Exploring safe and intelligent multi-application liquid nitrogen cooling via auto-fill technology

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The handling of Liquid Nitrogen (LN_2) is not easy and can be extremely hazardous. Scientists are unable to avoid using LN_2 as there are applications requiring the cooling power of the liquid. A system enabling the use of cryogenic cooling without the risk of coming into contact with LN_2 is therefore desirable.

The LN_2 microdosing system is a cryogenic cooling system in which Liquid Nitrogen (LN_2) is used as the cooling medium. LN_2 is transferred from the Dewar to the desired application.

The Norhof LN_2 cooling systems stores the Liquid Nitrogen in pressureless cryogenic Dewars. When a LN_2 transfer is required, a micro processor controlled heater element in the LN_2 generates a small overpressure 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 optimised to the application.

These autonomous cooling systems are designed for use in scientific instrumentation environments and/or in processes that require perfect control over the temperature required. LN_2 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 LN_2 under pressure in which a solenoid valve is used to switch the supply ON/OFF. The system instead delivers a pressure less flow of LN_2.

The pump is software driven and many control modes are already built-in; subzero temperature control, flow control by 0-5V input, control by RS232 line, etc. *Figure 1* shows a software screenshot. The temperature sensor(s) are plugged directly into the pump housing; and not into a separate control box.

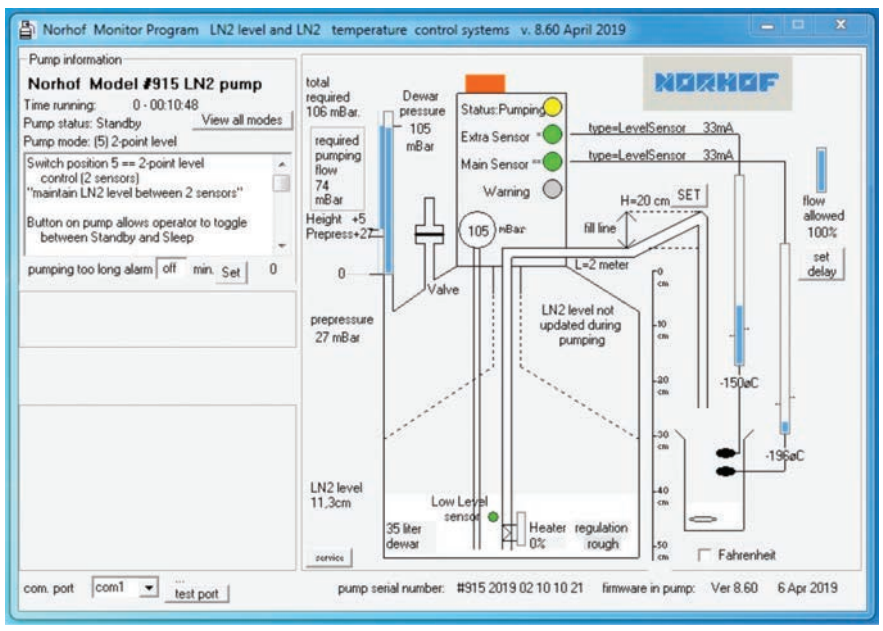

Figure 1: Screenshot Temperature Control Software

Advantages:

The advantages of the system described is that as a LN_2 valve is not required there is no unnecessary heat input and noise. The set up is clean and ergonomic as no additional control unit is required. The software controlled pump can deliver N_2 cold liquid with a flow optimised for the application without noise, vibration and excessive waste. A variable flow feature makes sub-zero temperature control extremely easy. The system is perfect for monitoring and data logging or remote control and connects to a PC quickly and easily. The P.E.D. 99/36/EC (Pressure European Directive) for pressurised vessels does not apply for this system.

As the maximum possible pressure is lower than 300m bar the system can be safely used inside the lab, near your working place.

Temperature controlled applications:

- thermal analysis systems
- computer controlled freezing
- gas chromatography
- temperature stages in microscopes
- sub-zero temperature control systems
- stopping chemical reactions
- cooling of FTIR detectors
- cooling of targets, IR cells, cuvettes etc.

