

## Norhof LN2 microdosing systems

**#600 series LN2 microdosing systems (liquid supply)**

**including: model #610 2 point level control,  
for DSC 131 or similar**

**User manual**



**for software version 8.1  
and higher,**

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## **INTRODUCTION**

### **GENERAL DESCRIPTION**

The Norhof LN2 microdosing system is a cryogenic cooling system in which Liquid Nitrogen (LN2) is used as the cooling medium. LN2 is transferred from the dewar to the desired application.

In the Norhof LN2 cooling systems the Liquid Nitrogen is stored in pressure less cryogenic Dewars. When LN2 transfer is required, a small overpressure is generated by a micro processor controlled heater element in the LN2, and liquid nitrogen flows out of the system like water from a tap, without spilling, noise and vibrations. The cryogenic transfer flow is variable and can be optimized to the application.

### **INTENDED USE**

The autonomous cooling systems are designed for use in an instrumentation environment (e.g. scientific instruments) and/or in processes that require perfect control over the "cold" required. Liquid Nitrogen (LN2) is used as the cooling medium and is taken from a storage vessel by a static pump and delivered through a fill line to the application in a micro dosing way.

The system is designed to overcome the drawbacks of LN2 under pressure in which a solenoid valve is used to switch the supply ON / OFF. The system instead delivers a pressure less flow of LN2

# **1. GUIDE LINES FOR THE USE OF LIQUID NITROGEN (LN2) ALUMINUM CRYOGENIC DEWARS**

## **1.1 GENERAL**

- The aluminum dewar as supplied with the cooling system has a fiberglass/epoxy neck and is insulated with multilayered superinsulation under vacuum.
- These lightweight and highly efficient dewars are designed to withstand the most severe working conditions. However certain precautions should be taken to protect personnel using these dewars and to increase the life of your dewar.

## **1.2 PRECAUTIONS DURING USE**

- always transport and store the vessel in an upright position on an even and level floor, also when the dewar is empty or out of use. When using a transport trolley, only use the original trolley from Norhof.
- avoid tilting the dewar, even to withdraw LN2. When not using your dewar, LN2 will evaporate by itself. If you absolutely want to empty it, do it outdoors and pour on earth or gravel. Remember that most materials become brittle when cooled with LN2.
- when handling the dewar, do not drop it and avoid impacts when placing the dewar on the ground
- either the pump or the separate plug must be on top of the dewar at all times. No compliance will increase the boil off of the dewar and can lead to ice plug forming in the neck
- the dewar must be filled by inserting in the neck either a flexible hose or a hand withdrawal pipe connected to a storage vessel. In the case of an installation using a transfer line and if the transfer line is warm, the flexible hose should be inserted into the neck only after appearance of the liquid at the end of the flexible hose
- when filling a warm dewar, pour liquid slowly to avoid any liquid being propelled out due to rapid vaporization of the liquid inside the dewar. Fill the dewar to approximately 50% of the total volume and allow cooling down some hours before topping up. Thermal stability will be reached only after 48 hours.
- during filling, it is important to avoid spillage of LN2 onto the top of the dewar. If any spillage occurs, check during 24 hours that there are no traces of frosting left before re-using the dewar

## **1.3 CHECKING THE DEWAR**

- if traces of frosting appear on the outer vessel or if the outer vessel is completely frosted over, this shows that the vacuum in the interspace has been damaged or broken and that the LN2 is evaporating very quickly. Contact us for all necessary instructions. No repairs should be done by yourself on the dewar.

## **1.4 PRECAUTIONS WHEN HANDLING LIQUID NITROGEN**

- Liquid Nitrogen stored in your dewar has a boiling point of  $-196^{\circ}\text{C}$  and has a very high refrigeration capacity. Strict regulations must be applied to handle this fluid.
- contact with LN2 may cause cryogenic burns
- the liquid must be handled, particularly during filling in such a way that splashing is prevented
- when handling LN2, protect your eyes with glasses, your hands with proper gloves and your body with clothes that completely cover your arms and legs
- in the event of LN2 burns, proceed as for a burn. In all cases call a doctor
- Do not rub the skinburns. Gradually bring the affected parts up to normal temperature by placing them against another warm part of the body
- Gaseous nitrogen produced by evaporation of LN2, is odorless and invisible. A concentration of gaseous Nitrogen in a closed room or in a poorly ventilated place may cause asphyxiation by lowering the oxygen. Always use and store your dewars in a well-ventilated place.

## 2 UNPACKING

- **Dewar:** The dewar has a closing plug, which is needed when filling the dewar. During transport of the dewar (empty or filled), this plug should be ON the dewar for safety, and to prevent entering too much water (ice) into the dewar. So keep the plug on a place that it could be taken when the dewar needs to be filled.
- **Pump:** The pump is packed in plastic. Unpack it. On top of the pump is an orange knob. Depending of the packaging, this orange knob is already mounted, OR, the knob is packed separately in a plastic bag to make the pump fit into the package. In that case mount the orange knob firmly on the pump, so that it closes the pump airtight.
- **Sensors:** The #610 is supplied with a set of two sensors, one one cable with connector. The sensor consists out of a thick cable from ab. 2.00 meter, and a thinner part of blue teflon tubing of ab. 20 cm. During transport the blue teflon tubing is covered by a black ribbed tubing, for protection only. This should be removed before use. You may use this black ribbed tubing to guide the sensor to the place where you need the sensor. For that purpose you may cut the ribbed tubing to the right length. Be careful with the sensors !! In the end of the blue tubing there is the sensor element itself. It is a glass sensor of 1.6 mm diameter, which is very fragile.
- **PowerSupply :** The transformer supplied has an EURO mainsplug connected if it is for 230 Volts. If the system is for use in a country where this plug is not suitable, cut the plug and mount a plug which is suitable for this country. If the system is for use at 100 to 115 Volts, a transformer is supplied with NO plug. Check the voltage which is printed on the transformer. Please mount a suitable plug. The green/yellow wire is the ground (shielding),.
- **Trolley:** (optional, if ordered) The trolley is already mounted.
- **Floorstand:** (optional, if ordered) Mount the tube to the baseplate with the bolt supplied. Put the stand on the floor and step on it. This will align the 5 feet with the floor.

