FRITSCH · VARIO-PLANETARY MILL ·

Operating Manual

Translation of the original

PULVERISETTE 4

FRITSCH Vario-Planetary mill

Fritsch GmbH

Manufacturers of Laboratory Instruments

Industriestrasse 8

D - 55743 Idar-Oberstein

Phone: +49 (0)6784/ 70-0
Fax: +49 (0)6784/ 70-11
E-Mail: info@fritsch.de
Internet: http://www.fritsch.de

Fritsch GmbH, has been certified by the
TÜV-Zertifizierungsgemeinschaft e.V.
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It was verified through an audit that Fritsch GmbH satisfies the requirements of DIN EN ISO 9001:2008.

The enclosed declaration of conformity calls the directives
which the „PULVERISETTE 4“ corresponds to.
This permitts us to mark the instrument with the CE-Sign.

Instrument number 04.1030.00

applies as of serial number 0139

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FRITSCH

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1 General Information / Introduction

1.1 Notes about Operating Instructions

- The copyright to these technical documents is the property of Fritsch GmbH, Manufacturers of Laboratory Instruments.
- These operating instructions are not to be reprinted or copied without the express approval of Fritsch GmbH.
- Please study these instructions carefully before operation.
- All operators must be familiar with the contents of the operating instructions.
- Please observe all notes concerning your safety.
- The mill was designed with the user's safety in mind, however inherent risks cannot be excluded. Follow the advices in these instructions to avoid risks to users.

Attention!

observe operating instructions

- The symbols in the right hand margin highlight the risks described in the text.
- Symbols are also to be found on the instrument warning users of possible risks.
- Warning symbols are surrounded by a triangle.
- These operating instructions do not constitute a complete technical description. They describe only the details required for safe operation and maintenance for usage under normal conditions.

1.2 Explanations of the signs at the instrument and in the operating instructions

Attention! warning against danger spot observe operating instructions	
Attention! Mains voltage	
Attention! risk of explosion	
Attention! hot surface	
Attention! Inflammable substances	
wear protective gloves!	
wear ear protectors!	
wear safety goggles!	
Do not step below lifted load!	

1.3 Short Description of the Machine

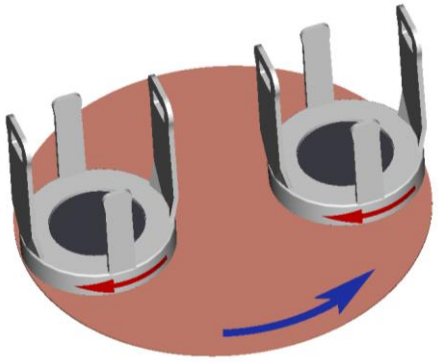
1.3.1 Applications

The "pulverisette 4" vario-planetary mill is capable of emulating ball mills of conventional design, simulating the types of stress entailed and thus reproducing or optimising grinding processes. Due to the high flexibility available for selecting the grinding parameters, it is possible to achieve results unattainable with any other ball mills.

This is the ideal mill for mechanical activation and alloying. The main applications are in the field of materials research and, of course, wherever a powerful, innovative planetary mill is required.

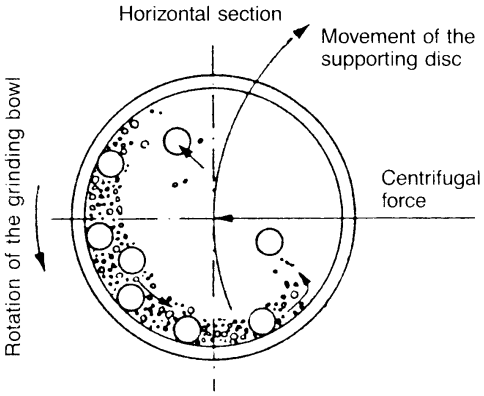
When particles < 10 mm are fed in, a final fineness up to 0.1 µm can be achieved. The useful capacity is between 2 x 5 ml in the case of 12 ml grinding bowls and 2 x 225 ml when 500 ml grinding bowl are used.

1.3.2 Method of Operation



With standard planetary ball mills the grinding bowls are rotating and mounted eccentrically on a rotating support disc. The rotational speed of the supporting disc can be selected at will; the grinding bowl rotates at a fixed transmission ratio.

Due to the overlapping of grinding bowls and supporting disc, the material to be ground and the grinding balls execute movements and trajectories in the grinding bowl whose form and effect depend on the transmission ratio. Comminution is the result of a combination of friction and impact at high energy. Planetary ball mills with a fixed transmission ratio are optimised for a single grinding process only.



But with the "pulverisette 4" vario-planetary mill the rotational speeds of grinding bowls and supporting disc can be adjusted completely independently of each other. By varying the transmission ratio it is possible to control the movements and trajectories of the grinding balls at will so that the balls strike the inner wall of the bowl vertically (high impact energy), approach each other tangentially (high friction) or just roll down the inner wall of the bowl (centrifugal mills).

All intermediate levels and combinations of frictional and impact pressures can be set as required. By changing the transmission ratio it is therefore possible for the first time to carry out mechanical activation as well as mechanical alloying.

Furthermore, it is also possible for the first time to optimally adjust a planetary ball mill to the material to be ground, the size of the grinding bowls and the grinding balls.

1.4 Technical Data

Dimensions and Weight

height:	130 cm
width:	70 cm
depth:	70 cm
weight:	320 kg

Operating Noise

The noise level is about 70dB (A). The value fluctuates greatly depending on the speed and the material being ground and on the type of grinding bowl and grinding balls.

see instruction manual

Voltage

400 V, 3 ~

230 V 3 ~ with specifically transformer (number 04.1800.00)

(See also section 3.7)

Transient overvoltages according to overvoltage category II allowed.

Current input at 400V

15 A

Power consumption

The maximum power consumption is approx. 10000 VA

Electrical Fuses

The fuses are depenting on the mains.

The characteristic must have slow or very slow.

Various fuses for the motor drives, fans and control transformers are located in the side drawer on the lower right.

ATTENTION:
Only trained experts may open and inspect the electrical equipment.

Material

- Maximum feed size approx. 10 mm
- Achievable mean final fineness (depending on material) down to $d_{50} < 1$ µm

Final Fineness

- Dry grinding
  down to $d_{50} < 20$ µm (depending on material)
- Wet grinding
  down to $d_{50} <$ 1 µm (depending on material)

2 Operating Safety

2.1 General Safety Instructions

observe operating instructions!!

- Read the operating instructions carefully before use.
- The instrument can only to be used for the purpose described in Section 1.3.1 Applications.
- Use only original accessories and original spare parts. Failure to do so may call into question the performance of the instrument.
- Do not use damaged accessories.
- The operators must be familiar with the contents of the operating instructions. To this end, for example, the operating instructions must with the instrument.
- Do not remove labels.
- Protective devices must not be made unserviceable or removed.
- Unauthorized modification of the instrument or any part thereof will result in the loss of the conformity to European directives which is asserted by Fritsch and the warranty.

wear protective gloves

- Wear protective gloves!
  Grinding bowls may be very hot after grinding.
- Wear safety glasses
  Wet grinding may cause high pressure in the grinding bowl- Danger of squirting!
- Wear ear protectors
- Don’t run the instrument several hours without cooling phases - Danger of overheating.

wear safety goggles!

