INFN-LNL-270/2022

Corso Nazionale INFN "Introduzione alla criogenia"

Chiara Vignoli, INFN-LNGS

Bologna, 28-30 ottobre 2019

- Introduction to cryogenics
- Cryogenic fluid properties
- Cryogenic sensors & instrumentation
- Safety issues in cryogenics

Cryogenic fluid properties

Chiara Vignoli

Corso Nazionale INFN "Introduzione alla criogenia"
Bologna, 28-30 ottobre 2019

Why 120 K?

It’s the temperature below which “permanent gases” start to condense (LXe is not “cryogenic”)

Fluid	Normal Boiling Point (K)
(Xenon)	(165.04)
Krypton	119.8
Methane	111.6
Oxygen	90.2
Argon	87.3
Air	78.9
Nitrogen	77.4
Neon	27.1
Hydrogen	20.3
Helium	4.2

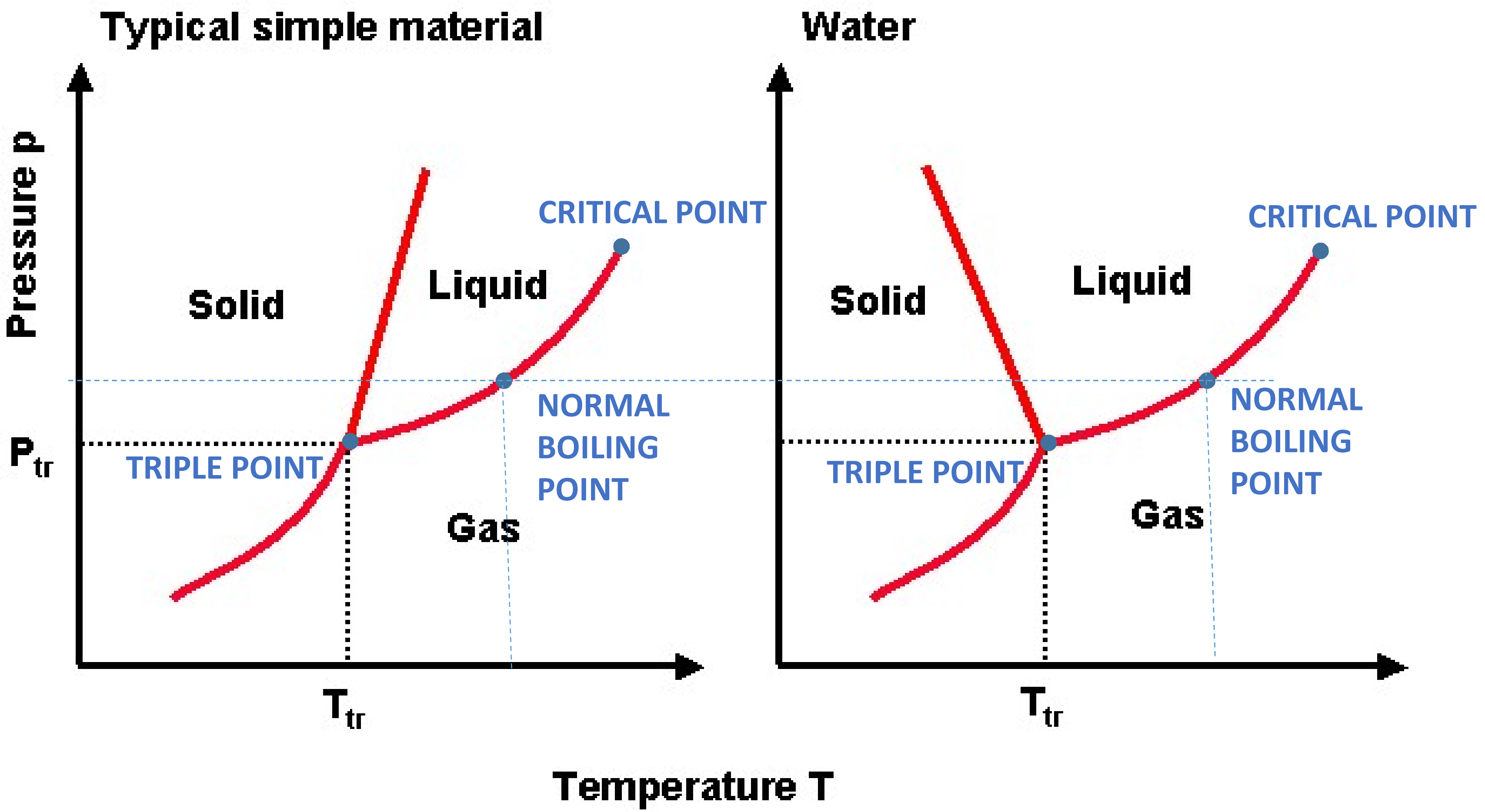
“Normal” means at ambient pressure 1 atm = 1.013 bar

Refrigeration

0 K	**120 K**	**273.15 K**

Cryogenics	Other kind of industrial refrigeration
Krypton	R134a (246.8 K)
Methane	R12 (243.3 K)
Oxygen	R22 (233 K)
Argon	Propane (231.1 K)
Nitrogen	Ethane (184 K)
Neon	CO_2 (195 K- solid)
Hydrogen	Ammonia NH3 (239.8)
Helium	…

P-T diagrams



Note: "Typical" diagrams.

Exception examples:

- Helium: no triple point.
- CO_2 is solid at atmospheric pressure

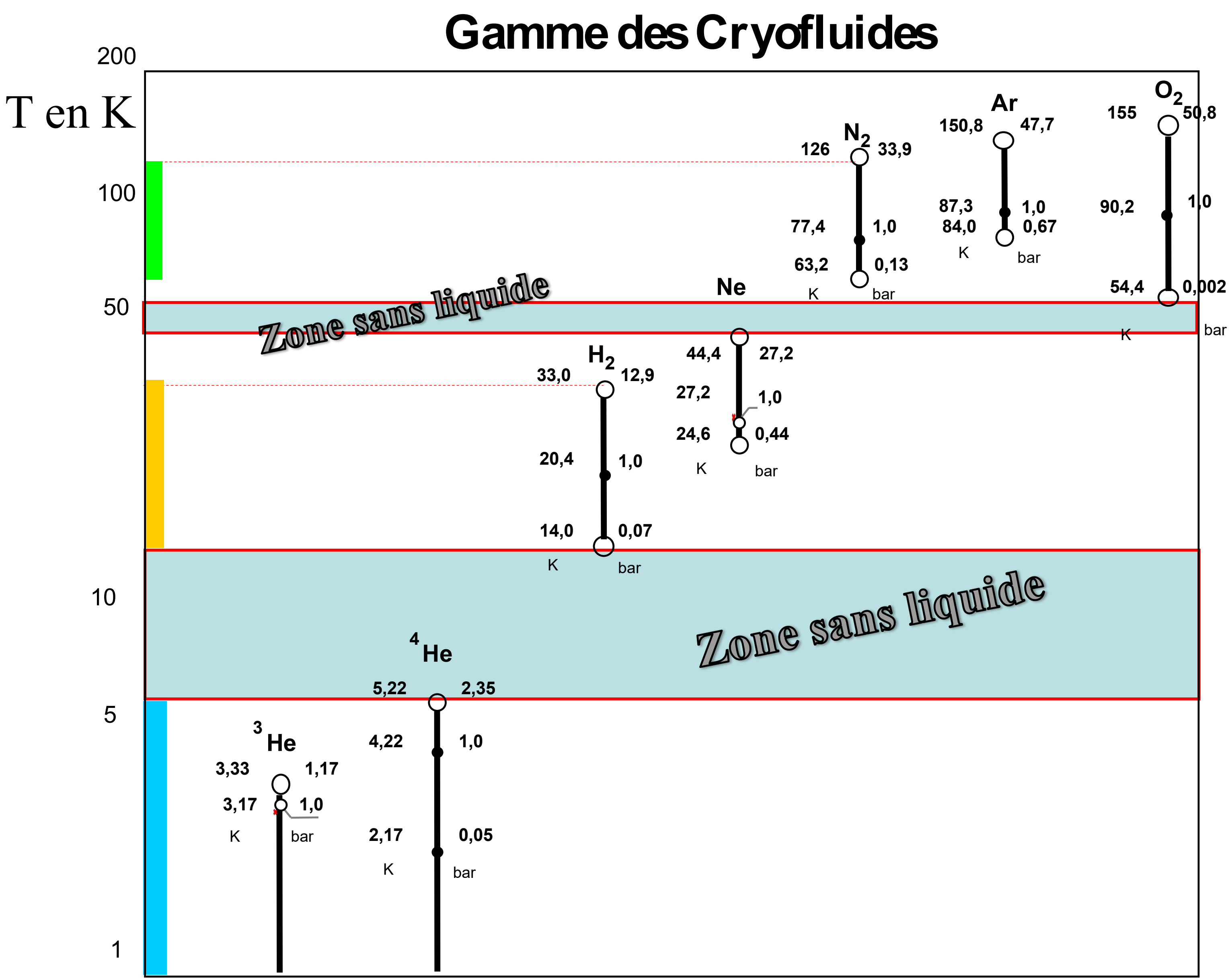
Characteristic temperatures of cryogenic fluids [K]

