

Manual Operation Manual Mixer Mill CryoMill





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1 Notes on the Operating Manual

This operating manual is a technical guide on how to operate the device safely and it contains all the information required for the areas specified in the table of contents. This technical documentation is a reference and instruction manual. The individual chapters are complete in themselves.

Familiarity (of the respective target groups defined according to area) with the relevant chapters is a precondition for the safe and appropriate use of the device.

This operating manual does not contain any repair instructions. If faults arise or repairs are necessary, please contact your supplier or get in touch with Retsch GmbH directly.

Application technology information relating to samples to be processed is not included but can be read on the Internet on the respective device's page at www.retsch.com.

Changes

Subject to technical changes.

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Infringements will result in damage compensation liability.



1.1 **Explanations of the safety warnings**

In this Operating Manual we give you the following safety warnings

Serious injury may result from failing to heed these safety warnings. We give you the following warnings and corresponding content.



WARNING

Type of danger / personal injury

Source of danger

- Possible consequences if the dangers are not observed.
- Instructions on how the dangers are to be avoided.

We also use the following signal word box in the text or in the instructions on action to be taken:



⚠ WARNING

Moderate or mild injury may result from failing to heed these safety warnings. We give you the following warnings and corresponding content.



CAUTION

Type of danger / personal injury

Source of danger

- Possible consequences if the dangers are not observed.
- Instructions on how the dangers are to be avoided.

We also use the following signal word box in the text or in the instructions on action to be taken:



CAUTION

In the event of possible property damage we inform you with the word "Instructions" and the corresponding content.

NOTICE

Nature of the property damage

Source of property damage

- Possible consequences if the instructions are not observed.
- Instructions on how the dangers are to be avoided.

We also use the following signal word in the text or in the instructions on action to be taken:

NOTICE



1.2 General safety instructions



CAUTION

Read the Operating Manual

Non-observance of these operating instructions

- The non-observance of these operating instructions can result in personal injuries.
- · Read the operating manual before using the device.
- We use the adjacent symbol to draw attention to the necessity of knowing the contents of this operating manual.



Target group: All persons concerned with the machine in any form

This machine is a modern, high performance product from Retsch GmbH and complies with the state of the art. Operational safety is given if the machine is handled for the intended purpose and attention is given to this technical documentation.

You, as the owner/managing operator of the machine, must ensure that the people entrusted with working on the machine:

- have noted and understood all the regulations regarding safety,
- are familiar before starting work with all the operating instructions and specifications for the target group relevant for them,
- have easy access always to the technical documentation for this machine,
- and that new personnel before starting work on the machine are familiarised with the safe handling of the machine and its use for its intended purpose, either by verbal instructions from a competent person and/or by means of this technical documentation.

Improper operation can result in personal injuries and material damage. You are responsible for your own safety and that of your employees.

Make sure that no unauthorised person has access to the machine.



CAUTION

Changes to the machine

- Changes to the machine may lead to personal injury.
- Do not make any change to the machine and use spare parts and accessories that have been approved by Retsch exclusively.

NOTICE

Changes to the machine

- The conformity declared by Retsch with the European Directives will lose its validity.
- You lose all warranty claims.
- Do not make any change to the machine and use spare parts and accessories that have been approved by Retsch exclusively.



1.3 Repairs

This operating manual does not contain any repair instructions. For your own safety, repairs may only be carried out by Retsch GmbH or an authorized representative or by Retsch service engineers.

Your supplier Retsch GmbH directly Your Service Address:

The Retsch representative in your country

In that case please inform:



2 Confirmation

This operating manual contains essential instructions for operating and maintaining the device which must be strictly observed. It is essential that they be read by the operator and by the qualified staff responsible for the device before the device is commissioned. This operating manual must be available and accessible at the place of use at all times.

The user of the device herewith confirms to the managing operator (owner) that (s)he has received sufficient instructions about the operation and maintenance of the system. The user has received the operating manual, has read and taken note of its contents and consequently has all the information required for safe operation and is sufficiently familiar with the device.

As the owner/managing operator you should for your own protection have your employees confirm that they have received the instructions about the operation of the machine.

	d and taken note of the contents of all chapters in this operating well as all safety instructions and warnings.	
User		
Surname,	first name (block letters)	
Position in	the company	
	, and company	
Signature		
Service to	echnician or operator	
Surname,	first name (block letters)	
Position in	the company	
Place, dat	e and signature	



3 Technical data

3.1 Protective equipment

The grinding chamber of the CryoMill laboratory mill is enclosed by a strong hood. It is only possible to start the machine if the hood is closed.

3.2 Emissions

Noise characteristic values:

The noise characteristic values are also influenced by the properties of the sample material.

Example 1:

Intensity of sound $L_{WA} = 71.4 \text{ dB(A)}$

Workplace-related

emission value L_{pAeq} = 61 dB(A)

Operating conditions:

Container:	1 steel grinding jar 50ml
Grinding organ:	1 steel ball 25mm
Feed material:	Quartz crushed pebbles approx. 4.0 – 6.0mm
Feed quantity:	8ml
Frequency:	25 Hz

Example 2:

Intensity of sound $L_{WA} = 76 \text{ dB(A)}$

Workplace-related

emmission value L_{pAeq} = 65 dB(A)

Operating conditions:

Container: 4 steel grinding jars 5ml	
Grinding organ:	2 tungsten carbide balls 8mm
Feed material:	Quartz crushed pebbles approx. 1.0 – 1.5mm
Feed quantity:	1.5ml
Frequency:	25 Hz





CAUTION

Damage to hearing

Depending on the type of material, the number of balls used, the grinding frequency set and the duration of grinding a high noise level may arise.



- Excessive noise in terms of level and duration can cause impairments or lasting damage to hearing.
- Provide suitable noise protection measures or wear hearing protection.

3.3 Degree of protection

IP30

3.4 Rated power

160 Watt

3.5 Dimensions and weight

Height: 370 mm to approx. 630 mm with hood opened

Width: 385 mm Depth: 570 mm

Weight: approx. 46 kg without grinding jar

3.6 Required floor space

Floor space: 500 mm x 650 mm



4 Transport, scope of delivery, installation

4.1 Packaging

Packaging is adjusted to the transportation route. It complies with the generally applicable packaging guidelines.

NOTICE

Storage of packaging

- In the event of a complaint or return, your warranty claims may be endangered if the packaging is inadequate or the machine has not been secured correctly.
- Please keep the packaging for the duration of the warranty period.

4.2 Transport

NOTICE

Transport

- Mechanical or electronic components may be damaged.
- The machine may not be knocked, shaken or thrown during transport.

4.3 Temperature fluctuations and condensed water

NOTICE

Temperature fluctuations

The machine may be subject to strong temperature fluctuations during transport (e.g. aircraft transport)

- The resultant condensed water may damage electronic components.
- Protect the machine from condensed water.

