

Operation Manual CryoMill
Typ Dewar

Retsch[®]

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1 Notes on the Operating Manual

This operating manual provides all the necessary information on the topics specified in the Table of Contents.

It instructs the target group(s) defined for the respective areas on the safe and purpose-conformant use. Familiarity with the relevant chapter is a precondition for the safe and purpose-conformant use of the machine.

This technical documentation is a reference work and learning guide. The individual chapters are complete by themselves.

This operating manual does not contain any repair instructions. In case of any faults or repairs being required, please contact your supplier or directly get in touch with

Retsch GmbH <http://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

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/operator of the instrument, must ensure that those persons entrusted with working on the instrument:

- have noted and understood all the regulations regarding safety,
- before starting work, are familiar with all the operation instructions and specifications for the target group relevant for them,
- have access to the technical documentation of this instrument at all times and without difficulty,
- new personnel are familiarized with the safe and purpose-conformant handling of the instrument before starting work on the instrument, either by a verbal introduction by a competent person and/ or by means of this technical documentation, .
- improper operation can result in harm to persons, damage to property and injuries. You are responsible for your own safety and that of your co-workers.
- Ensure that no unauthorized persons have access to the instrument.

For your own protection, have your induction into the operation of the instrument confirmed by your co-workers. The draft for a suitable form can be found in the chapter "Confirmation".



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 (Form for the operator)

I have taken note of the section „Notes on these operating instructions” and the section on “Safety”.

Signature of operating authority

Signature of service technician

3 Container for the storage of liquid nitrogen LN2

3.1 Design of a functioning container

3.1.1 Description

The container consists of two components:

- upright container for storing LN2,
- siphon head for transferring LN2, consisting of an LN2 valve and a vent valve, a manometer and an overpressure valve.

3.1.2 The container

The container consists of two shells made of aluminium alloy that are connected by a neck made of a fibreglass-epoxy compound material.

A high vacuum between the two shells and multilayer insulation (super-insulation foil) guarantee thermal insulation in the vacuum space.

Two handles and an NW 50 flange for attaching the siphon head with a clamp are located on the upper part of the container.

