

# Norhof LN2 microdosing systems

**#600 series LN2 microdosing systems**  
**model #606 DSC control**  
**model #608 FTIR detector control**

## 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 microprocessor 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:** One or two sensors are supplied with this systems (standard). The sensor consists out of a thick cable from ab. 1,80 meter, and a thinner part of blue Teflon tubing of ab. 50 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.
- **Power Supply :** The transformer supplied has an EURO mains plug 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**, no LN2 in the neck, so minimal 18 cm free. 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 cannot 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.

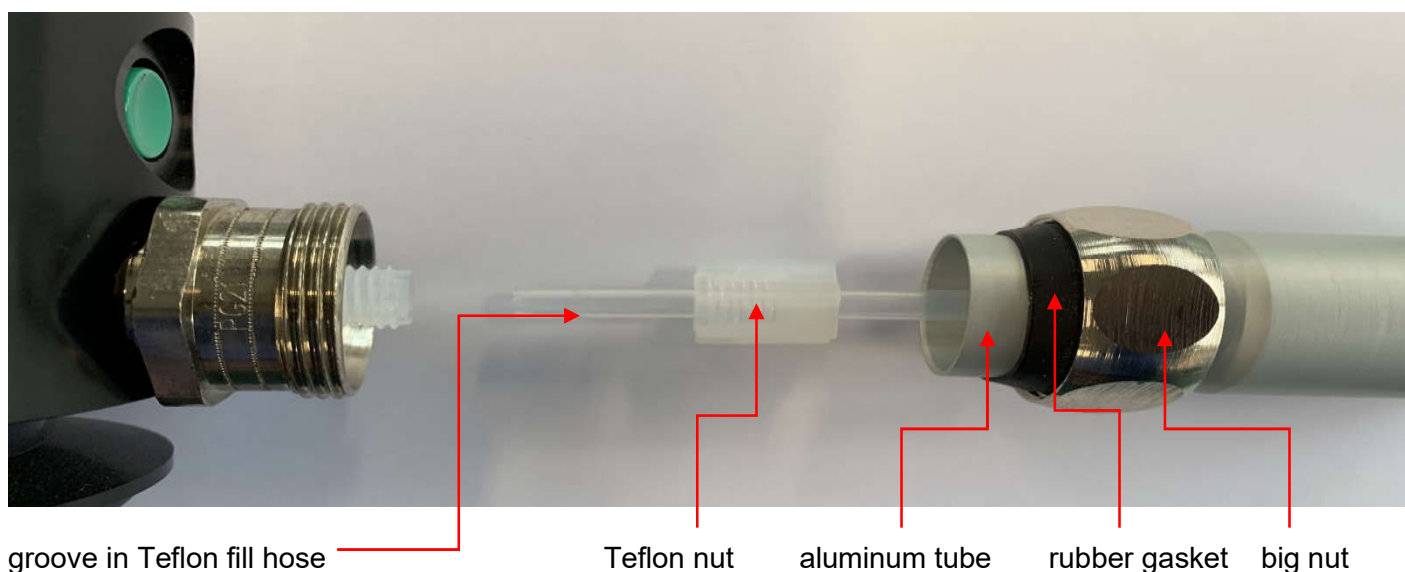
**3.4 lower the pump slowly into the Dewar**, in a way that the liquid does not splash too much. **WARNING:**

- 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! Hand tight is fine (after all the system works with only millibars of overpressure)

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



For a clear instruction how to Leak Free install the Cryo hose, please check our Norhof YouTube instruction Video at:

[https://youtu.be/xy8xilgeiJY?si=epIJ\\_bDJqx2Qbslk](https://youtu.be/xy8xilgeiJY?si=epIJ_bDJqx2Qbslk)



- 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 #608) It can be cut shorter for your application. The shorter the fill hose is, the faster the liquid will start to flow. Only make sure that the fill hose is long enough to make it possible to remove the pump from the dewar while the fill hose is still connected (for refilling the dewar)
- Slip the phase separator (if supplied) over the other end of the fill hose, over the inner tube, if you want a free falling LN2 supply. Do the installation when the tube is at room temperature. The phase separator is not mandatory for operation. It is just something that you might want to use.

## 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 will flash RED, indicating sensor is broken or not connected</li> <li>• Warning led will flash, indicating a general alarm</li> <li>• Beeper will beep 8 times to indicate main 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 sensor to socket (also restart the pump to reset the alarm)	<ul style="list-style-type: none"> <li>• for #606: Main sensor led will light steady RED to indicate sensor is warm</li> <li>• for #608: Main 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		



we are now going to test if the system will pump			
7	Put safety valve in place		<b>WARNING</b> 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		
we are now going to test if the level control works			
	Press button once to put pump in <b>standby</b> mode		pumping will start
10	Put the sensor in the LN2	<ul style="list-style-type: none"> <li>• for #606: Main sensor led will turn GREEN to indicate sensor is cold (in LN2)</li> <li>• for #608: Main sensor led will turn RED to indicate sensor is cold (in LN2)</li> </ul>	The first time you do this the sensor has to cool down which may take about 15 seconds.
		Pumping will go slower <ul style="list-style-type: none"> <li>• for #606: Pump will stay in "pumping" mode, but only drip.</li> <li>• for #608: Pump will switch to "active" after 80 seconds</li> </ul>	pumping will stop after the "delay time"
11	Raise the sensor above the LN2	The sensor will switch color to indicate sensor is warm (above LN2)	
12		<ul style="list-style-type: none"> <li>• for #606: Pumping will start again</li> <li>• for #608: Pumping will start after 4:00 hr is past (default repeat time)</li> </ul>	
13	Press button once to put pump in <b>sleep</b> mode		
we are now going to test the system built-in protection			
14	Press button once to put pump in <b>standby</b> mode		
15	Remove the clamp and raise	After a minute or two the built-in	By raising the pump

