

LN₂ Handling—Fact and Fiction

Bob Compton, Microscopy Today

"I will be getting my first SEM/EDS shortly, and to obtain the required operating permit from my safety group, I need a written procedure for handling LN₂. Specifically I need a written procedure for filling the 3 L dewar on the EDS detector from a 50 L dewar mounted on a cart. I plan to make the transfer by using a lab source of LN₂ to pressurize the 50 L dewar and have obtained all the valves and fittings required to do this from another SEM lab. I would appreciate copies of the procedure."

This question recently posed to the Microscopy List Server unleashed a flurry of comments, not only on the safety of various LN₂ procedures but on the safety of other materials as well. Also discussed was the necessity of formal LN₂ handling procedures.

Handling Concerns

The primary cause for many of the comments made in the LN₂ handling discussion is that LN₂ acts differently from most materials when it touches the skin. That is because when LN₂ touches human skin, a cushion of N₂ gas (Leidenfrost) is formed that temporarily prevents damage. This is why a typical demonstration of LN₂ is to stick ones finger into LN₂ for a few seconds and show that no harm occurs.

One major concern of LN₂ handling is the transfer of LN₂ from the storage dewar to the detector dewar. One of the most commonly used dewars is made from glass. However, as

Diane Ciaburri of General Dynamics noted, "I'm interested in why one would use a glass dewar for transporting LN₂ (cost?). Our transportation dewar is metal. I don't know how many walls but I assume there is a vacuum between them. It has been in use for over 20 years and had its share of bumps. I'm sure if it was glass, it would have been replaced a few times. It works great, has lasted 'forever' and doesn't have the safety issues of glass. Is glass that much cheaper?"

As Darrel Miles comments, "I believe the problems with glass dewars arise when scratches occur from filling and handling the glass dewars. These can be so fine to be difficult to see (when cutting glass, a fine, chip free scribe does a better job than a heavy one). When the conditions are just right (stress from cooling down, or tipping the dewar up), the vacuum bottle will implode. This is why we have some old metal outside/plastic inside dewars for transferring LN₂ from the storage/supply dewar, to the system dewar. They are not used to store LN₂, but are good and rugged for all of the handling they get."

Many do not employ dewars for filling EDX detectors. For example, Terry Ellis reports that "I fill up our EDX directly from the 160 L liquid nitrogen tank. I use a special liquid nitrogen flexible metal hose with a sintered brass end that lets the gas out the sides and liquid out the end. The 160 L tank sits on a special movable dolly, so I unchain it and move it next to the EDX or whatever I need to fill up. It saves spillage and no dewar is needed for EDX fillups." Also as reported by Michael Shaffer of the University of Oregon, "The 160 L dewar tank I fill from has a "nalgene" hose and I've never had a problem with it. However, one plastic hose (not nalgene) did explode on me while filling, which underlines the use of wearing protective eyewear while working with LN₂!"

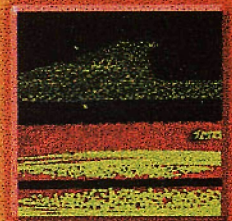
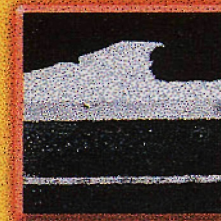
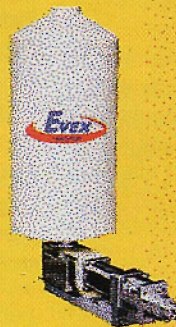
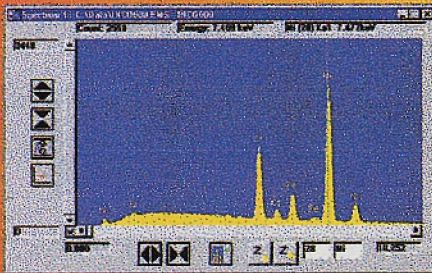
609-252-9192

