

Operator's Manual

Laboratory - Planetary - Mill „ pulverisette 5 “



Fritsch GmbH

Manufacturers of Laboratory Instruments

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(Diese Betriebsanleitung ist auch in Deutsch erhältlich)

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On June 24, 1995, Fritsch GmbH was certified by the TÜV-Zertifizierungsgemeinschaft e.V.



An audit was conducted (Report No. Q94/107) to verify that Fritsch meets the requirements of

DIN ISO 9001 / EN 29001

(Certificate No. 71 100 4 008)

Safety Instructions

- Do not remove information signs.
- Allow only trained personnel to perform maintenance.
- Replace covers after maintenance.
- Do not permanently disable safety devices.
- Check safety device regularly.
- "Out of Service" ⇒ Turn off master switch.
- Allow only trained specialists to work on the mill.
- Do not open planetary mill until it is completely stopped.
- Do not put your hands into mill while it is running.
- Always wear safety glasses when working.
- Set up the planetary mill indoors only.
- Ambient temperature 0 to 40°C
- Set it up on a sturdy work table.
- Dimensions: 1100 x 830 x 830 mm (H x W x D)
Weight (net): approx. 170 kg (4 carrying handles included)



- **Danger: Electricity:**
- The instrument contains a frequency converter with electricity in the low voltage range 230 Vrms
- **Grinding bowls can become very hot:**
- **Exercise caution while loosening the bowl holder.**
Pressure in bowl may be very high!
- **Exercise caution when working with combustible or poisonous materials.**



Always comply with valid safety regulations (MAC values) when handling samples or carrier liquids which are dangerous to your health and, where applicable, set up the planetary mill in a well-ventilated safety zone.

- Be careful not to be pinch yourself on lid cover.
- Be careful not to be pinch yourself on bowl holder.



Read and comply with Operator's Manual



Operators' Manual

Laboratory - Planetary - Mill

„ pulverisette 5 “

The laboratory planetary mill "pulverisette 5" can be used universally for high-speed grinding of liquid or solid inorganic and organic samples for analysis, quality control or material testing.

In the case of synthesis, the "pulverisette 5" is used to mix and homogenise dry samples, emulsions or pastes.

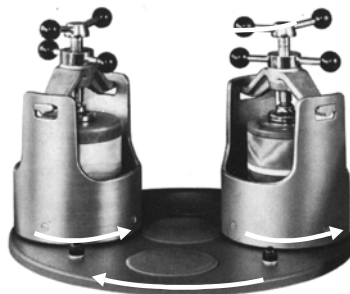
Approx. initial sample size for hard material is max. 10 mm

Approx. quantity of sample to be ground is max. 800 ml

(in four 500 ml grinding bowls each
with 10 grinding balls 30 mm in diameter)

Average final fineness $d_{50} < 1\mu\text{m}$ (depending on the materials).

1 The working method

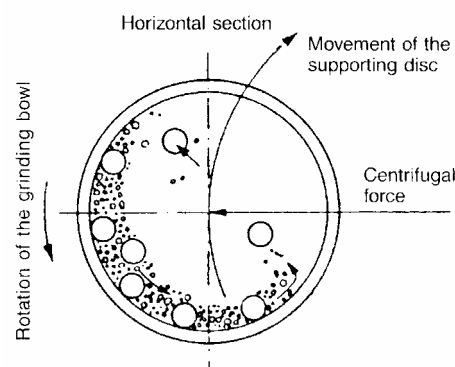


The material to be ground is crushed and torn apart by grinding balls in 2 or 4 grinding bowls. The centrifugal forces created by the rotation of the grinding bowls around their own axis and the rotating supporting disc are applied to the grinding bowl charge of material and grinding balls.

Since the directions of rotation of grinding bowls and supporting disc are opposed, the centrifugal forces are alternately synchronised and opposite. Thus friction results from the grinding balls and the material being ground alternately rolling on the inner wall of the bowl, and impact results when they are lifted and thrown across the bowl to strike the opposite wall. The impact is intensified by the grinding balls striking one another.

The impact energy of the grinding balls in the normal direction attains values up to 40 times higher than gravitational acceleration.

