

# Operating instructions

## PLANETARY MICRO MILL

### PULVERISETTE 7 *classic line*

Valid starting with: 07.4000/0487



Read the instructions prior to performing any task!

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Translation of the original operating instructions

Fritsch GmbH  
Milling and Sizing  
Industriestraße 8  
D - 55743 Idar-Oberstein  
Telephone: +49 6784 70-0  
Email: [info@fritsch.de](mailto:info@fritsch.de)  
Internet: [www.fritsch.de](http://www.fritsch.de)

## Certifications and CE conformity

### Certification

Fritsch GmbH has been certified by the SGS-TÜV Saar GmbH.



An audit certified that Fritsch GmbH conforms to the requirements of the DIN EN ISO 9001:2015.

### CE Conformity

The enclosed Conformity Declaration lists the guidelines the FRITSCH instrument conforms to, to be able to bear the CE mark and the UKCA mark!



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## 1 Basic structure



- 1 Hood handle
- 2 Latch
- 3 Hood
- 4 Membrane keyboard
- 5 Spindle clamping device
- 6 Lock

- 7 Voltage rotary switch
- 8 Main switch
- 9 Mains connection
- 10 Device fuse 2x 10 A T
- 11 RS232 - interface
- 12 Support disc



## 2 Safety information and use

### 2.1 Requirements for the user

This operating manual is intended for persons assigned with operating and monitoring the Fritsch PULVERISETTE 7 classic line. The operating manual and especially its safety instructions are to be observed by all persons working on or with this device. In addition, the applicable rules and regulations for accident prevention at the installation site are to be observed. Always keep the operating manual at the installation site of the PULVERISETTE 7 classic line.

People with health problems or under the influence of medication, drugs, alcohol or exhaustion must not operate this device.

The PULVERISETTE 7 classic line may only be operated by authorised persons and serviced or repaired by trained specialists. All commissioning, maintenance and repair work may only be carried out by technically qualified personnel. Qualified personnel are persons who, because of their education, experience and training as well as their knowledge of relevant standards, regulations, accident prevention guidelines and operating conditions, are authorised by those responsible for the safety of the machine to carry out the required work and are able to recognize and avoid possible hazards as defined for skilled workers in IEC 364.

In order to prevent hazards to users, follow the instructions in this manual.

Malfunctions that impair the safety of persons, the PULVERISETTE 7 classic line or other material property must be rectified immediately. The following information serves both the personal safety of operating personnel as well as the safety of the products described and any devices connected to them: All maintenance and repair work may only be performed by technically qualified personnel.

This operating manual is not a complete technical description. Only the details required for operation and maintaining usability are described.

Fritsch has prepared and reviewed this operating manual with the greatest care. However, no guarantee is made for its completeness or accuracy.

Subject to technical modifications.

## 2.2 Scope of application



### NOTICE

Fritsch laboratory mills are not intended for use in explosion-hazardous areas. Fritsch laboratory mills therefore fall outside the scope of the 94/9/EC Directive, but within the scope of the Machinery Directive 2006/42/EC. The use of Fritsch laboratory mills within explosion-hazardous areas is not permissible according to ATEX (94/9/EC) and is only allowed if additional explosion protection measures are taken. According to the Machinery Directive 2006/42/EC, there are no ignition sources in our mills that can become effective during normal operation. Nonetheless, there may be ignition sources in our mills that may become effective in the event of probable malfunctions.

Because Fritsch has only limited information on the composition of the ground product in use, its final fineness and therefore ultimately its ignition temperature, no statement regarding the explosion risk during intended use in conjunction with the occurring energy input can be made.

The occurrence of dust explosions can therefore never be completely ruled out. The user must create an explosion protection document in accordance with the ATEX 137 Directive (1999/92/EC) and define appropriate protective measures.



### NOTICE

This laboratory instrument is designed for an 8-hour shift operation at 30 % duty cycle and not for continuous operation.

The duty cycle is defined as the ratio of load duration to run time. The run time is defined as load duration plus pause time. According to DIN EN 60034-1 (VDE 0530, IEC34-1) a continuous operation already takes place after a standardised run time of 10 minutes. At 30 % duty cycle (DC = ratio of load duration to run time) a load duration of 3 minutes and a pause time of 7 minutes would be within standard.

If the standardised run time of 10 minutes is exceeded, then, by definition, there would be a continuous operation and disproportionate temperature increases may occur, possibly involving increased wear.

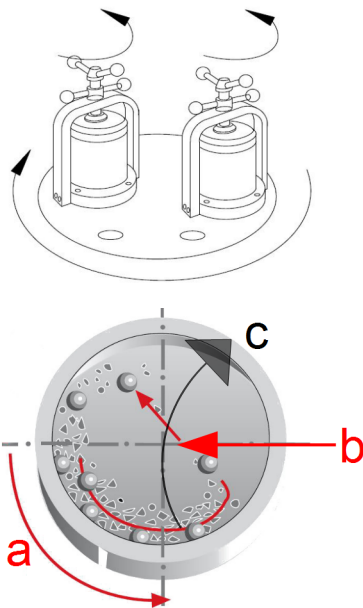
The micro mill, PULVERISETTE 7 *classic line*, can be applied universally for fast dry and wet grinding of inorganic and organic samples for analysis, quality inspection or material testing.

During synthesis, the planetary micro mill is used for mixing and homogenising dry samples, emulsions or suspensions.

### 2.2.1 Operating principle

The grinding stock is crushed and ground by grinding balls in a grinding bowl. The centrifugal forces from the rotation of the grinding bowls around their own axis and from the rotating support disc act on the contents of the grinding bowl which consists of grinding stock and grinding balls.

The grinding bowl and the support disc have opposite directions of rotation, so that the centrifugal forces alternate in the same direction and in the opposite direction. The result is that the grinding balls run down the inside of the bowl's wall providing a friction effect and the grinding balls hit the opposite wall of the grinding bowl providing an impact effect. The impact effect is amplified by the impact of the grinding balls against each other.



- a Rotation of the grinding bowl
- b Centrifugal force
- c Support disc movement

### 2.2.2 Drive motor and speed regulation

A maintenance-free three-phase motor operated via a frequency converter is used as the drive.

## 2.3 Obligations of the operator

Before using the PULVERISETTE 7 classic line, this manual is to be carefully read and understood. The use of the PULVERISETTE 7 classic line requires technical knowledge; only commercial use is permitted.

The operating personnel must be familiar with the content of the operating manual. For this reason, it is very important that these persons actually receive the present operating manual. Ensure that the operating manual is always near the device.

The PULVERISETTE 7 classic line may exclusively be used within the scope of applications set down in this manual and within the framework of guidelines put forth in this manual. In case of non-compliance or improper use, the customer assumes full liability for the functional capability of the PULVERISETTE 7 classic line and for any damage or injury arising from failure to fulfil this obligation.