Evex Analytical

www.evex.com

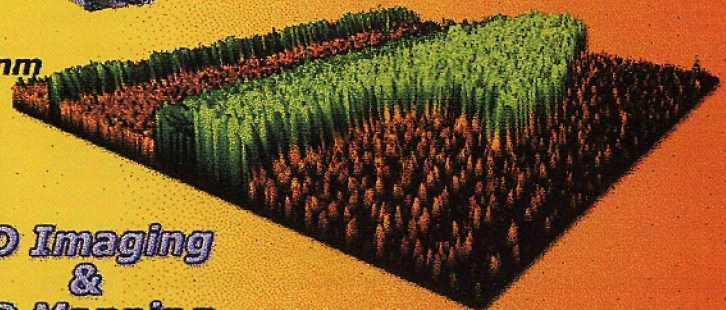
X-ray Microanalysis
&
Digital Imaging

X-ray Microanalysis



5 nm

3D Imaging
&
3D Mapping



0 nm



Allen R. Sampson offered one caution when using a hose feed system. He cautioned that "When using hoses, you may turn the pressure up too far at first, since there is only vapor passing through. When the fluid starts flowing, the pressure may be high enough to cause excessive 'splashing'. Another real problem with the use of cryogenic hoses is that the exterior metals can also achieve cryogenic temperatures. While they will generally get coated with ice, the underlying metal can burn if touched. When the contents of a dewar are first being drawn, the warm hoses will cause the fluid to evaporate. For the first minute or two, all that will be going into the EDS dewar will be cold vapor that can cause an accumulation of ice and affect performance.

Another options for filling EDX detectors is a glass thermos. As reported by Chuck Butterick of Engineered Carbon, "Besides being glass, a thermos poses several different kinds of hazards after breaking. Also, a 5 L dewar is very expensive. That is why just over a year ago I started using a 2 gallon plastic drink dispenser from WalMart that costs less than \$10. It lasted almost a year before the seal around the spigot failed. Of course the dispenser can't store LN₂ for any length of time, but for transport between tank and EDX detector, it does great. By the way, the top vent was permanently removed so no pressure buildup could occur."

The danger is more than just LN₂ burns

Most problems associated with LN₂ are caused by things other than the LN₂ itself, For example Ken Moran of Moran Scientific remembers, "When I first started using LN₂ over 25 years ago, I was filling a glass vacuum flask when it exploded and filled the room. I was quickly inundated with instant fog, tinsel and plenty of

LN₂ as a fine spray. I was unharmed apart from fine glass shards and aluminum foil in my hair. I was wearing no protective gear. The LN₂ was in my hair as a fine spray, thank goodness. I was amazed that I had no effect from the LN₂. After that I started to wear thermal type gloves but soon found that these were more dangerous than no gloves at all. There are times when one does have to be careful in handling LN₂ and these in my experience have been with 1) glass containers, 2) pressurized containers and 3) accidental contact from surfaces at very cold temperatures.

Should LN₂ be a Controlled Substances?

Arguably burning water is more dangerous than LN₂ because it has no insulating layer. So boiling water poured onto skin will burn, whereas LN₂ would only do so in extreme cases. Pressurized LN₂ must be compared with pressurized boiling water, as in a pressure cooker or autoclave. Boiling water is undeniably dangerous and many, many burns occur every day. Of course it's a very commonly used substance, but that means that all, except small children would know of the danger and should use care. Why then is boiling water not a tightly controlled substance and why is no operator's license required to make a cup of coffee? Ask your safety officer. Our discussion concerns LN₂ use in laboratories by people with more training and understanding of physical properties than that of the general public. Why should these people be required to submit a procedural outline for simple tasks, such as transferring some LN₂.

Also noted by Moran, "Water is also dangerous - to wit drownings. Danger is a comparative matter. For example, many