4.4 Conditions for the place of installation

NOTICE

Ambient temperature

- Electronic and mechanical components may be damaged and the performance data alter to an unknown extent.
- Do not exceed or fall below the permitted temperature range of the machine (5°C to 40°C / ambient temperature).

Atmospheric humidity:

Maximum relative humidity 80% at temperatures up to 31°C, decreasing linearly up to 50% relative humidity at 40°C



NOTICE

Atmospheric humidity

- Electronic and mechanical components may be damaged and the performance data alter to an unknown extent.
- Do not exceed the admissible range for atmospheric humidity.

4.5 Installation of the machine

Installation height: maximum 2000 m above sea level

NOTICE

Property damage

A very cold vapour plume escapes from the nitrogen gas outlet.

- The vapour plume can greatly cool objects. This is why the humidity in the air condenses.
- The area above the nitrogen gas outlet (B) up to the ceiling must be kept free.

NOTE

Installation

- Depending on the operating status of the mill, there may be slight vibrations.
- Place the mill on an even, flat and balanced supporting surface only.
 The supporting surface must be stable and must not vibrate.

NOTICE

Installation of the machine

- It must be possible to disconnet the machine from the mains at any time.
- Install the machine such that the connection for the mains cable is easily accessible.

4.6 Removing the transport safeguard

NOTICE

Transport safeguard

- Components may be damaged.
- Operate the machine only without the transport safeguard or transport the machine only with transport safeguard.



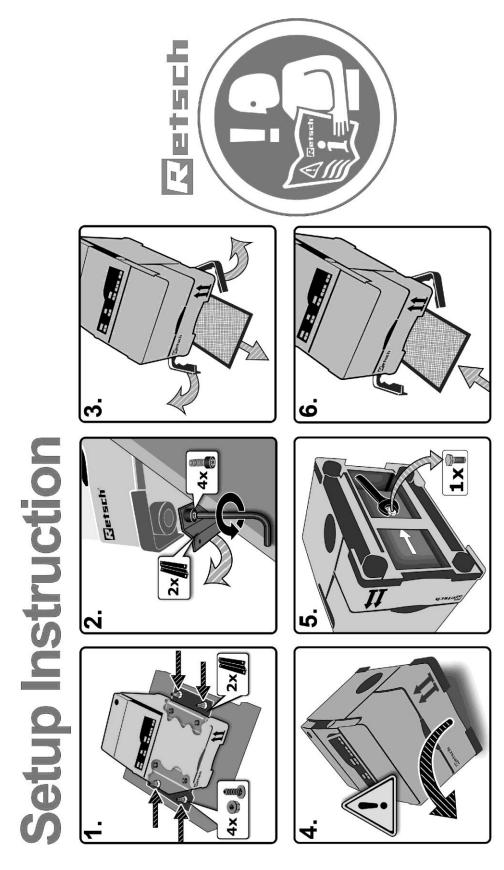


Abb. 1: Setup Instruction



A transport safeguard is located on the underside of the machine shown by an arrow

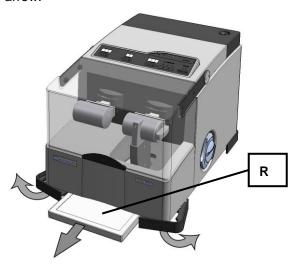


Fig. 2: Pulling out collecting filter for condensed water

- Push out the two front guards to the side.
- Pull the collecting filter for condensed water (R) completely from the machine.
- Tip the machine on its side.

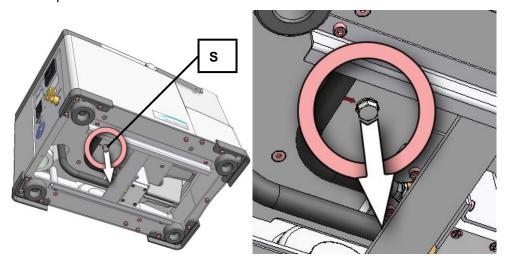


Fig. 3: Removing the transport safeguard

- Unscrew the screw and remove (S).
- Replace the collecting filter for condensed water (R).

Keep the transport safeguard for transportation at a later date!

4.7 Electrical connection

The voltage required and the frequency of the machine are provided on the type plate.

- Ensure that the values agree with the available power supply system.
- Connect the machine to the power supply system using the connecting cable supplied.





An external fusing at the connection of the mains cable to the power supply must be provided in accordance with the conditions of the place of installation.

The external fuse must have at least a T4A rating.

NOTICE

Electrical connection

- Mechanical or electronic components may be damaged.
- Please observe the information on the type plate.



5 Operating the machine

5.1 Use of the machine for the intended purpose

Target group: owners, operators **Machine type designation**: CryoMill

This machine is a laboratory device and is suitable for the particularly gentle grinding and homogenization of very temperature-sensitive, soft, fibrous, hard and brittle materials in dry and wet state.

The CryoMill is designed exclusively for special screw-top jars (not standard grinding jars) with steel casing and serves the purpose of fast pulverization of up to four samples simultaneously depending on size.

Available grinding jar volumes: 50ml; 35ml; 25ml and 4 x 5ml including adapter.

The closed grinding system guarantees a complete recovery of samples. In view of the extremely short grinding time and the high final fineness of the sample material, the CryoMill is also ideally suited to sample preparation for all spectral analyses.

Depending on grinding time and the specific properties of the sample material, final finenesses of up to $5 \, \mu m$ can be achieved.

The optimum grinding jar filling is usually 1/3 of the grinding jar volume. Exceptions to this are voluminous materials such as wool, leaves, grasses and similar. A filling level of 70 - 80% is necessary here.



