



PLANETEN-MIKROMÜHLE PULVERISETTE 7 *premium line*

Operating Manual



translation of the original

pulverisette
premium line



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Fritsch GmbH, Laborgerätebau has been certified by the TÜV-Zertifizierungsgemeinschaft e.V. Certificate registration number 71 100 4 008



It was verified through an audit that Fritsch GmbH satisfies the requirements of DIN EN ISO 9001:2000.

The enclosed conformity statement states the directives satisfied by the planetary micro mill "PULVERISETTE 7 *premium line*" in order to bear the CE mark.



Instrument number 07.5000.00

Applies as of serial number 0100

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1 General / introduction

1.1 Notes about the operating manual

- The copyright to these technical documents is held by Fritsch GmbH, Manufacturers of Laboratory Instruments.
- These operating instructions are not to be reprinted or copied without the express approval of Fritsch GmbH.
- Study the operating manual carefully.
- All operators must be familiar with the contents of the operating manual.
- Please follow the notes for your safety.
- The instrument was designed from a perspective of user safety; however, some residual dangers could not be eliminated. To prevent endangering of the users, the instructions in this operating manual must be followed.
- The symbols in the right hand margin highlight the risks described in the text.
Some symbols may also be found on the instrument and warn against possible inherent hazards.
Warning symbols are surrounded by a triangle.
- This operating manual does not constitute a complete technical description. It describes only the details required for safe operation and maintenance for usage under normal conditions.



Caution!
Follow the
operating manual

1.2 Explanation of the symbols used on the device and in the operating manual

<p>Attention! Warning against danger spot Observe operating manual</p>	
<p>Attention! Mains voltage</p>	
<p>Attention! Danger of explosion</p>	
<p>Attention! Hot surface</p>	
<p>Attention! Inflammable substances</p>	
<p>Wear protective gloves!</p>	
<p>Wear ear protection!</p>	
<p>Wear safety goggles!</p>	
<p>Do not stand under suspended load!</p>	
<p>Attention! Danger of crushing or pinching</p>	

1.3 Brief description of the machine

1.3.1 Fields of application

The planetary micro mill "PULVERISETTE 7 premium line" is universally applicable for quick dry or wet grinding of inorganic and organic samples for analysis, quality control and materials testing.

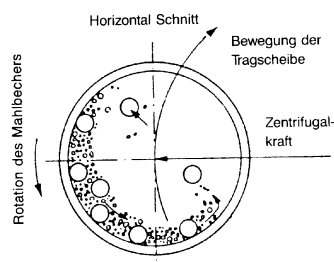
In synthesis, the planetary micro mill can be used for mixing and homogenisation of dry samples, emulsions and pastes.

1.3.2 Method of operation

The grinding material is pounded and ground by grinding balls in a grinding bowl. Centrifugal forces from the rotation of the grinding bowl around its own axis and the rotating supporting disc act on the grinding bowl filling consisting of the grinding material and grinding balls.

The grinding bowl and supporting disc rotate in opposite directions so that the centrifugal forces alternately act in the same and opposite directions.

This results in the grinding balls running along the inner wall of the grinding bowl as a frictional effect and the balls impacting against the opposite wall of the grinding bowl as an impact effect.



1.3.3 Driving motor and speed control

The machine is driven by a maintenance-free electric motor that is operated with a frequency converter.

1.4 Technical data

1.4.1 Dimensions

360 x 400 x 580 mm
(height x width x depth)

1.4.2 Weight

Net: approx. 44 kg

Gross: approx. 61 kg

1.4.3 Operating noise

The noise level is approx. 74dB (A). The value fluctuates greatly depending on the speed, the grinding material and the type of grinding bowl and balls.

The instrument is measured with IP 20.

1.4.4 Voltage

The machine can be operated in two voltage ranges:

- Input value range 100-240 V \pm 10%.

Transient excess voltage permissible as per excess voltage category II.

Caution:

If the instrument is to be operated with two different voltages, such as 115 V or 230 V, it must be disconnected from the mains current at least 60 seconds before switching of the voltage.

1.4.5 Current consumption

The maximum current consumption is approx. 10 A (115 V) or 5 A (230 V).

1.4.6 Power consumption

The maximum power consumption is approx. 1100 W.

1.4.7 Electrical fuses

- Fuse insert at the rear of the machine: 2x10 A T

1.4.8 Material

- Maximum input size for hard material approx. 5 mm
- Maximum input quantity 2 x 35 ml

1.4.9 Final fineness

- Dry grinding
down to $d_{50} < 20 \mu\text{m}$ (depending on material)
- Wet grinding
down to $d_{50} < 0.1 \mu\text{m}$ (depending on material)

2 Operating safety

2.1 General safety instructions

- Read the operating manual carefully before using the planetary micro mill “pulverisette 7” for the first time. Never allow anyone to use the instrument who has not already read and understood the operating manual. To this end, it must be ensured that the operating manual is situated with the instrument, among other measures.
- Only use the planetary micro mill for the purpose described in the operating manual (section 1.3.1).
- Every time before using the planetary micro mill, inspect that the safety equipment is undamaged and clean (see section 2.3, 2.4, 2.5, 4).
- Do not make any changes to the safety equipment, with the exception of the maintenance measures described in the operating manual. The valves on the grinding bowls may not be repaired.
- Do not disable safety devices.
- Only use original accessories and original spare parts. Failure to adhere to this may jeopardise the protection of the machine.
- Do not continue using damaged accessories.
- Do not remove the instruction labels.
- Independent alterations to the device negate the conformity with European directives declared by Fritsch and void the warranty.
- Wear ear protection! As of noise level 90 dB(A)
- Wear protective gloves!
The grinding bowls can be very hot. See section 2.4, 4.8.1, 4.14.10.
- Wear safety glasses!
An overpressure may result from the high temperature or chemical reactions in the grinding bowls during wet grinding. Spraying hazard! Explosion hazard! Always follow the instructions in section 4.1 and 4.8.2.
- Never use force to open the grinding bowls. Only open the grinding bowls if you are certain that the interior pressure has been completely released. Follow the instructions for opening in section 4.9.2 and 4.8.2.
- Do not grind with the machine for several hours at a time without a pause for cooling. Danger of overheating!
- Care must be taken during all work to prevent accidents.
- The maximum workplace concentrations given in the valid safety instructions must be observed and, if necessary, ventilation should be provided or the machine should be operated under a hood.
- The machine should be operated indoors only. The ambient air should not contain any electrically conductive dust particles.





- When grinding oxidisable materials (e.g. metals or coal), there is a risk of spontaneous ignition (dust explosion) if the fine particles exceeds a certain percentage of the material. A chemical reaction can also occur during wet grinding. While such materials are being ground, it is therefore necessary to take special safety precautions (e.g. wet grinding), and the work must be supervised by a specialist.
- The instrument is not explosion-proof and is not suitable for grinding of explosive substances.
- Do not allow the machine to operate unattended. Due to the vibrations, the machine may creep along its supporting surface under certain operating conditions.
- If the planetary micro mill or parts of it are damaged or its functioning does not correspond to the descriptions in the operating manual, the device may not be put into operation. In this case, contact Fritsch GmbH, the representative office that covers your region or dealer who sold you the instrument.
- If you still have questions or problems after reading this operating manual, please contact our technical personnel.

2.2 Operators

- The planetary micro mill may only be operated by authorised persons and may only be maintained and repaired by trained experts.
- No one suffering from medical ailments or under the influence of medications, drugs, alcohol or excess fatigue may operate the planetary micro mill.

2.3 Protective devices

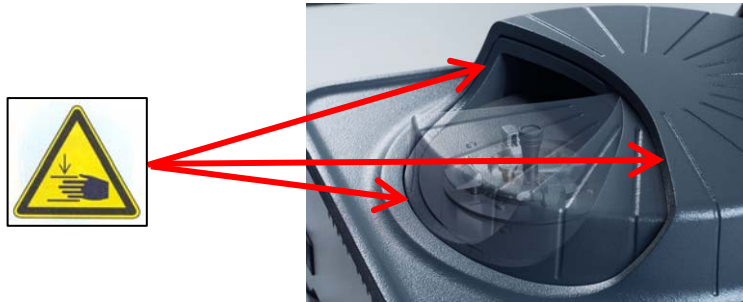
Protective devices should be used for the intended purpose and must not be made unserviceable or removed.

All protective devices must be regularly inspected for completeness and proper function. See section 7 Maintenance.

- The grinding chamber cover of the instrument can only be opened or closed by motor and cannot be operated without a mains power connection. See section 2.5.2.
- It can only be opened once the mill has come to a complete stop.
- The grinding chamber cover must always be closed during grinding.
- The mechanical components of the mill are locked against starting up while the grinding chamber cover is open.

2.4 Danger points

- Danger of crushing when the grinding chamber cover is being opened and closed (the closing force is adjustable, please contact Fritsch GmbH).



- Danger of crushing when adjusting the operating display.



- Danger of crushing when operating the locking lever.



- Danger of burns from the grinding bowl after grinding and during grinding pauses. See section 4.3, 4.8.1, 4.14.10.
- Explosive opening of the grinding bowls due to excess pressure. Never open the bowls with force. Always follow the instructions in sections 4.8.1 and 4.8.2.



2.5 Electrical safety

2.5.1 General

- The mains switch disconnects the machine from the mains supply at both the poles.
- Switch off the mains switch when the planetary micro mill will be out of operation for an extended period (e.g. over night).

2.5.2 Protection against restarting

In event of a mains failure during operation or after switching off with the main switch, the grinding chamber cover will remain locked. When the mains voltage is returned, the grinding chamber will be opened only after recognition by the software that the drive is not in motion. For safety reasons, however, the planetary micro mill will not restart.

2.5.3 Overload protection

- In event of overloading, the speed of the machine is reduced. This special operating state is indicated in the display.
- If the drive motor becomes too warm, the machine switches off.
- If the drive is obstructed, the machine switches off.

2.5.4 Imbalance detection

In event of an excessive imbalance, the machine switches off. See section 4.14.8.

3 Installation

3.1 Transport

- Transport the machine on the transport pallet with a fork lift or lift truck.



3.2 Unpacking and setting up

- Open the binding bands of the packaging.
- Remove the cover of the crate or open the top of the box.
- Remove the accessories box and additional padding.
- Lift the top packaging piece off the transport pallet.
- The machine can now be lifted from the pallet and out of the foam padding.

Caution!

The machine must always be lifted by 2 people. Grip under the edge of the housing to lift the machine.



- Compare the contents of the delivery with your order.

Tip!

Grinding bowls of hardened steel can exhibit surface indentations caused by the manufacturing process. These do not influence the grinding or the grinding result and generally disappear after the first grinding. When present, these surface indentations lie within the permissible manufacturing tolerance ranges. Accordingly, complaints regarding such grinding bowls cannot be accepted.

- Place the planetary micro mill in an indoor room on a level, stable surface.

Caution!

Operation of the planetary micro mill while standing on the transport pallet is not permitted!

- Take care that:
 - ⇒ All switches / operating elements are freely accessible
 - ⇒ The ventilation slots are not covered



Caution!

Covering of the housing openings can result in reduced air supply and thereby overheating of the machine.

⇒ The ports (USB / Ethernet) are freely accessible

- Please store the transport packaging so that it can be reused in case of any return of the instrument. Fritsch GmbH is not responsible for damages resulting from improper packaging (no original packaging).

3.3 Ambient conditions

- Only use indoors.
- The ambient temperature must range from 5 - 40°C.
- Height up to 2000 m above sea level
- Maximum relative humidity 80% for temperatures up to 31°C, linearly decreasing to 50% relative humidity at 40°C.
- Pollution level 2 as per IEC 664.

3.4 Electrical connection

Before making the connection, compare the voltage and current values shown on the type plate with the values of the mains supply used.

Single-phase alternating voltage with protective conductor (see section 1.4).

The connector cable may only be changed by a trained expert.

3.5 Switching on the first time / function test

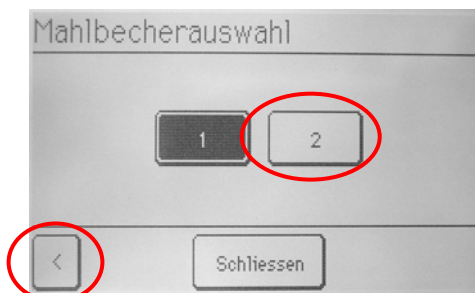
Only switch on the machine once all work described in section 3 Installation has been performed!

