Retsch®
Solutions in Milling & Sieving

the sample

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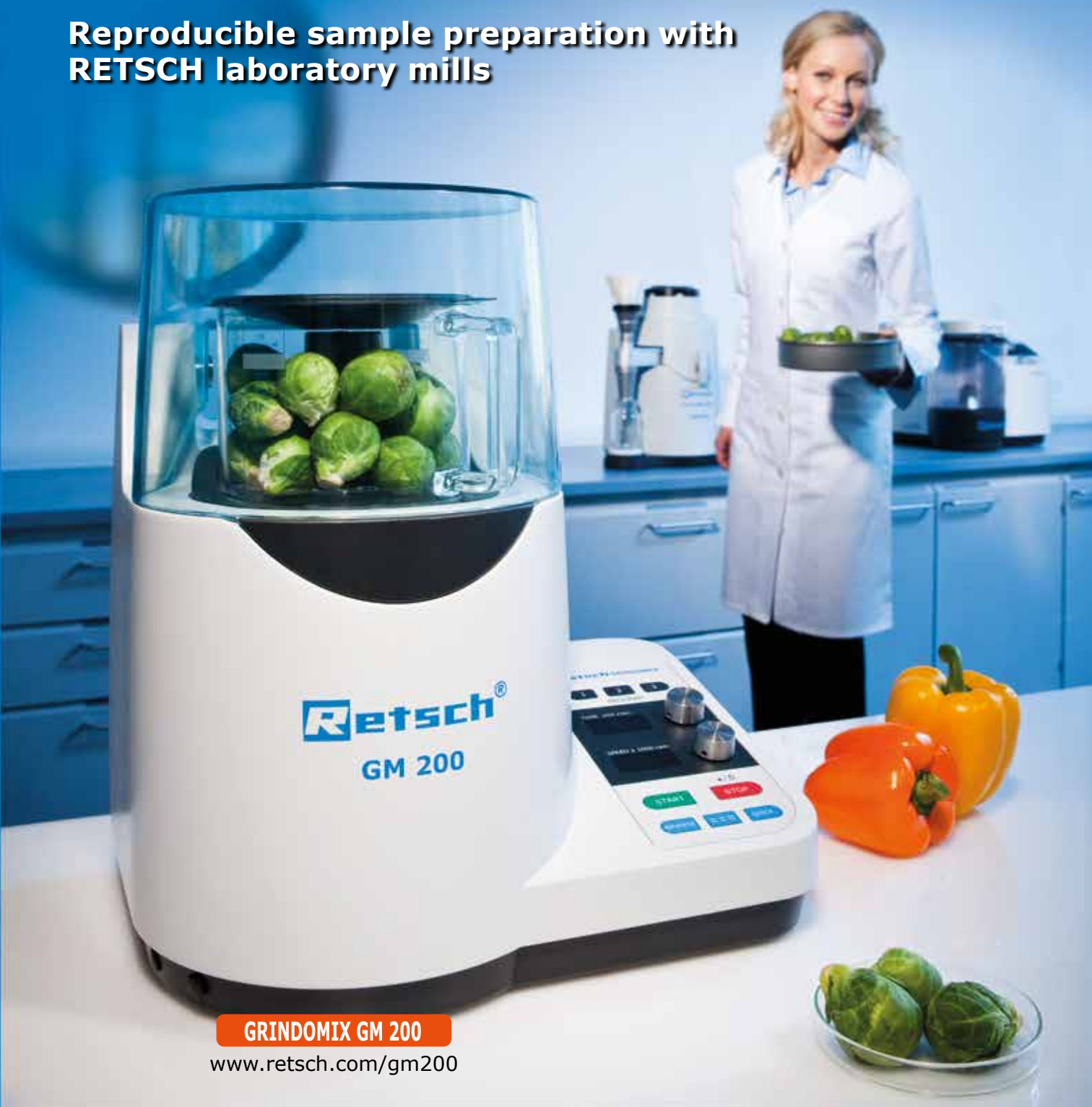
FOOD ANALYSIS

Reproducible sample preparation with RETSCH laboratory mills



GRINDOMIX GM 200

www.retsch.com/gm200

Dear Readers, Customers and Business Partners,

Nutrition is a central issue in our daily lives: from cooking shows on TV to the latest diet or food scandal. More and more consumers aim to make their diet healthy, balanced and sustainable and apply high standards to the quality of their food. The EU has launched a number of directives regulating permissible ingredients as well as limit values of potentially hazardous substances in comestible goods. To ensure the safety and quality of food, a comprehensive and reliable quality control process must be observed. Typical food testing techniques include, for example, chromatographic methods, Kjeldahl protein assay, NIR or other spectrometric techniques such as hydride generation atomic fluorescence spectrometry (HG-AFS). **All methods have in common that they require a homogeneous sample which is representative of the original material to provide meaningful and reliable results.** This is where **RETSCH mills** for reproducible, neutral-to-analysis sample preparation come into play. In this issue of "the sample" we present you the full range of RETSCH equipment suitable for the food industry.

The **determination of particle size distributions** is another important aspect of quality control of food products which we demonstrate using the examples of coffee and cereals.

We hope you enjoy reading this issue!

Yours

Dr. Jürgen Pankratz
Director VERDER SCIENTIFIC

Sample preparation
for food testing

QUICK AND REPRODUCIBLE HOMOGENIZATION AND CHARACTERIZATION OF COMPLEX SAMPLES

Food products are available in a great variety of forms and consistencies and are usually inhomogeneous. To determine nutritional values or detect hazardous substances, for example, homogeneous and representative samples are required to obtain reproducible and meaningful results. Samples, which are often very complex, need to be homogenized and reduced to a suitable particle size prior to analysis. Laboratory mills of different designs are ideally suited for this process.

Most analysis techniques only require a few milligram or gram of sample material which, however, must represent the entire original sample. The composition of the analysis sample may vary, depending from which part of the original sample it was extracted. Cereal bars, for example, consist of a variety of ingredients which all need to be represented in the part sample to ensure correct determination of the nutritional values. This is achieved by thoroughly homogenizing the cereal bars prior to analysis. Basically, the sample preparation process should be adapted to the sample characteristics as well as to the subsequent analysis technique to avoid falsified results. Grinding parameters and accessories should be selected with regard to preserving the sample properties to be analyzed. **A basic rule is to only grind the sample as fine as necessary and not as fine as possible**, as this always generates more effort (energy input, time, heat, wear). The sample preparation process and the required particle size depend on the chosen analysis method. A particle size of 0.5 mm is ideally suited for most digestion and extraction methods.