Cryogen	Triple Point	Normal boiling point	Critical Point
Methane	90.7	111.6	190.5
Oxygen	54.4	90.2	154.6
Argon	83.8	87.3	150.9
Nitrogen	63.1	77.3	126.2
Neon	24.6	27.1	44.4
Hydrogen	13.8	20.4	33.2
Helium	2.2*	4.2	5.2

∗ λ point

The simplest way of cooling equipment with a cryogenic fluid is to make use of its latent heat of vaporization, e.g. by immersion in a bath of boiling liquid. As a consequence, **the useful temperature range** of cryogenic fluids is that in which there exists latent heat of vaporization, i.e. **between the triple point and the critical point**, <u>with a particular interest in the normal boiling point,</u> i.e. the saturation temperature at atmospheric pressure.

Cryofluid temperature inventory



Comparison with water

The most famous cryogens:

- Helium which is the only liquid at very low temperature
- Nitrogen for its wide availability and ease of use for pre-cooling equipment and thermal shielding

To develop a feeling about properties of these cryogenic fluids, it is instructive to compare them with those of water.

PROPERTY	HELIUM	NITROGEN	WATER
Normal boiling point [K]	4.2	77	373
Critical temperature [K]	5.2	126	647
Critical pressure [bar]	2.3	34	221
Liquid density* [kg/m^3]	125	808	960
Liquid/vapor density ratio*	7.4	175	1600
Heat of vaporization* [kJ/kg]	20.4	199	2260
Heat of vaporization* [kJ/l]	2.6	161	2170
Liquid viscosity* [μ Pl]	3.3	152	278

* at normal boiling point

Selected properties of cryogenic fluids

Characteristics	LHe-4	LH_2	LN_2	LAr	LO_2
Normal Boiling Point (K)	4.214	20.27	77.36	87.3	90.18
Density at NBP (kg/m^3)	124.8	70.79	807.3	1400	1141
Latent Heat at NBP (kJ/l)	2.6	31.4	161	220	243
Entalpy of warmed gas to 300 K (kJ/kg)	1550	3800	233	112	193
Ratio of entalpy of vapour at 300 K and latent heat at NBP	74.2	8.6	1.2	0.7	0.9

Selected properties of cryogenic fluids

Table 2.1. Selected Properties of Cryogenic Liquids at Normal Boiling Point

	Liquid helium-4	Liquid e-hydrogen	Liquid neon	Liquid nitrogen[a]	Liquid air	Liquid fluorine	Liquid argon	Liquid oxygen[b]	Liquid methane
Normal boiling point (K)	4.224	20.268	27.09	77.347	78.9	85.24	87.28	90.18	111.7
Density (kg/m^3)	124.96	70.78	1204	808.9	874	1506.8	1403	1141	425.0
Heat of vaporization (kJ/kg)	20.73	445.6	86.6	198.3	205.1	166.3	161.6	212.9	511.5
Specific heat (kJ/kg K)	4.56	9.78	1.84	2.04	1.97	1.536	1.14	1.70	3.45
Viscosity ($kg/m \cdot s \times 10^6$)	3.57	13.06	124.0	157.9	168	244.7	252.1	188.0	118.6
Thermal conductivity ($mW/m \cdot K$)	27.2	118.5	113	139.6	141	148.0	123.2	151.4	193.1
Dielectric constant	1.0492	1.226	1.188	1.434	1.445	1.43	1.52	1.4837	1.6758
Critical temperature (K)	5.201	32.976	44.4	126.20	133.3	144.0	150.7	154.576	190.7
Critical pressure (MPa)	0.227	1.293	2.71	3.399	3.90	5.57	4.87	5.04	4.63
Temperature at triple point (K)	—	13.803	24.56	63.148	—	53.5	83.8	54.35	88.7
Pressure at triple point ($MPa \times 10^3$)	—	7.042	43.0	12.53	—	0.22	68.6	0.151	10.1

[a] Reference 3.
[b] Reference 1.

Selected properties of cryogenic fluids

P norm = 101.3 kPa
(1 atm = 1.013 bar)

Propriétés de quelques fluides		He3	He4	H2	D2	Ne	N2	O2	Ar	CH4	H20
Température d'ébullition à p normale ($1.013 .10^5$ Pa) (K) – Téb -		**3.2**	**4.2**	**20.4**	**23.6**	**27.1**	**77.3**	**90.2**	**87.3**	**111.7**	**373.15**
POINT TRIPLE	Tt en K	-	-	13.95	18.70	24.50	63.14	54.40	84.00	90.70	273.16
	Pt en hPa	-	-	72	170	424	125	2	670	116	6
POINT CRITIQUE	Tc en K	3.33	5.20	33.20	38.30	44.40	126.10	154.40	150.80	191.00	647.14
	Pc en 10^5 Pa	1.16	2.23	12.80	16.50	26.60	33.10	49.50	47.70	45.80	220.60
Volume de gaz provenant de l'évaporation d'1 litre de liquide	à Téb et pnormale (l)	2.5	7.3	54.6	70.0	127.0	180.0	260.0	240.0	250.0	
	à T et p normale (l)	455	700	790	900	1355	646	798	784	595	
Chaleur latente L de vaporisation à Téb et p normale (kJ/kg)		**8.2**	**21**	**452**	**305**	**86**	**199**	**213**	**157**	**510**	**2250**
Enthalpie sensible entre Téb et 300 K (kJ/kg)		**2080**	**1550**	**3800**	**2048**	**280**	**233**	**193**	**112**	**402**	
Taux d'évaporation (Wh/l) = nombre de watts à déposer pour vaporiser 1 litre en 1 heure		**0.14**	**0.7**	**9.0**	**13.6**	**29**	**45**	**68**	**61**	**60**	**624**
Capacité calorifique à pression constante (kJ/kg.K) à 0°C et 1 bar		-	5.20	14.05	-	1.03	1.038	0.909	0.52	2.19	1.842 (à 100°C)
Conductibilité thermique du gaz à Téb (mW/m.K)		-	10	15	< 40	8	7.6	9	8	8.7	22
Conductibilité thermique du gaz à p normale et 300 K (mW/m.K)		-	152	181	137	50	26	27	18	31	
Masse volumique du liquide à p normale (kg/m³)		59	125	71	161	1210	810	1140	1400	425	998
Masse volumique de la vapeur saturante à p normale (kg/m³)		24	17	1.3	2.3	9.5	4.5	4.4	5.8	1.7	0.77
Masse volumique du gaz à p et T normales (kg/m³)		0.13	0.18	0.09	0.18	0.9	1.25	1.43	1.8	0.55	
Viscosité du liquide à Téb (μPa.s)		2	3.6	13	16.2	125	160	190	260	120	278
Viscosité du gaz à Téb (μPa.s)		1.2	1	1	1.5	4.5	5	7	8	4.4	12.5
Viscosité du gaz à Tambiante (μPa.s)		-	20	9	13	30	17	20	22	11	-
Permittivité du liquide		-	1.05	1.23	1.27	1.19	1.44	1.48	1.54	1.68	80

Selected properties of cryogenic fluids

Characteristics	Krypton	Argon	Nitrogen	Neon	Helium
Boiling point at 1 bar	-153.4 ℃ (120 K)	-185.8 ℃ (87.3 K)	-195.8 ℃ (77.3 K)	-246 ℃ (27.1 K)	-268.9 ℃ (4.2 K)
Density of liquid at boiling point [kg/m³]	2413	1400	810	1210	125
Litres of gas at 20 ℃ produced by 1 litre of liquid, 1 bar	700	841	693	1454	751
Density at 20 ℃ compared to density of air	2.9	1.4	1.0	0.7	0.14
Latent heat [kJ] of evaporation for 1 litre of liquid	260	220	160	104	2.6
Ratio of enthalpy of vapour at 20 ℃ and latent heat of evaporation	0.67	0.7	1.14	3.2	72

Some simple exercises

Example n.1

- We have a dewar of 100 l in our lab filled with a cryogenic liquid.
- Let’s calculate liquid loss for evaporation in case of 10 W heat input due to electrical power dissipation in the liquid:
  - LHe =
  - LN_2 =
  - LAr =

Example n.1 - cont

- Let’s calculate now how many liters of gas are produced when warmed up to ambient temperature

Example n.1 - cont

- And what happens if I let them stratify in the lab (no ventilation)?