3.5.1 Switching on

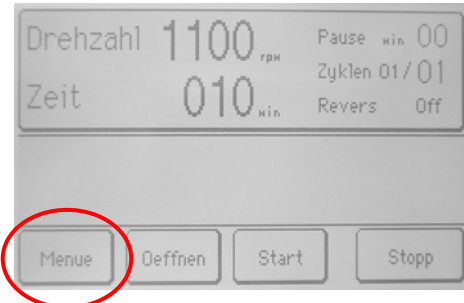
1. Connect the machine to the mains supply using the supplied cable (device cable with IEC 320/C13 plug).
2. Switch on the machine at the main switch.
3. The display lights up.



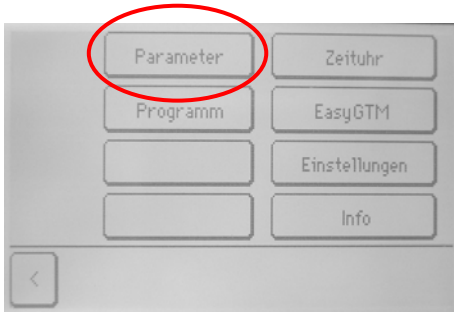
4. Grinding station 1 is moved to the loading position.
5. The grinding chamber cover opens.
6. Check whether grinding station 1 is empty (no bowl inserted).
7. Press BUTTON "2" on the display.



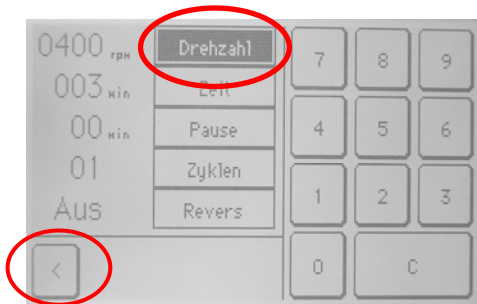
8. Grinding station 2 is moved to the loading position.
9. Check whether grinding station 2 is empty.
10. Confirm and return to the main menu with the "<" button.
11. Press the "Menu" button.



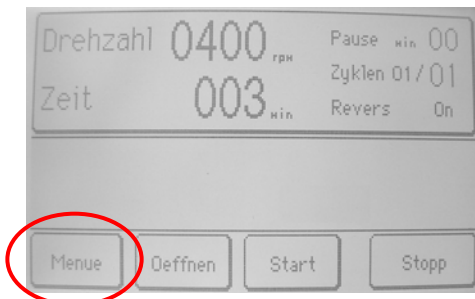
12. In the next menu, you can configure various settings. Select the "Parameters" menu item.



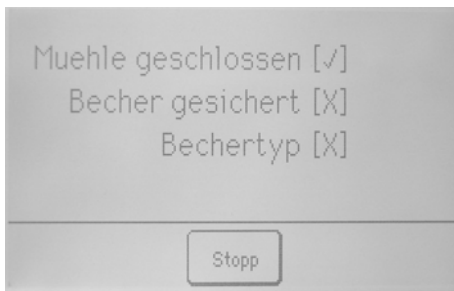
13. A submenu appears in which you can set the rotational speed using the input buttons. Set the speed to 100.



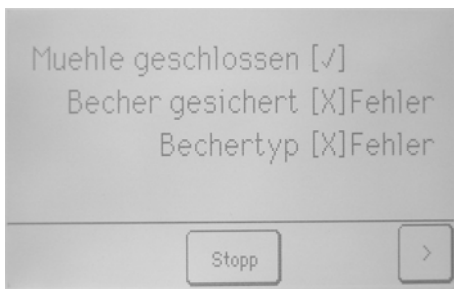
14. Save the entry and return to the main menu with the "<" button.
15. Press the "Start" button to close the grinding chamber cover and start the safety check.



16. Various sensors inspect the bowls and the closing mechanism.
This takes a few moments.



17. The sensors determine that the grinding bowls are not present.



18. Press the ">" button to open the grinding chamber cover and bring grinding station 1 to the loading position.
19. The functions of the machine are OK.

3.5.2 Switching off

If the machine will not be used for an extended period, close the grinding chamber cover with the "Close" button and switch off the machine at the main switch.

4 Working with the planetary micro mill

4.1 Safety Instructions

Danger!

The PULVERISETTE 7 premium line planetary mill from Fritsch allows speeds up to 1100 rpm. Due to the high energy application, very high temperatures and high pressures can be reached within the grinding bowls.

Failure to observe the following safety instructions could result in grinding bowls that explode, sending pieces flying that could cause injuries or damage to buildings and equipment.

We disclaim any responsibility for the consequences and the occurrence of injuries, building damage or equipment damage caused by reactions of the sample material inside the grinding bowl that could neither be anticipated nor accounted for by us.

We expressly state that test grindings with unknown parameters must always take place within a protected room that reliably prevents damage from exploding grinding component.

4.1.1 General

This grinding bowl & lid are tested and approved for static internal pressures up to 20 bars.

For normal use where a slow pressure increase inside the bowls occurs, the safety devices incorporated into the grinding bowl lid prevent pressure rising above the safety limit of 20 bar, thus preventing inadmissible positive pressure and subsequent damage to the milling bowl and the P-7 Ball Mill.

Where fast, dynamic pressure surges (e.g. explosions, very fast chemical reactions, sudden liquid compression etc.) occur, these will not cause damage providing the pressure surge does not exceed 30 bars maximum. However sudden pressure changes greater than 30 bar can lead to failure of the grinding bowl and it's lid, causing subsequent damages to the Ball Mill itself.

In this specific case, the grinding bowls & lids, together with the complete P-7 Ball Milling machine are no longer covered by the standard Fritsch warranty.

During the cleaning of the grinding bowls and grinding bowl parts it is most important to adhere to the operating instructions (section 6.1).

4.1.2 Relieving Excess Pressure in Grinding Bowls

The grinding bowl seals act simultaneously as seals and excess pressure relief elements. In event of a slow pressure rise during grinding, the special seals open at a specific pressure to release the pressure in a controlled fashion.

Important:

The excess pressure relief does not function in event of rapid pressure rises (e.g. explosions).



A semicircular notch with a 2 mm diameter has been punched out of the outside diameter of the gasket. The excess pressure relief of the grinding bowl functions with gaskets of FKM as of a static grinding bowl internal pressure of ~10 bar. Gaskets of silicone open as of a static internal grinding bowl pressure of ~18 bar. The position of the notch on the circumference relative to the grinding bowl wall is does not matter.

Danger:
When gaskets without these defined notches are used, very high pressures arise in the grinding bowl, which can result in bursting of the grinding bowl (injuries and material damage).

Due to the high application of energy during grinding, high temperatures arise in the grinding bowls during grinding. In an 80 ml grinding bowl filled with 60 ml of water, the following pressure and temperature curves result:

Temperature grinding bowl wall [°C]	Pressure in grinding bowl [bar]
101	1
109	2
127	4
141	6
152	8
159	10
166	12
171	14
177	17
180	19
183	21

When using water as an additive, the excess pressure relief with FKM gaskets is tripped at 159°C, with silicone gaskets at 180°C. No effects from the material being ground are taken into account here.

Great caution is required when using solvents as an additive. 60 ml of isopropanol in an 80 ml grinding bowl results in the following pressure-temperature curve:

Temperature grinding bowl wall [°C]	Pressure in grinding bowl [bar]
50	1
73	2
86	5
109	8
124	10.5
132	13
139	14
148	19
149	20

The excess pressure limit for FKM gaskets is reached at 120°C and for silicone gaskets at 149°C.

Here is an example of pressure and temperature curves in a real grinding application:

Grinding balls: 120g 0.4-0.7mm ZrO₂

Grinding bowl: 80 ml ZrO₂

Grinding material: 10 g aluminium oxide with input size d₅₀ = 20 µm

Additive: 30 ml water

Rotation speed: 1100 rpm

Time [min]	Bowl 1 [°C]	Bowl 2 [°C]
10	124	119
15	140	136
20	144	143
25	143	141
30	136	138
40	124	126
60	114	111
128	102	102
180	103	104
240	120	130

With other configurations or grinding material, high temperatures can be reached already at a much earlier point in time.

Tip:

Because gases and solids can mix completely in the grinding bowl, pure gas never escapes; instead it escapes along with grinding material. For instructions on cleaning your mill, please consult section

4.1.3 Grinding Bowl Lid

The grinding bowl lids achieve a gas-tight seal with manual pretensioning of the grinding bowl (see also section 4.8.2). The locking hooks are designed to withstand an internal grinding bowl pressure of up to 40 bar.

Since the excess pressure relief no longer functions in event of rapid pressure increases, we explicitly state that the hook can tear off in event of an explosion.

Important:

An explosion in the grinding bowl can result in severe damage to the instrument.

Risk of fatal injuries:

An explosion in the grinding bowl can result in severe injuries.



As part of our product monitoring, we can relate the following instances of damage that were reported to us:

- 1) During dry grinding of a light metal / resin mixture, a severe grinding bowl explosion occurred after 64 cycles (5 min. grinding + 10 min. pause; 800 rpm) that completely destroyed the mill.
- 2) During wet grinding of Si powder in DEGBE (solvent), a grinding bowl explosion occurred after 11 cycles (10 min. grinding + 30 min. pause) that tore off the locking hook of the grinding bowl lid. The grinding bowl lid destroyed the mill hood, the safety interlock stopped the mill.
- 3) During dry grinding of a Si compound, an explosion resulted during grinding conditions kept secret by the customer and not reported to us. This explosion tore off the locking hook of the grinding bowl lid.

4.2 Selecting grinding bowls and grinding balls

Caution!

When using grinding elements that are not original accessories of the machine, no guarantee is provided and no complaints will be accepted in event of damage to the machine.

The hardness and density (specific gravity) of the grinding bowls and grinding balls used must be greater than that of the materials being ground in order to prevent excessive wear.

Material (Bowl and balls)	Main components of the material	Density* in g/cm ³	Abrasion resistance	Used for grinding material
Agate	(99.9% SiO ₂)	2.65	good	soft to medium-hard samples
Sintered corundum	(99.7% Al ₂ O ₃)	3.7	somewhat good	medium-hard, fibrous samples
Zirconium oxide	(95% ZrO ₂)	5.7	very good	fibrous, abrasive samples
Stainless steel	Bowl: (17-19% Cr + 8-10% Ni) Balls: (12.5-14.5% Cr + 1% Ni)	7.8	somewhat good	medium-hard, brittle samples
Hardened steel	Bowl: (11-12% Cr) Balls: (1.0-1.65% Cr)	7.9	good	hard, brittle samples
Tungsten carbide	(93%TC+6% Co)	14.7	very good	hard, abrasive samples
Silicon nitride	(90% Si ₃ N ₄)	3.1	extremely good	abrasive samples, iron-free grinding

The grinding bowls and grinding balls of zirconium oxide are resistant to acids – except for hydrofluoric acid.

Generally use grinding bowls and grinding balls of the same material.

Exception: Tungsten carbide balls can be combined with grinding bowls of hardened steel.

4.2.1 Grinding bowl capacity

Grinding bowl	20 ml	45 ml	80 ml
Capacity (grinding material)	1-9 ml	3-20 ml	10-30 ml

* High density means high impact energy

4.2.2 Grinding ball size

Type of material to be ground	Appropriate ball diameter
Hard samples with maximum input size of 5 mm	15 mm / 20 mm
Medium input size of 0.5 – 1 mm	15 mm / 10 mm
Fine material 0.1 – 0.5 mm	10 mm / 5 mm
Very fine material < 0.1 mm	3 mm and smaller
Homogenising of dry or liquid samples	10 mm / 5 mm

Caution!

Mixing of balls with different diameters is not recommended. (When using various ball diameters, increased wear on the balls must be expected!)

4.2.3 Number of balls per grinding bowl

A larger number of balls reduces the grinding time, and the grinding results lie within a narrower range of grain sizes.