By using the PULVERISETTE 7 classic line the customer agrees with this and recognizes that defects, malfunctions or errors cannot be completely excluded. To prevent risk of damage to persons or property or of other direct or indirect damage, resulting from this or other causes, the customer must implement sufficient and comprehensive safety measures for working with the PULVERISETTE 7 classic line.

## Safety information and use

Neither compliance with this manual nor the conditions and methods used during installation, operation, use and maintenance of the PULVERISETTE 7 classic line can be monitored by Fritsch GmbH. Improper execution of the installation can result in property damage and thus endanger persons. Therefore, we assume absolutely no responsibility or liability for loss, damage or costs that result from errors at installation, improper operation or improper use or improper maintenance or are in any way connected to these.

The applicable accident prevention guidelines must be complied with.

Generally applicable legal and other obligatory regulations regarding environmental protection must be observed.

## 2.4 Information on hazards and symbols used in this manual

### Safety information

Safety information in this manual is designated by symbols. Safety information is introduced by keywords that express the extent of the hazard.



#### **DANGER**

This symbol and keyword combination points out a directly hazardous situation that can result in death or serious injury if not avoided.



#### **WARNING**

This symbol and keyword combination points out a possibly hazardous situation that can result in death or serious injury if not avoided.



#### **CAUTION**

This symbol and keyword combination points out a possibly hazardous situation that can result in slight or minor injury if not avoided.



#### **NOTICE**

This symbol and keyword combination points out a possibly hazardous situation that can result in property damage if not avoided.

### Special safety information

To call attention to specific hazards, the following symbols are used in the safety information:



#### **! DANGER**

This symbol and keyword combination points out a directly hazardous situation due to electrical current. Ignoring information with this designation will result in serious or fatal injury.



## **DANGER**

This symbol and keyword combination designates contents and instructions for proper use of the machine in explosive areas or with explosive substances. Ignoring information with this designation will result in serious or fatal injury.



## **DANGER**

This symbol and keyword combination designates contents and instructions for proper use of the machine with combustible substances. Ignoring information with this designation will result in serious or fatal injury.



## **WARNING**

This symbol and keyword combination points out a directly hazardous situation due to movable parts. Ignoring information with this designation can result in hand injuries.



## **WARNING**

This symbol and keyword combination points out a directly hazardous situation due to hot surfaces. Ignoring information with this designation can result in serious burn injuries due to skin contact with hot surfaces.

### Safety information in the procedure instructions

Safety information can refer to specific, individual procedure instructions. Such safety information is embedded in the procedure instructions so that the text can be read without interruption as the procedure is being carried out. The keywords described above are used.

Example:

1. ➔ Loosen screw.

2. ➔



## **CAUTION**

**Risk of entrapment at the lid.**

Close the lid carefully.

3. ➔ Tighten screw.

### Tips and recommendations







*This symbol emphasises useful tips and recommendations as well as information for efficient operation without malfunction.*

## Safety information and use

### Further designations

To emphasise procedure instructions, results, lists, references and other elements, the following designations are used in this manual:

Designation	Explanation
 1., 2., 3. ...	Step-by-step procedure instructions
	Results of steps in the procedure
	References to sections in this manual and relevant documentation
	Lists without a specific order
[Button]	Operating elements (e.g. push button, switch), display elements (e.g. signal lamps)
'Display'	Screen elements (e.g. buttons, function key assignment)

## 2.5 Device safety information

### Please observe!

- Only use original accessories and original spare parts. Failure to observe this instruction can compromise the safety of the machine.
- Accident-proof conduct is to be strictly followed during all work.
- Comply with all currently applicable national and international accident prevention guidelines.



#### CAUTION

##### Wear hearing protection!

If a noise level of 85 dB(A) is reached or exceeded, ear protection should be worn to prevent hearing damage.



#### WARNING

The maximum accepted concentration (MAC) levels of the relevant safety guidelines must be observed; if necessary, ventilation must be provided or the machine must be operated under an extractor hood.



**DANGER**

**Explosion hazard!**

- When grinding oxidizable substances, e.g. metals or coal, there is a risk of spontaneous combustion (dust explosion) if the share of fine particles exceeds a certain percentage. When grinding these kinds of substances, special safety measures must be taken and the work must be supervised from a specialist.
- The PULVERISETTE 7 classic line is not explosion protected and is not designed to grind explosive materials.

- Do not remove the information signs.



**NOTICE**

Immediately replace damaged or illegible information signs.

- Unauthorised alteration of the PULVERISETTE 7 classic line will void Fritsch's declaration of conformity to European directives and void the guarantee.
- Only use the PULVERISETTE 7 classic line when it is in proper working order, as intended and in a safety- and hazard-conscious manner adhering to the operating manual. In particular, immediately rectify any malfunctions that could pose a safety hazard.
- If, after reading the operating manual, there are still questions or problems, please do not hesitate to contact our specialised personnel.

## 2.6 Protective equipment



*Protective equipment must be used as intended and may not be disabled or removed.*

*All protective equipment must be checked regularly for integrity and proper functioning.*

For start-up, the hood (3) has to be closed.

The hood (3) is locked:

- without mains connection
- during operation



*The hood (3) can only be opened if the mill's drive is at a standstill.*

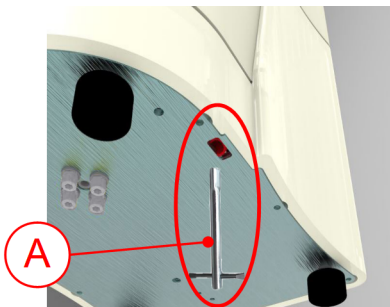
## Safety information and use

### 2.6.1 Opening the hood without mains connection



#### CAUTION

The emergency release must not be activated while the machine is running! Disconnect the machine from the mains before the emergency release. Failure to observe this will render void the guarantee, and releases us from liability for any resulting damage to the device as well as for any resulting personal injury.



1. ➤ Insert the included triangular key (A) into the bore hole on the bottom side of the PULVERISETTE 7 *classic line* and turn it to the right.
2. ➤ Unlock the latch (2) by turning it.
3. ➤ Pull the hood (3) up to open it.
4. ➤ The mill can only be switched on again if the hood (3) is closed and the safety lock (7) has been enabled again by turning the triangular key to the right.

### 2.7 Hazardous points



#### CAUTION

- Crushing hazard when closing the hood (3).
- Crushing hazard at the spindle clamping device (5).

### 2.8 Electrical safety

#### 2.8.1 General information

- The main switch (8) separates the device from the mains on two poles.
- Switch off the main switch (8) if the planetary micro mill will be idle for an extended period of time (e.g. overnight).

