## THE ULTIMATE GUIDE TO DEWAR MONITORING AND

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**ALARM** 

# XILTRIX



### The Ultimate guide to Dewar Monitoring and Alarm

**Central question:** How to properly monitor liquid Nitrogen filled dewars, detect issues and send out alarms to prevent damage to the stored samples.

**WARNING:** The test performed and discussed in this paper are carried out by trained professionals in a controlled environment using all necessary safety precautions. Working with liquid Nitrogen is dangerous and has an inherent risk of suffocation when used in a poorly ventilated environment. The destructive tests performed in this paper also carry the risk of explosion because due to a sudden build-up of pressure. Do NOT repeat these tests without the help and supervision of a trained professional and using all appropriate safety precautions!

#### Introduction

Liquid Nitrogen filled cryo storage dewars are the standard for long term tissue storage in many laboratories. Tissues may vary from cord blood to human eggs, embryos or even stem cells, all of which represent a great financial as well as irreplaceability value. These samples need to be kept at a very low temperature to stop biological processes and essentially freeze the tissues in time. The liquid Nitrogen continually evaporates from the dewars lowering the liquid level in the vessel. The dewars are therefore (manually) filled frequently to prevent the liquid from evaporating completely. The temperature of the dewar will only remain low if liquid Nitrogen is inside the vessel. After the liquid Nitrogen has fully evaporated, the temperature will start rising quickly. If the temperature of the storage vessel rises above a critical temperature, tissues inside will incur viability damage or will simply perish.

#### **Storage risks**

Labs using a quality management system, work with a riskbased approach. This means the risks of operating dewars and liquid Nitrogen need to be mapped together with the consequences of the risks. If the risks are great and consequences dire, a mitigation measure will have to be implemented. This white paper will focus on a number of key questions.

- 1. What can go wrong with a dewar from a user perspective?
- 2. How to accurately measure liquid Nitrogen in a dewar?
- 3. What can go wrong with a dewar from a technical perspective?
- 4. How can all these risks be monitored with high accuracy giving the user time to respond and prevent damage?

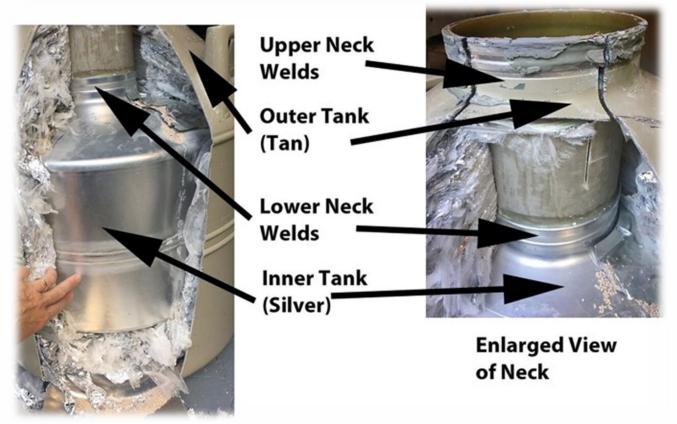


Figure 1: Dewar with ice formation on the cap.

#### How does a dewar work?

A dewar is in essence a very well insulated thermos flask. The inner tank holds the liquid Nitrogen which, in its liquid form, has a temperature of approximately -196°C, depending on atmospheric pressure. If the liquid Nitrogen was to be poured in an uninsulated vessel, it would evaporate in no-time. Therefore, the inner dewar is wrapped in many layers of an aluminium insulating material. This insulated dewar is then placed inside a larger tank with spacers to prevent touching of the two tanks with a material meant to prevent a further transfer of heat.

After completely sealing the space between the outer and inner tanks, the outer tank is pulled to a vacuum to remove all air from the double walled chamber. This final step eliminates air that can carry heat from the outside to the inside of the dewar which would speed up the evaporation process (vacuum insulation).



*Figure 2:* Picture of internal insulation materials of a dewar (source: https://link.springer.com/article/10.1007/s10815-019-01597-5#citeas).

The assumption of many labs is that the temperature inside the dewar is exactly -196°C across the dewar (top and bottom) and that the temperature change will be equal with or without liquid. This is not true; Firstly, the temperature in the dewar is not exactly -196°C across the dewar (something we will demonstrate later). Secondly, the temperature inside the dewar will hold steady for a very long time, only to rise very fast after all liquid has evaporated. This second fact is often overlooked when looking for a monitoring and alarm solution. When the liquid Nitrogen is gone, the time reaction for the user is often so short, with delay times of an alarm and travel time to the lab taken into account, they will be too late to prevent viability damage to the samples.



#### 1. What can go wrong from a user perspective?

Dewars are often stored in relatively cramped spaces with very little room to move around in which there are many identical dewars that need to be filled manually. Aside from being a time consuming and risky task, forgetting to fill one or multiple dewars is a real risk. If a dewar would for example not be filled every week would not mean the samples inside will immediately perish. The most commonly used dewars have a static hold time of up to 2 months.



Figure 3: example of large number of identical dewars on a small footprint.

The smaller the dewar, the shorter the static hold time. Also, the more often the dewar is opened the quicker the liquid Nitrogen will evaporate. Having established that the temperature inside a dewar remains low as long as liquid Nitrogen is still present, monitoring the presence of liquid inside the dewars would be a good mitigation method. Knowing the temperature of the dewar at any given time though would even be better.