- Behaviour at all times must be such as to strictly preclude any accidents.
- Furthermore, the MAC values at place of work specified in the pertinent safety regulations must be adhered to. Where applicable, ventilation must be provided or the instrument must be operated under an exhaust hood.
- When oxidizable materials such as metals, organic materials, wood, coal, plastic, etc. are ground or sieved, the risk of spontaneous ignition (dust explosion) exists whenever the fine particles exceed a specific percentage. 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.

wear ear protectors!

- The instrument is not explosion-proof and is unsuitable to grind or sieve materials which are explosive, combustible or promote combustion.

Attention! risk of explosion

- Do not allow the instrument to run unsupervised. Due to the vibrations, under certain operating conditions, the machine may creep along the surface on which it is located or mounted.
- max. temperature inside bowl 130°C.(Agate max. temperature inside 100°C)

2.2 Operators

- No one other than authorized persons should operate the instrument and it must be serviced and repaired by trained specialists.
- No one suffering from medical problems or under the influence of medications, drugs, alcohol or overtiredness should be permitted to operate the instrument.

2.3 Protective Devices

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

All protective devices should be regularly checked for completeness and to ensure that they are functioning correctly. See section 6 Maintenance.

The hood must be closed when the machine is started up.
The hood is locked:

- when the machine is disconnected from the mains supply
- during operation

The hood can be opened only when the drive of the mill has come to a standstill.

Opening the hood when the machine is disconnected from the mains supply

1. Unscrew cover plate at the upper left side of the housing
2. Insert the attached triangular wrench through the bore, and turn clockwise.
3. The closure hatch can now be opened after the spring-loaded lock at the front of the hood is opened.
4. The instrument can now not be switched on. If it is to be switched on, the safety lock must be activated by turning the triangular wrench anticlockwise, and the hood must be closed.

2.4 Danger Points

- Danger of crushing when the hood is being closed.
- Danger of crushing at the grinding bowl holder.

Attention!

2.5 Electrical Safety

General

- The main switch disconnects the machine from the mains supply at all poles.
- Switch off the main switch if the instrument is to be inoperative for an extended period (e.g. over night).

Protection against Restarting

In the event of a mains failure during operation or after switching-off with the main switch, the lid will remain locked. When the mains voltage is restored, the lock of the lid will open.

For safety reasons, however, the instrument will not start again. At power failure the remaining milling time is stored to the machine. After pressing the START-Button on Control Panel, the milling cycle will be continued, showing a „0“ in the display of the mill. If you restart the process via PC-software or select program 1..9 on control panel, you will lost the remaining time.

Overload Protection (see 8 Troubleshooting Checklist)

In the event of an overload, the device lowers the rotational speed. If the main disk is overloaded, the error code “7” or “8” blinks on the display. If the drive motor overheats, the device shuts down. If the drive is blocked, the device shuts down.

3 Installation

3.1 Unpacking

- Check that the items supplied correspond to your order. Grinding bowls of hardened steel may 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.

3.2 Transport

- Transport the mill on the transport pallet with a fork lift truck or a hand fork lift truck.
- Lift the mill from the transport pallet with a crane.

Do not step below lifted loads!

Procedure:

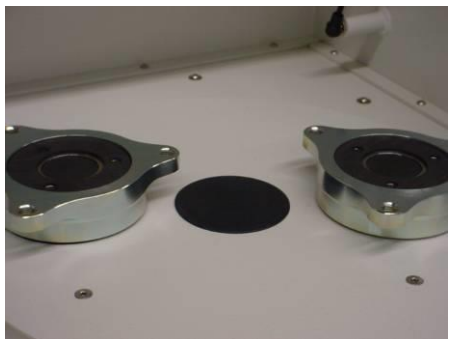
1. Open the hood (see section 2.3 Protective Devices)

2. Hang the crane hook into the eyelet and position the device.

3. Lift and set-up the device carefully.

4. Remove the eyelet screw after the set-up.

5. Fix the cover



3.3 Erection

- Place the instrument on a level, stable surface, indoors.
- Fix the two front wheels by pressing their pedals.

It is inadvisable to operate the instrument while it is standing on the transport pallet.

Do not block the exit of air from the ventilator louver at the side.
There is a danger of overheating if the louver is blocked.

3.4 Ambience conditions

- Use the instrument only inside.
- The air must not contain any electrical conductive dust.
- The ambient temperature must be between 5 and 40°C.
- Height up to 2000m M.S.L.
- Maximum relative humidity of air 80% temperature up to 31°C, linear decreasing down to 50% relative humidity of air at 40°C
- Contamination level 2 (IEC 664)

3.5 Electrical Connection

CAUTION

Electrical Safeguard!

Danger of damages due to short-circuit.

- Ensure that the socket is connected to a power line secured with a circuit breaker.

Before making the connection, compare the voltage and current values show on the nameplate with the values of the mains supply to which the instrument i to be connected.

Caution

Non-observance of the values on the type plate may damage electrical as well as mechanical components.

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

3.6 Connection of the computer

At the face of the p-4 there is a 9 pin DSUB connector located. Connect this by means of the including connecting cable with your computer. Plug the cable to one RS232 of your computer.

3.7 Adaptation to Mains Supply Voltage

The pulverisette 4 was constructed for a main voltage supply of 400 V 3~. Any adaptation to foreign supply voltages must be done using an external transformer (04.1800.00).

Technical Data : - primary 200V 3~ or 230V 3~
- secondary 400V 3~
- power 10000 VA
- Cable 5m without plug

3.8 Switching On for the First Time / Test for Correct Functioning

Switch on the machine only when all the work described in section 3 on Installation has been done.

Switching On

1. Connect the machine to the mains supply (or transformer).
2. Switch on the machine with the main switch .
3. Open the hood.
4. If the grinding bowl holder or any wood are present, take them out. There should be nothing in the grinding bowl holder.
5. Close the hood.
6. **Press button programm until** 1 **is shown in the LED-display** (Upon delivery, only program slot 1 is occupied with a low motor rotational speed.)
7. Press START .
8. The hood will be locked and the mill will run at the **programed** speed.

Switching Off

- Press STOP .
- After a short period (after the mill has come to a standstill), the hood is unlocked and can be opened.

3.9 Rotation Direction Detection

It is necessary to connect the phase sequence of the supply voltage in the correct order.

If the phase sequence is correct, the mill will function correctly.

If it is incorrect, the mill can be operated from the keyboard, but the power supply is not connected internally. Error code 7 or 8 appears on the display (see section 8 Troubleshooting Checklist).

4 Working with the mill

Warning!!!
Before starting the machine, ensure that the grinding bowl is correctly braced and there are no loose parts inside the device. There is a risk that loose grinding bowls or parts will be thrown out. →If this instruction is not observed, no guarantee or claims will be accepted for damages to the device or injuries to persons.