3.1.3 The siphon head

3.1.4 View of the container with siphon

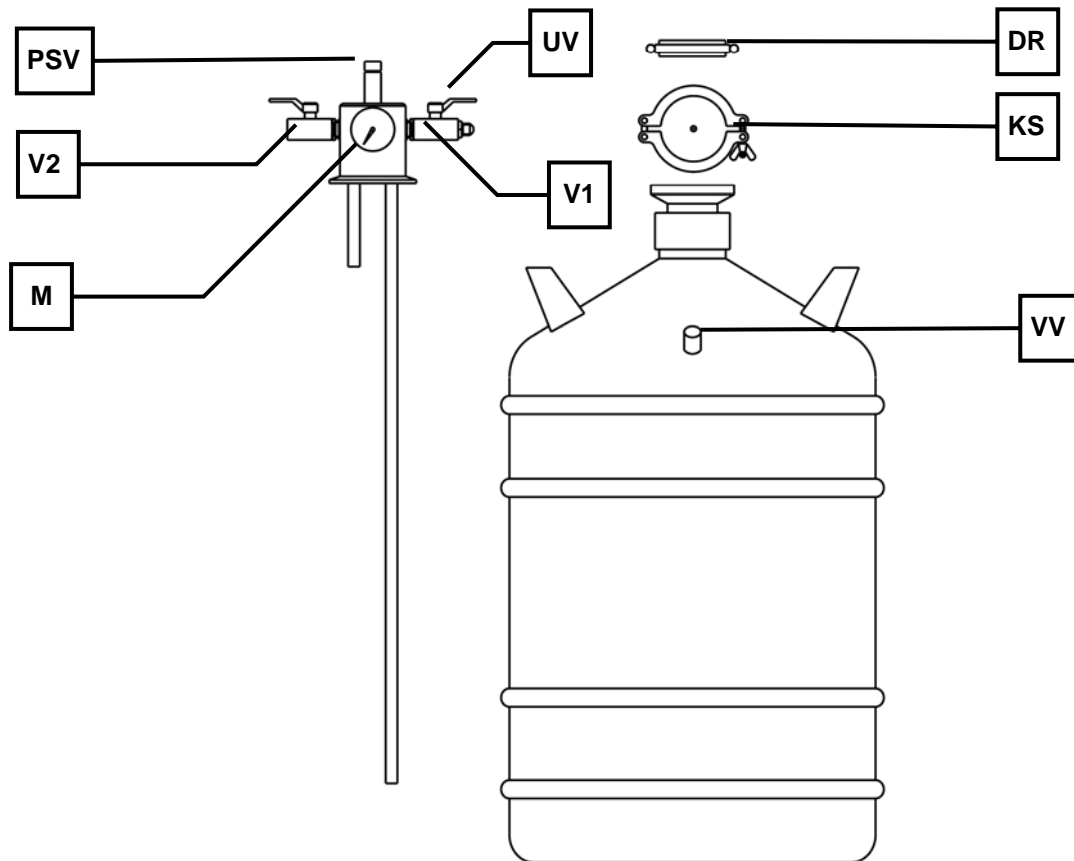


Fig. 1: View of Dewar for liquid nitrogen with siphon

Element	Description	Function
DM	Manometer	Display of the
PSV	Safety valve	Safety valve 0.5 bar
V1	Transfer valve	Valve for liquid nitrogen transfer
V2	Vent valve	Valve for liquid nitrogen venting
KS	Quick release clamp	Sealing and fixing of the siphon to the Dewar
DR	O-ring with centering ring	Sealing
UV	Screw adapter	Connection of liquid nitrogen line to the CryoMill
VV	Vacuum valve	Sealing of the high vacuum in the intermediate cavity

3.1.5 Technical data of the Dewar

Liquid container made of aluminium for the storage and transport of liquid nitrogen

Type	ALU 10
Capacity	12 l
Operating overpressure	0.5 bar
Weight empty	7.5 kg
Weight full	17.5 kg
Overall height	584 mm
Diameter	308 mm
Evaporation rate static	0.2 L / day
Static holding time	67 days

Daily evaporation and static holding time were determined at 20°C, 1013 mbar, container stationary, lid closed and without transfer siphon. These are nominal values that may change depending on the age and use of the container

3.2 Dewar setup instructions

3.2.1 Precautions when setting up the container

You must read the attached Safety Regulations prior to setting up the container or prior to first use. Please pay particular attention to the following important items:

Nitrogen is neither toxic nor flammable, but it may cause an oxygen deficiency in closed spaces.

For that reason, we recommend that containers be used that are filled with liquefied gas (especially if liquefied gas is being transferred to an open container) in a room with sufficient ventilation.

Always have either the siphon head or the plug inserted in the container.

Store it protected from weather damage.

3.2.2 Mounting the siphon head

The siphon head is mounted on the LN2 container as follows:

- a) Open vent valve V2 and close transfer valve V1 (these steps are especially important if the container is filled with liquid nitrogen).
- b) Put the centering ring with O-ring (**DR**) on flange DN 50 of the LN2 container.
- c) Place the siphon head on the centering ring, making sure not to bump against the neck of the inside container and that the siphon head sits in the center of the sealing ring.
- d) Attach the quick release clamp (**KS**) and connect the siphon head to the container.



Risk of ice forming

- There is a risk of ice forming in the tubing or in the safety equipment. These may get plugged with ice and consequently cause operating failures.
- **Before assembling the siphon head, remove any traces of moisture by blowing out the tubes and valves with nitrogen or dry air.**

3.3 Operating instructions of the Dewar

3.3.1 Transport of the Dewar

LN2 containers may be transported only as open, depressurized containers, i.e., without transfer siphon (with the necktube core), if they are filled with LN2.

The pressure inside the container must be equivalent to the atmospheric pressure. To achieve this, remove the siphon head and put on the plug. This will reduce leakage and prevent moisture from entering.

3.3.2 Handling the Dewar

The containers have been designed to be resistant to minor blows that cannot be avoided during handling. However, to minimize leakage and to ensure a long service life of the container, we strongly recommend the following:

- avoid major impacts,
- keep the container always in an upright position,
- transport the containers only within the laboratories (do not use them as containers for mechanically robust transports).