5.2 Operating elements and displays

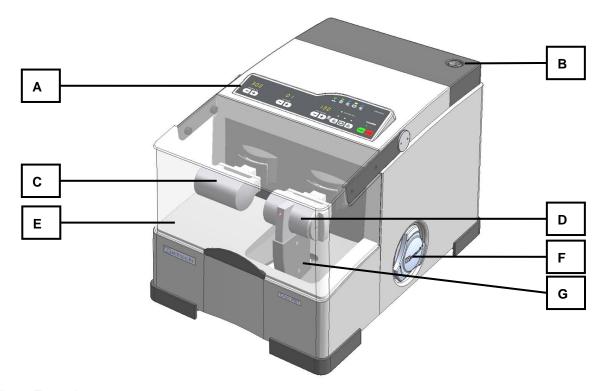


Fig. 4: Front view

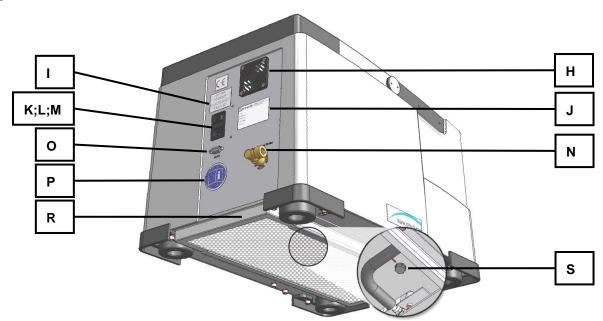


Fig. 5: Rear view



5.3 Overview table of the machine parts

Element	Description	Function
Α	Display and operating unit: for explanations see below	Time preselection, frequency preselection and start / stop of the machine
В	Nitrogen gas outlet	Outlet opening for gaseous nitrogen
С	Balance weight, left	Counterweight to cooling casing with grinding jar
D	Cooling casing with grinding jar	Forms the cooling system together with the special grinding jar
E	Hood	Closes the grinding chamber
F	Easy opener	Can be pushed on to the grinding jar lid to easily remove the grinding jar
G	Cooling agent feed line and cooling agent drain line	Feeds the liquid nitrogen from the rear cooling agent connection to the grinding jar
н	Fan	Ventilates the motor and the inner chamber of the mill
1	Sign: Caution! Unplug mains plug	Safety notice
J	Type plate	Information on the machine and installed loads
К	Switch ON / OFF	To switch the MM 400 on and off
L	Fuse drawer	To take two visible type fuses
М	Machine socket	Connection for mains cable of the machine
N	Cooling agent connection	Connection for liquid nitrogen
0	Interface RS232	Permits the operating software to be updated
Р	Plate: observe Operating Manual	Safety notice
R	Collecting filter for condensed water	Collects the condensed water from cooling system and permits it to evaporate
s	Transport safeguard (lower side)	Prevents damage of components during transportation



5.4 Operating elements and displays

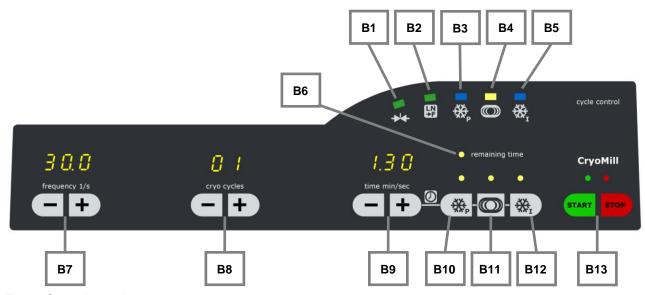


Fig. 6: Operating unit

Button	Description	Function
B1	LED on – valve open	Shows whether the internal solenoid valve of the liquid nitrogen feed line is opened or closed
	LED off – valve closed	Thirrogen reed line is opened or closed
B2	LED liquid nitrogen flowing	Shows that liquid nitrogen is flowing into the machine
В3	LED	Precooling in progress
В4	LED	Grinding in progress
B5	LED	Intermediate cooling in progress
В6	LED remaining total grinding time	Lights up during grinding and signalises that the time min/sec display shows the remaining total grinding time
		Flashes at the end of the running time if an error arose during the grinding time, e.g. interruption to the liquid nitrogen feed line
В7	Frequency – minus button	In setting mode and in operating mode: reduction of the set frequency by 0.1 oscillations per second. Continuous pressing activates the rapid digit display.
	Frequency – plus button	In setting mode and in operating mode: increase in the set frequency by 0.1 oscillations per second. Continuous pressing activates the rapid digit display.
В8	Cooling cycle - minus button	Reduces number of total cycles by 1
	Cooling cycle – plus button	Increases number of total cycles by 1
В9	Time – minus button	In setting mode and in operating mode: reduction of the set: recooling time



		 grinding time intermediate cooling time by one second. Continuous pressing activates the rapid digit display.
	Time – plus button	In setting mode and in operating mode: increase of the set: precooling time grinding time intermediate cooling time by one second. Continuous pressing activates the rapid digit display.
B10	Setting of precooling	Preselection button to set the precooling time
B11	Setting of grinding	Preselection button to set the grinding time
B12	Setting of intermediate cooling	Preselection button to set the intermediate cooling time
B13	Start – button	Starts the grinding operation
	Green LED	Shows grinding operation
	Stop – button	Interrupts or ends the grinding operation,
	Red LED and ON	places the machine in stand-by mode



5.5 Inserting the grinding jar



CAUTION

Danger of injury and frostbite

Escape of liquid nitrogen

- Liquid nitrogen has a temperature of –196 °C and may cause injuries similar to burns on skin or eye contact or frostbite.
- Never switch the machine on without tightly closed cooling casing.

5.5.1 Inserting and removing CryoMill special grinding jar



CAUTION

Clogged piping

Formation of ice

 Ice or foreign matter can clog the pipe and subsequently cause malfunctioning.

Keep the cooling system and the supply pipe dry and free of foreign matter.

- Do not insert the grinding jar if it is damp or covered with frost.
- Use the cooling jacket sealing plug (KA) if you are not going to use the device for a long time.
- Do not let the device run without supervision.

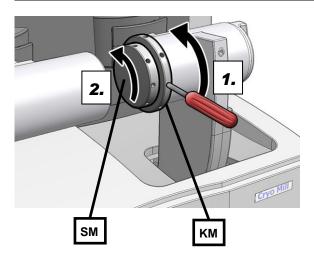


Fig. 7: Loosening the grinding jar safety catch

- Loosen the lock nut (KM).
- Loosen the screw (SM).



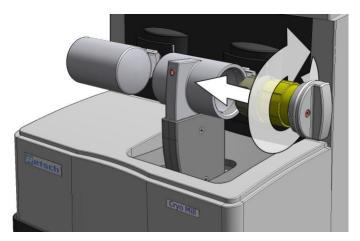


Fig. 8: Inserting the grinding jar

- Push the special grinding jar filled with the comminution material and the grinding balls into the cooling jacket.
- Screw the grinding jar in completely.

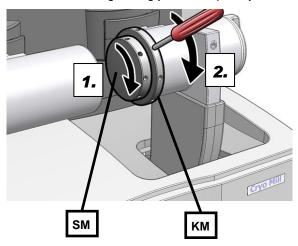


Fig. 9: Securing the grinding jar

- First tighten the screw (SM) by hand (to finger tightness).
- Then tighten the lock nut (KM) against the cooling jacket. Use the provided spring pin for this purpose.

Make sure that the grinding jar is screwed tightly to the cooling jacket; otherwise liquid nitrogen can escape.

The lock screw (SM) must be tightened and secured with the lock nut so that no sample material will escape from the grinding jar.



CAUTION

Danger of injury to eyes and skin

The temperatures of the cooling jacket and the grinding jar become very low during the grinding.



