Safety Training for high pressure gases

Safety Manual for Handling High Pressure Gases and Cryogens

May 2013

Liquefaction and Supply Division

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1 Safe handling of the cryogens

1.1 Basic properties of cryogens

Cryogen like liquid nitrogen (LN_2) and liquid helium (LHe) characterized by their large gas/liquid volume ratio, low temperature, and an extreme high purity tend to cause accidents such as frostbite, explosion (rupture), and oxygen starvation (hypoxia) etc. It is important to understand their basic properties to handle them safely.

Table 1 shows physical and chemical properties of typical cryogens.

Table 1: Physical and chemical properties of cryogens often used
(0 degrees Celsius=273.15K (Kelvin) NBP: normal boiling point)

Gases	Boiling tempera-ture(K)	Mole-cular weight	Color	Smell	Taste	Category	Liquid density NBP (kg/L)	Specific gravity of gases (air=1)
Nitrogen N_2	77.3	28	No	No	No	Non flammeble	0.808	0.967
Oxygen O_2	90.1	32	Light blue	No	No	Combustion enhancing	1.144	1.105
Helium He	4.2	4	No	No	No	Non flammable	0.125	0.138
Neon Ne	27.1	20	No	No	No	Non flammable	1.207	0.696
Hydrogen H_2	20.3	2	No	No	No	flammable	0.071	0.069
Argon Ar	87.2	40	No	No	No	Non flammable	1.374	1.380
Air	78.8	29	Light blue	No	No	Combustion enhancing	0.874	1.000
Carbon dioxide CO_2	194.7	44	No	No	No	Non flammable	1.030 (-20℃, 1.967MPa)	1.530

1.2 How to handle cryogenic vessels

Cryogenic vessels are often called 'Dewars'. Use exclusively liquid nitrogen vessels to store liquid nitrogen, as well exclusively liquid helium vessels for liquid helium. The following notices are appropriate for both cases

1 Never apply a shock
2 Do not tip, because their construction is fragile against shock
3 Do not increase the inner pressure unintentionally -> To avoid blockage
4 Do not let the vessels open to the air for a long time -> To avoid blockage
5 Wear appropriate gloves for low temperature and safety goggles
6 Ensure the room has good ventilation
7 Waste residual liquid nitrogen in a safe place immediately after use (water and/or oxygen frozen at the lip of the vessels)

1.2.1 Liquid nitrogen vessels

Liquid nitrogen vessels often used for transport and store in small amount, are classified into the open- and self-pressurized vessels.

(1) Open vessels

They are in most cases relatively small in size (active volume less than 100L). It may be possible to tilt the vessels in order to take out the cryogen, but it is highly recommended to use a syphon in the upright position, since their construction is not robust against the lateral forces. For storing cryogen, use a properly designed cap in order to avoid condensing water inside, which will eventually causes blockage of the neck-tube. This cap is designed to keep a certain clearance to enable venting the boil-off gas. In case you lost the proper cap, take care not to seal the vessels and avoid opening to the air as well, for instance put dry clothes (waste) gently.

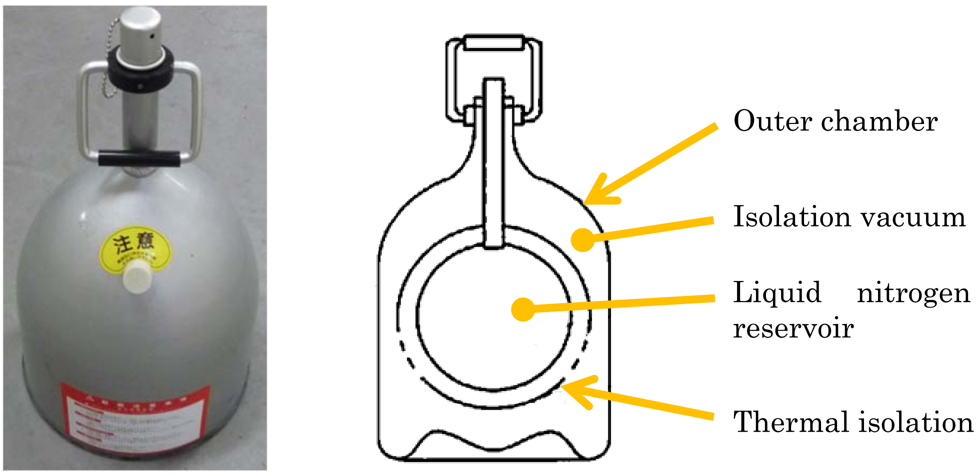
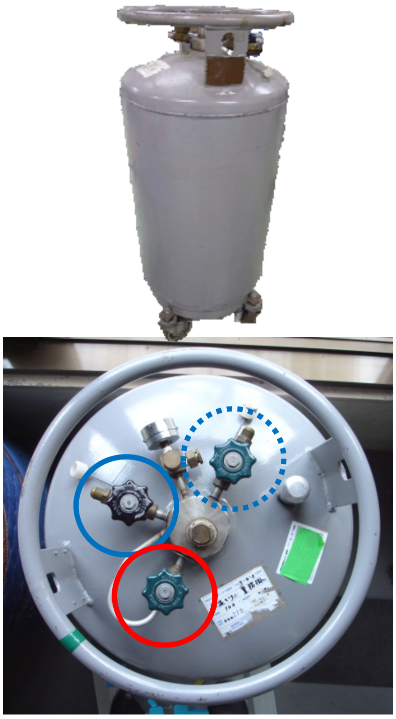