Figure 4: Test setup at the cryo room of Cryo Products.



## 2. How to accurately measure liquid Nitrogen level and temperature in a dewar?

Detecting Liquid Nitrogen in an enclosed dewar is challenging due to a number of reasons. Firstly, there is very little space to place sensors. This means the sensor need to have thin cables and must be an minimum obstruction when trying to get to the samples. Secondly, the sensors (including thin wiring) will have to withstand the extremely low temperatures inside the dewar for a very long time. As samples are moved in and out of the dewar, the sensors and wiring undergo extreme temperature differences which puts a lot of stress on components. The following sensing principles can be determined:

#### 1. Weight

liquid Nitrogen level inside dewars can be measured based on full and empty weight of the dewar. By placing a set of scales underneath every dewar, the weight of the dewar is a reliable indicator of the liquid level.

#### **PROS:** Reliable indicator of liquid level

**CONS:** Dependent on dewar size, type, and inventory system, expensive, bulky (takes up large footprint), mostly no connection to remote monitoring, no temperature measurement of samples

#### 2. Differential pressure sensor

Actual measurement of liquid Nitrogen is often done by differential pressure. The weight of the liquid Nitrogen moves air in a pressure tube measured by a pressure sensor. This value is then translated into a level measurement for the vessel.

**PROS:** Reliable indicator of liquid level, actual level measurement **CONS:** Expensive, does not fit inside small dewars, prone to breaking, mostly no connection to remote monitoring, no temperature measurement of samples

#### 3. Capacitive sensors

Indicative measurement of liquid Nitrogen is also done by several capacitive temperature-based sensors. The level can be presented in steps (i.e., 25% - 50% - 75% - 100%).

**PROS:** Indicative indicator of liquid level, actual level indication **CONS:** Expensive, does not fit inside small dewars, prone to breaking, mostly no remote monitoring, no temperature indication of samples

#### 4. Temperature/minimum level sensor inside the dewar

An accurate PT100 temperature sensor is installed inside the dewar, placing the tip of the sensor at a predetermined minimum level. If the level drops below the tip of the sensor, the temperature change is measured. This sensor acts as a combined temperature and minimum level sensor for dewars.

**PROS:** Reliable indicator of liquid level, minimum level measurement, very cheap, long lasting **CONS:** no actual level measurement.

#### Conclusion

Weighing all the pros and cons of dewar monitoring solutions yields several technical, space constraint, cost, and robustness issues. The only solution which is both relatively inexpensive and robust is the temperature sensor that can double as a minimum level sensor. The main question is if this type of monitoring is sufficiently capable of triggering an alarm during all perceivable issues (both user and technical).



#### 3. What can go wrong with a dewar from a technical perspective?

Dewars, being large thermos flasks with a high level of insulation, are capable of holding liquid Nitrogen in for a long time. Looking at the design of a dewar there is a number of things that can happen to the dewar itself that will have an effect on the performance of the unit. The impact on the hold time of the dewar will be profound, but without proper measurements, it is unclear whether temperature/ (minimum) level sensors will be able to detect issues and send an alarm quickly enough to prevent damage.



Figure 5: CryoLow level sensing device.

In order to test whether the temperature sensor is a sufficiently robust instrument to monitor dewar failures, a series of tests has been setup. With help of the company <u>Cryo Solutions</u>, the <u>XiltriX - Real-time monitoring & alarm system</u> was used to measure 3 temperature sensors placed in a set of test vessels. The test vessels had a liquid Nitrogen volume of 47L & 10L. The 10L dewars is one of the smallest dewars used for long term tissue storage. The smallest dewar also has the lowest surface-area-to-volume ratio (SA:V), where heat conduction has an increased impact on evaporation (source: <u>https://en.wikipedia.org/wiki/Surface-area-to-volume ratio</u>).

Two PT100 temperature sensors were placed inside the liquid Nitrogen. One sensor was placed on the outside of the dewar and one <u>CryoLow</u> sensor was used to detect liquid level drops.

Sensing device	Measurement principle	Placement level
PT100 sensor 310 mm	Temperature sensor	Placed 10 cm from dewar bottom
PT100 sensor 160 mm	Temperature sensor	Placed 25 cm from dewar bottom
CryoLow level detection	Temperature level sensor	Placed 10 cm from dewar bottom
PT100 surface sensor	Temperature sensor	Placed at neck level of dewar



*Figure 6:* PT100 temperature sensors placed inside and on the outside of a 10L dewar.

#### **TEST 1 - Liquid level inside dewar drops below tip of temperature sensor**

In order to determine a monitoring baseline, the temperature sensors were placed inside the dewar together with the <u>CryoLow</u> level detection device. This device uses a responsive and sensitive temperature sensor to determine when the liquid Nitrogen level drops below the tip of the sensor. This sensor is placed alongside the PT100 temperature sensor at the same height. This means the expected behaviour is that both sensors would detect a liquid Nitrogen level drop at the same time.

#### **Results**

The results of this test took a very long time (multiple days). Dewars are designed to keep the liquid Nitrogen in its liquid form for a long time. The CryoLow level detection sensor was the first to detect the drop in Liquid Nitrogen level, closely followed by the PT100 temperature sensor.