Example n.2

- Suppose that the previous dewar of 100 l was built to contain liquid helium.
- Let’s discuss if it is possible to operate it with:
  - LN_2
  - LAr

Example n.2 (cont)

- Viceversa, if the previous dewar of 100 l was built to contain liquid nitrogen.
- Let’s discuss if it is possible to operate it with:

  - LHe

  - LAr

Example n.2 (cont)

- Suppose that the manufacturer specifications of the heat losses of the dewar nitrogen are 1% per day.
- What happens if I use it for LHe?

General properties of cryofluids

- Due to the wide temperature & pressure ranges covered by cryogenics the **properties of fluids vary greatly**
  - generally **we can't assume constant properties**
- Understanding changes in the thermodynamic state of the fluids allows us to describe refrigeration and liquefaction cycles
- **Pure cryogenic fluids act as classical fluids**
  - **with the exception of Helium and Hydrogen**
- Fluid properties are well known (mostly) & many resources exist

Thermophysical cryofluid properties

1. **State properties**

   related to a fluid inperturbed by any temperature gradient or fluid motion

   - Phase Diagrams
   - Equations of State (imperfect gases)
   - Condensed phase properties (ρ, β, κ, c_p, h_{fg}, σ)
   - Density
   - Specific heats
   - Thermal expansivity
   - Compressibility (isothermal and isoentropic)
   - Sound velocity
   - Joule-Thomson coefficient
   - Optical, electric and magnetic properties

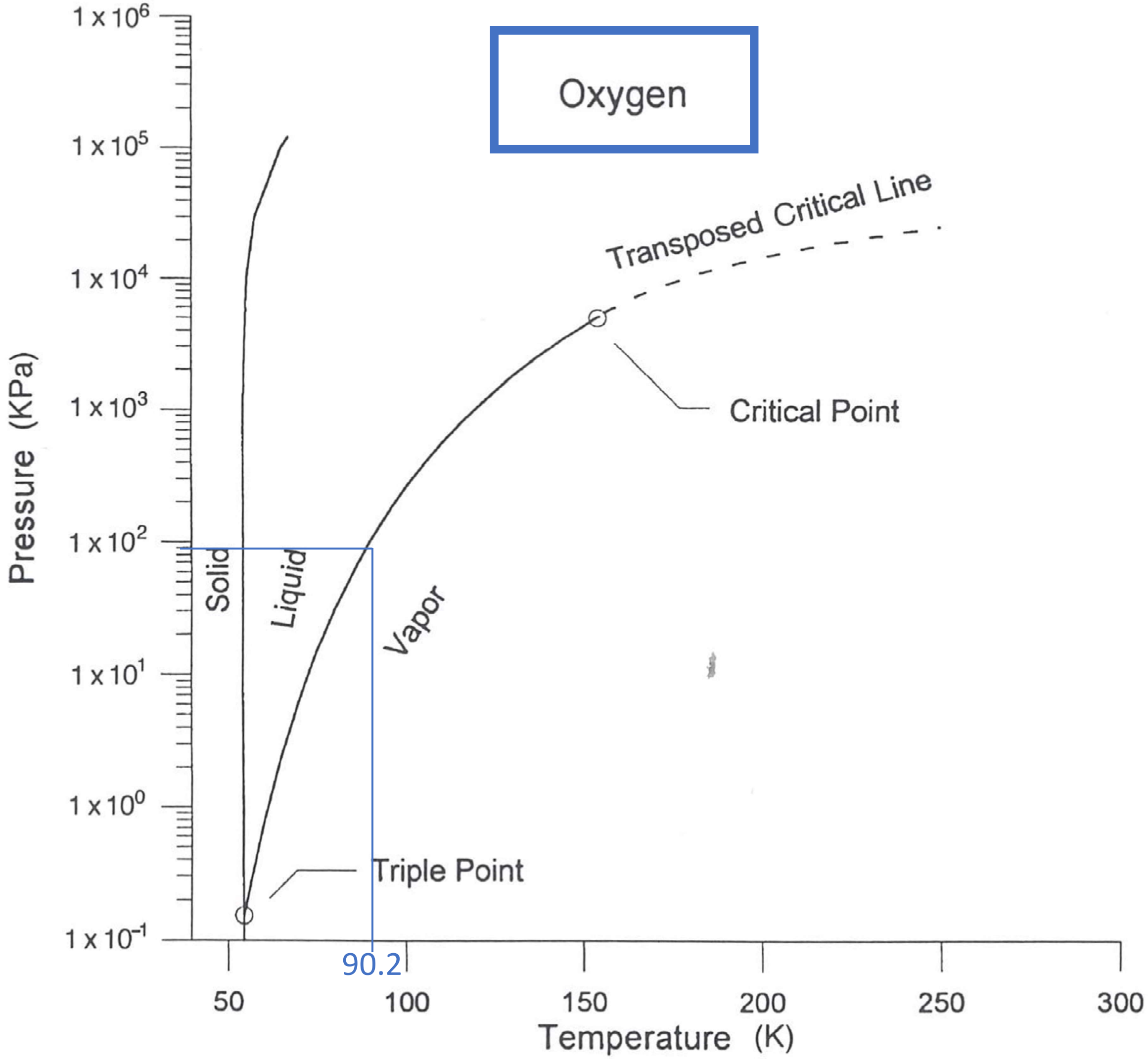
2. **Transport properties**

   related to the transport of energy or momentum through the fluid

   - Thermal conductivity (k_{cond})
   - Viscosity (μ)
   - Prandtl number ($Pr = \mu c_p/k$)

These properties depend on the molecular configuration and inter-molecule interactions