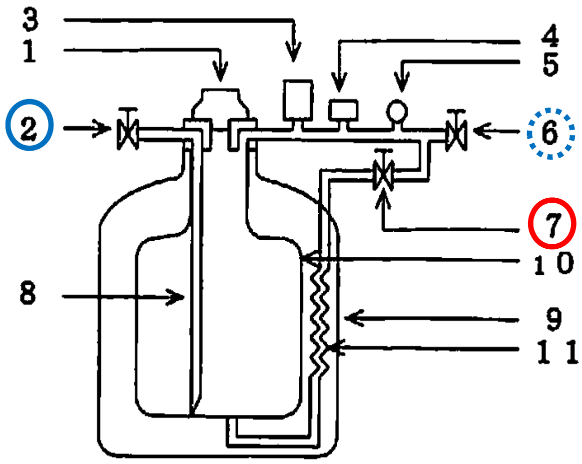
Fig. 1: External view of open type vessel for liquid nitrogen (left). Sectional view (right).
Leaving liquid nitrogen in a bucket type Dewar results in a deviation of the boiling

temperature due to the mixing of liquefied oxygen in the air or in high concentration oxygen condition around the Dewar lip. It is recommended to empty out the liquid in a safe place immediately after use, because high concentration oxygen enhances combustion accompanied by an explosion if the oxygen contact with, e.g., oils or greases.

(2) Self-pressurized vessels

The self pressurized vessels are equipped with pressure building coil-tube (evaporator) to increase the pressure inside the reservoir. In most cases they contents are larger than 50 liters.





1: Liquid inlet
2: Liquid outlet valve
3: Safety valve
4: Rupture disk
5: Pressure gauge
6: Gas vent valve
7: Pressure building valve
8: Syphon tube
9: Outer chamber
10: Inner bath
11: Evaporator

Fig.2 External view (left top), an example of valve configuration (left bottom) and construction (right) of self-pressurized vessels.

[How to use the self-pressurized vessels / Cautions]

1 Close the gas vent valve.

2 Slowly open the pressure building valve and confirm the increase of inner bath pressure

3 Open the liquid outlet valve and take out the liquid nitrogen. Care has to be taken not to increase the pressure too much. It is recommended to keep the pressure within the range 0.02 – 0.05MPa depending on your purpose.

4 Close the liquid outlet valve after you take out the required amount.

5 Close the pressure building valve and open the gas vent valve to reduce the pressure.
6 Vessels containing liquid nitrogen should be stored in a well-ventilated place, avoid exposure to sunshine. To prevent tipping, lock the wheels. In addition, the gas vent valve may be opened in order to avoid excessive overpressure. You may use a rubber with a slit to terminate the gas vent valve. This precaution may prevent the blockage caused by leaking air inside.

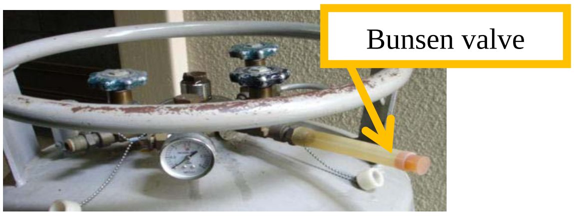


Fig.2: The Bunsen valve

[Re-inspection of self-pressurized vessels]

The high pressure gas safety act obliges re-inspection of self-pressurized vessels for liquid nitrogen and prohibit filling (and blowing out) the liquid nitrogen in such a vessel that expired the inspection period. Confirm the carved month/year on the production tablet (Fig.4). If your vessels require inspection, consult your local vessel supplier and proceed with the inspection at a registered vessel inspection station.

Table2: Period of vessel re-inspection (As of Apr. 2013)

Production year	Re-inspection period
Before 31. Mar. 1989	Every year
Not less than 20 years after production	Every 2 years
Less than 20 years after production	Every 5 years

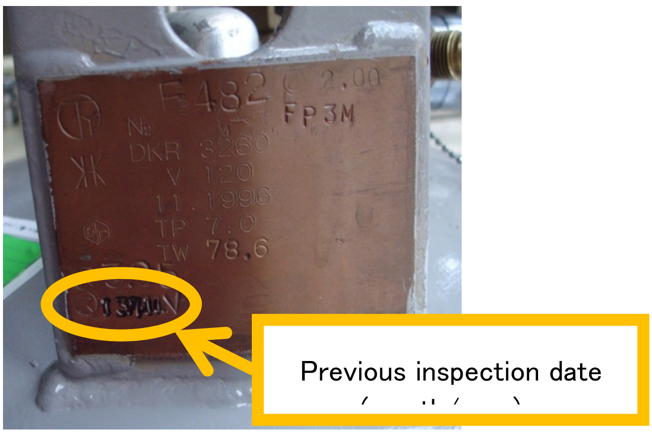


Fig. 3: An example of carved date of inspection

1.2.2 Vessels for liquid helium

The latent heat of liquid helium is very small. Even a tiny heat load boils off the liquid in a short time. Therefore multi-layer insulation type vessels are used which show improved isolation performance compared to a liquid nitrogen vessels.