Food products vary greatly with regards to hardness, moisture or fat content. RETSCH offers a variety of mills and grinders suitable for the different requirements of food analysis. In many laboratories a large number of samples is analyzed each day. **Thanks to simple and intuitive handling, effective size reduction and quick and easy cleaning, RETSCH instruments help to increase the efficiency in the food lab.**

SELECTION GUIDE FOR MILLS SUITABLE FOR FOOD PROCESSING

	 Ultra Centri-fugal Mill	 Cyclone Mill	 Rotor Beater Mill	 Cross Beater Mill	 Knife Mill	 Cutting Mill	 Mortar Grinder	 Disc Mill	 Mixer Mill	 Ball Mill
Bread	•	•	•	•	✔	✔	–	–	•	–
Cheese	•	–	-	–	✔	–	–	–	•	–
Cocoa nibs	–	–	–	–	•	–	✔	–	•	•
Coffee beans	✔	✔	✔	•	✔	–	–	–	•	–
Fish	–	–	–	–	✔	•	–	–	•	–
Fruit	•	–	–	–	✔	•	–	–	•	–
Grain	✔	✔	✔	•	✔	•	✔	•	✔	✔
Herbs	✔	✔	•	•	✔	✔	✔	•	✔	✔
Meat	–	–	–	–	✔	•	–	–	•	–
Mushrooms	•	–	–	–	✔	•	–	–	✔	–
Nuts	•	–	–	–	✔	•	•	–	•	–
Olives	–	–	–	–	•	–	–	✔	–	–
Pasta	✔	✔	✔	✔	✔	✔	✔	•	✔	✔
Potatoes	–	–	–	–	✔	•	–	–	•	–
PralineS	–	–	–	–	•	–	•	–	•	–
Rice	✔	✔	✔	✔	–	–	✔	•	✔	✔
Salt	✔	•	✔	✔	–	•	✔	•	•	•
Spices	✔	✔	•	•	✔	✔	✔	•	✔	✔
Tea	✔	✔	✔	•	✔	✔	✔	✔	✔	✔
Tobacco	✔	✔	✔	•	•	✔	✔	✔	✔	✔
Sugar	✔	•	✔	✔	–	–	✔	•	•	•
Vegetables	•	–	–	–	✔	•	–	–	•	–

✔ = suitable • = suitable to a limited extent – = not suitable

The diversity of foodstuffs with their often very different product properties represents a real challenge for food testing laboratories. Before the actual analysis, the sample materials – which can vary strongly with regards to hardness and moisture – need to be homogenized and reduced to a sufficiently small particle size. RETSCH's GRINDOMIX knife mills are the ideal tools to meet the complex requirements of the sample preparation of food. The model GM 200 has proven itself for the homogenization of small sample volumes of up to 700 ml. For larger volumes RETSCH offers the GM 300 model with a grinding chamber volume of 5,000 ml.

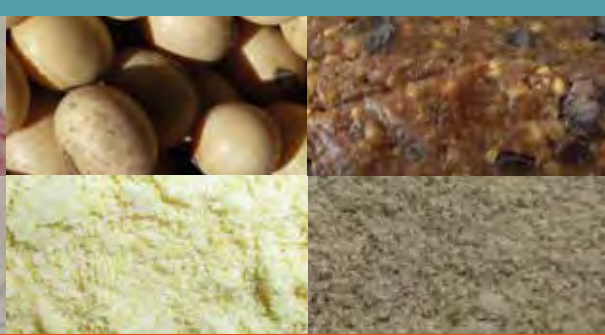
From muesli to streaky bacon

Complete homogenization of complex samples

GRINDING TOUGH MEAT

According to forecasts the worldwide meat consumption will increase substantially from today's 42.5 kg per year and person due to improved living conditions and changes in consumer behavior. Consequently, the importance of quality controls such as the determination of fat content of meat will increase. **Tough sample materials like fatty, inhomogeneous, streaky bacon pose quite a challenge to the homogenization process prior to analysis.** However, representative sample preparation is an essential step to obtain reliable analysis results. If larger parts of the rind or skin remain uncut, the sample is not homogeneous and the following analysis may yield false results. Another important aspect of size reduction is the **high water content** of the sample. Knife mills have proven to be best suited for thoroughly homogenizing this type of sample.

The knife mill GRINDOMIX GM 200 is equipped with a strong motor, to make use of the full cutting capacity of the blades for the size reduction process. **The new serrated blade knife is ideally suitable for homogenizing tough meat samples in a very short amount of time.** In addition, the short grinding times ensure low heat build-up.



GRINDOMIX GM 200
www.retsch.com/gm200

PERFORMANCE DATA

KNIFE MILLS GRINDOMIX GM 200/GM 300

Applications:	size reduction, homogenization
Feed material:	soft, medium-hard, elastic, containing water / fat / oil, dry, fibrous
Feed size*:	< 40 mm / < 130 mm
Final fineness *:	< 300 µm

*depending on feed material and instrument configuration

TYPICAL SAMPLE MATERIALS

Cereal bars, cheese, cocoa nibs, deep-frozen food, dried fruit, feed pellets, fish, grain, ham, meat, nuts, oil seed, pharmaceutical products, plant materials, salad, sausages, spices, sweets, vegetables, etc.

GRINDOMIX GM 300

www.retsch.com/gm300

The GRINDOMIX GM 300 processes sample volumes up to 4.5 l (e.g. bread, salad, pizza) without preliminary size reduction quickly and reproducibly.

HOMOGENIZATION IN 3 STEPS

1. 250 g pork shoulder are processed in the GM 200 with the serrated blade knife for 30 seconds with interval mode at 3,000 min^{-1}.
2. The first step is followed by 2 cycles of 30 seconds, each at 7,000 min^{-1}.
3. Complete homogenization of the sample is achieved after another 30 seconds at 10,000 min^{-1}.

Part of the sample sticks to the grinding container wall above the blades and needs to be removed with a scraper and returned to the grinding process from time to time.