### 3 PREPARATION FOR 1<sup>ST</sup> TIME OPERATION

**3.1 remove clamp and remove pump from the dewar**

**3.2 “park” pump in floorstand or lay it on a table**

**3.3 fill the storage Dewar for max 90% with LN2**

allow Dewar to cool down. A ‘fresh’ filled dewar will degas for more than 12 hours, which is not a big problem. You may use it in this stage, but during this cooling down of the dewar the detecting of the liquid level which is in the pump can not be very accurate. The level as shown on the computer screen in the monitor program, may vary. However, the detection of the warning in the pump for ‘too low level’ is NOT depending of this. It is a separate sensor, which is NOT influenced by the degassing of a ‘fresh’ filled dewar.

**3.4 lower the pump slowly into the Dewar, in a way that the liquid does not splash too much.**

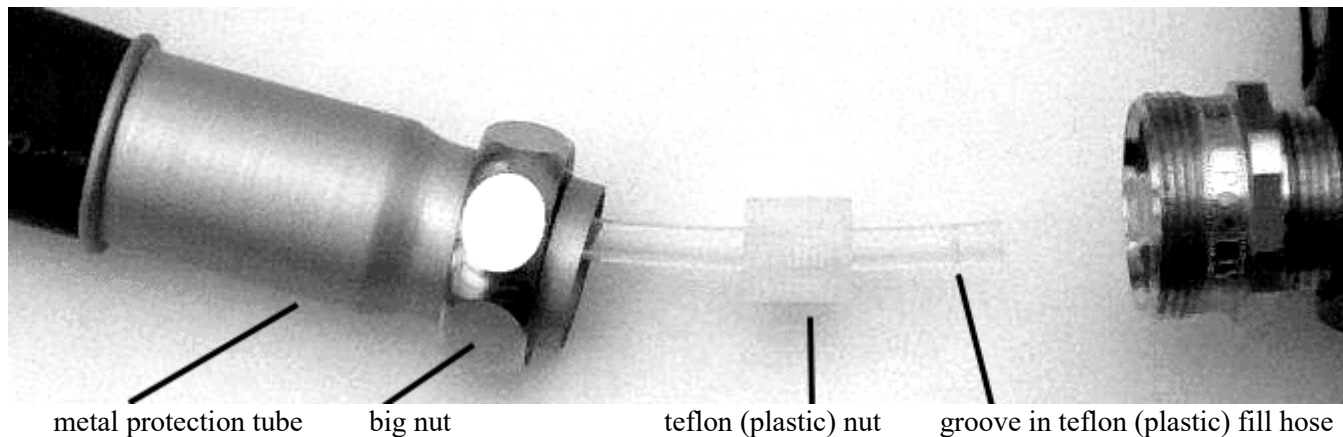
**WARNING:**

- the pump is at ambient temperature and when the “hot” protection pipe hits the liquid, a fair amount of “clouds” are generated
- also liquid might escape through the Teflon coupling, so don’t stand in front of it

**3.5 keep lowering the pump until it rests on the dewar.**

- put the clamp in place.
- make sure the pump + clamp is placed properly to make an airtight seal. Do not use tools! Handtight is fine (after all the system works with only millibars of overpressure)

**3.6 mount the fill hose to the pump**



- Loosen the big nut on the LN2 outlet, and put in on the metal protection tube of the fill hose. Loosen the teflon (plastic) nut 2 turns. Push the teflon (plastic) fill hose in the nut. Tighten the teflon nut 2 turns, and gently pull on the fill hose until the groove clicks in the nut. This is easy to feel. Now tighten the nut by hand as tight as possible. Then take your 16 mm. spanner and give the nut a half turn with the spanner. DO NOT TIGHTEN IT TOO MUCH. It is plastic, and can break if tightened too much.
- Move the insulation on the filling hose so, that the metal protection tube fits into the pump’s LN2 outlet. Tighten the big nut so that the protection tube is fixed. Tighten by hand only.
- The fill hose is standard ab. 2,20 meter. (1,60 m for #610) For the #180 it can be NOT be cutted shorter by yourself, because on both end there is a special groove to fit in the connector and on the tubing.