Continued on following page

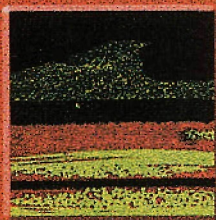
609-252-9192

Evex Analytical

www.evex.com

*X-ray Microanalysis
&
Digital Imaging*

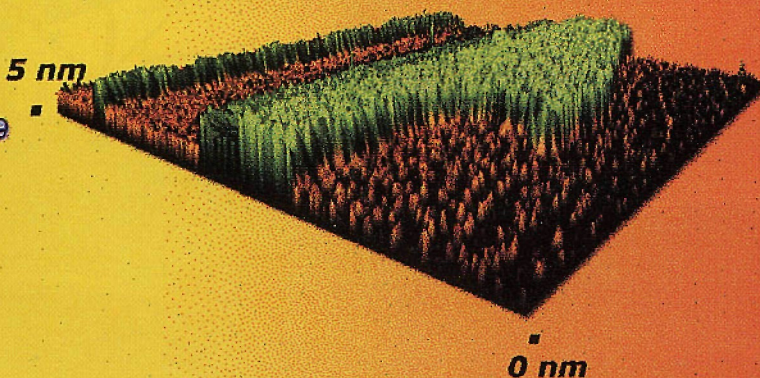
**People once
thought maps
were flat.**



**Evexium,
Proved them wrong !**

**3D Imaging
&
3D Mapping**

**Discover Evexium,
the most advanced technology for
electron microscopy. Evexium is the
next generation of products from
Evex Analytical.**



**View images from our prespective.
Visit us on the web
www.evexium.com**

LN₂ Handling—Fact and Fiction

Continued from preceding page

common substances are dangerous when mishandled. Petrol/gas in my opinion is rather more dangerous than liquid nitrogen. Gas is much more common than LN₂, but few gas accidents are reported. LN₂ accidents become folklore, not because of the greater danger of LN₂ but because those accidents demonstrate ingenious, applied stupidity. Although gas and its dangers are well known, people inhale it (brain damage and death), start fires with it, store it in crummy plastic containers, siphon it by mouth, use it to rekindle a fire, etc. I must agree though, gas is not a good comparison to LN₂."

Also Arthur Day of Ansto notes: "I would also like to counter the thought that gasoline is more dangerous than liquid nitrogen. It's different - I carry a 4.5 litre sealed container of gasoline/petrol around in my car quite safely but I wouldn't seal LN₂ like that. Open or poorly sealed storage can lead to icing or condensation of oxygen from air (now there's a good one to debate - has anyone heard of that causing an accident?) with possible disastrous consequences. Surfaces in prolonged contact with gasoline present less danger than with LN₂; and gasoline has a strong odor so I wouldn't expect to be asphyxiated by it so easily. (OK, it might ignite more easily and become harmful). The point being that even though LN₂ use might seem less of a risk than boiling water when used "properly" and "common sense" is applied, it cannot be assumed without question that everyone's initial mental picture behind these two terms is exactly the same!

How is safety enhanced by a single task written procedure

Jim Darley of ProSciTech notes that most LN₂ accidents are caused by lack of knowledge of the material's physical properties, or temporary "insanity"? A six-minute briefing of LN₂ properties and don'ts would benefit the neophyte and safety more than a cabinet full of paper. The required written procedure on decanting LN₂ is a fig leaf for the safety officer. Unfortunately, safety officers have become part of a bureaucratic system and frequently are required to perform nonsensical tasks.

As Layer Stofer stated, "As many people around the industry are aware, gloves, goggles, masks (and shoes) are actually more dangerous when handling LN₂ than sandals and no protection because they tend to eliminate the insulating layer of N₂. And clothes are actually more dangerous than being naked. Get the safety officer to experiment. With a little persuasion you can probably convince the safety officer that when handling LN₂, everybody should be naked."

As Darly suggests, try getting your safety officer to conduct an experiment:

1. Hold out hand.
2. Pour a small volume of LN₂ over hand.
3. Now the interesting bit, put on a glove, and pour the same quantity of LN₂ into glove.
4. Phone for ambulance.

The point is that a brief contact causes no problems, but if the contact is continued you get a nasty burn.

But what about the safety officers point of view?

Dr Eric Lachowski, University of Aberdeen, can report from both sides of the safety officer fence. Dr. Lachowski reports, "I am



Premium EDS/Digital Imaging Systems

4pi Analysis provides EDS and digital imaging systems, components, and software for both the Mac® OS and Windows® 98 or NT. Whether you need a complete system or want to upgrade outmoded electronics, 4pi's products meet the high standards you demand.