#### 2.8.2 Protection against restart

If a power failure occurs during operation or if the device is switched off with the main switch (8), the hood (3) is locked. The hood lock (6) is automatically released when the power returns. For safety reasons, however, the mill does not restart.



### 2.8.3 Overload protection

- In the event of an overload, the device reduces the speed in a controlled manner. The REDUCED SPEED light is lit as a warning. The device can be used again with the reduced speed.
- The device switches off if the drive motor becomes too hot.
- The device switches off if the drive is blocked. (See ➔ *Chapter 9 'Repairs' on page 43*)

## Technical data

### 3 Technical data

#### 3.1 Dimensions

500 x 370 x 530 mm (height x width x depth)

#### 3.2 Weight

Net: approx. 35 kg

Gross: approx. 55 kg

#### 3.3 Operating noise

Emissions value of workplace according to DIN EN ISO 3746:2005 is up to 96dB (A). The value fluctuates strongly, depending on the speed, the grinding stock and the type of grinding bowl and grinding balls.

#### 3.4 Voltage

The device can be operated in two voltage ranges:

- Single phase alternating current 100 - 120 V  $\pm$  10%, and,
- Single phase alternating current 200 - 240 V  $\pm$  10%.

(See also ➡ *Chapter 4 'Installation' on page 20*)

Transient overvoltages in accordance with overvoltage category II are permitted.

#### 3.5 Current consumption

Depending on the mains voltage, the maximum current consumption is in the ranges:

- 115 V  $\rightarrow$  8.8 A
- 230 V  $\rightarrow$  3.7 A

#### 3.6 Protection class

IP 21

#### 3.7 Power consumption

Depending on the voltage range, the maximum power consumption is approx. 880 W.

#### 3.8 Electrical fuses

Device fuse (10): 5 x 20 A T

Micro-fuse 10 A T in the frequency converter

### 3.9 Material

- Maximum feeding size 5 mm
- Maximum feed amount 2 x 20 ml

### 3.10 Final fineness

- Dry grinding up to  $d_{50} < 20 \mu\text{m}$  (depending on the material)
- Wet grinding up to  $d_{50} < 1 \mu\text{m}$  (depending on the material)

## 4 Installation

### 4.1 Transport

The device is delivered on a transport pallet with a wooden cover. We recommend using a forklift or pallet truck for transporting the packed device.



#### DANGER

Do not step under the transport pallet during transport.



#### WARNING

Improper lifting can lead to personal injury or property damage. The machine is only to be lifted with suitable equipment and by qualified personnel.

The guarantee excludes all claims for damage due to improper transport.

### 4.2 Unpacking

- Remove the nails that fasten the wooden hood to the transport pallet.
- Lift the hood off the transport pallet.
- The pre-perforated segments can be detached so that the foam parts can be removed more easily.
- Please store the transport packaging so that it can be reused if you need to return the product. Fritsch GmbH accepts no liability for damage caused by improper packaging (packaging that is not from Fritsch).
- Compare the contents of the delivery with your order.



*Grinding bowls made of hardened steel may have recesses on the surface caused during production. They do not have an impact on grinding or the grinding results and usually disappear after the first grinding operation.*

*These recesses on the surface, if present, are within the range of the permissible production tolerances. Complaints relating to such grinding bowls therefore cannot be accepted.*

### 4.3 Setting up



#### NOTICE

Allow the device to acclimatise for two hours before commissioning. High temperature differences can lead to condensation in the device and damage to the electronics after switching on.

Strong temperature fluctuations can occur during transport or interim storage. Depending on the temperature difference between the installation site and the transport or storage environment, condensation can form inside the device. This can damage the electronics if the devices are switched on too early. Wait for at least two hours after setup before switching on the device.



**DANGER**

Do not step under the transport pallet during transport.



**CAUTION**

The weight of the planetary micro mill is approx. 35 kg!



**CAUTION**

**Crushing hazard!**

Always lift with 2 persons.

Hold the bottom edge of the housing when lifting.



**NOTICE**

Never operate the mill while it is standing on the transport pallet!

- Lift the mill from the transport pallet with at least 2 persons.
- Place the mill on a flat, stable surface.
- Make sure that the planetary micro mill can be easily accessed. There has to be sufficient space to reach the main switch on the back of the device.
- Keep any obstructions away from the air outlet on the side ventilation grate. Risk of overheating!

## 4.4 Ambient conditions



**WARNING**

**Mains voltage!**

- The device may only be operated indoors.
- The surrounding air may not carry any electrically conductive dust.
- Maximum relative humidity 80% for temperatures up to 31°C, linearly decreasing down to 50% relative humidity at 40°C.

- The room temperature has to stay between 5 - 40°C.
- Altitudes up to 2000 m
- Degree of pollution 2 according to IEC 664.

### 4.5 Electrical connection



#### DANGER

##### Provide short-circuit protection!

Risk of damage due to short-circuits.

- Make sure that the socket is connected to a mains line protected with a residual current circuit breaker.



#### DANGER

##### Mains voltage!

Changes to the connection line may only be made by a qualified person.



#### CAUTION

Ignoring the values on the type plate may result in damage to the electrical and mechanical components.

Before establishing the connection, compare the voltage and current values stated on the type plate with the values of the mains system to be used.



*The mains voltage has been set at the factory to that of the specific country. The mains voltage only has to be adjusted if it deviates from the value on the type plate. If adjustment is necessary, proceed as in ➔ Chapter 4.5.1.1 'Adjusting the mains voltage with the rotary switch (7)' on page 23 and ➔ Chapter 4.5.1.2 'Adjusting the mains voltage in setup mode' on page 23.*



#### NOTICE

Fritsch mills are speed controlled. The devices are equipped for this with frequency converters. In order to comply with the EMC directive, many measures must be taken to prevent operational transient emissions.

The possible leakage currents resulting from filtering measures can trigger a conventional residual current circuit breaker in the mains line. **This is no defect!**

To prevent this, special residual current circuit breakers, which are adapted for operation with frequency converters, are commercially available.

Operation without a residual current switch is possible, but must be done in accordance with the relevant regulations.

## 4.5.1 Adjusting the mains voltage

### 4.5.1.1 Adjusting the mains voltage with the rotary switch (7)



#### CAUTION

Only qualified personnel may change the voltage range on the device!



#### CAUTION

The voltage range may only be adjusted after the mains has been disconnected. Disconnect the device from the mains!

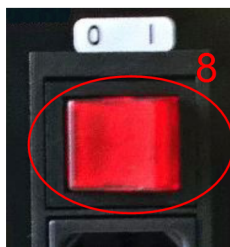


1. ➔ The rotary switch (7) for adjusting the mains voltage is located on the back of the device. Rotate this switch to the required voltage. The slit in the rotary switch indicates the chosen voltage.
2. ➔ Connect the device to the mains.