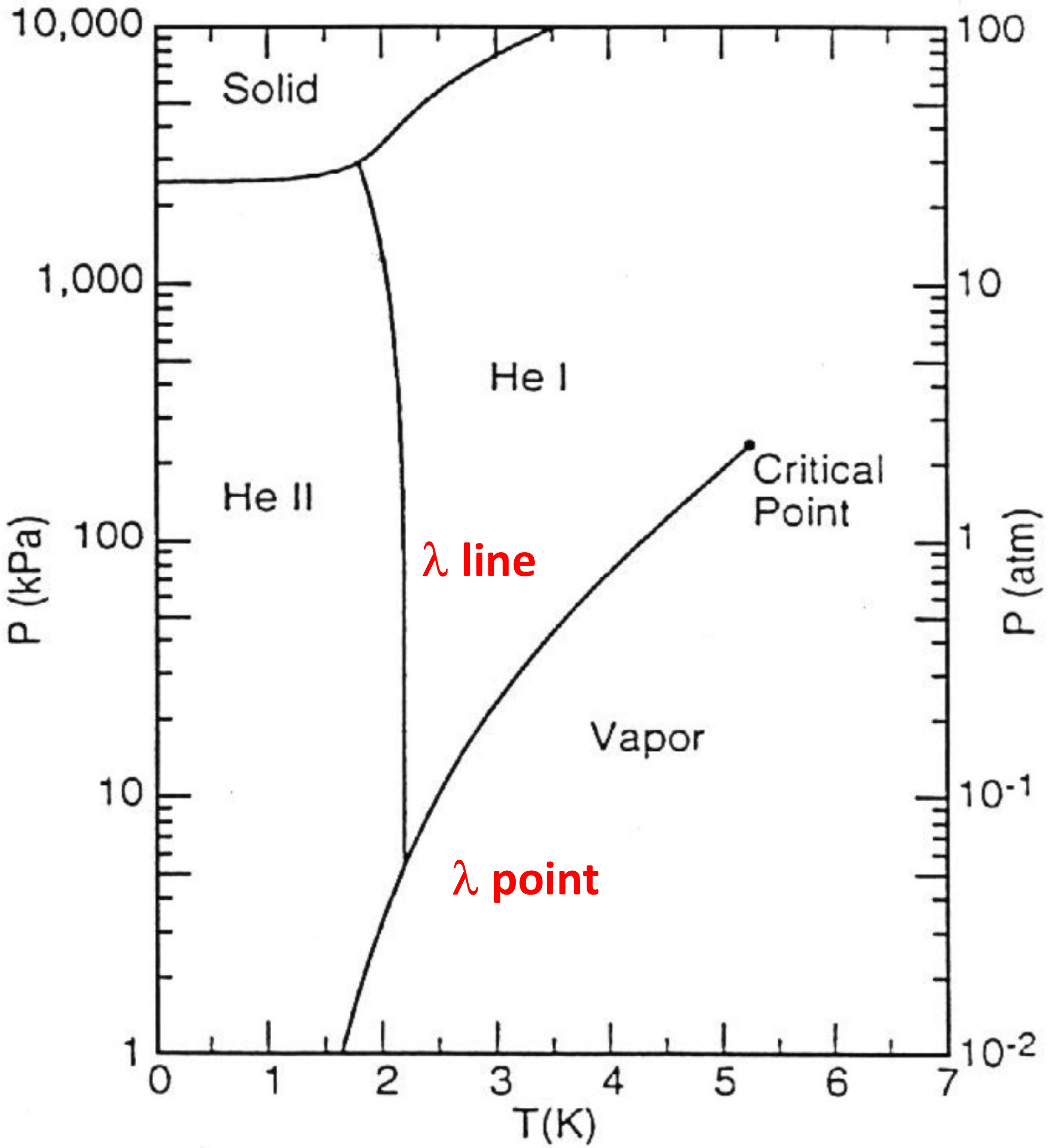
Fluid phase diagram: P - T



- P-T diagram
- Normal Boiling Point (T_B, P_B)
- Critical Point (T_C, P_C)
- Triple Point (T_T, P_T)
- Melting Line
- Saturation Line
- Transposed Critical Line

Fluid	Normal Boiling Point (K) @ 1 bar	Triple Point (K)	Critical Temperature (K)	Critical Pressure (kPa)
Oxygen	90.2	54.4	154.6	5043

P-T Diagram for Helium 4



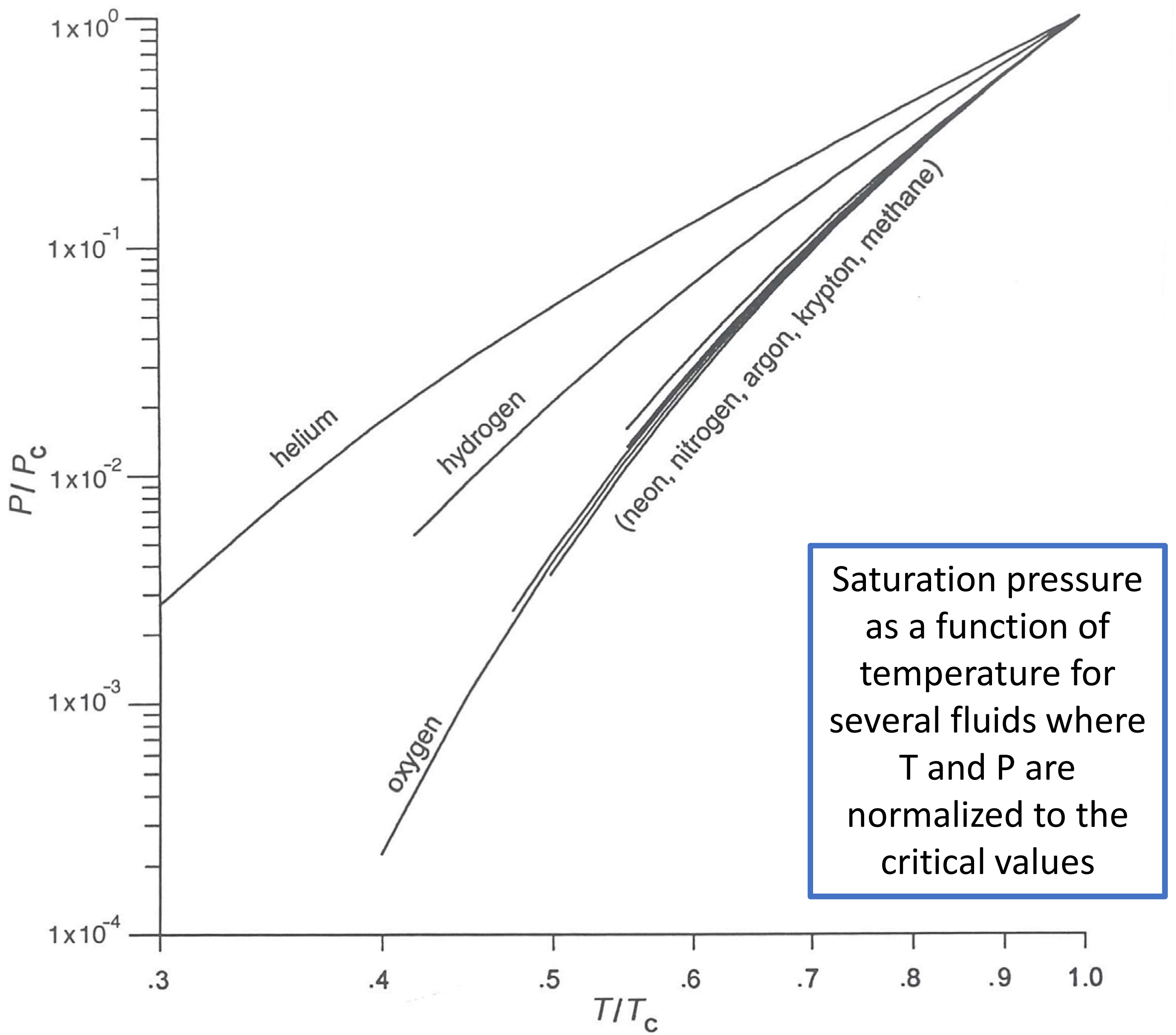
- **The Helium melting line and the saturation line do not intersect** even at absolute zero.
- No Triple point
- Lambda (λ) line and lamba point
- He II: superfluid
- Above the critical temperature Helium exhibits properties consonant with those of the other fluids

2.17 K, 50 mbar: λ point

Some Key Parameters of Cryogenic Fluids

Fluid	Normal Boiling Point (K)	Triple Point (K)	Triple Point (Kpa)	Critical Temperature (K)	Critical Pressure (kPa)	Critical Density (kg/m3)
Krypton	119.8	115.8	73.2	209.4	5496	910.8
Methane	111.6	90.7	11.7	190.6	4599	162.6
Oxygen	90.2	54.4	0.1	154.6	5043	436.1
Argon	87.3	83.8	68.9	150.9	4906	535.7
Nitrogen	77.4	63.2	12.46	126.3	3399	313.1
Neon	27.1	24.6	43.4	44.4	2703	483.2
Hydrogen	20.3	13.8	7.0	32.9	1283.8	31.6
Helium	4.2	N/A (2.17 λ point)	5048	5.2	227.5	69.6