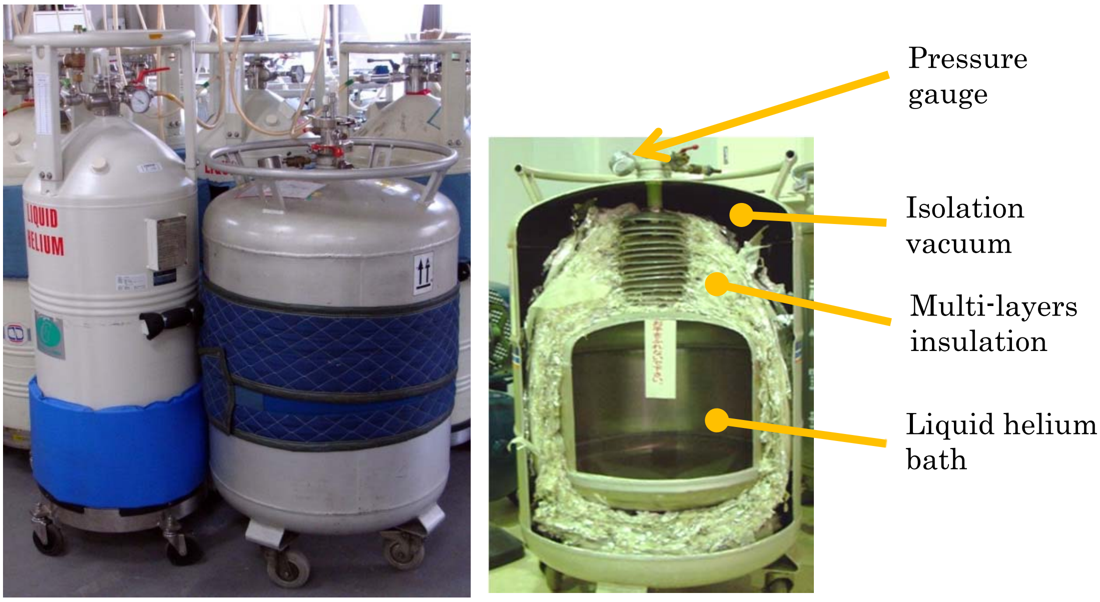


Fig. 5: External view (left) and the cross section (right) of liquid helium vessels. Thermal isolation based on the aluminum-evaporated miler films are wrapped in multilayers in order to prevent the heat transfer via radiation.

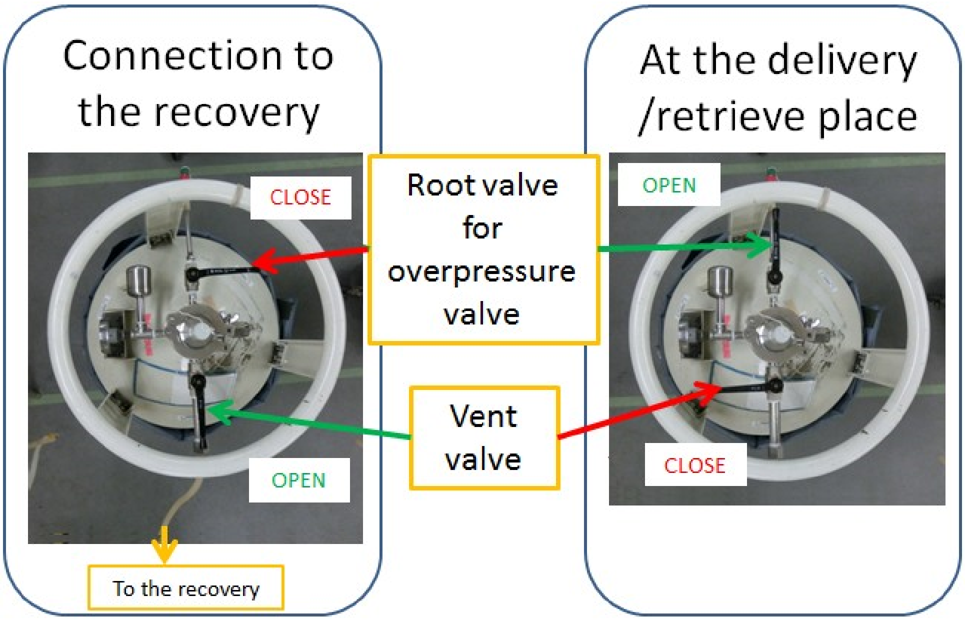


Fig. 6: An example of valve operation of the liquid helium vessel.

[How to use liquid helium vessels / Cautions]

1 Operate valves appropriately in order to prevent air leakage inside the vessels or excessive overpressure.

In the Lab. -> Connect the vent port to the helium recovery line and open the vent valve

At delivery/retrieve space -> Open the root valve to the overpressure valve and close the vent valve.

2 Liquid outlet port has to be firmly sealed.

[Liquid helium transfer]

1 Wear cryogenic gloves whilst you are working

2 Recover the boil off helium gas as much as possible

3 Warm up the boil off gas up to around room temperature … cold gas may eventually cause damage to the flow meter.

4 Do not fully insert the transfer tube into the vessel.

5 Return cryogenic vessels in cold condition, with not less than 10% cryogen remaining.

6 Do not apply too much overpressure. It may cause the boiling of all the residual cryogen inside the cryostat.

1.3 Notice about cryogenic vessels

[In case of blockage]

1 Wear cryogenic gloves whilst you are working.

2 Confirm the pressure inside the cryogenic vessels, safeness and ventilation of your surroundings.

3 In case of high pressure inside the vessels,

(N_2) Vent to the air carefully

(He) Vent to the recovery as much as possible (vent to the air, if not possible)

4 Remove the ice carefully …Keep distance your face from the liquid He outlet port

(LN_2) Flow He gas at room temperature

(LN_2 LHe) Touch gently with a warm copper rod or tube and remove the ice. A drier may be used to warm up the rod/tube

[In case of carrying cryogenic vessels with lift (elevator)]

There is an increasing risk to turn over the vessels at the entrance of a lift (an elevator), because of the finite clearance and the floor-level discrepancy between the building and the lift. In case you turn over the vessels cryogen may spill out and then it may injure or stifle the transporter and fellow passengers. Handle the cryogenic vessels with greatest care, when you get on and off the elevator, and comply with the rules of your faculty or building. Do not be panic, when you were confined in a lift during the transportation, since you are safe so long as the cryogenic vessels are standing in the upright position. Countermeasures in case you are confined in a lift:

Get off at the nearest floor (e.g.: Press emergency stop button)

Keep the door open at the floor stopped (e.g.: Press open prolongation button)

Announce that no other persons should get in the lift (e.g.: call disaster prevention center)

1.4 Use a liquid helium in Hongo campus

Helium is a noble element. In Japan the supply of helium depends 100% on import from abroad. In Hongo campus therefore, we repeatedly recover the boil off gas from the liquid helium and liquefy the recovered gas in the Cryogenic Research Center. Decrease of recovered amount, i.e. the increase of loss, affects the supply price reflecting the increase of the supplemental helium gas purchased. In addition, the decrease of purity of the recovered gas may eventually cause damage to the liquefying facility. To maintain reliable liquid helium supply, Cryogenic Research Center requires all the users improving the recovery rate and keeping high purity. Your cooperation will be highly appreciated.

