Operating Manual

ALU 7 - ALU 10 - ALU 20 - ALU 26 - ALU 35 - ALU 60 - ALU 100

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General safety regulations for handling LN2 are to be followed !

1 Design of a Workable Container

1.1 Description

The container consists of two components:

* **Upright container for storing LN2**
* **Siphon head for transferring LN2, consisting of a LN2-transfer valve, a vent valve, a manometer and an overpressure valve**

1.2 Container

The container consists of two shells out of aluminium alloy that are connected by a neck out 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.

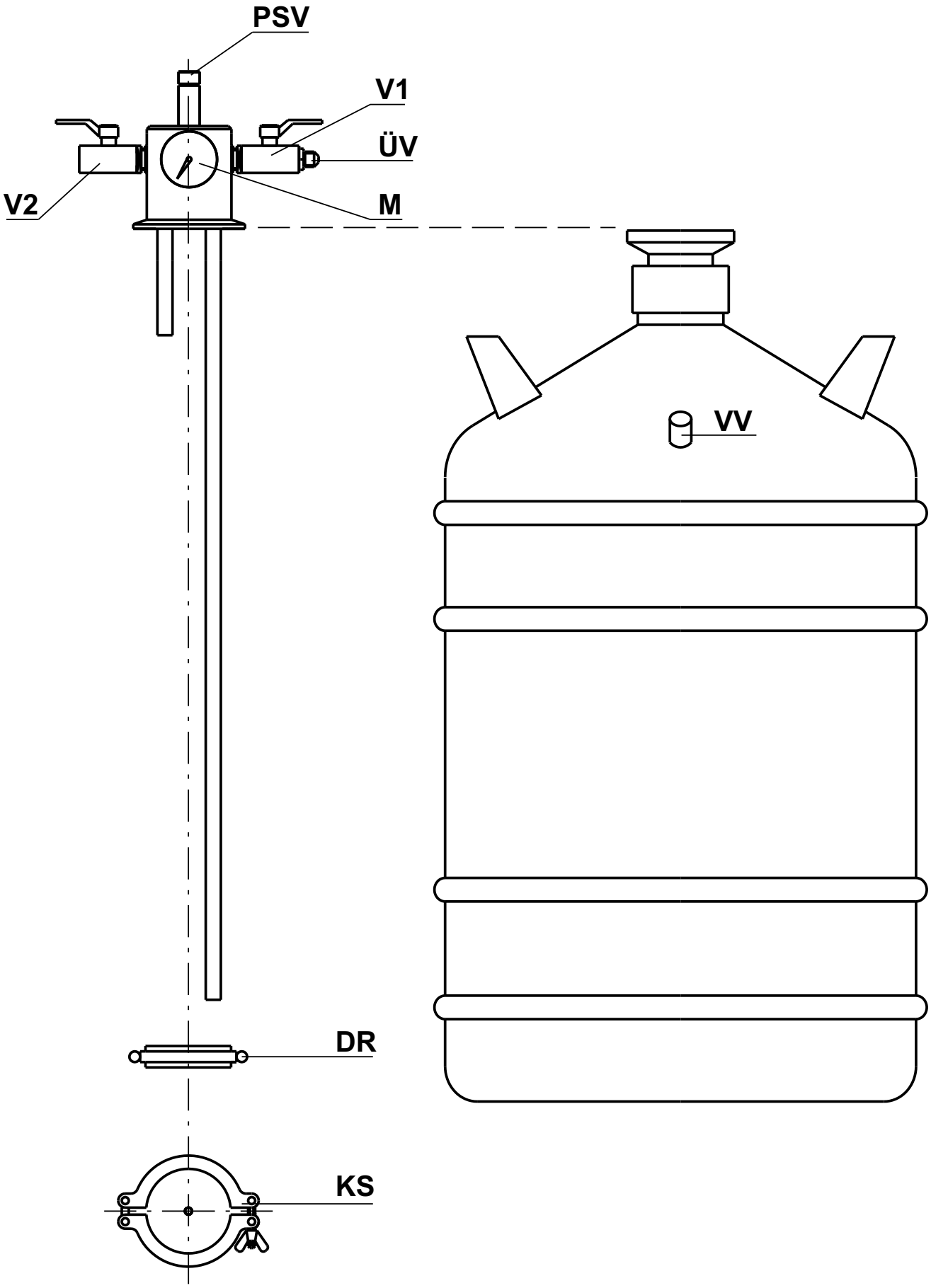
Two side grips (ALU 7 and ALU 10 with carrying handle) and a KF-NW50 flange for mounting a siphon head with a clamp are located on the upper part of the container. The container is equipped with a combined vacuum/overpressure valve (VV).