## 4 CHECK PUMP OPERATION

**\*\* Each pump is carefully tested before shipment. You may skip the procedure below, but may use it later to periodically check proper working of the system.**

### 4.1 GENERAL NOTES

- Have a small vessel, dewar, tin or alike ready to collect some LN2 for testing the sensors. The vessel does not have to be thermally isolated, almost anything that can hold some liquid will do. Do not use glass because of breakage risk
- the majority of the signals mentioned below are also elucidated on the label of the pump

### 4.2 START

- **DO NOT CONNECT SENSOR(S)** (use pump as it comes out of packing and with the fill hose you have just installed)
- **DO NOT PUSH BUTTON**

step	Do the following	result	elucidation
1	Remove safety pressure relief valve (orange knob on top)	System will remain pressure less	this prevents LN2 to come out from outlet
	<b>we are first going to test if all electrical signals work properly</b>		
2	Put 25D connector in place, connect to power supply and connect power supply to mains	Status led will flash at slow speed (every 5 seconds); indicating that the pump is in sleep mode	
3	Press button once to put pump in <b>standby</b> mode	<ul style="list-style-type: none"> <li>• Status led will flash at fast speed; indicating pump is in standby mode</li> <li>• Main sensor led and extra sensor led will flash RED, indicating sensors is broken or not connected</li> <li>• Warning led will flash, indicating a general alarm</li> <li>• Beeper will beep 16 +8 times to indicate main sensor + extra sensor circuit has a problem</li> </ul>	<p>this is of course true as no sensor is connected</p> <p>this is of course true as no sensor is connected</p> <p>this is of course true as no sensor is connected</p>
4	Connect the sensors to socket (main sensor)	<ul style="list-style-type: none"> <li>• Main sensor led will light steady RED to indicate sensor is warm</li> <li>• extra sensor led will light steady GREEN to indicate sensor is warm</li> </ul>	
5		• You will notice cold gas escaping from the top - after some delay - indicating that the heater is switched on and working	in normal operation the pumping will stop or go slower when the sensor is touched by the liquid nitrogen , while the color of the LED will change.
6	Press button once to put pump in <b>sleep</b> mode		
	<b>we are now going to test if the system will pump</b>		
7	Put safety valve in place.		<b>WARNING</b>



	Tighten it firmly, by hand		If you press the button now, the system builds up pressure to transport LN2. So be aware that LN2 escapes from the fill hose when you put the pump in standby mode. Put your test vessel under the end of the Teflon cryotubing
8	Press button once to put pump in <b>standby</b> mode	LN2 will flow through outlet	Be aware that it initially takes about 60 seconds before the tubing is cold and a steady flow is achieved. Observe that there comes a small steady stream of LN2
9	Press button once to put pump in <b>sleep</b> mode		
<b>we are now going to test if the level control works</b>			
	Press button once to put pump in <b>standby</b> mode		pumping will start
10	Put the low sensor in the LN2	Main sensor led will turn GREEN to indicate sensor is cold (in LN2)	The first time you do this the sensor has to cool down which may take about 15 seconds.
	put also the high level sensor in LN2	Extra sensor led will turn RED to indicate sensor is cold (in LN2)	
		Pumping will go slower Pump will switch to "active" after 15 seconds	pumping will stop after the "delay time" of 15 seconds
11	Raise both sensors above the LN2	The sensors will switch color to indicate sensors are warm (above LN2)	
12			Pumping will start again
13	Press button once to put pump in <b>sleep</b> mode		
<b>we are now going to test the system built-in protection</b>			
14	Press button once to put pump in <b>standby</b> mode		
15	Remove the clamp and raise pump above the dewar with all connections in place. You may put the pump in the floor stand (or keep it raised)	<p>After a minute or two the built-in sensor senses that it is no longer in LN2:</p> <ul style="list-style-type: none"> <li>• Warning led lights continuously red to warn for refilling the dewar</li> <li>• beeper double beeps every 30-sec.</li> </ul> <p>Keep the pump raised</p> <ul style="list-style-type: none"> <li>• After another delay of about 1 minute the pump switches itself off:</li> <li>• beeper will beep 1 time to indicate that pump is not in LN2</li> </ul>	<p>By raising the pump above the dewar you simulate that the liquid level drops below the built-in level sensor</p> <p>This double beep reminds you to refill the dewar even if you do not watch the front LEDS.</p> <p>In other words when the dewar really runs empty the pump switches itself off.</p>
16	Press button once to put pump in <b>sleep</b> mode		



## RESULT

By doing above test, you assured yourself

- that the system does pump (step 8)
- that a broken sensor is detected (step 3)
- that the supplied sensors work (step 10)
- that the level control works
- that you are timely warned to refill the dewar (step 15)
- that the system is protected when the dewar runs dry (step 15)

## 5 PUMP ON COMPUTER SCREEN

With the pump comes a software package that makes the pump visible on a computer screen. This is not a static schematic, on the contrary. The picture on display e.g. changes with the various pump models, the number of sensors in use, etc.

The screen allows you to:

- see the status of each active component
- read pressure and temperature of each sensor
- read the level of LN2 in the dewar
- read the (calculated) level in your application
- adjust the pump to the height of your application (relative to the exhaust height of the pump) (default = 50 cm)
- adjust the output flow for your application (default = 60 mBar)
- etc.

Also with this software it is possible to adjust the pump to a longer filling line than the standard 1.6 meters line. The external sensor heats himself (for level control). In this way the sensor is able to detect the difference between the liquid, and the gas just above the liquid.

It is highly advisable to connect your PC to the pump for educational purposes during your familiarization with the system.

If you do, make sure that the pump + PC are on the same mains, otherwise groundloops may prevent proper operation, or cause damage.

The PC is by no means mandatory to operate the system, only for adjusting the fill application height (once).