- Danger of injuries to eyes and skin from extreme freezing.
- Always wear goggles and protective gloves when opening the cooling jacket and the grinding jar.





To take out the grinding jar, use the opening aid (F) that is kept on the side of the device.

- Loosen the lock nut(KM)
- Loosen the screw (SM)
- Put the opening aid (F) onto the grinding jar and open it.

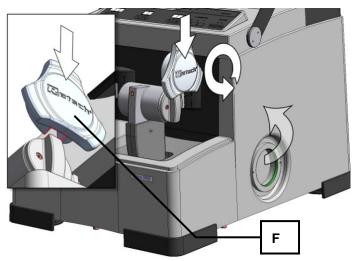


Fig. 10: Using the opening aid

5.5.2 Inserting the cooling jacket sealing plug

NOTE

Clogged piping

Formation of ice

- Ice or foreign matter can clog the pipe and subsequently cause malfunctioning.
- Insert the cooling jacket sealing plug (KA) as soon as you stop using the device.

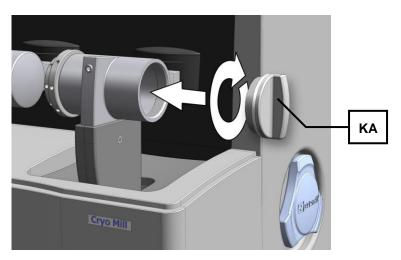


Fig. 11: Inserting the cooling jacket sealing plug

5.5.3 Insert the adapter for 4/2 x 5 ml grinding jar



You can operate the CryoMill with 4 or 2 grinding jars (5 ml).

• Insert the 5 ml grinding jars into the adapter as shown in the following diagram.

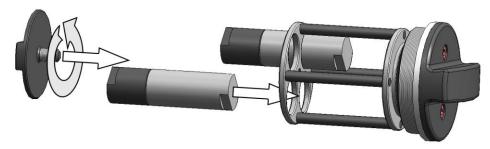


Fig. 12: Inserting 5ml grinding jars

Please pay attention to the positioning of the grinding jars. If you use two grinding jars they must be positioned in the opposite openings.

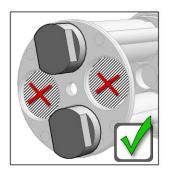




Fig. 13: 5ml grinding jar position

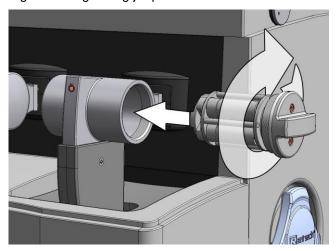


Fig. 14: Inserting the grinding jar adapter

5.5.4 Insert the adapter for 4/2 x 2-ml Eppendorf reaction vial

NOTE

Shattering of Eppendorf reaction vials

Changing material properties

 The Eppendorf reaction vials, available as accessories, change their material properties during cryogenic grinding.

Eppendorf reaction vials after cryogenic grinding:



- do not use them again
- · do not insert into centrifuges

You can operate the CryoMill with 4 or 2 Eppendorf reaction vials (2-ml).

• As shown in the following diagram, insert the 2-ml Eppendorf reaction vials into the adapter for reaction vials.

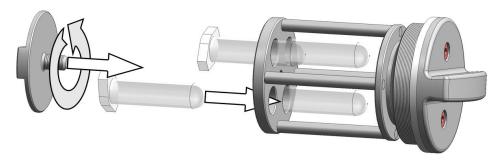


Fig.15 Insert 2-ml Eppendorf reaction vials

Make sure the Eppendorf reaction vials are positioned correctly. If you use two reaction vials, you must insert them into the opposite openings.

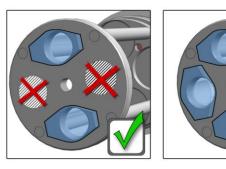


Fig. 16 Position 2-ml reaction vials

5.6 Connecting the cooling agent feed



Clogged piping

Formation of ice

 Ice or foreign matter can clog the pipe and subsequently cause malfunctioning.

Keep the cooling system and the supply pipe dry and free of foreign matter.

- Do not insert the grinding jar if it is damp or covered with frost.
- Use the cooling jacket sealing plug (KA) if you are not going to use the device for a long time.
- Do not let the device run without supervision.



NOTICE

Damage to the machine

Wrong cooling agent

- If cooling agents other than liquid nitrogen are used, the cooling system will lose its tightness.
- Liquid nitrogen (LN2) may be used exclusively in this machine as cooling agent.

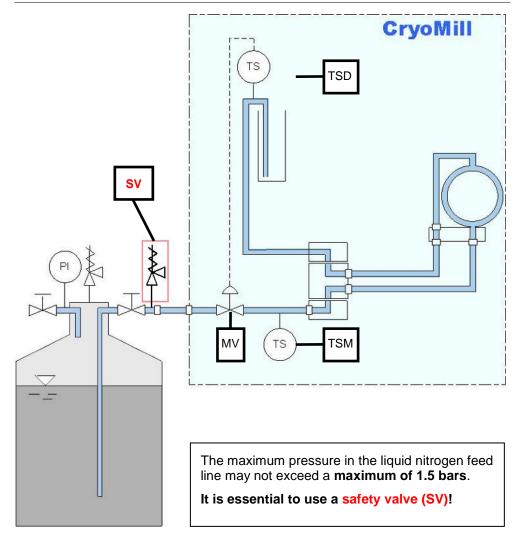


Fig. 17: Schematic drawing of the cooling system

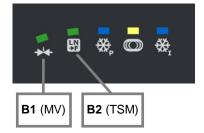


Fig. 18: LED displays

The temperature sensor (**TSD**) in the Dewer vessel controls the solenoid valve (**MV**). The LED (**B1**) lights up if the solenoid valve (**MV**) is open.



The temperature sensor (TSM) that is switched downstream of the solenoid valve registers whether liquid nitrogen flows into the CryoMill. The LED (B2) lights up if liquid nitrogen flows into the cooling system.



⚠ VORSICHT

Do not move the device when it is in a cryogenic state. Do not allow any additional forces to affect the coolant connection (N). The coolant supply hose may not be moved as long as the device is in a cryogenic state.



CAUTION

Danger of injury and frostbite

Bursting of the feed line hose

- Depending on the operating state of the machine, liquid nitrogen may be enclosed in the feed line. In the case of heating-up this may lead to the feed line hose bursting.
- The maximum pressure in the liquid nitrogen feed line may be a maximum of 1.5 bars. It is essential to use a safety valve!

The connection for the liquid nitrogen (LN2) is a G 1/4 inch thread connection.

The working pressure in the liquid nitrogen supply pipe should be at least 0.5 bar.