1.3 Siphon head

The siphon head consists of the following:

* **Manometer (M)**
* **0.5 bar overpressure valve (PSV)**
* **LN2-transfer valve (V1)**
* **Vent valve (V2)**
* **Clamp (KS)**
* **O-ring with centring ring (DR)**
* **Screw adapter (ÜV)**

1.4 Drawing of container with transfer siphon



1.5 Specifications

Technical data								
Type ALU		7	10	20	26	35	60	100
Art.-No.		2515	2516	2517	2518	2519	2521	2522
Capacity	(l)	7	12	21	26	34	60	99
Operating overpressure	(bar)	0,5	0,5	0,5	0,5	0,5	0,5	0,5
Weight empty	(kg)	6	7,5	11	13,5	16	21,5	29,5
Weight full	(kg)	12	17,5	28,5	34,5	43	70	110
Overall height	(mm)	480	584	605	670	655	870	986
Diameter	(mm)	308	308	388	388	468	468	510
Evaporation rate static	L / Tag)	0,2	0,2	0,18	0,2	0,24	0,4	0,55
Static holding time (days)		36	67	119	130	140	150	180
Roller base				2640	2640	2641	2641	2642

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.

2 Setup Instructions

2.1 Precautions for setting up the container

You must follow general (and company internal) safety regulations for handling with LN2 prior to setting up the container or using it for the first time. Please pay particular attention to the following important items:

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

This is why we recommend to use containers holding liquefied gas (especially if liquefied gas will be transferred to an open container) in a room with sufficient ventilation, only.

Container should always be equipped either with a siphon head or a plug.

Store it protected from weather damage.

2.2 Mounting the siphon head

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

a) Open vent valve (V2) and close LN2-transfer valve (V1) (these steps are especially important, if the container is already filled with liquid nitrogen).

b) Put the centring ring with o-ring (DR) on KF-NW50 flange of the LN2 container.

c) Place the siphon head on the centring ring, making sure not to bump against the neck of the inner container and that the siphon head sits in the centre of the o-ring.

d) Attach the clamp (KS) and connect the siphon head to the container.

Warning:

Before assembling the siphon head, remove any traces of moisture by blowing out the tubes and valves with nitrogen or dry air. This precaution is absolutely necessary so as to prevent any risk of ice forming in the tubing or in the safety equipment, which might be plugged by ice and consequently cause operating failures.

3. Operating Instructions

3.1 Transport

LN2 containers may be transported only as open, depressurised containers, i.e., without transfer siphon (but with transport plug), 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 the loss and prevent moisture from entering.

3.2 Handling

The containers are designed to be resistant to minor impacts that cannot be avoided during handling. However, to minimise the loss and to ensure a long service life of the container, we strongly recommend the following:

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

For ease the transport of containers in the laboratory, we can provide a roller base as an accessory (see chapter 6). This detachable roller base is connected to the container. The container with roller base can be lifted to move it over small uneven areas (steps, edges etc.).

3.3 Connections

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

The screwthread connection of screw adapter (ÜV): UNF - 3/4" / 16 convolutions.

Always wear protective gloves and goggles when handling liquefied gas.

3.4 Filling

The LN2 container is filled either with:

1) a transfer hose inserted into the neck of the container

2) help of LN2-Level Control Unit

3) help of a filling flange with a filling hose (2606 and 2607).

3.4.1 Filling by using a transfer hose on an open container

a) Make sure that the container is depressurised and that vent valve (V2) on the siphon head is opened.

b) Remove the clamp (KS).

c) Carefully remove the siphon head.

d) Insert the transfer hose and let the liquid nitrogen flow into the container up to the desired level. (maximum level`: lower end of the neck.)

Make sure that liquid nitrogen will not run over the vacuum valve (VV); if necessary, cover the vacuum valve.

e) After filling the container with LN2, carefully and slowly put on and connect the siphon head and clamp as described in chapter 2.2.

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

The amount of inserted 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 7	**= approx**	**5.6 kg**
ALU 10	**= approx**	**8 kg**
ALU 20	**= approx**	**16 kg**
ALU 26	**= approx**	**21 kg**
ALU 35	**= approx**	**28 kg**
ALU 60	**= approx**	**47 kg**
ALU 100	**= approx**	**78 kg**

has been transferred.

3.4.2 Filling with help of LN2-Level Control Unit

The siphon head is attached to the container. Make sure that the container is depressurised. If not, please:

a) Open vent valve (V2) and release the overpressure slowly .

b) Open the clamp (KS) and remove the siphon head from the container.

c) Put on the level-filling head and secure it with the clamp (KS). There is no need to use the centring ring with the o-ring.

d) Insert the level probe into the level-filling head and secure it. Connect the filling hose (2607) to the level-filling head.

e) Slowly move the LN2-reservoir tank with transfer siphon and the LN2-magnetic valve to the container that should be filled and secure it.