Ball diameter (mm)	Grinding bowl volume (ml)	20	45	80
20	Number of balls (pcs.)	-	-	5
15	Number of balls (pcs.)	-	7	10
10	Number of balls (pcs.)	10	18	30
5	Number of balls (pcs.)	80	180	250

Ball diameter 3 mm and smaller				
Material	Grinding bowl volume (ml)	20	45	80
Zirconium oxide	Ball quantity (g)	30	70	100
Hardened steel	Ball quantity (g)	40	90	150
Tungsten carbide	Ball quantity (g)	80	200	300

The number of balls must be adhered to in order to avoid unnecessary wear.

4.2.4 Calculated ball weight

Kugeldurchmesser in mm		5	10	15	20
Werkstoff	Dichte in g/cm ³	errechnetes Gewicht einer Kugel in g			
Achat	2,65	0,17	1,39	4,68	11,1
Sinterkorund	3,8	0,25	1,99	6,72	15,92
Zirkonoxid	5,7	0,37	2,98	10,07	23,88
rostfreier Edelstahl	7,8	0,51	4,08	13,78	32,67
gehärteter Stahl	7,9	0,52	4,14	13,96	33,09
Wolframkarbid	14,7	0,96	7,70	25,98	61,58
Siliziumnitrid (Syalon)	3,1	0,20	1,62	5,48	12,99

To calculate the weight of the required balls, the "calculated ball weight" is multiplied by the "number" of balls required.

Example: A 45 ml agate bowl should be filled with 185 agate balls with 5 mm diameter.

Calculation: $0.17 \text{ g} * 185 \text{ balls} = 31.45 \text{ g}$

31.45 g of grinding balls can be weighed out and placed in the grinding bowl in order to save the time it takes to count the balls.

4.3 Effects of the ball size and material during grinding

Large balls of 10 mm – 20 mm diameter result in a high mechanical load on the system. The high mass of the balls in connection with the high accelerations results in a high mechanical load → **impact effect**.

The higher the density of the material used for grinding, the higher the mechanical load.

The highest mechanical load is achieved with balls of tungsten carbide with 20 mm diameter.

When using large agate balls, these are capable of destroying themselves and the grinding bowl.

This is also the case for sintered corundum and silicon nitride balls.

Balls with 5 mm diameter and smaller result in a high thermal load on the system.

Due to the large surface of many small balls, the frictional effects result in a high thermal load. After even 5 minutes of grinding, temperatures exceeding 100°C can be observed on the surface of the lid. You must measure it with a surface temperature sensor. Inside the grinding bowl, the temperature is then many times higher. The adhesive used has a sustained temperature resistance of 200°C. If an external temperature of 100°C is not exceeded, the adhesive will hold.



4.4 Grinding bowl filling quantities

Grinding bowl	Min. sample quantity	Max. sample quantity
20 ml	1 ml	9 ml
45 ml	3 ml	20 ml
80 ml	10 ml	30 ml

Caution!

During wet grinding with large ball diameter > 10 mm, at least half the maximum sample quantity must be used. A suspension with a viscosity that is too low offers no resistance to the balls and can result in damage to the balls and grinding bowl. The result is the same as if no grinding material were placed in the bowl at all. The same also applies to dry grinding with less than the minimum sample quantity.

Caution!

When using less than the minimum filling quantity, increased wear must be expected! This can result in destruction of the grinding parts.

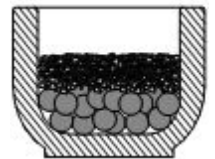
4.5 Filling the grinding bowl

Always observe the following order:

1. Place the grinding balls in the empty bowl.
2. Pour the grinding material onto the balls.

Caution!

Never operate the machine without grinding material because the grinding balls and grinding bowl would be damaged!



4.6 Factors influencing the grinding

4.6.1 Duration (grinding time)

- Longer grinding time increases the share of fine particles.

4.6.2 Speed

- Higher speed reduces the grinding time and increases the share of fine particles.

4.6.3 Reversing operation

(regular reversal of the direction of rotation after every grinding cycle)

- Useful in mechanical alloying.
- Improves the homogeneity of the sample.

4.6.4 Number and size of the balls

- Pre-grind coarse, hard material with large balls → Results in a small share of fine particles.
- A large number of small balls increases the share of small particles with a correspondingly longer grinding time.

4.6.5 Mass of the balls (material type)

- A higher mass (density) of the grinding balls accelerates the grinding. (See the table in section 4.2 Selecting grinding bowls and grinding balls.)

4.7 Dry grinding

Below approx. 20 µm particle size, surface forces dominate and the grinding material starts to "stick".

You can achieve additional dry comminution by adding surface-active substances to the grinding material.

Examples (maximum quantity to be added in % by mass)

- Stearic acid 2-3%;
- Aerosil (highly dilute silicic acid) 0.5-2%;
- Quartz sand ~ 2%;
- Glass powder ~ 2%.

4.8 Wet grinding (grinding in suspension)

In the transition to grinding in suspension, you can ideally add liquids with high boiling point and low vapour pressure.

Caution!

The machine is not explosion-proof. When using flammable liquids, it must be ensured that the heating in the grinding bowl will not reach the boiling point of the solvent. Corresponding cooling phases must be programmed in. If the vapour pressure is too high, escaping vapours may ignite. If possible, we recommend using either non-flammable liquids or liquids with a high boiling point. The boiling point should be over 80°C or, for long grinding times, over 100°C.



4.8.1 Effects of high temperature

Due to high internal temperature, the enlargement of the particle surfaces through comminution and possible chemical reactions in the bowl, very high pressure can arise in the grinding bowls during wet grinding.

This pressure can be so large that the hooks holding the lid to the bowl tear off, causing the lid to "fly off". During grinding tests, chemical reactions were identified between steel parts (hardened steel and stainless steel) and tungsten carbide parts with water as suspension fluid. After a grinding time as short as 20 minutes, a sufficient pressure is produced that locking hooks could be torn off. The grinding bowl temperature may still be relatively low.



Caution!

In event of long grinding times, always include cooling pauses to reduce the internal pressure. For certain grinding materials, it is also necessary to release the pressure after the cooling pause since this can remain within the bowl even despite cooling. This is also particularly true in event of a chemical reaction in the grinding bowl. See section 4.3, 4.8.2, 4.14.10!



The bowl can also stick in the mount if the bowl temperature is too high. In this case, it is necessary to wait until the bowl has cooled enough for it to be released from the mount.

4.8.2 Safety measures against high pressure

When a long wet grinding session is performed without cooling pauses or ventilation during the pauses, very high pressure can arise in the grinding bowl. To prevent damage to the grinding bowls and the device, a small groove has been created on the flat seal around the outer diameter. The pressure can escape at this point if the insert is lifted by the pressure inside the bowl. This takes place at about 20 bar of internal pressure.

Only use original seals!



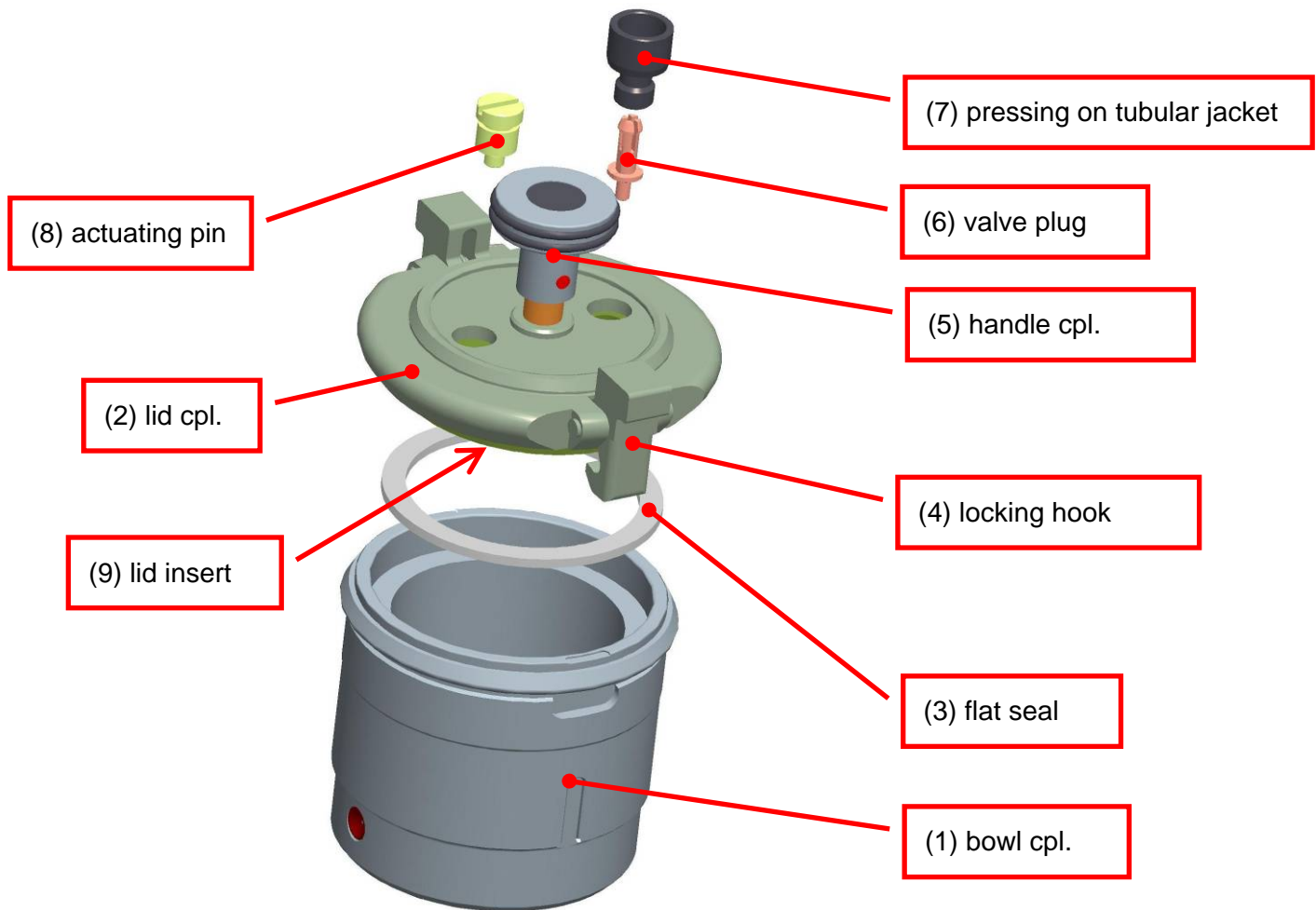
small groove

**Attention!**

Never use force to open bowls.

4.9 Grinding bowl handling

4.9.1 Layout



4.9.2 Opening the grinding bowl

1. Allow the bowl (1) to cool.
2. Release pressure by loosening the pressing on tubular jacket (7).



3. Unscrew the handle (5).



4. If the lid insert (9) sticks to the flat seal (3), pry the lid insert (9) away from the flat seal (3) with a screw driver at the actuating pin (8).



5. Open the lid (2)



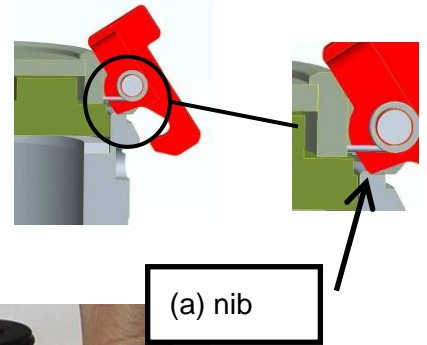
4.9.3 Closing the grinding bowl

1. Insert the flat seal (3) so that the groove is situated in the area of one of the locking hooks (4).



Attention!

2. Press the locking hooks (4) on the upper end entirely inward, and place the lid (2) on the bowl (1). The lid (2) now lies on the small nibs (a) of the locking hooks (4) on the rim of the bowl. Turn the lid (2) on the bowl until the small nibs (a) of the locking hooks (4) snap into the indentations of the bowl rim. Release the locking hooks (4) and press together on the lower end until the locking hooks (4) snap into place and stand perfectly vertically.



3. Screw the handle (5) firmly downward.
The pressing on tubular jacket (7) must be unfixed, so that air can escape from the bowl.



4. Turn pressing on tubular jacket (7) firmly downward



Caution!
After the locking hooks snap into place, the lid may no longer be turned with respect to the bowl. The position of the hooks and the grooves in the bowl beneath the hooks must match.