TIP **Video of meat homogenization in the GM 200:**
www.retsch.com/gm-meat

Thanks to **three different operation modes** – with forward motion the sample is cut, in reverse mode it is submitted to impact, and the interval mode ensures a thorough mixing of the sample – the size reduction process can be optimized with regards to the sample properties. A wide selection of accessories allows for adaptation of the GRINDOMIX mills to individual requirements. Grinding containers are available in steel, glass, polycarbonate or polypropylene. A special **gravity lid** reduces the sample volume during grinding to improve the homogenization of samples with high water content. The liquid which flows up the container walls (capillary effect) is returned to the grinding chamber via overflow channels, thus ensuring that no sample material is lost and that results are not falsified. A **lid for grinding chamber reduction** downsizes the volume of the PP grinding container to 0.5 l so that smaller amounts of up to 0.35 l are continuously subjected to the grinding process and cannot escape the blades.

KNIFE MILLS

GRINDOMIX

- Perfect homogenization
- Results with minimum standard deviation
- Variable speed
- For sample volumes up to 700 ml or 4,500 ml
- Autoclavable grinding tools
- Unique lids for volume adaptation of grinding chamber
- Accessories for heavy-metal-free grinding

Tracing poison

Sample preparation for analysis of toxic elements

Reports on findings of hazardous substances in food products are nothing unusual these days: pesticides in fruit, arsenic in rice or toxic plant parts in tea, to name but a few. Therefore, permanent quality control is an indispensable tool of consumer protection. Apart from the actual food testing, sample preparation by homogenization is essential to obtain reliable results.

Rotor Mill ZM 200
www.retsch.com/zm200

ARSENIC IN RICE

There have been a number of recent reports published concerning the levels of arsenic found in rice and rice products. Rice is an essential ingredient of a large number of products such as baby food, breakfast cereal or rice cake. Consumer Reports in the United States already recommended in 2012 that people limit their rice consumption, after finding arsenic in over 60 rice products they tested. Arsenic is a contaminant and a known carcinogen which exists in both toxic and nontoxic forms. Rice absorbs arsenic from soil or water much more effectively than most other plants. This is primarily because rice is grown in water-flooded conditions, which allow arsenic to be more easily taken up by its roots and stored in the grains. Meanwhile the EU has also suggested stipulating limit values for arsenic between 0.10 and 0.30 mg/kg, depending on the area of application.

Once a representative bulk sample is taken for sample preparation, the rice must be ground to a suitable size range. The grinding process serves firstly to homogenize the sample, while the subsequent sample preparation steps or

analysis method typically require a certain fineness of material. RETSCH's Ultra Centrifugal Mill ZM 200 is best suited to pulverize rice to analytical fineness.

Sample	Rice
Feed quantity	100 g
Feed size	3 – 7 mm
Speed	18,000 min^{-1}
Grinding tools	24-teeth push fit rotor, 0.12 mm distance sieve, cyclone
Grinding time	< 3 min
Final fineness	< 100 µm

The analysis of total arsenic is most commonly achieved using acid digestion followed by a spectroscopic detection method such as hydride generation atomic fluorescence spectrometry (HG-AFS).

PYRROLIZIDINE ALKALOIDS IN TEA

The group of pyrrolizidine alkaloids comprises 500 chemical compounds which are mostly found in composite flowers, borage family and leguminous plants. Some of the better known compounds are senecionine, senkirkine or lycopsamine which can be found in ragwort. Unfortunately, these toxic plants repeatedly found their way into comestible goods such as herbal tea. The German Federal Institute for Risk Assessment demands zero tolerance for pyrrolizidine alkaloids, particularly in teas for pregnant and breast-feeding women and small children, due to their toxicity.

Dried camomile flowers were processed with the following parameters:

Sample	Dried camomile flowers
Feed quantity	25 g
Feed size	5 mm
Speed	18.000 min^{-1}
Grinding tools	12-teeth push-fit rotor, 0.5 mm ring sieve, then 0.2 mm ring sieve, cyclone
Grinding time	< 2 min
Final fineness	< 100 µm

The use of a **cyclone** ensures continuous material discharge and also **cooling of the sample**. Thus the characteristics of the heat-sensitive pyrrolizidine alkaloids are preserved during sample preparation.

ULTRA CENTRIFUGAL MILL ZM 200

The ZM 200 is a high speed rotor mill which pulverizes the sample by impact and shearing between rotor and fixed ring sieve. The sample falls through the hopper onto the rotor and is thrown outward by centrifugal acceleration. When hitting the wedge-shaped rotor teeth, which rotate with high speed, the sample is subjected to primary size reduction. Fine size reduction occurs between rotor and ring sieve with the final fineness being determined by the aperture size of the ring sieve. The sample does not remain long enough in the grinding chamber to heat up so that the properties to be determined are preserved.

ULTRA CENTRIFUGAL MILL
ZM 200

- Rapid and gentle size reduction by 2-step rotor/screen system
- Speed range up to 18.000 min^{-1}
- Patented cassette system for maximum sample recovery and easy cleaning
- Defined final fineness due to ring sieves with aperture sizes from 0.08 - 10 mm
- Comfortable parameter setting via display and ergonomic 1-button operation
- Wide range of accessories including various collection and feeding systems, rotors and sieves

PERFORMANCE DATA

ROTOR MILL ZM 200

Applications:	fine grinding
Feed material:	soft, medium-hard, brittle, fibrous
Feed size *:	< 10 mm
Final fineness*:	< 40 µm

*depending on feed material and instrument configuration

TYPICAL SAMPLE MATERIALS

Cocoa beans, coffee beans, corn, dried fruit and vegetables, fruit skin, gelatin, grain, muesli, pasta, pepper, rice, seeds, spices, sugar, tea, etc.

A healthy and balanced diet becomes increasingly important to more and more people. Fish, for example, is considered to be very healthy due to the Omega-3 fatty acids it contains; these cannot be produced by the human organism and therefore need to be obtained through food. The popularity of fish is reflected in the figures published by Germany's Statistical Office: between 2012 and July 2014 there was an increase of 4.2% in aqua cultures in Germany alone. Like for all comestible goods, the analysis of nutritional values and substances is an essential part of the quality control of fish, with a focus on protein and fat analysis. Some parts of the fish are rather fatty; therefore a thorough homogenization of the sample is vital to obtain reliable analysis results. Moreover, fish is examined for pollutants such as heavy metals, carcinogenic polychlorinated biphenyls or pharmaceutical residues as part of consumer protection. These substances accumulate in fish and are therefore considered reliable indicators for water quality; with appropriate analysis methods even smallest traces of contamination, for example with polychlorinated biphenyls, can be reliably detected.

