Retsch®

MILLING SIEVING ASSISTING

RETSCH BALL MILLS

TIPS & TRICKS FOR JAR FILLING, HANDLING & CLEANING OF ACCESSORIES

part of VERDER scientific

GUIDELINES FOR SAMPLE AMOUNT, GRINDING BALLS AND SPEED

The rule of thumb is that the grinding balls should be approximately 3 times larger than the largest particle in the sample to be ground. In addition to the machine settings, the filling level of the grinding jars is also essential for the success of a grinding process. When grinding bulk materials, a jar filling should consist of approximately 1/3 sample material and 1/3 ball quantity. The remaining third is free volume, which is required for the movement of the balls.

If volume increase or decrease is to be expected during grinding, the sample quantity can be adjusted within the range shown in the table (in case of volume decrease possibly beyond).

For wet grinding with ball sizes <3 mm, the ball filling should be approx. 60 % of the grinding jar volume, while the sample filling should be 30 %, just like for dry grinding. Based on the density of the respective material, the required number of balls can be converted as the mass to be weighed in.

CONTENT

HIGH-ENERGY BALL MILL EMAX

The Emax is an entirely new type of ball mill for high energy milling. The unique combination of high friction and impact results in extremely fine particles within the shortest amount of time.

HIGH-ENERGY BALL MILL EMAX

			Dry grinding Recommended ball filling (pieces)					**Wet grinding** Recommended ball filling (mass, g)
Jar volume	**Sample amount**	**Max. Feed size**	**Ø 5 mm**	**Ø 7 mm**	**Ø 10 mm***	**Ø 12 mm***	**Ø 15 mm***	**Ø ≤ 3 mm**
50 ml	5–20 ml	4 mm	160	45	16	8–12	-	Zirconium oxide: 110 g Stainless steel: 145 g Tungsten carbide: 276 g
125 ml	15–50 ml	5 mm	400	110	50	35	15–18	Zirconium oxide: 275 g Stainless steel: 364 g

	Ball filling Ø > 10 mm
Grinding jar material	**Recommended max. speed (rpm)**
Stainless steel	1500
Tungsten carbide (WC)	1200
Zirconium oxide	

Grinding jars in 3 different materials

Aeration lids

PLANETARY BALL MILLS
PM 100 / PM 200 / PM 300 / PM 400

Planetary Ball Mills meet and exceed all requirements for fast and reproducible grinding to analytical fineness. They are used for the most demanding tasks in the laboratory, from routine sample processing to colloidal grinding and advanced materials development.

PLANETARY BALL MILLS PM 100 / PM 200 / PM 300 / PM 400

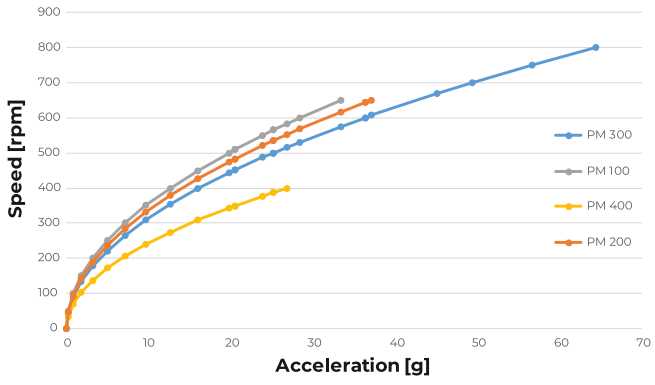
			Dry grinding Recommended ball filling (pieces)						**Wet grinding** Recommended ball filling (mass, g)
Jar volume	**Sample amount**	**Max. Feed size**	**Ø 5 mm**	**Ø 7 mm**	**Ø 10 mm***	**Ø 15 mm**	**Ø 20 mm**	**Ø 30 mm**	**Ø ≤ 3 mm**
12 ml	< 5 ml	< 1 mm	50	15	5	-	-	-	Hardened stainless steel: 35 g
25 ml	< 10 ml	< 1 mm	95–100	25–30	10	-	-	-	Hardened stainless steel: 75 g
50 ml	5–20 ml	< 3 mm	200	50–70	20	7	3–4	-	Zirconium oxide: 110 g Hardened stainless steel: 145 g Tungsten carbide: 275 g
80 ml	10–35 ml	< 4 mm	250–330	70–120	30–40	12	5	-	Hardened stainless steel: 235 g Tungsten carbide: 440 g
125 ml	15–50 ml	< 4 mm	500	110–180	50–60	18	7	-	Zirconium oxide: 275 g Hardened stainless steel: 365 g Tungsten carbide: 690 g
250 ml	25–120 ml	< 6 mm	1100–1200	220–350	100–120	35–45	15	5	Zirconium oxide: 550 g Hardened stainless steel: 730 g Tungsten carbide: 1380 g
500 ml	75–220 ml	< 10 mm	2000	440–700	200–230	70	25	8	Zirconium oxide: 1100 g Hardened stainless steel: 1450 g

