UNIVERSITY of WASHINGTON

REPORT | LIQUID NITROGEN

Liquid Nitrogen and Alarms in University Research Space June 18, 2014

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I. PURPOSE

This report is intended to provide guidance on identifying and evaluating potential risks related to storage of liquid nitrogen in laboratory space, and how to best mitigate those risks at the University of Washington (UW). This paper provides an analysis of the available literature on the subject, example calculations of risk, and suggestions of best practices to detect an unsafe environment from liquid nitrogen and other cryogenic material spills and releases in rooms and spaces.

II. BACKGROUND

Cryogenic liquids are usually taken to be those that boil below some specified temperature at one atmosphere (atm) of pressure₁. Temperatures vary depending on the application or reference from 123 to 300oK (K = -273oC = -460oF)_{1, 2}. The National Institute of Standards and Technology (NIST) defines a cryogenic as a liquid with a normal boiling point below - 130 oF (-90oC)₃.

Although there are numerous cryogenic liquids, this paper will focus only on liquid nitrogen for several reasons: the large number of users of LN2 at the UW, the large quantities stored and used in several locations, the volume of liquid stored in open containers or Dewars compared to gas cylinders, and hazardous characteristics surrounding LN2.

Liquid nitrogen (LN2) characteristics:

- Clear liquid, resembling water in appearance, under pressure in vessel or in an open Dewar; material will rapidly turn to gas upon release or spill forming a vapor cloud
- Colorless, odorless, tasteless; cannot be detected by the normal human senses
- As a liquid, LN2 is inert, a chemically inactive and noncorrosive₃, nonflammable, nontoxic liquid (or gas)
- Boiling point: -196oC or -320 oF₃
- Rapid expansion occurs when changing state from a liquid to gas (1:696.5) which can displace oxygen.
- LN2 is denser than air as a gas when released. Condensed, a vapor cloud forms during LN2 release (common to all cryogens)₃ which remains in low areas rather than mixing evenly with air.

Oxygen deficiency hazard (ODH) is one of the most significant hazards with storage and use of LN2. Factors that could lead to ODH include₄:

- Equipment failure (container, lab equipment)
- Piping failure (transporting LN2 to equipment)
- Human error (leaving valves open)

- Safety equipment failure (oxygen monitors or ventilation alarms)
- Ventilation failure (power outage, breakdown of ventilation system)

Liquid nitrogen is stored and transported in double walled, sealed vacuum storage containers, which can be either pressurized or non-pressurized. <u>Dewar flasks</u> are non-pressurized open-topped vessels with loose fitting covers to minimize evaporation, or off-gassing, and range from one to 50 liters in size. These Dewars are commonly used for immediate use of LN2, for placing instruments into the vessels, or for pouring the LN2 into equipment or other containers.

The U.S. Department of Transportation (DOT) refers to the larger, pressurized Dewars as *Liquid Cylinders* (referred to as cylinders in this report). Liquid cylinders tend to be larger, containing 160, 180, and 230 liters of LN2, and are common at the UW. They are often provided with vents, dispensing hoses and pressure gauges. Cylinders in laboratory settings are typically transported and distributed by compressed gas manufacturers to the labs or storage sites near the labs. Pressure relief valves are spring loaded devices set at a specific pressure that relieve excessive pressure, reclose and reseal to prevent further release of product₅. Gas releases from the pressure relief valves or other fittings are continuous.

III. REGULATORY REQUIREMENTS AND BEST PRACTICES

Regulations and Codes (adopted):

<u>Employer Responsibilities: Safe Workplace</u> – Washington State Department of Labor and Industries₆:

<u>WAC 296-800-11005</u>: Provide a workplace free from recognized hazards. "You must provide your employees a workplace free from recognized hazards that are causing, or likely to cause, serious injury or death."

<u>WAC 296-800-11010</u>: Provide and use means to make your workplace safe. "You must provide and use safety devices, safeguards, and use work practices, methods, processes, and means that are reasonably adequate to make your workplace safe."