The lower the pressure in the liquid nitrogen feed line, the longer the precooling time will be. At 0.5 bars feed line pressure and at a room temperature of the system the precooling time will be approx. 10 minutes. Depending on application. you can conduct up to 4 grinding operations with 10 litres of liquid nitrogen.

During the entire cooling time the atmospheric humidity of the room will be reflected as an ice layer on all cooled parts.

After cooling, this condensed water will drop into the collecting filter and evaporate.



CAUTION

Danger of injury and frostbite

Uncontrolled escape of liquid nitrogen

- In the event of an uncontrolled escape of liquid nitrogen the danger of injury exists.
- Immediately close the liquid nitrogen feed line!

NOTICE

Depending on ambient temperature, atmospheric humidity and duration of use, small quantities of water may drip from the collecting filter.

5.7 Removing the liquid nitrogen feed line

Before you remove the liquid nitrogen feed line, the pressure must be relieved for safety reasons.

Press the (B10) pre-cooling and (B13) START keys simultaneously for 2 seconds.

As long as you keep the keys depressed, the solenoid valve will remain open.

Cold air outlet 5.8



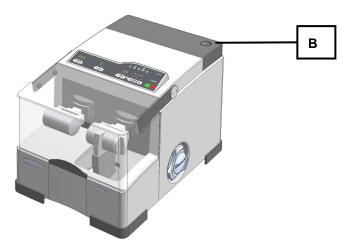


Fig. 19: Outlet opening for gaseous nitrogen (B)



Danger of injury to eyes and skin through frostbite

Outlet opening for gaseous nitrogen

- Liquid nitrogen has a temperature of –196 °C and may cause injuries similar to burns on skin or eye contact or cause frostbite.
- Do not hold any part of the body over the outlet opening for gaseous nitrogen (B).

5.9 Explanations of the grinding cycles

You can use the CryoMill for grinding with cooling or for grinding without cooling.

5.9.1 Grinding without Cooling

- Turn on the CryoMill by pressing the main switch.
- Set the pre-cooling time to zero (B9) + (B10).
- Set the desired grinding time (B9) + (B11).
- Press the START key.

The START LED and the REMAINING TIME LED light up. The remaining grinding time and the set frequency are displayed. The 01 value appears in the B9 display.

5.9.2 Grinding with Cooling

When grinding with cooling the following program items can be set.

- Pre-cooling time (Vkz)
- Grinding time (Mz)
- Intermediate cooling time (Zkz)
- Cryo Cycles (Number of Cooling /Grinding Cycles)
- Frequency 1/s (Grinding Frequency)

A single grinding cycle consists of the pre-cooling time and the set grinding time. There is no intermediate cooling time for single grinding cycles.

Vkz + Mz = total grinding time



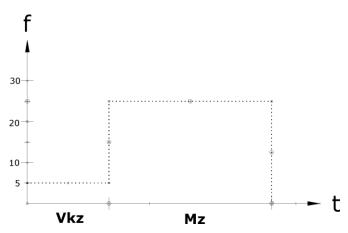


Fig. 20: Sequence in a single grinding cycle

Several grinding cycles consist of the pre-cooling time, the set grinding time and the intermediate cooling time.

Number of cryo cycles: n

Vkz + (n-1)(Mz + Zkz) + Mz = total grinding time

Pre-cooling time + [(Number of the set cryo cycles) - 1] X (grinding time + cooling time) + grinding time = total grinding time

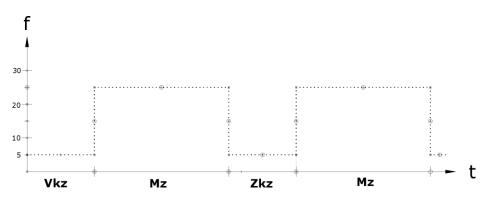


Fig. 21: Sequence in two grinding cycles

5.10 Setting the grinding frequency



Fig. 22: Frequency 1/s

Switch on the CryoMill at the main switch.

The grinding frequency can only be set if the button (B11) has been pressed to set the grinding parameters and the LED (B11) has lit up.

- Press the button (B11).
- Press the corresponding buttons (B7) to set the grinding frequency



The adjustable frequency range is between 3 and 25 Hz.

- + short pressing increases the frequency.
- short pressing decreases the frequency.

If the buttons are pressed for longer the display runs faster.

The frequency of precooling and intermediate cooling is set at a constant 5 Hz. During the setting of the precooling or intermediate cooling time the frequency cannot be adjusted.

5.11 Setting grinding cycles

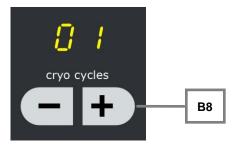


Fig. 23: Setting the grinding cycle

Switch on the CryoMill at the main switch.

After switching on the last used grinding parameters are shown and can be used.

Press the corresponding button (B8) to set the grinding cycles.

You may adjust 1 to 9 grinding cycles.

- + short pressing increases the number of cycles.
- short pressing decreases the number of cycles.

If the buttons are pressed for longer the display runs faster.

5.12 Setting the precooling time

NOTE

During the pre-cooling and intermediate cooling processes the machine runs at a frequency of 5 Hz.

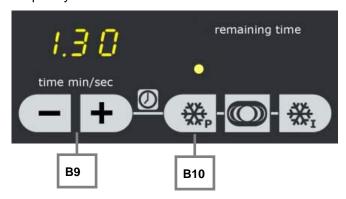


Fig. 24: Setting the pre-cooling time

• Turn on the CryoMill by pressing the main switch.



When the device is switched on, the grinding parameters last used are displayed and can be used.

To adjust the pre-cooling time, press the (B10) key.

The pre-cooling time LED lights up.

You have a choice of three pre-cooling variants:

5.12.1 Pre-Cooling with Definable Pre-Cooling Time

- Use the (B9) keys to set the intermediate cooling time.
- Briefly pressing and releasing + increases the duration.
- Briefly pressing and releasing reduces the duration.

Pressing and holding causes the display to run up or down more quickly.

5.12.2 Automatic Pre-Cooling

In automatic pre-cooling the grinding starts as soon as the entire system has cooled down. This is monitored by a filling level sensor in the Dewar vessel.

- Set the pre-cooling time to -- .
- Use the (B9) keys to set the intermediate cooling time.
- Briefly pressing and releasing + increases the duration.
- Briefly pressing and releasing reduces the duration.

Pressing and holding causes the display to run up or down more quickly.

5.13 Setting the grinding time

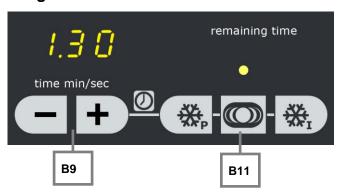


Fig. 25: Setting the grinding time

Switch on the CryoMill at the main switch.

After switching on the last used grinding parameters are shown and can be used.