If you have any question or feel a hesitance to do something, contact us, preferably by e-mail: [norhof@ziggo.nl](mailto:norhof@ziggo.nl)

## 6 Changing defaults for your application

The #610 pump is factory preset for use with the Differential Scanning Calorimeter 131.

For this application the LN2 feeding height is preset on 50 cm above the pump exhaust, counting on that your application stands on a table.

If your application is much higher, you could set this height according your application height. (see below)

For this application a flowrate of about 60 mBar is the most optimal setting. If the flowrate is too high, the system could switch pumping OFF too soon because of the splashing on the sensor before the actual level is high enough.

In that case the flowrate could be set a little lower to create a more gentle filling.

If the flowrate is set too low, it will take longer before the application is filled.

**Readjusting the feeding height** is done in the Monitor software by clicking on the SET button. This opens a screen in which you can enter the height, measured from the exhaust on the pump. For a 35 Liter dewar on a trolley the exhaust is about 70 cm from the floor. If your application is standing on a table of 70 cm, and is 50 cm high, this value should be 50 cm. If your actual feeding height is much higher, you should enter here the correct height. The length of the fill line should be on 2 meters if the fill line is 2 meters or shorter. If the filling line is longer, this could be adjusted here. Also when a very thin filling line is used, and a "frozen" alarm occurs while the fill line is NOT frozen, this value can be set a little higher to indicate to the pump that it takes more pressure to reach a steady flow, before the "frozen" alarm occurs.

**Readjusting the delay time before sensor stops filling** is done in the "set delay" button. If the sensor is touched by the LN2 level, the pump only stops filling if the sensor is below -180 during this xx seconds. Also during counting down this time, the flowrate is lowered. This prevents the pump to switch off during splashes on the sensor, while the detector is not yet full. The default 15 seconds will be OK for the DSC131.

**Readjusting the pumping flow rate** is done in the "service" and "show calibrate screen".

In the bottom there is the "flow preset" slide bar, which is on 60 mBar. If you slide this bar, and click "write in pump", the new value will be stored in the pump. Do NOT click on other button here unless you know what you do. (for calibration see later)

## 7 Working for Differential Scanning Calorimeter (DSC131 ), pump model #610 (or similar application)

For the #610, when the pump is switched to STANDBY, (by hand on the push button, or automatic by the DSC instrument), and the sensors are warm, the pump switches to "PUMPING". The pump starts building pressure and will fill up the DSC dewar until both sensors touch LN2 for more than 15 seconds (default). When the "high" sensor is touched by the liquid, the pumping flowrate will be lowered, allowing the boiling effect of reaching the -196 degrees to slow down.

After this 15 seconds the pump will stop pumping and switches to "STANDBY" state.

In this "STANDBY" state it will wait until both sensors are dried out of LN2. If the low sensor is also above -180, so warm, the pump will switch to "PUMPING" state again, and will fill up the dewar again.

So after pumping when the level is reached the pump will go back to "STANDBY", doing nothing.

If in between the pump is switched to SLEEP (by the push button, or with a signal from the DSC instrument), and switched to STANDBY again, a new fill cycle will start. This will happen also when the low sensor is still cold. In this way you may start a new filling cycle, to be sure that for that moment the DSC dewar is completely filled. This could be useful before a long measuring cycle in which you do not want an automatic fill cycle.

When the pump is switched to SLEEP (by hand on the push button, or automatic by the DSC instrument), the pump stops pumping (if pumping was busy) and releases all pressure.

Adjusting filling pressure:

If the height of the DSC instrument is set correctly in the pump, the fillpressure should be so, that the DSC dewar is filled in about 8-20 minutes. The default 60 Mbar should be about good. If the filling is too fast, LN2 may splash on the sensor and switch the pump OFF before the wanted level is reached. If the fill pressure is too low, it will take much more time, or the LN2 will not reach the dewar.

Fill pressure can be set in the SERVICE-CALIBRATION screen. (see later)

**NOTE:** When the pump is in the LN2, the power should be kept on the pump. When the power is OFF, the leading hose of the pump may freeze because waterdamp will enter the pump slowly and freeze the exhaust. There is a small heater element in the pumphead to prevent this. If the pump is longer (several hours) without power, the pump could internally freeze. Only solution then is to warm up the pump completely.

## 8 Remote control

The #610 can be remotely switched OFF and ON (= in SLEEP or STANDBY), in 4 ways:

- 1 With a 5 volt signal on pin 4 of the 25p subDconnector, the pump can be switched to SLEEP.  
 0 Volt = SLEEP, 5 Volt is no-operation.  
 With a 5 volt signal on pin 5 of the 25p subDconnector, the pump can be switched to STANDBY.  
 0 Volt = STANDBY, 5 Volt is no-operation.  
 Connecting these pins to ground (pin 17=18=19=20), is also enough to make the signals switch.  
 So two pushbuttons could do the job also. SLEEP overrides the STANDBY.  
 (pull up resistor is 1 kohm, with 100nF capacitor)
- 2 With the optocoupler signals (see below)

For programmers:

- 3 If NO monitor software is standby, so the serial port is free for your own application software, the serial port can be opened at 19200,N,8,1  
 is Baudrate 19200, No parity, 8 databits, 1 stopbit.  
 Sending a "pon" command (PumpingON) will set the pump in STANDBY, sending a "pof" command (PumpingOFF) will set the pump to SLEEP.  
 Sending "rm 19" will give back the pump status register. result and 2 = pumping, result and 16 = standby, result and 32 = sleep, result and 128 = alarm

- 4 If the monitorsoftware is running, so the port is occupied, there is a way to tell the monitor software to switch the pump. This is not a very gentle solution, but the only easy way.  
 The monitor software will check continuously in his working directory (c:\Program Files\NorhofPumpMonitor as default) for a (empty) file "PON.txt" or "POF.txt" (or "pon.txt" or "pof.txt" ). If found, the pump will switch ON or OFF, and delete the file.  
 creating a "RM19.txt" file gives back a "RM19.dat" file with the pumpstatus result.