A successful grinding process in a RETSCH planetary ball mill is not only influenced by the machine settings, but also by the filling level of the grinding jars. The usable capacity of the jars depends on the type of material. The indicated numbers of balls are the minimum quantity per jar. Better results are achieved by using the larger number of suitable grinding balls, if specified. In exceptional cases, the number of balls can be reduced by up to 15 %, however, increased wear of the grinding tools is to be expected.

The mill automatically regulates its maximum speed depending on the individual grinding process (jars and balls, sample, etc.). The table shows the maximum speeds to be expected for dry grinding processes in steel jars. Please note that using materials other than steel (especially with grinding balls >15 mm in diameter) can lead to increased wear or even damage.

Due to the high energy input from mill to sample, but also to the grinding jar and balls, the information on speed limits should be observed when working with materials other than steel. When using grinding balls larger than Ø 15 mm, it is recommended to select lower speeds. This will prevent material caking on the jar walls and balls during dry grinding. In addition, signs of wear and damages to jars and balls are reduced.

In practice, an upper limit of approx. 500 rpm has proven to be favorable for grinding with balls larger than Ø 15 mm. For the natural material agate, even lower speeds are recommended.



Speed-dependent acceleration in different planetary ball mills

MIXER MILLS
MM 400 / CRYOMILL / MM 500 VARIO

Mixer mills grind and homogenize small sample volumes quickly and efficiently by impact and friction. These ball mills are suitable for dry, wet and cryogenic grinding as well as for cell disruption for DNA/RNA or protein recovery. For special applications such as mechanosynthesis, they offer unique solutions. Mixer mills are well known for their ease of use and small foot-print compared to other types of ball mills.

MIXER MILLS MM 400 / CRYOMILL / MM 500 VARIO

			Dry grinding Recommended ball filling (pieces)			
Jar volume	**Sample amount**	**Max. Feed size**	**Ø 5 mm**	**Ø 7 mm**	**Ø 10 mm**	**Ø 12 mm**
1.5 ml	0.2–0.5 ml	1 mm	1–2	-	-	-
5 ml	0.5–2 ml	2 mm	5–6	1–2	1	-
10 ml	2–4 ml	4 mm	17–20	9–12	1–2	1–2
25 ml	4–10 ml	6 mm	35–40	16–20	5–6	2–4
35 ml	6–15 ml	6 mm	55–60	25–30	6–9	4–6
50 ml	8–20 ml	8 mm	80–90	45–50	12–14	6–8

			Dry grinding Recommended ball filling (pieces)			**Wet grinding** Recommended ball filling (mass, g)
Jar volume	**Sample amount**	**Max. Feed size**	**Ø 15 mm**	**Ø 20 mm**	**Ø 25 mm***	**Ø ≤ 3 mm**
1,5 ml	0.2–0.5 ml	1 mm	-	-	-	Hardened stainless steel: 4,5 g
5 ml	0.5–2 ml	2 mm	-	-	-	Hardened stainless steel: 15 g
10 ml	2–4 ml	4 mm	-	-	-	Zirconium oxide: 20 g Hardened stainless steel: 30 g Tungsten carbide: 55 g
25 ml	4–10 ml	6 mm	1–2	-	-	Zirconium oxide: 55 g Hardened stainless steel: 75 g Tungsten carbide: 140 g
35 ml	6–15 ml	6 mm	2–3	1	-	Zirconium oxide: 75 g Hardened stainless steel: 105 g
50 ml	8–20 ml	8 mm	3–4	1	1	Hardened stainless steel: 145 g