Press the button (B11) to set the grinding time.

The grinding time LED lights up.

- Use the buttons (B9) to set the grinding time.
- + short pressing increases the time.
- short pressing decreases the time.



If the buttons are pressed for longer the display runs faster.

5.14 Setting the duration of intermediate cooling



Fig. 26: Setting intermediate cooling

Switch on the CryoMill at the main switch.

After switching on the last used grinding parameters are shown and can be used.

Press the button (B12) to set the intermediate cooling time.

The intermediate cooling time LED lights up.

- Use the buttons (B9) to set the intermediate cooling time.
- + short pressing increases the time.
- short pressing decreases the time.

If the buttons are pressed for longer the display runs faster.

5.15 Starting the grinding process



Danger of injury and frostbite

Escape of liquid nitrogen

- Liquid nitrogen has a temperature of –196 °C and may cause injuries similar to burns on skin or eye contact or frostbite.
- Never switch the machine on without tightly closed cooling casing.

NOTICE

The grinding process can only be started if the hood is closed.



Fig. 27: Starting the grinding process



Press the START button to start the grinding process.

The Start LED (green) lights up. At the same time the solenoid valve opens and liquid nitrogen can flow into the cooling system.

5.16 Stopping the grinding process



Fig. 28: Stopping the grinding process

Press the STOP button to end the grinding process.

Die Stop LED (red) lights up. At the same time the solenoid valve closes.

If during operation the feed of liquid nitrogen was interrupted this is shown by the flashing of the remaining time LED (B6) at the end of the grinding time.

Pressing the button once interrupts the grinding process to assess the sample material for example. The remaining grinding time remains visible in the display.

Pressing the start button again causes the mill to continue until the grinding time has been completed.

Pressing the stop button twice stops the grinding process. The machine is now in stand-by mode.

Pressing the start button reactivates the display and the grinding time is reset to the last starting value.

During the grinding process the time can be adjusted. When the grinding time has elapsed the grinding process is automatically ended. The display is reset to the last started value.

5.17 Operating hours display



Fig. 29: Display of the operating hours

Press the STOP button to switch the machine to stand-by mode.

The stop LED (ret) lights up.

Press the frequency 1/s = and cryo cycles + buttons simultaneously.

The overall running time is shown in hours distributed over the three display windows. The maximum value that may be displayed is 999999 hours. The letters bS are displayed at the first two places.

Examples:



 $bS0\ 00\ 012 = 12$ operating hours

bS1 23 456 = 123456 operating hours

Press the STOP button to leave the operating hours display.

5.18 Operating software display



Fig. 30: Operating software display

Press the STOP button to switch the machine to stand-by mode.

The stop LED (red) lights up.

Press the frequency 1/s + and cryo cycles + buttons simultaneously.

The current version of the operating software is shown distributed over the centre and right hand display window. The letter <S> is shown at the first place.

Examples:

S 1.23 = version 1.23 of the operating software

S 2.34 = version 2.34 of the operating software

Press the STOP button to leave the operating software display.

5.19 Replacing the machine fuses

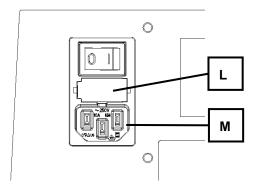


Fig. 31: Replacing the machine fuses

The following visible-type fuses are required for the Cryomill: 2 fuses MT 3.15 A

- Unplug mains plug from the machine socket (M).
- By pressing the locking devices on the side of the fuse compartment (L) these are released and can be pulled out.
- · Replace fuses.
- Insert fuse holder (L) so that it engages.



6 Working instructions

6.1 General

The CryoMill is an ultra modern, high performance product from Retsch GmbH.

Owing to the large selection of accessories the CryoMill is a machine with many different application possibilities in laboratories in industry and research.

It is used mainly in the chemical and pharmaceutical centres and in mineralogical and biological applications etc.



CAUTION

Danger of explosion or fire

Changing sample properties

- Please note that the properties, and therefore the dangerous nature of your sample, can change during the grinding process.
- Do not grind any substances in this machine which can cause an explosion or a fire.



CAUTION

Danger of personal injury

Dangerous nature of the sample

- Depending on the dangerous nature of your sample, take the necessary measures to rule out any danger to persons.
- (i)
- Observe the safety guidelines and datasheets of your sample material.

6.2 The grinding process

- Place the grinding jar filled with sample material and grinding ball(s) into the grinding jar holder as described in the section on "Inserting grinding jar".
- Close the hood.
- Set the grinding parameters.
- Open your external liquid nitrogen feed line.
- Start the machine.

Do not open the hood during the grinding process.

Although the grinding jars are brought to a standstill immediately by the built-in brake, the grinding process can no longer be continued with the remaining running time.

The machine must be restarted and the initial parameters are then available again.

It is possible to grind without cooling. For this purpose, the precooling and intermediate cooling times are set to zero.



6.3 Quantities of sample material and feed sizes

			Recommended ball filling (piece)					
Grinding jar volume	Sample material quantity	Max. feed size	Ø7 mm	Ø10 mm	Ø12 mm	Ø15 mm	Ø20 mm	Ø25 mm
5.0 ml	0.5 - 2.0 ml	2 mm	1 or 2	-	-	-	-	-
25.0 ml	4.0 – 10.0 ml	6 mm	-	5 or 6	2 to 4	1 or 2	-	-
35.0 ml	6.0 - 15.0 ml	6 mm	ı	6 to 9	4 to 6	2 or 3	1	-
50.0 ml	8.0 - 20.0 ml	8 mm	-	12 to 14	6 to 8	3 or 4	1	1

6.4 Working instructions

The grinding jar and its content are cooled by liquid nitrogen (LN2) until it becomes possible to grind soft material such as rubber.

Precooling will be necessary until the ideal grinding temperature has been reached. The motor runs at a low frequency (5Hz) to prevent the moving machine parts from freezing. During precooling the fan is switched on and blows the air heated by the motor through the front openings of the oscillating arms into the grinding chamber. This warm air prevents the oscillating arm bearing from cooling down. Therefore, it is also necessary for the fan to continue to run for a period of time after the grinding process has been discontinued.

In preliminary tests an average precooling time of approx. 10 to 15 minutes has been determined. The grinding time was between 4-5 minutes.

The valve is switched off on reaching the ideal temperature. Starting from this time, the grinding process can be commenced and the valve is switched on or off depending on temperature.

The grinding process can also be conducted without cooling whereby the precooling and intermediate cooling times are set to ZERO.

Manual cooling is started by pressing the button combination (B10) and (START B13). The valve remains open for as long as the buttons are pressed.