Loss-free comminution is guaranteed by a hermetic seal between grinding bowls and lid - even if suspensions are being ground.



2 Operating safety

The planetary mill is secured with a personnel safety switch.

This switch locks the lid during operation and prevents the mill from starting when the lid is open.

⇒ The planetary mill cannot be opened during operation.

⇒ The planetary mill will not start when the lid is open.

Locking the lid:

When power to the mill is switched off, the lid is locked.

To open the lid, connect the mill to the AC power and turn on the master switch.

In emergencies only:

Using a tool to open the mill when the power is switched off:

After removing the cover on the front left of the housing, turn socket hex key 2.5 mm

⇒ 90° in clockwise direction to open the personnel safety switch.

After opening the lid, reset the safety switch with a ⇒ 90° turn in counter clockwise direction and put the cover back on.

After the **"STOP"** key is pressed, the mill decelerates: standstill in seconds.

⇒ Lid can be opened .

Stop

In case of power failure during operation
(or if mill turned off at the master switch)

⇒ Lid remains locked.

Recovery AC power opens the lid lock.

⇒ Mill is secured against restarting.

⇒ Press the **" START "** key : mill starts up.

Start

Turning off the master switch results in a bipolar separation of the entire mill from the AC power.

Turn the master switch off whenever the mill is "Out of service" for a protracted period.

⇒ night-time shutoff

For overload protection the speed of the mill is automatically reduced by an electronic monitoring of its current and speed. Simultaneously a red warning lamp „**Overload**“ lights up.

If with long term working the maximum permissible temperature of the motor is reached an automatic thermal sensor switches off the power. After cooling down the motor can be switched on by pressing the **Start**-key.

3 Setting up the planetary mill

Set up the planetary mill indoors on a flat, sturdy surface.
It is not necessary to fasten it in place.

The room temperature must be between 0 and 40°C.

Make certain that the mill is easy to access.

Keep ventilation grille on back unobstructed for air discharge.

4 Electrical connections

120 / 240 V 10% VAC single-phase with PE conductor, 50/60Hz,
2300 watt max. power consumption (under normal load significantly lower)

100 - 120 volt fuse: 25 A; 200 - 240 volt fuse: 16 A.

Turn on the master switch (black knob-operated switch) on the left-hand side. The green Ready lamp (Power Supply) will light up.

4.1 Adapting the planetary mill to the AC power

Only a trained specialist is permitted to switch from 240 to 120 VAC power voltage and/or to change the connecting cable (see "Technical documentation").

4.2 Electrical Fuses

- Mains fuse 16 A
- Automatic circuit-breaker for short-circuit protection in instrument 16 A
(to left of master switch; to reset: push in the red button).
- Overload fuse 10 A
(accessible after unscrewing the left-hand cover grille on the PC board of the frequency converter. Replacement: finewire fuse, slow-blow fuse 10; 5 x 20)
- Thermal motor protecting switch for drive motor
(resets itself after motor cools down).

4.3 Drive motor and speed control

Driven by 3-phase AC motor.

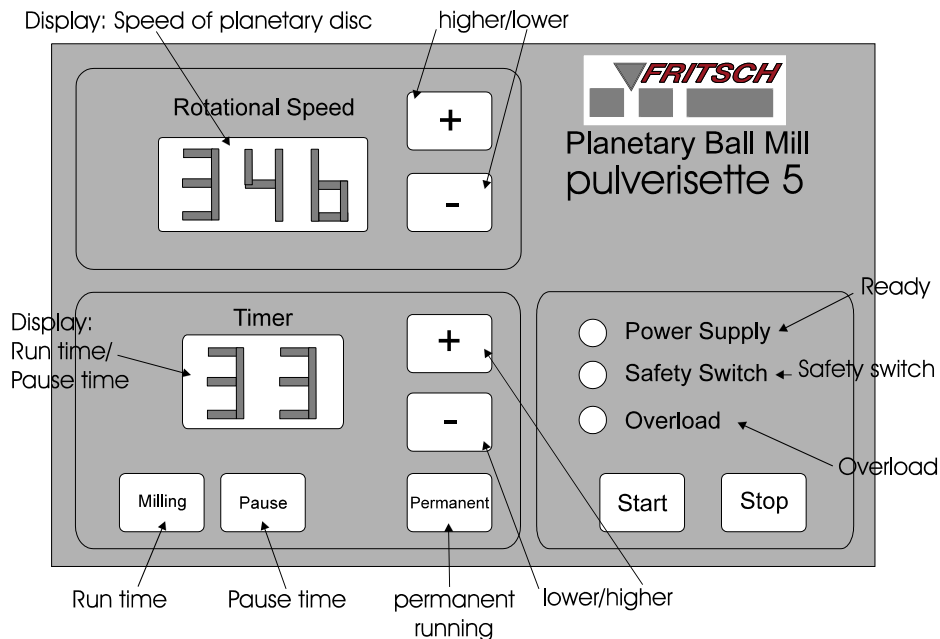
Speed control by frequency converter:

transforms single-phase voltage into 3-phase 220 V 3-phase AC
with variable frequency. Pressing the "+" key (or "-" key) sets the speed of the
planetary disc from the control panel and thus the desired speed is forced on the
drive motor via the associated frequency.



5 Operating the Planetary Mill

Membrane keyboard



Power Supply: **Green** lamp on: master switch "ON"

Lid Lock Activated : **Green** lamp on : "START" pressed.

Safety switch closed

Flashing red : "START" pressed

Lid not reliably closed

Safety switch **not** locked

Overload: **Steady red:** Mill overloaded, speed limited

Milling: Key pressed: flashing **red:** display: run time, time adjustable

Pause: Key pressed: flashing **red:** Display: Pause time, time adjustable

Start: Key pressed: mill starts up, if "Lid Lock Activated" is green.

Stop: Key pressed: mill decelerates,
lid released when mill comes to a complete stop.



6 Selecting milling bowls and milling balls

Take into account: Hardness of the material to be ground
Permissible contamination of sample by abrasion from grinding elements.

Material (bowls and balls)	g/cm ³	Density	Abrasive
Agate	(99.9% SiO ₂)	2.6	good
Sintered corundum	(99.7% Al ₂ O ₃)	3.7	conditionally good
Sintered corundum 2	(85% Al ₂ O ₃)	3.5	satisfactory
Zircon oxide	(97% ZrO ₂)	5.7	very good
Polyamid bowls		1.4	acceptable
Teflon balls with steel core		5.0	acceptable
Chromium nickel steel stainless		7.8	conditionally good
Chromium steel hardened		7.8	good
Tungsten carbide (93%WC+6% Co)		4.7	very good
„Syalon“ silicon nitride (90% Si ₃ N ₄)		3.2	extremely good

(High density ~ **high impact energy**)

6.1 Instructions for assembling the grinding set

Normal case:

Select grinding bowls and grinding balls made of same material
(only one type of contamination by abrasion)

Special case:

To shorten the grinding period, select grinding balls of a higher density.
(e.g. tungsten carbide or zircon oxide balls, but do not use them in agate grinding bowls).

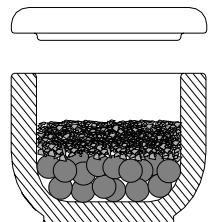
Determine through experiment: Size of grinding bowls and grinding balls

- Size of grinding bowls:**

Fill only $\frac{2}{3}$ of the bowl volume with grinding material and grinding balls.
Leave $\frac{1}{3}$ of the bowl volume free as space to develop centrifugal force.

- Size of grinding balls (rule):**

A relatively large number of balls shortens the grinding time and the ground material has a tighter range of grain sizes.



- Recommendation:**

Normal case:

Hard samples with relatively large pieces	30 or 40 mm balls
Medium size pieces (2-5 mm)	30 mm balls
Fine material (0.5 mm)	20 mm balls
To homogenise dry or liquid samples	10 mm balls
To homogenise viscous samples	20 mm balls

- Recommended **number of balls** per grinding bowl (maximum):

Bowls		500 ml	250 ml	80 ml
Balls	10 mm	100	50	30
or	20 mm	20	15	5
or	30 mm	10	6	
or	40 mm	4		

- Special case:**

Grinding relatively large pieces down to a few µm:

Mix balls of different diameters, 20 mm and 30 mm Ø:

Large balls quickly comminute the coarse material.