3.3.3 Connections of the Dewar

The screw adapter (ÜV) attached to transfer valve V1 allows different accessories, e.g. transfer hose or transfer tube, to be connected to the LN2 container:

Thread of the screw adapter (ÜV) :
3/4" / 16 UNFpitch thread.

3.3.4 Filling of the Dewar



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.**



3.3.5 Filling

The LN2 container is filled either with

- a) a filling hose introduced into the neck of the container, or
- b) a filling flange with a filling hose

3.3.5.1 Filling using a filling hose on an open container

- Make sure that the container is depressurized and the vent valve V2 on the siphon head is open.
- Remove the quick release clamp (KS).
- Carefully remove the siphon head.
- Insert the filling hose and let the liquid nitrogen flow into the container up to the desired filling level. (maximum level: lower end of the neck).

Make sure that liquid nitrogen cannot get on to the vacuum valve (VV); if necessary, cover the vacuum valve.

- After filling the container with LN2, put on and connect the siphon head and quick release clamp carefully and slowly.



Do not forget to first open valve V2 and to close valve V1.

The amount of introduced liquid can be checked by weighing the container. One litre of liquid nitrogen weighs approx. 0.808 kg under atmospheric pressure. The maximum filling level has been reached if in case of ALU 10 approx. 8 kg have been transferred.

3.3.5.2 Filling using a filling flange

Siphon head and filling flange are attached to the container.

Make sure that the container is depressurized. If not:

- Open vent valve V2 on the siphon head and slowly release the excess pressure.
- Also open the vent valve (BV2) on the filling flange.
- Connect the filling hose to the screw adapter (ÜV) of the filling flange and open the LN2 filling valve (BV1).en.
- Slowly push the LN2 reservoir tank with transfer siphon to the container to be filled and secure it.



The pressure in the reservoir tank should not exceed 1.3 bars.

- Connect the filling hose to the screw adapter (ÜV) of the transfer siphon of the reservoir tank.
- Open the LN2 valve (V1) on the reservoir tank and fill the container. The pressure in the container may not exceed 0.4 bars during the filling process (see manometer on the transfer siphon)
- If LN2 appears in addition to N2 gas on the vent valve of the filling flange, the LN2 valve (V1) on the reservoir tank is closed immediately.



Carry out LN2 filling under supervision only.

- After completion of the filling process, remove the transfer hose from the full container and close the filling valve (BV1).
- Close the vent valve on the siphon head of the container (V2) and on the filling flange (BV2).

- Remove the reservoir tank and wait until the container has reached its service pressure.

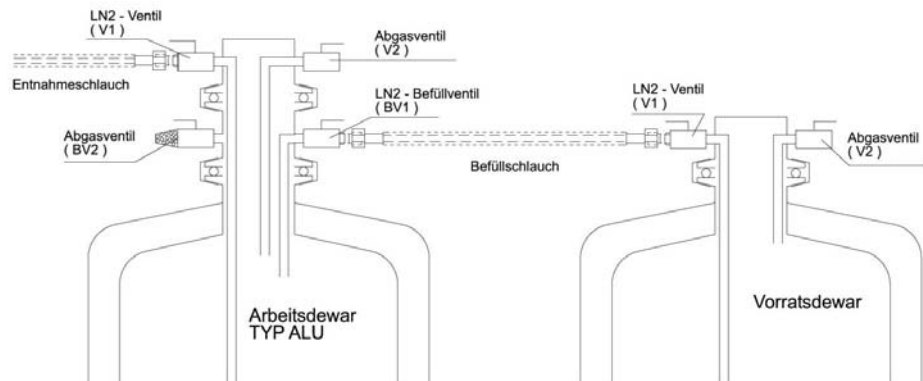


Fig. 2: Dewar fill connection diagram

3.4 Extraction of liquid nitrogen

If the container is not connected to the transfer siphon yet, do the following:

- Place the LN2 transfer siphon on the container where the centering ring and the O-ring have already been placed. Secure it with the quick release clamp.
Do not forget to first open vent valve V2 and close LN2 transfer valve before placing the transfer siphon on the container.
- Connect the transfer hose or tube to LN2 transfer valve V1.
- Close vent valve V2.
- Wait for internal pressure to build up.
- Can be extracted after the internal pressure has been built up.

INSTRUCTIONS:

Do not use the container unprotected outdoors during rain. The container and the transfer siphon must never come into contact with water while being used.