Caution!!!
The grinding tools are subject to normal wear. Prior to each comminution the thickness of the grinding bowl wall is to be inspected. With severe wear the grinding bowl is to be changed. If not, the possibility exists that the grinding ball due to high centrifugal forces which occur during the grinding may strike through the grinding bowl wall, damaging the mill. We don't honour the warranty or accept complaints for instrument damage or personal injuries occurring when disregarding the above information.

NOTE

During milling, high temperatures can build up in the grinding bowl.

In encased grinding bowls, the inserts are glued into the casing with a two-component construction adhesive.

The adhesive is temperature-resistant up to approx. 140°C. Above 140°C, the adhesive becomes liquefied and collects below the insert in the casing. When the adhesive cools down, it solidifies again and presses the casing upward. As a result, the insert could get damaged. This always makes the grinding bowl unusable.

At temperatures above 200°C, the adhesive is ruined. The same applies for encased grinding bowl lids.

4.1 Choice of Grinding Bowls and Grinding Balls

Caution!!!
No warranty or claims shall be accepted in case of damages caused on account of using grinding bowls and grinding balls that are not original accessories of the appliance.

In order to prevent excessive abrasion, the hardness of the grinding bowl used and of the grinding balls must be higher than that of the material used.

Material (bowl and balls)		**density**[1] in g/cm^3	**restistance to abrasion**
Agate	(99.9% SiO_2)	2.65	Good
Sintered corundum	(99.7% Al_2O_3)	3.7	Fairly good
Zirconium dioxide	(95% ZrO_2)	5.7	Very good
Stainless steel		7.8	Fairly good
Tempered steel		7.9	Good
Hardmetal tungsten carbide	(93%WC+6% Co)	14.7	Very good
Silicon nitride (Syalon)	(90% Si_3N_4)	3.1	Extremely good

The grinding bowls and grinding balls of zirconium dioxide are resistant to acids with the exception of hydrofluoric acid.

Normally, grinding bowls and grinding balls of the same material should be chosen.

Exeption: Tungsten carbide balls (<20mm) may be combined (just a few minutes) with steel bowls.

The size of the grinding bowls and grinding balls should be determined experimentally.

4.1.1 Size of the Grinding Balls

Type of feed material	Ball diameter
Hard samples feed particle size <10 mm	30 mm or 40 mm
Medium piece size (<5 mm)	20 mm
Fine material (0.5 mm)	10 mm / 5 mm
Homogenisation of dry or liquid samples	10 mm
Homogenisation of viscous samples	20 mm

This are just clues: the size of the grinding bowls and grinding balls should be determined experimentally if necessary.

Attentione:
Mixing balls with different diameters is not recommended.
(If different ball diameters are used there is the danger of higher abrasion of the balls!)

[1]High density means high impact energy

4.1.2 Number of Balls per Grinding Bowl

larger number of balls reduces the grinding time, and the grinding result will lie within a narrower grain band width.

Ball Ø (mm)	Grinding bowl volume (ml)	12	45	80	250	500
5	Number of balls (piece)	50	180 - 200	250 - 300	1200 - 1300	2000 - 2500
10	Number of balls (piece)	6 - 8	18 - 20	25-30	50 - 150	100 - 250
15	Number of balls (piece)		7	10	45 - 50	70 - 100
20	Number of balls (piece)			5	15 - 20	25 - 35
30	Number of balls (piece)				5 - 6	10
40	Number of balls (piece)					4

These values are just clues: the number of balls should be determined experimentally if necessary.

While using balls with diameter Ø 30 and Ø 40 mm please look after the unit permanently, because the vibrations could cause the machine to move around.

4.1.3 Calculated Ball Weight

Ball diameter in mm		5	10	15	20	30	40
Material	**density in g/cm³**	**Calculated ball weight in g**					
Agate	2,65	0,17	1,39	4,68	11,10	37,46	88,80
Sintered corundum	3,8	0,25	1,99	6,72	15,92	53,72	127,34
Zirconium oxide	5,7	0,37	2,98	10,07	23,88	80,58	191,01
Stainless steel	7,8	0,51	4,08	13,78	32,67	110,27	261,38
Tempered steel	7,9	0,52	4,14	13,96	33,09	111,68	264,73
Hardmetal tungsten carbide	14,7	0,96	7,70	25,98	61,58	207,82	492,60
Silicon nitride (Syalon)	3,1	0,20	1,62	5,48	12,99	43,83	103,88

To calculate the weight of the required balls, the “calculated ball weight” is multiplied by the “number” of balls needed.

Example: A 250 ml agate bowl must be filled with 1221 agate balls of 5 mm diameter.

Calculation: 0.17 g * 1221 balls = 207.57 g

207.57 g of grinding balls can be weighed out and placed in the grinding bowl; this saves the time required for counting out the balls.

4.2 Filling the Grinding Bowl

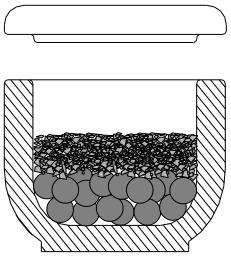
Grinding bowl	Min. filling[2]	max. filling
500ml	80ml	225ml
250ml	30ml	125ml
80ml	1ml	30ml
Grinding bowl	Min. filling	max. filling
45ml	3ml	20ml
12ml	0,5ml	5ml

Using less sample material than recommended will cause higher abrasion!

It is imperative that the following sequence be observed:

1. Place the grinding balls in the empty bowl.
2. Fill the material to be ground on the balls.

Never use the mill without sample material!
Danger of badly damage of the grinding balls and bowl!



filling the grinding bowl

4.3 Influencing Quantities During Grinding

Running time (grinding time)

A longer grinding time will increase the fine fraction. To reduce the grinding time you can use grinding bowls and balls with a higher density and therefore with a higher impact energy.

Speed

A higher speed will reduce the grinding time and increase the fine fraction.

Reversing operation

(regular reversal of the direction of rotation)

- useful for mechanical alloying
- improves homogenizing of the material

Number and size of the balls

Pregrinding coarse, hard material with large balls:
small fine fraction.

Use of many small balls will increase the fine fraction if the running time is increased.

Mass of the balls (type of material)

A higher mass (spec. weight) of the grinding balls will accelerate the grinding.
(see 4.1 Choice of Grinding Bowls and Grinding Balls)

[2] Filling = material to be ground

4.3.1 Dry Grinding

Below a particle size of approx. 20 µm, surface forces predominate and the material will start to "stick".

Further dry grinding can be achieved if surface-active substances are added to the material.

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

- Stearic acid 2-3%
- Aerosil (microdispersed silicic acid) 0.5-2%
- Silica sand ~ 2%
- Glass powder ~ 2%

Attention! inflammable substances

4.3.2 Wet Grinding (Grinding in Suspension)

When grinding in suspension, you can add auxiliary substances in liquid form with a high boiling point and low vapour pressure.

Flammable liquids with a boiling point <100°C should not be used.