The CryoLow is able to provide an alarm when it detects a low level but has no temperature output to provide temperature data. When zooming into the detailed graph of the PT100 temperature sensor the sensor stays at roughly -196°C until the liquid level drops below the tip of the sensor. As soon as the tip of the sensor is no longer submerged in the liquid Nitrogen, within 5 minutes the measured temperature increases a little over **2.2°C**. After the increase in temperature step, the temperature remains stable again for a long period of time. Only until all the liquid had evaporated did the temperature rise further.

To confirm this 'sudden temperature increase', the test was repeated with a different brand and type of dewar, but of the same 10L size. Also another set of measuring hardware and sensors was used to validate the results. It turned out the results were identical, with the same >2°C temperature rise in a <10 minutes time frame.

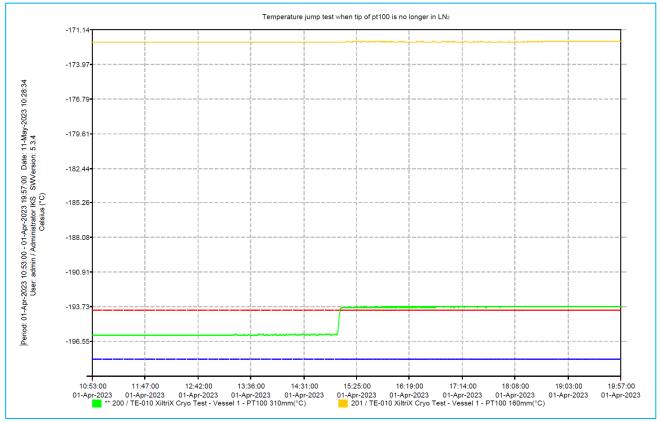


Figure 7: Results of all temperature sensors attached during the hold test.

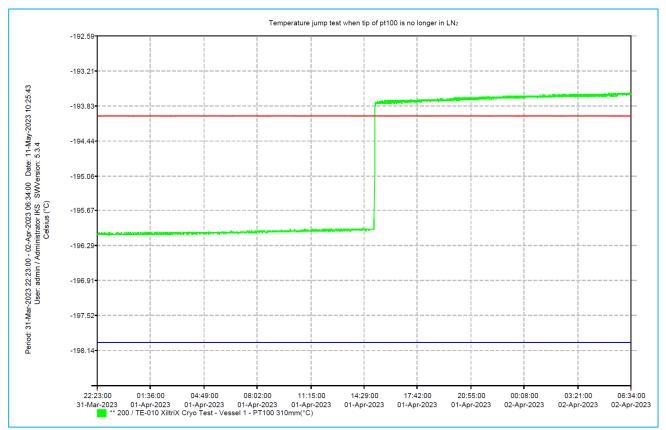


Figure 8: Temperature jump after liquid Nitrogen level drops below temperature sensor tip.