WARNING: The pressure in the reservoir tank should not exceed 1.3 bar.

f) Connect the filling hose (2607) to the LN2-magnetic valve.

g) Connect the LN2-magnetic valve and the min/max probes with the Level Control Unit.

h) Turn on the LN2-Level Control Unit. The filling process will be started.

Automatic filling a container under supervision, only.

The LN2-magnetic valve is open during the filling process. As soon as the LN2 level in the container being filled has reached the maximum probe of the Level-Control Unit, this unit closes the magnetic valve and stops the filling process.

Once the maximum filling level has been reached in the container and the Level-Control Unit has closed the magnetic valve, the container can be disconnected from the LN2-reservoir tank.

a) After the filling process is completed, remove the filling hose from the full container and disconnect the level probe from the Level-Control Unit.

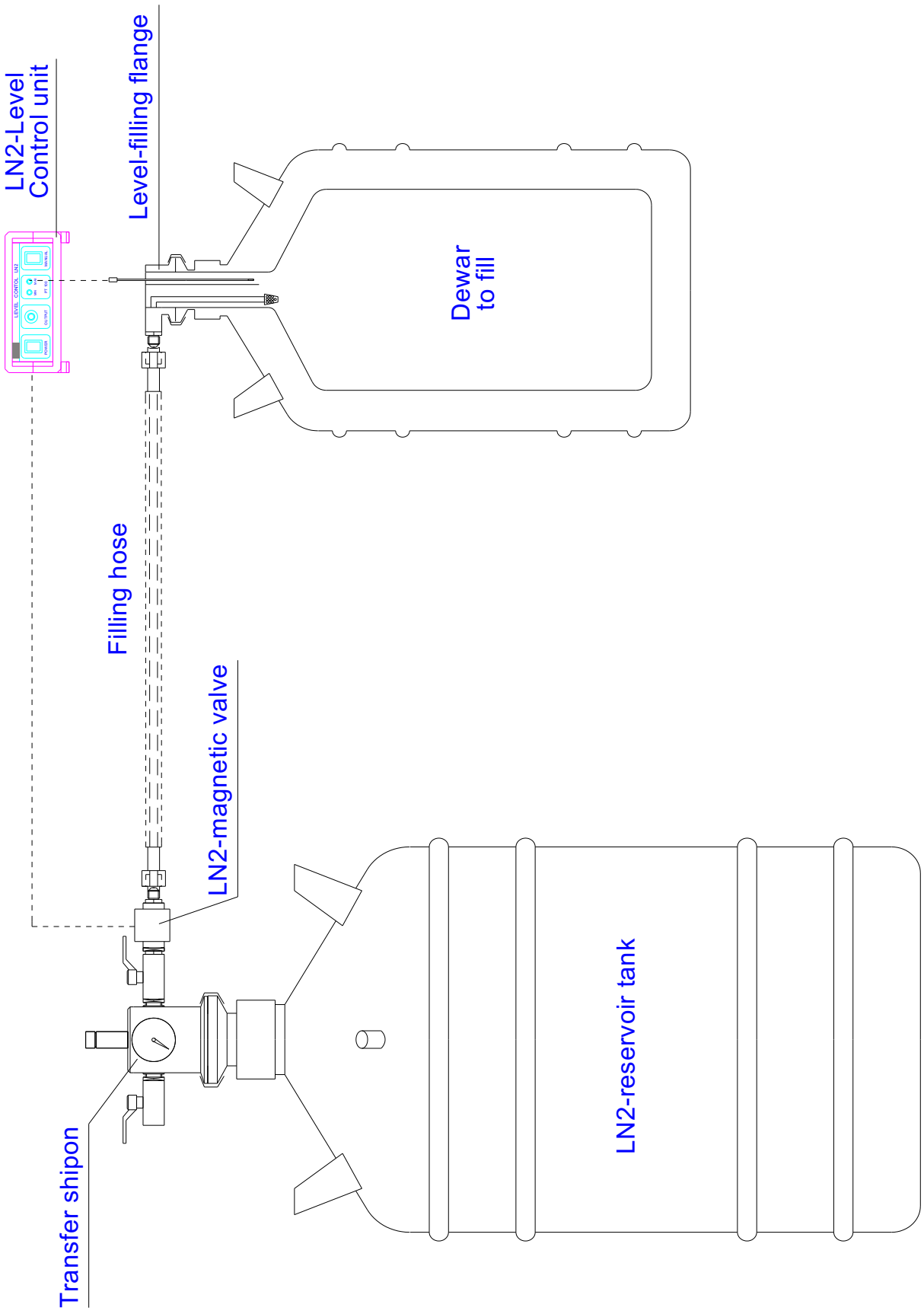
b) Open the clamp (KS) and remove the level-filling head from the container.

c) Close LN2-transfer valve (V1) on the EKI-siphon head and open vent valve (V2).

d) Place the siphon head with the centring ring on the container flange and secure it with the clamp. Close the vent valve (V2).

e) Connect a transfer tube or transfer hose and wait until the container has reached a suitable working pressure.