4.10 Inserting grinding bowls into the grinding bowl mount

Perform the following checks before inserting the grinding bowls:

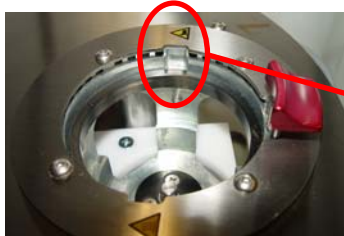
1. Outside of the grinding bowl is clean → Remove any coarse dirt.
2. Inside of the grinding bowl mount is clean → Remove any coarse dirt.
3. Grinding bowl properly sealed (see section 4.9.3)

4.10.1 Inserting grinding bowls

1. Hold the grinding bowl at the handle (5) and insert into it into the grinding bowl mount.



2. Turn the grinding bowl until the grinding bowl noticeably snaps into place and moves several millimetres down into the grinding bowl mount.



3. Press down the locking element on the bowl mount. → The grinding bowl moves farther down in the mounting mechanism.



4. With vertical pressure on the grinding bowl, lock it into the mount. The grinding bowl is properly locked in place if the locking element on the mounting mechanism has moved back up into its original position.



5. As long as the locking element is not in its original position, the rotation of the entire mechanical mill component is blocked.

4.10.2 Removing grinding bowls

1. Push down the locking element on the bowl mount.



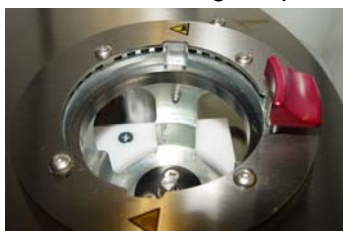
2. The grinding bowl is unlocked and moves several millimetres upward.



3. The grinding bowl can now be removed (upward) from its mount. (The grinding bowl should preferably be lifted with the grip provided for this).



4. Once the grinding bowl has been removed, the locking element returns to its original position.



5. Since a locking element that is not in its original position blocks the mechanical mill components, the loading position can only be changed once the locking element has properly returned to its original position.

After several minutes of grinding and during the cooling phases, inspect the proper condition of the grinding bowls (see section 4.8, 4.9, 4.10)

4.11 Mass balance

Planetary ball mills are subject to a certain amount of imbalance as a result of their method of operation. To keep this imbalance low, it is necessary to balance out all rotating masses in the system as completely as possible.

To ensure optimal mass balance (minimal imbalance) of the machine, identical grinding bowls and identical grinding bowl fillings must always be used at both grinding stations.

The machine can certainly be operated with different loads (such as different grinding bowl fillings); however, this results in performance losses and, if the imbalance is too great, switching off of the machine. The request "Please check the imbalance" appears on the display. See section 4.14.8.

4.12 Grinding time

Depending on the application, the grinding time should be adapted to the heating of the bowls. When grinding at high speeds, a grinding time of 1 hour should not be exceeded; then allow for 0.5 to 1 hour of cooling.

If a chemical reaction takes place in the grinding bowl, a very high pressure can arise in the grinding bowl without the temperature rising above 100°C. In this case, the pressure must also be released after cooling of the grinding bowl.

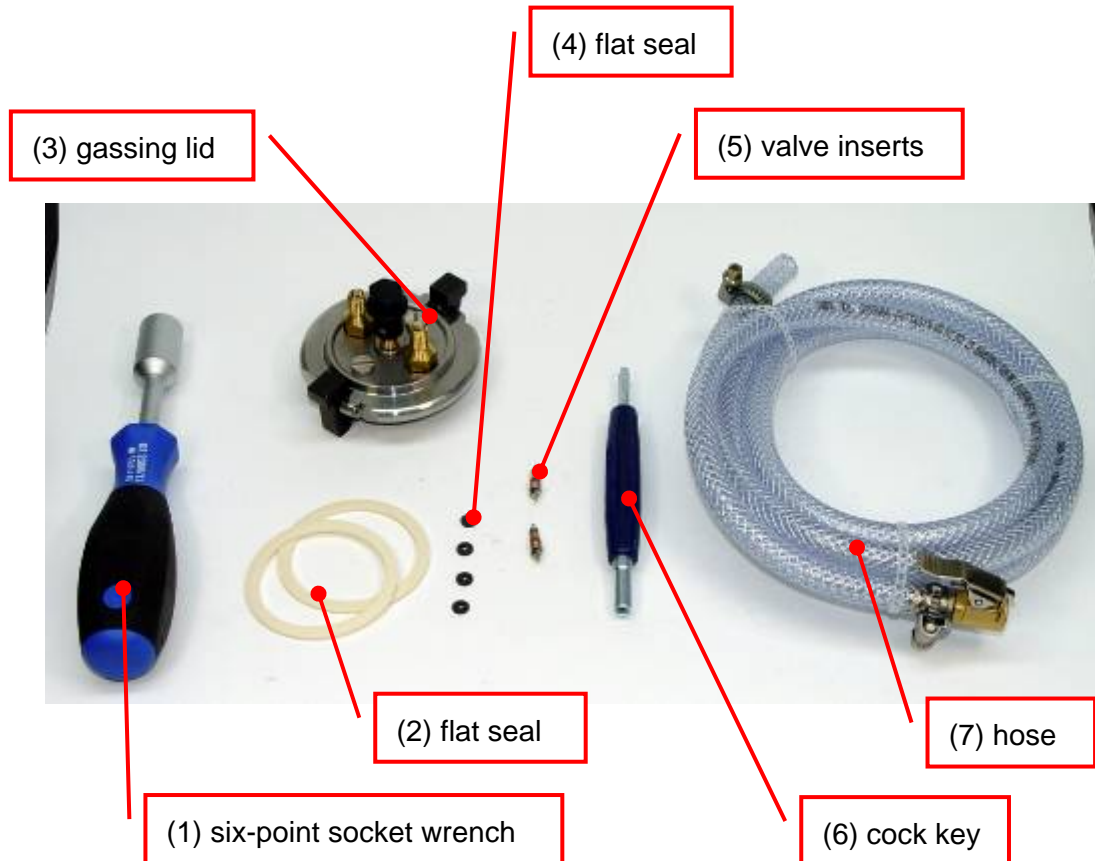
The pulverisette 7 premium line is not suitable for grinding with steel or tungsten carbide grinding bowls with water as suspension fluid. The extremely high energy application results in wear particles at the nano scale. This can lead to uncontrollable chemical reactions.

The extent to which the heating of the grinding material itself must be observed naturally depends on the specific sample being ground. It should be noted that → longer durations should also be accompanied by long pauses for cooling!

If bowls are removed during a grinding pause, the proper seat of the bowls must be inspected before reactivation.

4.13 Gasing Lid

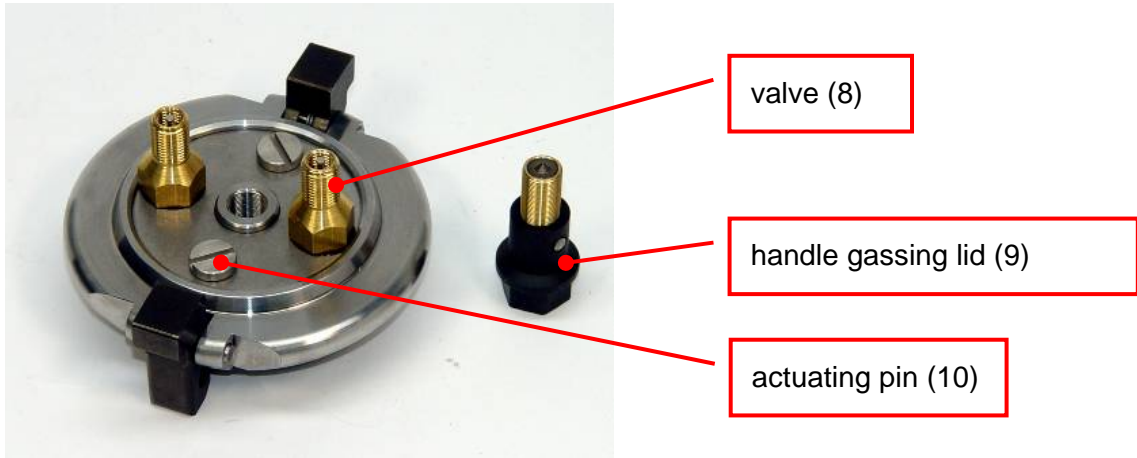
4.13.1 List of all parts from the packing from left to right:



- (1) Six-point socket wrench 13mm
- (2) 2 pieces silicone flat seals, 57,5x48x2mm, for grinding beaker (1 spare part)
- (3) Gassing lid
- (4) 4 pieces viton flat seals for valves, 6,3x1,8x1mm (spare)
- (5) 2 pieces valve inserts (spare)
- (6) Cock key for unscrewing the valve inserts from the valve
- (7) Hose with fitting for valve

4.13.2 Disassembly of the gasing lid step by step

1. Manual unscrewing the central handle (9).



2. Unscrewing the two actuating pins (10) using a broad slot screwdriver (tightening torque approx. 4-5Nm = maximum hand force).



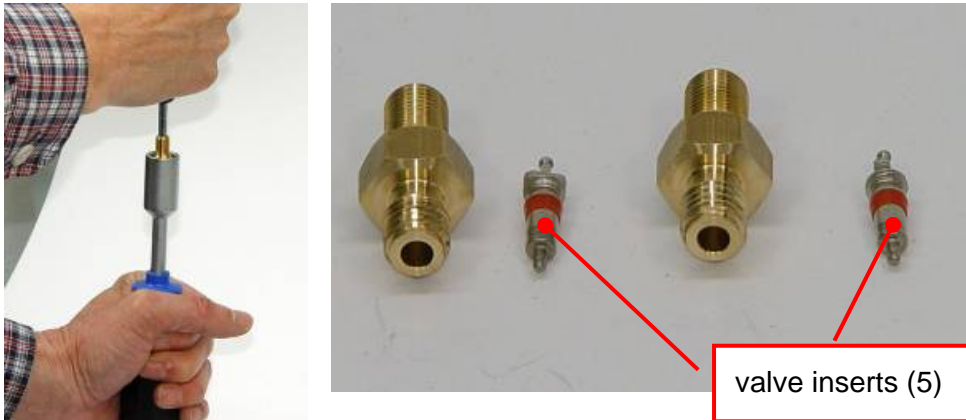
3. Unscrewing the two valves (8) using the six-point socket wrench (1) (tightening torque approx. 3.5-4Nm = maximum hand force).



4. Removal of the flat seals (4) underneath the valves.
The flat seals are deformed by the clamping pressure.

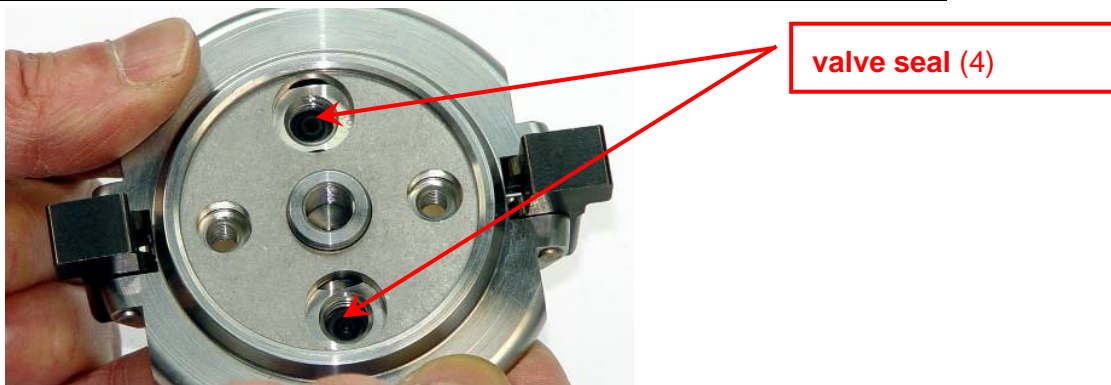


5. Unscrewing the two valve inserts (5) from the valves using the cock key (6)
(tightening torque approx. 1.5-2Nm = medium hand force)
The valve is held by the six-point socket wrench (1), the valve insert (5) is unscrewed using the cock key (6).



6. Assembly of the gasing lid (3) is done in reverse order

Tip:
Do not forget to insert the two valve seals (4).