### 4.5.1.2 Adjusting the mains voltage in setup mode



1. ➔ Press and hold the STOP button on the front of the control panel.



2. ➔ Switch on the device with the main switch (8) on the back of the device, and release the STOP button.
3. ➔ If POWER SUPPLY is flashing, the device is in setup mode. If POWER SUPPLY is not flashing, repeat the procedure.

## Installation



4. ➤ Use the +/- ROTATIONAL SPEED buttons to adjust the level of the mains voltage (90-260 V) for the existing mains system.



5. ➤ To save and exit setup mode, press the STOP button.

### 4.5.2 Setting device specifications



#### NOTICE

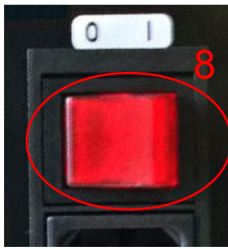
"P7" must always be displayed in the REPETITIONS field. The Fritsch company assumes no guarantee for damage resulting from changing this setting.



## 5 Initial start-up

Perform initial start-up only after all work as described in ➔ *Chapter 4 'Installation' on page 20* has been carried out.

### 5.1 Switching on



- The device must be connected to the power supply if this has not been done already.
- Switch on the device with the main switch (8) on the back of the device.
- The POWER SUPPLY lamp lights up.

### 5.2 Function check



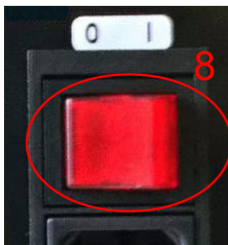
#### CAUTION

Only perform the function check at a speed of 100 1/min. Use two empty grinding bowls of equal weight to prevent too great an imbalance.



- Open the hood (3).
- Clamp two empty lidded grinding bowls with of the same weight.
- Close the hood (3).
- Set the speed on the control panel as 100 1/min. (see ➔ *Chapter 6.8 'Setting the speed' on page 35*)
- Press START on the control panel.
- The hood (3) is locked and the mill starts up at the preselected speed.

### 5.3 Switching off



- Press the STOP button on the control panel.
- Shortly after the mill comes to a standstill, the hood is unlocked and can be opened.
- Switch off the device with the main switch (8) on the back of the device.

## 6 Using the device



### DANGER

Before starting the machine, make sure that the grinding bowl has been tensioned correctly and that there are no loose parts inside the device. There is a risk of loose grinding bowls or parts being projected. Failure to observe this will render void the guarantee, and releases us from liability for any resulting damage to the device or personal injury.



### NOTICE

During grinding, high temperatures and high pressure may appear in the grinding bowl.

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

The adhesive is resistant to temperatures up to approx. 140 °C. Above 140 °C, the adhesive will liquefy. That can cause irreparable damage to the insert. The grinding bowl will definitely be rendered unusable.



*The device requires a start-up phase at the beginning in order to reach maximum performance. A well-filled and heavy grinding set may initially operate at a lower speed than after running for approximately 1 - 2 hours.*

### 6.1 Choice of grinding bowls and grinding balls



### CAUTION

If the grinding elements used are not genuine accessories, we assume no guarantee and exclude all liability for damage to the device or for personal injury.



### CAUTION

The grinding element is subject to normal wear when used. Before every grinding operation, check the wall thickness of the grinding bowls. In the event of severe wear, replace the grinding bowl. If this is not done, the prevailing high centrifugal forces during grinding may cause the grinding balls to penetrate the bowl's wall and damage the mill. Failure to observe this will render void the guarantee and release us from liability for any resulting damage to the device or personal injury.


**NOTICE**

Each grinding process means wear to the grinding element. Therefore, please pay attention to what components the material of the grinding element contains and whether these could react with the sample. Such reactions may have consequences. A simple reaction with minor consequences may be, for example, the grinding of samples containing sulphur in the steel bowl, which contains iron. The iron released by abrasion can combine with sulphur and react to form iron sulphide. This can lead to black deposits on your grinding set.

The hardness and density (specific weight) of the grinding bowl and grinding balls used have to be greater than that of the material used to prevent excessive wear by abrasion.

Material (bowl and balls)	Main components of the material	Density in g/cm <sup>3</sup> High density means high impact energy!	Abrasion resistance	Use for grinding stock
Agate	(99.9% SiO <sub>2</sub> )	2.65	Good	Soft to medium-hard samples
Silicon nitride	(90% Si <sub>3</sub> N <sub>4</sub> )	3.25	Extremely good	Abrasive samples, metal-free grinding
Sintered corundum	(99.7% Al <sub>2</sub> O <sub>3</sub> )	3.9	Fairly good	Medium-hard, fibrous samples
Zirconium oxide	(96.2% ZrO <sub>2</sub> )	5.7	Very good	Fibrous, abrasive sam- ples
Hardened, stainless steel	(16.0 - 18.0% Cr)	7.7	Fairly good	Medium-hard, brittle samples
Tungsten carbide	(93% WC + 6% Co)	14.9	Very good	Hard, abrasive samples

The grinding bowls and grinding balls made of zirconium oxide are resistant to acids - apart from hydrofluoric acid.

Normally choose a grinding bowl and grinding balls that are made of the same material.

Exception: Tungsten carbide balls (<20 mm) may be temporarily (a few minutes) combined with grinding bowls made of hardened steel.

## Using the device

### 6.1.1 Size of the grinding balls

Type of feed material	Suitable ball diameter
Coarse feed size of $\leq 5$ mm	15 mm or 10 mm
Fine material 0.5 mm	10 mm or smaller
Homogenisation of dry or liquid samples	10 mm or smaller

These are reference values: The size of bowls and grinding balls may need to be determined through experimentation.



#### NOTICE

It is not advisable to mix balls of different diameters. If balls with different diameters are used, increased wear and damage to the grinding elements is to be expected.

### 6.1.2 Number of balls per grinding bowl (independent of the material quantity)

A higher number of balls will reduce the grinding time and the grinding result will have a smaller particle size distribution.

Ball diameter (mm)	Grinding bowl volume (ml)	12	45
5	Number of balls (pcs)	50	180
10	Number of balls (pcs)	6	18
15	Number of balls (pcs)	-	7

These are reference figures: The number of balls may need to be determined through experimentation.

Tab. 1: Grinding balls  $\leq 3$  mm: Recommended ball mass per grinding bowl in grams

Grinding bowl / Useful capacity (material to be ground)	12 ml 0,5 - 5 ml	45 ml 3 - 20 ml
Material		
Zirconium oxide	20 g	70 g
Hardened, stainless steel	30 g	90 g
Hardmetal tungsten carbide	50 g	200 g

### 6.1.3 Calculated weight of a ball

Ball diameter in mm		5	10	15
Material	Density in g/cm <sup>3</sup>	Calculated weight of a ball in g		
Agate	2.65	0.17	1.39	4.68
Silicon nitride	3.25	0.20	1.7	5.48
Sintered corundum	3.9	0.25	1.99	6.72
Zirconium oxide	5.7	0.37	2.98	10.07
Hardened, stainless steel	7,7	0,50	4,03	13,60
Tungsten carbide	14,9	0,96	7,70	25,98

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

Example: A 45 ml agate bowl is to be filled with 185 agate balls with a diameter of 5 mm.