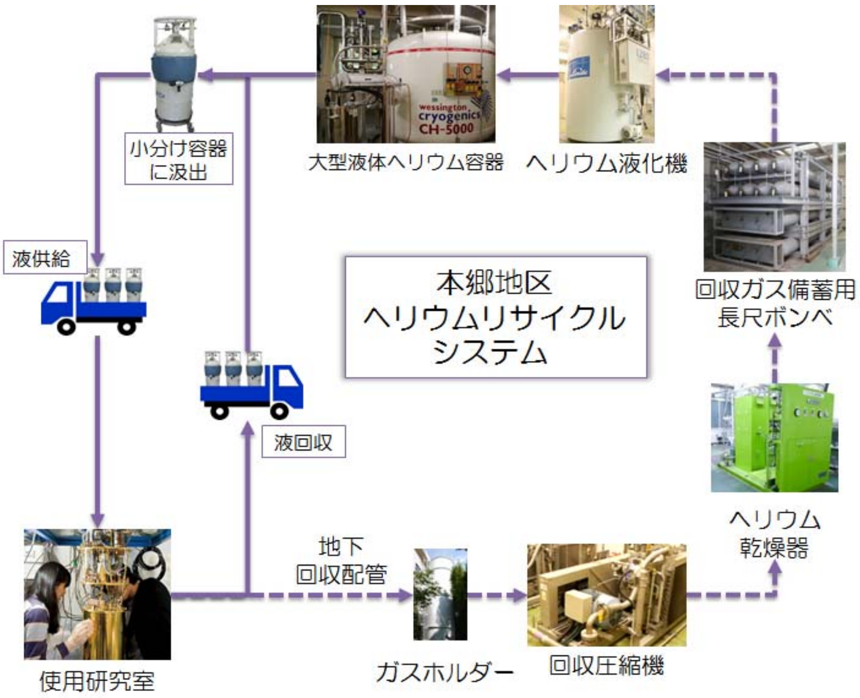


Fig. 7: Liquid helium cycle in Hongo campus

2 Prevention of accidents

2.1 Oxygen starvation (hypoxia)

Breathing the air with a dearth of oxygen causes severe damage to the brain and various symptoms appear to the human body (table 3). The symptoms may depend on the oxygen concentration, and are categorized in two cases:

A) Acute hypoxia: Caused by breathing the air with almost no oxygen.

B) Moderate hypoxia: Caused by breathing the air with a moderate oxygen deficiency.

In case A) those who try to rescue may suffer from oxygen starvation (secondary accident). In case B) although an operator would have a symptom such as headache or collapse, the operator may not be able to recognize that the symptom is arising from oxygen deficiency.

In contrast, the damage to the brain comes in a few ten seconds. In general, the sooner lifesaving first aid results in lower probability of death (Fig. 8). In case of Oxygen starvation, more careful and prompt action is required than in the general case. To prevent accidents caused by oxygen starvation (hypoxia), it is most effective to prevent oxygen starvation condition in working place. The operator should always take necessary precautions:

1 Open windows and doors and confirm the ventilation condition when you use cryogen.
2 Install oxygen monitors to confirm oxygen concentration.
3 Store an air respirator, get first aid training and take precautions for accidents.

In case the room is supposed to be apparently starved of oxygen due to sudden evaporation of liquid nitrogen or liquid helium, purge the working area with air first and confirm the oxygen concentration before you enter the room.

(This rule, however, does not apply to the cases when some other alarms for flammable gases or toxic gases are working)

Table 3： Symptoms of the human body due to reducing oxygen concentration

Concentration (%)	Symptom
18	Safety lower limit. Prepare ventilation, measurement of oxygen concentration and breath aiding devices.
16～12	Increase of the pulse and respiratory count. Concentration requires effort and fine muscle control doesn't work. Headache
14～9	Hard to make a decision. Exaltation condition, unstable mind condition and insensitive to pain etc. Feeling intoxicated, unable to know anything, rising body temperature and cyanosis.
10～6	Lose consciousness, an obstacle in central nerve, convulsions, cyanosis and chain Stokes respirations.
10～6 (continuously) or less	A coma →respiration slow →respiration stop → heartbeat stops after 6～8minutes

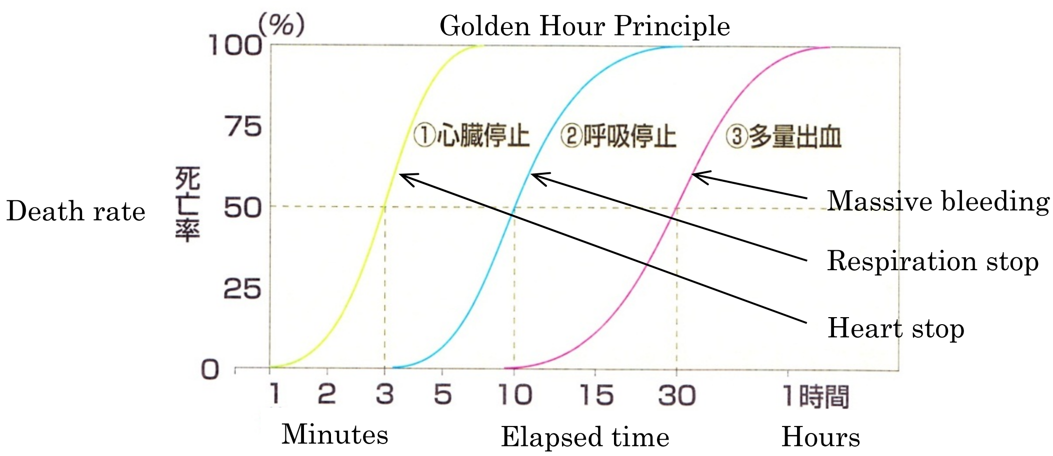


Fig. 8: The life-saving curve by Cara.