				Numerica	I data			
	Analogu	e channel 2007	re-010 Xiltri)	K Cryo Test - Ve	ssel 1 - PT100 310	mm	-	
Local (CEST)	Raw value	Evaluated value	Lo	Hi	Door switch	High/low switch	Start/stop switch	GMT
01-Apr-2023 14:50:04	-20,465.0	-196.0	-198.0	-194.0				01-Apr-2023 12:50:04
01-Apr-2023 14:51:04	-20,463.0	-196.0	-198.0	-194.0				01-Apr-2023 12:51:04
01-Apr-2023 14:52:04	-20,464.0	-196.0	-198.0	-194.0				01-Apr-2023 12:52:04
01-Apr-2023 14:53:04	-20,463.0	-196.0	-198.0	-194.0				01-Apr-2023 12:53:04
01-Apr-2023 14:54:04	-20,463.0	-196.0	-198.0	-194.0				01-Apr-2023 12:54:04
01-Apr-2023 14:55:04	-20,463.0	-196.0	-198.0	-194.0				01-Apr-2023 12:55:04
01-Apr-2023 14:56:04	-20,463.0	-196.0	-198.0	-194.0				01-Apr-2023 12:56:04
01-Apr-2023 14:57:04	-20,463.0	-196.0	-198.0	-194.0				01-Apr-2023 12:57:04
01-Apr-2023 14:58:04	-20,463.0	-196.0	-198.0	-194.0				01-Apr-2023 12:58:04
01-Apr-2023 14:59:04	-20,464.0	-196.0	-198.0	-194.0				01-Apr-2023 12:59:04
01-Apr-2023 15:00:04	-20,464.0	-196.0	-198.0	-194.0				01-Apr-2023 13:00:04
01-Apr-2023 15:01:04	-20,463.0	-196.0	-198.0	-194.0				01-Apr-2023 13:01:04
01-Apr-2023 15:02:04	-20,464.0	-196.0	-198.0	-194.0				01-Apr-2023 13:02:04
01-Apr-2023 15:03:04	-20,463.0	-196.0	-198.0	-194.0				01-Apr-2023 13:03:04
01-Apr-2023 15:04:04	-20,463.0	-196.0	-198.0	-194.0				01-Apr-2023 13:04:04
01-Apr-2023 15:05:04	-20,464.0	-196.0	-198.0	-194.0				01-Apr-2023 13:05:04
01-Apr-2023 15:06:04	-20,445.0	-195.8	-198.0	-194.0				01-Apr-2023 13:06:04
01-Apr-2023 15:07:04	-20,354.0	-195.0	-198.0	-194.0				01-Apr-2023 13:07:04
01-Apr-2023 15:08:04	-20,272.0	-194.3	-198.0	-194.0				01-Apr-2023 13:08:04
01-Apr-2023 15:08:41	-20,243.0	-194.0	-198.0	-194.0				01-Apr-2023 13:08:41
01-Apr-2023 15:08:51	-20,236.0	-193.9	-198.0	-194.0				01-Apr-2023 13:08:51
01-Apr-2023 15:09:01	-20,236.0	-193.9	-198.0	-194.0				01-Apr-2023 13:09:01
01-Apr-2023 15:09:04	-20,236.0	-193.9	-198.0	-194.0				01-Apr-2023 13:09:04
01-Apr-2023 15:09:11	-20,236.0	-193.9	-198.0	-194.0				01-Apr-2023 13:09:11
01-Apr-2023 15:09:21	-20,231.0	-193.9	-198.0	-194.0				01-Apr-2023 13:09:21
01-Apr-2023 15:09:31	-20,228.0	-193.8	-198.0	-194.0				01-Apr-2023 13:09:31
01-Apr-2023 15:09:41	-20,226.0	-193.8	-198.0	-194.0				01-Apr-2023 13:09:41
01-Apr-2023 15:09:51	-20,226.0	-193.8	-198.0	-194.0				01-Apr-2023 13:09:51
01-Apr-2023 15:10:01	-20,226.0	-193.8	-198.0	-194.0				01-Apr-2023 13:10:01
01-Apr-2023 15:10:04	-20,226.0	-193.8	-198.0	-194.0				01-Apr-2023 13:10:04
01-Apr-2023 15:10:11	-20,226.0	-193.8	-198.0	-194.0				01-Apr-2023 13:10:11
01-Apr-2023 15:10:21	-20,224.0	-193.8	-198.0	-194.0				01-Apr-2023 13:10:21
01-Apr-2023 15:10:31	-20,224.0	-193.8	-198.0	-194.0				01-Apr-2023 13:10:31
01-Apr-2023 15:10:41	-20,224.0	-193.8	-198.0	-194.0				01-Apr-2023 13:10:41
01-Apr-2023 15:10:51	-20,223.0	-193.8	-198.0	-194.0				01-Apr-2023 13:10:51
01-Apr-2023 15:11:01	-20,223.0	-193.8	-198.0	-194.0				01-Apr-2023 13:11:01
01-Apr-2023 15:11:04	-20,220.0	-193.8	-198.0	-194.0				01-Apr-2023 13:11:04
01-Apr-2023 15:11:11	-20.220.0	-193.8	-198.0	-194.0				01-Apr-2023 13:11:11

Figure 9: Numerical values indicating the rapid rise in temperature in a very short time frame.

#### Conclusion

Both the CryoLow & temperature sensor can be used effectively for the minimum level sensing of liquid Nitrogen. The CryoLow has the benefit of reacting slightly faster to a drop of liquid level compared to the PT100 temperature sensor but is has no temperature output and requires a connection to mains power to remain functional.

The PT100 temperature sensor is very capable in sensing the decrease in liquid level as long as the sensor and monitoring system used are accurate and fast responding to detect the rise in temperature. The high alarm threshold will need to be set no higher than -194°C for it to effectively sense the temperature step which enables it to raise an alarm.

#### **TEST 2 - Loss of vacuum**

Dewars have a double wall design that is highly insulated. The insulating capabilities come both from the aluminium wrapping of the inner dewar, as well as the vacuum insulation between the inner and outer dewar. The vacuum is not absolute and will decrease over time. Next to the deterioration of the vacuum, physical damage or corrosion are not uncommon. This can be a puncture of the outer aluminium skin, or damage to the plastic vacuum seal (one-way valve) used to extract residual air. Both issues will lead to a loss of vacuum. We have accurately timed how fast liquid Nitrogen will evaporate and the rise of temperature inside the dewar.

#### **Test setup**

Device	Measurement principle	Level
PT100 sensor 310 mm	Temperature sensor	Placed 10 cm from dewar bottom
PT100 sensor 160 mm	Temperature sensor	Placed 25 cm from dewar bottom
PT100 surface sensor	Temperature sensor	Placed at neck level of dewar

The 10L dewar was filled to the brim with liquid Nitrogen, to a higher level than users would normally fill it. After filling, the XiltriX temperature sensors, together with the foam cap, were inserted back into the dewar. When all temperatures stabilized the outer wall of the dewar was punctured using a ø 6mm drill. When the skin was fully punctured a loud hissing sound of air rushing into the insulated wall was perceived, directly followed by a violent evaporation of the liquid Nitrogen inside the dewar. A movie clip is available: <u>What happens with a dewar when the vacuum is removed?</u> This was followed by a violent evaporation of the Nitrogen inside the dewar.



Figure 10: PT100 surface temperature sensor taped to the outside of the dewar surface.

#### **Results**

The loss of vacuum jumpstarts a lot of energy transfer between inside and outside dewar walls, boiling aggressively and at the same time lowering the temperature of the outside wall to just below freezing at -2°C. The evaporation speeds up and is first detected at the liquid level for the 160 mm PT100 sensor, followed sometime later by the PT100 sensor sitting lower inside the dewar.