Features

- light element x-ray detection
- detector resolution of 133eV or better
- peak to background ratio of 20,000:1
- slow and fast x-ray mapping
- x-ray and SEM line scans
- x-ray image mapping
- qualitative and quantitative analysis
- real-time data acquisition
- micron markers on acquired images

4pi Analysis, Inc.
 3500 Westgate Dr. #403
 Durham, NC 27707
 (919) 489-1757
 www.4pi.com

a microscopist who also has the misfortune to be the Departmental Safety Adviser and I have a few comments to make from the point of view of the enemy. While the jokey remarks about the advisability of wearing clothing, protective or otherwise, do have an element of truth, liquid nitrogen has the potential to do a great deal of harm. Recently there was a prosecution in Edinburgh where the Medical Research Council was found guilty of breaches of a Health and Safety law after a technician died of asphyxiation while dispensing LN₂ in a room with inadequate ventilation. Also three of his colleagues came close to suffering the same fate while trying to rescue him. The room was fitted with a low oxygen alarm, that had been switched off because it went off too frequently and annoyed him. This is an example of familiarity leading to dangerous practices. (His bosses got hammered because they knew about the alarm being switched off)."

"Therefore when you write a procedure you need to think of what could go wrong, as well as the routine safe handling of the LN₂, which is basically covered by - would you believe it - common sense. What would happen if the dewar shattered? A full shield should stop LN₂ from getting up your nose or in your mouth and is better than goggles which might trap liquid inside them. Drain holes in the bottom of the outer case of the dewar will reduce the risk of your hand freezing to it. What would happen if the main tank ruptured or fell over? I know that this is very unlikely, but a simple calculation based on the volume of the room and the capacity of the tank will tell you if oxygen depletion to a dangerous level is a significant possibility - and it will look good on your paperwork.

Also remember that LN₂ burns are particularly nasty because you may not be aware of them until the frozen bit thaws out again,

by which time it may be too late to prevent serious damage. Therefore you might want to consider loose fitting gloves if you are to handle cold parts of the apparatus. These may be proper cryo-gloves, but many claim that they offer limited protection and can in certain circumstances be worse than not wearing gloves at all." In fact Ken Compton of NORAN reports that they use welder gloves that can be flicked off immediately if a spill occurs on or in the gloves.

An LN₂ Horror story as told by Earl Weltmer

Also takes the point of view of the safety officer's conservative position. Weltmer says: "I am all for getting bureaucrats out of our hair but not at the expense of safety. I agree the extra clothing, goggles and what have you are cumbersome and most of the time unnecessary but I am only reminded of an incident that happened to me about 25 years ago. I was routinely filling a portable LN₂ tank from a larger source. The portable tank had a pressure gauge that would measure tank pressure: when full the gauge read 20 psi; and when empty it would read 0 psi. The assembly was attached to the tank via a rubber vacuum hose clamped at each end. The normal procedure was to release the "vent" valve, unclamp the rubber hose, remove the valve assembly, then refill the portable tank from the larger LN₂ source. I am sure most of you have seen a similar assembly. Early one morning (before my coffee), I went through the normal procedure. After unclamping the rubber hose, I proceeded to remove the assembly from the tank using both hands to pry it loose. Unfortunately there was a little pressure left and as soon as the hose was released, a stream

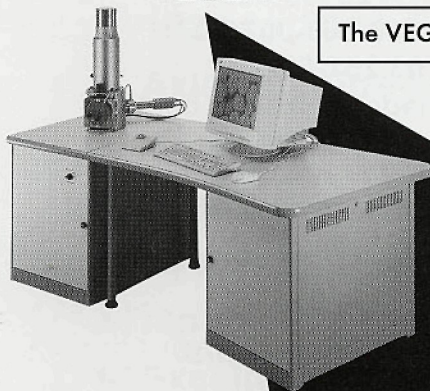
Continued on following page

CamScan USA

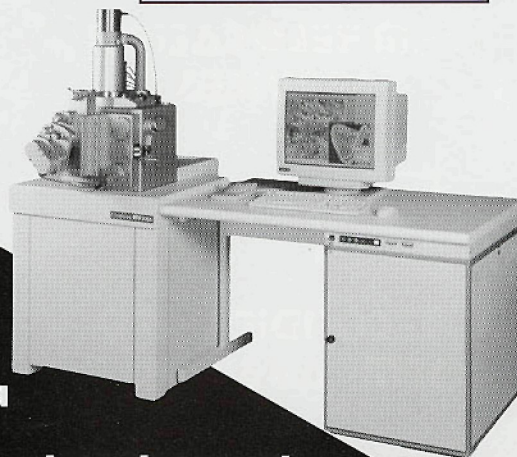
Scanning Electron Microscopes

508 Thomson Park Drive
Cranberry Twp, PA 16066-6425
Tel: 724.772.7433 Fax: 724.772.7434
email: info@camscan-usa.com
visit our website: www.camscan-usa.com