Cryogenic fluid saturation line



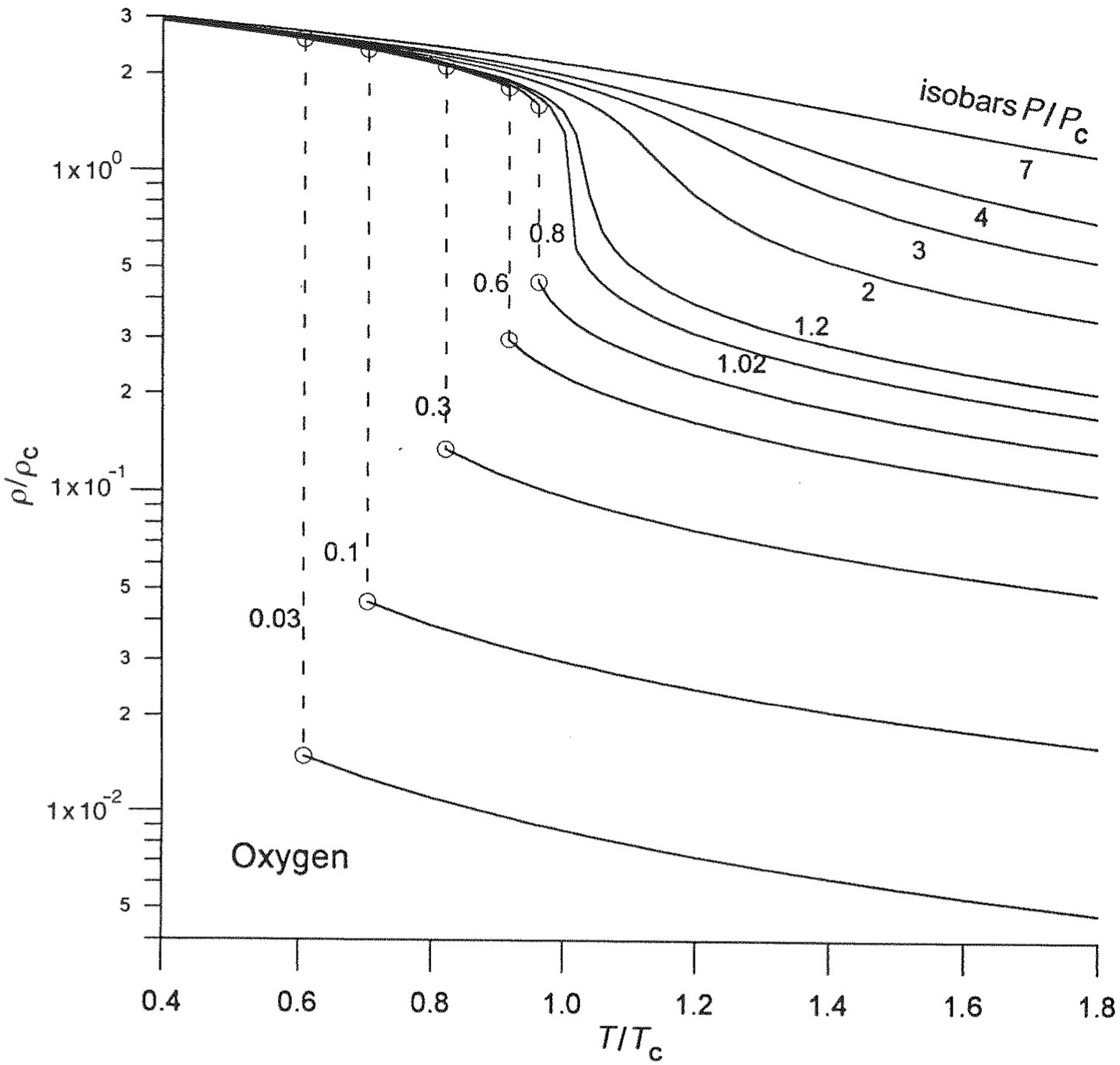
Saturation pressure as a function of temperature for several fluids where T and P are normalized to the critical values

- Saturation pressures differ by no more than few percent from each other, except for Helium and Hydrogen.
- The location of the triple points along the quasiuniversal saturation line shows no obvious correlation with nay other fluid property.
- The slope is about 7 for most fluids and 4 for He.
- The critical parameters provide the scaling by which state properties of various fluids may be compared one with another.

The Law of Corresponding States

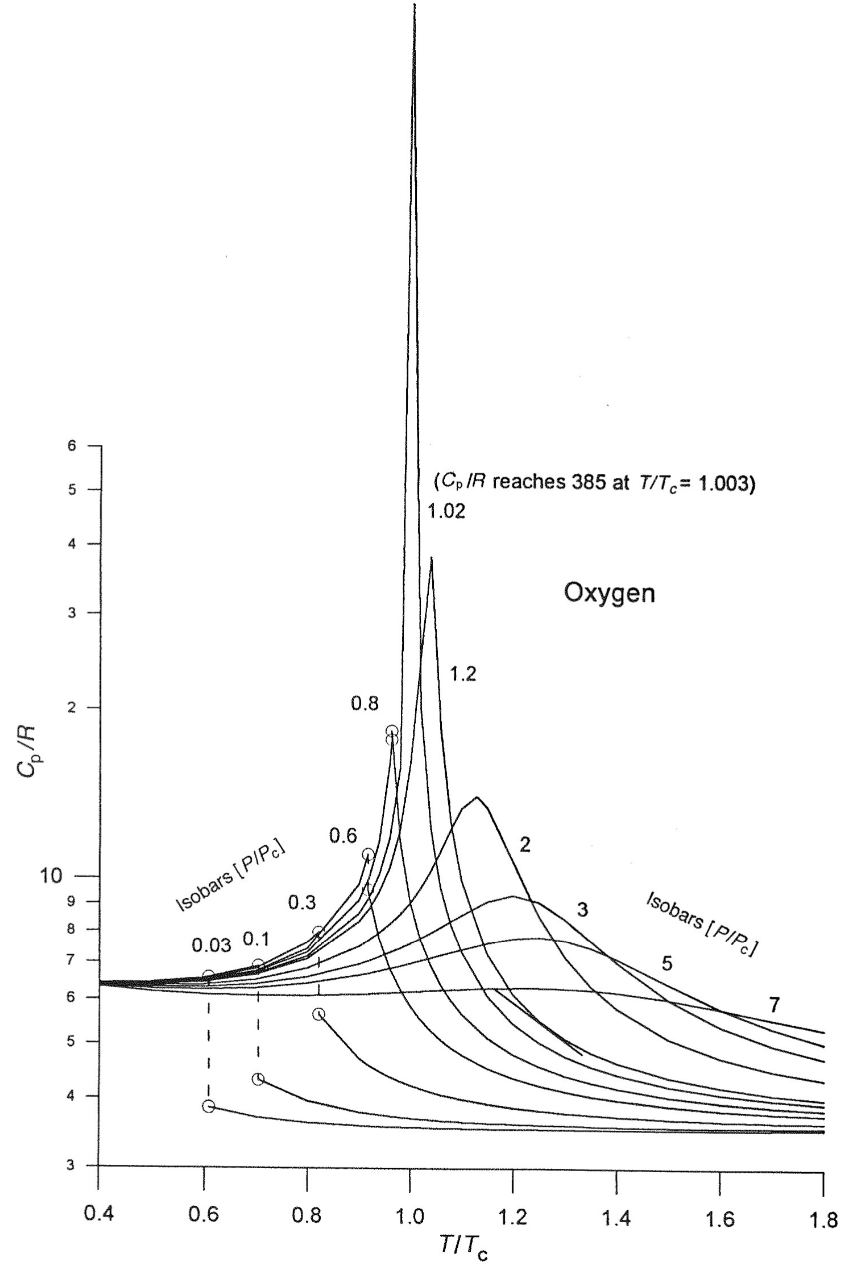
- With the exception of Helium and Hydrogen, the properties of cryogenic fluids can be scaled from one fluid to another with a fair accuracy provided the properties have been normalized by the critical properties of the fluid
- This is useful in looking at the general shape of properties

