

Manual Dewar LN2



Translation

Retsch[®]

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| | | |
|----------|--|-----------|
| 1 | Notes on the Operating Manual | 6 |
| 1.1 | Explanations of the safety warnings | 7 |
| 1.2 | General safety instructions | 8 |
| 1.3 | Repairs..... | 9 |
| 2 | Confirmation | 10 |
| 2.1 | Type plate description..... | 11 |
| 3 | Safety and occupational health instructions..... | 11 |
| 3.1 | General | 12 |
| 3.2 | Safety instructions on the handling of liquid nitrogen | 13 |
| 3.3 | Oxygen deficiency | 13 |
| 3.3.1 | Dangers | 13 |
| 3.3.2 | Causes..... | 13 |
| 3.3.3 | Recommendations | 13 |
| 3.3.4 | General conduct in the case of an accident | 14 |
| 3.4 | Cryogenic burns..... | 14 |
| 3.4.1 | Dangers | 14 |
| 3.4.2 | Causes..... | 14 |
| 3.4.2.1 | Burns through splashes | 14 |
| 3.4.2.2 | Burns through contact..... | 14 |
| 3.4.3 | Recommendations | 14 |
| 3.4.4 | General rules of conduct for splashes with liquid nitrogen | 15 |
| 3.4.4.1 | To the eyes | 15 |
| 3.4.4.2 | On the skin..... | 15 |
| 3.5 | Danger of explosion..... | 15 |
| 3.5.1 | Dangers | 15 |
| 3.5.2 | Causes..... | 15 |
| 3.5.3 | Recommendations | 15 |
| 3.5.4 | General conduct in the case of an accident | 16 |
| 3.6 | Oxygen enrichment..... | 16 |
| 3.6.1 | Dangers | 16 |
| 3.6.2 | Causes..... | 16 |
| 3.6.3 | Recommendations | 16 |
| 3.7 | Surrounding area of the machine | 16 |
| 3.7.1 | Rooms..... | 16 |
| 4 | Operating the machine | 18 |
| 4.1 | Views of the Instrument | 18 |
| 4.2 | Overview table of the parts of the device | 19 |
| 4.3 | Technical Data | 20 |
| 4.4 | Vacuum closure and safety device | 20 |
| 4.5 | Level Display | 21 |
| 4.6 | Filling the vessel | 22 |
| 4.7 | Pressure build-up..... | 23 |
| 4.8 | Withdrawal of liquid nitrogen..... | 23 |
| 4.9 | Pressure relief..... | 23 |
| 4.10 | Transport..... | 24 |
| 4.11 | Service..... | 24 |

| | | |
|----------|--------------------------------------|-----------------------|
| 4.12 | Admitting the test pressure | 25 |
| 4.13 | Checking the seat tightness..... | 25 |
| 4.14 | Checking the response pressure | 25 |
| 5 | Index | 26 |
| | Appendix | following page |

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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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.**
-

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.

I have read and taken note of the contents of all chapters in this operating manual as well as all safety instructions and warnings.

User

Surname, first name (block letters)

Position in the company

Signature

Service technician or operator

Surname, first name (block letters)

Position in the company

Place, date and signature

2.1 Type plate description



Fig. 1: Type plate marking

- 1 Instrument name
- 2 Year of manufacture
- 3 Article number
- 4 Serial number
- 5 Manufacturer's address
- 6 CE marking
- 7 Waste disposal marking
- 8 Barcode

When making enquiries, please quote the name of the instrument (1) or the article number (3) and serial number (4) of the instrument.

3 Safety and occupational health instructions

 **CAUTION**

Danger of injury to eyes and skin

Frostbites through liquid nitrogen

- Liquid nitrogen has a temperature of $-196\text{ }^{\circ}\text{C}$ 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

- Retsch GmbH rules out any liability claims that may arise in the use of liquid nitrogen or similar cooling agents.
- **Please observe the safety regulations of the cooling liquid supplier.**



3.1 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.**
-

3.2 Safety instructions on the handling of liquid nitrogen

3.3 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.