Time	Sensor
11:00 (t=0)	drilling the hole into the skin
11:33 (33 mins)	Surface temperature sensor reaching 0°C
12:16 (1h 16 mins)	160 mm PT100 temperature sensor reaching -194°C
15:51 (4h 51 mins)	160 mm PT100 temperature sensor reaching -180°C
16:00 (5 hours)	310 mm PT100 temperature sensor reaching -194°C
18:48 (7h 48 mins)	310 mm PT100 temperature sensor reaching -180°C
19:50 (8h 50 mins)	All liquid Nitrogen has evaporated
22:25 (11h 25mins)	Temperature sensors >0°C



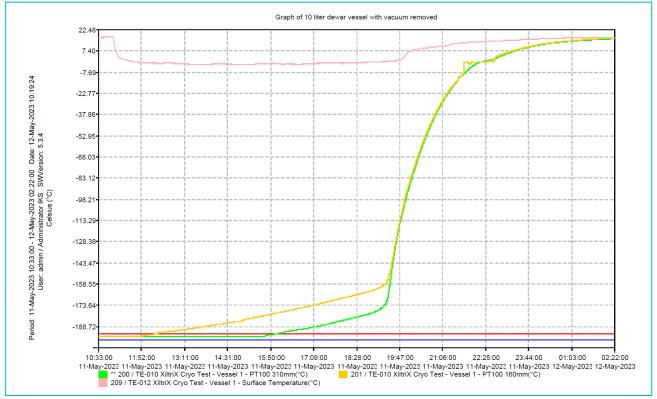


Figure 11: Graph of all temperature sensor with the vacuum removed.

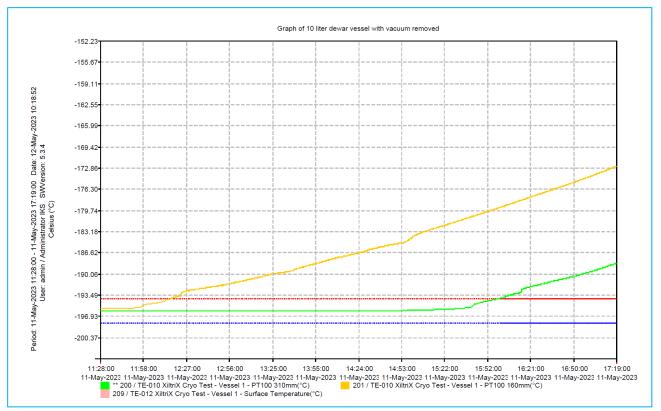


Figure 12: Detailed view of the two temperature sensor inside the dewars at different levels.

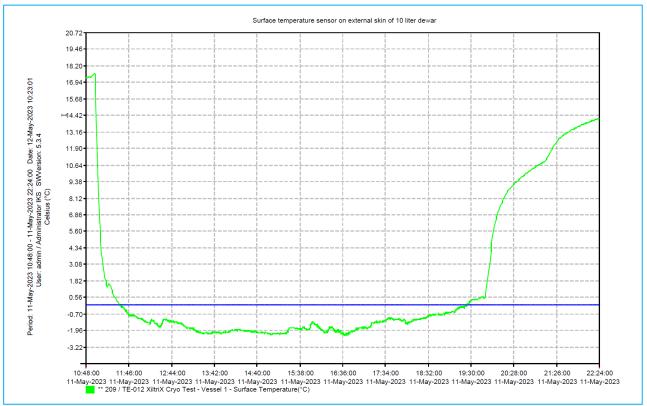


Figure 13: Temperature graph of the sensor on the outer skin of the dewar.

#### Discussion

The PT100 sensor placed on the outside of the dewar was the quickest to respond to the loss of vacuum. The test though was however performed with a sudden and complete loss of vacuum. If a slow loss of vacuum was to occur, the speed and extent of the temperature change will be different. Analysing the data shows that just a surface temperature on the outside of the dewar is good way to predict a catastrophic problem. There is however no way to predict how the temperature measurement will change in other situations. For this reason, an external temperature sensor alone would not mitigate all the risks.

Both PT100 sensors installed inside the dewar are effective in detecting the decrease in level. The sensor placed high up in the dewar neck (160 mm) detects the level drop the fastest. The longer sensor (310 mm) logically emerges from the liquid level at a later time. Relying on a single 310 mm long PT100 sensor monitoring precious samples, the increase in temperature from -194°C to -180°C provides the on-call or user with roughly **2 hours and 48 minutes** response time. Having a proper real-time monitoring solution in place, this should give the on-call ample time to response and save sensitive material.

When using a 160 mm sensor placed higher up in the tank for monitoring, the response time will be extended up to **6 hours and 30 minutes**. This would mean dewars would have to be topped up more frequently to prevent false alarms as the liquid level would drop more quickly below the threshold level.



#### **TEST 3 - Puncture of internal dewar at neck level**

By removing the vacuum the insulating qualities of a dewar are drastically lowered. This is not the most catastrophic of failures that can happen to a dewar. If the internal tank of the dewar would crack or rupture it would allow liquid Nitrogen to infiltrate the outer skin, amplifying the surface available for heat transfer. The welds at the neck of the dewar experience the added stress of temperature fluctuations more than the lower welds. Metal fatigue at the welds increases the chance of cracks at this location. The following test will showcase the behaviour of a dewar with a puncture of the inner skin at neck level.