<u>WAC 296-800-11040</u>: Control and prevent chemical hazards and exposure. "Control chemical agents in a manner that they will not present a hazard to your workers; or protect workers from the hazard of contact with, or exposure to, chemical agents."

<u>Seattle Fire Code</u>: The storage and use of Cryogenic materials is regulated by the 2012 Seattle Fire Code, a locally amended version of the 2012 International Fire Code. This code does not require alarms in rooms or spaces where cryogens are stored or dispensed.



National Standards (not adopted):

<u>National Fire Protection Association:</u> NFPA maintains a national standard on Compressed and Cryogenic Fluids, NFPA 55, and Fire Protection for Laboratories using Chemicals, NFPA 45.

<u>NFPA 55, 6.8, Employee Alarm Systems</u>: When required by government regulations, an employee alarm system shall be provided to allow warning for necessary emergency action as called for in the emergency action plan required by 4.2.1.1, or for reaction time for safe egress of employees from the workplace or the immediate work area, or both.

<u>NFPA 45. 11.4.3. Cryogenic Fluids:</u> The space in which cryogenic systems are located shall be ventilated commensurate with the properties of the specific cryogenic fluid in use.

Industry Guidelines:

<u>Compressed Gas Association</u>: The CGA maintains a publication for Safe Handling of Cryogenic Liquids, "CGA P-12₃" and Safe Handling of Liquefied Nitrogen and Argon, AV-5 (video). The CGA Safe Handling of Cryogenic Liquids states, "If low oxygen atmospheres are possible, installation of analyzers equipped with alarms should be used to monitor the oxygen content. Whenever personnel enter enclosed areas, the breathing atmosphere shall be constantly monitored by appropriate instrumentation.₃"

IV. SUPPORTING DATA

Past Incidents

According to the U.S. Chemical Safety and Hazard Investigation Board (CSB), 85 nitrogen incidents occurred in the workplace resulting in 80 deaths and 50 injuries from 1992 to 2002. An estimated 11% of these incidences occurred in laboratories and miscellaneous industries, such as medical and transportation. Over 60 percent of the victims were working in or next to a confined space₇.

Of various LN2 incidents around universities and hospitals, a lack of ventilation allowing nitrogen concentrations to accumulate appears to be the primary cause of death or injury.

Date	Location	Incident	
1995	Sapporo Medical University	Two researchers died due to asphyxia from	
		oxygen deficiency in a cold experiment room.	
1998 University of Gottingen, A 24 year		A 24 year old lab worker died of asphyxiation	
	Germany	while filling Dewars in a lab that had no working	
		ventilation or open windows.	
1999	Western General Hospital,	One died and four were injured from an LN2 spill	
	Edinburgh, Scotland	in a basement storage room.	





2001	Australian Animal Health	One worker died of asphyxiation in an air lock		
	Laboratories, Geelong,	containing LN2 where there were multiple		
	Australia	deficiencies: back up fans were inoperable, failure		
		of the air pressure switch, no oxygen alarm		
		procedures and no audible low-oxygen alarm		
		system in the air lock.		
2008	Korean university	Postgraduate student died in an unventilated,		
		underground, open-topped dry area while filling a		
		Dewar with LN2.		
2011	Chelsea and Westminster	One lab worker died; suspected to be from liquid		
	Hospital, London, England	nitrogen.		

Other University Practices

The cryogenic/LN2 safety programs of the following universities were reviewed; some institutions have policies on this topic but there appears to be no consistent approach.

- Stanford University
- University of Florida
- University of Vermont
- University of Louisville
- Perdue University
- Grand Valley State University
- University of Kentucky
- Northeastern University
- Texas A&M University
- Bristol University
- University of Auckland

These universities incorporate various engineering and administrative controls to mitigate the hazard of LN2.

Engineering controls include: ventilation, storage, safety relief valves for equipment and piping, and oxygen monitors; generally as a recommendation.

Administrative controls include: written programs, web sites, training, and the "buddy system" while pouring or working with LN2.