	<p>pump above the dewar with all connections in place. You may put the pump in the floor stand (or keep it raised)</p>	<p>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>• beeper will beep 1 time to indicate that pump is not in LN2</li> </ul>	<p>above the dewar you simulate that the liquid level drops below the built-in level sensor</p> <p>In other words when the Dewar really runs empty the pump stops pumping</p>
16	Press button once to put pump in <b>sleep</b> mode		

## RESULT

By doing above test, you assured yourself

- that 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 Monitor Software package (On the USB stick delivered with the pump) 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 = 20 cm)
- adjust the output flow for your application (default = 40 mBar)
- adjust the interval time for refilling (#608 model) (default = 4:00 hr)
- etc.

Also with this software it is possible to adjust the pump to a longer filling line than the standard 2 meters line.

The external sensor heats itself (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. All necessary parts are included.

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

The PC is by no means mandatory to operate the system after the system is installed.

The PC does not need to be connected when all is working fine.

On the USB stick is also a folder "608monitor" for the recorder for the sensor behavior of the #608 (see later)

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

## 6 The Monitor software

The pump is delivered including an USB stick containing the monitor software. During installation, or if there is a problem with the pump, of course it is the best to connect the pump to the PC and read out all of the behavior on screen.

The pump can be connected with the (supplied) 5 meter cable with a small black 4 pin round connector on one end, and the 9 pin subD connector on the computer side. The pump should be connected to a (serial) COM port on the PC.

If the computer does not have a COM port (such as many laptops have nowadays), you can simply use a USB to COM converter cable. (cost ab 10\$). With this adaptor there is created a virtual COM port on the PC.

In the hardware setup of the computer you can set this COM port to a fixed port number. The monitor software can handle port numbers form 1 to 8. When possible, assign your USB to COM port adaptor to COM1, because the monitor software starts default on COM1.

### 6.1 Other version software

You should use ONLY the monitor software version supplied with the pump. During the years we make changes in the pumps and the software. Only the same version number as the pump has will correspond fully. It can do no harm if you just connect another pump to older (or newer) software to check for the version number, but you should NOT write any calibration values or feeding height into the pump with software which does not correspond to this pump. Writing in a pump with the wrong version could make the pump unusable.

Also, when a wrong version number software is connected to the pump, the readout on screen can give numbers which are not for real. For example, the readout for the feeding height was changed somewhere in 2008. When connecting an older pump to newer software, the readout for the feeding height on screen could give for that pump 26 meters, but inside the pump the construction of this number is built in a new way. So do NOT write in this case a new value of e.g. 20 cm in this pump, because probably the real feeding height in this pump was OK, but the readout with the newer software was wrong only on screen.

### 6.2 Firmware version number of the pump

On the right bottom in the monitor screen is the firmware version of the pump. (the firmware is the software which is inside the microprocessor in the pump). This is shown once when the software is started up or when a pump is connected. If you connect another pump and you want to read the firmware version, you should close the monitor software and start it up again.

The firmware version should correspond with the version number of the monitor software itself (in the top blue bar, before the version date.) It will read e.g. "Norhof Monitor Program v. 7.51 21 March 2012".

The firmware version of the pump will read also : "firmware in pump: ver 7.51 12 March 2012".

The dates does not need to be the same exactly, because the date gives the date of the last change. There can have been made a small change in the same version number while not changing the version number.

### 6.3 Pump model

The monitor software will recognize the pump model automatically. There is one version of the monitor software which is suitable for all pump models (series #600, #800 and #900 models)

In the left part it will display the pump model, and what working mode the pump is in.

For the 800 and 900 models the working mode can be selected by yourself with the mode selector switch, mounted inside the pumphead. For the #600 models pumps there is only one fixed working mode.

### 6.4 Pump drawing

On the right part is a picture of the pump in the dewar and a symbolic drawing of the application. The form of this drawing depends on in what working mode the pump is. If it is a working mode with one sensor, only one sensor is drawn on screen, but if it is a working mode with two sensors, both sensors are drawn on screen.

## 7 Changing defaults for your application

The pump is factory preset for use with the Differential Scanning Calorimeter (#606) or FTIR detector (#608).

For this application the LN2 feeding height is preset on 20 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 30 to 40 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.

For the #608, the interval time for refilling is factory set on 4:00 hours. If your detector has a much longer, or shorter stand-time, you could readjust this to about  $\frac{3}{4}$  of the stand-time, so that refilling starts when there is about 25% of LN2 left in your detector.