3.3.1 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.

3.3.2 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.

3.3.3 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.
- 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.

3.3.4 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.

3.4 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.

3.4.1 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

3.4.2 Causes

There are two types of cryogenic burns:

3.4.2.1 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.

3.4.2.2 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.

3.4.3 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.

3.4.4 General rules of conduct for splashes with liquid nitrogen

3.4.4.1 To the eyes

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

3.4.4.2 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.

3.5 Danger of explosion

3.5.1 Dangers

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

3.5.2 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.

3.5.3 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.

3.5.4 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.

3.6 Oxygen enrichment

3.6.1 Dangers

Oxygen enrichment may increase the danger of explosion and fire.

3.6.2 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).

3.6.3 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

3.7 Surrounding area of the machine

3.7.1 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.

4 Operating the machine

The APOLLO® vessel is a vacuum super-insulated pressure vessel made of corrosion-resistant stainless steel for storing refrigerated liquid nitrogen in conformance to the European Pressure Equipment Directive 97/23/EG, Category I module A, with CE marking.

4.1 Views of the Instrument

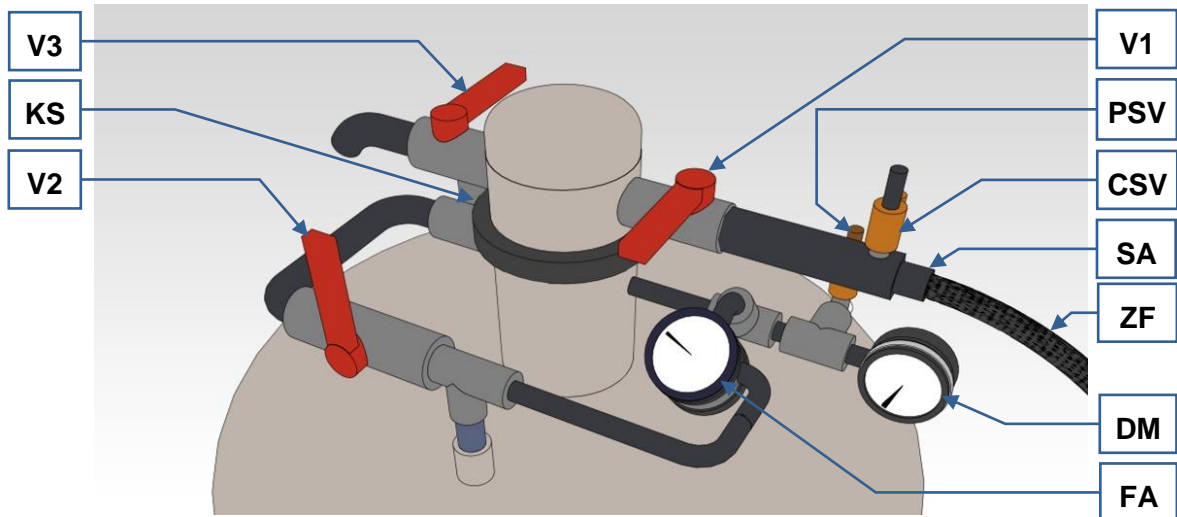


Fig. 2: Front view

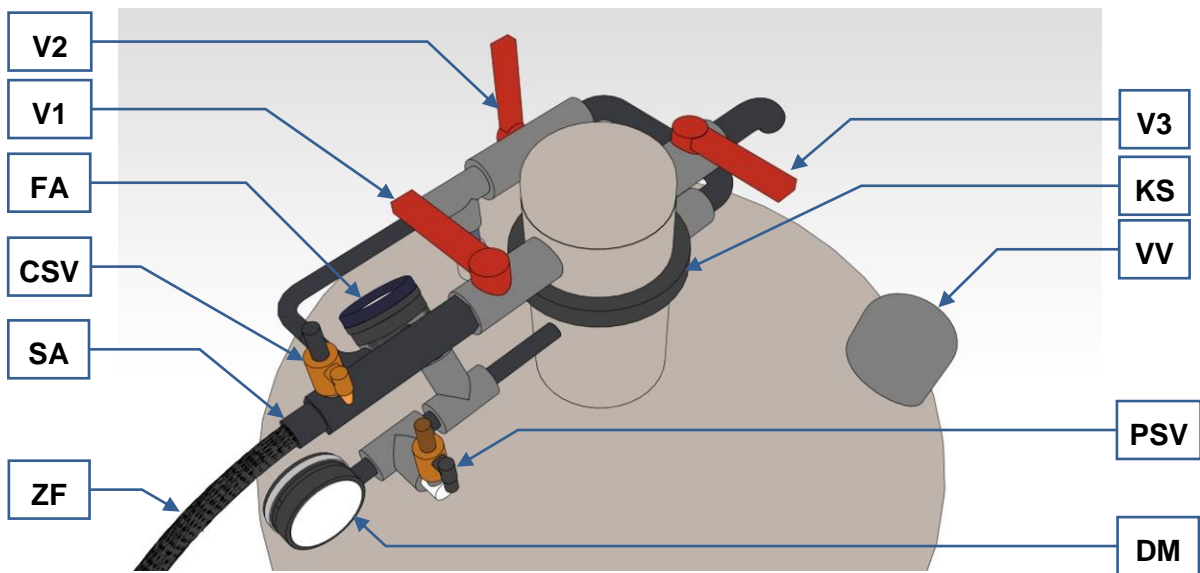


Fig. 3: Rear view

4.2 Overview table of the parts of the device

| Element | Description | Function |
|------------|-----------------------------------|--|
| V1 | Withdrawal valve | Valve for taking out liquid nitrogen |
| V2 | Pressure build-up valve | Valve for building up working pressure quickly |
| V3 | Waste gas valve | Valve for draining liquid nitrogen |
| CSV | Overflow valve | Safety valve for the feed line to the CryoMill |
| DM | Pressure indicator | Gas pressure display |
| PSV | Dewar flask pressure relief valve | Safety valve 1.3 bar of the Dewar flask |
| KS | Quick-release clamp | Sealing and attachment of the siphon to the Dewar flask. |
| VV | Vacuum closure and safety device | Sealing of high vacuum in the intermediate hollow space |
| ZF | Withdrawal hose | Liquid nitrogen supply line to the CryoMill |
| FA | Level display | Shows the liquid nitrogen level |

4.3 Technical Data

Type: APOLLO® 50

| | |
|----------------------------|--------------|
| Overall height: | 80cm |
| Outside diameter: | 50cm |
| Overall width: | 76cm |
| Immersion depth | 58.5cm |
| Neck diameter: | 5.0cm |
| Weight empty: | 44kg |
| Weight full: | 85 kg |
| Geometrical capacity: | 49.5 litres |
| Stat. rate of evaporation: | 2% / day |
| Operating pressure: | max. 1.3 bar |

4.4 Vacuum closure and safety device

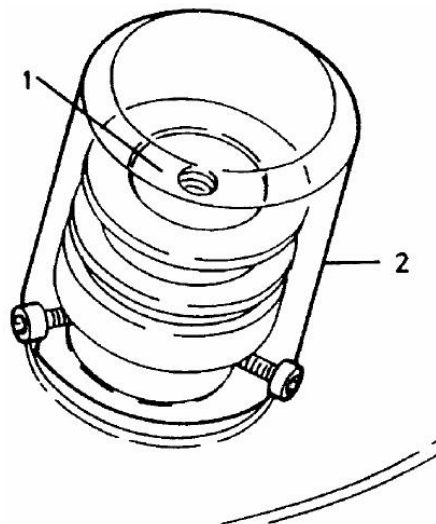


Fig. 3: Vacuum closure and safety device

⚠ WARNING

The vacuum closure and the safety device protect the vacuum space from positive pressure. Only the manufacturer's qualified staff may conduct the re-evacuation.