What's in the fish?

Effective size reduction by cutting

QUICK AND SIMPLE HOMOGENIZATION OF DRIED FISH

The homogenization of fish is a challenge; scales, skin and bones are fairly resistant to size reduction so that the sample still contains some larger pieces after grinding in most mills. A high fat content of the fish makes the process even more difficult, as fatty particles stick together to form large lumps which block the mill and keep the sample inhomogeneous. A knife mill like the GRINDOMIX is suitable for successfully homogenizing fresh fish. Michael Schlachter of GMA (Gesellschaft für Marine Aquakultur mbH) addressed RETSCH to find a solution for **pulverizing freeze-dried fish**. For this type of fish the powerful cutting mill SM 300 proved to be the best option:

Sample	**Freeze-dried fish** (turbot, carp)
Feed quantity	125 g (= 4 fish of each type, pre-cut once or twice)
Speed	3,000 min^{-1}
Grinding tools	V rotor, 1.0 mm bottom sieve, cyclone
Grinding time	< 2 min
Final fineness	< 1 – 2 mm

The mill's **V rotor** is recommended for use with fibrous and tough samples and cuts very effectively through the freeze-dried fish. Its particular shape reduces the dead volume inside the grinding chamber and **promotes the quick discharge** of the

Cutting Mill SM 300
www.retsch.com/sm300

THE PERFECT CUTTING MILL FOR EVERY APPLICATION

Cutting Mill SM 100

www.retsch.com/sm100

The budget-priced basic model for routine applications

Cutting Mill SM 200

www.retsch.com/sm200

The versatile standard model for a wide range of application

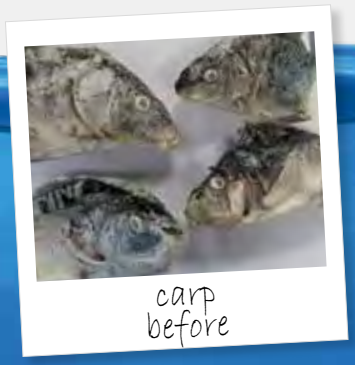
Cutting Mill SM 300

www.retsch.com/sm300

The high-performance powerful model processes even the most difficult samples

CUTTING MILL SM 300

- Powerful size reduction thanks to 3 kW drive with high torque and RES technology
- Perfect adaptation to application requirements by variable speed from 700 bis 3.000 min^{-1}
- Optimum cutting effects thanks to double acting cutting bars
- Quick and easy cleaning due to fold-back hopper, smooth surfaces and push-fit rotor
- Max. rotor peripheral speed of 20.3 m/s
- Defined final fineness due to bottom sieves with aperture sizes from 0.25 - 20 mm
- Wide range of accessories including various hoppers, collection systems, rotors and sieves
- Highest safety standards due to engine brake, central locking device, electronic safety check



pulverized fish, thus accelerating the grinding process. Moreover, this rotor also cuts the major part of the scales which leads to a better homogenization of the sample. The use of a cyclone improves material discharge. The grinding process in the SM 300 is carried out without any noteworthy heat build-up so that the fatty parts of the fish don't block the bottom sieve and the sample is thoroughly homogenized. The mill is quickly cleaned thanks to the fold-back housing and the easily removable push fit rotor.

The SM 300 grinds up to 5 liters of sample material in one run. After grinding in the SM 300, the sample can be further pulverized in the ultra centrifugal mill ZM 200, if required. For the analysis of fish the fineness achieved in the SM 300 is sufficient. Although the freeze-dried fish had a high fat content, the homogenization process in the cutting mill yielded very good results thanks to the powerful drive, the cutting performance of the V rotor and the optimized material discharge achieved by using a cyclone.

PERFORMANCE DATA

CUTTING MILL SM 300

Applications:	size reduction
Feed material:	soft, medium-hard, tough, elastic, fibrous
Feed size*:	< 60 x 80 mm
Final fineness*:	< 0.25–20 mm

*depending on feed material and instrument configuration

TYPICAL SAMPLE MATERIALS

Bones, carrots, cocoa beans, coffee beans, corn, deep-frozen chicken legs, dried fruit and vegetables, freeze-dried fish, fruit skin, kohlrabi, nuts, pasta, plants, spices, tobacco, etc.

Sticky, tough or elastic samples are generally difficult to pulverize. Cheese, for example, can be ground with a knife mill but only to a final fineness of about 1 – 2 mm which is not small enough for most analysis techniques. Sweets, on the other hand, often consist of various components like elastic foamy sugar with a sticky, liquid filling. If these types of sample are not embrittled before grinding, they tend to clog the mill. Cryogenic treatment, however, improves the breaking properties of cheese or candy so that even elastic materials can be successfully pulverized.

Cold, sticky and tough

Cryogenic grinding improves breaking properties

CRYOMILL

- Powerful cryogenic grinding by impact and friction, up to 30 Hz
- Cryogenic grinding, dry and wet grinding at ambient temperature
- Closed LN_2 system (autofill) for enhanced safety, avoids contact of the user with liquid nitrogen
- Screw-top grinding jars for convenient, leak-proof operation
- Wide range of accessories
- Low LN_2 consumption
- Clearly structured user interface, memory for 9 SOPs
- Programmable cooling and grinding cycles (10 s to 99 min)

RETSCH's CryoMill is the perfect tool for fine size reduction under continuous cooling with liquid nitrogen. The grinding jar oscillates horizontally with up to 30 Hz so that the sample is mainly pulverized by the grinding balls through impact and friction. It produces significantly finer grind sizes than other cryogenic mills. The continuous embrittlement of the sample prevents the soft, elastic particles from simply deforming or smearing. The grinding jar of the CryoMill is cooled via an integrated cooling system with liquid nitrogen before and during the grinding process. The mill is particularly efficient and safe as liquid nitrogen is refilled from the autofill system in exactly the amount which is required to keep the temperature consistently at -196 °C, without the user having to get in contact with LN_2. The CryoMill is also ideally suited to process materials containing volatile components.