4.4 Clamping and unclamping the grinding bowls

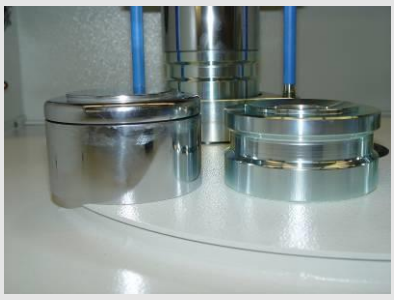
The following tests should be performed before the grinding bowls are clamped in the machine before each grinding operation:

- Check the rubber disc in the bowl holder for damage.
  Replace rubber discs that are flattened (rough side up!).
- The flat Teflon seal (for sealing between the lid and the bowl) must not be damaged or dirty.
  Replace severely deformed flat Teflon seals.
- The surfaces of the lid and of the bowl on which the flat Teflon seal rests must be clean.

4.4.1 Clamping 500ml, 250ml and 80ml grinding bowl

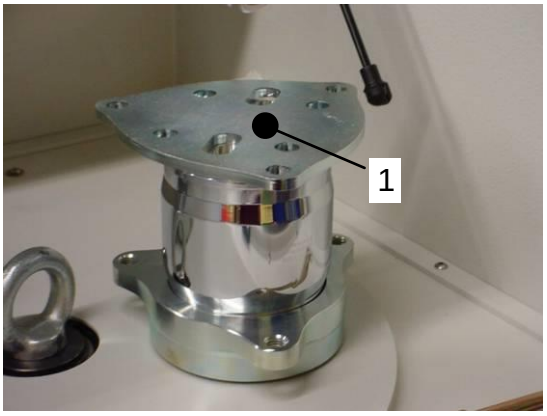
Attentione!!!

- **Either use the 80 ml with the reducing piece fitted (order no. 90.1120.09) or**

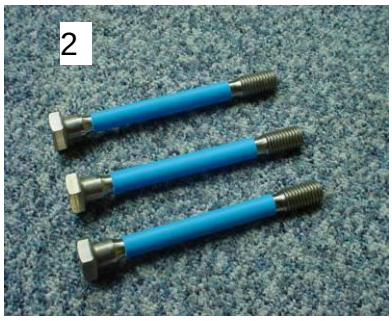


- **use two 80 ml grinding bowls, one on top of the other.**

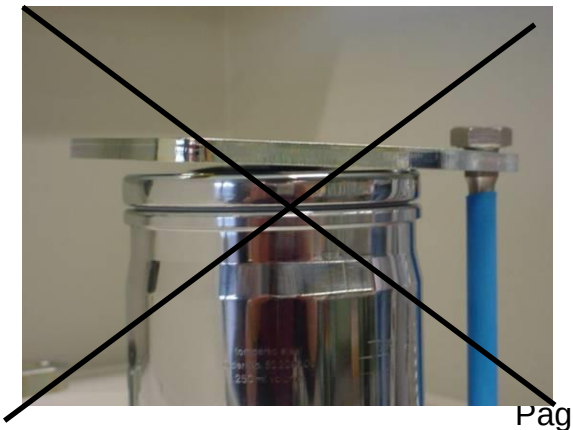
1. Put the grinding bowl into the holding device

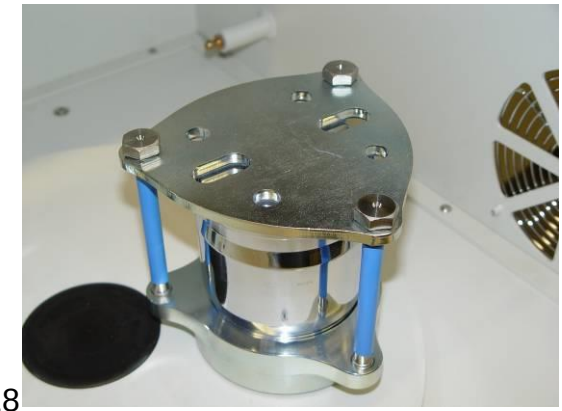


2. Then put the clamping plate 1 on the lid of the grinding bowl.



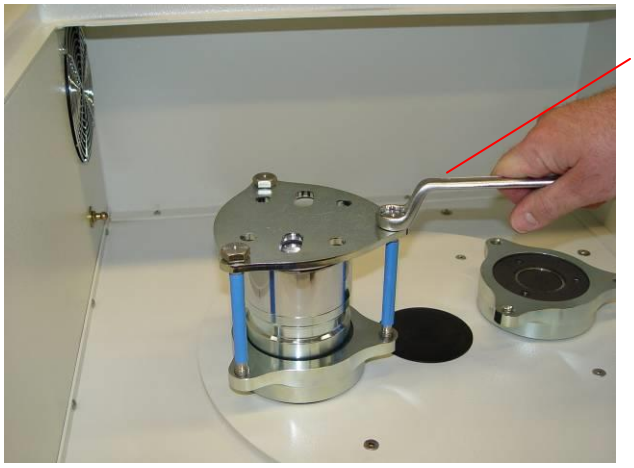
3. Insert the delivered blue hexagon head cap screws (2) in the corresponding bores.



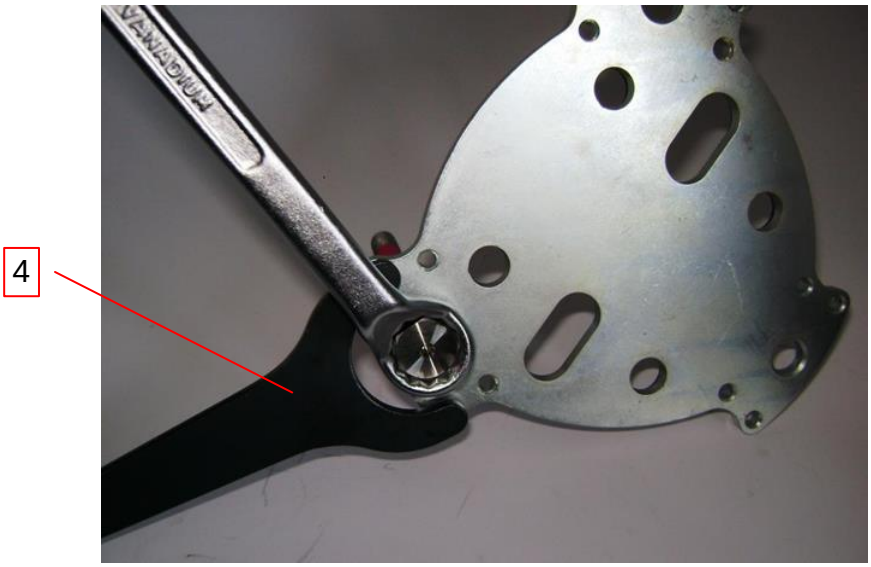


Caution!!
Hexagon screw with:
blue shaft for the standard bowl with standard lid and standard bowl with de-gassing lid.
red shaft for the GTM system, additional clamping system and combination of degassing lid with additional clamping system .