4.13.3 Mounting the aeration cover onto the grinding beaker

1. Put the silicone flat seal (2) onto the rim of the bowl insert.
2. Touch the lid via the hooks, push the hooks outward and place the lid onto the bowl.
3. The central handle (9) must be unscrewed as shown in the image.



4. Screw the lid with hooks pressed outward onto the bowl until the noses of the hooks snap into place in the indentations on the rim of the bowl mount (see section 4.9.3). After releasing the hooks, these must completely sink into the indentations on the external diameter of the bowl mount and must be in an exact vertical position.



5. Manually screw the central handle (9) downwards until the resistance increases. Tighten with maximum hand force using the six-point socket wrench (1). The maximum hand force equals approx. 3.5-4Nm torque.



6. This is what the closed grinding beaker looks like.



4.13.4 Hose connection for aeration of the grinding beaker

1. Push the locking lever (11) inwards, slide the hose connection onto the valve (8) and press it downwards onto the interior seal. At the same time, the valve is opened by an interior pin in the hose connection. When the locking lever (11) is released, it snaps into place on the valve thread.



2. For rinsing, the second valve can be opened using a pen, e. g. the tip of a ballpoint pen.



Tip:

The gas must be fed very slowly in order to prevent dispersions in the grinding beaker or the swirling up of the grinding stock during filling.

4.14 Performing a grinding

Caution:

The following must be observed during grinding of materials for which no values are available based on experience with the premium line:

Initially, a grinding time of 5 minutes should be set, and after this time, a surface thermometer should be used to measure the temperature on the lid. If this is below 80°C, grinding can continue for another 5 minutes until a temperature of 90°C is reached. If this temperature is reached, insert a cooling pause of approx. 15-30 minutes. At the end of the cooling pause, the knurled screw should be opened to check whether pressure has built up within the bowl. If no high pressure is present, the grinding and pause times can be programmed so that the temperature does not exceed 90°C.

Attention:

During the pause, the mill continues to run at a very low speed for better cooling. (Firmware V1.08)

If a high internal pressure is apparent after cooling due to prolonged blowing noises and the escape of grinding material suspension, great care is required during continued grinding. The pressure must be regularly released after the cooling pauses (at least every 30 minutes so that the suspension can settle) so that it does not become too great. At some point, the build-up of pressure will cease.

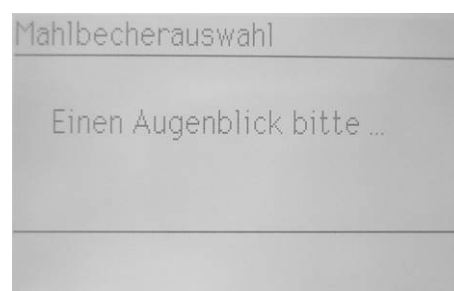


4.14.1 Program sequence after switching on

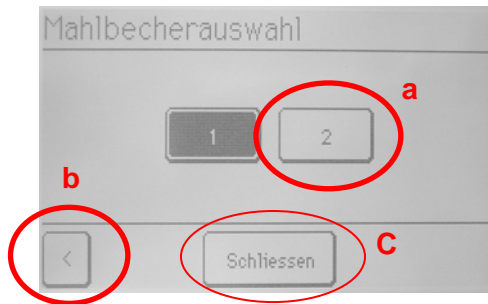
1. Switch on the machine at the main switch.
2. The display lights up.



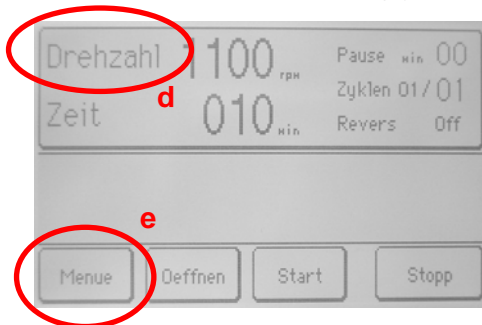
3. During the start and initialisation phase, the INFO button **can** be pressed; an info display appears with the contact address. The display can be exited with the continue button.



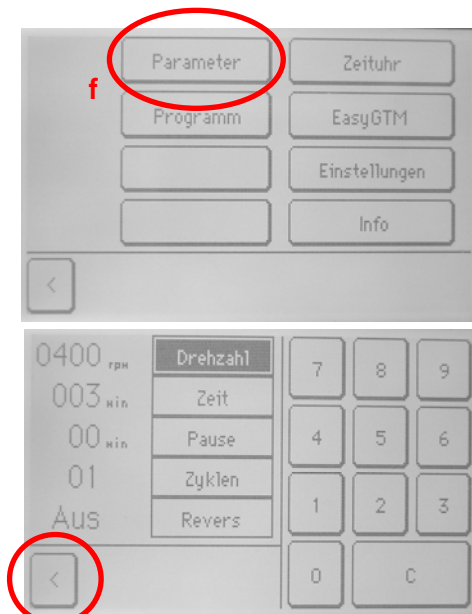
4. Grinding station 1 is moved to the loading position. The grinding chamber cover opens automatically.
5. Place the grinding bowl containing the sample (section 4.5) and properly sealed (section 4.9) into the grinding bowl mount (section 4.10).
6. Bring grinding station 2 to the loading position with the 2 button (a). (With the 1 button, grinding station 1 can be brought back to the loading position).



7. Place the grinding bowl containing the sample (section 4.5) and properly sealed (section 4.9) into the grinding bowl mount (section 4.10).
8. The grinding chamber **can** be closed with the Close button (c).
9. Press the Continue button (b) to enter the main menu.



10. To enter the process parameters, either press the display (d) of the data (button function) or use the MENU button (e) → PARAMETER button (f) to open the parameter entry function.

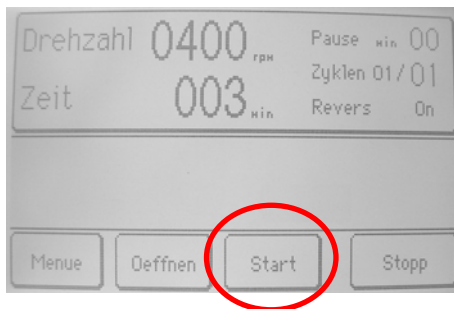


11. The field coloured black is active. When you start entering a number, the previous value is deleted, or you can delete the current parameter with the C button.

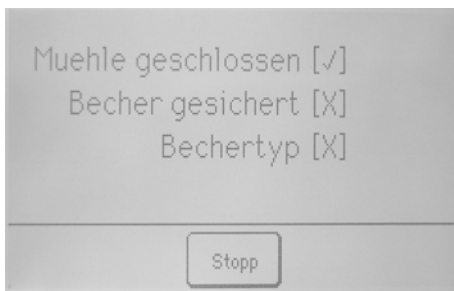
The maximum possible entries are:

- Speed = 1100 rpm,
- Time = 999 min, Pause = 99 min,
- Cycles = 99.

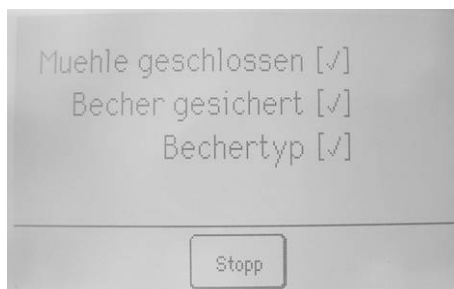
12. After the parameters have been input, return to the main menu with the "<" button.



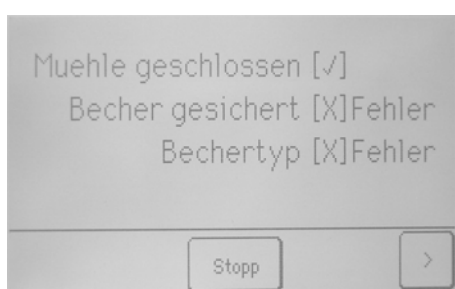
13. Press the Start button → The grinding chamber is closed and the safety check is started.



14. Various sensors inspect the bowls and the closing mechanism of the machine. This takes a few moments. The Stop button interrupts the process.



15. If the inspection is successful, this is confirmed and the grinding process is started immediately afterward.

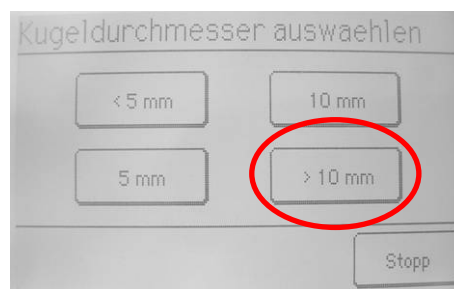
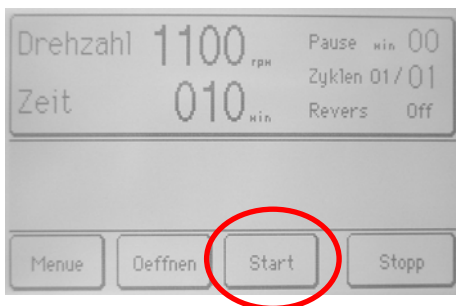


16.If the sensors detect insufficient safety, the start process is interrupted and the cause is displayed. The ">" button opens the grinding chamber and grinding station 1 is moved to the loading position. For measures to correct faults, see section Checklist for fault correction. If the fault is corrected, the grinding can be continued as described under item 9.

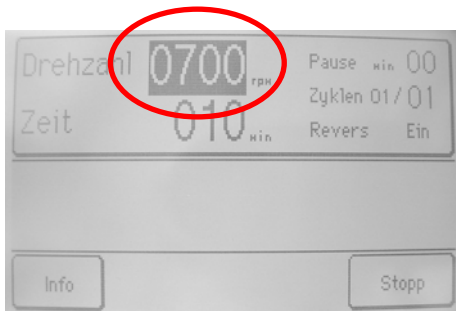
4.14.2 Starting the grinding at high speed

If the speed exceeds 600 rpm, the correct ball size must be selected.

Caution!
The high grinding energy can destroy the balls if they are selected incorrectly!



The maximum speed is reduced based on the selection of the ball size.



A reduced speed is signalled by blinking of the set value for a short time.

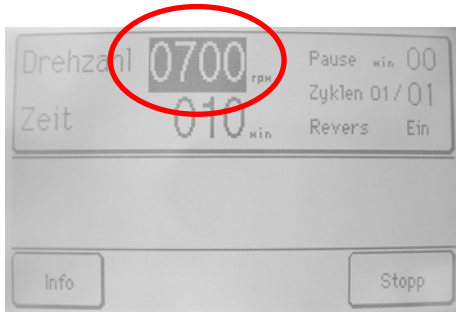
The Stop button ends the grinding process.

The Info button displays some system information as well as the type of grinding elements.

Guide values for speed restriction		
Ball diameter (mm)	Speeds (rpm) for agate	Speeds (rpm) for all other materials
< 5	1100	1100
5	900	1000
10	750	850
> 10	600	700

4.14.3 Overloading

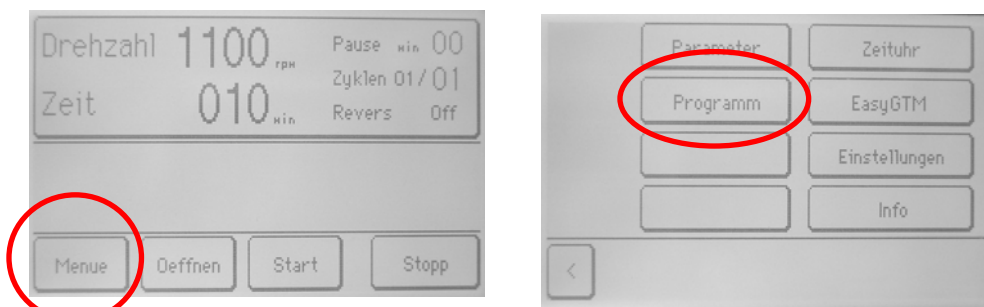
In event of overloading of the planetary micro mill, the speed is reduced. The reduced speed blinks in the display.



4.14.4 "Program" menu item

The grinding sequences can be programmed, saved and loaded here.