**Readjusting the feeding height** is done in the Monitor software by clicking on the SET button next to the fill line. 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 20 cm high, this value should be 20 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 "shut off" time (#606)** is done in with the "SET" button, after entering the new time in the shut off time window.

**Readjusting the repeat interval filling time (#608)** is done in with the "SET" button, after entering the new time in the repeat time window.

**Readjusting the delay time before sensor stops filling (#608)** 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 -191 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 80 seconds will be OK for a small FTIR detector.

**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 30 mBar.(40mBar for #608). 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)

## 8 Working for Differential Scanning Calorimeter (DSC), model #606

For the #606, when the pump is switched to STANDBY, (by hand on the push button, or automatic by the DSC), the pump starts building pressure and will fill up the small dewar in the DSC until the sensor touches LN2.

While the sensor is cooled down, the built-in PID regulation will regulate the flowrate back when the sensor temperature comes closer to the "wanted temperature" of -192 C. This is visible in the right upper corner at the "flow allowed" bar. This is on 100% in the beginning, but will go lower when the "wanted temperature" is approached.

Also when the temperature drops fast, the flowrate is regulated back. For the cooling down period of the application this can mean that the flowrate becomes so low that filling is almost stopped, while the level is not yet reached. This is still OK, because the flowrate will increase if the temperature stays higher than the "wanted temperature" and this takes too long. But, in this way a soft landing on the "wanted temperature" is created.

If the sensor temperature becomes lower than -191 (sensor halfway in the LN2) the pumping will go slower, trying to keep the temperature of the sensor on -191.

If the temperature on the sensor drops too low, pumping will go lower, and if the temperature comes above the -191, pumping will increase. This will keep the level in the application always on about the same.

If the sensor stays too cold for more than the "shut off time" (20 minutes default), pumping shuts off completely, but will be turned ON when the sensor is too warm again. In most applications this will never happen.

Important in this is to have the maximum flowrate (possible to adjust in the calibration screen) not too high, so that the PID is capable of regulating back fast enough. The optimal adjustment would be, that in the "stable" situation (when the level is reached), the "flow allowed" bar is at 30 to 50%. Probably in the stable situation the flowrate needed to keep the level constant will be around 10 mBar only. So if the maximum flowrate would have been set on 200 mBar, the PID should regulate back to 5%, which is a lot. When 10 mBar is needed to keep the level steady, 30 mBar should be high enough as maximum flowrate setting, and the PID can regulate back to 30%, which is a nice setting.

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

Note: For optimal regulation the highest point of the filling hose should be at the end, so that the filling hose will be completely filled with LN2, and a little more pressure "overflows" the filling hose. Do not have the hose in the middle somewhere higher than at the end.

### Pumping too long alarm:

This alarm setting is in the main screen of the monitor software, and is default on OFF.

For the #606 this alarm is of no use.

This alarm can be set if the filling time of your application is known, but for the #606, because it will pump almost continuously, there is no certain time after which the pump should switch off.

**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 water damp 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



## 9 Working for FTIR detectors, model #608

For the #608, when the pump is switched to STANDBY, (by hand on the push button, or automatic by the FTIR instrument), the pump starts building pressure and will fill up the small FTIR dewar until the sensor touches LN2 for more than 80 seconds (default). When the sensor is touched by liquid, the flowrate will be lowered, allowing the boiling effect of reaching the -196 degrees to slow down.

After this the pump will stop pumping, switch to "active" state, and start to count down the interval timer (time remaining value, default on 4:00 hr). After this 4:00 hr, pumping will start again, and pump until the sensor again is touched by the LN2. In this way the detector is always cold.

If in between the pump is switched to SLEEP (by the push button, or with a signal from the FTIR instrument), and put to STANDBY again, a new fill cycle will start, and after that a new wait cycle of 4:00 hr (default). In this way you may start a new filling cycle, to be sure that for the next 4 hours no automatic fill cycle will start. This could be useful before a long measuring cycle in which you do not want an automatic fill cycle.

EXTRA: If the interval time is set on 0 minutes, the pump will switch to SLEEP after the fill action, and should be started manual or with a signal for the next fill action. This can be useful for half automatic purposes.

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

### Adjusting fill pressure:

If the height of the FTIR instrument is set correctly in the pump, the fill pressure should be so, that the small dewar of the detector is filled in about 8-20 minutes. 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 detector.

On the USB stick is an extra program, the 608monitor, with which you can monitor the sensor during filling. The sensor will be cooled during filling to about -175 degrees, and drop below -192 when it is reached by the level. If the fill pressure is too high, you can see that sometimes the sensor drops below the -180, and even below -192 too long, so that the pump can switch off too soon. In that case you should lower the fill pressure a little.

Fill pressure and shut off time can be set in the SERVICE-CALIBRATION screen.

See appendix D for more details of adjusting the fill pressure and how the delayed switching off works.

### Pumping too long alarm:

For most FTIR detectors the normal filling time will be around 10 minutes.

Normally, pumping will continue until the sensor is cold.

In case of a major error, or when there is a leak, and the LN2 does not reach the detector, the sensor will never become cold, so pumping would be endless.