Small balls accelerate the grinding of the fine material.

7 Putting the mill into operation

Important instruction:

Symmetrical charging:

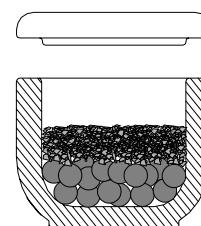
To balance the mill, always clamp a corresponding heavy grinding bowl with cover and gasket into the opposed pot fastener (use empty bowl if necessary).

7.1 Filling the grinding bowls

- Put grinding balls into the empty bowl.
- Pour material to be ground onto the balls.

Reminder: Fill grinding bowls only $\frac{2}{3}$ full (see 6.1).

- Gasket and sealing surfaces must be clean.
- Put gasket and cover in place.
- Place grinding bowl in pot fastener on cork gasket.



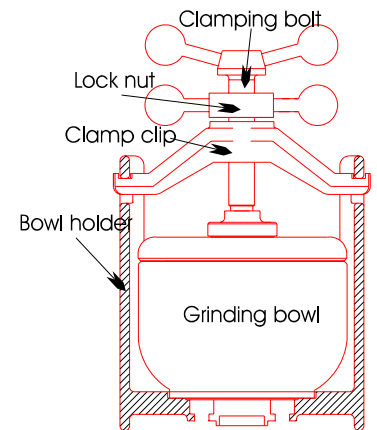
7.2 Clamping in the grinding set

- Loosen the lock nut.
- Hook the clamp clip into the bowl holder.
- Screw the clamping bolt down tight.
- Tighten the lock nut.,

Check: Make certain clamping bolt cannot be turned by hand

Instructions:

Clamp 80 ml grinding bowl in place with a reducer (Order No.53.050.00) on top of it, or place two 80 ml grinding bowls on top of one another.



7.3 Achievable Final fineness

Achievable: Dry grinding, average final fineness approx. 20 μm .

Wet grinding, average final fineness approx. 1 μm .
(Depending on the materials)

Influenced by:

- **Run time** (duration of grinding)
Grinding longer increases the percentage of fine particles
- **Speed**
Higher speed increases the percentage of fine particles.
- **Number and size of ball**
Pregrind coarse, hard material with large balls:
⇒ percentage of fine particles low
Many small balls
⇒ increases the percentage of fine particles
when run time extended.
- **Density of the balls (type of material)**
Balls of higher mass (density) **accelerate** the grinding process.
- **Size of grinding bowls**
Large bowls increase the grinding process (higher centrifugal forces).

Attention: Stickiness of material during dry grinding

When the particles are less than approx. 20 μm in diameter, surface forces pre-dominate and the material to be ground begins to "stick".

Further reduction in size is only possible by grinding in suspension. In some cases, further improvement is possible by adding tenside.

**Lightly combustible liquids as for example
ketone or benzine with boiling point < 120°C
must not be used.**



7.4 Activating the planetary mill

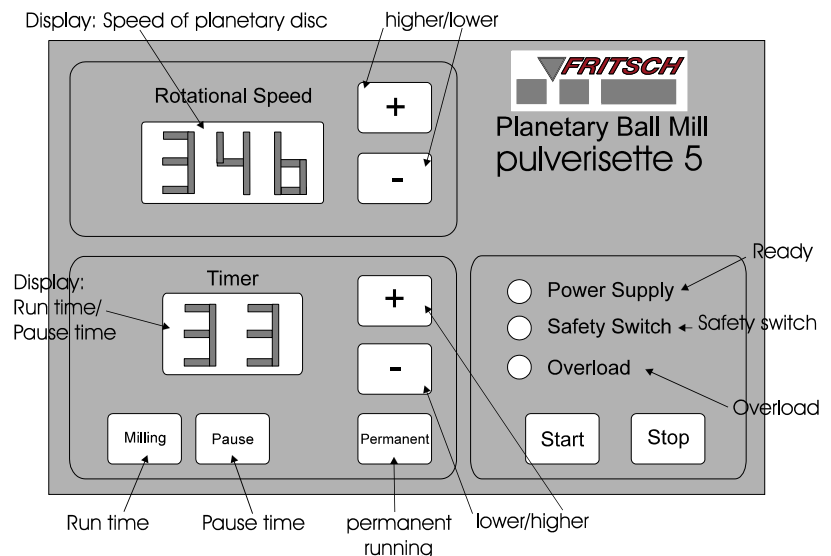
Instructions:

- Before closing lid, make certain that grinding sets and locking set are firmly seated.
- Close lid securely - safety switch locks home.
- Pay attention to **heating of material** to be ground:
in case of relatively long run time, possible stop to cool down!

(1) Setting the speed: Press „+“ or „-“ key, (speed in RPM),

(2) Setting the run time:
Press „Milling“: key will flash,
Press „+“ or „-“ key: set run time in minutes.

*If pause time required
for cooling:*



(3) Setting the pause time: Press „Pause“ key: key will flash,
Press „-“ or „+“ key, set pause time in minutes.

(4) Setting continuous operation: Press „Permanent“ key.
Run time and pause time settings have no effect.

⇒ **Maximum grinding period: 1 hour.**

⇒ Prior to restart: Allow to cool for 1/2 hour with lid open and fan running.

(5) Grinding: Press „Start“ key, **Stop:** Press „Stop“ key

When operation with external timer, allow to cool for at least one hour.

Information: If the mill is overloaded (large bowls, many large balls, tough materials) the speed is automatically reduced in order to protect the drive motor; simultaneously the red warning lamp „**Overload**“ lights up. The reduced speed is indicated on the display.

8 Maintenance:

Attention: Disconnect the mains plug before beginning any maintenance work !!
Secure instrument against unintentional operation.
(Eventual installation of a warning sign.)

Functional component	Function	Test	Maintenance interval
Safety devices	Ready display	green lamp lights up	Before each use
Safety switch	Lock	Lid secure	Before each use
Safety switch	In operation	green lamp lights up	Before each use
Safety switch	Malfunction warning	Flashing red lamp	1 x month
Rotating bearings	Permanent lubrication	Bearing clearance	After each 2.000 hrs
Drive motor	Permanent lubrication	Bearing clearance	After each 4.000 hrs
V-belts	Motor planetary disc	Voltage	1 x annually
V-belts	Planetary disc	Voltage	1 x annually
Fans	Cooling of grinding chamber	Function dirt	2 x annually
Grinding bowl holder	Toggle-type fastener	Ease to move	After each 1.000 hrs
Grinding bowl holder	Cork gasket	Traces of wear	After each 1.000 hrs
Grinding bowl holder	Thrust piece: rubber gasket	Traces of wear	After each 1.000 hrs

Cleaning the grinding elements

Clean grinding elements after each use:

- ⇒ Hold grinding elements under running water and brush with conventional scouring materials.
- ⇒ Cleaning in ultrasonic bath is permitted.
- ⇒ In case of sterilisation in the drying chamber, heat only to 250°C.
- ⇒ Do not heat grinding elements made of agate over 110°C; cool slowly and carefully.
- ⇒ Simple cleaning by grinding quartz sand and water for a short time.

9 Warranty

The warranty card enclosed with this shipment must be returned to the supplier completely filled out in order for the warranty to take effect.

Fritsch GmbH of Idar-Oberstein and our "Application technology laboratory" and/or our agent in your country will be glad to advise and aid you.

In order to answer any questions you may have, we will require the serial number stamped on the nameplate.