In these applications a system is used in a micro-dosing way to bring just enough cooling to the application as is needed, more at first to achieve the required sub-zero temperature and later less to maintain the temperature. This means that flow control in an analogue fashion of 'more-less' based on temperature measurement is necessary. Each system is designed to be truly universal. Various built-in modes of operation is of paramount importance for OEM and research applications as one system covers almost all possible needs for cooling, without the need for additional valves or control instruments. This means that standardisation within the facility is no longer wishful thinking, despite the varied application demands.

For any system, a host of control possibilities are prerequsites so that users can control it from existing analytical equipment, PLC's, simple switches, computers etc. almost without any adaptations.

Example 1: FTIR detector

The response time and sensitivity of photonic detectors can be much higher if the detector is cooled to decrease the thermal noise. Normally you have to spill LN_2 in a small Dewar to cool the detector down. The LN_2 microdosing system provides a universal automatic filling system for FTIR detectors. It allows long-term measurements and the FTIR spectrometer is able to run overnight. The system is fully safe to use inside the laboratory. It allows an easy setup to most FTIR detectors. *Figure 2* shows a FTIR detector setup.

Figure 2: FTIR detector setup

The pressure above the liquid level inside the Dewar is built by heating a small amount of liquid in the bottom of the Dewar.

With only up to 100 mBar of overpressure, the liquid will gently rise out of the rise pipe and fall into the fill hose. *Figure 3* shows the LN_2 Dewar setup.

Figure 3: LN_2 Dewar setup

Example 2: High-purity germanium (HPGe) detectors

High-purity germanium (HPGe) detectors used for high-resolution spectrometry of gamma radiation need to be cooled well below the ambient temperature to work. Historically, this has been achieved by thermally connecting the detector to a liquid nitrogen (LN_2) pool at -196°C (77 K). In practice, a solid copper rod attached to the detector crystal mount would dip into a Dewar flask filled with LN_2. The need to periodically manually refill the LN_2 from a storage Dewar lead to the development of electrical cryostats in recent years. However, they can still not completely match the performance and security of LN_2 based systems. In applications where performance and/or security are key, an automated LN_2 refill system is the answer.

One such automated, robotic, non-destructive waste assay system was recently developed by DuAl GmbH in cooperation with Norhof. RoboCount(TM) 2020 is used for measurement of radioactive waste packages. The detector carried by the robot is cooled by a Dewar with a LN_2 capacity of 7 l and a 5 day hold time which would normally necessitate a LN_2 refill once or twice a week. This is where the Norhof 900 LN_2 pump came in. Equipped with a 50 l Dewar, it could extend the hold time to (50/7)x5 = 35 days or more than a month. The pump could not be permanently attached to the detector Dewar because if positioned at the base of the robot, the fill line would be too long and its freezing following a refill would prevent robot operations until it warmed up again. To solve this problem a docking station was designed with two cryogenic couplings. The inlet coupling is connected to the Norhof 900 pump and the outlet coupling houses a temperature sensor, also connected to the pump electronics. The mating parts of the couplings are part of the detector assembly and connected to the inlet and outlet ports of the 7 l Dewar, respectively. When the time comes to refill LN_2, the robot positions the detector assembly such that the cryogenic couplings mate. LN_2 flow is initiated and continues until the temperature sensor in the outlet of the docking station detects it. The robot then uncouples the detector assembly from the docking station and operations can continue immediately.

Example 3: Controlled cooling for a thermomechanical analyser (TMA)

A thermomechanical analyser allows a high number of product property measurements.

For example: coefficient of thermal expansion, glass transition determination and thermal stress analysis. For a high number of materials it is necessary to cool the analyser down to low temperatures. The LN_2 microdosing system will be hooked up to a TMA via an adaptor plate and allows temperatures down to -150°C (or lower if necessary).

The benefits include: no manual filling (and spilling); automated system for your safety; delivers LN_2 liquid with a flow optimised for the application - without noise, vibration and excessive waste.

Figure 4: Robot setup with docking station

Now the TMA can be left to run overnight.

New Applications

A solar panel company use the LN_2 microdosing system to autofill the cold traps in the vacuum lines in order to protect the pump from solvents condensating in the pump oil and for extra low vacuum.

A crystal cleaning company use the LN_2 microdosing system to spray/drip LN_2 over crystals in a machine for cleaning purposes.

Conclusions

The systems are extremely flexible and can be used for most LN_2 cooling applications.