CARNEGIE SCIENCE | Geophysical Laboratory

(Room 137)
(Gas Loading Laboratory)

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Purpose:

The gas loading facility is designed to load gases such as H_2, He, Ne, Ar, CH_4 and N_2 at high densities into the diamond anvil cell. It also has a glove box for loading air-sensitive samples.
All laboratories and facilities on the Broad Branch Rd. campus are controlled areas. Specific training must be completed and documented before working in this laboratory / facility.

Known hazards and safety policies:

The major source of hazards in the gas loading room is the gas loading facility. However, the room contains other units for sample loading, including a conventional glove box and a plastic glove box for cryogenic loading of aggressive and reactive materials in liquid form at low temperatures. The cryogenic glove box is a source of specific hazards and should be used only by its current qualified user (Maddury Somayazulu)*. The conventional glove box does not pose significant known hazards, however, the potential user should get specific instructions from the responsible staff member before using the glove box to keep the instrument in proper condition after use.

We are trying to keep at minimum the hazardous or reactive chemicals in the gas loading room. However, some specific samples require storage for a prolonged period of time in a glove box to be available for day-to-day work. The placement of such sensitive samples into the glove box should be approved by the responsible staff member. Any samples placed in the glove box without prior permission may be removed and discarded at any time without prior notice.

The gas loading facility is designed to load gases such as H_2, He, Ne, Ar, CH_4 and N_2 at high densities into the diamond anvil cell. This corresponds to about 25000 psi (about 1.5 kbars). The system consists of the following subunits;
(a) Two stage compressor for compressing gases from 1000 psi to 25000 psi.
(b) The pressure vessel housing the diamond anvil cell.
(c) The inlet and exhaust valves for introducing the gas to the compressor and venting the gas after loading.
(d) The pressure-sealed stem for engaging the gear and increasing the pressure in the diamond cell.
(e) The large threaded nut that seals the entrance to the pressure vessel.
Each of these parts listed above has certain safety issues related to it that need to be checked and followed before any loading is attempted. The biggest safety concern about the gas loading facility is the stored energy that is due to the compressed gas in the pressure vessel. Typically, when raising the pressure of about 1.5 moles of a gas from 1 bar to 2 kbars, an energy of the order of roughly 20 kJ is stored in compressed gas. Compare this with the muzzle energy of a bullet, which is about 3 kJ, or a stick of dynamite, which is about 2500 kJ. At the same time, this value is about 10 times less than the stored energy in a 300 liter gas cylinder containing H_2 at 2000 psi. The safety of the system is thus enhanced when minimizing stored energy and ensuring that there are no leaks, which could act as violent vents for the stored gas. Thus, you should minimize the stored energy by

keeping free volume minimal inside of the pressure vessel. Leaks must always be checked and notified so that they can be resolved before any loading is attempted.

We provide below a checklist, which should be followed for any gas loading procedure. Please note that these steps were designed for minimizing the risks associated with the issues listed above. First of all, we recommend all those who are using the gas loading lab to wear protective goggles, gloves and shoes.

(1) Is the gas cylinder properly tied down to the stand? Read the part above about how much stored energy the cylinder has. Please get familiar with gas cylinder safety requirements on GL safety *webpage/general document/safety seminar record*?

(2) Are you using the proper regulator for the type of gas being loaded? Please don't try to make do by mix-and-match. We do have the correct regulators for the gases we load. You cannot load oxygen using this compressor and if you are using gas mixtures that you have made on your own, it must be certified before you can use it to load.

(3) Is the copper piping used to connect the cylinder to the compressor looking good? Most often we find that the pipe is twisted, bent, crinkled and would end up leaking at these points. If the Swagelok connection on the pipe looks bad, please replace it or seek help in replacing it.

(4) Is the gas pressure in the cylinder above 600 psi? By using input pressure below 600 psi, you are overloading the compressor and also not going to attain high enough loading density. If you are replacing the cylinder, please use the cart to transport the cylinder. Label the cylinder empty, place it in the empty cylinder area downstairs near the loading dock and only then bring the full cylinder up to the loading lab. Please make a note in the log file that you have brought the new cylinder up. It is for this purpose that we strongly urge you to write in the log file the pressure in the cylinder before and after any loading. It not only helps us track the usage of the cylinder but also helps us notice if there are any abnormal leaks that need to be checked.