The VEGA Series - Standard & Midsize Chambers



MV2300 - Large Chamber



- 3.5 nm resolution
- 500V to 30 KV operation
- 6 axis eucentric stage
- fully computer controlled
- turbo pumped
- unique 4-lens column, four imaging modes:
#1 resolution, #2 depth, #3 field, #4 fish eye

LN₂ Handling—Fact and Fiction

Continued from preceding page

of LN₂ burst from the tank. The noise startled me and in less than a half second I removed my hands from the tank. In less than one second, I realized that I was burned and immediately immersed both hands into water from a nearby sink. Too late. In less than one-half second, I received second and third degree LN₂ burns on the bottom of my hands. The blisters extended from the bottom of both hands to halfway up my small finger. Worse yet, both hands were bleeding. Apparently, both hands immediately froze and I had cracked the skin by moving them to the sink. I wish I had gloves at that time. I worried for the next two weeks about getting gangrene. Still today, I don't use gloves as they are cumbersome but I would hate to think what would have happened if I were sprayed in the eyes from LN₂ even for a split second."

Another LN₂ Escapade

Paul Grover also reports, "I'd like to share my experience with getting naked in a hurry. I was working around midnight in the biochem lab at Oklahoma State University in '79 and was freezing samples to put in the lyophilizer. My arms were full, carrying numerous flasks plus a dewar of LN₂ which I held against my shoulder. As I turned a corner, I slipped on some spilled water. I was wearing those flip flop sandals and the entire contents of the dewar poured down my back. Fortunately, there were no witnesses to my rapid striptease. I suffered only a minor burn on my upper back. Definitely, nudity is the way to go if you plan on spilling LN₂ on yourself."

Another horror story

Marten Harris recalls: My associates and I were having a coffee break in a portacabin room used as a makeshift lab when a delivery lorry, which was about to top off our cryogenic LN₂ tank, reversed into and broke the connecting refill valve. We were observing the resulting stream of LN₂ spill over the tarmac when one of the guys remembered that his new car was parked in the enclosed area and his new tyres may be damaged. Without a word he pushed through the fire door, ran the few yards through the increasingly heavy fog and managed to reverse his car in the few feet available to be out of the way of the liquid nitrogen. But when he went to return he found the fog too heavy to see the few yards to the portacabin, and quickly became disorientated. As panic set in he fell to his knees and, becoming increasingly short of breath, started crawling - finally to the fire door which by now was firmly closed, as we had all run for safety. Fearing asphyxiation he managed to crawl around the building through the fog and emerged gasping a minute or so later. The contrast to one minute, quietly having a coffee and the next to witness this life and death (?) struggle and accusations of attempted murder as someone had closed the fire door caused much merriment at the time. All except for the poor 'victim'.

Summary

Thus there are many sides to the question of LN₂ safety. As indicated in these real life anecdotes and opinions, there are many concerns that need to be resolved before you can consider your LN₂ handling procedures safe. By doing a good job of planning and education you can make sure that your lab doesn't become the source of one of these "horror stories". ■

**SEM SERVICE
CONTRACTS**

**SEM RELOCATION
SERVICES**

**RECONDITIONED
SEMs AVAILABLE**

IMAGE CONTROL INC.

"Your Image Is Our Concern"

P.O. Box 838
Bristow, VA 20136-0838
Phone: 703 369-0021
Fax: 703 257-6321

Web: www.imagecontrolinc.com

TOPCON/ISI HITACHI JEOL

We Have Hard-To-Find TOPCON/ISI Parts

Service Contracts available at reasonable cost:

**Unlimited Calls
Fixed Number of Calls
Hourly Rates**

- **Servicing Electron Microscopes since 1966**
- **Engineers located strategically thru the U.S.**
- **Prompt and economical service**
- **Move units room to room or state to state**
- **Quality prime used equipment for sale**