#### **Test setup**

Device	Measurement principle	Level
PT100 sensor 310 mm	Temperature sensor	Placed 10 cm from dewar bottom
PT100 sensor 160 mm	Temperature sensor	Placed 25 cm from dewar bottom
PT100 surface sensor	Temperature sensor	Placed at neck level of dewar

**Please note:** The outside skin of the dewar was drilled to prevent a dangerous increase in pressure inside the dewar. Without these extra holes the test was deemed too dangerous, due to an explosion risk. Do not try to replicate this test without taking ALL necessary safety precautions!

A 10L dewar was prepared with a puncture of the inner skin at neck level. The dewar was filled to the brim with liquid Nitrogen, to a higher level than users would normally fill it. After filling, the XiltriX temperature sensors, together with the foam cap, were inserted back into the dewar. The liquid Nitrogen was able to partially drain through the hole at neck level infiltrating the space between the inner and outer skin. The raised surface area allows for a higher heat transfer and should therefore result in a quicker heat up of the whole dewar.



**Figure 14:** Position of neck puncture. X marks the spot of the neck level puncture.

#### Results

The puncture of the neck resulted in a very fast evaporation of the liquid Nitrogen. The temperature of the surface temperature sensor was fastest to respond and dropped to -27°C fast. The PT100 sensor high up in the dewar was again the first to show a decrease in liquid Nitrogen level, followed by the lower sensor in a similar manner. After the complete evaporation of all liquid Nitrogen the outside surface temperature sensor heated quickly again.

Time	Sensor
10:56 (t=0)	Filling the prepared tank with liquid Nitrogen
10:59 (3 mins)	Surface temperature sensor reaching 0°C
13:13 (2h 17 mins)	160 mm PT100 temperature sensor reaching -194°C
17:48 (6h 52 mins)	160 mm PT100 temperature sensor reaching -180°C

19:03 (8h 7 mins)	310 mm PT100 temperature sensor reaching -194°C		
20:52 (9h 56 mins)	310 mm PT100 temperature sensor reaching -180°C		
20:55 (9h 58 mins)	All liquid Nitrogen has evaporated		
23:37 (12h 41mins)	Temperature sensors >0°C		

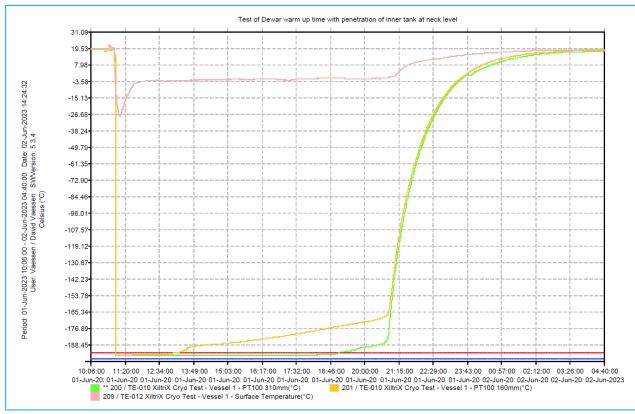


Figure 15: Behaviour a dewar with puncture of inner skin at neck level.

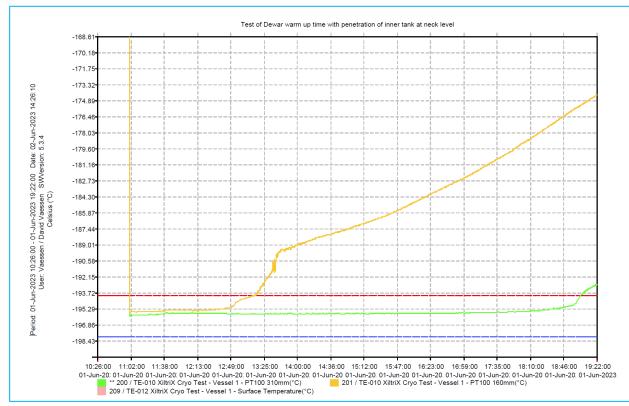


Figure 16: Detailed view of internal temperature sensor in a dewar with a puncture at neck level.

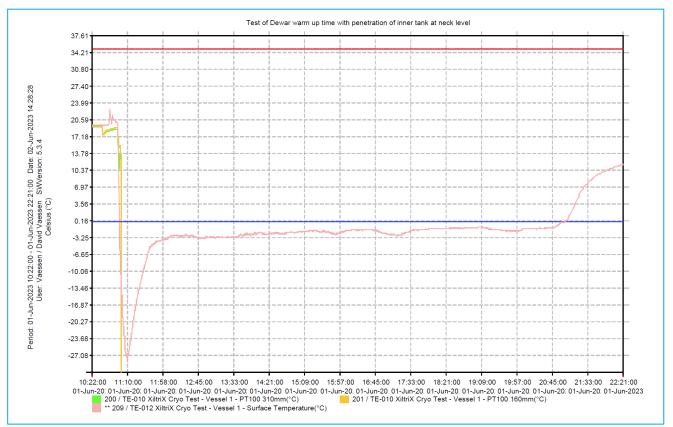


Figure 17: Behaviour of surface temperature sensor in dewar with a puncture at neck level.