⚠ CAUTION

The protective cap (2) retains the valve insert (1) if excessive pressure builds up in the vacuum space.

- Do not remove the protective cap (2).
- Protect the valve from heat or cooling because embrittlement can lead to the loss of operating vacuum.

4.5 Level Display



Fig. 4: Level Display

Determination of the level in the vessel

- Read the Dewar flask level on the scale in the level display.
- Alternatively, you can use a dipstick to determine the level and compare it with the level curve (see picture of Apollo® 50 level curve).

The black area on the level display scale marks 25% of the remaining contents.

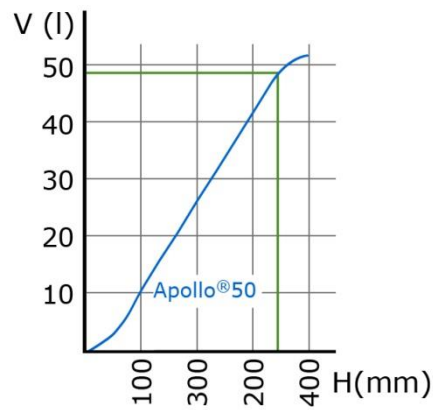


Fig. 5: Level curve Apollo ® 50

4.6 Filling the vessel

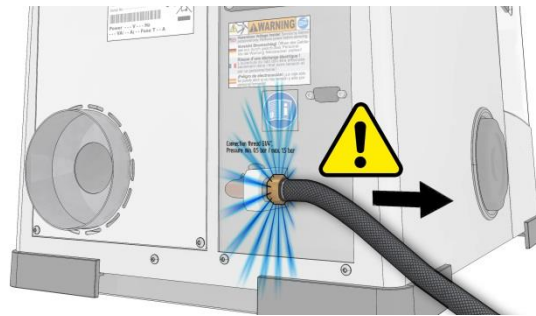


Abb. 4: Removing the liquid nitrogen feed line

CAUTION

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

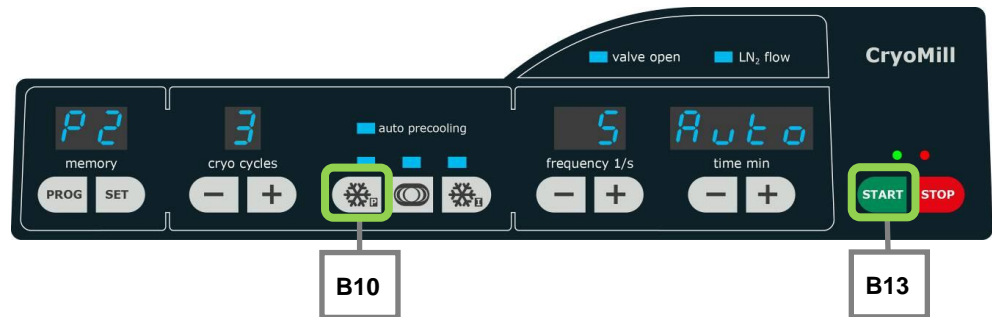


Abb. 5: pressure relief

- 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.

