans, Kenaf seeds, Leather, Mayonnaise, Nylon, Oil, Polyethylene, Quark, Rock core, Stents, Textiles, Upper wisdom tooth (no, just joking), Vegetables, Wool, Xylene, Yarn, Zurich Lake water.

Products

SpeedExtractor E-916 and E-914 are part of BUCHI's vast extraction offering. SpeedExtractors are fast, as you may run up to 6 samples in parallel. Identical extraction conditions reduce the number of replicates. The fail-safe and reliable extraction cells seal automatically and guarantee reproducible results.

Samples

Soil, sludge, polymers, textiles, chicken eggs, fish, thyme, milk powder etc.

Pressurized Solvent Extraction

Pressurized solvent extraction (PSE) is fast and highly efficient thanks to elevated temperature and pressure. Flexible temperature, pressure and extraction cell parameters of the SpeedExctractors allow for optimized methods.

Tip 1: Dry the samples

Extraction of dry samples is upmost efficient. Hence, drying the sample prior to extraction is recommended. The drying agent of choice is diatomaceous earth (DE). For very wet samples, oven drying is a suitable alternative. Freeze-drying or spray drying are also appropriate options.

Tip 2: Always disperse the samples

Dispersion of the sample with inert materials avoids aggregation of sample particles and is recommended for almost all applications. The most commonly used dispersing agents are quartz sand and DE. For most samples, a w/w ratio of 1 : 5 with sand and 1 : 1 with DE is suitable.

Tip 3: Pack extraction cell properly

Various techniques of packing the cell are used to reduce solvent consumption, increase extraction efficiency, avoid clogging of the cell, or to simplify the cleaning process after extraction.

1. Standard filling of sample mixed with drying or dispersing agent
2. Sand bed below sample and above to fill void volume
3. Expansion element with voluminous fluffy samples
4. Paper or glass fibre thimble topped with glass wool

Tip 4: Choose the right temperature

Temperature has a high impact on extraction speed and recovery. Start at 20 °C above the solvent's boiling point or at 100 °C for environmental samples. To avoid decomposition of the sample, apply 50 °C for plant materials and other natural products and ≤ 100 °C for fat extraction. Set temperature carefully for polymers to avoid melting during extraction.

Tip 5: Choose the right pressure

A pressure of 100 bar is a good starting point for a very broad range of applications. Set 150 bar for wet samples.

The Expert
David Vinzent

My motto

It's all about the good old standard methods!

About my job

After working in the food industry for several years, I studied food and beverage technology. The experiences I've gained during this time helps me to understand the customer's needs in daily business.

Products

- BUCHI K-350 and K-355 units: steam distillation in the most efficient way.
- BUCHI K-360 unit: the modular setup allows to adapt the K-360 to customer's needs and even includes a titration kit.
- SO_2 absorption accessory with two receivers.

Samples

Shrimps, wine, beer, spices, herbs, salt meat etc.

SO_2 Determination

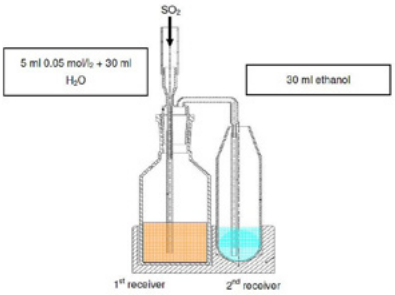
Sulfites are frequently applied to preserve food and beverages. Hence, strict regulations of the sulfite content require frequent and reliable determinations.

Tip 1: Apply the BUCHI method

For the quantitation, sulfites are converted into volatile sulfur dioxide SO_2 which is steam-distilled and thus, separated from the sample. The iodine in the receiver oxidizes SO_2 and is consumed. The residual iodine after all SO_2 is distilled then is titrated with sodium thiosulfate.

Tip 2: Crucial points to consider

Some hints to reach good SO_2 determination practice:



- Homogeneous samples are necessary to obtain repeatable results.
- Grind solid samples to fine powder before weighing.
- If foaming or heavy splashing occur during distillation, reduce steam power or add an antifoam tablet.
- To avoid corrosion by HCl, use H_3PO_4 to release SO_2.
- Start distillation immediately to prevent loss of iodine and sulfur dioxide.
- Stop distillation by set distillation time or when distillate level reaches the neck of the receiver bottle.

Tip 3: How to optimize recovery

The recommended way to optimal recovery is the use of BUCHI SO_2 absorption glassware with two flasks. The first receiver contains the iodine solution, the second one is filled with ethanol to catch evaporating iodine. Before titration, both containers are combined.