Personal protective equipment (PPE) include: cryogenic gloves, face shields, smocks or lab coats (required), closed-toe shoes or boots.

Other safety factors include emergency response guidelines, first aid and proper disposal of LN2.

V. UNIVERSITY OF WASHINGTON-SPECIFIC INFORMATION LN2 IN LABORATORIES

LN2 in Laboratories

An estimated 96 laboratories, research and medical facilities use and store LN2 as identified in the UW MyChem chemical inventory tracking system. Quantities of LN2 varying from 2 to 50 liters are typically transported and used in open-top Dewars to 160 liters or more (for large-quantity storage). Laboratory quantity breakdown is as follows:

LN2 Quantities in labs on campus	Number of labs
Labs greater than 100 L	48
Labs from 100 to 35 L	23
Labs less than 35 L	25

Departments using LN2 include Engineering, including Aeronautics and Astronautics, Applied Physics, Molecular, Mechanical and Electrical Engineering; medical departments including Hematology, Pathology, Periodontics, Pulmonary and Critical Care, Genome Sciences, Immunology and Pediatrics; and sciences including Biochemistry, Environmental and Occupational Health Sciences, Microbiology, Oceanography and Biology.

In an *ad hoc* phone interview of various labs and research centers, most labs or facilities use LN2 for cryogenic processing of samples, including tissues or cells. Others use LN2 as a coolant for equipment, such as gas chromatographs or to create high vacuum environments, and as a source of pure nitrogen with no water or other volatile organic compounds.

UW Ventilation in Rooms and Labs

The UW has over 50 laboratory buildings used for scientific teaching and research. Many of the buildings have non-recirculating ventilation provided at a rate of 6 to12 air changes per hour. At the lower end of this range, labs on campus have complete air changes every 10 minutes. This equates to roughly one air change per square foot of floor space per minute.

VI. ANALYSIS

Oxygen Deficient Atmosphere Health Effects

There are two major risks associated with LN2; health hazards and physical hazards. Only the asphyxiation hazard will be discussed here, consistent with the purpose of this paper.

Normally air is comprised of nitrogen (78.08%), oxygen (20.95%), argon (0.93%) and carbon dioxide (0.13%), with the remainder being a mixture of gases₁. An oxygen deficient atmosphere is "an atmosphere containing less than 19.5% oxygen by volume"₈. Oxygen

deficient atmospheres could cause asphyxia in two ways: sudden and gradual₂. Humans vary considerably in their reaction to oxygen-deficient atmospheres and it is not possible to predict exactly how they will react₆. However, a general indication of what could happen is listed below:

- 1. Sudden and acute asphyxia would occur from the inhalation of little to no oxygen. Unconsciousness without warning would be immediate upon one breath of oxygen concentrations at levels below 10% to the point that the person could not evacuate or use an air-line respirator or SCBA₅, _{9, 10}. At levels below 16%, impaired perception and judgment, fatigue, and poor muscular coordination would impede self-rescue due to being wholly unaware that anything is wrong₂, ₁₁. Sudden asphyxia could occur with a large release of LN2 or if someone entered a room when the oxygen is depleted.
- 2. Gradual asphyxia can occur at any level below 20.9%, which is normal oxygen content. Symptoms of hypoxia (i.e., reduced oxygen to tissue), such as accelerated breathing and heart rate, increase at levels closer to 17%, although these symptoms may vary with individuals_{2, 5}. A feeling of euphoria can set in during hypoxia, making the victim unaware of the danger and thus making no attempt to self-rescue_{3, 9}. Gradual asphyxia could occur during an increased use of nitrogen, gaseous or liquid, as cylinders normally relieve pressure, or if valves or fittings freeze and stick open.

Cylinders and Dewars

Released gas or liquid nitrogen can result in a vapor fog cloud. Atmospheric water vapor condenses by the cooling effect of the liquid nitrogen being vaporized₂. Vapor clouds can travel considerable distances causing visibility problems₂, making escape difficult. All cryogenic liquids and gases will have negative buoyancy at their normal boiling temperature at 1 atm and 20 oC, thus the vapors will settle to the floor or ground₉. Fog and gases could collect in low-lying areas causing an asphyxiation hazard, especially on floors where there is limited draw from ventilation.