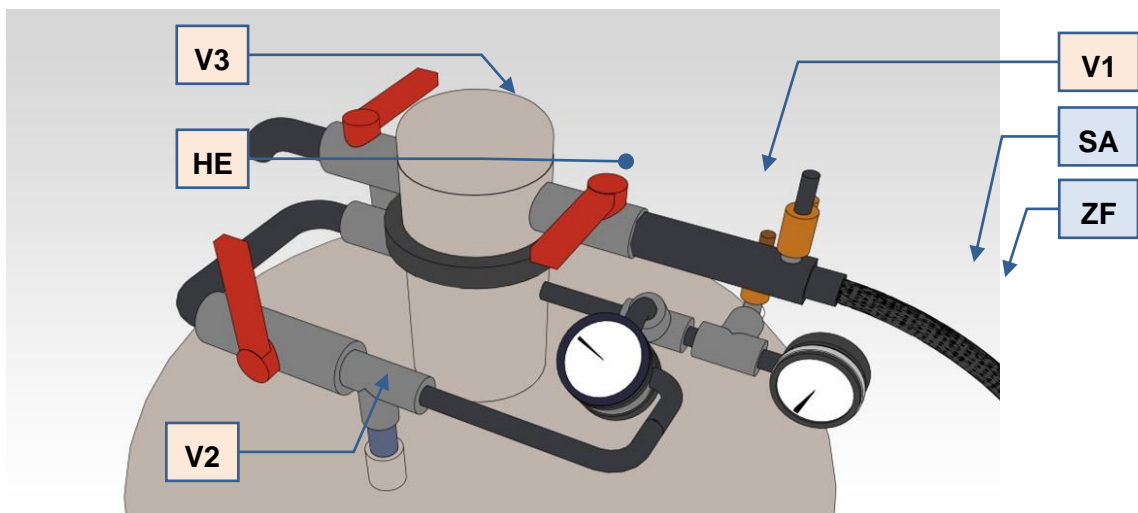


Fig. 6: Front view

NOTICE

Damage to the device

Incorrect filling of the tank

- Foreign particles or humidity and thus the formation of ice crystals may block the pipes and valves of the cooling system, and cause malfunctions.
- **Do not remove the lever (HE) when filling.**
- **Adhere to the specified order when filling the tank.**

- Close all valves. (**V1, V2, V3**)
- Remove the transfer hose (ZF) from the connector (**SA**).
- Connect the filling pipe to the connector (**SA**).
- Open the exhaust and overflow valve (**V3**).
- Open the extraction valve (**V1**).
- Fill the Dewar until liquid nitrogen escapes from the exhaust and overflow valve (**V3**).
- Close the extraction valve (**V1**).
- Close the exhaust and overflow valve (**V3**).

4.7 Pressure build-up

- Close the withdrawal valve (V1) and the waste gas / overflow valve (V3).
- Open the pressure build-up valve (V2).
- Watch the pressure indicator (DM).
- Close the pressure build-up valve (V2) as soon as the needle reaches the red marking or nitrogen comes out of the pressure relief valve (PSV).



Fig. 6: Pressure gauge

4.8 Withdrawal of liquid nitrogen

- Attach the liquid nitrogen line to the CryoMill and to the connection (SA).
- Open the filling and withdrawal valve (V1).
- After taking the liquid, close the filling and withdrawal valve (V1) again.

4.9 Pressure relief

- Close the pressure build-up valve (V2).
- Open the waste gas valve (V3).

4.10 Transport

CAUTION

Risks of liquid or gaseous nitrogen

Positive pressure in the vessel

- The introduction of energy during transport can cause a sudden surge in pressure. Nitrogen is blown off then and this can cause a lack of oxygen, cryogenic burns, oxygen enrichment and associated risks.
- **Make sure the vessel is depressurised before you transport it,**
- **Remove the siphon and put in the transport plugs.**
- **Transport the vessel only in an upright position.**
- **Avoid impacts and vibrations.**
- **Secure the vessel against falling down, rolling away and damage.**

4.11 Service

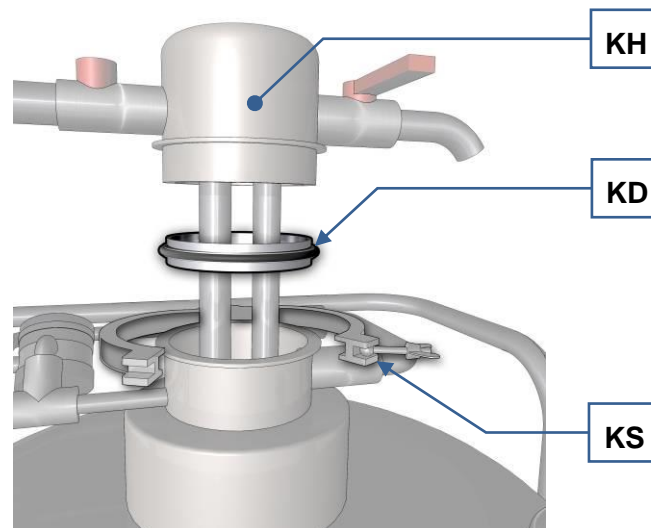


Fig. 4: Sealing ring on the siphon

The sudden temperature differences when filling the dewar with liquid nitrogen can result in wear or signs of aging on the sealing ring (KD) .