4. Turn the 3 hexagon screws **evenly** by hand until all the screw heads lie on the clamping plate.



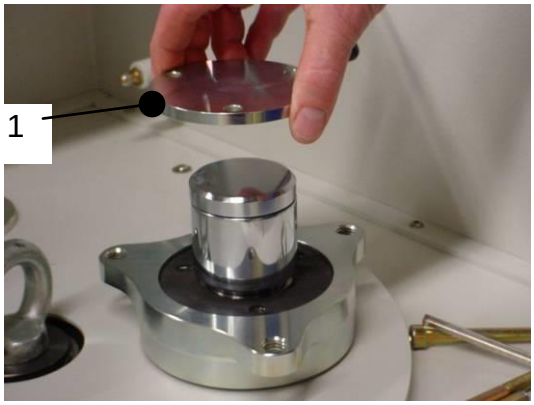
5. Then tighten the 3 hexagon screws **evenly** with the two-hole face pin wrench (4) and the spanner (3)**.**



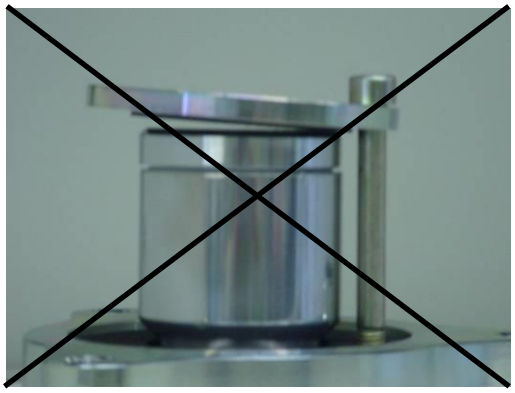
- **Attention!!! Do not twist screw heads!**
- **Caution!!!**
  If the clamping plate is not clamped tightly enough, the sample can push out of the grinding bowl or in the worst scenarios, the grinding bowl may come off the bracing and this may damage the appliance. Fritsch GmbH shall not assume liability for improper handling.
- **After a few minutes of grinding, and in the cooling phases, check that the clamping is secure.**

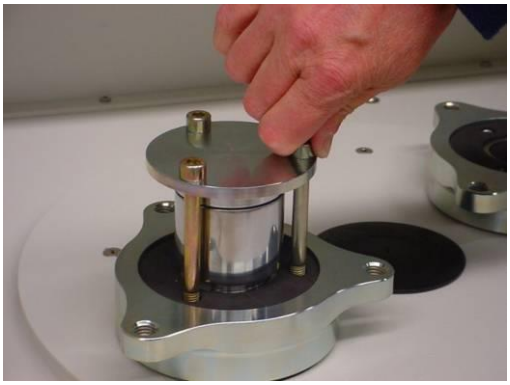
4.4.2 Clamping 45ml and 25ml grinding bowl

1. Put the grinding bowl into the holding device.

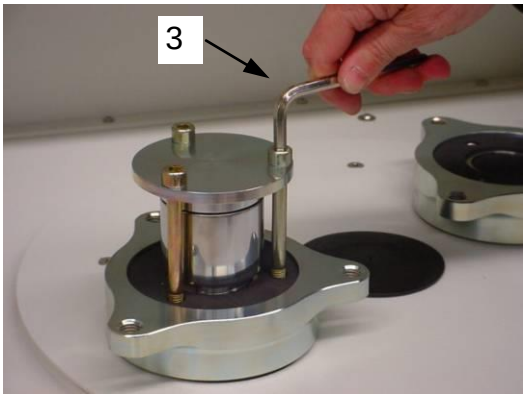


2. Then put the clamping plate (1) on the lid of the grinding bowl.





3. Insert the delivered allan screws (2) in the corresponding bores.
4. Turn the 3 socket screws **evenly** by hand until all the screw heads lie on the clamping plate.



5. Then tighten the 3 socket screws **evenly** with the socket screw wrench (3).

- **Attention!!! Do not twist screw heads!**
- **Caution!!!**
  If the clamping plate is not clamped tightly enough, the sample can push out of the grinding bowl or in the worst scenarios, the grinding bowl may come off the bracing and this may damage the appliance. Fritsch GmbH shall not assume liability for improper handling.
- **After a few minutes of grinding, and in the cooling phases, check that the clamping is secure.**

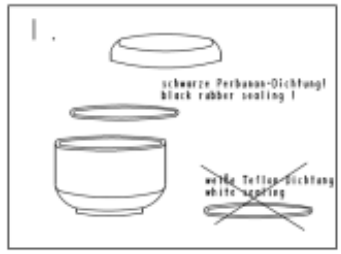
4.4.3 Unclamping

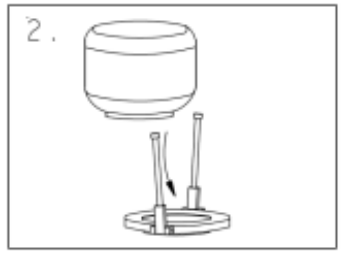
The unclamping is made in reverse order.

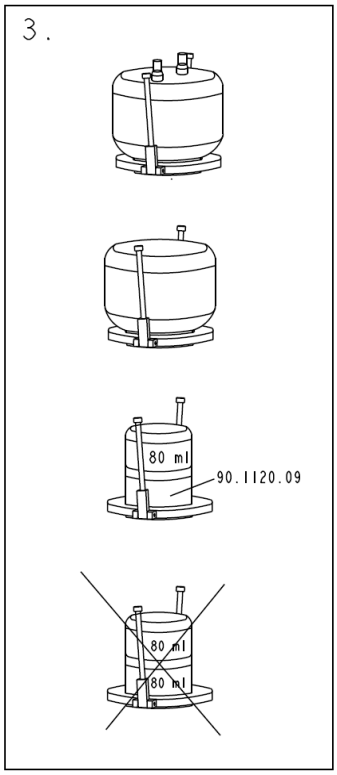
4.4.4 Clamping with Additional lock-system

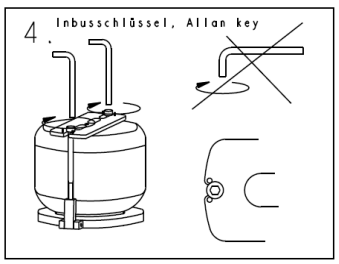
Grinding bowls can be filled and then closed with the help of the Additional Clamping System in a box with protective gas, without beeing clamped in the mill.

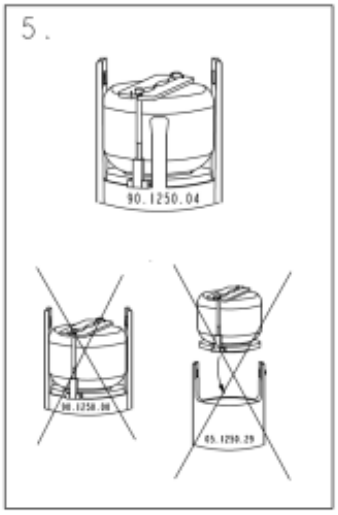
It's also possible to clamp the gas lids with the Additional Clamping System. So they can be filled with protective gas out of the mill.