Cylinder and Dewar failures are highly unlikely to occur spontaneously. Cylinder failures can occur from blunt force accidents, such as forklift tines puncturing the double walls of the cylinder or someone striking the fittings at the head of the cylinder. Cylinders meet DOT drop test specifications and should be able to withstand tip overs and falling on their sides. However, if the head fittings are struck, the valves, fittings or gauges can break free and release LN2 in liquid or gaseous form causing a vapor cloud. These clouds should not pose an immediate threat to human health unless contacted by skin. It is unlikely that the whole liquid cylinder valve neck would be impacted to the point of failure.

Cylinder designs prevent overfilling by providing a ullage space of about 10% so they cannot be completely filled per DOT regulations 49 CFR 173.302(c)_{5,9,12}. They readily vent nitrogen gas due to continuous expansion from liquid to gas nitrogen. During transport, cylinders are chained to and transported by a four-wheeled hand truck or dolly. Dollies with full cylinders can weigh up to 700 pounds and require proper handling during manual transport. These dollies may require two people to handle and balance the weight as it may be necessary to transport dollies up ramps or up-grade walkways outside of buildings.

A pressure relief burst disc, or rupture disc, is a non-reclosing pressure operated device that prevents excessive pressure from building up in the cylinder. The metallic membrane ruptures under abnormal operation or during emergancies_{3,13}, causing the entire cylinder contents to vent to the surrounding atmosphere₅. Relieved pressure must be vented away or far enough away from personnel to prevent cryogenic liquid or gas contact. All lines, piping or associated connections to the cylinders, other storage containers of LN2 should be provided by the supplier or the transfill equipment vendors.

Pressure relief valves periodically release at set pressures to prevent over pressurization of the cylinder. At times, these valves freeze open, continually releasing nitrogen gas. A gentle tap or adjustment will allow the valves to reseat themselves. If a burst disc was to rupture, only gaseous nitrogen would be released to the atmosphere. This would occur only if the inner tank became pressurized beyond the pressure relief valve and burst disc capacities. In this event, something catastrophic is occurring and personnel will most likely be alerted to the failure by a combination of noise from escaping gas and the sight of a vapor cloud; all personnel should evacuate.

During LN2 use and operations, nitrogen release is nominal. As LN2 is poured into Dewars, pumped into equipment such as gas chromatographs, or drawn into instruments, nitrogen gas generation is unlikely to cause oxygen depletion in the laboratory. The largest potential cause for concern is if LN2 were pumped or drawn through non-cryogenic tubing or piping, allowing the LN2 to expand enough to burst the piping or vessels. If cylinders were connected in succession, this would greatly increase the chances of pressure buildup.

Two large LN2 tanks are located on campus in the Chemistry building and Fluke Hall. In these cases, gaseous nitrogen is piped to specific labs for use. Liquid cylinders are also filled from these tanks; however, this occurs outside in open ventilated spaces.

The release of LN2 can occur from several types of events; however, the most likely releases would be from the following: over pressurization of cylinders or piping, releases from fittings (normal operation or accidental), transportation accidents with cylinders or Dewars, or dispensing of LN2.

LN2 Calculations - Volume Expansion and Oxygen Depletion

The most common size pressure cylinders in which liquid nitrogen is stored are 160, 180 and 230 liter. If a complete release occurred of these sized cylinders and the cylinders emptied their full contents, the gas would expand to the volumes indicated in Table 1.