PERFORMANCE DATA

CRYOMILL	
Applications:	size reduction, mixing, homogenization, cell disruption
Feed material:	hard, medium-hard, soft, brittle, elastic, fibrous
Feed size*:	< 8 mm
Final fineness*:	< 5 µm

*depending on feed material and instrument configuration

TYPICAL SAMPLE MATERIALS

Baby food, bones, cheese, chocolate, dietary supplements, feedstuff, fish, fruit, gummy bears, hard candy, meat, oil seeds, plants, pralines, toffee, vegetables, etc.

PULVERIZATION OF CHEESE

The quality control of cheese not only involves determination of nutritional values such as proteins or amino acid content but also of mould and mycotoxins produced by mould. If a product such as hard cheese is further processed to soft cheese, reliable analysis and quality control is particularly important to ensure that potential quality defects will not be carried on to the next production step.

Hard cheese was processed with the following parameters:

Sample	Hard cheese
Feed quantity	5 g
Feed size	8 mm
Grinding tools	50 ml steel grinding jar, 25 mm steel grinding balls
Pre-cooling	7 min at 5 Hz
Grinding time	2 min
Oscillation frequency	30 Hz
Final fineness	< 300 µm (90 % of the sample)

After extraction the sample is analyzed with High Performance Liquid Chromatography (HPLC) for ingredients and nutritional values.

STICKY PRALINE WITH LIQUID FILLING

Like all comestible goods, confectionary is subject to strict quality control. Parameters to be determined range from nutritional value, moisture or fat content to the quantification of certain ingredients such as vitamins or alkaloids. Chromatographic methods like HPLC are typically used to analyze the ingredients; these generally require a particle size of 0.3 – 0.75 mm.

The praline was processed with the following parameters:

Sample	Praline with liquid filling
Feed quantity	1 praline
Grinding tools	50 ml steel grinding jar, 25 mm steel grinding ball
Pre-cooling	5 min at 5 Hz
Grinding time	2 min
Oscillation frequency	30 Hz
Final fineness	< 400 µm (90 % of the sample)

The sample was completely homogenized and did not stick on the grinding jar walls.

CryoMill
www.retsch.com/cryomill

White Gold

Grinding large sample volumes

ROTOR BEATER MILL
SR 300

- Suitable for batchwise operation of larger quantities
- Grinding chamber, feed hopper and material inlet and outlet of stainless steel
- Removable hopper and push fit rotor for easy cleaning
- Material feed size up to 25 mm
- Optional grinding inserts 180° for grinding of hard-brittle materials by additional impact
- Distance rotor (optional) reduces frictional heat
- Defined final fineness due to bottom sieves with aperture sizes from 0.08 - 10 mm

"White gold" is a figure of speech for salt and reflects its enormous importance. Archeological discoveries helped to prove that salt was commonly used as early as 10,000 B.C.. Salt not only serves to flavor food but also to preserve it. It is won on the coast where sea water evaporates in shallow areas and the minerals crystallize (sea salt). Rock salt is quarried from natural salt deposits and makes up about 70 % of the worldwide salt production. These natural deposits built up over millennia of desiccation of the sea. The various minerals crystallized one after the other, first forming a lime layer, followed by gypsum, salt (sodium chloride) and clay minerals. Due to tectonic effects these places were flooded by the sea again and, in the course of time, sediments, which turned to sedimentary rock, covered the salt layers. These were partly pushed upwards leaving the salt right below the surface. As it was not possible to win the salt in all places it had to be transported over long distances. Many cities along the transport routes acquired great wealth through the salt trade by collecting tolls and taxes.

PERFORMANCE DATA

ROTOR BEATER MILL SR 300

Applications:	size reduction, deagglomeration
Feed material:	soft, medium-hard
Feed size*:	< 15 mm
Final fineness *:	< 50 µm

*depending on feed material and instrument configuration

TYPICAL SAMPLE MATERIALS

Beans, caraway, cinnamon sticks, cocoa beans, dried prawns, dried fruit and vegetables, grain, grape seeds, hard candy, herbs, nut shells, pepper, potato flakes, resins, rice, saffron, salt, seeds, spices, sugar, tea, etc.

PULVERIZING LARGE AMOUNTS OF SALT

Rock salt and sea salt not only consist of sodium chloride but may also contain other minerals and silicates, depending on the mining area and method. To analyze the composition of salt, the sample needs to be sufficiently homogenized, considering that larger lumps of rock salt are usually very inhomogeneous. The element concentrations in salt are usually very low so that it is frequently necessary to process amounts in the kilogram range. **The Rotor Beater Mill SR 300 easily pulverizes charges of several kilogram and is therefore ideally suited for this application**. Size reduction and deagglomeration of the sample is effected by impact and shearing. The feed material falls through the hopper into the grinding chamber where it is pulverized between rotor and sieve insert. As soon as the sample is smaller than the aperture size of the sieve, it falls into a collecting vessel attached to the mill. Grinding chamber, hopper and material inlet and outlet are made completely of stainless steel. The quick action door lock permits easy access to the grinding chamber for easy cleaning.

Salt was processed with the following parameters:

Sample	Salt
Feed quantity	5 kg
Feed size	< 10 mm
Speed	8,100 min^{-1}
Grinding tools	0.25 mm ring sieve, distance rotor
Grinding time	< 6 min
Final fineness	< 200 µm

A **distance rotor** was used to reduce frictional heat. Thanks to the 5liter collecting vessel, 5 kg of sample were pulverized in one run.

Rotor Beater Mill SR 300
www.retsch.com/sr300

From cacao tree to chocolate bar

Homogenization with mortar and pestle

What would the world be without chocolate? The production of chocolate is a fairly elaborate process, an important step of which is the reliable quality control of the raw material.

The way from the cacao tree to the chocolate bar is a long one. Due to the many processing stages – harvest, fermentation, drying, cleaning, roasting, pre-crushing and finally grinding the cocoa nibs to liquor from which cocoa powder and cocoa butter are pressed – **there may be significant differences in the quality.**

The quality control process not only includes testing the innocuousness of the product (e. g. fungi, heavy metal contamination) or the ingredients (fat, carbohydrate content), it also involves size reduction to an adequate particle size for a sensory test of the cocoa liquor. That is important because the human palate perceives particles as small as 30 microns as unpleasant.