Disposable 1.5 ml / 2 ml reaction vials are also used in mixer mills. In the MM 400, 5 ml reaction vials, 30 ml wide-mouth bottles or 50 ml conical centrifugal tubes can be employed as well. The MM 500 vario accommodates 1.5 ml / 2 ml / 5 ml reaction vials. The 50 ml centrifugal tubes are suitable for dry grinding only to a limited extent. For special applications, such as homogenization of tissue samples or dried plants in centrifugal tubes, please contact our Applications Team.

			Dry grinding Recommended ball filling (pieces) Stainless steel or zirconium oxide				**Cell disruption of biological cells**
Vial	**Sample amount**	**Max. Feed size**	**Ø 4 mm**	**Ø 5 mm**	**Ø 7 mm**	**Ø 10 mm**	**Glass beads** (0.1–0.25 mm/0.25–0.5 mm/0.75–1 mm/ 1–1.5 mm) **Zirconium oxide grinding balls** (< 3 mm)
1.5 ml	0.2–0.5 ml	< 1 mm	2–4	-	-	-	~ 0,75 ml
2 ml	0.3–0.75 ml	< 2 mm	3–6	2–4	1–2	-	~ 1 ml
5 ml	0.5–2 ml	< 2 mm	12	-	-	-	~ 2,5 ml
30 ml*	5–12 ml	< 5 mm	40–45	20–22	10–14	6–10	~ 15 ml
50 ml*	8–20 ml	< 4 mm	-	-	-	-	~ 25 ml

** Please note that a total weight of 650 g per grinding jar holder should not be exceeded in the MM 400.*

MIXER MILLS
MM 500 NANO / CONTROL

Please note that the maximum permissible ball size may vary depending on the material!
The MM 500 nano is particularly suitable for wet grinding.
Dry, wet and cryogenic grinding down to - 100 °C can be carried out in the MM 500 control.

MIXER MILLS MM 500 NANO / CONTROL

				Dry grinding Recommended ball filling (pieces)						
Material	**Jar volume**	**Sample amount**	**Max. Feed size**	**Ø 5 mm**	**Ø 7 mm**	**Ø 10 mm**	**Ø 12 mm**	**Ø 15 mm**	**Ø 20 mm**	**Ø 25 mm**
Hardened stainless steel	10 ml	2–4 ml	4 mm	32	12	3	1	1	-	-
	25 ml	4–10 ml	6 mm	116	35	12	4	2	1	-
	50 ml	5–20 ml	8 mm	160	45	16	8–12	-	1	1
	80 ml	10–32 ml	10 mm	260	70	32	23	12	3	1
	125 ml	15–50 ml	10 mm	400	110	50	35	15–18	8	-
Zirconium oxide	50 ml	5–20 ml	8 mm	160	45	16	8–12	-	-	-
	80 ml	10–32 ml	10 mm	260	70	32	23	12	-	-
	125 ml	15–50 ml	10 mm	400	110	50	35	15–18	-	-
Tungsten carbide	50 ml	5–20 ml	8 mm	160	45	16	8–12	-	-	-
	80 ml	10–32 ml	10 mm	260	70	32	23	12	-	-

			Wet grinding Recommended ball filling (pieces)
Jar volume	**Sample amount**	**Max. Feed size**	**< Ø 3 mm**
10 ml	5–20 ml	8 mm	Hardened stainless steel: 32 g
25 ml	5–20 ml	8 mm	Hardened stainless steel: 90 g
50 ml	5–20 ml	8 mm	Zirconium oxide: 110 g Hardened stainless steel: 145 g
80 ml	10–32 ml	10 mm	Zirconium oxide: 176 g Hardened stainless steel: 232 g
125 ml	15–50 ml	10 mm	Zirconium oxide: 275 g Hardened stainless steel: 364 g

Grinding jars for MM 500 nano / control

GRINDING JARS – HANDLING AND CLEANING

The following instructions for handling and cleaning the grinding jars must be observed to prevent damage.