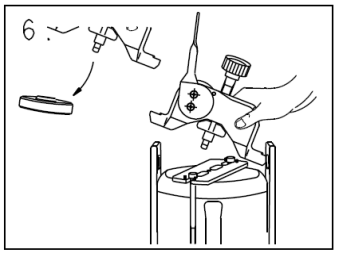


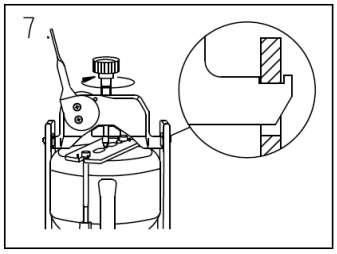


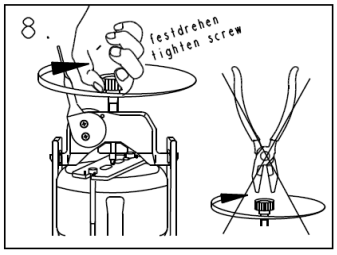


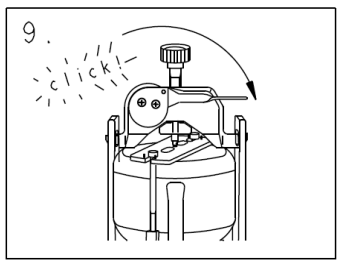


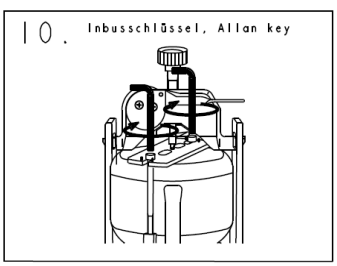








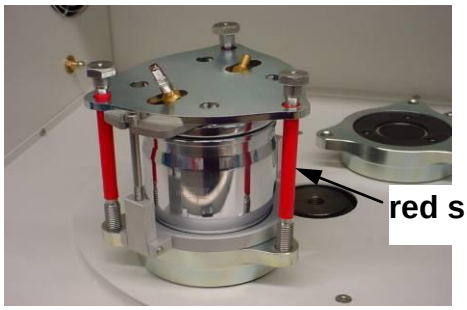


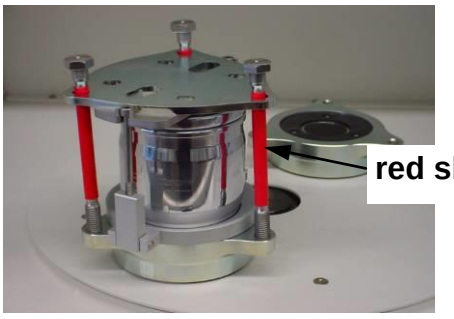












The clamping and unclamping takes place as described in section 4.4 Clamping and unclamping the grinding bowls. Hexagon screws with **red** shaft are nevertheless used

Attention!!
After tightening the clamping plate, check whether the socket screws of the additional clamping system is firmly fixed and if required, tighten up the socket screw.

4.5 Mass Balance

Mill symmetrical charging!

For weight balancing, always clamp a grinding bowl of the same weight with a cover and sealing ring in the opposite pot mounting.

CAUTION

Additional weights like „GTM“ and additional clamping systems must also be balanced.

4.6 Grinding Time

In accordance with the application, the grinding time should be adapted to the heating of the bowl. The temperature inside the bowl is about 20-30°C higher than the outside of the grinding bowl.

The max. temperature at the outside walls of the bowl is about 100-110°C (Agate max. 70-80°C). The grinding time is determined by this temperature. The grinding time wich does not exceed this temperature depends on the sample material, the balls und the speeds. For this reason the grinding time has to be determined experimentally by the user.

clue

In the case of grinding at high speeds and with large bowls, the grinding time should not exceed 1 hour (temperature-dependent). Then allow to cool for 0.5 to 1 hour.

- Pay attention to the heating of the material;
- In the case of extended running times, if necessary set a break time for cooling.
- Before switching on again after a cooling phase, check that the clamping is secure.

To reduce the grinding time you can use grinding bowls and balls with a higher density and therefore with a higher impact energy.

In the case of operation for mixing and homogenisation at low speeds, the mill may run for several hours without harm being done.

The machine cannot be operated with an external timer

4.7 control panel

- Switch on the main switch on the right side of the device
- The LED display on the control panel will come on.

4.7.1 Choose Program

Push button **program** to choose program 1 to 9.

Defined rotational speeds of main disk and planet disks, milling times, pause times, repetitions and reversing actions are stored to defined program places one to nine. Directly from the mill these programs only can be loaded and executed. Changing the programs needs a connection to a computer running the p-4 control program.

4.7.2 Start of Mill

- After everything has been set up as described in section 4 on Working with the mill, close the hood.
- Press the START button on the control panel. The hood will be locked and the mill will run.

Only the rotational speeds stored to this program place are executed.

4.7.3 Stop Milling

When the stop button **O** is pressed, the mill brakes the main disk and the planets until they stop. This process may take several seconds. Also see the section 4.8 Performing a Grinding Operation.

4.7.4 LED-Display

Display 0

After a power failure during milling the remaining milling time is stored to program place 0 (max. inaccuracy: one hour). After the mill is ready to continue, a remaining time is shown with „0“.

Display 1-9

These are the program places one to nine.

Display 5-9 blinking

Look for section 8 General Information / Introduction.

4.8 Performing a Grinding Operation

- After everything has been set up as described in section 4 on Working with the mill, close the hood.
- Press the START button on the control panel.
- The hood will be locked and the mill will run.
- The mill will rotate at the speed set (set speed) – if the load is too great, e.g. if the grinding bowl is too large, the machine will be run at a lower speed (actual speed) so as to prevent overloading.

If the mill does not start, see section 8 Troubleshooting Checklist.

Overloading

When the mill is overloaded, the rotational speed is reduced

If the overload continues, the mill switches off, see section 8 Troubleshooting Checklist.

Miscellaneous

- During operation, the hood remains locked even during pauses and the fan cools the interior.

Interruption of a grinding

- After a program is selected and the start button on the mill is pressed, an internal timer starts to run. This makes it possible to check the mill progress by pressing the stop button on the device, checking the grinding and pressing the start button on the device again to continue the grinding without losing any time. If you stop the mill during the pause, the timer will also be continued after pressing the start button.
- Pressing the start button of the PC program transfers all total times. No remaining times or pause times are included. The grinding begins again from the start.
- After every hour, the remaining times are saved as a precaution. This means that if milling is performed for a long time and the power supply is interrupted, the remaining time can be made up (max. error <1 hour).

  Function: Once the mill is again supplied with power, the software detects the saved remaining time and indicates this with a “0” in the display. If you now press the start button on the device, the grinding will be continued.

  The remaining time can be seen in the program (see operating instructions for the program pulverisette 4 “32-bit for Windows“).

Attention
As soon as the program button “START” is used, the remaining times are lost.

Switching Off

- Press STOP on the control panel.
- Once the drive has come to a stop, the hood is unlocked and can be opened.

  It may take some time before the drive is braked, depending on the rotational speed of the main disk.
- Switch off the main switch (right side of device).

4.9 Cooling the Grinding Bowls

- with the hood open or
- at the programmed break times with the hood closed (locked) and the ventilator running.