Table 1:Complete Displacement				
Cylinder Size (liters)	Volume (CF)	Lab Space (SF)		
		(assuming 10 foot		
		ceiling)		
160	3933	394		
180	4427	443		
230	5657	566		

Calculation: 160 L x 696.5 expansion to gas = 111,440 L; 111.44 m3 x 35.315ft3/m3 = 3935.5 ft3 Notes:

- CF = cubic feet

- SF = square feet

Release Scenarios

1) Catastrophic Release of Liquid Cylinder

There are several manufacturers of liquid cryogenic cylinders; however, only Chart Industries' cylinders were observed in the labs on the UW campus. The Chart technical advisor, Sam Cook, was contacted to discuss possible worst case and most likely scenarios for a liquid nitrogen release. He stated he had never seen or heard of a complete cryogenic cylinder failure in 30 years of the new DOT-type cylinders. Failures would most likely be due to human error or accidents like a forklift tine hitting the cylinder or another blunt force puncture of the double walled cylinder. The cylinders are designed to withstand the standard DOT drop tests of four feet, so tipping or falling on their sides will unlikely release contents. Fermilab calculated Equipment Failure Rate Estimates for Dewar (cylinder) leak or rupture at 1 x 10-6/hr₁₄, which equates to one failure in one million hours of use.

Over pressurization could release the inner contents. This would most likely be due to extreme heat such as a fire. In this case, personnel would be evacuated. Severe accidents and explosions can occur from placing LN2 into unapproved cylinders, Dewars or vessels. Closing off or circumventing pressure relief valves of liquid cylinder heads may allow the pressure to build up in the container to the point of catastrophic rupturing. This scenario was not calculated due to the extreme unlikelihood of this event occurring.

2) Open-Head Dewar Spill

The most likely immediate release of LN2 would be the dropping or spillage of an openheaded Dewar during filling or hand carrying in the lab. The largest observed Dewar was 50 liters (13.2 gallons). A complete release, when full, would expand to 34.825 m³ (1,229.8 ft³).



In a 5,000 ft³ lab, oxygen concentrations would decrease to 15.83% (See Appendix A for analysis calculations). In this case, it would be advised for occupants to leave the room long enough for the building ventilation system to clear the space, allowing the ventilation to return the room to normal oxygen levels. With basic room ventilation of 500cfm, a 5,000 sf space would be ventilated within 10 minutes. At a 16% oxygen level occupants could lose consciousness before the room is purged.

Most Dewars observed in the lab being hand transported were between one to ten liters. If a ten liter Dewar was spilled and immediately flashed into vapor, oxygen concentrations would decrease to 19.95%, which is within safe levels.

3) Rupturing a Port or Valve

One possible scenario of a release from a cylinder would be if someone attached inferior or non-cryogenic rated tubing to the cylinder and the tubing or piping ruptured. Gas releases from the gas valves would be a continuous liquid flow due to the cylinder trying to build pressure. This gas flow would be continuous; however, there would be no expansion to the nitrogen since it would be in its gaseous state. This gas volume release is estimated at 20 liters per minute₁₅. This rate of release would not be expected to result in a significant decrease in oxygen content under conditions of normal ventilation.

Two cryogenic programs calculated nitrogen flows from cylinders: Argonne National Laroratory₁₂ (ANL) and Fermi National Accelerator Laboratory (Fermilab)₁₆. ANL measured LN2 flow from a cylinder's liquid valve being open releasing about 0.3 L/sec at 23 psi, after vaporizing and coming to room temperature, nitrogen gas volume calculates to 0.20 m³/sec or 423.78 ft³/min (See Appendix A for analysis calculations). Fermilab estimated nitrogen gas flow to be 5 scfm at 22 psi from 200 ft of 3/8 inch outer diameter (OD) copper tubing. Fermilab estimates a fluid line leak or rupture 3 x 10-6/hr₁₄. Fermilab release rates are less than the ANL estimated release, so the ANL release rate will be used in this scenario.

Graphs 1 and 2 show oxygen concentrations decrease (percent) over time (seconds) for 160 and 230 liter cylinder volumes. Calculations include room ventilation as dilution air (500 cfm) and room ventilation with the assistance of a fume hood of 1,500 cfm (2,000 cfm total). See Appendix A for analysis calculations.