7 Safety functions and fault display

7.1 Fault messages

F01	Motor temperature too high	The F01 fault message appears. The fault message is reset by pressing the stop button.
F02	Zero speed at the eccentric shaft	There are no signal from the speed sensor.
F03	Overspeed	The speed is >1500 rpm. Motor brake monitoring.
F04	Hood is opened	The magnetic switch sends a signal that the hood is open.
F05	Contact monitoring	This fault message is displayed if the start, stop, SET, time +, time -, frequency + and – keys are pressed for longer than 10 seconds. Fault messages for time and frequency are only emitted when the maximum levels are reached.
F06	Magnetic switch monitoring, hood	Service necessary.
F07	Wire break monitoring, magnetic switch, hood	Service necessary.
F08	Monitoring PT100 LN2 inlet	Service necessary.
F09	Monitoring temperature sensor in the liquid nitrogen collection container (PT100 Dewar)	
F32	Leakage monitoring/no LN2 during the cooling process	The device is stopped, a continuous acoustic alarm is given and the F32 error message appears.
	CAUTION	
	Close the hand valve on the Dewar immediately!	
	Leakage inside the machine or interruption in the liquid nitrogen feed line (storage container empty or hand valve closed)	
F33	Solenoid valve monitoring	The valve is switched on and off several times in order to
	CAUTION	remove the foreign body or tap the valve free.
	Close the hand valve on the	Press the STOP key. Press the START levels and times the principle.
	Dewar immediately!	Press the START key to continue the grinding.
	Solenoid valve does not close. Foreign matter (ice or dirt) inside the magnet valve.	If the ice is not eliminated after the second attempt of knocking it free, the machine is switched off with F11 and an acoustic alarm is heard.
		Switch off the device.
		The reason for the machine being switched off must be checked every time it is switched off by F33.
		A restart is only permitted if the cooling system has dried out completely.



F34	There is no flow of liquid	The device is switched off after 15 minutes with this error
	nitrogen	message.

Any interruption in the supply of liquid nitrogen during operation is indicated by the flashing of the remaining time (B6) LED at the end of the grinding time.

All other fault alarms cause the machine to be switched off. The fault alarm is indicated in the time display, the red stop LED lights up and all other displays are off.



8 Cleaning, wear and service

8.1 Cleaning

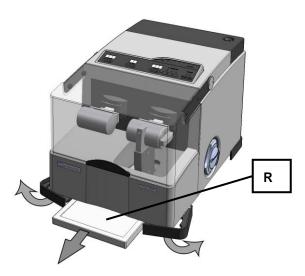


Fig. 32: Pulling out collecting filter for condensed water

Depending on the ambient conditions, the degree of use and general atmospheric humidity the collecting filter for condensed water must be cleaned regularly.

- Push out the two front guards to the side.
- Pull out the collecting filter for condensed water (R).
- Clean the collecting filter for condensed water with a little commercially available detergent under running water.
- Allow the collecting filter to dry and then replace it into the machine.

8.2 Wear

The grinding tools may become worn, depending on the frequency of the grinding operation and the sample material. The grinding jars and the balls should be regularly checked for wear and replaced if necessary.

8.3 Wearing parts



Personal injury

Incorrect repairs

- This operating manual does not include instructions for repair.
- For your own safety repairs should be carried out only by Retsch GmbH or an authorised representative (service technicians).

8.4 Service



The grinding tools may become worn, depending on the frequency of the grinding operation and the sample material. The grinding jars and the balls should be regularly checked for wear and replaced if necessary.

8.5 Checks

Correct functioning of the hood switch must be checked regularly.

- Switch on the machine with switch (K).
- Start grinding operation with start button (B13 Start).
- · Lift the hood.

If there is an opening gap of a few cm the machine switches itself off and F04 appears in the display.

Cancel F04 with stop button (B13 - Stop).

If this switching-off function does not take place the CryoMill must be checked immediately by Retsch-Service.

9 Accessories

Piece	Art. No.	Designation	DIN /Material	Weight
1	01.462.0284	Grinding jar 50ml	Stainless steel	0.76kg
1	01.462.0288	Grinding jar 35ml	Stainless steel	0.74kg
1	01.462.0289	Grinding jar 25ml	Stainless steel	0.64kg
1	01.462.0290	Grinding jar 5ml (for use with adapter 02.706.0272)	Stainless steel	0.065kg
1	02.480.0001	Autofill with LN2 container, 10 litre		10kg
1	02.706.0272	Adapter for use of 2 or 4 grinding jars, 5ml stainless steel (01.462.0290)	Stainless steel	0.48kg
1	05.368.0029	Grinding ball 5mm ø	Hardened steel	
1	05.368.0030	Grinding ball 7mm ø	Hardened steel	
1	05.368.0031	Grinding ball 9mm ø	Hardened steel	
1	05.368.0032	Grinding ball 12mm ø	Hardened steel	
1	05.368.0033	Grinding ball 20 ø	Hardened steel	
1	05.368.0034	Grinding ball 5mm ø	Stainless steel	
1	05.368.0035	Grinding ball 7mm ø	Stainless steel	
1	05.368.0036	Grinding ball 9mm ø	Stainless steel	
1	05.368.0037	Grinding ball 12mm ø	Stainless steel	
1	05.368.0062	Grinding ball 20mm ø	Stainless steel	
1	05.368.0105	Grinding ball 25mm ø	Stainless steel	

10 Safety and occupational health instructions





CAUTION

Danger of injury to eyes and skin

Frostbites through liquid nitrogen

- Liquid nitrogen has a temperature of -196 °C and may cause injuries similar to burns on skin or eye contact or cause frostbite.
- Always use goggles and wear protective gloves when opening the cooling casing and the grinding jar.

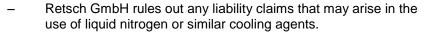






CAUTION

Use of liquid nitrogen





Please observe the safety regulations of the cooling liquid supplier.



10.1 General

This section summarises the general occupational health and safety guidelines for the handling of liquid nitrogen.



CAUTION

Handling liquid nitrogen

General hazardous situations

- The following hazardous situations may arise in the handling of liquid nitrogen: oxygen deficiency situations, cryogenic burns, danger of explosion, oxygen enrichment
- All users must be informed about the dangers of working with liquid nitrogen in order to be able to work safely.



10.2 Safety instructions on the handling of liquid nitrogen

10.3 Oxygen deficiency

The main components of air according to volumes are as follows:

- Oxygen O2 21 %
- Nitrogen N2 78 %
- Argon Ar 1 %

The gases contained in the atmosphere are not toxic. However, a change in concentration (in particular changes to the oxygen concentration) may have effects on life and burns. It is therefore essential for the air breathed in to contain sufficient oxygen (> 19 %).

Humans cannot detect changes in the composition of air within the time that will actually be necessary because the components are colourless and odourless.