Calculation:  $0.17 \text{ g} \cdot 185 \text{ pcs} \approx 31.45 \text{ g}$

31.45 g of grinding balls can be weighed and inserted in the grinding bowl, thus avoiding the time required for counting the balls.

## 6.2 Filling quantities of grinding bowls



#### CAUTION

For grindings with large balls with a diameter of >10 mm, at least half of the maximum sample quantity must be inserted. If the suspension is highly fluid or the material quantity is too small, the balls will not have any resistance and the balls and the grinding bowl could be damaged. The result is the same as if it had been filled with no grinding stock.



#### NOTICE

Never operate the mill without grinding stock! This can lead to grinding balls and grinding bowls getting damaged.



#### NOTICE

If the minimum filling quantity is fallen short of, increased wear due to abrasion is to be expected. This can cause irreparable damage to the mill components.



#### NOTICE

The filled in volume can increase during the grinding. Check the volume after a brief grinding time.

## Using the device

Grinding bowl	min. filling	max. filling
45 ml	3 ml	20 ml
12 ml	0,5 ml	5 ml

### 6.3 Filling the grinding bowl



#### CAUTION

**Do not fill in any dry ice or liquid nitrogen in the grinding bowls!**

Adding dry ice or liquid nitrogen into the grinding bowls can lead to a sudden increase in volume with a high static pressure. This can result in an explosion of the grinding bowls.

**Do not fail to comply with the following sequence:**

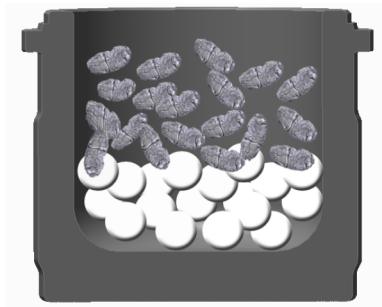
1. ➔ Place the grinding balls in the empty bowl.
2. ➔ Fill grinding stock onto the balls.



#### NOTICE

Make sure the sealing surface is clean and the seal is not damaged.

3. ➔ Place the lid on the grinding bowl.



### 6.4 Factors with an impact on grinding

#### 6.4.1 Running time (grinding duration)

To reduce the grinding time, you can use a grinding bowl and grinding balls with a higher density, and thus a higher impact energy.

## 6.4.2 Speed

Higher speeds shorten the grinding time and increase the percentage of fine particles.

Lower speeds increase the grinding time and lower the temperature, which can lead to fewer pauses. This means that the overall working time may remain the same. The wear is increased in this case though.



*Fritsch recommends however to use the maximum speed and to plan for enough pauses so that the wear is minimised.*



*For thermally sensitive materials, the optimal rotational speed needs to be determined through experimentation.*

## 6.4.3 Reverse mode

- Useful for mechanical alloying.
- Improvement of the homogeneity of the sample during mixing.



*A low to medium speed is recommended for mixing (dry or wet).*

## 6.4.4 Number and size of the balls



### NOTICE

It is not advisable to mix balls of different diameters. If balls with different diameters are used, increased wear and damage to the grinding elements is to be expected.



*You can find the recommended number and size of balls in ➔ Chapter 6.1 'Choice of grinding bowls and grinding balls' on page 26.*

Larger balls are used for pre-crushing. (See ➔ Chapter 6.1.2 'Number of balls per grinding bowl (independent of the material quantity)' on page 28).

To increase the proportion of fine particles, the large balls need to be replaced by smaller ball sizes during the course of the grinding process.

## Using the device

### 6.4.5 Weight of the balls (type of material)

A higher mass (specific weight) of the grinding balls accelerates grinding. (see table in ➔ Chapter 6.1 'Choice of grinding bowls and grinding balls' on page 26).

### 6.4.6 Dry grinding



#### DANGER

##### Dust explosion!

There is a risk of spontaneous combustion especially for very fine metal oxides and a thus resulting dust explosion. Mind the external temperature and the pressure that can develop in the grinding bowl during the dry grinding.



#### NOTICE

Dry grindings need to be checked in briefer intervals. Longer dry grindings, without pauses and checks, can cause the grinding set to gum up and to become damaged. In particular with agate, the grinding bowl and grinding balls can become damaged after a short grinding time.

At a particle size of less than 20 µm, the surface forces prevail. The grinding stock begins to stick.

Additional dry comminution can be achieved by adding surface-active substances to the material to be ground.

Examples (maximum amount to be added in mass %)

- Stearic acid 2-3 %
- Aerosil (fine-particle silicic acid) 0.5 - 2 %
- Quartz sand ~ 2 %
- Glass powder ~ 2 %
- Glycol (Ethylene glycol) ~ 0.1 - 0.5 % (Δ 5 - 25 droplets)
- Triethanolamine ~ 0.1 - 0.5 %

### 6.4.7 Wet grinding (grinding in a suspension)



#### DANGER

##### Explosion hazard! Ignition hazard!

The device is not explosion-protected. If flammable liquids are used, make sure that the heat developing in the grinding bowl does not reach the solvent's boiling point. Program appropriate cooling phases. If the vapour pressure is too high, vapours may escape and ignite.

If it can be avoided, we recommend using non-flammable liquids or liquids with a high boiling point. The boiling point should be above 80 °C and above 100 °C for a long grinding duration.



During the transition to grinding in suspension, you can add a liquid auxiliary agent with high boiling point and low vapour pressure, e.g. water, white spirits (boiling point 100 - 140°C), or alcohols with a high boiling point (e.g. isopropanol)

We recommend that you only use so much liquid that the suspension has the same consistency as motor oil. With this viscosity the best results can be achieved in most cases.

## 6.5 Clamping the grinding bowls

### 6.5.1 Clamping with the spindle clamping device (5)

Carry out the following checks before clamping the grinding bowls:

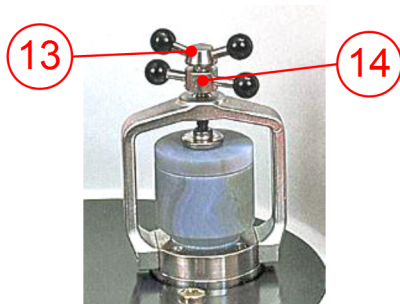
- Check the cork disc for damage  
Replace the cork discs if they have been pressed flat
- The Teflon flat seal (for the seal between the lid and the bowl) should not be damaged or soiled.  
Replace heavily deformed Teflon flat seals.
- Check the rubber disc for damage!  
Replace rubber discs that have been pressed flat and are protruding laterally from the pressure piece.
- The end faces of the counter plates and the clamping bracket should not be dirty.
- The lid and bowl surfaces that contact the Teflon flat seal must be clean.