- The sealing ring (KD) must be replaced if you notice nitrogen escaping in the area of the tension lock (KS).

NOTICE

Increased frost formation on the tension lock (KS) can already indicate damage to the sealing ring (KD).

CAUTION

Always discharge pressure before replacing the sealing ring.

- Close the pressure build-up valve (V2).
- Carefully open the vent valve (V3).

It is impermissible to use the following agents and tools:

- pliers
- striking tools
- lubricating spray

- hemp
- open flame
- steam
- leak detection spray
- suds

Only the bubble method outlined below may be used to test that the safety valve is securely in place and has the correct response pressure. This prevents the valve mechanics becoming dirty and rusty. The response pressure is marked on the safety valve.

4.12 Admitting the test pressure

Use a suitable test device to introduce the test pressure. Where the safety valves are not to be removed, the vessel's inherent pressure is used for the test.

Do not conduct the tests with oxygen or inflammable or corrosive gases.

4.13 Checking the seat tightness

Increase the test pressure to 90% of the response pressure. The valve must remain leak-tight.

4.14 Checking the response pressure

Slowly increase the test pressure to 100%. The safety valve blows off. The response pressure can deviate from the nominal level by +/- 10%.

5 Index

A

Article number 11

B

Barcode 11

Bubble method 25

C

Causes 13, 14, 15, 16

CE marking 11

Changes 6

Confirmation 10

Copyright 6

Cryogenic burns 14

D

Danger of explosion 15

Dangers 13, 14, 15, 16

Description 19

Dirt 25

E

Explanations of the safety warnings 7

F

Filling the vessel 22

Front view 18, 22

Frost formation 24

Function 19

G

General 12

General conduct in the case of an accident 14, 16

General rules of conduct for splashes with liquid nitrogen 15

General safety instructions 8

Geometrical capacity 20

I

Immersion depth 20

Inherent pressure 25

Instrument name 11

L

Level display 21

Level Display 21

Levels in the vessel 21

M

Manufacturer's address 11

Moderate or mild injury 7

N

Neck diameter 20

Nominal level 25

Notes on the Operating Manual 6

O

Operating pressure 20

Operating the machine 18

Outside diameter 20

Overall height 20

Overall width 20

Overview table of the parts of the device 19

Oxygen deficiency 13

Oxygen enrichment 16

P

Positive pressure 20

Pressure build-up 23

Pressure gauge 23

pressure relief 22

Pressure relief 23

pressure relieve 22

Prohibited agents 25

Prohibited tools 25

property damage 7

R

Rear view 18

Recommendations 13, 14, 15, 16

Re-evacuation 20

Removing the liquid nitrogen feed line 22

Repairs 9

Response pressure 25

Rooms 16

Rust 25

S

Safety and occupational health instructions 11

Safety instructions on the handling of liquid nitrogen 13

Safety warnings 7

Sealing ring 24

Seat tightness 25

Serial number 11

serious injury 7

Service 24

Service Address 9

Static rate of evaporation 20

Surrounding of the machine 16

T

Target group 8

Technical Data 20

Test device 25

Test pressure 25

Transport 24

Type 20

type plate description 11

Type plate marking 11

V

Vacuum closure and safety device 20

Vacuum closure and safety device 20

Vacuum space 20

Valve mechanics 25

Views of the Instrument 18

W

Waste disposal marking 11

Weight empty 20

Weight full 20

Withdrawal of liquid nitrogen 23

Y

Year of manufacture 11



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