Drawing of filling with help of LN2-Level Control Unit



3.4.3 Filling with help of a filling flange

The siphon head and the filling flange are attached to the working Dewar. Make sure that the container is depressurised. If not, please:

a) Open vent valve (V2) on the siphon head and release the overpressure slowly.

b) Open the vent valve (BV2) on the filling flange.

c) Connect the filling hose (2607) to the screw adapter (ÜV) of the filling flange and open the LN2-filling valve (BV1).

d) Slowly move the LN2-reservoir tank with transfer siphon to the container to be filled and secure it.

WARNING: The pressure in the reservoir tank should not exceed 1.3 bar.

e) Connect the filling hose (2607) to the screw adapter (ÜV) of the transfer siphon of the LN2-reservoir tank.

f) Open the LN2 valve (V1) on the LN2-reservoir tank and fill the container. The pressure in the container may not exceed 0.4 bar during the filling process (see manometer on the transfer siphon).

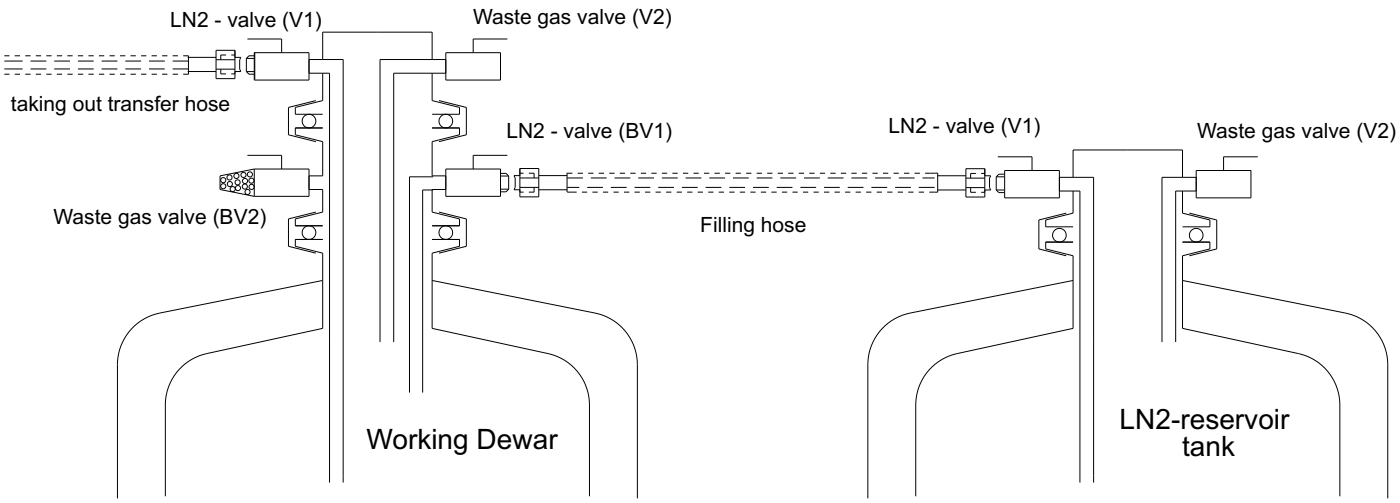
g) If LN2 appears in addition to N2 gas on the vent valve (V2) on the LN2-transfer siphon of working Dewar, the LN2 valve (V1) on the reservoir tank must be closed immediately.

Fill LN2 into containers under supervision, only.

h) After the filling process is completed, remove the transfer hose from the full container and close the filling valve (BV1).

i) Close the vent valve on the siphon head of the container (V2) and on the filling flange (BV2).

j) Remove the LN2-reservoir tank and wait until the container has reached a suitable working pressure.



4. LN2 Transfer

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

a) Place the EKI-transfer siphon on the container where the centring ring with o-ring have already been placed. Secure it with the clamp.

Do not forget: Before placing the transfer siphon on the container, you first have to open vent valve (V2) and close LN2-transfer valve (V1)!

b) Connect the transfer hose or transfer tube to LN2-transfer valve (V1).

c) Close vent valve (V2).

d) Wait for the internal pressure to be built-up.

e) LN2 can be extracted after the internal pressure has been built up.