13 Spindle screw

14 Lock nut

Clamping

- Place the sealing ring and the lid onto the bowl.
- Place the grinding bowl in the grinding bowl holder on the cork disc; do not force it!
- Turn the spindle screw (13) all the way down until the pressure piece of the rubber disc lies flush on the lid.
- Screw the spindle screw by hand until tight.
- Turn the lock nut (14) all the way down until it lies flush on the clamping bracket.
- Tighten the lock nut by hand.



#### **DANGER**

After several minutes of grinding and in the cooling-down phases, check that the spindle screw and the lock nut are still firmly connected.

## 6.6 Mass balance

For mass balancing always clamp a bowl with a lid and sealing ring of the same weight opposite as a counterweight. This bowl can be empty (without balls or grinding stock), if the weight difference does not exceed 70 - 100 g.

## Using the device



### NOTICE

If the weights of the bowls differ too much, this may result in the planetary micro mill shifting position on the supporting surface.

## 6.7 Grinding duration



### WARNING

#### Burn hazard!

Grinding bowls can get very hot after long grinding durations. Wear protective gloves for removal after grinding or during the grinding breaks.

Depending on the application, the grinding duration should be adapted to the development of heat in the bowls. The temperature inside the bowls is 20 - 30 °C warmer than the outer casing temperature.



### CAUTION

The maximum permissible temperature of the grinding bowl outer casing is 100 - 110 °C (agate, max. 70 - 80 °C).

The grinding duration is therefore based on the maximum bowl temperature. The grinding duration at which the temperature is not exceeded depends on the material, ball, and speed. For this reason, the user should determine it through experimentation.

The temperature of the outer casing of the grinding bowl during wet grinding must be at least 30 °C below the boiling point of the liquid used.



### NOTICE

- Monitor the warming of the grinding stock.
- A longer running time may require pause times for cooling down.
- Check that the clamping device is firmly connected when switching on again after a cooling-down phase.



### NOTICE

If grinding bowls are removed during a grinding pause, check that they fit correctly before the device is switched back on.



*The extent to which the heating up of grinding stock needs to be observed depends of course on the sample used in each individual case. Note → a longer running time may also require a longer pause time for cooling down.*

To reduce the grinding time, you can use a grinding bowl and grinding balls with a higher density, and thus a higher impact energy.

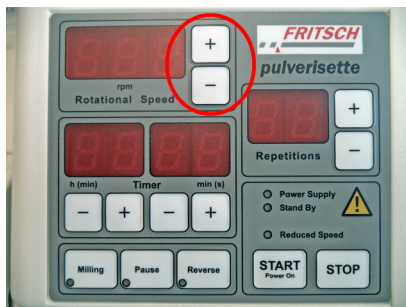
The mill can also run for several hours during low-speed operations for mixing and homogenisation.

Operation with an external time switch is not possible.

## 6.8 Setting the speed



- Switch on the main switch (8) on the back of the device (I).
- The green POWER SUPPLY ready status indicator lights up on the control panel.



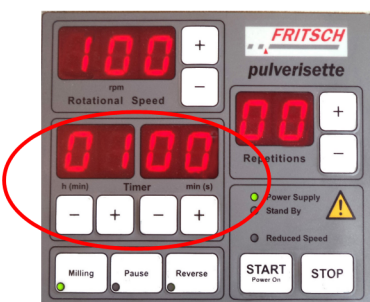
### ROTATIONAL SPEED control panel area

Press and hold the + or - button.

The speed can be selected in steps of 10 between 100 and 800 1/min.

The actual speed is displayed during operation. The nominal speed is briefly displayed when the + or - button is pressed.

## 6.9 Setting the running time



### TIMER control panel area

- Press the MILLING button, it lights up; press the + or - button and select the running time in minutes (0..99) and seconds (0..59).
- Set the pause time if cooling down is necessary. Press the "Pause" button, it lights up; press the + or - button and select the pause time in minutes (0..99) and seconds (0..59).

If no pause time is required, set the pause time to 0.

## Using the device

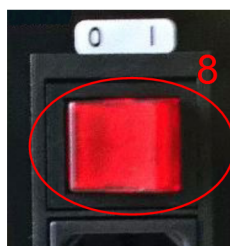


- If the combination minutes/seconds is set in setup mode instead of hours/minutes (see ➡ Chapter 6.9.1 'Changing the time unit in setup mode' on page 36), the numbers at **h** indicate the minutes and at **min** the seconds!  
The **factory setting** for the time unit is minutes and seconds. (Display: 1)
- The remaining running times and the pause times are displayed during operation.
- Operation with an external time switch is not possible.
- Running times ➡ Chapter 6.4.1 'Running time (grinding duration)' on page 30.
- Interrupt grinding by pressing the STOP button. Continue grinding by pressing the START button. This takes into account the previous grinding duration and the number of repetitions.

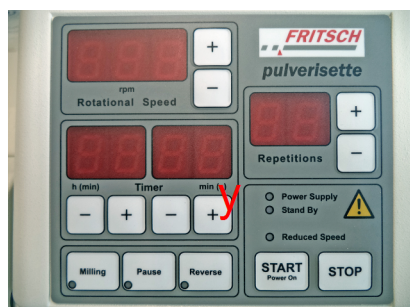
### 6.9.1 Changing the time unit in setup mode



- ➡ When the device has been switched off, press and hold the STOP button on the front control panel.



- ➡ Switch on the device with the main switch (8) on the back of the device.
- ➡ If POWER SUPPLY is flashing, the device is in setup mode. If POWER SUPPLY is not flashing, repeat the procedure.



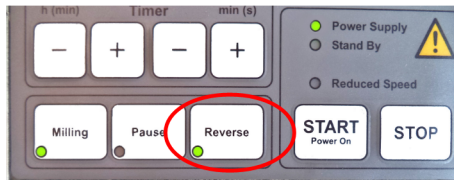
- ➡ To perform changes, press the right "+" button (y) in the TIMER field:  
Time unit, hours and minutes → display: -  
Time unit, minutes and seconds → display: 1 (factory setting)
- ➡ To save and end setup mode, press the STOP button.

## 6.10 Reverse mode

→ Press the REVERSE button. If the REVERSE button is illuminated, reverse mode is activated.

After the selected running time expires, the mill will change its direction of rotation. For this, REPETITIONS has to be at least 1.