To prevent this, there is the "pumping too long" alarm, from which the default time is set on 30 minutes for the #608. This alarm setting is in the main screen of the monitor software.

This means that when the pump was pumping for more than 30 minutes, this alarm comes in, which blocks all pumping and let the pump beep every 5 seconds until the operator shuts the pump off.

If your application needs a longer filling time than 10 minutes, you may set this alarm value some higher. When the alarm time is set on 0 minutes, this alarm is off.

**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 water damp 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.

## 10 Remote control

The #606 and #608 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.

(Pin 4 is connected with 5 Volt in the standard setup, connecting it to 0 Volt will switch the pump in SLEEP)

With a 5 volt signal on pin 5 of the 25p subDconnector, the pump can be switched to STANDBY.

(Pin 5 is connected with 5 Volt in the standard setup, connecting it to 0 Volt will switch the pump in STANDBY)

Where SLEEP overrides the STANDBY.

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. (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 64 = dewar level lower than 5 liters, 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 pump status result.

For the #606 in one of these ways the application can switch the pumping ON and OFF, keeping the DSC cold or not.

For the #608, the application can switch the pump ON, starting a fill cycle. After 4:00 hours(default) the fill cycle will repeat, until the pump is switched OFF. Switching the pump OFF, and some later again ON, will start a fill cycle, and after that a new wait cycle.

If you want to steer the fill cycles from your application, you could switch the pump OFF and ON, starting a fill cycle, and after some time (5-10 minutes ?), the detector 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 ( < 4:00hr default) . In this way the detector is filled during your pauses, and if the pause should come too late, the automatic fill cycle will make sure that the detector cannot become warm.



## 11 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 password protection. (=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")

## 12 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.)

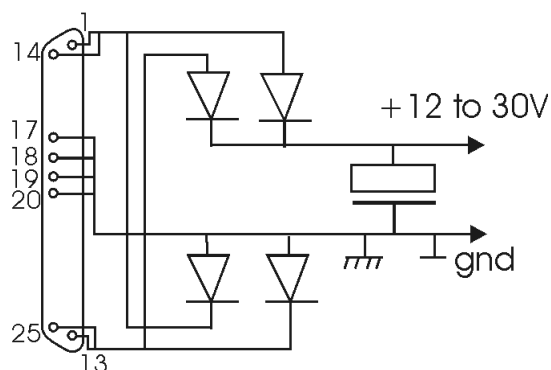
17=18=19=20 system ground

\*\* If you want to use another 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.

## 13 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 is 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 #606 and #608 in the left screen there is only one button, for the external sensor as "self heating" sensor, on 33 mA.

### SELECTION OF EXTERNAL SENSOR TYPE

The external sensor is a PT100 element. The sensor itself is a 1.6 mm glass sensor, 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 sensor is used for level-detection, and is heated continuously by a small current (33 mA) to heat the sensor approx. 20 degrees Celsius above the environment temperature. So if the sensor is in the N<sub>2</sub> 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, 470)

### 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 sensor

For the external sensor, 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). If the value is much lower or higher, 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 self-heating). If you want a very accurate calibration, you may put the sensor in a glass of water, and heat the water up to 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 up to -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. Here for the sensor must be put into LN<sub>2</sub>. Make sure that the sensor is deep into the LN<sub>2</sub>, 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 reset (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. (default may vary between 32 and 52, was factory calibrated)

### **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 +40 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. (default = 237 and 637)

### **Readjusting the pumping flow rate**

In the bottom there is the "flow preset" slide bar, which is on 40 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 internal clock (by reading the calibration byte, making it slower or faster, and writing a new value)

reading the setpoints, binary, voltage and degrees. Use only on factory instructions.

"calibrate ALL 606 (or 608) parameters"

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

## 14 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 some gas to build a small overpressure. Because the fill hose 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 flowrate 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 Pre pressure

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 'pre pressure'.

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 pre pressure is fixed and adjusted to the dewar height. (27 mBar. for 35 Liter dewar, or 32 mBar. for a 50 liter dewar)

\* The second value is the pressure to reach 20cm below 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 20 cm feeding height is 8 mBar, needed to rise from the bottom of the pumphead to the outlet, keeping the level at 20cm lower than the highest point.

\* The third value is the pressure wanted to have a flow suitable for your application. This is preset in the #606 and #608 on 40 mBar. The higher this pressure is set, the faster the LN2 comes out of the fill hose, but too fast may cause the 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 pre pressure

#### 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). For the #606 and #608 the working mode is fixed.

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, a small overpressure is used to raise the LN2 level in the rise pipe to the bottom of the pumping head, to make it as fast as possible to respond on a pumping request later. The pump will create the pressure equal to the "pre pressure", depending on the height of the dewar. (for a 35 liter dewar this is ab. 27 mBar, equal to a level of 13 cm under the dewar top.)

This is kept until the pump goes into PUMPING mode.

Note: the #608 (only) will NOT keep the pre pressure in STANDBY mode, since for FTIR filling it is not important to react as fast as possible.

So for the #608 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 monitor program at the HEATER. It will start at 5% and increases every 2 seconds, up to 100%. It will increase pressure until the pressure is equal to the requested total pressure, depending on 3 values.