Note:

Do not use the container unprotected outdoors during rain. The container and the transfer siphon must never get in contact with water during use.

When mounting or removing transfer tubes or transfer hoses, always make sure that the tubes or hoses being attached to the LN2-transfer valve (V1) of the container are blown out first. You can use N2 gas or dry air so any moisture that may have accumulated in there will be removed.

5 Maintenance

5.1 Inspecting the valves and fittings

The valves and fittings must be inspected regularly and after any operational incident.

Inspection frequency: at least once a year.

5.1.1 Checking the manometer

Tools required: a calibrated manometer as reference.

Proceed as follows:

Close valves (V1 and V2) of the transfer siphon.
Let the pressure in the container build up to a certain value, e.g., 0.4 bar.
Connect a calibrated reference manometer to the valve (V2) and open this valve.
The two manometers should show the same value.
If both pressure valves differ more than 0.1 bar, the manometer should be replaced.

5.1.2 Checking the overpressure valve

Tools required: none, if manometer has been checked before

Proceed as follows:

Close valves (V1 and V2).
Wait for the internal pressure to build up.
Note the pressure value at the time the overpressure valve (PSV) is opened.
If this pressure value is not in between 0.45 and 0.55 bar, the overpressure valve must be replaced.
The internal pressure of container has to be released carefully by opening valve (V2) slowly in small steps (make sure nobody is standing in the axial direction of valve (V2)).

5.2 Leak test

Connect an N2-gas cylinder with pressure-reducing device to vent valve (V2) and set the pressure at 0.4 bar. Close valve (V1) and open valve (V2) until the container has reached nitrogen-gas pressure of 0.4 bar.

Prepare a soap-water mixture in a cup or use a ready-to-use solution (leak detector spray).

Apply the soap-water mixture to the individual connections by using a brush. Bubbles are a sign for an existing leak.

Mark the leak and replace the leaking part.

5.3 Checking the evaporation rate

The evaporation rate is checked by weighing the LN2-filled container. You will need a scale with a weighing range that corresponds to the weight of container and that weighs with sufficient accuracy (i.e., where the weighing inaccuracy is insignificant compared to the differences between the actual weighings, see below).

Proceed as follows:

- **Put the plug on the container (the evaporation rate is always checked under atmospheric pressure).**
- **Weigh the empty container.**
- **Fill the container with liquid nitrogen, e.g., 30 kg / LN2 for the ALU 60.**
- **Wait 3 to 4 days until the temperature of the container has stabilised.**
- **Weigh the container: mass M1.**
- **Weigh the container again after 24 hours, : mass M2.**
- **For a new container, the difference between the two weighings M1 and M2 must equal the following: 330 g +/- 10% for the ALU 60.**

If the precision of scale is not sufficiant (e.g., if its readability is 100 grams), both weighings must be done in a time frame that will render the measurement inaccuracy negligible with respect to the difference between M1 and M2.

Note:

To receive a significant measurement result, the room temperature must be at a constant 15°C, and the atmospheric pressure has to be 1013 mbar consistently during the measuring interval. Of course, the conditions and the age of container will have a significant influence to the evaporation rate.

5.4 Replacing the valves and fittings

The container has to be empty and warm (room temperature) whenever the valves or fittings should be replaced.