1. Press the "Menu" button in the main menu.



2. Then press the "Program" button → A new window is opened.



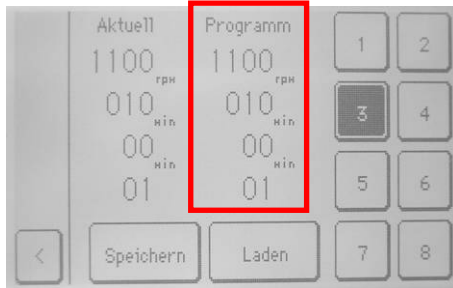
3. The "Current" column shows the current parameters from previous entries.
4. The "Program" column shows the data that was previously saved under the 1 button.

4.14.5 Saving the current data

1. First select the program slot that is open or that should be overwritten.



2. Then save the current parameters in the desired program slot with the Save button.



3. After successful saving, the new parameters are now displayed in the "Program" column.

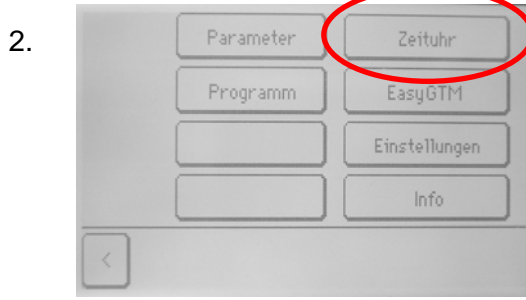
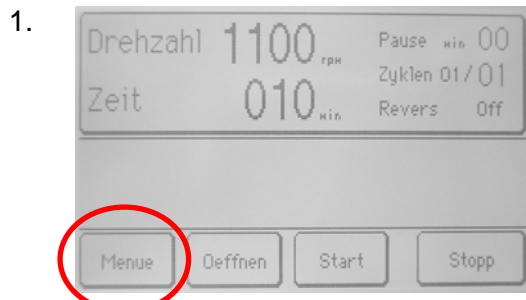
4.14.6 Loading a program

1. Select one of the programs 1...8; the various parameter data are displayed in the Program column.

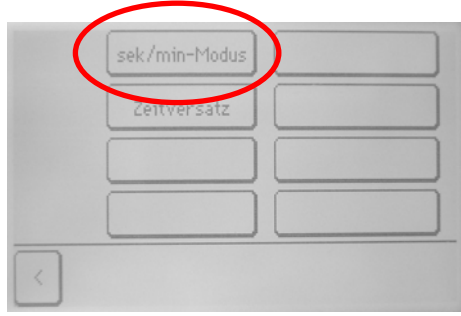


2. Transfer the program data with the "Load" button.
3. Press the "<" button to return to the main menu.

4.14.7 Setting the time control



3. Select time basis for the timer.



- 4. Confirm entry with "<".
- 5. Enter time delay. With entry of a time delay, the grinding process is only started after this amount of time has elapsed. This function is actively displayed in the main menu.



6. Confirm entries with "<".

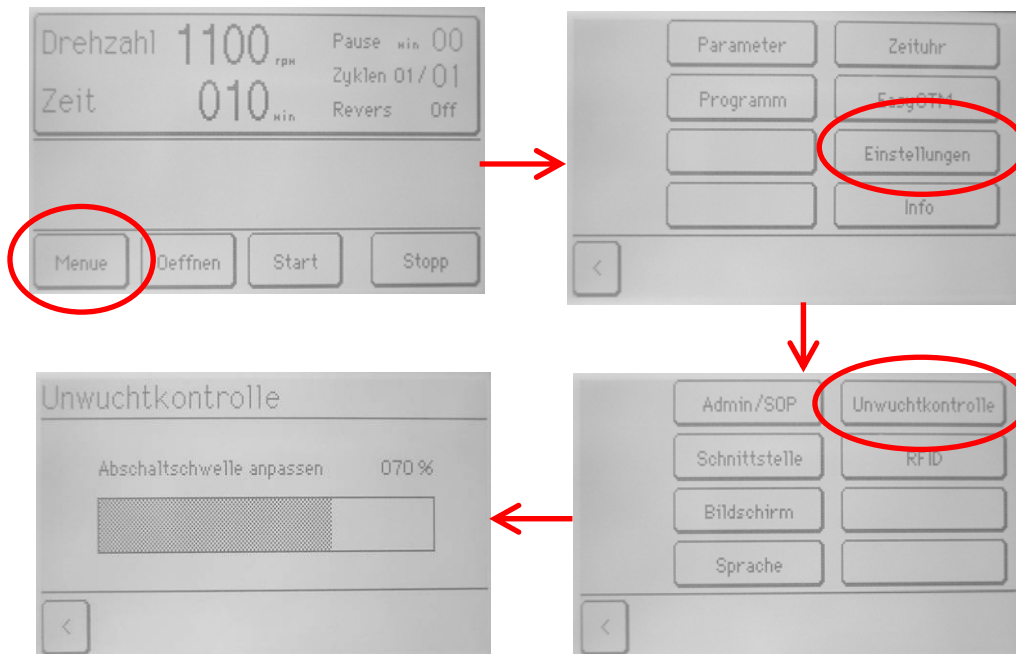


7. The time delay is reset to 0000 after grinding.

4.14.8 Imbalance monitoring

A different weight distribution between the two bowls always leads to different levels of vibrations; this also depends on the speed.

To keep this imbalance within certain limits, the shut-off threshold can be set.



Touch the setting bar or move it back and forth under light pressure.

Caution!

At small setting values, the measurement system is more sensitive; at high values it is less sensitive.

Confirm entries with "<"

4.14.9 Switching off

1. Press "STOP" on the display.
2. When the drive comes to a stop, grinding station 1 is moved to the loading position and the grinding chamber cover opens automatically.
3. If the machine will not be used for an extended period, close the grinding chamber cover and switch off the main switch.

4.14.10 Cooling the grinding bowls

The grinding bowls can be cooled

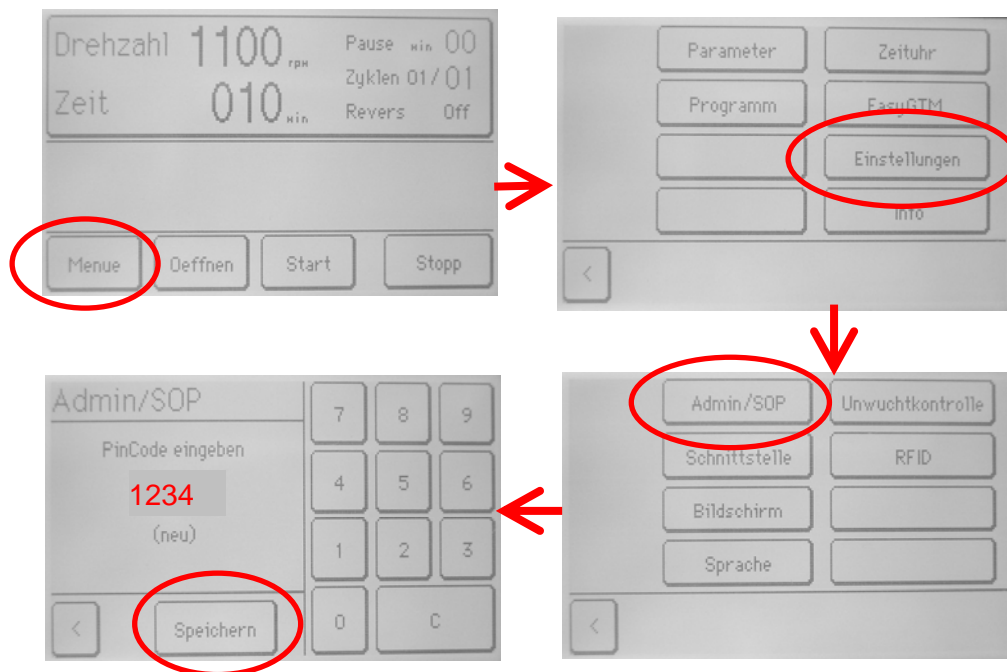
1. with open grinding chamber cover and running fan (fan runs for 1 minute after grinding);
2. or during programmed pause times with closed (locked) grinding chamber cover and running fan.

5 General and optional settings

5.1 SOP mode (Standard Operating Procedure)

It is possible to protect the machine using the SOP mode. If this mode is activated by the administrator, only saved programs can be loaded for operation; changing of the parameters is no longer possible. Parts of the menu structure can no longer be accessed.

5.1.1 Activating the SOP mode



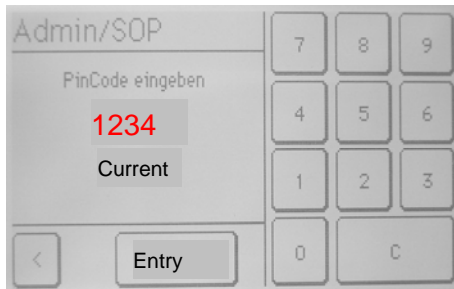
1. Enter a number from 1-9999.
2. Then press "Save".
3. Return to the main menu with the "<" button.
4. The SOP mode is displayed in the main menu.



5.1.2 Disabling the SOP mode

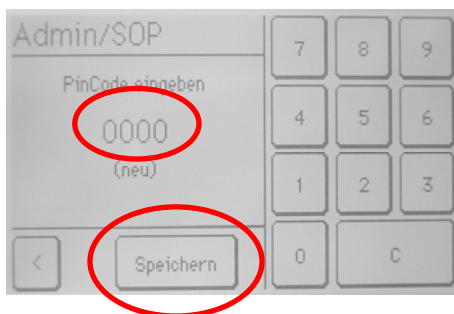
To remove the restrictions on parameter changes, enter the pin code and conclude the entry with the "ENTER" button. Return to the main menu with the "<" button.

If the device is switched off, the SOP mode becomes active again.



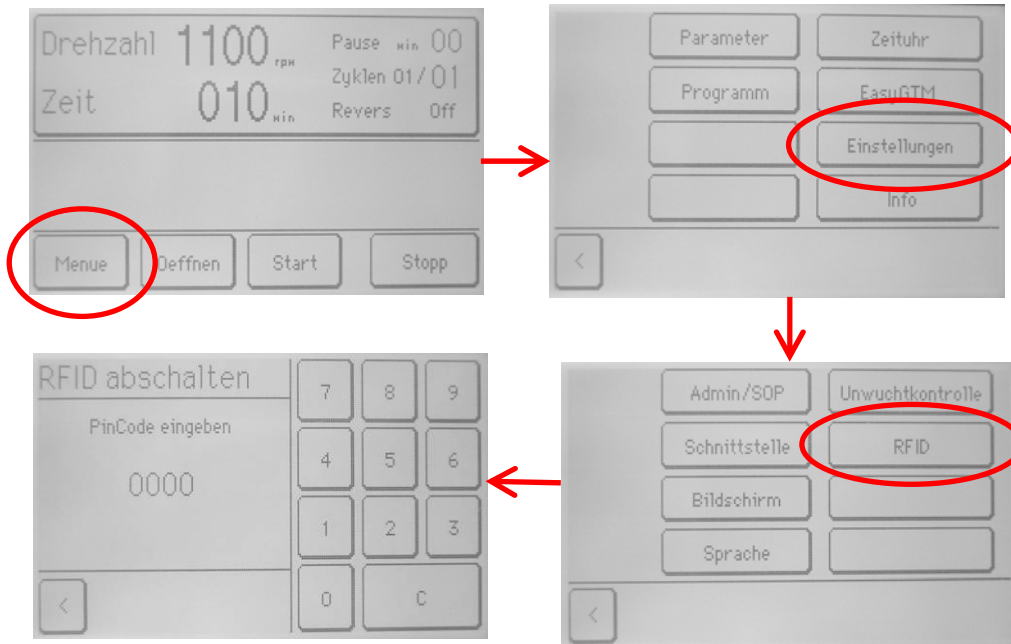
5.1.3 Completely deactivating the SOP mode

1. Disable the mode (section 5.1.2)
2. Enter pin code 0000 and press "Save"
3. Return to the main menu with the "<" button.



5.2 RFID

In the event of damage to a bowl, it is possible to deactivate the bowl recognition.