When mounting or removing tubes or hoses, you should always make sure that the tubes or hoses being attached to the LN2 transfer valve (V1) of the container are blown out first with N2 gas or dry air so that any moisture that may have collected in them is removed.

3.5 Checks

3.5.1 Inspection of the fittings



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.**



3.5.2 Inspection of the fittings

Fittings and valves must be inspected regularly or after any operational incident. We recommend having the inspection carried out by a specialist.

Inspection frequency: at least once a year.

3.5.3 Checking the manometer

Tools required: a manometer calibrated as a reference

- There must be enough liquid nitrogen in the Dewar.

Procedure: close valves V1 and V2 of the transfer siphon. Let the pressure in the container build to a certain value, e.g. 0.4 bar. Connect the calibrated reference manometer to the V2 valve and then open this valve.

Both manometers (**Ma**) should read out the same value.

If the pressure values differ by more than 0.1 bar, the manometer should be replaced.

3.5.4 Checking the overpressure valve V3

Tools required: a manometer calibrated as a reference

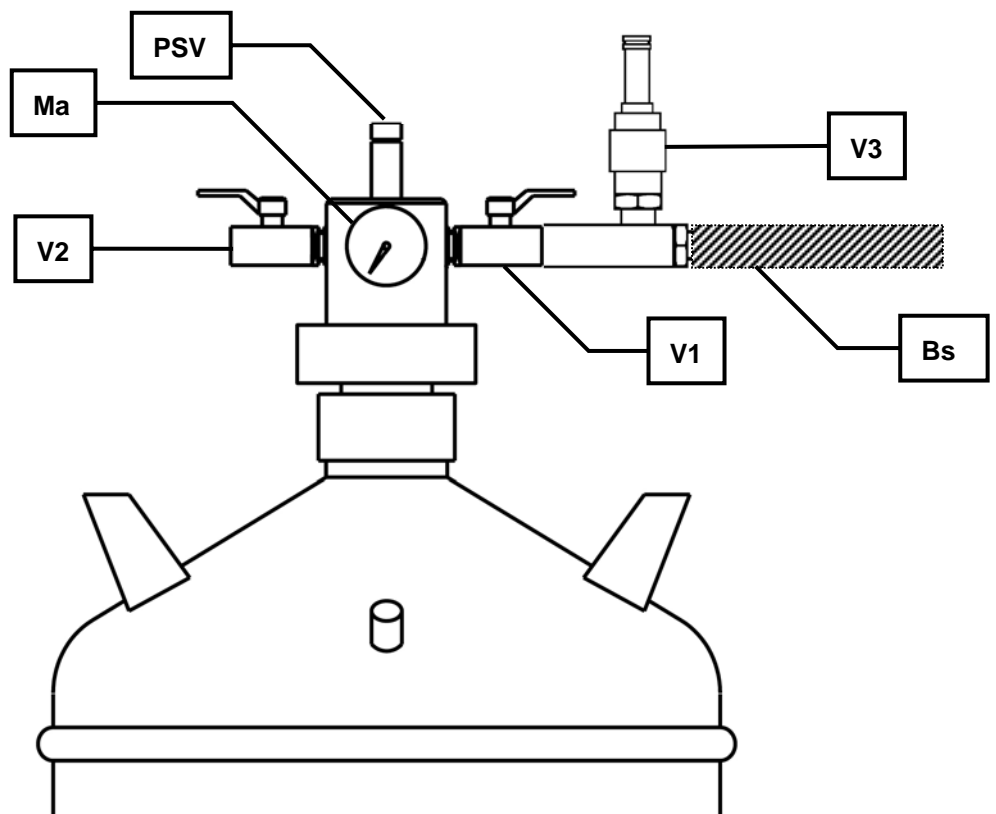


Fig. 3:

- Close valves (V1) and (V2).
- Press the buttons precooling (B10) and START (B13) on the CryoMill simultaneously until liquid nitrogen no longer flows from the cooling system.

The filling hose (**Bs**) between the liquid nitrogen storage container and the CryoMill is now empty.