5.4.1 Replacing the manometer and the overpressure valve

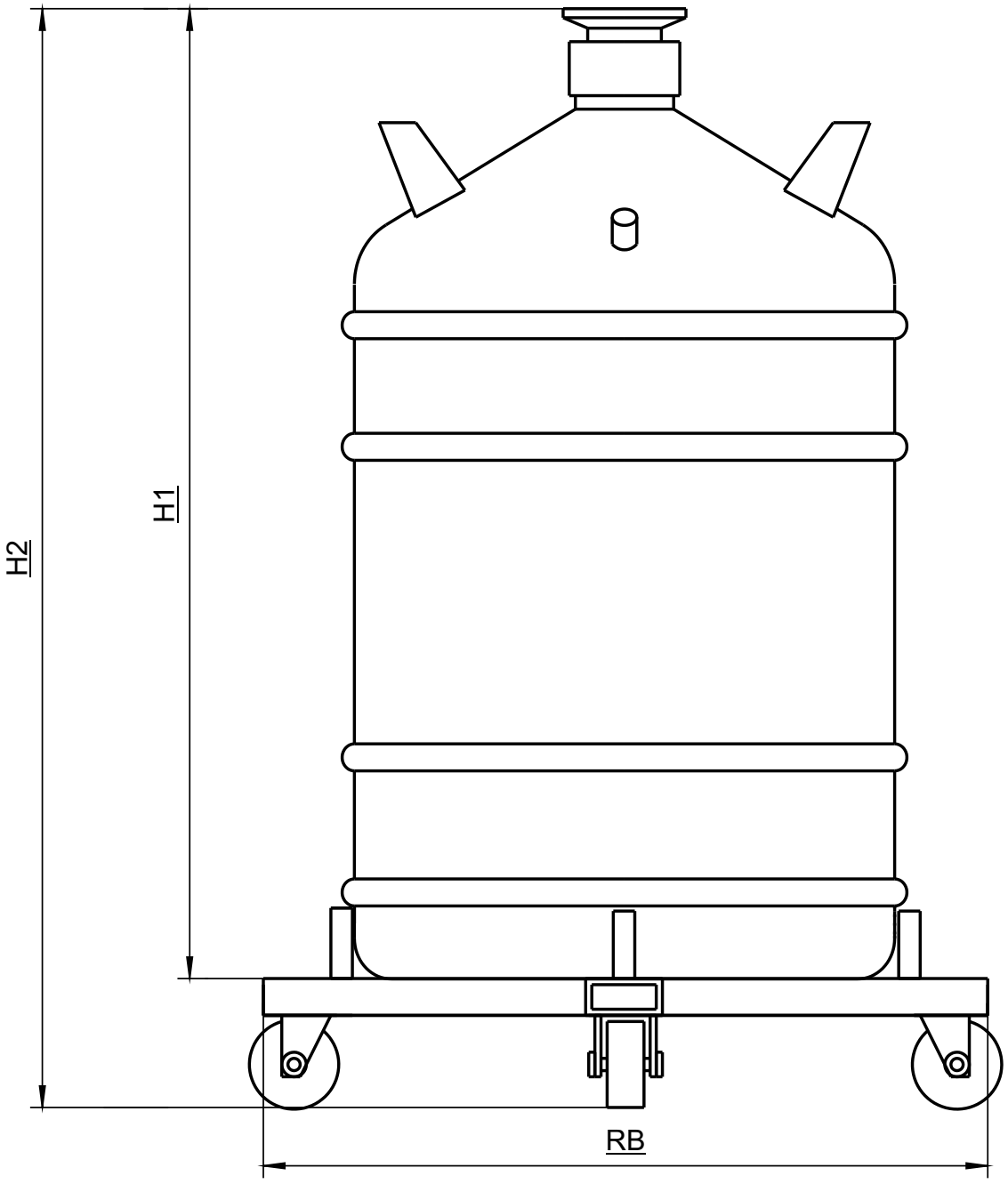
- **Open vent valve (V2) to release any pressure.**
- **Remove the defective part, making sure not to loosen any other parts.**
- **Thoroughly clean the female thread to remove all traces of the sealing compound.**
- **Apply PTFE tape or sealing compound, making sure not to clog the openings on the spare part's threads.**
- **Screw on the part without damaging the threads. Perform a leak test (please see chapter 5.2).**

5.4.2 Replacing the valves

- **Remove the vent valve or the LN2-transfer valve and thoroughly clean the female thread to remove all traces of sealing compound.**
- **Mount the vent valve or the LN2-transfer valve (inclusive already mounted screw adapter) using PTFE tape or sealing compound.**
  Mounted valves have to be secured by lock nuts so that they will not loosen when mounting or dismounting a transfer hose afterwards.
- **Place the siphon head with the new valves on the container and secure it with the clamp.**
- **Afterwards, a leak test has to be made (please have a look at chapter 5.2).**