Mortar Grinder RM 200
www.retsch.com/rm200

PERFORMANCE DATA

MORTAR GRINDER RM 200

Applications:	grinding, mixing and triturating
Feed material:	soft, hard, brittle, pasty, dry and wet
Feed size*:	< 8 mm
Final fineness*:	< 10 µm

*depending on feed material and instrument configuration

TYPICAL SAMPLE MATERIALS

Cocoa nibs, freeze-dried dough, lactose powder, nuts (without shell), oil seeds, spices, vitamin granulate

Before the actual quality control, the roasted beans are pre-crushed and liberated of impurities such as peel, germ and silver skin. The cocoa nibs thus gained are processed in roller mills to cocoa liquor. For the laboratory-scale quality test the mortar grinder RM 200 is ideally suited because its grinding principle is very similar to that of the industrial roller mills. Grinding the cocoa nibs produces a spreadable paste instead of a powder which is due to the high fat content of 54%. The process is facilitated by warming the grinding tools (mortar and pestle) and the sample material to 50 °C in a drying cabinet before grinding. This helps to reduce caking of the material to the grinding tools and the fat inside the nibs becomes thinner. The procedure allows to **produce a pourable cocoa mass in the RM 200 with a particle size smaller than 20 microns**. Now a representative sample can be extracted from the homogeneous paste for subsequent analysis. The same procedure is applied for grinding chocolate to analyze its fat, water and carbohydrate content.

MORTAR GRINDER
RM 200

- Dry, wet and cryogenic grinding
- Reproducible results due to adjustable pestle and scraper pressure
- Closed, dust tight grinding chamber with windows
- Easy exchange of mortar and pestle without tools
- Performance display for load control
- Adjustable pestle and scraper pressure
- Wide range of materials for contamination free grinding

MORTAR GRINDER RM 200

The Mortar Grinder RM 200 **mixes and homogenizes powders, suspensions and pastes even with high viscosity**. It is used for the reproducible sample preparation of soft, brittle, pasty and also hard products (up to 9 on the Mohs scale) for subsequent analysis and is suitable for dry as well as wet grinding.

The RM 200 **substitutes cumbersome hand mortars** by a high performance drive with electronic control. Thanks to the digital timer and scale for the pestle pressure reproducible results can be achieved. For processing difficult materials, the grinding set **can be cooled and heated**; it is also possible to use grinding aids. Grinding sets, consisting of mortar and pestle, are available in various material qualities ensuring neutral-to-analysis sample processing. The latest generation of the classic "Retsch Mill" is exceptionally powerful, safe and easy to operate and clean.

Cocoa nibs were processed with the following parameters:

Sample	Cocoa nibs
Feed quantity	150 g
Feed size	10 mm
Grinding tools	mortar and pestle made of agate
Grinding time	20 min
Final fineness	homogeneous paste

SIEVE SHAKER
AS 200 CONTROL

- 3-D throwing motion, powerful electromagnetic drive
- Excellent separation efficiency even with short sieving times
- Setting of sieve acceleration "g" for comparable and reproducible sieving results worldwide
- Free digital adjustment of all process parameters

In food processing companies quality control serves to ensure and maintain the requirements posed on the products. To meet consumer expectations food safety is not only required during the manufacturing process, the quality of the raw materials is also crucial. Continuous inspections of incoming raw materials are a precondition for a consistently high product quality of the end product.

Measuring Dust

Incoming inspection with sieve analysis

Graduate student Jennifer Franz has developed a new inspection procedure during her work at the German food producer Lebensgarten GmbH. With the help of sieve analysis the company can now reliably **ascertain the fines and dust fractions of incoming cereal flakes**; these have a negative influence on the mixing and packaging process of muesli. The dust fraction consists of particles < 500 microns and prevents tight sealing of the packaging by sticking to the welding seam. Another negative effect occurs during the production of so-called "crunchy" products. Crunchies are crisply baked cereal flake products; by adding honey, for example, the ingredients are formed into a compact mass and are then baked. The higher the dust fraction, the more crumbly and fine-pored the consistency of the crunchies becomes. Separating the flakes into individual fractions by sieve analysis reduces these negative effects on the product quality by allowing reliable quality evaluation.

The mixture of cereal flakes can be divided into different particle size fractions, roughly whole flakes, half flakes, broken flakes and fines. The aperture sizes of the sieves used are 4 mm, 2 mm, 1 mm, 0.5 mm and < 500 µm (sieve bottom). This last fraction is considered as the dust fraction which has a particularly negative influence on the packaging and production process. Before selecting the time intervals for sieve analysis, the structural composition of the flakes was evaluated. Based on the fact that cereal flakes are categorized as a fragile raw material which breaks easily and is subjected to natural deviations, a time interval of 2 minutes was defined. To ascertain a suitable amplitude for each flake type, care was taken that the flakes were not thrown with too high or too low intensity. If the throwing intensity is too low, the flakes are not properly dispersed resulting in low separation efficiency. If the amplitude is set too high the low-density particles are held in suspension and a comparison of individual flakes with the sieve apertures is not possible. For flakes of oat, wheat, rye, spelt or barley amplitudes of 0.9 or 1.00 mm (instable flakes) and 1.5 mm (stable flakes) were chosen according to the stability of the flakes. 4 different sieving protocols were defined for the cereal flakes by experimental sieve analyses. This test series shows that sieve analysis is a highly suitable method to control the quality of the raw materials by separating the fines and dust fractions.

PERFORMANCE DATA

VIBRATORY SIEVE SHAKER AS 200 CONTROL

Applications:	separation, fractioning, particle size analysis
Feed material:	powders, bulk materials, suspensions
Measuring range*:	20 µm – 25 mm
Max. batch:	3 kg

*depending on feed material and sieve stack used

TYPICAL SAMPLE MATERIALS

Citric acid, coffee, flour, food granulates, gelatin, herbs, hop, lentils, liver sausage, nuts, paprika, rice bran, seeds, spices, sunflower seeds, sugar, tobacco, etc.

VIBRATORY SIEVE SHAKER AS 200 CONTROL

Jennifer Franz carried out the test series with a Sieve Shaker AS 200 control. The **patented controllable electromagnetic drive** of RETSCH's vibratory sieve shakers allows for opti-mum adaptation to the material to be sieved. The drive produces a 3D throwing motion that moves the sample equally over the whole sieve surface and effectively separates particles in a size range from 20 µm to 125 mm. The sieve shakers accept a variety of sieve diameters from 100 mm to 450 mm. Sieve stacks with a maximum height of 450 mm allow for separation of up to 17 fractions in one analysis. **The "control" series features digital setting and control of amplitude, time, sieve acceleration and interval**. Up to 9 parameter combinations can be stored to conveniently repeat routine analyses.