1. The pre pressure ( 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 almost the highest point, if set)
- 3 The real pumping pressure (preset on 40 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 on how much LN2 is really pumped.

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

After the pump has finished pumping (depending on what mode the pump is in) and the pump is in STANDBY , the dewar pressure is released to the pre pressure. This means that the LN2 is in the rise pipe at the bottom of the pumphead. This is to have the fastest response for when again LN2 is needed again.

### **FREEZE PROTECTION:**

If the pump is OFF (= in SLEEP mode), a small heater element in the top of the rise pipe will be heated up to 50 degrees, to block the water damp 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 water damp 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 length of the fill hose could be set some longer, to prevent this alarm. This value only sets the sensitivity of the alarm. It has further NO influence on any pumping behavior.

The frozen alarm will also be set when the (blank) pressure measuring tube (tube going down in the LN2) is frozen. Only solution then is to warm up the pump and make sure all water is out of this tube.

### **Pumping too long alarm:**

This alarm setting is in the main screen of the monitor software, and is default on OFF for the #606 and on 30 minutes for the #608.

If you work with an application that should be filled within a certain time, you can set here the time after which the pump will switch OFF and give an alarm when this time is reached.



## 15 Appendix D: detailed working of the 608

The #608 is specially made for filling a small detector, like an FTIR, but also other types of small detectors can be filled with this #608.

The sensor is in self heating mode, so the sensor is warmed a little by electric current. This means that the sensor itself is some warmer than the surrounding air, but when the sensor is IN the LN2, the sensor shows the right temperature of -196 C. In this way the difference between gas and real liquid is detected.

There is an extra mechanism for making sure that the detector is fully filled before the filling shuts off.

This is done by looking to the sensor, and lowering the flowrate when the sensor comes below -192 C.

In the right upper hand corner of the monitor software is the "flow allowed" bar. During filling this is on 100%, allowing 100% of the preadjusted flowrate (40mBar. default?) to be as flowrate pressure.

When the sensor is touched by the LN2 level, OR by splashing on the sensor, and the sensor is below -192 C, the "flow allowed" is reduced by 3%, and every 1 seconds 3 % lower.

If the lowering on the sensor was caused by splashing, this will reduce the splashing so that the sensor will become some warmer again.

If the lowering on the sensor was caused by the real LN2 level reaching the sensor, the reducing of the flowrate will not cause the sensor to become some warmer, but the sensor will stay below -192 C.

When the sensor is below -192 C, also the count down for switching off starts.(visible in the monitor screen next to the sensor). When the sensor is 80 seconds below -192 (while the flowrate then is reduced to 0%), the pump switches to STANDBY mode and starts counting down the interval time for the next filling action.

If the sensor below -192 C was NOT caused by the LN2 level reaching the sensor, but was caused by splashing on the sensor, there will be a moment that the sensor will rise again above the -192 C.

This moment will be before the counting down to shut off is finished.

Then also the "flow allowed" will increase slowly again, while also the counting down for the shut off is reset. This means that the flow will keep increasing until the "flow allowed" is 100% again and filling on 100% flowrate continues.

After this, there will be a moment that again the temperature on the sensor will come below -192 C. Again the "flow allowed" will reduce, and the countdown for shut off will start.

Notice that then the detector will be fuller than the first time.

This mechanism will continue until the detector is fully filled, and the temperature of the sensor is below -192 longer than 80 seconds.

So, also when the flowrate is too high and the sensor is reacting too much on the filling, the detector should be fully filled before the pump shuts off.

But it would be better that the filling is so, that the sensor comes below -192 only when the real LN2 level reaches the sensor.

Trick in this is to make sure that the LN2 stream from the filling comes not directly on the sensor.

Therefor the sensor should be mounted so, that the stream of LN2 is NOT reaching the sensor.

If you have the adaptor for your detector from us, you have also the (black) fill tube of 5mm with the end blocked and the opening in the side. Make sure that it is mounted so, that the fill opening is to one side, and the sensor in the opposite side. In this way the filling should not splash on the sensor directly.

Also make sure that the sensor and the fill opening in the fill tube are below the neck of the detector.

Most detectors have a small neck over more than 20mm, and the detector inside becomes wider below this neck.

Make sure that the sensor and fill opening are in the wide part, and not still in the neck.

If both are in the neck, and the neck is 10 mm wide or so, the LN2 from the filling will reach the sensor directly, making the sensor going below -192 much too fast.