For the #610 in one of these ways the application can switch the pumping ON and OFF, keeping the DSC cold or not.

Switching the pump OFF, and some later again ON, will start a fill cycle, and after that a new wait cycle for the sensors to be warm again.

If you want to steer the fillcycles from your application, you could switch the pump OFF and ON, starting a fill cycle, and after some time (5-10 minutes ?), the DSC will be ready filling. ( Better is reading the status register to detect when pumping stopped. ) Then your measurements can continue. Then you can build-in in your measuring cycles pauses for refilling.

Simply let your measuring software pause your measurement, and switch the pump OFF and ON, to restart a new filling cycle.

Be sure that this moments are sooner than the automatic fill cycle. In this way the DSC is filled during your pauses, and if the pause should come too late, the automatic fill cycle will make sure that the DSC can not become warm.

## 9 Password Protection

In the monitor software all the editable functions can be password protected.

In the Main screen - service screen - calibration screen - extended setup is a button "password protection".

Here you can set a password of maximum 4 characters or numbers, which will be asked before entering all the editable functions.

Also here you can delete the passwordprotection. (=NO password).

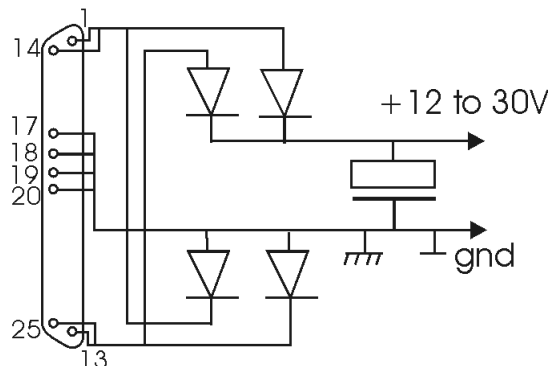
If you have forgotten the password, you can simply delete the file "NORpassword.sys" in the working directory of the PumpMonitor. (probably c:\Program Files\NorhofPumpMonitor\NORpassword.sys")

## 10 Appendix A: pin connections of 25p subD connector

1=14 AC1 AC or DC 12 to 24 Volts Power supply (min. 4 A.)  
13=25 AC2 AC or DC 12 to 24 Volts Power supply (min. 4 A.)

17=18=19=20 system ground

**\*\* If you want to use an other power supply than we supplied, and if it is a DC power supply of 12 to 30 Volt:**  
connect - (minus) of the DC power supply to 17,18,19 and 20  
connect + (plus) of the DC power supply to 1,14,13 and 25  
Power supply must be DC12V, 4 amps, or 24 Volt, 2 amps.



8 RXD serial connection

9 TXD serial connection

4 TTL\_1 input, 0 or 5 Volt, 0 Volt = switch pump to SLEEP (pull up resistor is 1 kohm, with 100nF capacitor)

5 TTL\_2 input, 0 or 5 Volt, 0 Volt = switch pump to STANDBY (pull up resistor is 1 kohm, with 100nF capacitor)

15 opt1C\* optocoupler input 1 neg.

2 opt1A \* optocoupler input 1 pos. : 0 or 5-24 Volt input. 5-24 V. to switch pump to STANDBY (1 Kohm resistor for 5 Volt, 5K for 24 Volt)

16 opt2C\* optocoupler input 2 neg.

3 opt2A\* optocoupler input 2 pos. : 0 or 5-24 Volt input. 5-24 V. to switch pump to SLEEP (1 Kohm resistor for 5 Volt, 5K for 24 Volt)

(\*) connect C to ground and supply positive signal with resistor to A to switch (\*) OR, connect A to ground to use negative signal on C .

21 optAL2E optocoupler output emitter ALARM (\*)

22 optAL2C optocoupler output collector ALARM (\*)

conductive = Alarm, OPEN is no alarm

(\*) connect E = emitter to ground to switch a positive signal,

then connect C=collector with 10K to +5V to switch the signal, or with 33k to +24V

(\*) OR, connect C = collector to ground to switch a negative signal,

then connect E=emitter with 10K to -5V or with 23k to -24 V.

## 11 Appendix B: calibration

Usually the system will work as mentioned above and you do not have to do any adjustments.

If the system is recently delivered, all calibration is already done at the factory.

However, if you change the setup, or have the feeling that the sensors may not give the accurate temperature, you may calibrate the internal pressure sensor and internal and external sensor(s).

This procedure is very simple.

In the monitor program, there a button to go to the 'service' screen. There in the bottom is a button to go to the 'calibrate' screen.

On the left are some buttons to make a 'rough' calibration, to select what type of external sensors are connected. In the right part is the fine calibration. For the #610 in the left screen there are two buttons, for the external main sensor and external extra sensor, as "selfheating" sensor, on 33 mA.