MEAN VALUES AND STANDARD DEVIATIONS

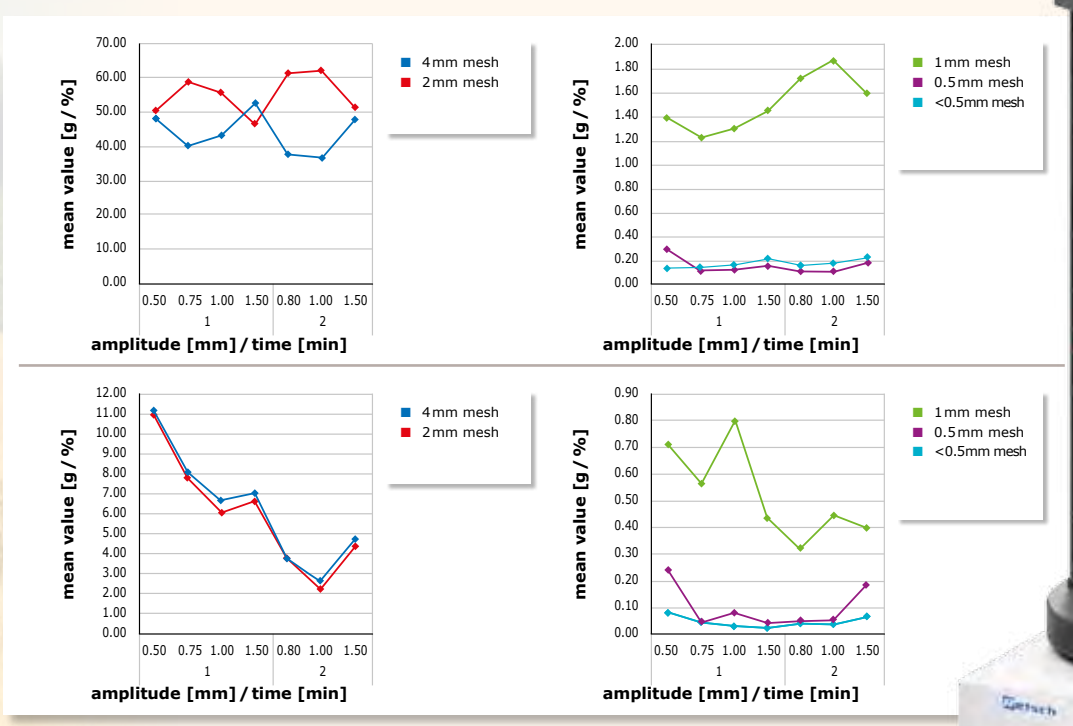


Fig. 1: Mean values (above) and standard deviations (below) of the sieve fractions of *Bio oat flakes grbl. 480er*, sieved with varying parameters (amplitude and time).

The lowest standard deviations occur when sieving for 2 min and with an amplitude of 1 mm. Hence, this is the most suitable sieving protocol for cereal flakes and is now used at Lebensgarten for their quality control process.

Vibratory Sieve Shaker AS 200 control
www.retsch.com/as200

Retsch TECHNOLOGY

PARTICLE ANALYZER
CAMSIZER® XT

- Digital image processing with patented two-camera-system
- Dynamic measuring range from 1 µm to 8 mm
- Reliable detection of smallest amounts of "undersize" and "oversize"
- Very short measurement time of 1 - 3 minutes
- Modular system X-Change for dry and wet dispersion
- Measurement results are 100% compatible to sieve analysis

With an estimated 1.4 billion cups of coffee consumed worldwide every day, there is a great and ever increasing demand for coffee beans in the global market. The taste of coffee is influenced by the roasting of the beans, their grinding degree and the method and quality of the preparation. Different types of preparation by brewing and filtering (e. g. espresso machine, filter coffee or mocca) require different grinding degrees of the coffee powder to obtain an aromatic result. When roasted beans are ground to powder, the determination of the particle size is a critical aspect because the particle size influences the brewing and filtering properties and thus the taste and salubriousness of the coffee.

Particle Analyzer CAMSIZER® XT
www.retsch-technology.com/camsizerxt

Aromatic Beans

Particle size and shape analysis of coffee powder

FINENESS MATTERS

The preparation of coffee involves optimum extraction of the ingredients which is achieved, for example, by applying hot water when brewing the coffee. The finer the coffee is ground the more ingredients can be extracted in less time. If the grinding degree is not perfectly attuned to the time and temperature of the brewing procedure, there is a danger that the coffee is "over- or under-extracted". An under-extracted coffee (= ground too coarsely) is not very aromatic whereas an over-extracted coffee (= ground too finely) tastes bitter due to too many bittern and tannic acids.

With the reliable determination of the particle size a reproducible grinding degree can be set in accordance with the respective preparation method to produce an aromatically balanced coffee. Due to the high oil content, wide particle size distribution and irregularly shaped particles coffee powder is difficult to pour, i.e. the particles strongly tend to agglomerate, don't flow easily and can hardly be conveyed. These aspects need to be taken into account by the mechanical and optical measuring methods.

Whereas traditionally, the particle size distribution of coffee powder was determined by test sieving, today laser diffraction has become the established standard method. Both sieve analysis and laser diffraction have methodical and technical limits. With sieve analysis the strongly agglomerating coffee powder is not sufficiently dispersed to exactly determine the coarse and fine fractions. Laser diffraction permits better dispersion of the powder with air pressure or in liquid. However, the limited dynamic measuring range and the low resolution and sensitivity for larger particle sizes prevent this method from reliably detecting the coarse fraction. Moreover, neither of these methods provides information on the particle shape.

RETSCH Technology's optical particle analyzer CAMSIZER XT is based on Dynamic Image Analysis and has become a superior alternative to laser diffraction, not only for measuring coffee powder.

The typical particle size distribution of coffee powder includes distinctive coarse and fine fractions. The fine fraction lies below 200 microns, the coarse particles can be up to 2 mm in size. Thanks to the patented Dual Camera System and the resulting wide dynamic measuring range both fractions can be determined with high resolution and good statistical validation by CAMSIZER XT. This can be seen in figure 1 which shows coffee powders of various grinding degrees. The measurement was carried out with air pressure dispersion in the X-Jet module (fig. 2). RETSCH Technology has developed an appliance for vibratory feeding of the powder on a chute which allows smooth conveyance to the measurement area even of coffee powder.