2.2 Frostbite

[Main causes]

1 Contact to the scattered cryogens, contact to ejected cold gases.

2 Contact to the cryogens with wet hands or feet.

3 Contact to the metals at cryogenic temperatures.

[Preventive measure]

1 Wear cryogenic gloves with good thermal isolation, which should be easy to get on and off. Cotton and wool gloves are prohibited.
2 Wear safety goggles.
3 Do not handle cryogens with wet hands
4 Use metal tube for transfer when possible and avoid fragile materials such as vinyl chloride or glass.

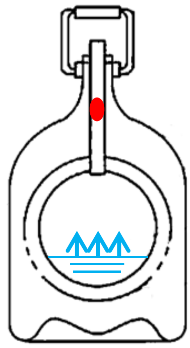
[In case of frostbite]

Worm up the affected part with tepid water. Immediately consult a doctor. Do not heat up the affected part with a drier.

2.3 Rupture / Explosion

[Cause]

Examples of the rupture of liquid nitrogen vessels and liquid helium vessels are following,



An example of causes: Ice blocks neck tube and prevents evaporation.

All the valves are closed which results in anomalous overpressure.

Sudden degrade of thermal isolation causing evaporation which could not be vented in time.

Vessel has been broken by an external shock.

*Combustion may occur by contact of high concentration oxygen (e.g. liquid oxygen) with organic material such as greases. In the case of a flammable cryogen such as liquid hydrogen an explosion may occur by electrostatic discharge.

[Countermeasure]

1 Use vessels with good thermal isolation, the inside of which should be sufficiently dry.
2 Do not close the vessel tightly.
3 Keep cryogens away from fire (especially for flammable gases).
4 Vent the evaporated gas in an outdoor safe place without flammables
5 Do not wear gloves or clothes contaminated with oil or greases.

3 Safe handling of gas cylinders

3.1 Gas cylinder

Gas cylinders (high pressure vessels) are classified by external color according to Table 4 (High pressure gas safety act , vessels rule article 10). At the shoulder of the gas cylinder a carved stamp informs the contents and specification (Table 4). Cylinders unable to carve a stamp are labeled showing the species of filling gases.

Fig.9: External view (left) and carved stamp (right) of gas cylinders.

Table 4: External color of gas cylinders (left). Contents of carved stamp (right). This classifications do not apply to, e.g., gas cylinders imported from abroad.

Species of gases	External color
Oxygen	Black
Hydrogen	Red
Liquefied Chlorine	Yellow
Acetylene	Brown
Liquefied Carbondioxyde	Green
Liquefied Ammonia	White
Other high pressure gases	Gray

Description	Example
Gas type	He
Registration number of the vessel	ABC23456
Volume（L）	V 47.2
Empty weight (kg)	W 60.2
Month year of passed pressure test	0 4.98
Hydrostatic test pressure	TP 24.5
Maximum allowing filling pressure (at 35℃)	FP 14.7

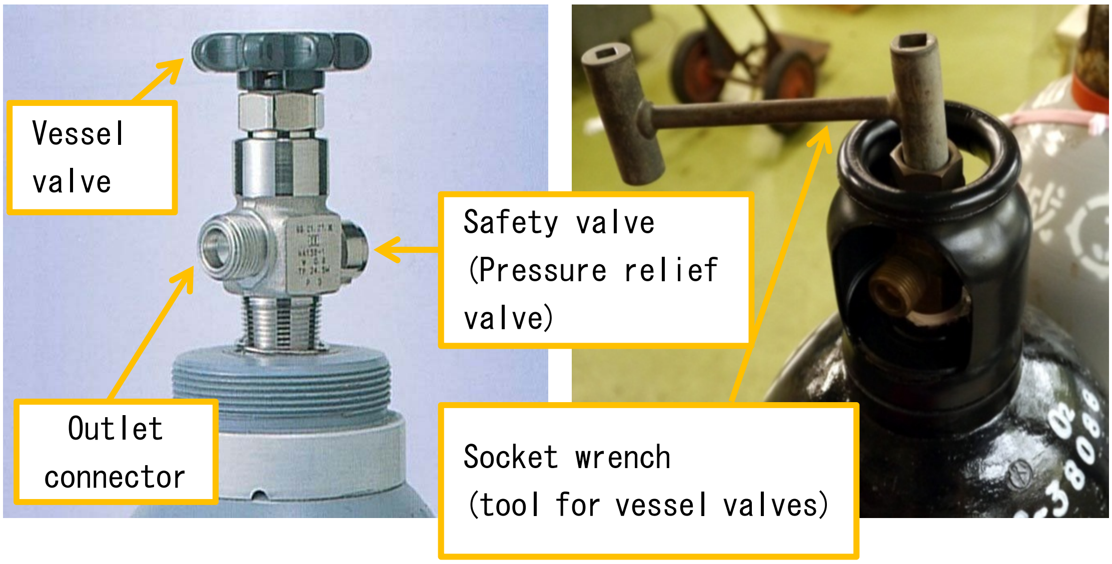


Fig. 10: External view of vessel valves with knob (left) and without knob (right).

[Cautions for storing gas cylinders]

1 Keep in the upright position and fasten the vessels firmly to the proper frame using two sets of chains in upper and lower position.