All grinding jars, including those with glued-in ceramic inserts, and grinding balls can be cleaned with alcohol, petroleum ether or normal household detergent.

It is also possible to clean them in a dish washer.

DRYING

Drying of the grinding jars after cleaning can be carried out in a drying cabinet at the temperatures specified below:

Jar material Temperature
Hardened steel up to 200 °C
Hardened stainless steel up to 200 °C
Tungsten carbide (WC) up to 120 °C
Zirconium / Agate / Sintered corundum up to 120 °C

USAGE

Cryogenic grinding may only be carried out with grinding balls and jars made of stainless or hardened steel. Balls and jars made of zirconium oxide or tungsten carbide are not suitable for grinding at cryogenic temperatures.

Wet grinding using highly flammable materials is permitted in grinding jars provided certain precautions are observed. When using grinding aids such as isopropanol, ethanol, petroleum ether or similar, the interior of the jar must be classified as Zone 0, i. e. a permanently present explosive mixture.

GENERAL

Acceptable liquids include: **ethanol, isopropanol, petroleum ether, water, mild soap suds, up to 5% acetic acid** or **up to 5% Na_2CO_3**

Grinding jars for MM 400 and MM 500 vario

Accessories for MM 400 and MM 500 vario

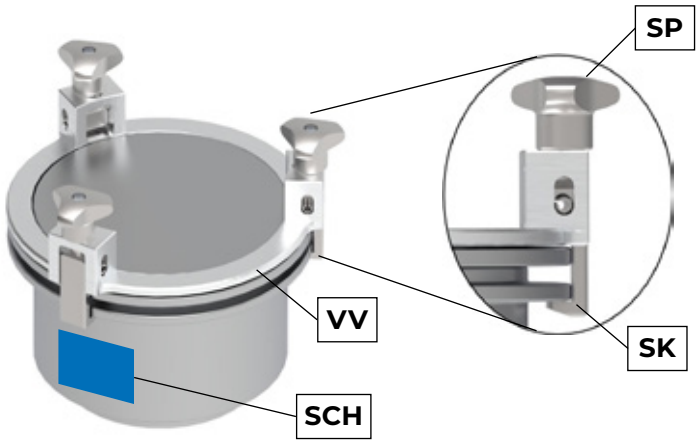
SAFETY CLOSURE DEVICE FOR PM 100 / PM 200 / PM 300 / PM 400

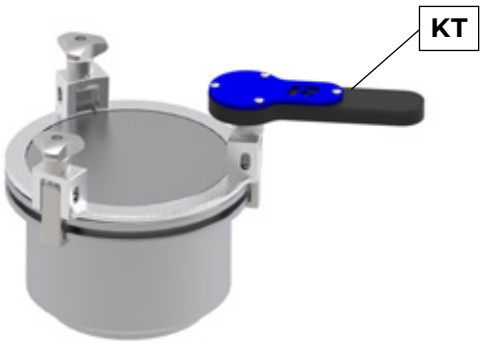
In the cases of wet grinding, an expected pressure increase in the grinding jar or when using aeration lids, the grinding jar needs to be closed with a safety closure device during the grinding process.

For the ball mills Emax and MM 500 nano / control, the safety closure device is no optional accessory but integrated in the jar lid.

PLEASE NOTE

- For wet grinding processes, please only use the type of closure device as illustrated below.





Close the grinding jar with the lid and place the closure device (VV) centrally on the jar lid. Align the closure device (VV) so that one of the safety clamps (SK) is at the level of the labeling field (SCH) of the grinding jar.

PLEASE NOTE

- The safety clamps (SK) of the closure device need to fully enclose the rim of the grinding jar to avoid unwanted opening of the jar.

Tighten the three clamping screws **(SP)** of the closure device evenly with the opening and closing aid **(KT)** to at least 8 Nm. Internal pressures of up to max. 5 bar are only permissible with this pretension. After tightening the closure device, check whether the three screws of the safety clamps **(SK)** and the closure device **(VV)** are still tight. The jar lid must lie on the jar without a gap.