Normalized Density ρ/ρ_C as a function of T/T_C for different P

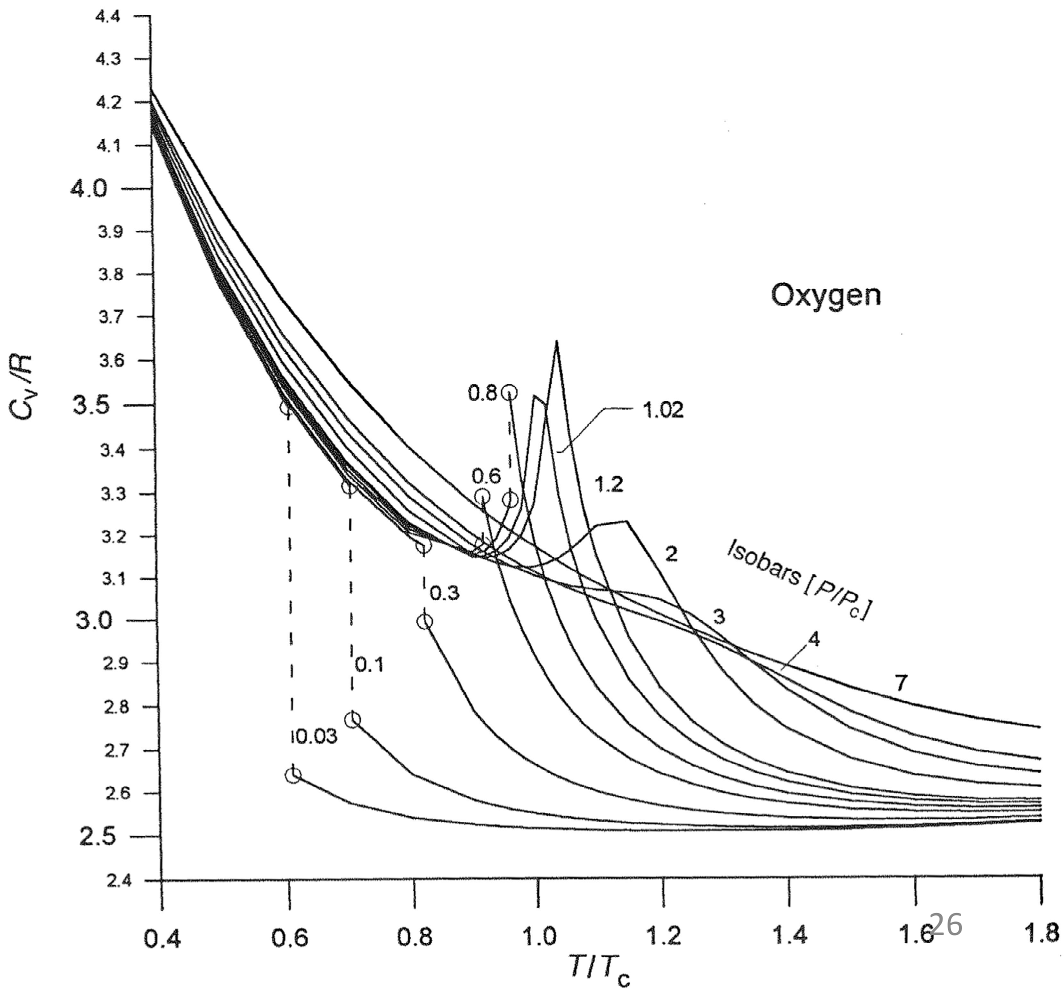


- The circles connected by dotted lines are saturated liquid and vapour states on the saturation line.
- The curve is for Oxygen.
- For the law of corresponding states we can derive values for density for the other cryogenic fluids (except He and H_2)

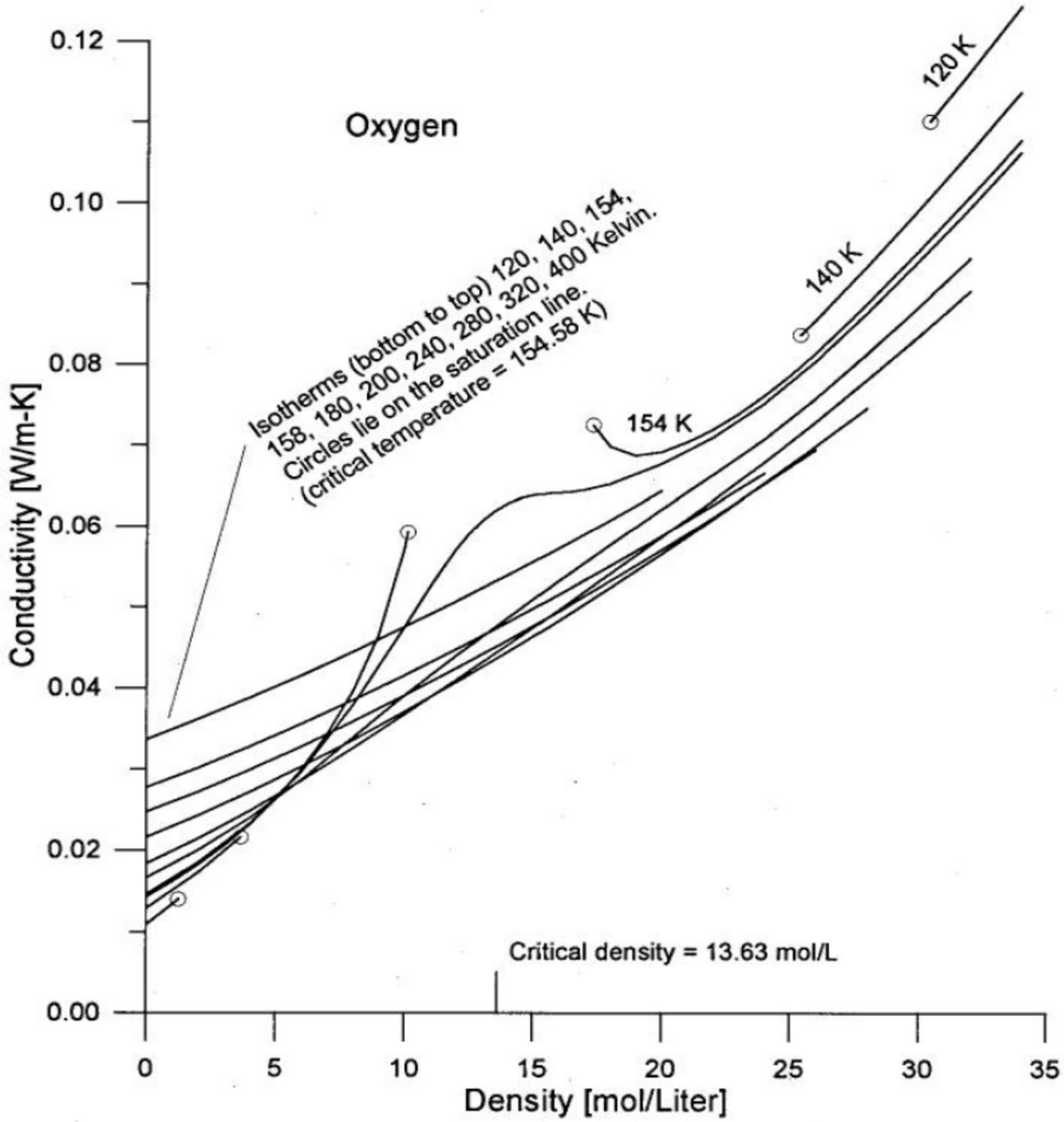
C_P/R & C_V/R as a function of T



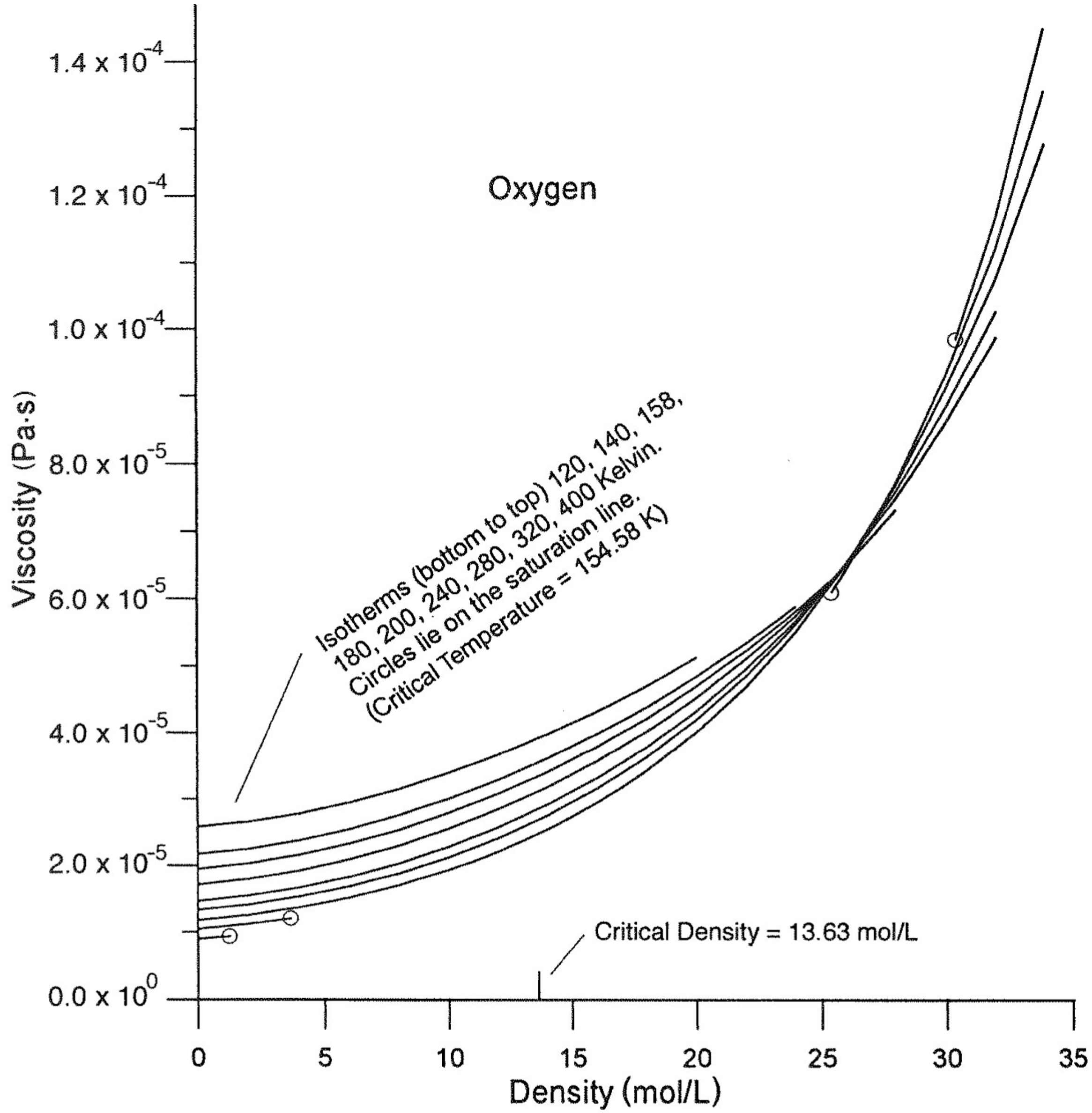
- C_P/R & C_V/R as a function of T/T_C for different P
- Dramatic difference