2 Make the frame stable and anchored firmly to the floor.

3 Avoid the overtemperature.

4 Avoid exposure to the sun. Do not place in the vicinity of the welding working place.

5 Store in a well ventilated place.

6 For storage in lateral position owing to unavoidable circumstances, take necessary precautions to prevent rolling.

[Cautions for using gas cylinders]

1 Operate valves slowly and carefully. Do not apply excessive force (torque).

2 Do not stand or face in the way of the gas flow.

3 Open a valve once fully and then turn it back a half turn

Opening a valve to the end with too much torque may damage the thread, making it impossible to close again. When you have no idea if the valve is open or closed, then turn to close-direction first.

4 When you have finished using the gas, close the vessel valve firmly and put on a proper cap.

[Cautions for transport gas cylinders]

1 Use proper carriage for gas cylinders.

2 Do not touch the valve and attach the protection cap adequately during the transportation.

3 Put on safety shoes.

4 After unloading the cylinder from the transport carriage, move the cylinder gently.

Fig.11 The carriage of gas cylinders.

3.2 Pressure regulators

A pressure regulator (pressure reducing valve) is an apparatus which supplies gas, e.g., from a gas cylinder with constant pressure. Different pressure regulators are required for different gas types, since the outlet base connections depend on the type of gases. A left thread is used for flammable gases such as hydrogen and for helium. A right thread is used and for the other gases. Prepare a proper pressure gauge, hose, tubing etc. for each gas type. Pay attention especially to the oxygen case, since oil or fat inside the system causes an explosion.

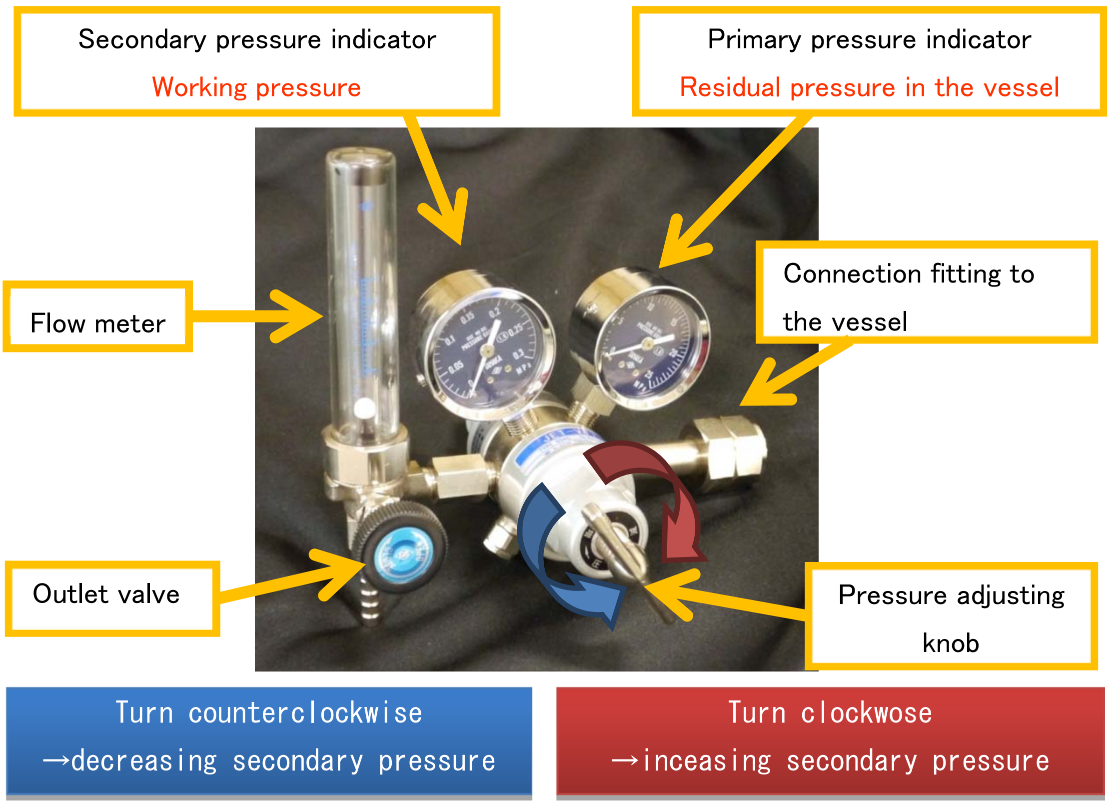


Fig.12 Pressure regulator

[How to use the pressure regulator / Cautions]

1 Before you attach the pressure regulator to the gas cylinder, turn the pressure reducing valve counterclockwise until the knob becomes lose to prevent any gas flow.

2 Clean up the dust inside the fitting and attach it to the gas cylinder. Do not use the pressure regulator, if the thread of the gas cylinder and pressure regulator does not fit (too much clearance),.

3 Pay an attention to the gas flow direction and confirm nobody is in the way and then open the vessel valve slowly.

*Do not face towards the pressure indicator

*Operate valves (open/close) sufficiently slowly and carefully

* Confirm that there is no sound of leaking gas whilst you read the primary (high) pressure indicator.

4 Put bubbling solution on the gas-connector fitting to confirm that no gas is leaking from the connection. In case there is a leak from the fitting, close the vessel valve first and reduce the primary pressure down to zero. Then check the condition of the gasket and replace it if necessary.

5 Set secondary (working) pressure by turning the pressure adjusting knob clockwise. In case of the high purity gas, purge inside the pregulator and the tube sufficiently.

6 Open the outlet valve and take the gas out

7 When you have finished using the gas, turn the pressure adjusting knob counterclockwise until it becomes loose and then close the vessel valve.

8 Release all the residual gas inside the regulator before removing the regulator from the gas cylinder.

4 High Pressure Gas Safety Act

4.1 The aim of the high pressure gas safety act

The high pressure gas safety act is a law that regulates the handling of high pressure gases to prevent disaster (High pressure gas safety act article 1). Many of the liquid cryogens and gas cylinders used in the university come under this regulation. We are required to understand the aim of the law and we shall endeavor to prevent accidents and ensure the security and safety of the public.