Only open the grinding jar at a safe place (suction device) after it has cooled down.

Please note that the grinding jars may get warmer than 100 °C, depending on the size, ball filling, machine speed and grinding time.

AERATION LID FOR PM 100 / PM 200 / PM 300 / PM 400 AND MM 500 NANO / CONTROL & EMAX

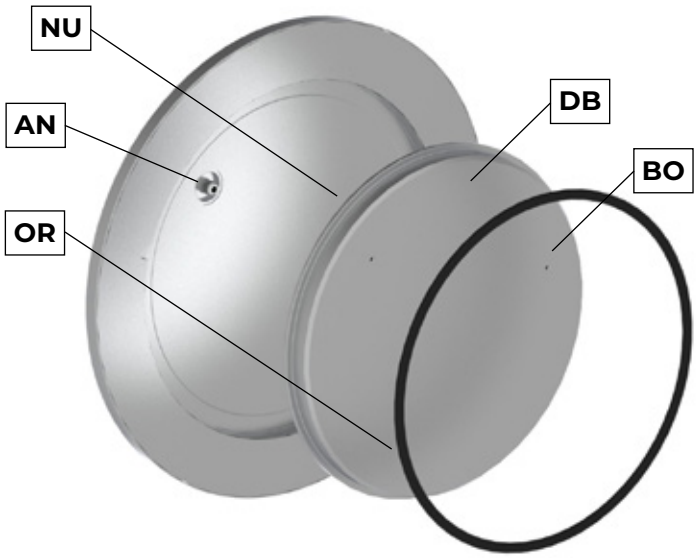
The handling described below applies to grinding jars of the PM 100 / PM 200 / PM 300 / PM 400 and MM 500 nano / control as well as Emax.

Instead of the regular grinding jar lids, aeration lids can also be used. These have special connections that enable grinding in a protective atmosphere. The handling of the aeration lid is identical to the standard lids.

To ensure that the material of the lid matches the material of the bowl, the lid base plate can be replaced. Always make sure that the size of the lid base plate matches the selected jar volume. Removing the lid base plate allows easy cleaning of the connections. Lid base plates are available from Retsch GmbH in various material types and sizes.

Follow these steps to exchange the lid base plate.

1. Carefully remove the O-ring (OR) from the notch (NU) of the lid base plate.
2. Carefully remove the lid base plate.
3. Insert the new lid base plate in such a way that the holes (BO) in the lid base plate match the connections (AN) in the jar lid.
4. Insert the intact O-ring (OR) into the notch. Tip: Alternately press the O-ring in crosswise to prevent stresses in the material.



GRINDCONTROL FOR PM 100 / PM 300 / PM 400, MM 500 NANO / CONTROL AND EMAX

The handling described below applies to grinding jars of the PM 100 / PM 300 / PM 400 and MM 500 nano / control as well as Emax.

The sensor unit and the GrindControl software are used for continuous recording of the status variables pressure and temperature in a grinding jar. The measured values of the sensors located in the jar lid are transmitted wirelessly to a receiving system on the PC. The temperature sensor is thermally decoupled from the jar lid and measures the gas temperature inside the grinding jar. The pressure sensor measures the difference in gas pressure between the jar interior and its surroundings.

To enable usage of the sensor unit for grinding in a protective atmosphere, the lid has connections for gas flushing (15.3) (M8x1 connection thread).

"Comfort" grinding jar design (left) and EasyFit design (right)

PLEASE NOTE

- The GrindControl system for Planetary Ball Mills can only be used with the EasyFit grinding jars. Grinding jars in "comfort" design have different dimensions and are therefore not compatible.