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Graph	1
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160 L cylinder would be empty after 533.33 seconds (8.89 minutes). Assumptions:

- 1) Complete mixing of air in room.
 - 2) Spill rate and ventilation rate remain constant.
 - 3) Initial oxygen concentration of 21%. .
 - 4) Room volume of 5,000 cf.





Note:

230 L cylinder would be empty after 766.67 seconds (12.78 Minutes). Assumptions:

- Complete mixing of air in room.
 Spill rate and ventilation rate remain constant. ٠
- 3) Initial oxygen concentration of 21%. •
- 4) Room volume of 5,000 cf. •



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Graph 3 shows recovery times after 160 and 230 L cylinders have emptied into a 5,000 sf room with 500 and 2,000 cfm ventilation rates after oxygen depletion scenarios in Graphs 1 and 2. Note that the 160 and 230 L cylinder at 2,000 cfm events have overlapping lines.





L – liters
cfm – cubic feet per minute

Table 1 calculates the total time of oxygen depletion during the cylinder release and the recovery of oxygen concentrations of the room through ventilation for both 160 and 230 L cylinders. The second part of the table calculates the time when the room is below 18% oxygen, the point when it would be considered potentially fatal.

Table 1

Cylinder Size (L)		Cylinder Depletion Time (min)1	Room Recovery Time (min) ₂	Total Time of Event (min) ₃
160				
	Ventilation 500 cfm	8.89	10	18.89
	Ventilation 2,000 cfm	8.89	2.5	11.39 ₄
230				
	Ventilation 500 cfm	12.78	10	22.78
	Ventilation 2,000 cfm	12.78	2.5	15.28₅

TOTAL TIME OF LN2 RELEASE EVENT

Cylinder Size (L)		Cylinder Depletion Time (min)1	Room Recovery Time (min) ₂	Total Time of Event (min) ₃
160				
	Ventilation 500 cfm	6.88	8.57	15.45
	Ventilation 2,000 cfm	6.88	2.14	9.024
230				
	Ventilation 500 cfm	10.77	8.57	19.34
	Ventilation 2,000 cfm	10.77	2.14	12.91₅

TOTAL TIME OF EVENT BELOW 18% OXYGEN

Notes:

- L = liters
 cfm = cubic feet per minute
- min = minutes
- 1) Cylinder Depletion Time = time of cylinder to evacuate LN2 contents (Graph 1 and 2)
- 2) Room Recovery Time = time for room to return to normal oxygen levels (Graph 3)
- 3) Total Time of Event = Depletion + Recovery times
- 4) Oxygen concentrations at 17.37%.
- 5) Oxygen concentrations at 17.33%.

Fermilab considers ODH atmospheres above 18% oxygen concentration to be safe levels and concentrations below 18% would be considered fatal. Fermilab calculates a fatality rate and incorporates a fatality factor, which is not conducted in this report₁₆. If 18% oxygen concentration levels are implemented and calculated (as in Table 1), the total time of fatal ODH atmospheres would be 9 to 20 minutes in duration. Although these times represent ODH levels below 18%, a series of events would need to occur to put someone into such a hazardous situation:

- The rupture or breakage of the LN2 valve, where the force would have to navigate around the protective ring to access the critical fittings
- Complete release of the cylinder contents where cylinder would have constant pressure
- Someone present or entering when the atmosphere is less than 18% oxygen
- Immediate vaporization of LN2 to gas and complete air mixing in the room;

4) LN2 Releases During Maintenance or Power Outages

Risks of possible LN2 releases increase during power outages and ventilation shutdowns. Building ventilation systems are periodically shut down for cleaning and maintenance. Power outages occur at least once each year. During these times, LN2 could be released from the equipment to the labs potentially causing oxygen deficient atmospheres.

However, during scheduled ventilation maintenance, labs are contacted to stop use of hazardous chemicals or equipment that may release toxic vapors to the local atmosphere. Science buildings are generally unoccupied during power outages. If cylinders do release



due to pressure, the cylinders are designed to maintain cryogenic temperatures and would only release approximately 2% of their contents per day₁₅.