Attention! hot surface of grinding bowl

4.10 Grinding under protective gas with gas lid

NOTE

All our gas lids are subjected to a bubble test (Werker water bath test, approved test method): The part under inspection is sealed, pressurized to 5.5 bar and submerged in a water basin. If there is a leak, bubbles will form. The bubbles that form within a certain period of time are evaluated by the operator/inspector.

Only gas lids with leak rates $<10^{-4}$ [mbar l/s] receive approval.

Important Load the mill symmetrically! See section 4.5 Mass Balance.

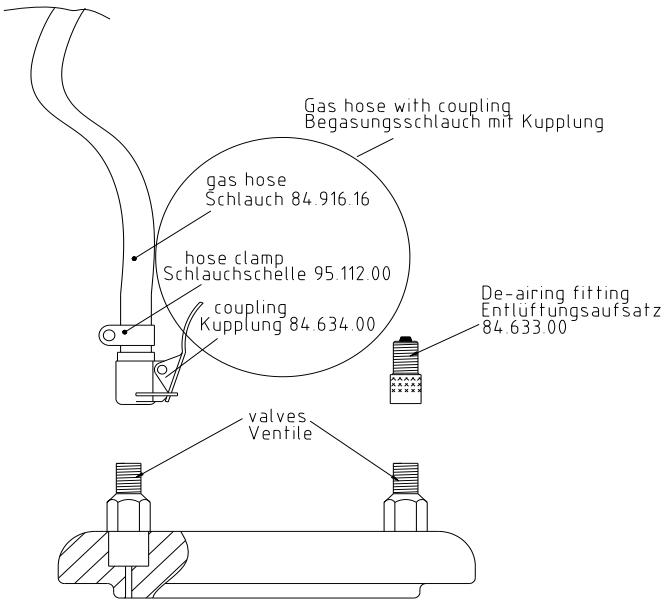
For grinding under protective gas, the same conditions apply for the selection of the grinding set and grinding balls.

Two valves are screwed onto the gas lid. Before switching on the laboratory planetary mill, you can introduce protective gas, e.g. nitrogen, through these.

A Viton flat seal is used instead of the Teflon flat seal.

Preparation for gassing

- Fill the grinding bowl with grinding balls and the material to be ground.
- Put on the lid with seal (use Viton seal).
- Insert the grinding bowl in the grinding bowl holder.
- Clamp the grinding bowl.



- Connect the gas hose to an insert gas supply with the aid of the attached hose clip.
- Screw the de-airing fitting onto one of the two valves.
- Fit the coupling of the gas hose onto the free valve. To do so, press the lever of the coupling and push the coupling onto the valve shaft as far as it will go. Release the lever.

Gassing

- Slowly open the inert gas feed.
- Press on the top of the de-airing fitting so that the air can escape from the grinding bowl.
- The inert gas will now flush the air out of the grinding bowl.
- The flushing period must be determined experimentally;
  the flushing period is dependent on, inter alia, the grinding bowl size, the filling and the gas feed.
- To end the flushing, close the inert gas feed and release the de-airing fitting.
- Unscrew the de-airing fitting.
- Pull off the coupling of the gas hose. For this, press the lever.

Warning
Switch the machine on only when both the coupling and the de-airing fitting have been removed.
Excess pressure can occur during grinding.

De-airing after grinding

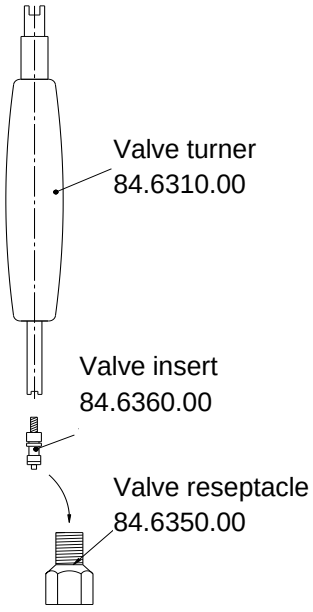
- After grinding, screw on the de-airing fitting.
- For pressure equalisation, carefully press on the de-airing fitting.
- Release the grinding bowl clamping only after de-airing.

Note
Either valve can be used for airing and de-airing.

Cleaning the valves

Both valves should be cleaned after each grinding operation.

- For this, unscrew the valve insert with the attached valve turner. Insert the thin end of the valve turner into the valve from above and turn anticlockwise.
- Unscrew the valve insert.
- Depending on the contamination, clean the valve insert with compressed air or place the valve insert in a small glass vessel filled with alcohol, clean it in the ultrasonic bath (laborette 17) and carefully dry it.
- After both valve inserts have been removed, the two valve receptacles can be cleaned from the upper side of the lid with compressed air.



Fitting the valve inserts

- Place the valve insert (spring pointing upwards) in the valve receptacle.
- Screw the valve insert in clockwise with the valve turner.

The following gas lids, each with two valves and a soft sealing ring, are available for the grinding sets:

Material	Order number
Agate 250 ml	50.8100.00
Agate 500 ml	50.8000.00
Tungsten carbide 250 ml	50.8600.00
Hardened chrome steel 80 ml	50.8700.00
Hardened chrome steel 250 ml	50.8500.00
Hardened chrome steel 500 ml	50.8400.00
Stainless Cr-Ni steel 80 ml	50.8800.00
Stainless Cr-Ni steel 250 ml	50.8300.00
Stainless Cr-Ni steel 500 ml	50.8200.00

The black, soft sealings made of Viton are temperature stabil up to 200°C.
The valves are temperature stabil up to 180°C for one hour max.

4.11 GTM System

The GTM system, order no. see price list, is available as an accessory for measuring the pressure and temperature in the grinding bowl during grinding. Instructions are provided with the GTM system.

5 Cleaning

5.1 Grinding accessories

- Clean the grinding bowl and grinding balls after each use:
  e.g. brush them clean under running water with usual cleaning agents.
- Fill the grinding bowl with grinding balls and some sand and water half-full and run the planetary motormill for 2 to 3 minutes (with the grinding bowl correctly clamped in position).
- Cleaning in the ultrasonic bath is permissible.
- When sterilising the grinding bowl and grinding balls in the heat cabinet, heat only to 100°C.

Parts of agate, sintered corundum, zirconium oxide should being cooled down carefully and slowly.
Agate parts must never be heated in the microwave (they heat up too rapidly).
They must never be subjected to temperature shocks, such shocks may destroy the parts → They burst apart explosively.

5.2 Mill

- When switched off, the mill can be wiped down with a damp cloth.

Do not allow any liquids to seep into the machine.

6 Maintenance

Before commencing maintenance work, disconnect the mains plug and secure the machine against being switched on again unintentionally.
When maintenance work is being performed, this should be indicated with a warning sign.

Attention! mains voltage

Regular cleaning is the most important part of maintenance.