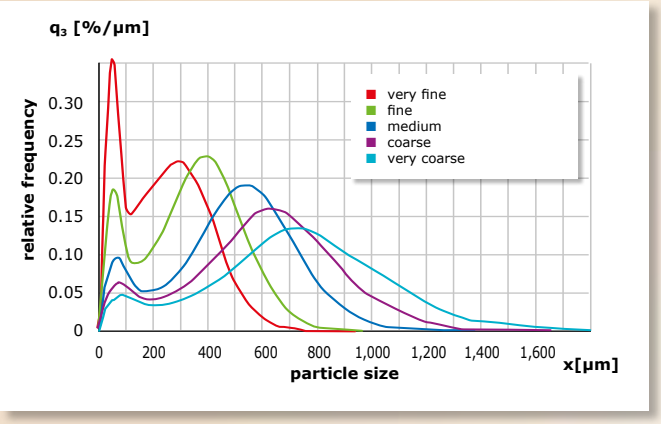


Fig. 1: Measurement of 5 coffee samples with different degrees of coarseness with CAMSIZER XT

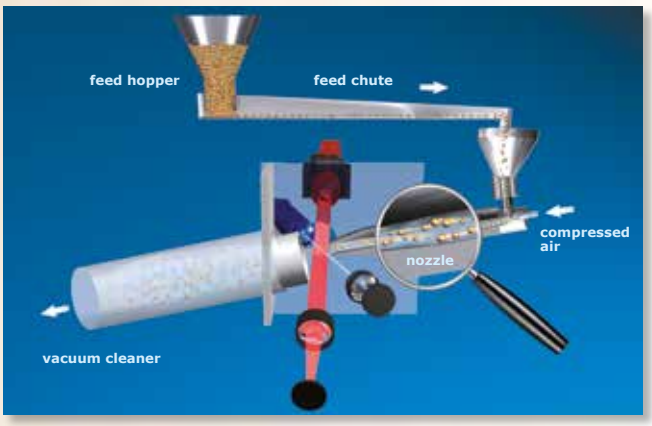


Fig. 2: Schematic drawing of the X-Jet dispersion module

INFLUENCE OF THE PARTICLE SHAPE

The degree of roasting influences the brittleness of the coffee beans. Ground coffee from brittle beans often contains edgy or spiky grains which lead to a lower packing density of the compacted coffee. Both particle size distribution and particle shape have an impact on the bulk density, the filter and extraction properties of the powder and thus also on the quality of the prepared coffee. Figures 3a and 3b show that CAMSIZER XT simultaneously determines the width, length and also the diameter of equivalent circle of the particles with image analysis. The results for these parameters differ greatly which is typical for irregular particle shapes. The result of laser diffraction, in comparison, which defines all particles as spheres, only provides a cross section of the width and length of the particles. Fig. 3b clearly shows that laser diffraction does not correctly detect particles which are larger than 1 mm.

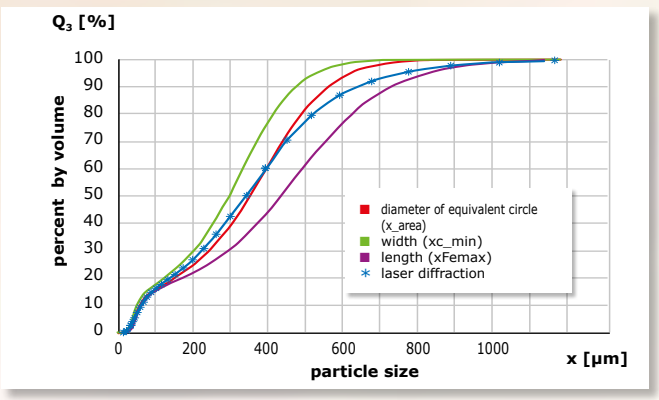


Fig. 3a: Measuring results of a coffee sample, ~ 10 µm – 1000 µm, comparing CAMSIZER XT and laser diffraction.

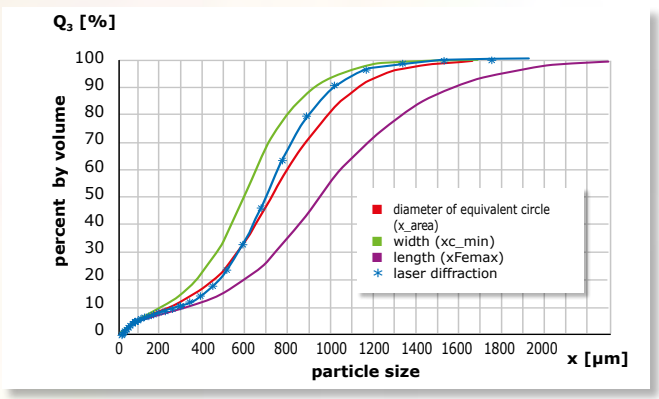
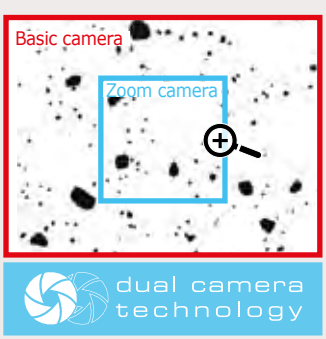


Fig. 3b: Laser diffraction does not correctly detect the fraction >1 mm in a coffee sample with particles from ~ 10 µm – 2000 µm



PERFORMANCE DATA

PARTICLE ANALYZER CAMSIZER® XT

Measuring principle:	Dynamic Image Analysis (ISO 13322-2)
Measuring range:	1 µm – 8 mm 10 µm – 8 mm (free fall dispersion) 1 µm – 4.5 mm (air pressure dispersion) 1 µm – 600 µm (dispersion in liquid)
Type of analysis:	dry and wet measurement
Measuring time:	~ 1 – 3 minutes

TYPICAL SAMPLE MATERIALS

Additives (vitamins, citric acids, sulfites etc.), coffee (powder, freeze-dried and instant granulate), food powder and granulate, granulated products (spray and fluid bed), salt, spices, sugar, etc.

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