10.3.1 Dangers

A danger of suffocation exists as a result of the normal evaporation of the liquid nitrogen that pushes out the oxygen in the air. An oxygen deficiency is dangerous and can cause death through suffocation. The reaction of the organism to oxygen deficiency will differ greatly depending on the individual. It is not possible to provide precise and generally applicable information on the symptoms of an oxygen deficiency.

Example: under normal conditions (20° C; 1013 mbars) 1 l liquid nitrogen evaporates to produce 680 l nitrogen gas.

10.3.2 Causes

An oxygen deficiency may arise during the following work or in the following conditions:

- Nitrogen as liquid or gas
- Natural evaporation of liquid nitrogen
- Refilling of liquid nitrogen
- Leaks in containers for liquid or gaseous nitrogen
- Defect in the air feed or outlet
- Tipping over of the container

This list is not complete.

10.3.3 Recommendations

In order to prevent the danger of an oxygen deficiency, the following measures must be taken.

The vessel:

- must be kept in a vertical position.
- must be provided with a suitable insulating lid.
- must be protected from direct sunlight and may not be set up near heat sources.

Safety and occupational health instructions

- may not be transported in vehicles in filled state.
- must be protected from impact, knocks and rapid movements.
- Ventilate all installation rooms constantly and appropriately.
- Wear protective clothing (suitable gloves, goggles or face protection and safety shoes).
- Check the oxygen content of the room constantly.
- Always carry an oxygen metre.
- Only trained personnel may work with liquid nitrogen.

This list is not complete.

10.3.4 General conduct in the case of an accident

In the case of an accident from oxygen deficiency the following regulations should be observed.

- Secure the surrounding area to avoid any subsequent accidents.
- Act quickly.
- The rescuers must take measures to protect themselves (respiratory protection device).
- Move the injured persons from the hazardous area.
- Observe the company instructions for emergencies.
- Ventilate the rooms affected sufficiently.
- Investigate the cause of the accident.

This list is not complete.

10.4 Cryogenic burns

Liquid nitrogen is very cold (-196° C).

The surfaces of vessels that were in contact in liquid nitrogen (in particular during the filling process) may cause skin burns on contact.

10.4.1 Dangers

Cryogenic liquids may:

- bring about burns to the human body
- make specific materials (metal and plastic) that are not suitable for low temperatures brittle
- generate strong misting depending on atmospheric humidity

10.4.2 Causes

There are two types of cryogenic burns:

10.4.2.1 Burns through splashes

When handling samples and in general when handling liquid nitrogen, personnel must protect themselves from splashes. They can cause cryogenic burns with serious consequential damage, in particular to eyes and face.



10.4.2.2 Burns through contact

Contact of the skin with cold material causes frostbite or cryogenic burns.

The interior of vessels or the samples may never be touched or held with bare hands.

10.4.3 Recommendations

In order to prevent the danger of burning the following points must be observed:

- Never bring cryogenic liquids in contact with the skin.
- Never touch the cold non-isolated or iced walls of a vessel.
- Wear personal protective equipment (suitable gloves, goggles or face protection and safety shoes).
- Keep the vessel upright.
- Use suitable material (e.g. metal hose or PTFE hose) to refill.
- Train personnel.

This list is not complete.

10.4.4 General rules of conduct for splashes with liquid nitrogen

10.4.4.1 To the eyes

- Rinse eyes with much water for 15 min.
- Follow the company instructions for emergencies.
- Consult a doctor

10.4.4.2 On the skin

- Do not rub.
- If possible remove or loosen clothing.
- Slowly and gradually warm the parts affected.
- Do not apply anything to the burnt area.
- Follow the company instructions for emergencies.
- Consult a doctor.

Both lists are not complete.

10.5 Danger of explosion

10.5.1 Dangers

The evaporation of liquid nitrogen may lead to an overpressure in the vessel.

10.5.2 Causes

An increase in the vessel may be attributable to:

- incorrect set-up (use of a closable lid)
- icing on the neck and on the insulating lid



This list is not complete.

10.5.3 Recommendations

To avoid the danger of explosion:

- Always use suitable insulating lids (pay attention to waste gas opening).
- Observe filling levels to avoid the formation of ice on the insulating lid.
- Set up the vessel in dry and roofed rooms.
- Monitor atmospheric humidity in the installation room.
- Check vessel regularly for the collection of condensed water.
- Check vessel regularly for damage to surface or material damage

This list is not complete.

10.5.4 General conduct in the case of an accident

In the case of an accident from oxygen deficiency the following regulations should be observed.

- Secure the surrounding area to avoid any subsequent accidents.
- Act quickly.
- The rescuers must take measures to protect themselves (respiratory protection device).
- Move the injured persons from the hazardous area.
- Observe the company instructions for emergencies.
- Ventilate the rooms affected sufficiently.
- Investigate the cause of the accident.

This list is not complete.

10.6 Oxygen enrichment

10.6.1 Dangers

Oxygen enrichment may increase the danger of explosion and fire.

10.6.2 Causes

Oxygen may be condensed from the air if liquid nitrogen is used and similarly be liquefied because the boiling point of oxygen (approx. -183° C) is above that of nitrogen (-196° C).

10.6.3 Recommendations

The following points must be considered in the case of oxygen enrichment:

- Do not smoke.
- Where possible, keep easily inflammable materials away from the vessel.



- Remove all sources of fire (naked flame and light, spark producers, matches, lighters etc.).
- Ventilate installation rooms constantly and appropriately.
- Clean floor regularly.
- Train personnel.
- Wear personal protective equipment.
- Check oxygen content constantly.
- Always carry an oxygen metre.

This list is not complete

10.7 Surrounding area of the machine

10.7.1 Rooms

The room in which the machine is located must:

- permit operation without danger to the employees
- have a constantly running and appropriate ventilation system
- have a level and non-porous floor that is also able to bear the load of the vessel
- have safety datasheets on liquid nitrogen visible for all to see
- · prevent unauthorised access
- permit the safe filling of the vessel
- · permit accessibility to the vessel for inspection, cleaning and maintenance

This list is not complete.



11 Disposal

Please observe the respective statutory requirements with respect to disposal.

Information on disposal of electrical and electronic machines in the European Community.

Within the European Community the disposal of electrically operated devices is regulated by national provisions that are based on the EU Directive 2002/96/EC on Waste Electrical and Electronic Equipment (WEEE).

Accordingly, all machines supplied after 13.08.2005 in the business-to-business area to which this product is classified, may no longer be disposed of with municipal or household waste. To document this they have the following label:

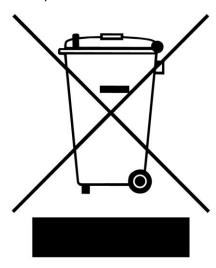


Fig. 33: Disposal label

Since the disposal regulations within the EU may differ from country to country we would request you to consult your supplier.



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