VII. OXYGEN SENSOR TECHNOLOGIES AND OTHER ALARMS

The requirements of oxygen sensors as a means of identifying oxygen depletion environments depend on the organization or facility. Sensors in universities and facilities are located in low ventilation areas, high volume LN2 storage or areas where LN2 is poured into Dewars. Two main types of oxygen sensors are available: electrochemical or zirconium oxide sensors. Typically, the electrochemical sensors are in hand-held instruments with limited sensor life span (on average up to two years with some to three years). The zirconium oxide sensors are installed in plug-in style instruments or hard-wired into electrical systems (some with battery backup) and/or alarm systems with a life span up to 10 years, depending on manufacturer. Oxygen sensors require training, and at least annual calibration and maintenance for batteries, alarms and verification of operation.

No cryogenic safety plan reviewed as part of this evaluation included alarm systems for ventilation systems. One facility employs backup fans in the event of the main ventilation system failing or shutting down. In the case of ventilation shut down, safety awareness was communicated in the written programs.

Several labs at the UW have installed oxygen monitors, following the advice from compressed gas distributors. Brands purchased have not been consistent. All equipment is considered to be departmental responsibility; none are connected to the building alarm system, and few, if any, are remotely monitored. Labs have stated that their alarms have sounded during LN2 use due to valves or fittings sticking open, which were easily closed. Once the leak was corrected, alarms stopped and oxygen levels returned to normal in a short amount of time.

VIII. CONCLUSION

Safety is a high priority for the University of Washington in the labs; for the students and faculty, and around the campus. The UW stores, transports and utilizes quantities of LN2 in nearly 100 laboratories and facilities. Liquid nitrogen can pose a significant risk when released, leading to oxygen deficient hazards if not properly managed and controlled. The following general conclusions reflect the outcome of this evaluation.

- 1. Training on the hazards of LN2 is important; general training should be provided to all personnel working with and transporting LN2. Lab specific training should also be provided.
- 2. Regulations don't require alarms in rooms where liquid nitrogen is stored

and used, but an alarm may add value and improve safety in some circumstances. Standards and best practices indicate alarms may be necessary but there is no clear guidance on the topic for a laboratory setting.

- 3. LN2 should be used and stored in well ventilated areas. If large quantities of gases are present, risk can be mitigated through ventilation.
- 4. Oxygen alarms are not necessary in laboratories if the lab has sufficient continuous mechanical ventilation. Off gassing from cylinders and during general, dispensing and even of a small spill can be effectively managed by building ventilation.
- 5. A hazard assessment should be conducted for unventilated or under ventilated locations and other locations such as environmental rooms where there may be a possibility of an ODH to determine if oxygen alarms or other mitigation is needed.
- 6. A hazard assessment should be conducted for all bulk storage areas to determine the need for oxygen alarms or other mitigation. Several factors should be considered, including room size, ventilation rate, the frequency of transporting materials, nature and condition of the space and equipment, concentration of storage, and other factors that may influence the likelihood of a spill and the potential for a spill to make the space untenable for a period of time. These spaces can be rooms with multiple liquid cylinders, multiple cylinders connected through manifolds, or multiple cylinders equal to or greater than 230 L in size.
- 7. If alarms are installed, it is essential that they be calibrated, tested and maintained per the manufacturer's recommendation. This type of alarm system is considered departmental equipment so organizational units cannot rely on central service units to perform this service.

Further information

The American Welding Society, American Gas Association, American Petroleum Institute, and National Fire Protection Association have detailed literature on purging, testing, and working on potentially hazardous jobs₃.

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X. APPENDIX: ANALYSIS CALCULATIONS

Open-Head Dewar Spill, 50 liter

The following equations calculate expansion of LN2 from liquid to gas and the displacement of oxygen in an average sized laboratory of 5,000 cubic feet.