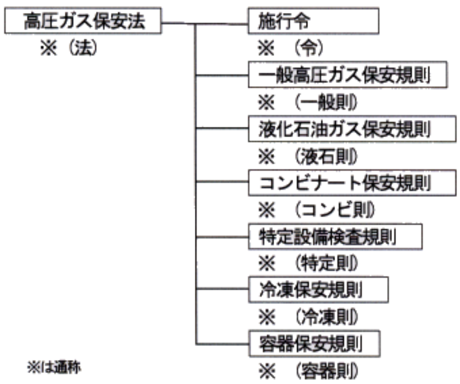


Fig.13: The high pressure gas safety act and related rules

4.2 Definition and classification of high pressure gas

The high pressure gas safety act defines "high pressure gases" as follows (High pressure gas safety act article 2). The pressure mentioned hereinafter is in gauge pressure (= absolute pressure –atmospheric pressure).

1.Compressed gas, the pressure of which is not less than 1 mega Pascal at its normal operating temperature and which is currently not less than 1 mega Pascal, or compressed gas, the pressure of which is not less than 1 mega Pascal at a temperature of 35 degrees Celsius (except compressed acetylene gas in both cases);
2. Compressed acetylene gas, the pressure of which is not less than 0.2 mega Pascal at its normal operating temperature and which is currently not less than 0.2 mega Pascal, or compressed acetylene gas, the pressure of which is not less than 0.2 mega Pascal at a temperature of 15 degrees Celsius;
3. Liquefied gas, the pressure of which is not less than 0.2 mega Pascal at its normal operating temperature and which is currently not less than 0.2 mega Pascal, or liquefied gas, the temperature of which is 35 degrees Celsius or less in the case that the pressure is 0.2 mega Pascal; or
4. In addition to what is listed in the preceding item, those liquefied gases, the pressure of which exceeds zero Pascal at a temperature of 35 degrees Celsius, and which, inclusive of liquefied hydrogen cyanide and liquefied methyl-bromide, are specified by a Cabinet Order.

The pressure of liquefied gases is judged by their saturated vapor pressure. The saturate pressure of liquid nitrogen and of liquid helium exceed 0.2 mega Pascal at -189 degrees Celsius and -268 degrees Celsius respectively and, therefore, they define themselves as high pressure gas. The object and contents vary from each other depending on the classification (flammable, combustion enhancing, non-flammable, / poisonous, not poisonous) of the gases. A notification to the corresponding supervisory office is required in certain cases before use of gas cylinders or in design stage of facilities.

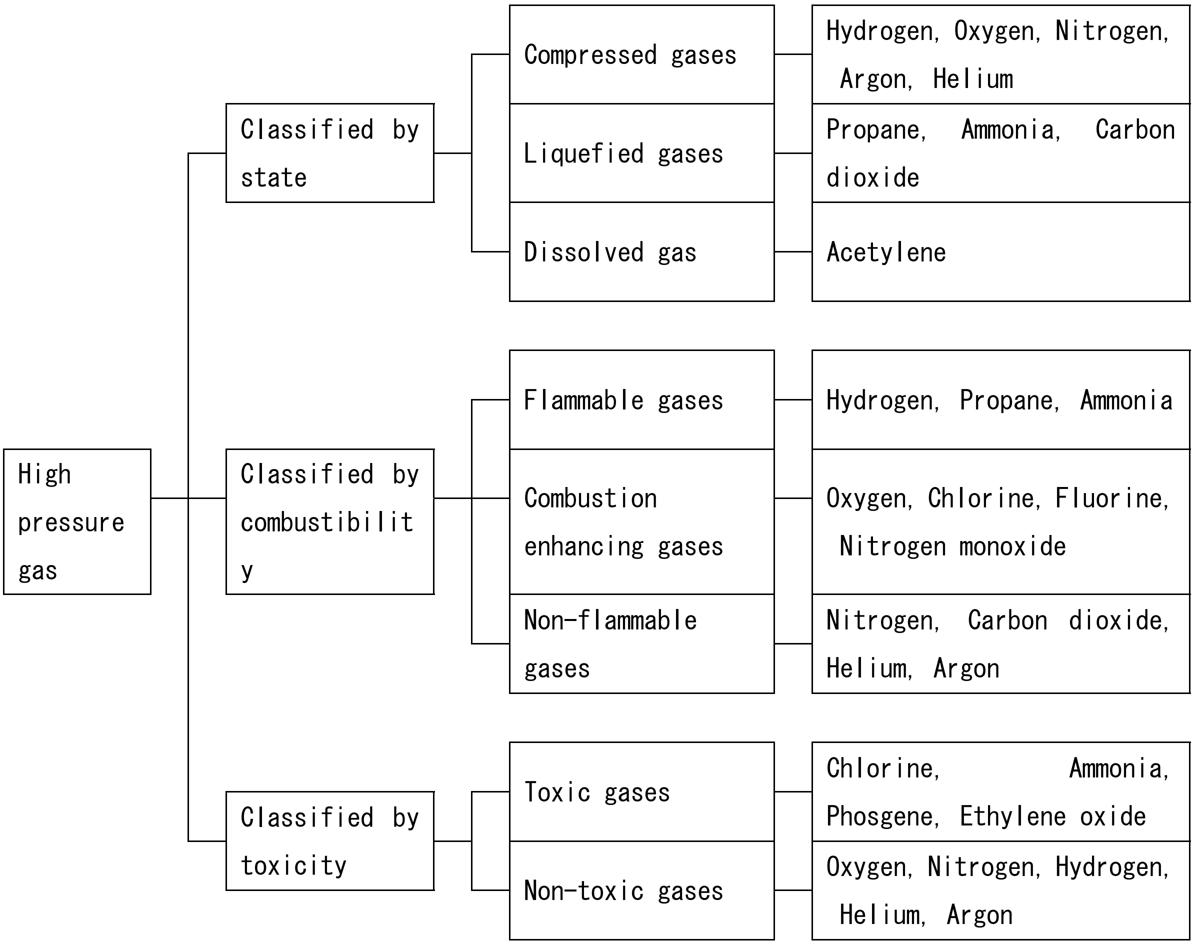


Fig. 14: Classification of high pressure gases

4.3 Production, Storage, and Consumption of high pressure gases

An application and a notification to the supervisory office are required in certain case, when you produce, store, and consume high pressure gases. Please understand well the construction of your experimental equipment, and the process concerned, and consult your local faculty administrative office for safety and health in advance.