## SELECTION OF EXTERNAL SENSOR TYPE

The external sensor is a PT100 element. The sensor itself is a 1.6 mm glassensor, in a small teflon tubing of 2,8 mm. (the tip is very fragile). You also could use other sensors yourself, as long as it is a PT100 element.

The sensors is used for level-detection, and is heated continuously by a small current (33 mA) to heat the sensor approx. 30 degrees Celsius above the environment temperature. So if the sensor is in the N2 gas, the temperature measured is a little higher than for real. If the sensor is touched by the liquid, it will cool down to the liquid temperature. In this way it is possible to have an accurate level detection.

Clicking on the "main 33 mA" button presets the calibration values for this sensor to factory default. (137, 463)

## FINE CALIBRATION

After the rough calibration is entered into the pump, you may use the fine calibration to calibrate the sensor more accurate. In the right side of this screen you see the fine calibration buttons.

### External sensors

For the external sensors, you can read the actual temperature in the middle.

When the sensor is at room temperature, after switching the mains power on the pump, or after connecting the sensor, the sensor would be at ambient temperature (about 20 degrees), but only in the beginning.

Beacuse the sensors are in self-heating mode, the temperature will raise quickly.

If the value is much lower or higher than the room temperature, you may press the '20 degrees' button to enter this value into the pump. When the sensor is connected some time longer, the temperature rises (the sensor is at 33 mA, because of the selfheating). If you want a very accurate calibration, you may put the sensor in a glass of water, and heat the water upto 30 degrees (with a hairdryer?) and then press the '30 degrees' button. Note that this temperature does not need to be absolute accurate when the sensor is only used for level control. For level control the sensor is used at -196 upto -172 only, so if the top temperature is some degrees too high or low, it has almost no influence. What is important, is the calibration for the 'cold' value. Herefor the sensor must be put into LN2. Make sure that the sensor is deep into the LN2, and look at the temperature on screen that it is not dropping any longer (one or two minutes). If the reading is not exactly -196, press the '-196' button to enter this value into the pump. Now the reading should be -196 exactly.

Calibration is now ready, but before use, the pump should be resetted (power off and on) to work with the new values.

### Internal pressure sensor

Inside the pump is an internal pressure sensor which measures the pressure in the dewar, 4cm from the bottom, so UNDER the LN2. Calibration of this is very simple. The pump should be out of the LN2, so in open air. If the reading on screen is not 0 mBar (+\_ 0.5 mBar), you may click the '0 mBar' button to enter this value. Now the reading should give 0 mBar +- 0,5 mBar. After the pump is placed back in the LN2, the LN2 level should correspond with the actual LN2 level. Also here, before use, the pump should be resetted (power off and on) to work with the new value.

### Internal vessel sensor.

Inside the pump , down in the dewar, there is a set of sensors to measure the level in the dewar. If the pump is in room temperature, the reading should be around +30 degrees (due to heating because of 33 mA through these sensors also). When the pump is in the LN2, and there is more than 10 cm LN2 in the dewar, the reading should be -196 degrees. If not, you may click the '-196 button' to calibrate this value.

### Readjusting the pumping flow rate

In the bottom there is the "flow preset" slide bar, which is on 60 mBar. If you slide this bar, and click "write in pump", the new value will be stored in the pump. This is the value which is used during pumping.

### Factory defaults

The calibration screen can be made more wide to the right. In the right part it is possible to: calibrate the inernal clock (by reading the calibration byte, making it slower or faster, and writing a new value) reading the setpoints, binear, voltage and degrees. Use only on factory instructions.

"calibrate ALL 610 parameters" button:

When clicking this button, all calibration values are back to default (but make sure that he pump is OUT of LN2 while clicking)



## 12 Appendix C: detailed working of the pumping

When the pump is not standby, there is NO overpressure in the dewar and the small release valve is open. This release valve is a small hole to connect the dewar to the outside. It is closed when overpressure is needed.

When the pump is switched to 'STANDBY', the pump uses a heater element in the bottom of the dewar to create gas to build a small overpressure. Because the fillhose is in fact coming from the bottom of the dewar, when a small overpressure is inside the dewar, the liquid will raise in this fill hose. When the pressure is enough, the liquid will reach the highest point, and will flow out of the fill hose. You can imagine, that how higher the pressure is, the higher the flow is. Note that there is NO valve in the fill hose, it is just a hose coming from the bottom of the dewar, going to the application.

So, when switched to STANDBY, the release valve will close and the heater will build some pressure to create some LN2 flow.

There can be 3 situations of the pump:

- 1 SLEEP the pump is doing nothing, except keeping an internal small heater warm to prevent the pumphead from freezing
- 2 STANDBY a small overpressure is used to keep the pump standby for the next pumping action
- 3 PUMPING a pressure is used to realize the requested flow of LN2

### Pumping Prepressure

For pumping, the pumping pressure consists of 3 variable values.

\* First, a certain pressure is needed to get the level high enough to reach the pumphead. This is called the 'prepressure'.

Since the pressure is measured in the bottom of the dewar, the pressure needed here is not depending of the dewar level. Only when the dewar is more empty, it may take some more time to reach that pressure. This prepressure is fixed and adjusted to the dewar height. (27 mBar. for 35 Liter dewar)

\* The second value is the pressure to reach the highest point in the fill hose. You can imagine that if the fill hose goes 1.5 meters high, that a higher pressure is needed to let the LN2 come out of the fill hose. This can be set in the monitor program with the 'feeding height compensation'. The value for 0 cm feeding height is 8 mBar, needed to rise from the bottom of the pumphead to the outlet.