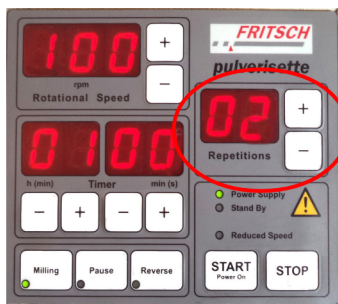
"Reverse" is selected when mixing dry samples or suspensions, for example.



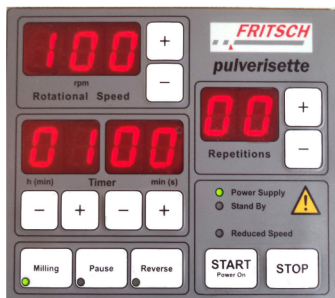
## 6.11 Repetition of grinding / pause cycles

### REPETITIONS control panel area

Press + or - button; select the number of repetitions (0..99). The number of remaining cycles is displayed during operation.



## 6.12 Conducting a grinding operation



- After the conditions specified in ➔ Chapter 6 'Using the device' on page 26 have all been observed, close the hood (3).
- Press the START button on the control panel.
- The hood is locked and the mill starts up.
- The mill turns at the set speed (nominal speed). If the load is too great, for example due to heavy grinding bowls, the speed is reduced (actual speed) so that the machine is not overloaded.

→ If the mill does not start, see ➔ Chapter 9 'Repairs' on page 43



*While in operation, the hood (3) remains locked, even during pause times, and the fan cools the interior.*

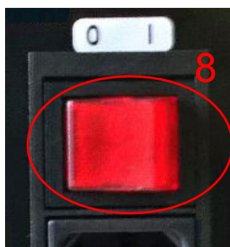
### 6.12.1 Overload

If the mill is overloaded, the speed is reduced and the REDUCED SPEED light flashes.

The mill switches off if the overload continues for too long; see ➔ Chapter 9.1 'Checklist for troubleshooting' on page 43.

## Using the device

### 6.12.2 Switching off



- Press the STOP button on the control panel.
- Shortly after the mill comes to a standstill, the hood is unlocked and can be opened.
- Switch off the device with the main switch (8) on the back of the device.

### 6.13 Cooling the grinding bowl



#### **WARNING**

#### **Burn hazard!**

Grinding bowls can get very hot after long grinding durations. Wear protective gloves for removal after grinding or during the grinding breaks.

- When the hood is open (3), or
- In the programmed pause times with closed (locked) hood and the fan running.

### 6.14 Stand-by

If the mill is not in operation and the hood (3) is open, it switches to the energy-saving stand-by mode after one hour. The STAND-BY lamp lights.

The stand-by function is not possible when the hood is closed.

## 7 Cleaning



### DANGER

#### Mains voltage!

- Before beginning with cleaning work, disconnect the mains plug and protect the device against being unintentionally switched back on!
- Do not allow any liquids to flow into the device.
- Indicate cleaning work with warning signs.
- Put safety equipment back into operation after cleaning work.



### NOTICE

Cool grinding elements made of agate, sintered corundum, zirconium oxide and silicon nitride slowly and carefully.

Do not heat agate elements in a microwave under any circumstances (heating is too fast).

They must never be exposed to thermal shocks as this could cause irreparable damage to the parts → They will burst apart with force.



*When cleaning any part of the device, adhere to the guidelines of the Accident Prevention Regulation (BGV A3) - especially if the device has been set up in a dusty environment or if processing grinding stock that produces dust.*

### 7.1 Grinding elements



### NOTICE

Cool grinding elements made of agate, sintered corundum, zirconium oxide and silicon nitride slowly and carefully.

Do not heat agate elements in a microwave under any circumstances (heating is too fast).

They must never be exposed to thermal shocks as this could cause irreparable damage to the parts → They will burst apart like in an explosion.

- Clean the grinding bowl and grinding balls each time after using them: Clean them, e.g., under running water using a brush and a commercially available cleaning agent.
- Half fill the grinding bowl with grinding balls, sand and water, and run with maximum speed for 2 to 3 minutes (correctly tensioned) in the Planetary Micro Mill.
- Cleaning with an ultrasonic cleaner is permitted.
- For sterilisation in the heat cabinet, only heat the grinding elements up to 100 °C.

## 7.2 Mill

- The Planetary Micro Mill can be wiped down with a damp cloth when it is switched off.



## 8 Maintenance



### DANGER

#### Mains voltage

- Before beginning with maintenance work, unplug the mains plug and protect the device against being unintentionally switched back on again!
- Indicate maintenance work with warning signs.
- Maintenance work may only be performed by specialised personnel.
- Put safety equipment back into operation after maintenance or repair work



### NOTICE

Send all Safe-Lock clamping systems to Fritsch GmbH once a year for inspection. The customer must bear the costs of this maintenance itself.



*We recommend keeping a safety logbook → Chapter 13 'Safety logbook' on page 50, where all work (maintenance, repairs.....) performed on the device is entered.*



*The most important element of maintenance is regular cleaning!*

Functional part	Task	Test	Maintenance interval
Safety lock	Hood lock	Is the closed hood locked in place when the main switch is off?  If this test is failed, do not continue to work until the fault has been rectified.	Before each use
LID LOCK ACTIVATED light on the control panel	Shows that the hood is closed	When the hood is closed the LID LOCK ACTIVATED light should light up green	Once a month
Rotating bearings	Permanent lubrication	Check bearing clearance	Every 2,000 h or annually
Drive motor	Permanent lubrication	Check bearing clearance	Every 4,000 h or annually

## Maintenance

Functional part	Task	Test	Maintenance interval
V-belt	Motor to planetary disc	Check the voltage; remove the housing; the belt must not slacken by more than approx. 10 mm when pushed with your thumb.	Once a year
Fan	Grinding chamber cooling and electronics	Proper function, clean when soiled	Twice a year
Spindle clamping device (5) for the grinding bowl clamping device	Clamping the grinding bowls	Mobility, oil if necessary	Every 1,000 h
Grinding bowl holder	Teflon of the thrust piece, grinding bowl lid seal and rubber insert of the grinding bowl holder	Signs of use; replace if pressed flat and no longer elastic	Every 1,000 h

## 9 Repairs


**DANGER**
**Mains voltage!**

- Before beginning with repair work, unplug the mains plug and protect the device against being unintentionally switched back on.
- Indicate repair work with warning signs.
- Repair work may only be performed by specialised personnel.
- Put safety equipment back into operation after maintenance work.