Thermal Conductivity as a function of density



Viscosity as a function of density



Equations of State

- Allow calculation of all thermodynamic state properties
- In theory, are based on the interactions of a molecule with its neighbors
- In reality, are highly empirical
  - A simple example is the ideal gas law:
    $A(\rho,T) = RT\ (\log \rho - a \log T + S_0)$
    a = 3/2 for a monatomic gas, 5/2 for a diatomic etc.
- Best calculated via computer codes

Fluid Property Computer Codes

- The use of computer codes to generate properties (based typically on equations of states and empirical data) is the most common way to find fluid properties today
- Examples include:
  - NIST – 12 National Institute for Standards & Technology http://www.nist.gov/srd/nist23.cfm
  - GASPAK & HEPAK from CryoData http://www.htess.com/software.htm
  - An interactive website (free) from NIST should suffice for us http://webbook.nist.gov/chemistry/fluid/

Air composition

Table III. Normal Composition of Clean, Dry, Atmospheric Air[1]

Constituent	Vol. %	Mol. Wt.[3]
Nitrogen	78.084	28.013
Oxygen	20.9476	31.999
Argon	0.934	39.948
Carbon dioxide	0.1314[2]	44.0
Neon	0.001818	20.183
Helium	0.000524	4.003
Methane	0.002[2]	16.04
Krypton	0.000114	83.80
Hydrogen	0.00005	2.016
Nitrous oxide	0.00005	44.01
Xenon	0.0000087	131.30
Sulfur dioxide	0–0.0001[2]	64.06
Nitrogen dioxide	0–0.000002[2]	46.01
Ozone Summer:	0–0.000007[2]	47.998
Winter:	0–0.000002[2]	—
Iodine	0–0.000001[2]	253.809
Ammonia	0–trace[2]	17.03
Carbon monoxide	0–trace[2]	28.01
Air	100	28.96

Nitrogen

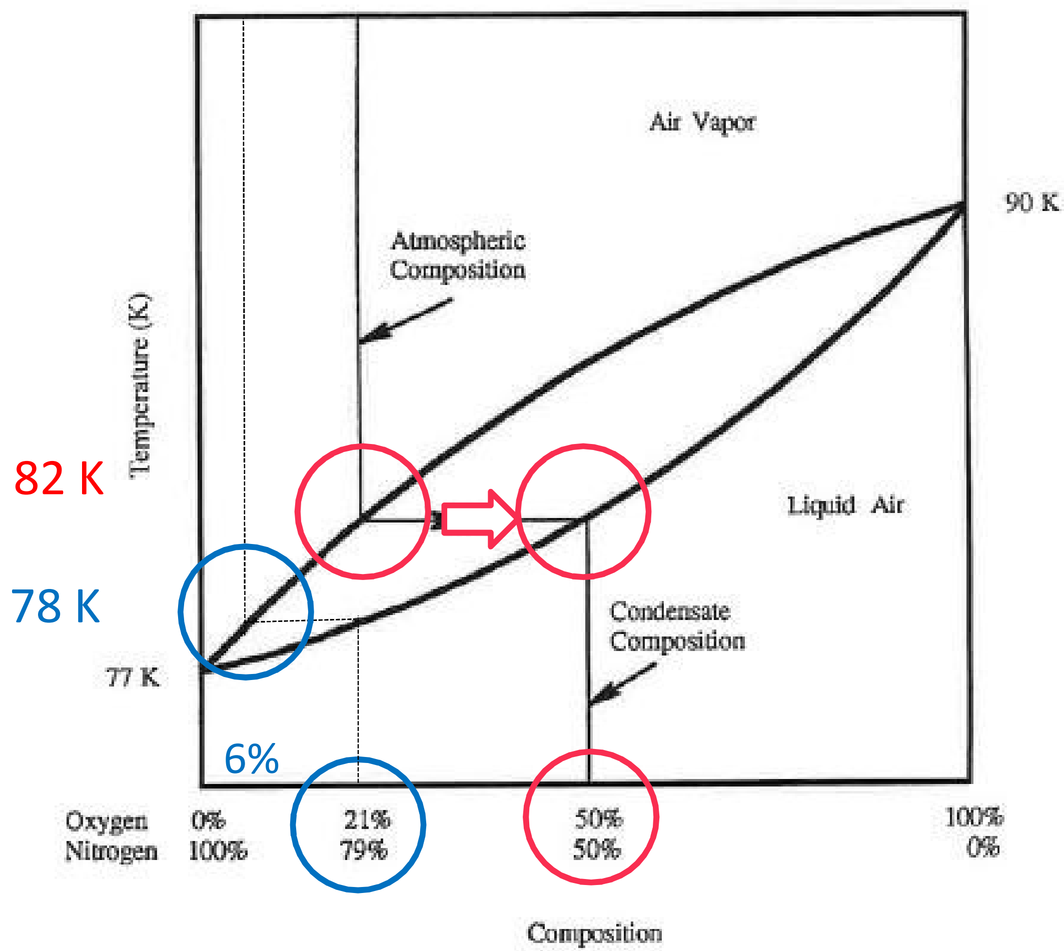
- **Boils at 77.36 K and freezes at 63.2 K.**
- Resembles water in appearance - 807 kg/m^3 (water ≈ 1000 kg/m^3).
- Exists in 2 stable isotopes - N^{14} & N^{15} in ratio of 10000 : 38.
- Heat of vaporization is 199.3kJ (water 2257kJ/kg).
- It is produced by distillation of liquid air (N_2 is 78% of air). It is a “by-product” of oxygen production (N_2 is 21% of air) and its cost is low (≈ transport cost).
- Easy handling (insulation). Cryogenic coolenat or pre-coolant (LHe).
- High temperature superconductivity.
- Nitrogen is primarily used to provide an inert atmosphere in chemical and metallurgical industries. It is widely used in a petrochemical site.
- Food preservation, beverage (pressure), blood, cells preservation, cryoterapy.
- It provides an inert atmosphere in the electronics prodiction.
- It is used in civil engeeniring (concrete cooling, soil freezing, ..).
- It is used in glass production.

Oxygen

- **Boils at 90.18 K and freezes at 54.4 K.**
- Blue in color – due to long chains of O_4.
- Has a density of 1141kg/m^3 (water – 1000 kg/m^3).
- O_2 is slightly magnetic and exists in 3 stable isotopes - O^{16}, O^{17}, O^{18} in ratio of (10000 : 4: 20).
- Not used in cryogenics.
- Safety issues: very reactive. It can react violently with common materials, such as oil and grease. Other materials may catch fire spontaneously. Nearly all materials, including textiles, rubber and even metals, will burn vigorously in oxygen.
- Because of the unique properties of oxygen, there is no substitute for oxygen in any of its uses – widely used in industries and for medical purpose:
  - Food preservation and packaging, breathing systems, iperbaric chambers, oxygenation systems, O_2 masks, …
- It is largely used in iron and steel manufacturing industry.
- Oxidizer propellant for spacecraft rocket applications.

Properties of the mixtures

- Mixtures properties:
  - Condensation/boiling is not an isothermal process
  - Both temerature and composition of the mixture change as the condensation/boiling proceeds
  - The components of the mixtures are partially separated during the condensation process
  - By repeating partial condensation a large number of times, almost complete separation of components can be achieved.
  - This is the basis of **fractional distillation**.