#### Discussion

The PT100 sensor placed on the outside of the dewar was quickest to respond to the puncture at neck level. The test though was done with a larger puncture at the neck level. If a smaller puncture were to occur, the speed and extent of the temperature change will be different. Analysing the data shows that just a surface temperature on the outside of the dewar is good way to predict a catastrophic problem. There is however no way to predict how the temperature measurement will change in other situations. For this reason, an external temperature sensor alone would not mitigate all the risks.

Both PT100 sensors placed inside the dewar are effective in detecting the level drop. The sensor placed high up in the dewar neck (160 mm) detects the level drop the fastest. The sensor placed lower down (310 mm) takes longer to sense the liquid level drop. If a user would only use the 310 mm sensor to monitor their samples, the response time from reaching the -194°C to -180°C would be **1 hours and 49 minutes**. Having a proper real-time monitoring solution in place, this should give the on-call ample time to response and save sensitive material.

When using a 160 mm sensor placed higher up in the tank for monitoring, the response time will be expanded up to **7 hours and 39 minutes**. This does mean dewars would have to be topped up more frequently to prevent false alarms because the liquid level would drop more quickly below the threshold level.



#### **TEST 4 - Puncture of dewar at bottom level**

By removing the vacuum, the insulating qualities of a dewar are drastically lowered. This is not the most catastrophic of failures that can happen to a dewar. If the internal skin of the dewar would crack or rupture it would allow liquid Nitrogen to infiltrate the outer skin, amplifying the surface available for heat conductivity. The bottom of the dewar would be the most critical area of a tank to crack with liquid draining at the highest rate. This test will showcase the behaviour of

*Please note:* The outside skin of the dewar was drilled to safely release the pressure that was built-up in the dewar. Without these holes the pressure build up could have resulted in an explosion. Do not try to replicate this test without taking the necessary safety precautions!

a dewar with a puncture of the inner skin at bottom level.

#### **Test setup**

Device	Measurement principle	Level
PT100 sensor 310 mm	Temperature sensor	Placed 10 cm from dewar bottom
PT100 sensor 160 mm	Temperature sensor	Placed 25 cm from dewar bottom
PT100 surface sensor	Temperature sensor	Placed at neck level of dewar

A 10L dewar was prepared with a puncture of the inner skin at the bottom level. The dewar was filled to the brim with liquid Nitrogen, to a higher level than users would normally fill it. After filling, the XiltriX temperature sensors, together with the foam cap, were inserted back into the dewar. The liquid Nitrogen was able to partially drain through the hole at bottom level infiltrating the space between the inner and outer skin. The bigger surface area allows for a faster heat transfer and should therefore result in a quicker heat up of the whole dewar. A clip of the filling can be viewed here: XiltriX - Filling a dewar with puncture of the inner skin.

#### Results

The puncture of the neck results in a very fast evaporation of the liquid Nitrogen. The temperature of the surface temperature sensor was fastest to respond and dropped to -127°C fast. The PT100 sensor high up in the dewar was again the first to show a decrease in liquid Nitrogen level, followed by the lower sensor in a similar manner. After the complete evaporation of all liquid Nitrogen the outside surface temperature sensor heated quickly again.

Time	Sensor
13:20 (t=0)	Filling the prepared tank with liquid Nitrogen
13:21 (1 mins)	Surface temperature sensor reaching 0°C
13:26 (6 mins)	160 mm PT100 temperature sensor reaching -194°C
13:29 (8 mins)	160 mm PT100 temperature sensor reaching -180°C
13:50 (29 mins)	310 mm PT100 temperature sensor reaching -194°C
14:20 (60 mins)	All liquid Nitrogen has evaporated
15:06 (1h 45 mins)	310 mm PT100 temperature sensor reaching -180°C
18:16 (4h 56 mins)	Temperature sensors >0°C

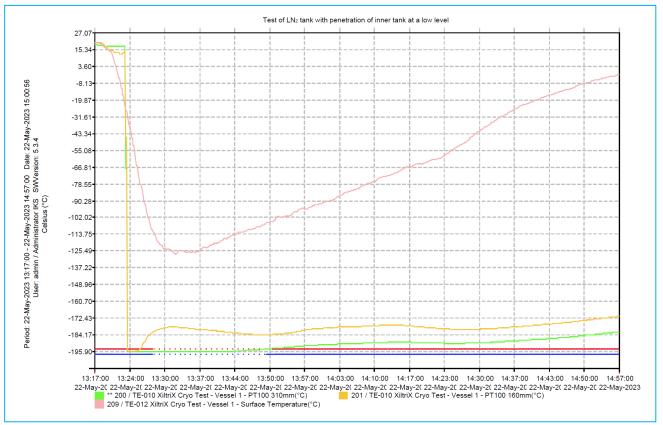


Figure 18: Test of dewar with a puncture of the inner skin at bottom level.

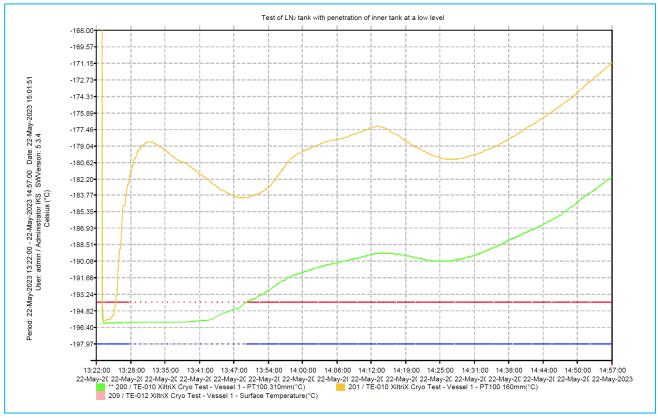


Figure 19: detailed view of internal temperature sensors inside a dewar with a puncture at bottom level.