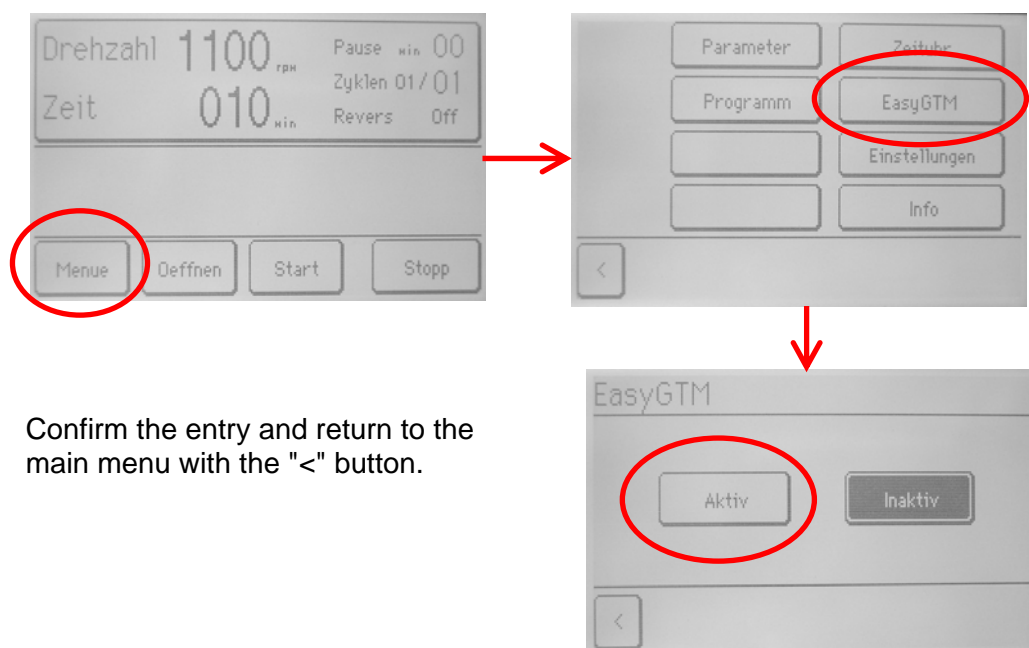


Because the RFID recognition is a safety function that may not be easily deactivated, this is protected by a pin code.

You can only obtain the pin code by contacting Fritsch GmbH.

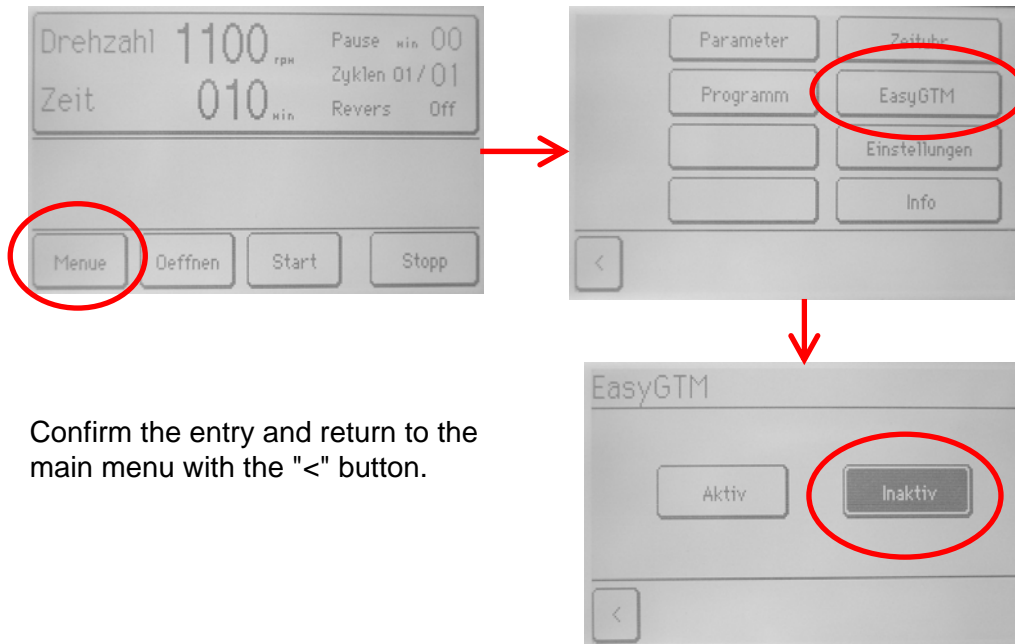
5.3 EasyGTM

5.3.1 Activating

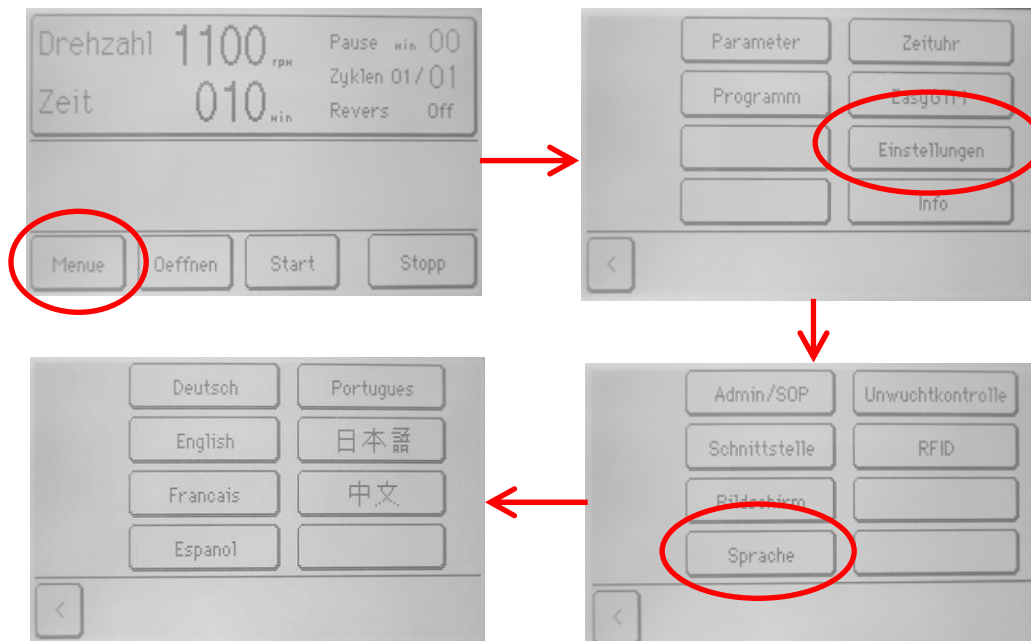


Confirm the entry and return to the main menu with the "<" button.

5.3.2 Deactivating



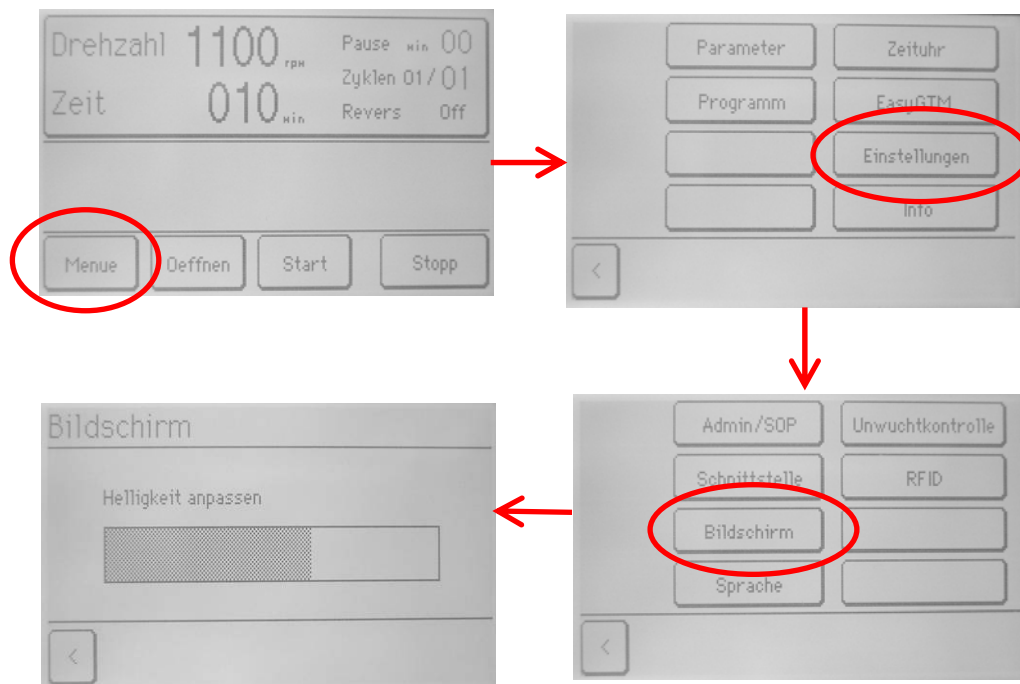
5.4 Language setting



After selecting the national language, all further text displays are shown in the selected language.

Confirm the entry and return to the main menu with the "<" button.

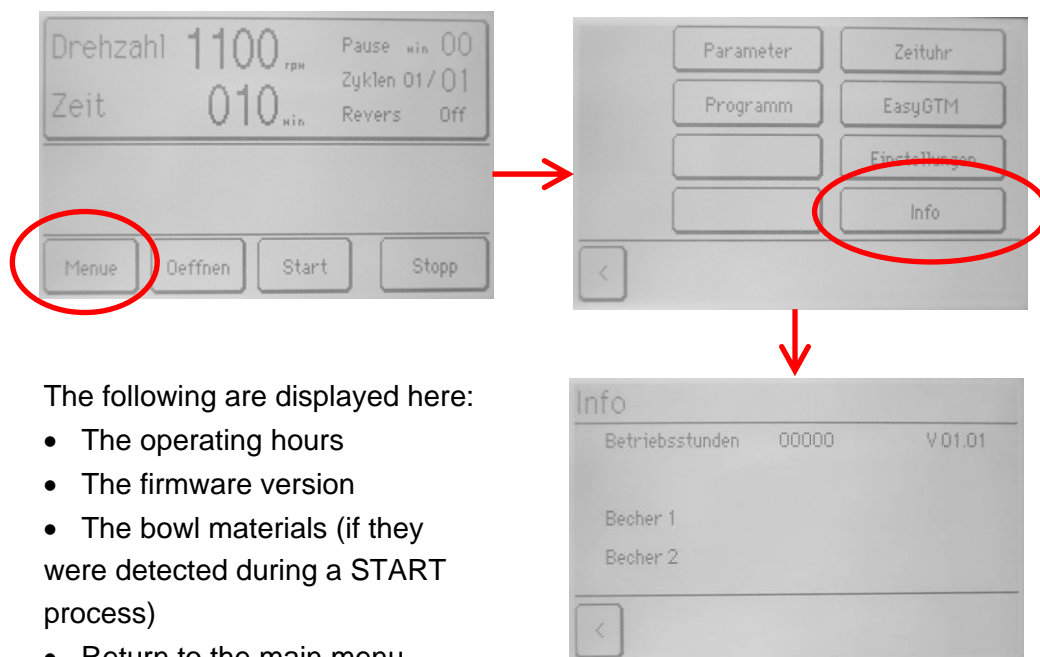
5.5 Screen brightness



The brightness of the screen can be individually adjusted. To do this, touch the setting bar or move it back and forth under light pressure. Save the entry and return to the main menu with the "<" button.

5.6 Information

System information can be accessed with the "INFO" button.



The following are displayed here:

- The operating hours
- The firmware version
- The bowl materials (if they were detected during a START process)
- Return to the main menu with the "<" button.