10 Checklist for troubleshooting

Malfunction	Possible cause	Remedy
Ready indicator does not light		
1	Not connected to mains	Insert mains plug
2	Master switch	Turn on master switch
3	Mains cut-off	Push in cut-off
4	Overload fuse (PCB)	Replace fuse
Press „Start“ key Mill does not start		
1	If red „Lid Lock“ lamp flashes, open safety switch	Close lid tightly
2	If green „Lid Lock“ is lit, overload fuse on PCB is faulty	Replace the fuse
3	Pause time active	Press „Milling“ key
Mill automatically reduces speed		
	If „Overload“ lamp is red: overload	Press „Stop“ key select lower speed Press „Start“ key or accept speed set automatically
Mill stops running		
1	Overload Overload fuse on PCB shut mill off	Exchange fuse, lower speed, press „Start“ key
2	Overload Thermal fuse in motor has shut mill off	Let motor cool down, choose lower speed, press „Start“ key
Mill rotates at only 50 RPM		
1	Speed sensor faulty	Call Customer Service
2	V-belt loose/faulty	Check V-belt
Ground material escapes		
1	Clamping bolt loose	Tighten clamping
2	Gasket faulty	Exchange gasket
Irregular running with strong vibration		
	Bowls not balanced	Arrange the bowls symmetrically (equal weights in opposed positions)

11 Examples:

The following examples from the Fritsch „Application Technology Laboratory“ provide information about the selection of milling sets, favourable grinding durations and the final fineness attained with the various grinding samples:

Material	Grinding bowl	Grinding balls	Grinding time	Results
Quantity	Material	Material		
Edge length	Volume	Qty x dia mm		

Ruby (stone)				
140 g	Cr-Ni-steel	Cr-Ni-Steel	3 min	100%
12 mm	250	6 x 30		<250 μ m
Titanium dioxid TiO₂ (Dry- and Wet grinding in Water)				
40 g	Cr-Ni-Steel	Cr-Ni-Steel	30 min	100%
2 mm	250	6 x 30		< 40 μ m
Titanium dioxyd TiO₂ (Dry- and Wet grinding in Water)				
40g/50 ml	Cr-Ni-Steel	Cr-Ni-Steel	60 min	100%
2 mm	250	6 x 30		< 10 μ m
Carbon (Dry- and Wet grinding in Water)				
5 g	zir.oxid	Zir.oxid	120 min	100%
0.5 mm	80	5 x 20		< 15 μ m
Aluminium oxide/Silicon oxide				
100 g	WC + Co	WC + Co	90 min	90%
0.1 mm	250	15 x 20		< 20 μ m
Ferrovandium				
70 g	WC + Co	WC + Co	20 min	70%
3 mm	250	5 x 30		<100 μ m
Glass				
50 g	agate	Agate	15 min	100%
4 mm	250	15 x 20		< 90 μ m
Silicon carbide (Dry- and Wet grinding in Water)				
15 g	WC + Co	WC + Co	30 min	100%
3 mm	80	5 x 20		<150 μ m

Material	Grinding bowl	Grinding balls	Grinding time	Results
Quantity	Material	Material		

Edge length	Volume	Qnty x dia mm		
Silicon carbide (Dry- and Wet grinding in Water)				
15g/5 ml	WC + Co	WC + Co	45 min	100%
3 mm	80	5 x 20		< 71 μ m
Raw-phosphate				
40 g	Cr-Steel	Cr-Steel	2 min	100%
3 mm	250	15 x 20		<250 μ m
Manganous oxide MnO ₂ (Dry- and Wet grinding in Water)				
50g/40 ml	WC + Co	WC + Co	60 min	100%
0.1 mm	250	15 x 20		< 20 μ m
Sewage sludge				
180 g	Al ₂ O ₃	Al ₂ O ₃	30 min	100%
8 mm	500	10 x 30		<250 μ m
Activated charcoal (Wet grinding in Water)				
150 ml	Cr-Ni-Steel	Cr-Ni-Steel	30 min	100%
0.025 mm	250	15 x 20		< 5 μ m
Gypsum				
300 g	Cr-Steel	Cr-Steel	20 min	100%
10 mm	500	10 x 30		<200 μ m
Protein				
50 g	Sint. cor.1	Sint. cor.1	90 min	90%
20 mm	250	6 x 30		< 50 μ m
Grain (barley)				
100 g	Sint. cor.1	Sint. cor.1	20 min	100%
3 mm	500	3 x 40		<150 μ m
Dough				
100 g	Sint. cor.1	Sint. cor.1	3 min	100%
5 mm	500	10 x 30		<250 μ m
Sugar (Wet grinding in alcohol)				
200 g	Agate	Agate	45 min	100%
1 mm	500	10 x 30		< 10 μ m