### 9.1 Checklist for troubleshooting

Fault description	Cause	Remedy
The POWER SUPPLY ready status indicator does not light up	No mains connection	Plug in mains plug
	Main switch	Switch on main switch.
	Device fuse	Check device fuse.
START button is pressed but mill does not start up	Check above if POWER SUPPLY is not lit up	See above
	if LID LOCK ACTIVATED is not lit up	Close the hood firmly
	Pause time active	Wait for end of pause or press STOP
	Fuse blown	Set the fuse to 0.063 A T on the board or 10 A T in the frequency converter
Mill reduces speed	If OVERLOAD lights up: Overload	Press STOP; reduce load or accept the reduced speed
Mill stops running	Switched off due to thermal overload of the drive	Allow device to cool down and select a lower speed
	Drive was blocked	Rectify malfunction in grinding chamber
	Motor's V-belt loose or ripped	Check V-belt, replace if necessary
	Speed sensor is defective	Call customer service.
The hood cannot be opened	The button on the front of the hood was not activated when opening	Open the hood by using the hood handle
	Micro-fuse on the board blown	To check the micro-fuse the housing has to be removed.
Grinding stock escapes	Clamping device loose	Tighten clamping device

## Repairs

Fault description	Cause	Remedy
Grinding stock escapes	Sealing ring defective	Replace the sealing ring
	Soiling on sealing ring, or dirt has penetrated it	Clean or replace if necessary
Runs unevenly with strong vibrations	Inadequate mass balancing	Correct the mass balancing

## 10 Disposal

It is hereby confirmed that FRITSCH has implemented the directive 2002/95/EC of the European Parliament and Council from 27th January 2003 for the limitation of the use of certain dangerous substances in electrical and electronic devices.

FRITSCH has registered the following categories according to the German electrical and electronic equipment act, section 6, paragraph 1, clause 1 and section 17, paragraphs 1 and 2:

**Mills and devices for the preparation of samples have been registered under category 6 for electrical and electronic tools (except for large stationary industrial tools).**

**Analytical devices have been registered under category 9, monitoring and control instruments.**

It has been accepted that FRITSCH is operating only in the business-to-business area. The German registration number for FRITSCH is WEEE reg. no. DE 60198769

### **FRITSCH WEEE coverage**

Since the registration of FRITSCH is classified for bilateral transactions, no legal recycling or disposal process is described. FRITSCH is not obliged to take back used FRITSCH devices.

FRITSCH declares it is prepared to take back used FRITSCH devices for recycling or disposal free of charge whenever a new device is purchased. The used FRITSCH device must be delivered free of charge to a FRITSCH establishment.

In all other cases FRITSCH takes back used FRITSCH devices for recycling or disposal only against payment.

## 11 Guarantee terms

### Guarantee period

As manufacturer, FRITSCH GmbH provides – above and beyond any guarantee claims against the seller – a guaranty valid for the duration of two years from the date of issue of the guarantee certificate supplied with the device.

Within this guarantee period, we shall remedy all deficiencies due to material or manufacturing defects free of charge. Rectification may take the form of either repair or replacement of the device, at our sole discretion. The guarantee may be redeemed in all countries in which this FRITSCH device is sold with our authorisation.

### Conditions for claims against the guarantee

This guarantee is subject to the condition that the device is operated according to the instructions for use / operating manual and its intended use.

Claims against the guarantee must include presentation of the original receipt, stating the date of purchase and name of the dealer, together with the complete device type and serial number.

**For this guarantee to take effect, the answer card entitled "Securing of Guarantee" (enclosed with the device) must be properly filled out and despatched without delay after receipt of the device and be received by us within three weeks or alternatively, ➔ *online registration* must be carried out with the above-mentioned information.**

### Reasons for loss of the guarantee

**The guarantee will not be granted in cases where:**

- Damage has arisen due to normal wear and tear, especially for wear parts, such as: Crushing jaws, support walls, grinding bowls, grinding balls, sieve plates, brush strips, grinding sets, grinding disks, rotors, sieve rings, pin inserts, conversion kits, sieve inserts, bottom sieves, grinding inserts, cutting tools, sieve cassettes, sieve and measuring cell glasses.
- Repairs, adaptations or modifications were made to the device by unauthorized persons or companies.
- The device was not used in a laboratory environment and/or has been used in continuous operation.
- Damage is present due to external factors (lightning, water, fire or similar) or improper handling.
- Damage is present that only insubstantially affects the value or proper functioning of the device.
- The device type or serial number on the device has been changed, deleted, removed or in any other way rendered illegible
- The above-mentioned documents have been changed in any way or rendered illegible.

### Costs not covered by the guarantee

This guarantee excludes any costs for transport, packaging or travel that accrue in the event the product must be sent to us or in the event that one of our specialist technicians is required to come to your site. Any servicing done by persons not authorised by us and any use of parts that are not original FRITSCH accessories and spare parts will void the guarantee.

### Further information about the guarantee

The guarantee period will neither extend nor will a new period of guarantee begin in the event that a claim is placed against the guarantee.

Please provide a detailed description of the type of error or the complaint. If no error description is enclosed, we shall interpret the shipment as an assignment to remedy all recognisable errors or faults, including those not covered by the guarantee. Errors or faults not covered by the guarantee shall in this case be rectified at cost.

We recommend reading the operating manual before contacting us or your dealer, in order to avoid unnecessary inconvenience.

Ownership of defective parts is transferred to us with the delivery of the replacement part; the defective part shall be returned to us at buyer's expense.

**NOTICE**

Please note that in the event that the device must be returned, the device must be shipped in the original Fritsch packaging. Fritsch GmbH denies all liability for any damage due to improper packaging (packaging not from Fritsch).

Any enquiries must include a reference to the serial number imprinted on the type plate.

## 12 Exclusion of liability

Before using the product, be sure to have read and understood this operating manual.

The use of the product requires technical knowledge; only commercial use is permitted.

The product may be used exclusively within the scope of applications set down in this operating manual and within the framework of guidelines put forth in this operating manual and must be subject to regular maintenance. In case of non-compliance, improper use or improper maintenance, the customer assumes full liability for the functional capability of the product and for damage or injury arising from violating these obligations.

The contents of this operating manual are subject in entirety to copyright law. This operating manual and its contents may not be copied, further distributed or stored in any form, in part or in whole, without the prior written consent of Fritsch.

This operating manual has been prepared to the best of our knowledge and checked for accuracy at the time of printing. FRITSCH GMBH assumes no guarantee or liability whatsoever for the accuracy or completeness of the contents of this operating manual, including but not limited to the implied warranties of merchantability and fitness for a particular purpose, unless liability is expressly prescribed by applicable laws or jurisprudence.

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## Safety logbook

### 13 Safety logbook

Date	Maintenance / Repair	Name	Signature

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Milling and Sizing

Industriestraße 8

D - 55743 Idar-Oberstein

Telephone: +49 6784 70-0

Email: [info@fritsch.de](mailto:info@fritsch.de)

Internet: [www.fritsch.de](http://www.fritsch.de)

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