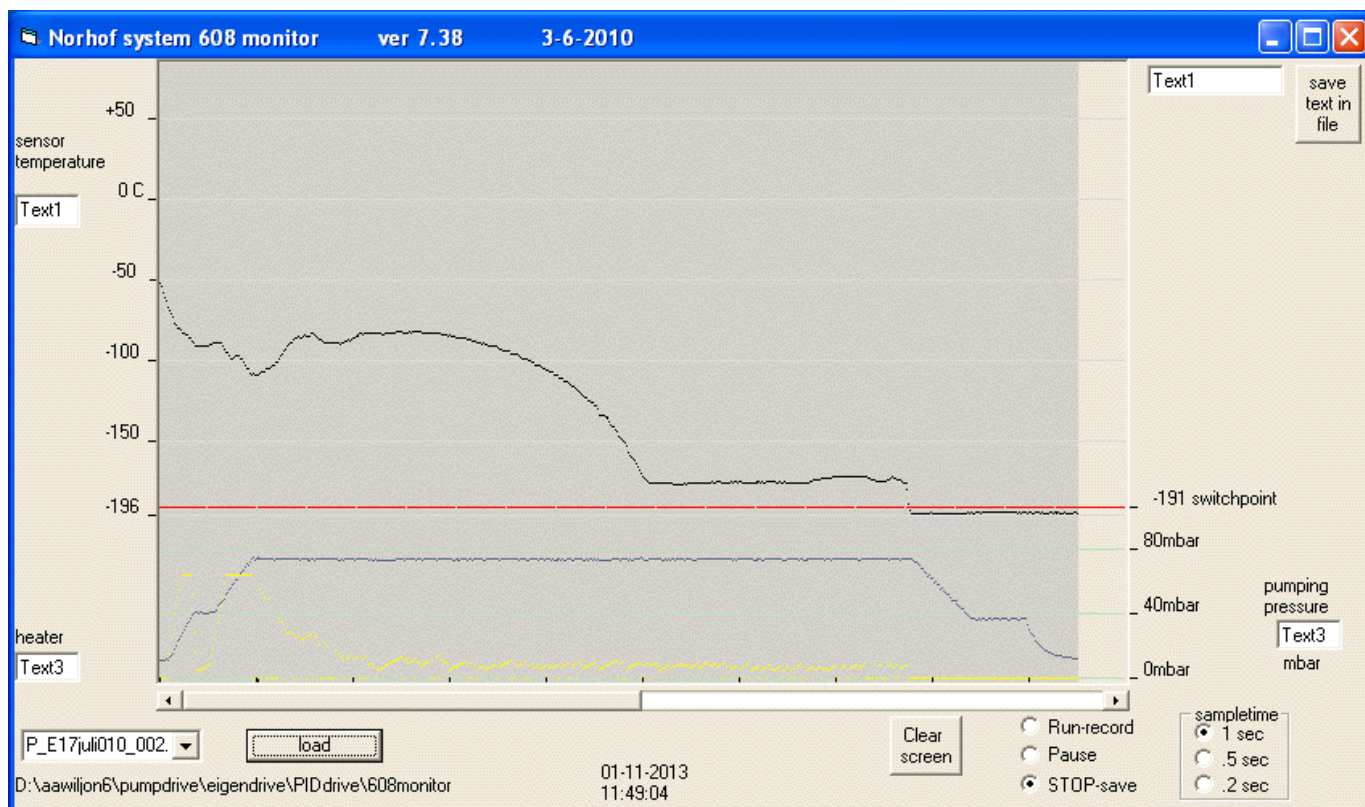


### The 608monitor program.

On the USB stick there is a map "608monitor", which contains the setup of the 608monitor program.

With this program the behavior of the sensor can be watched and logged, while at the same moment the actual flowrate and the heater can be seen.

See here a graph of the filling of a Bruker detector.



This setup was with a 50 liter dewar and the detector on a table. So the pre pressure was 32mBar and 8 mBar was added by the filling height adjustment (20cm). So on 40 mBar the filling flowrate starts. Pressure during sleep mode is about 15 mBar, because the dewar was about half filled. Horizontal scale is 1 minute per stripe (low).

Note that during the first 5 minutes the detector is cooling down, while mostly gas enters the detector, until the sensor is at about -175 C.

During this filling in the next 3 minutes liquid comes and the LN2 level rises in the detector, and the sensor is NOT touched by the LN2 because it stays at about -175 C.

After almost 8 minutes, the LN2 level reaches the sensor, making it drop to -196.

From here, in the next 30 seconds, the LN2 flowrate drops from 80 to 40 mBar, (by the "flow allowed" parameter), so the filling flowrate is zero (dewar pressure 40) in the end, but the sensor does not go higher, meaning that the sensor is really in the LN2 level.

After total 9 minutes, the pump switches off filling, going to STANDBY mode, releasing all the LN2 pressure until 15 mBar.(= NO flow) , and starts counting down the time for the next filling action.

This 608monitor program could help you to make visible the behaviour of the sensor.

It should be possible for you to make the same kind of graph, so that the temperature at the end stays -196.



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**\*\*Note that it is important that you do not set the adjustment for the feeding height to a too higher value than it actual is. When the "flow allowed" parameter is on 0%, there should NOT come any LN2 from the pump.**

If you set the feeding height to the height from the pump exhaust to your detector top, or a little lower, it should work. (mostly the default 20 cm will do, when the detector is on a table)

**\*\*Note that if you increase the filling flowrate, the filling can be faster, but that then the sensor may be splashed sooner, or that the cold gasflow cools down the sensor too much, so that the sensor reaches -192 before the detector is fully filled. This will start the mechanism of regulating down the flowrate, and restarting count down for shut off.**

So it can happen that a lower flowrate will fill your detector sooner than a higher flowrate.