\* The third value is the pressure wanted to have a flow suitable for your application. This is preset in the #610 on 60 mBar. The higher this pressure is set, the faster the LN2 comes out of the fill hose, but too fast may cause the high sensor to switch too early because of splashing on this sensor.

\* Cooldown boost pressure. During the first pumping action, the fill line must cool down. During this time an extra overpressure of 30 mBar (default) is added to the pumping pressure, which decreases when the sensor comes closer to the -155C. This helps in faster cooling down the fill line, while the actual filling will be at the filling pressure.

The value for the cool down boost can be changed in the extended setup in the calibration screen.

### Dewar level detection and prepressure

#### SLEEP mode:

When the pump is connected to the power, 9 beeps sounds, and the pump will begin in SLEEP mode (yellow LED flashing slowly every 5 seconds)

When the pump is started for the first time, the LN2 level in the dewar is unknown to the pump. During SLEEP (when all is OFF) the pump measures the pressure in the bottom of the dewar, and after some seconds, when this is stable, the LN2 level will be drawn on the screen.

Since this LN2 level can only be detected when the pump is in SLEEP, this drawing on screen can be some inaccurate during PUMPING, but it will NOT influence pumping behavior. After pumping is done and the pump is in SLEEP mode, the drawing on screen of the LN2 level is accurate again.

The warning for that the dewar is almost empty, or really empty, is measured with other sensors, so this is always accurate.

#### STANDBY / PUMPING mode:

When the green button is pushed, the pump switches ON, depending of what working mode and sensor temperature(s).

When sensor(s) are not yet cold enough, the pump will go into PUMPING mode. (Yellow LED burning steady)  
When the sensor(s) are cold, or the temperature is reached, the pump will go into STANDBY mode. (yellow LED flashing every 1 sec.)

#### **STANDBY mode:**

In the standby mode, there is no overpressure in the dewar. This will stay until the pump goes into PUMPING mode.

For the #610, STANDBY mode has the same pressure as SLEEP mode, and also updates the LN2 level in the dewar.

#### **PUMPING mode:**

When starting pumping, the pump starts to build up pressure. You can see this in the monitorprogram at the HEATER. It will start at 5% and increases every 2 seconds, upto 100%. It will increase pressure until the pressure is equal to the requested total pressure, depending on 3 values.

1. The prepressure ( to reach the bottom of the pumphead, for a 35 Liter dewar ab. 27 mBar)
2. The pressure to reach the highest point in the fill line. (8 mBar for the 13 cm. to reach the outlet of the pump, PLUS the pressure needed to reach the highest point, if set)
- 3 The real pumping pressure (preset on 60 mBar)

If this pressure is reached, the heater shuts OFF, until the pressure is too low again. It will then be switched ON with a little lower capacity (95%) each time. In the end this will be a stable situation and the heater will switch ON and OFF at 30% to 50 %, depending of how much LN2 is really pumped.

( On screen the visualibilty is slower than it is in real. This value is sampled on screen every 1 second only, to limit datatransfer. This is also for many other values on screen, because the monitor is just for indication)

After the pump has finished pumping and the pump is in STANDBY , all the dewar over pressure is.

#### **FREEZE PROTECTION:**

If the pump is OFF (in sleep mode), a small heater element in the top of the rise pipe will be heated upto 50 degrees, to block the waterdamp which could go into the pump and freeze the risepipe.

Therefor, the pump should not be IN the LN2 and disconnected from the mains power in the same time.

If mains power is disconnected, the pump should NOT be in the dewar. (for some hours it is no problem, but for a night long, it can be too long and the pump could freeze) This also depends on the humidity of that moment, and on the length of the fill hose. If the fill hose is long, it takes more time for the waterdamp to go into the tube and reach the pumphead.

#### **BLOCKED (frozen) alarm:**

The pump looks if the LN2 is really going out of the rise pipe, counting on that the exhaust of the LN2 is in the free air. When the pressure in the dewar is higher than 60 mBar, and the temperature measured in the exhaust of the pump is still higher than -20 C., the "exhaust blocked" alarm is set.

If the application is too much airtight, thus the exhaust is blocked too much, this could also cause a 'blocked exhaust' alarm. This is the same as a frozen exhaust. The application should have an opening wide enough to let expand all the gas. If the pump gives this alarm often, the value for the lenght of the fill hose could be set some longer, to prevent this alarm.



## 16 Alarm list

### Almost empty alarm:

Warning LED ON + double beep every 30 seconds = less than 4 liters LN2 left

- This is measured with a sensor 4 cm from the bottom of the dewar.