(5) Is the gearbox appropriate for the cell you are using? We have two kinds of gear boxes and they correspond to two different kinds of cells.

(6) Is the gearbox good? Are all bearing working well? If the bearings are broken, most probably there are some miniature balls and steel pieces floating about in the gear box. This will not only result in not allowing you to close the cell but will also destroy the gearbox, possibly beyond repairs.

(7) Have you calibrated your cell to know how many turns are required to close the cell? Remember, the gear box is not meant for high loads. One of the common problems we face is that people who do not calibrate their cells end up loading over and over and sometimes overload the gearbox destroying it, and even worse, breaking the delicate stem. This is not only going to result in down time but could be dangerous since the stem is like a projectile with more than 5 kJ of energy (a normal bullet would carry) sitting behind it!

(8) Have you checked with the dummy how much stacking you need to use to minimize free volume?

(9) Have you checked with the dummy the alignment of the hex key inside the pressure vessel?

(10) Have you used the vacuum cleaner to make sure that the inside of the pressure vessel and more importantly, the threading for the big nut is free from metal grit and dust? Presence of such dust and grit results in the wearing down of the threading on the big nut, and most often jams the nut while it is being threaded shut.

(11) Have you aligned the cell on the threaded holder such that the pins on the gear box would engage and the hex key also engages properly? If care has been taken, you will find that the cell assembly with the gear box engages correctly with a distinctive pair of clicks and you cannot turn the assembly clockwise. This is a very important step and most failure to load can be due to this step not being executed properly.

(12) Have you wiped the big nut with the special tissue paper to ensure that it is clean and would engage well? Have you checked the steel ring and the rubber O ring and ensured that they are not damaged?
(13) Have you engaged the big nut all the way so that it is flush with the outer rim of the pressure vessel?
(14) Have you closed the vent valve before you start pumping?
(15) Are the steel guard walls in place?
(16) Have you turned ON the vent fan? Have you closed the entrance door before starting the pump?
(17) Have you checked at 5000 and then at 10000 psi if there are any leaks?
(18) Have you closed the regulator on the gas cylinder before venting the remnant gas?
(19) Have you replaced the spacers and screws in their boxes so that the next user would not have to search around for them?
(20) Before leaving the gas loading lab, please ensure that you have filled the log book with the details. This has nothing to do with the fact that your loading may not have worked. Please report any problems with gas loading to the safety officer, responsible staff member, and your supervisor. Do not attempt to proceed with gas loading before you make yourself familiar with the procedures under the supervision of responsible staff member, or your supervisor (if he is qualified to perform the gas loading). Only after successful loading under the supervision you will be allowed to operate the facility.

We are trying to keep up-to date manual for gas loading procedures, however, your major source of information should be your training person, either responsible staff member or your supervisor, who will train you to use the facility. While it is usually sufficient to make 1-2 loadings under supervision to get familiar with the procedure, your supervisor or responsible staff may require you to make as many supervised loadings as the see fit to get you comfortable with the procedure.

Please do not perform gas loading while being alone at the facility. All after hours operations are not allowed without supervision or without a second qualified companion who can watch over your loading experience.

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* The cryogenic loading must be refurbished or removed when Maddury Somayazulu leaves the Geophysical Laboratory.

Laboratory User

I agree that I have thoroughly read and understood this laboratory safety document. I have access to this safety information at all times when I am working. I have been trained to be able to identify the hazards to which I may be exposed and to follow the work practices and procedures discussed in this document. I certify that I will conduct my research work safely and that I will be responsible for following stated safety policies.

User Name (Print)	User Signature	Date

Principal Investigator

I certify that the information presented in this safety document is accurate and complete. I agree to comply with all safety procedures and to fully train and supervise all researchers under my direction.

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PI Signature Date