**\*\*Note that when the detector was warm before the filling, the hold time of the detector is some lower than when the detector was already cold. Adjust the parameter for refill time so, that also with a warm detector at the start, the next filling action comes before the detector is dried out.**

## 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 maybe 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.

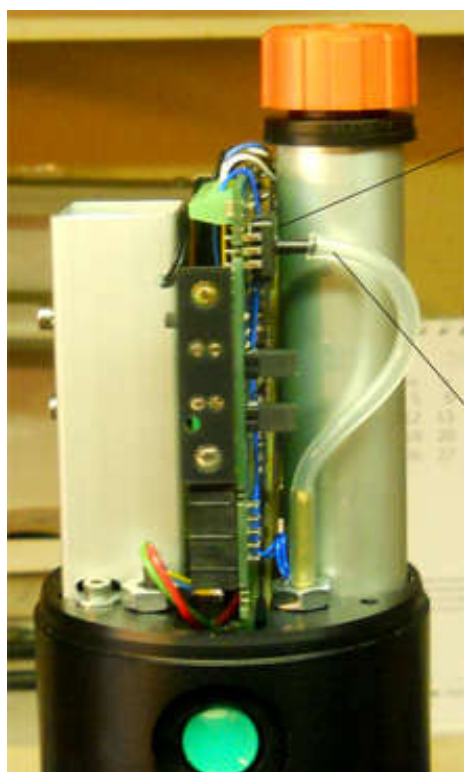
You may blow with dry air from the fill line into the pump, to blow the last water downwards out of the rise pipe. Of course the air should flow freely through this when the ice block is removed.

### **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 trough this tube. Here for, 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 through 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 P.E.D. 99/36/EC compatibility

According to P.E.D. 99/36/EC (Pressure European Directive) for pressurized vessels, systems which are working with a pressure of 0.5 Bar and higher are affected by this directive, and are not allowed in a laboratory. The Norhof system can produce maximum 300 mBar, and therefor this directive does not apply for this system.

## 19 Country of Origin

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

## 20 RoHS compliance



This product does not contain any of the restricted substances referred to in Article 4(1) of the RoHS Directive at concentrations in excess of those permitted under the RoHS Directive EC directive 2002/95/EC and 2002/96/EC



## 21 EU- Declaration of Conformity , RoHS compliance

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

Date: 3 July 2017 Signature: 

## 22 Mounting sensor and fill hose for Bruker detectors

23-9-2016

for use with #608 pump, or #905 pump in mode 3

A #608 pump is preset at the right values for use of a Bruker detector.

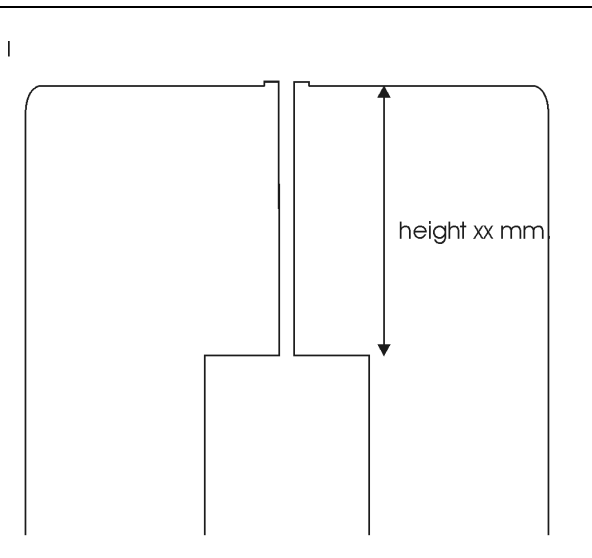
For a #905 pump, mode 3 must be set, at a flowrate of 40 mBar, filling height 20cm, refill time 4:00 hrs (or other depending on the hold-time of the detector).

This sensor and fill hose are universal for all Bruker detectors, but should be adjusted for the different types of detectors which are in the market.

### STEP 1

First measure the height from the top of the inner dewar to the surface of the cover of the machine.

We will adjust the sensor and fill hose so, that the LN2 system will fill up this dewar to 10 mm below the top of this dewar.



### STEP 2

First look where the little hole is in on the end of the (blue) sensor tube.  
Make sure the little hole is on the right side (as in this picture)

Next, make a knick in the blue Teflon hose from the sensor, so that this knick is at the measured height from step 1, + 10 mm, + the thickness of the black cover (=14mm.if it is a standard cover)

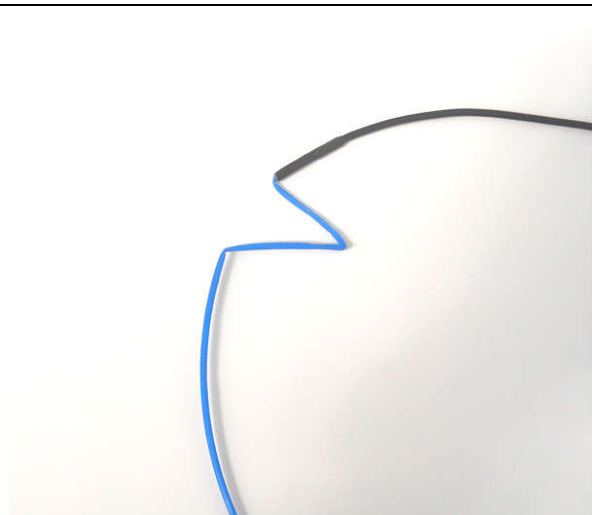
So if the measured height from step 1 = 90 mm, then  
 $90 + 10 + 14 = 114$  mm



### STEP3

Careful fold the blue tube two times so that it will fit for the picture from step 4, so that the black cable is clamped so that there can not come any force on the blue tube when it is pulled.