GrindControl PM lid

PLEASE NOTE

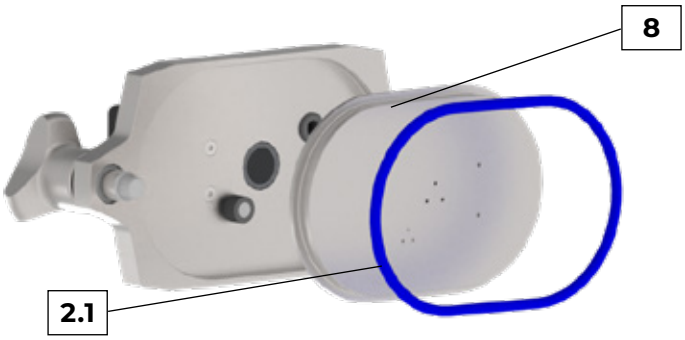
- Applies to all units compatible with planetary ball mills: To avoid deposits in the openings of the valve ports, a sinter filter (identical to the sinter filter protecting the pressure sensor) can be used. This protects the valve ports and reduces the cleaning effort.

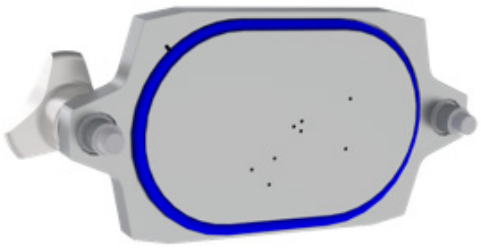
When using the gassing function, make sure that the ventilation ducts on the underside of the sensor unit are clear.

PLEASE NOTE

- The ventilation ducts have to be cleaned with the enclosed tools when required.

To match the material of lid and jar, the base plate of the lid can be exchanged. Care must be taken to select a base plate that fits the jar size.
The base plate is exchanged by following these steps:





1. Carefully remove the outer sealing ring (2.1) with the help of the enclosed tool.
2. Remove the lid base plate (8).
3. Insert new lid base plate (8).
4. Re-attach the previously removed sealing ring (2.1) to the base plate (8). Use the enclosed tool to firmly press the sealing ring into the notch.

PLEASE NOTE

- The radio module of the sensor unit, which is located on the top side, must not get wet! Contact with water will damage the electronics.
- Acidic or oxidizing samples should not be homogenized in the grinding jar or come into contact with the sensor unit, as this will lead to damages.

WARNING

- **Carefully read the operation manual of the ball mill you are using!**
  The document at hand only refers to the correct usage of GrindControl.
- Do not use grinding balls with a diameter smaller or equal to 1 mm! These could block the ventilation ducts.

Lock the grinding jar as follows:

1. Ensure that the joining surface between the grinding jar (14) and lid (15) is free of foreign bodies to guarantee tightness.
2. Place the lid (15) on the jar (14) to fit and close the grinding chamber.
3. Tighten both clamping screws (15.5) of the jar lid evenly to avoid tilting and to close the grinding jar (14).
4. For tight sealing, use the opening aid to tighten the clamping screws.

Hand-tightening the clamping screws is insufficient to seal the grinding jar absolutely tight.

To ensure reliability and operational safety of the sensor unit, cleaning must be carried out after each grinding.

PLEASE NOTE

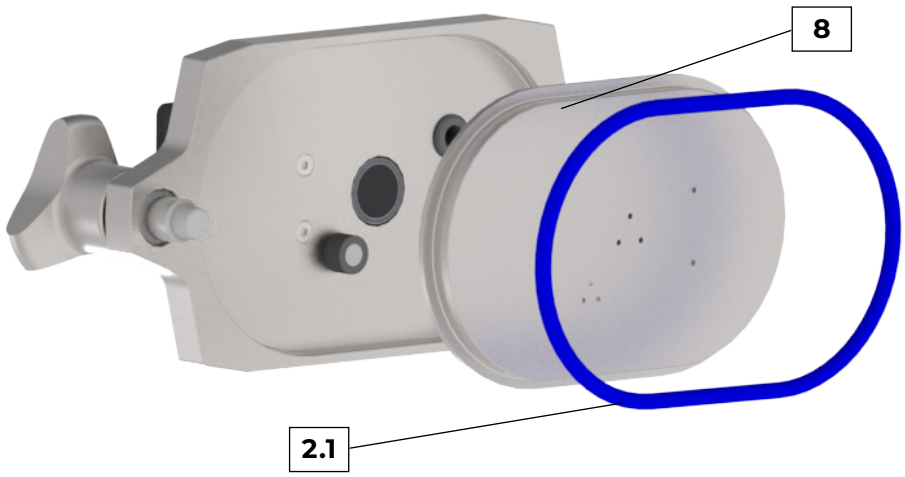
- The radio module of the sensor unit, which is located on the top side, must not get wet!