Functional part	Task / Description	Test	Maintenance interval
Safety lock	Locking the hood	Hood grip cannot be opened If this test fails, you must not continue to work before the error is corrected	Before each use
Rotating bearings	Permanent lubrication	Bearing play	Every 2,000 hours or annually
Drive motor	Permanent lubrication	Bearing play	Every 4,000 hours or annually
Fan, Ventiduct	Cooling the grinding chamber and electronics	Operation; clean when dirty	2 x annually
Grinding bowl holder and clamping	rubber disk of the clamping plate and rubber disk in the grinding bowl holder	Signs of use; when it has been pressed flat and is thus non-elastic, replace it	Every 1,000 hours
seal grinding bowl	grinding bowl lid seal	if dirt pressed in the sealing, replace it!	Every 100 hours

7 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).

8 Troubleshooting Checklist

Malfunction	Possible cause	Elimination of error
Display not Illuminated	Not connected to mains	Plug in mains plug
	Main switch	Switch on the main switch
START button pressed but mill does not Start	If display not illuminated check see above	see above
	Break time active	wait for the break to end or press STOP
	safety lock was opened by hand	see section 2.3 Protective Devices
Mill automatically reduces the rotational speed	When display blinking: overload	Reduce load or accept automatically configured rotational speed
Mill stops	Overheating of the drive motor	Let the mill coll down and select lower speed
	Machine imbalance too great	Improve weight distribution: see section 4.5 Mass Balance
	drive is obstructed	eliminate trouble in grinding chamber
	belt broken	check or change belt
Hood cannot be opened	Not connected to mains	Plug in mains plug
	Main switch	Switch on the main switch
Material Escapes	Tensioning is loose	Check
	Sealing ring defective or dirty	Replace or clean the sealing ring
Uneven running with se vere vibration	Mass balance not adequate	improve mass balance: see section 4.5 Mass Balance
Error code 5 is blinking on display	Emergency off switch	Unlock emergency off switch
Error code 6	No valid program parameters (e.g. no milling time is greater than zero)	See operating instructions : 32-bit program for Windows
Error code 7	No rotational speed feedback from planetary drive	See section 2.5 Electrical Safety
Error code 8	No rotational speed feedback from main drive	See section 2.5 Electrical Safety
Error code 9	Cover not closed correctly.	Close hood and lock mechanically with rotary grip.

9 Examples of Comminution Tasks

Material			
Quantity	Material bowl and balls	balls Qnty x dia mm	Results
Edge length	Volume bowl	Grinding time	final fineness
Ruby (Stone)			
140 g	Cr-Ni-steel	6 x 30mm	100%
12 mm	250 ml	3 min	<250µm
Titandioxid TiO_2 (Dry- and Wet grinding in water)			
40 g	Cr-Ni-steel	6 x 30mm	100%
2 mm	250 ml	30 min	< 40µm
Titandioxid TiO_2 (Wet grinding in water)			
40g/50ml water	Cr-Ni-steel	6 x 30mm	100%
2 mm	250 ml	60 min	< 10µm
Carbon (Dry- and Wet grinding in Water)			
5 g	Zirkonoxid	5 x 20mm	100%
0,5 mm	80 ml	120 min	< 15µm
Aluminium oxide / Silizium oxide			
100 g	WC + Co	15 x 20mm	90%
0,1 mm	250 ml	90 min	< 20µm
Ferrovanadium			
70 g	WC + Co	5 x 30mm	70%
3 mm	250 ml	20 min	<100µm
Glass			
50 g	Achat	15 x 20mm	100%
4 mm	250 ml	15 min	< 90µm
Silicon carbide (Dry- and Wet grinding in Water)			
15 g	WC + Co	5 x 20mm	100%
3 mm	80 ml	30 min	<150µm
Silicon carbide (Dry- and Wet grinding in Water)			
15g/5 ml Wasser	WC + Co	5 x 20mm	100%
3 mm	80 ml	45 min	<71µm
Raw-phosphate			
40 g	Cr-Stahl	15 x 20mm	100%
3mm	250 ml	2 min	<250 µm

Material			
Quantity	Material bowl and balls	balls Qnty x dia mm	Results
Edge length	Volume bowl	Grinding time	final fineness
Manganous oxide MnO_2 (Wet grinding in Water)			
50g/40ml Wasser	WC + Co	15 x 20mm	100%
0,1 mm	250 ml	60 min	<20µm
Sewage sludge (dry)			
180 g	Al_2O_3	10 x 30mm	100%
8 mm	500 ml	30 min	<250µm
Activated charcoal (Wet grinding in Water)			
150 ml	Cr-Ni-Stahl	15 x 20mm	100%
0,025 mm	250 ml	30 min	< 5µm
Gypsum			
300 g	Cr-Stahl	10 x 30mm	100%
10 mm	500 ml	20 min	<200µm
Protein			
50 g	Sinterkorund 1	6 x 30mm	90%
20 mm	250 ml	90 min	< 50µm
Grain (barley)			
100 g	Sinterkorund 1	3 x 40mm	100%
3 mm	500 ml	20 min	< 150µm
Dough			
100 g	Sinterkorund 1	10 x 30mm	100%
5 mm	500 ml	3 min	< 250µm
Sugar (Wet grinding in alcohol)			
200 g	Achat	10 x 30mm	100%
1 mm	500 ml	45 min	< 10µm

10 Disclaimer

Before using this product, these operating instructions are to be carefully read and be understood. Use of the product requires expertise and it is to be carried out only by commercial users. The product may be used exclusively for the applications outlined in these instructions and within the scope of the regulations set out in these operating instructions, and it shall subject to regular maintenance. In the event of infringements of these instructions and/or improper use or maintenance, the customer assumes full liability for the functionality of the product and for such damage or injury as may occur as a result of breaching these obligations.

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FRITSCH GMBH expressly reserves the right to amend and/or to update these operating instructions without prior notice. The same applies to changes and improvements to the products described in these operating instructions. The onus for obtaining information about the current status of these operating instructions lies with the individual user. In this regard, please contact the FRITSCH GMBH distributor in your area or apply directly to Fritsch GmbH, Industriestrasse 8 D-55473 Idar-Oberstein, Germany.

Not all parts illustrated must be built into the product. A right to delivery of these parts does not exist. If interested in them, please contact the FRITSCH GMBH distributor in your locality or Fritsch GmbH, Industriestrasse 8, D-55743 Idar-Oberstein directly.

FRITSCH GMBH endeavours, with the greatest of care, to continually improve the quality, reliability and safety of their products and to conform with the state of the art. The products supplied, as well as these operating instructions, correspond with, at the time of transfer from the influence area of FRITSCH GMBH, the respective state of the art.

Customers agree and acknowledge that, through usage of the product, defects, faults or errors cannot be ruled out entirely. To avoid the risks thereof, of damage to persons or property being incurred, or of any other direct or indirect damages, customers must provide for adequate and comprehensive safety measures whilst working with the product.

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Both adherence with these operating instructions and the conditions and methods of installation, operation, usage and maintenance of the product cannot be monitored by Fritsch GmbH. Improper execution of the installation can lead to property damage and, as a result, also place people at risk. Accordingly, we accept no responsibility or liability for losses, damages or expenses resulting from, or in any way connected with, defective installation and improper operation, as well as from incorrect utilisation and maintenance.


FRITSCH