(1) Production

To produce high pressure gases artificially used in the method of compression or liquefaction.

Application and notification to the supervisory government agency are required depending on the processing amount.

Example: To extract the gas at secondary pressure not less than 1 mega Pascal from a gas cylinder using a high-pressure regulator, or produce a gas not less than 1 mega Pascal using a compressor.

(2) Storage

To keep gases in a state of high-pressure gas exceeding the limited amount at a certain place.

Application and notification to the supervisory government agency are required depending on the storage amount.

Example: liquid nitrogen cold evaporator

(3) Consumption

To use after making the transition from high pressure gas to the state of a non high pressure, using such as a pressure reducing valve.

Notification to the supervisory government agency is obligatory in advance, for consumption of the special high pressure gas such as

arsine, disilane, diborane, hydrogen selenide, phosphine monogermane and monosilane.

Otherwise no approval procedure is required for consumption of small amounts.

Example: Use the gas at a pressure less than 1 mega Pascal from a gas cylinder with a pressure regulator.

4.4 High pressure gas safety act and Cryogenic Research Center

Cryogenic Research Center proceeds with production of liquid helium and storage of liquid nitrogen as a first kind of producer as provided by the high pressure gas safety act. A safety inspection by the supervisory government agency (once a year), enactment and notification of the regulations for prevention of danger and injury, planning and proceeding of safety instruction, regular autonomy inspection, and daily inspection are obliged to a first kind of producer. The liquefying division of the Cryogenic Research Center endeavors for the reliable supply of liquid helium, and liquid nitrogen by daily operation and maintenance of production and storage facility, compliance with the requirement of the law, and autonomy safety activities as well. We are going to keep the safe research environment in corporation with all the users.

5 Administration of high pressure gases in the University of Tokyo

In April 2013 'The Regulation for high pressure gas administration in the University of Tokyo' and 'The criteria for autonomy administration of high pressure gas in the University of Tokyo' have been enacted. As provided in those regulation and criteria, all the high pressure gas users in the University of Tokyo are required to comply with the safety instruction of the environmental safety headquarter and of the local faculty administrative office for safety and health as well, to take necessary safety instruction before use of any high pressure gas and to attend safety instruction of Cryogenic

Research Center especially for all liquid cryogen uses.

Administration of the high pressure gases of the University of Tokyo outside of the Hongo campus and the external Research Institutes could vary from that of Hongo campus. We ask all of you to follow the local instructions and guidance and to handle the high pressure gases safely.

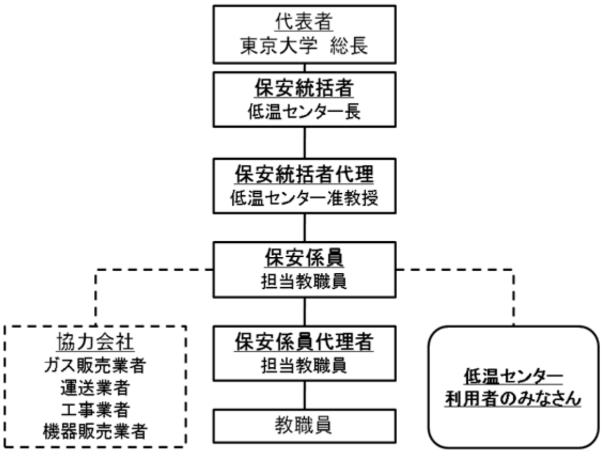


Fig 15: Organization of the safety administration

6 References

[1] e-Gov Data Supply System for Act http://law.e-gov.go.jp/cgi-bin/idxsearch.cgi

[2] Handbook on the Cryogenic Engineering edited by Japan Cryogenic Engineering Society Kansai Division (in Japanese)

[3] Chronological Science Tables (2006) Maruzen Publishing (in Japanese)

[4] S. Kobayashi and Y. Otsuka "Low Temperature Technique 2nd edition" University of Tokyo Press (in Japanese)

[5] Textbook for "High Pressure Gas Safety Instruction for the Newcomers" Low Temperature Liquefying Center, Institute of Solid State Physics, University of Tokyo (in Japanese)

[6] Instrucion for High Pressure Gas fiscal year2012 Environmental Safety HeadquarterUniversity of Tokyo. (in Japanese)

7 Appendix

Conversion table of the pressure units

The pressure is mentioned in Pa (gauge pressure) units in he high

Table 5: Pressure units often used

	Pa	MPa	kgf/cm^2	atm	bar	Torr	psi
1 Pa		1×10^{-6}	1.02×10^{-5}	9.87×10^{-6}	1×10^{-5}	7.50×10^{-3}	1.45×10^{-4}
1 MPa	1000000		10.2	9.87	10	7.50×10^{3}	145
1 kgf/cm^2	98066.5	0.0981		0.968	0.981	736	14.2
1 atm	101325	0.101	1.03		1.01	760	14.7
1 bar	100000	0.1	1.02	0.987		750	14.5
1 Torr	133.322	1.33×10^{-4}	1.36×10^{-3}	1.32×10^{-3}	1.33×10^{-3}		0.0193
1 psi	6894.76	6.90×10^{-3}	0.0703	0.0680	0.0689	51.7	
別表記	N/m^2				dyn/cm^2	mmHg	lbf/in^2

issued　May 2013

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