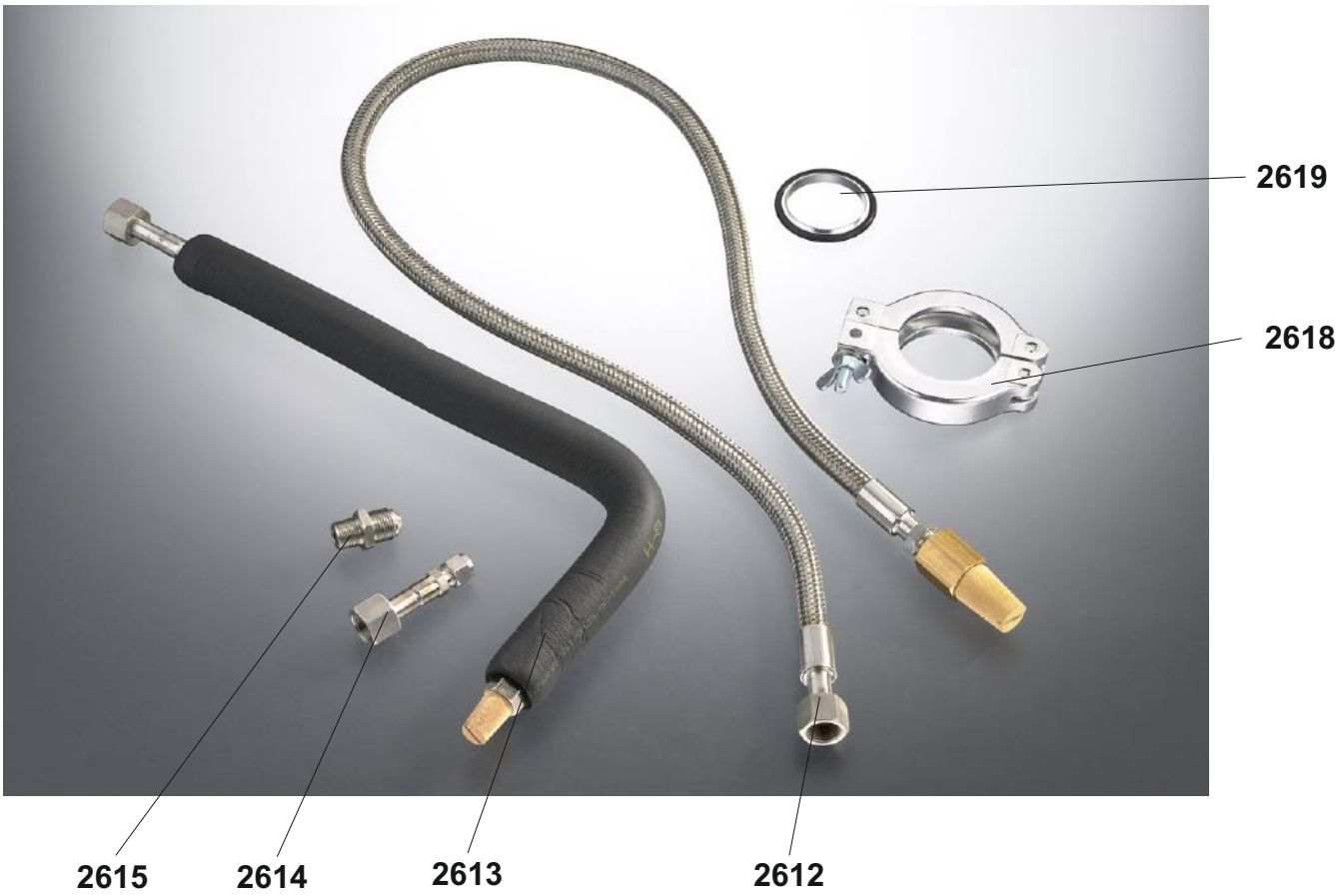
6. Roller Base

Containers can be equipped with a roller base for an easier in-house transport. The roller base is a detachable accessory; it is only attached to the container. The mounted roller base is fixed on the container.



Typ ALU	20	26	35	60	100
H 1 (mm)	605	670	655	870	986
H 2 (mm)	825	890	875	1090	1206
RB ca.mm	480	480	570	570	610

7. Spare Parts and Accessories



Special transfer siphon

This modified EKI-transfer siphon has a heating element; its purpose is to fasten the overpressure build-up inside of container by electric evaporation.