- Screw off the filling hose (**Bs**) from the CryoMill housing.
- Connect the free end of the filling hose (**Bs**) with a 3-way stopcock (thread type: G1/4 ")
- Connect a calibrated reference manometer to one of the two free ends of the 3-way stopcock.
- Set the 3-way stopcock such that the remaining end is open.

 **CAUTION**

Liquid nitrogen flows from the 3-way stopcock.

Make sure that no-one comes into contact with the escaping liquid nitrogen.

Always wear goggles and protective gloves when handling liquid nitrogen.

- Open the valve (**V1**).
- As soon as liquid nitrogen escapes close the 3-way stopcock.
- Close the valve (**V1**).
- Wait until internal pressure builds up.

At a pressure of 1.5 bars the overpressure valve (**V1**) must open.

If the pressure displays on the reference manometer deviates from this value by more than 0.1 bars the valve (**V1**) must be replaced.

3.5.5 Checking the overpressure valve (PVS)

Procedure: close valves V1 and V2.

Wait for internal pressure to build up.

Note the value of the pressure on opening the overpressure valve (PSV).

If this pressure value is not between 0.45 and 0.55 bars, replace valves.

Allow pressure to slowly drop in the container by gradually opening V2 (make sure that no one is located in the axial direction of valve V2).

Note pressure value on closing the valve. If this value is below 0.45 bars, replace valve.

4 Warranty conditions

1. If legitimate claims are made we shall remedy the defect or replace the goods free of charge.
The purchaser shall only have a right to rescind the contract or reduce the purchase price if we have decided that it is not possible to remedy the defect and a replacement delivery cannot be made or the time limit therefore cannot be complied with or if a reasonable additional time limit of six weeks granted by the customer has not been complied with due to our fault.
If the remedy or replacement delivery in fact fails the customer shall have the right to reduce the price or rescind the contract at his discretion. Further claims, in particular for damages in relation to damage not caused to the goods themselves, such as lost production, are excluded in so far as we have not acted wilfully or negligently. For goods produced by third parties we pass on the liability of the manufacturer.
2. We shall bear the costs directly incurred through the remedying of defects or the replacement delivery on the condition that claim is found to be legitimate. This also applies to the freight costs as well as the reasonable costs of removal and installation. The customer, however, undertakes to bear the reasonable costs of providing his own technicians and assistants on site.
If our customer carries on business overseas, however, we shall be entitled to pay the costs, in particular costs of transport, tolls, wages and materials, ex German border.
3. The warranty term for newly manufactured goods is two years, for used it is one year.
The guarantee refers to deployment in a laboratory in 1-shift operation. In case of multi-shift operation or other areas of application, the guarantee term is shortened accordingly.
No warranty is given for parts subject to wear and tear.
4. We warrant that our goods are free from manufacturing defects. The suitability, classification and function of our goods are determined exclusively on the basis of the performance descriptions contained in the order confirmation even if these differ from the order. In the latter event the customer may, within two weeks after receipt of the order confirmation, draw any possible difference from the order to our attention and come to an agreement on these with us. If the customer does not object to the specifications in the order confirmation then these shall be deemed to have been accepted.
Unless an agreement to the contrary has been reached, we shall not be held liable for the suitability of the goods delivered for the use to which the customer intends to put them. The same applies to performance figures expected by the customer unless we have been able to carry out appropriate preliminary practical experiments and have, in our order confirmation, declared in writing that these performance figures shall be binding.
5. Our warranty shall also become invalid if persons other than those employed by us carry out repairs or in any other way interfere with or make alterations to the goods delivered by us or do not use suitable parts to the extent that the defect is causally connected thereto. In addition, it is a condition of our warranty that our directions for use and operation be followed.
6. If, without a release having first been obtained from us, the goods are installed in and /or connected to, attached to or incorporated in other systems or production plants then our guarantee is limited exclusively to the parts delivered by us.
7. The remedying of defects or replacement of defective parts shall, at our discretion, be carried out on site or at the seat of our company. If the repair is carried out on site, the customer shall ensure that our employee has access, unlimited in either time or space, to the purchased item. In addition, the customer may only demand that work necessary in order to fulfil warranty obligations be carried out during the normal local business hours. If such work is carried out outside our normal business hours on request, the customer shall bear the additional costs. If he wishes to have other particular work performed which goes beyond the work warranted then these costs shall be payable at the actual valid price.

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