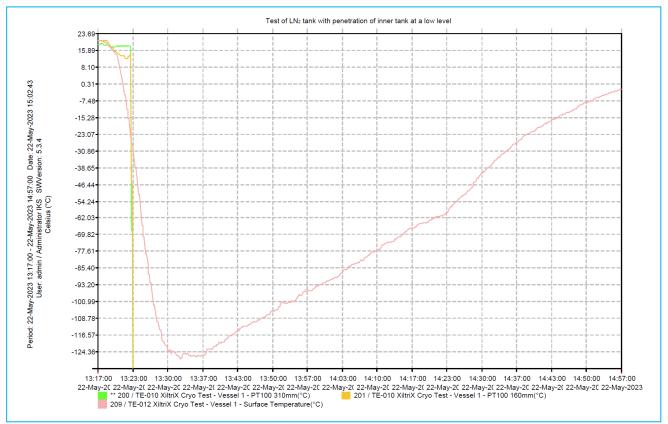


Figure 20: Surface temperature graph for dewar with a puncture of inner skin at bottom level.

#### Discussion

The PT100 sensor placed on the outside of the dewar was the fastest to respond to the puncture at bottom level. The test though was done with a large puncture at the bottom level. If a smaller puncture were to occur, the speed and extent of the temperature change will be different. Analysing the data shows that just a surface temperature on the outside of the dewar is good way to predict a catastrophic problem. There is however no way to predict how the temperature measurement will change in other situations. For this reason, an external temperature sensor alone would not mitigate all the risks.

Both PT100 sensors placed inside the dewar are effective in detecting the level drop. The sensor placed high up in the dewar neck (160 mm) detects the level drop the fastest. The sensor placed lower down (310 mm) takes longer to sense the liquid level drop. If a user would only use the 310 mm sensor to monitor their samples, the response time from reaching the -194°C to -180°C would be **1 hours and 16 minutes**. Having a proper real-time monitoring solution in place, this should give the on-call ample time to response and save sensitive material.

When using a 160 mm sensor placed higher up in the tank for monitoring, the response time will be expanded up to **1 hours and 40 minutes**. This does mean dewars would have to be topped up more frequently to prevent false alarms because the liquid level would drop more quickly below the threshold level.



#### **Final Conclusion**

The performed tests were to determine if using temperature sensors as a form of minimum level detection would be a robust solution for monitoring dewars. The results varied widely both temperature behaviour and time a user would have to effectively prevent damage if an issue was to happen in the middle of the night without anybody being in the facility.

All temperature sensors are effective at indicating catastrophic dewar failures in various forms. The temperature sensor placed on the outside of the dewar skin is always the quickest to respond. This sensor will provide the fastest alarm when connected to a remote monitoring system. It drops to a low temperature level quickly, but it is hard to predict what that level will be. Setting this sensor up correctly (both position and alarm limits) will be trial and error and it is impossible to test for every circumstance. It cannot be predicted to what level the temperature will drop if a failure will occur. Noteworthy, the ambient room temperature of the room will have an effect on the temperature measurement, so the chances of incurring false alarms are relatively high. Furthermore, the sensor placed on the outside is much more exposed and therefore prone to damage inflicted by moving around dewars. As the sensor is not placed among the actual samples, it has no added value for temperature measurement. It would not be recommended to **only** use this type of sensor for dewar monitoring, but it can have a **beneficial** effect on the response time to a catastrophic failure.

Using internally placed temperature sensors as minimum liquid level sensors always results in a reliable measurement and alarm event. The internal sensors are not affected by ambient conditions and their behaviour is therefore much easier to predict. By using an alarm limit at **-194°C** users have enough time to respond to an alarm **if the monitoring and alarm system is measuring and alarming in real-time**. In case of a major catastrophic failure of a dewar the time to respond can be as short as **1 hour and 16 minutes** before the lower samples reach a storage temperature above **-180°C**. This temperature is considered to be a critical temperature for storage of human eggs.

These test show how critical dewar failures can have a **catastrophic effect on the temperature conditions** of the stored samples. It also proves the importance of using a proper real time monitoring and alarm system in cryogenic storage facilities, for example IVF labs storing human seamen, eggs, and embryos. Using a non-real time monitoring system (for example data loggers) will result in a loss of response time that will dramatically increase the risk of damage to e.g. embryos. Beside measuring the temperatures, alarm delays should be set in a **flexible manner**. During office hours longer delay times are necessary to prevent false alarm. Outside office hours shorter delay times provide additional response time to the user. And finally, alarm notifications should allow for an **aggressive cascading mechanism with active acknowledgement**.

A system fit for this purpose is the <u>XiltriX real-time monitoring and alarm system</u>. For over two decades XiltriX successfully safeguards irreplaceable samples in cryogenic storage facilities all over the world.



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We hope that our information will help to make long term cryo storage in dewars a little safer and prevent unnecessary loss of tissue or even life.

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