### Other alarms:

When warning LED flashes, the number of beeps indicate the problem:

beeps

- 1 pump is not cold (empty?)
  - this is measured with a sensor close to the bottom of the dewar
- 2 dewar level sensor not OK
  - empty or almost empty internal sensor is broken?
- 3 pump flow sensor not OK
  - internal TMB sensor is broken?
- 4 no pressure building (leak?)
  - pump not airtight on dewar, or orange overpressure valve is not tight
- 8 main external sensor not OK
  - main sensor on the application is loose or broken
- 9 extra external sensor not OK
  - second application sensor is loose or broken
- 10 exhaust blocked (frozen?)
  - flow tube inside the pump is frozen, or fill tube on application is frozen, or
  - application is too much airtight
- 11 pressure measuring tube frozen
  - internal pressure measuring tube is frozen (or has water in)
  - then also the LN2 level indication of the dewar will be wrong
- 12 pumping was too long
  - pumping time has reached the adjusted time for pumping too long alarm

## 17 Warming up and drying the pump

**Notice:** when cleaning the pump, always remove the pump from mains during cleaning procedure

If you have the feeling that the risepipe or the pressure measuring tube is frozen, you need to warm the pump up to room temperature, and may be dry the rise pipe and measuring tube.

Please put the pump in its floorstand, or lay the pump on a table and wait for all ice and condense water has disappeared. You may help a little by warming it with a electrical hairdryer. But be careful. The protection pipe, around the heater and rise pipe, is made of PVC, and will deform at temperatures above 70 C.

### **Blocked exhaust:**

After all condense water is disappeared, it could be possible to see if there is an ice block in the rise pipe. The most obvious place is high in the risepipe, almost at the pumphead. So this may not be easy to see.

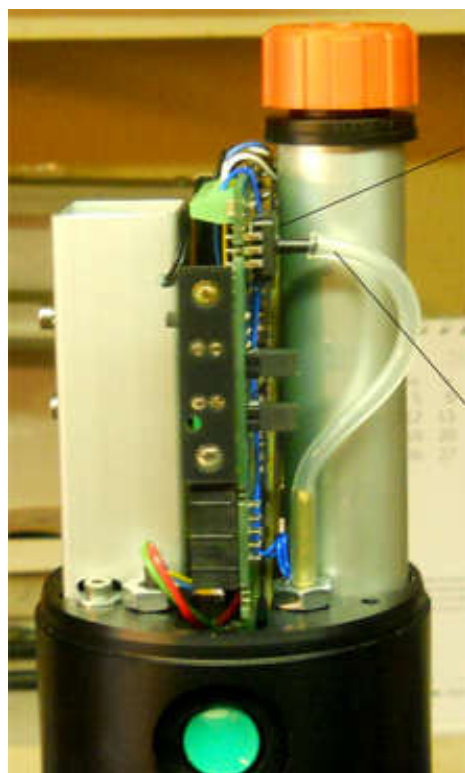
You may blow with dry air from the fill line into the pump, to blow the last water downwards out of the rise pipe. Ofcourse the air should flow freely through this when the ice block is removed.

### **Frozen pressure measuring tube:**

Second place of freezing is the pressure measuring tube. (red tube of 3,3 mm for pumps from before 2011, or transparent tube for pumps after 2011)

At the bottom, next to the heater, there is a set of two small resistors mounted in this pressure tube. These resistors evaporate LN2 during pumping, to make sure this pressure tube is fully filled with N2 gas all the time.

If the pump is out of the LN2, condense water may occur here, which will turn into ice when the pump is replaced in the LN2 before it was dried. If done many times, some ice may appear here, and even some water can go upwards in this tube. To make really sure that all water is out, you may careful blow with dry air from above true this tube. Herefor, the silicon tube in the pumphead could be removed from the pressure sensor on the PCB. Then you can blow in the silicone tube downwards true this pressure tube. Watch if any water comes out, and blow until there is no water left.



pressure sensor

pressure tube,  
make loose



pressure  
measuring  
tube

protection pipe

risepipe

## 18 Declaration of Conformity

**EU DECLARATION OF CONFORMITY**  
This declaration of conformity is issued under the sole responsibility of the manufacturer

**MANUFACTURER**

Business name: Norhof B.V.  
Address: Galileilaan 33U  
Postal code: 6716 BP  
Place: Ede  
Country: The Netherlands

**DESCRIPTION AND IDENTIFICATION OF THE ELECTRICAL EQUIPMENT**

Denomination: LN2 Microdosing Systems  
Function: Transfer of liquid nitrogen  
Type: Serie #400 Manual LN2 dosing systems  
Serie #600 Automatic LN2 microdosing systems  
Serie #800 Automatic N2 gasstream systems  
Serie #900 Advanced Automatic  
LN2 microdosing systems

Serial number: 400-XXXX-XX-XX-XX-XX  
600-XXXX-XX-XX-XX-XX  
800-XXXX-XX-XX-XX-XX  
900-XXXX-XX-XX-XX-XX

Year in which the CE marking  
was affixed: 2017

**COMPLIANCE**  
*The manufacturer declares that the above mentioned electrical equipment fulfills all relevant provisions of*


Low Voltage Directive (2014/35/EU)  
EMC Directive (2014/30/EU)  
RoHS Directive (2011/65/EU)  
General Product Safety (2001/95/EC)

*In conjunction with the following harmonised standards or technical specifications for the design and manufacture*

EN 12300:1998; EN 61010-1:2010; EN 61000-6-1:2007; EN 61000-6-3:2007

**SIGNED FOR AND ON BEHALF OF NORHOF B.V.**

Place: Ede  
Date: 3 July 2017

Identity: Mr. Emile Bisschop  
Position: General Manager  
Signature: 

## 19 Country of Origin

All the Norhof LN2 dispensers and pump models #400, #600, #800 and #900 are manufactured in the Netherlands.

- Original instructions -  
Norhof B.V., Ede 2017