Tip 4: How to save chemicals and samples

Expected SO_2 concentration [mg/kg]	Recommended sample weight [g]
7 – 10	150 – 100
10 – 20	100 – 50
20 – 100	50 – 10
100 – 200	10 – 5
200 – 500	5 – 2
> 500	< 2

The optimal SO_2 content per determination is 1 mg. Hence, the optimal sample weight depends on the expected total SO_2 concentrations.

Tip 5: Other methods

BUCHI steam distillation units can also be used for other sulfur dioxide determination methods such as the Monier-Williams method and the China National Standard (GB) method.

The Expert
Estefanía Pérez Fernández

My motto

All you need is… NIR.

My job

I am a biologist by training. Through a Ph.D. and postdoctoral studies, I gained 10 years of experience on NIR applications for environmental and agricultural research. I joined BUCHI in July 2017 to work on NIR development projects and to continue learning new aspects of this fascinating and versatile technology.

Products

NIRFlex and NIRMaster™ are BUCHI's choice of FT-NIR spectrometers. Models dedicated to QC, R&D and at-line provide optimal application performance. With NIR, many properties can be determined simultaneously.

Samples

Cereal grains (e.g. wheat, barley, rice), wheat flour, palm oil, olive oil, livestock feed, wet pet food, milk and dairy products (e.g. milk, milk powder, cheese, yogurt), meat, chemicals etc.

Near Infrared Spectroscopy

Near Infrared (NIR) spectroscopy, applied mainly to organic compounds, is an efficient tool to provide quick and cost-effective analytical results. Robust calibrations are required to achieve reliable results. Here are some calibration tips:

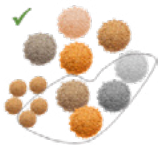
Tip 1: Get the samples right

Samples for analysis should be of the same nature as the calibration samples. For example, a calibration developed to predict protein content in wheat is not suitable to predict protein in other types of grains. Since sample moisture and particle size influence NIR, make sure all samples are presented in the same way.

Tip 2: 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.

Calibration set too narrow



Good calibration set

Principal components analysis (PCA) is a useful statistical tool to observe the variability of samples.

Tip 3: Reference values matter

Reliable NIR calibrations require reliable reference values.

- Always apply well-established reference methods.
- Consider implied standard errors and measurement uncertainty.

Tip 4: Calibrate like a pro

Calibrations correlate NIR spectral data with the reference values using statistical algorithms like PCR or PLS regression. Baseline and scatter corrections are also useful to enhance calibration results.

Tip 5: 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.

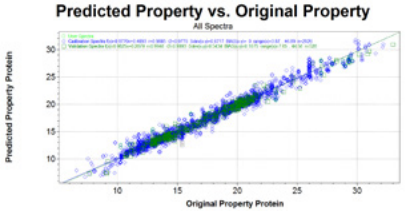


Figure 1: Regression of predicted and reference values

This way you can monitor your calibration performance regularly and apply corrections if necessary.

Further guides and handbooks

Kjeldahl Knowledge Base
Decode the mysteries of nitrogen and protein determination according to Kjeldahl,
76 pages, © 2017
https://secure.viewer.zmags.com/publication/1f5ce3a8#/1f5ce3a8/1

Freeze Drying Adviser
Basics and Application
20 pages, © 2017
www.buchi.com/lyovapor

Productivity Handbook for Industrial Evaporation
Optimize your system and achieve better results
25 pages, © 2017
www.buchi.com/tune

Guidebook to Proximate Analysis
6 pages, © 2017
www.buchi.com/guidebook-to-proximate-analysis-by-buchi

Cartridges in Flash Chromatography Infographic
Overview of the broad range of cartridges, particles and benefits
2 pages, © 2017
www.buchi.com/all-in-one

Reveleris® Advanced flexibility in purification
Scientific Reference Booklet
18 pages, © 2017
www.buchi.com/reveleris-advanced-flexibility-in-purification

5 Step Guide
To Make More Money in Feed Production
6 pages, © 2017
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www.buchi.com/parallel-evaporation-solutions

Rotary evaporation
www.buchi.com/laboratory-evaporation

Pressurized solvent extraction
www.buchi.com/products/extraction/speedextractor-e-914e-916

Industrial solutions

Besides laboratory products, BUCHI offers a variety of industrial solutions to master bigger sample sizes.

Rotary evaporation

Follow the challenging needs of scale-up and production labs.
Apply programmable methods and 24/7 operation.
www.buchi.com/content/industrial-evaporation-solutions

Units:
Rotavapor® R-220-Pro
Rotavapor® R-250
and more

NIR-Online Solutions

For real time process control: closely monitor key parameters such as moisture, fat and protein.
www.buchi.com/content/nir-online-solutions

Units:
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NIR-Online Multipoint System

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