Tie it together with a piece of rope, wire, or a tire-wrap, or tape.



## STEP 4

Mount the bracket so that the sensor is in place.

Make sure that the rest of the hole is free enough for step 6, the silicon tube of the fill hose.

So mount the sensor so, that the knick is close to the edge of this hole (in picture right below)



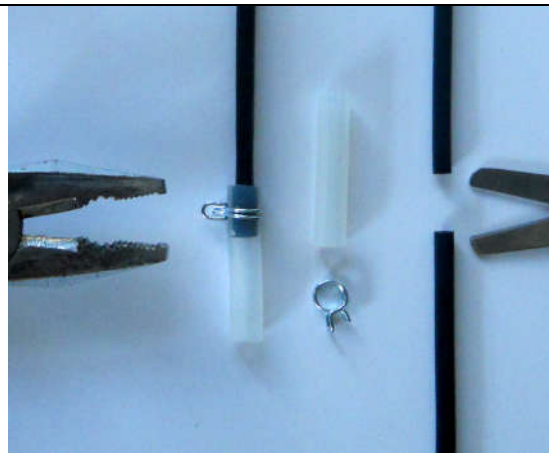
## STEP 5

Cut the (black) fill hose at the size of your measured height from step 1, plus 10 mm, plus the thickness of the black cover (14) , + 3 mm.

If your measured size from step 1 was 90, it will be  $90 + 10 + 14 + 3 = 127$  mm.

Mount the silicone tube of 40 mm over it, so that it is 12 mm !! over the black tube. (not less than 12), and use pliers to place the clamp in the middle of this 12mm

The hole for the silicone tube is 9 mm deep, so the black tube will come 3 mm higher than the top of the black cover.



## STEP 6

Look first where the small opening at the end of the black fill hose is.

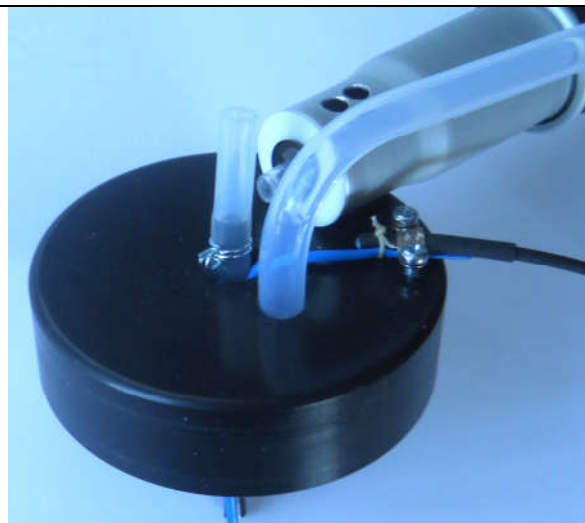
The hole comes at the left side.

Put in the black fill hose, while the blue sensor cable will be clamped.

!!! now the hole in the black fill hose is on the left side, and the little hole in the sensor is at the right side.

The length of the blue sensor tube is your measured size from step 1 + 10 mm, measured from the bottom of this black cover.

The black filling tube is about the same length as the blue sensor hose.



## STEP 7

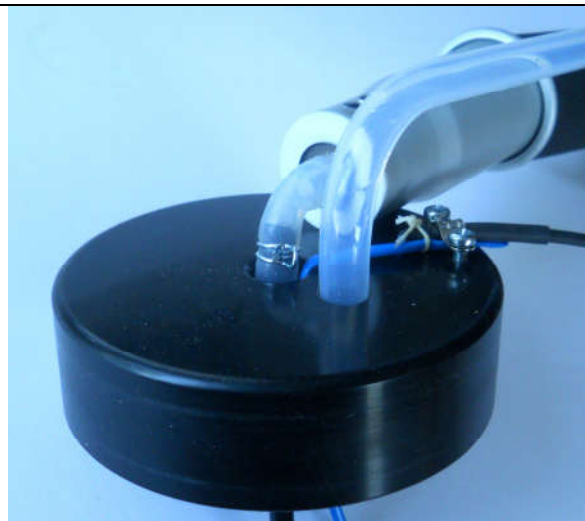
Mount the silicone tube firmly on the fill hose coming from the pump.

Make sure that the knick in the silicone tube is NOT blocking the LN2 flow.

Make sure that when the two hoses are inserted in the detector, that the openings in the hoses are in the opposite direction from each other.

The meaning of this is that the liquid nitrogen comes from the openings in the black tube, and will NOT splash in the opening of the blue tube for the sensor directly.

If LN2 splashes on this opening, the pump could switch off filling while the dewar is not yet fully filled.





- Original instructions -  
Norhof B.V., Ede 2025