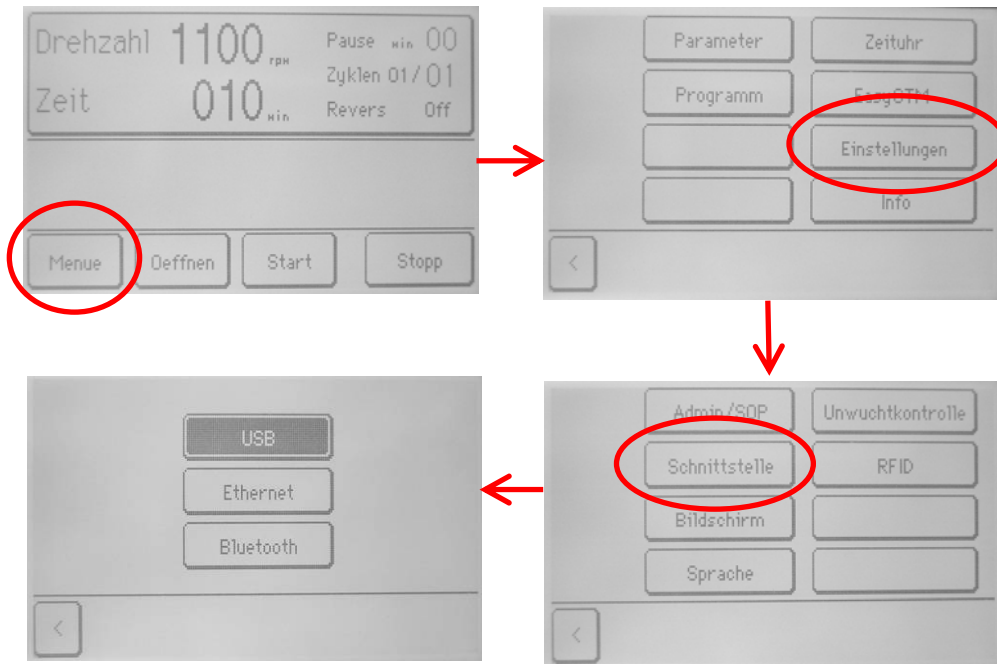
5.7 Interfaces

By default, the machine is equipped with a USB port.

Optionally, an Ethernet port or Bluetooth interface can also be delivered in the future.

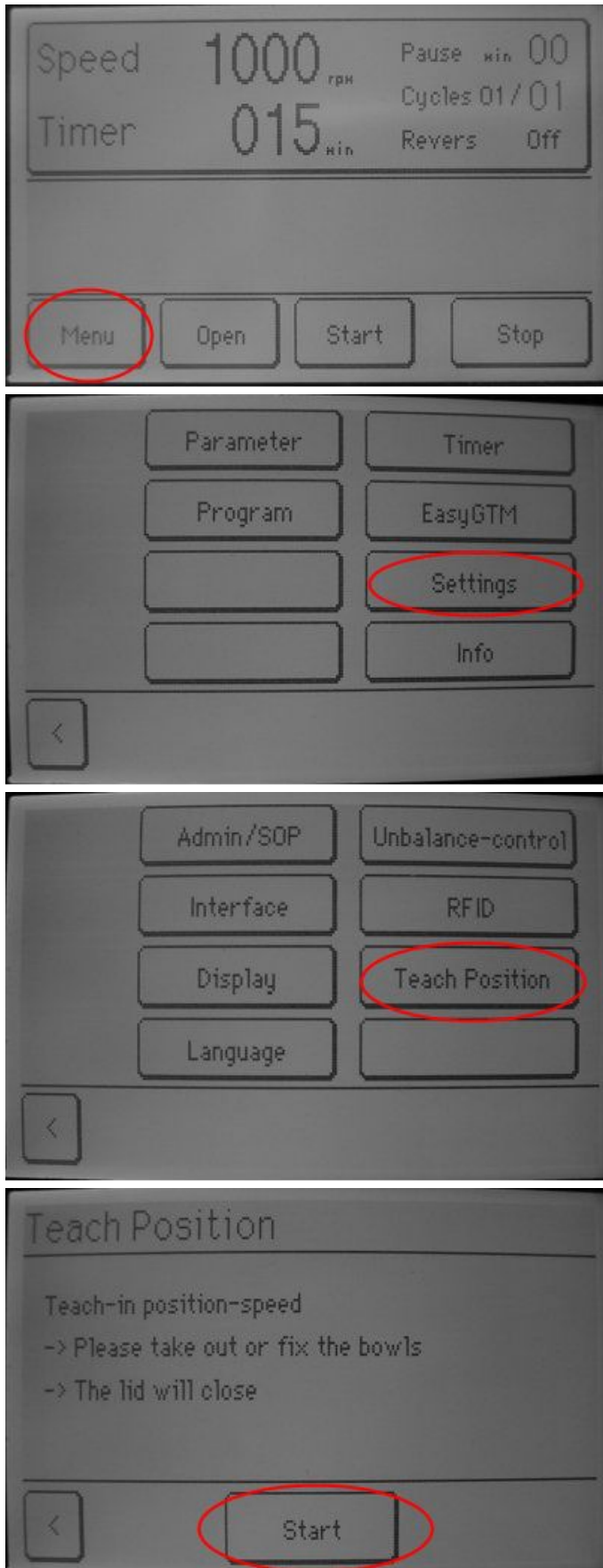
The interface selection must be switched accordingly.

Specific installation instructions are included with the delivery for each of the optional interfaces.



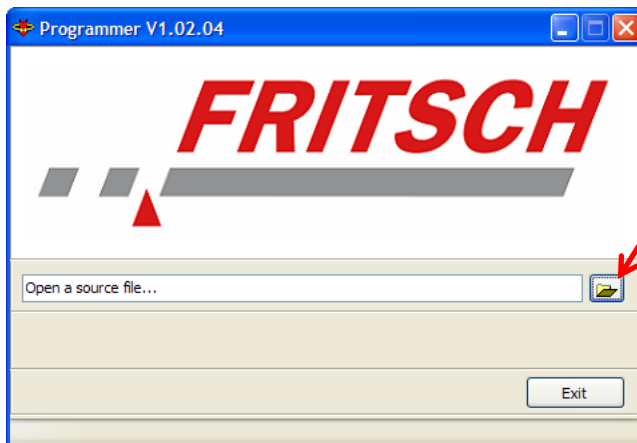
Save the entry and return to the main menu with the "<" button.

5.8 Set Position speed



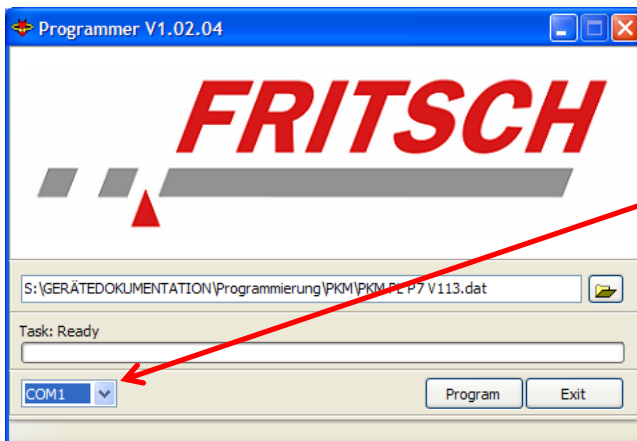
5.9 Firmware Update

1. **Switch OFF** instrument.
2. Install at PC / notebook the drivers for the new USB-device via „PL-2303 Driver Installer.exe“
3. Plug in USB-cable and switch on instrument subsequently. USB port will apply automatically.
4. Start ‚PROGRAMMER.EXE‘.



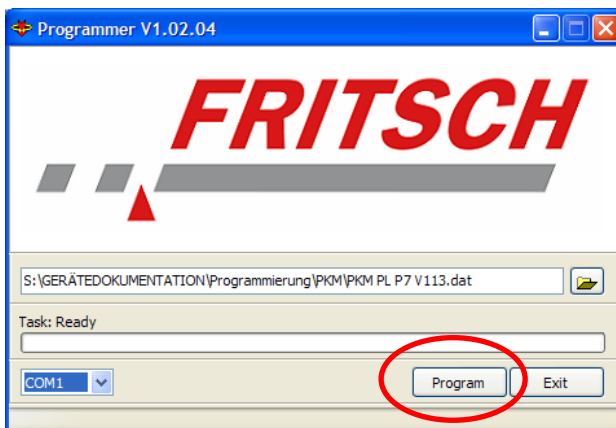
Select here the firmware-file for programming.

5. Select Com-port

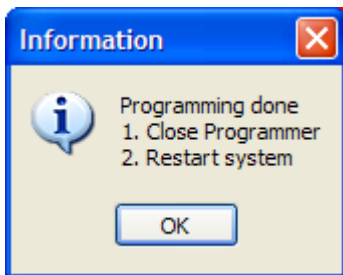


Select here the new installed COM-Port.
(Prolific USB-to-Serial-COMM-Port)

6. Start update with button 'Program'.



7. After programming:



8. Close the Flasher-software.

9. Switch off instrument (Not the personal computer!) for few seconds.

10. Switch on the instrument again → Ready !!

6 Cleaning

6.1 Grinding parts

Clean the grinding bowl and grinding balls after every use: For example, brush clean under running water with typical cleansers.

Fill the grinding bowl about half full with some sand and water and "grind clean" for 2 to 3 minutes in the laboratory planetary mill.

Cleaning in an ultrasound bath is permissible.

During sterilisation in a dry cabinet, only heat the grinding parts up to 120°C.

Important!!!

Do not heat grinding parts of agate, sintered corundum, zirconium oxide and silicon nitride over 110°C, and cool them carefully and slowly.

The grinding parts may not under any circumstances be heated in a microwave (heating too quickly).

They may not under any circumstances be subjected to temperature shocks, otherwise there is a risk that the parts may be destroyed → They break apart explosively.



6.2 Mill

The micro mill can be wiped off with a damp cloth while switched off.

Before the start of cleaning work, pull the power plug and secure the device against accidental reactivation!

The grinding chamber can be cleaned thoroughly with a damp cloth through removal of the grinding chamber cover.

6.2.1 Removal of the grinding chamber cover:

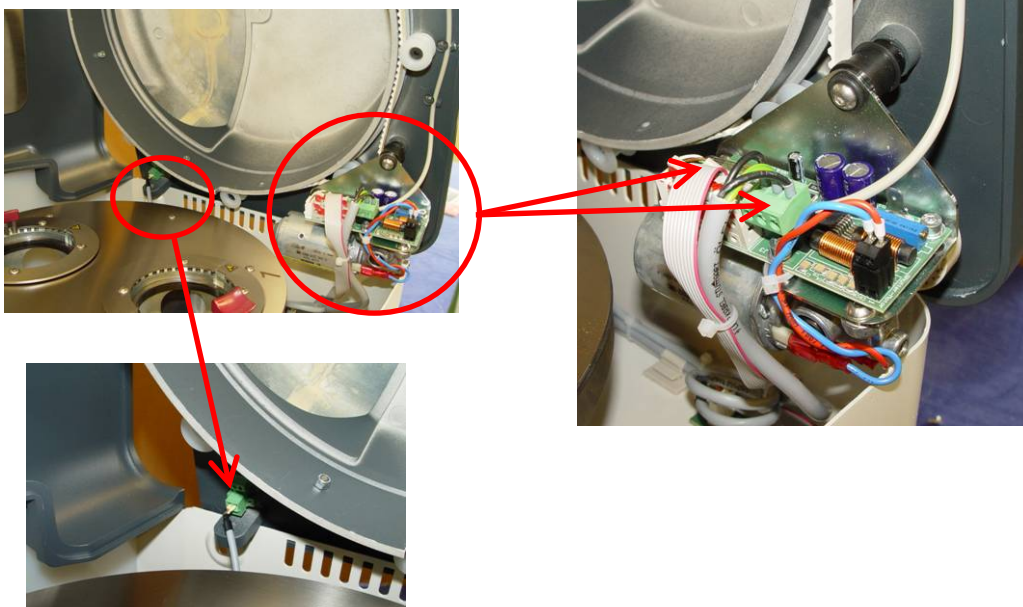
1. Remove the 4 screws on the side of the housing using a screwdriver.



2. Then carefully tip the grinding chamber cover to the right.



3. The 3 connector plugs must now be disconnected.



4. Once this is done, the cover can be lifted up entirely and the grinding chamber can be cleaned.
5. Follow the above steps in reverse to reinstall the grinding chamber cover.

Do not allow any liquids to flow into the device.



7 Maintenance

Before the start of maintenance work, pull the power plug and secure the device against accidental reactivation!

Warn about maintenance work with a sign.

Regular cleaning is the most important part of maintaining the planetary micro mill.

8 Warranty

The warranty card enclosed with the machine upon delivery must be completely filled out and returned to the delivering factory so that the warranty can enter into effect.

Online registration is also possible. More information can be found on your warranty card or on our website <http://www.fritsch.de>

The company Fritsch GmbH in Idar-Oberstein and your "Technical Application Laboratory" or the corresponding national representatives would be happy to provide you with advice and assistance.

Please include the serial number given on the type plate along with any questions.

Please note that the original Fritsch packaging must be used in the event that the machine is returned. Fritsch GmbH is not responsible for damages resulting from improper packaging (non-Fritsch packaging).



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