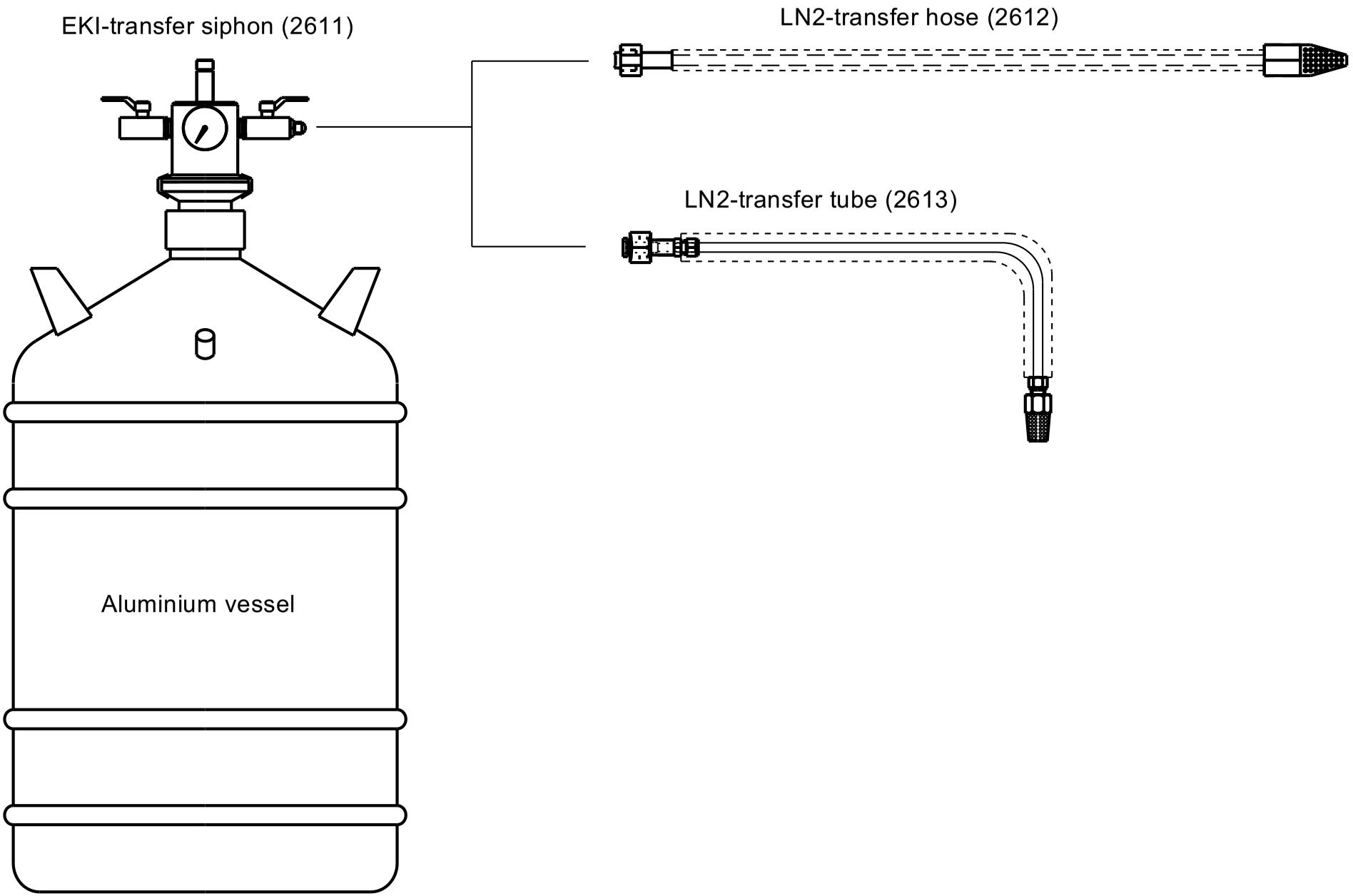
Art. no. 2611-H

7.1 Spare parts for EKI-transfer siphon

DESCRIPTION	ARTICLE NUMBER
LN2-transfer valve 3/8” female thread	2625
Vent valve 3/8” female thread	2625
Manometer 1/4” male thread	2626
Overpressure valve 0.5 bar	2621
O-ring with centring ring KF-NW50	2619
Clamp KF-NW50	2618
Loosely lied-on plug	2622

7.2 Accessories for container and LN2-transfer siphon

DESCRIPTION	ARTICLE NUMBER
EKI-transfer siphon 0.5 bar (specify container size)	2611 (adapted to size of container)
LN2-transfer hose 1.5 metres with PTFE piping and phase separator	2612
LN2-transfer hose 2 metres with PTFE piping and phase separator	2612-S3
Transfer tube with phase separator	2613
Screw adapter	2615
Screw coupling, dia. = 8 mm	2614
Roller base for ALU 20 or ALU 26	2640
Roller base for ALU 35 or ALU 60	2641
Roller base for ALU 100	2642
Filling flange	2606
Filling hose	2607
LN2-Level Control Unit	1301
Replacement probe PT100	1302
Magnetic valve 24V for LN2	1303
Level-filling head	1306


EKI-transfer siphon (2611)
LN2-transfer hose (2612)
LN2-transfer tube (2613)
Aluminium vessel

7.3 Transfer siphon with two separate LN2-fluid valves and two separate ascending pipes

EKI-2V
Art. No. 2611-D1

consisting of:

1 x EKI = transfer siphon with manometer and overpressure valve 0.5bar
2 x LN2-fluid valve with a screw adapter
1 x pressure-release valve

EKI-1V-1MAG
Art. No. 2611-D2

consisting of:

1 x EKI = transfer siphon with manometer and overpressure valve 0.5bar
1 x LN2-fluid valve with a screw adapter
1 x magnetic valve for LN2 with a screw adapter
1 x pressure-release valve

EKI-2MAG
Art. No. 2611-D3

consisting of:

1 x EKI = transfer siphon with manometer and overpressure valve 0.5bar
2 x magnetic valve for LN2 with a screw adapter
1 x pressure-release valve