Calculation of expansion of 50 liters of LN2 to gas:

 $50 L x 696.5 = 34,825L \text{ or } 34.835m^3$

 $34.825 m3 x 35.315 m^3/ft^3 = 1,229.84 ft^3$ of nitrogen gas

 $1,229.84 ft^3 / 5,000 ft^3 (lab size) = 0.246$

 $1 - 0.246 ft^3 = 0.754$

0.754 (percent nitrogen difference) * 0.209 (oxygen concentration) = .15834

= **15**. **834**% *Oxygen concentration*

Release Rate

The equation used to calculate oxygen concentration with ventilation is from the Fermilab report₁₃:

$$\frac{\mathsf{V}}{\frac{dt}{dt}} = 0.21Q - (R+Q)C$$

To solve for the oxygen concentration, the equation can be rewritten as

$$(t) = (0.21) + [Q + Re - (Q+V)t]$$

Q+R

Definitions:

C = oxygen concentration (0.XX = XX%) Q = ventilation rate of fan(s) (m^3 /sec) R = spill rate into confined volume (m^3 /sec)



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t = time (minutes), beginning of release as t = 0 V = confined volume, (m³)

The variables are: room of 5,000 sf (141.5 m³), ventilation rate of 500 cfm (0.236 m³/sec), release rate of LN2 is 0.3 L/sec (gaseous nitrogen is 0.2 m³/sec). For a 160 L (0.16 m³) cylinder leaking at 0.3 L/sec, the cylinder would empty in:

The equation would look like:

$$Ct = (\underbrace{0.21}_{0.236 + 0.2}) [0.236 + 0.2e^{-(\underbrace{0.2+0.2}_{141.5})533.33]}$$

Ct = 0.1323 *or* 13.23% *oxygen*

To calculate for 18% oxygen concentration, place 0.18 into Ct and solve for t.

$$0.18 = (0.21) [0.236 + 0.2e^{-\frac{0.2+0.2}{(141.5)}t]}$$
$$0.236 + 0.2$$

$$= 2.02 min$$

The oxygen concentration for a 160 L cylinder with a release rate of 0.3 L/sec into a 5,000 cf room with 500 cfm ventilation rate will reach 18% oxygen in 2 minutes as seen in Graph 1.

For other calculations, a 230 L cylinder will take 766.67 sec (t) to empty. With a fume hood of 1,500 cfm, room ventilation rates (Q) increase to 2,000 cfm (0.9433 m³/sec). Release rate (R) remains the same.

Oxygen recovery rates

A basic linear equation is used to calculate the room oxygen recovery rate:

 $\frac{room \ volume \ (cf)}{ventilation \ rate \ (cfm)} = time \ (minutes)$ $\frac{5.000 \ cf}{500 \ cfm} = 10 \ min$

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The room should return to normal oxygen levels in ten minutes. With increased ventilation from a fume hood (1,500 cfm or 2,000 cfm total), the room will return to normal oxygen levels in 2.5 minutes.

Calculating 18% oxygen concentrations

To calculate the time it takes for the room to return to safe levels of 18% oxygen, the following equations are used:

 $\frac{18 \%}{-1}$ = 0.857% difference

0.857 *x* 10 min = 8.57*min*

A 5,000 cf room will take 8.57 minutes to return to 18% oxygen, a concentration deemed safe.

Reference sizes a few labs with LN2 on UW campus:

- Johnson Hall, Rm 302A 30' x 27' x 8.5' ceiling = 6,885 ft³
- Magnuson Health Sciences Bldg F456 23' x 29' x 8.5' ceiling = 5,669.3 ft^3
- Chemistry Library, Rm 121 19' x 30' x 8.5' ceiling = 4,845 ft³
- Ocean Sciences, Rm 400AK (storage closet) 6' x 3' x 9' ceiling = 162 ft^3 Opens into a long hallway and a large 3-story open space.
- Rosen Bldg. Rm B45 9' x 12' x 10' ceiling = 1,080 ft³
- Aeroengineering lab $18' \times 25.7' \times 12.5'$ ceiling = 5,782.5 ft³