The lid base plate, the sinter filter and the O-rings can be cleaned in an ultrasonic bath.

The ventilation ducts of the lid base plate (10) can be cleaned carefully with the enclosed tool if they are dirty.

Cleaning of the ventilation ducts as follows:

1. Carefully remove the outer sealing ring (2.1) using the enclosed tool.
2. Remove lid base plate (8).
3. Carefully clean the ventilation ducts of the lid base plate (8) with the cleaning tool.



The sinter filter, the lid base plate and the O-rings can be cleaned in an ultrasonic bath if required.
To do this, disassemble the GrindControl as described in the previous text and place the items in the ultrasonic bath.

ATTENTION
These instructions do not contain any information on operating an ultrasonic bath.
Please refer to the operation manual enclosed with your ultrasonic bath.

GRINDING TOOLS MADE OF CERAMIC OR NATURAL STONE

In individual cases, grinding tools may be damaged during transport despite the packaging. Therefore, please check the goods directly when unpacking them. Grinding jars are not subject to the standard warranty, but are regarded as wear parts. We therefore ask for your understanding that we cannot accept complaints about ceramic or natural stone grinding jars that have already been used.

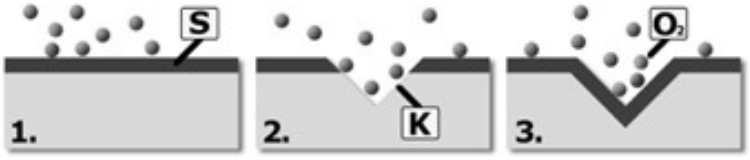
Unfortunately, it is not always possible to ensure that the color of the high-performance ceramics we supply is completely uniform. The color differences are due to phase boundary phenomena at the level of the submicroscopic crystalline microstructures and have no influence on the mechanical and chemical properties of RETSCH products.

Our products made of high-performance ceramic materials such as sintered corundum, zirconium oxide, tungsten carbide or silicon nitride are manufactured from high-purity starting materials. RETSCH guarantees purity in accordance with the applicable material specifications. This ensures high reproducibility with regard to properties like abrasion behavior, hardness, flexural strength, density, chemical inertness, corrosion resistance, etc.

Grinding jars made of natural stone or ceramic should be handled with utmost care. **Sufficient grinding media filling is of particular importance and absolutely necessary.**

If you operate your ball mill with high frequency or speed and large ball diameters, insufficient filling of the grinding media will inevitably lead to destruction of the grinding jar and balls. Only when the jars are sufficiently filled can the sample act as a protective jacket between the ball and the grinding chamber surface.

Ceramic grinding tools are inappropriate for pulverizing graphite or materials containing graphite.



GRINDING TOOLS MADE OF STEEL

Corrosion may occur in some steel grades under certain conditions. This does not represent a quality defect.

CORROSION-RESISTANT STEEL

This material is not completely corrosion-free despite its high chromium content. The resistance of "stainless" steels is due to an extremely thin, invisible protective oxide layer **S**, the so-called passive layer **(1.)**. As grinding tool surfaces are subjected to mechanical stresses, damage to this protective oxide layer results in minor corrosion spots **K (2.)**. In the case of steels with a chromium content of 13 % or more, this protective layer is constantly formed anew by reaction with the atmospheric oxygen **O_2 (3.)**.

This does **not represent a quality defect** and may occur despite **excellent material quality**.

PLEASE NOTE

- To avoid contamination, corrosion-resistant materials are not additionally treated with anticorrosive!

HARDENED STEEL

Hardened steel has a lower chromium content and does not form a protective oxide layer. Therefore, it is not characterized by corrosion resistance, but by its greater hardness. We protect these grinding tools with a special wax. Please remove the wax before the first use with water and washing-up liquid. Please dry the grinding tool well.

For longer storage times of the grinding tools, we recommend the use of an anti-corrosion oil. If corrosion has nevertheless formed, it can be removed with household scouring agents.

Notes

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