Air

- It has a boiling point of 78.9 K and 874 kg/m^3 as density (water density ≈ 1000 kg/m^3).

- For practical purpose, it is considered as a mixture of 78% N_2 + 21% O_2 + 1% Ar + others.

- Liquid air was earlier used as precoolant for low temperature applications.

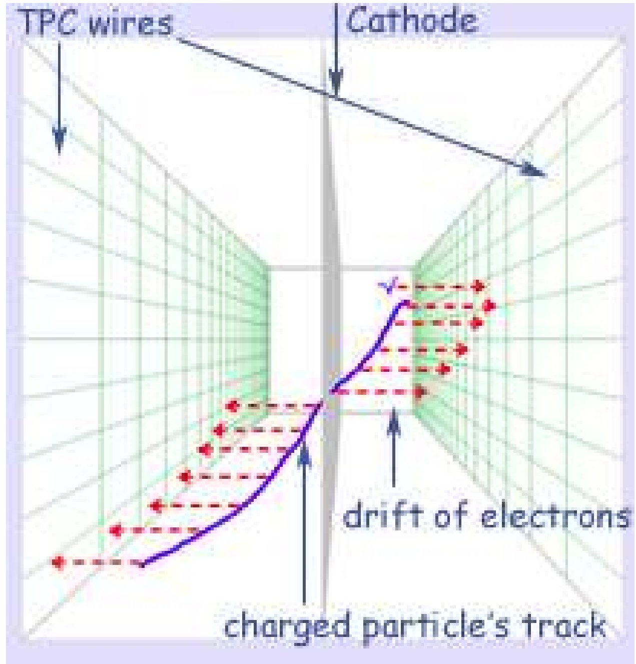
- Liquid air is primarily used in production of pure nitrogen, oxygen, and rare gases.

Argon

- It boils at 87.3 K and freezes at 83.8 K.
- It has a density of 1394 kg/m^3 (water ≈ 1000 kg/m^3).
- It is a colorless, inert and non toxic gas.
- Exists in 3 stable isotopes – Ar^{35}, Ar^{38}, Ar^{40} and in a ratio of (338 : 63 : 100000).
- It is widely used as detector media in high energy physics.
- It offers inert atmosphere for welding stainless steel, aluminum, titanium etc.
- It is widely used in semiconductors and electronics production.
- The property of inertness of argon is used to purge moulds in casting industry.
- It is used in Argon-oxygen decarburization (AOD) process in stainless steel industry.

Why LAr for TPC?

- Noble gas in liquid phase: dense (1.4 kg/l), chemical inert, homogeneous
- Available (Atmospheric Argon ≈ 1% of air) and relatively cheap to realize large masses, if we are not worried about cosmogenic ^{39}Ar contamination (Uderground Argon)
- Relatively easy to be insulated with "simple" solutions, it may be cooled by (pressurized) LN_2
- High ionization yield (important both for TPC single phase for ionization electrons and dual phase for secondary scintillation)
- Efficient scintillator (unfortunately in VUV range: 128 nm), self transparent
    - Fast and slow component
    - Light attenuation lenght, Rayleigh scattering
- Powerful Pulse Shape Discrimination (PSD) in the scintillation signal (S1 e S2)
- Need to be purified:
    - Long "electron lifetime" necessary for long drift distance (O_2, H_2O)
      $Q_{coll} = Q_0 \exp[-(T-T_0)/\tau_{el}]$, τ_{el} (ms) = 0.3 /N (ppb)   free electron lifetime
    - No scintillation quenching
        - $O_2 \leq 0.5$ ppm
        - $N_2 \leq$ few ppm (nitrogen is not removed by standard filters)
- Commercial techniques available to purify argon (for H_2O: Hydrosorb, for O2 now Cu, Oxysorb before)
- Similar charachteristics for LKr and Lxe.



ICARUS T600 @ LNGS
τ ≈ 15 ms
with 1.5 m gap, 500 V/cm

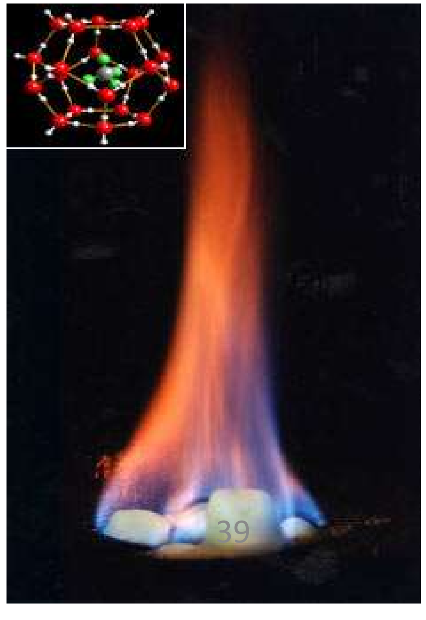
Corresponding to
15% attenuation
3.6 m gap @0.5kV/cm

Neon

- Boiling point at 27.1 K, triple point 24.6 K.
- It is a clear, colorless liquid, non-magnetic.
- It is compact, inert and less expensive as compared to liquid helium.
- Rare (1.8 10^{-5} in the air).
- Neon could be used as refrigerant for intermediate temperature and High Temperature superconducrivity.
- Neon is commonly used in neon advertising
- Liquid neon is commercially used as cryogenic refrigerant
- It is a contaminant in helium processes.
- It is used in some types of lasers.

Methane

- It boils at 111.7 K (NBP), triple point at 88.7 K.
- Naturally occurring methane is found both below ground and under the seafloor, and is formed by both geological and biological processes.
- The largest reservoir of methane is under the seafloor in the form of methane clathrates ("methane ice").
- CH_4 is the main constituent of natural gas. The relative abundance of methane on Earth makes it an attractive fuel In the form of Liquid (LNG) or Compressed Natural gas (CNG).
- "Green" fuel
- Flammable
  - Flammability limits CH_4: 5.3-15.0 (vol %)
  - Detonability limits CH_4: 6.3-13.5 (vol %)
  - Minimum ingnition energy: 0.29 mJ



Krypton

- NBP 119.9 K, $T_{melting}$ 115.8 K, TP 114.9 K
- Very very rare: 1.1 10^{-6} in air
- Noble gas in liquid form.
- Very dense ≈ 2.4 kg/l at NBP.
- Six stable isotopes.
- Used as detector media.

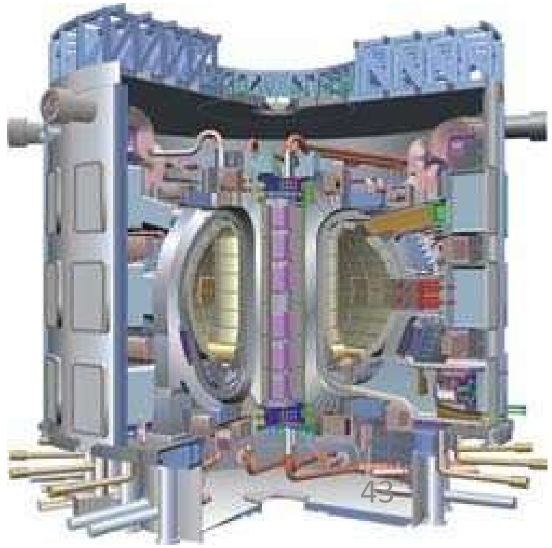
Xenon

- NBP 165.1 K, $T_{melting}$ 161.3 K, TP 161.4 K
- Very very rare
- Noble gas in liquid form.
- Very dense≈ 3 kg/l at NBP.
- Used as detector media.

Special Cases: H_2 and He

Hydrogen

- Diatomic Molecule (H_2). 5.5 10^{-7} in air.
- Lighter fluid: 70.79 kg/m^3 @ NBP (20.27 K), TP13.8 K (normal H_2)
- 3 Isotopes:
  - Ordinary hydrogen: 1 proton in nucleus
  - Deuterium (1/1600 natural H_2): 1 p and 1 n in nucleus
  - Tritium (^3H): 1 p and 2 n in nucleus, radioactive (half life 12.26 y)
- Natural forms (H2, HD, D2); HD (Hydrogen Deuteride) much more abundant than D_2.
- Applications for cryogenic hydrogen
  - Rocket fuel (LH_2+ LO_2)
  - Transportation fuel (vehicle fuel systems + GO_2)
  - Targets/detectors in high energy physics experiments
  - Semiconductor processing (high purity H_2 gas), optical fibers
  - Nuclear fusion reaction processes (D_2 and T_2)
    - ITER Project

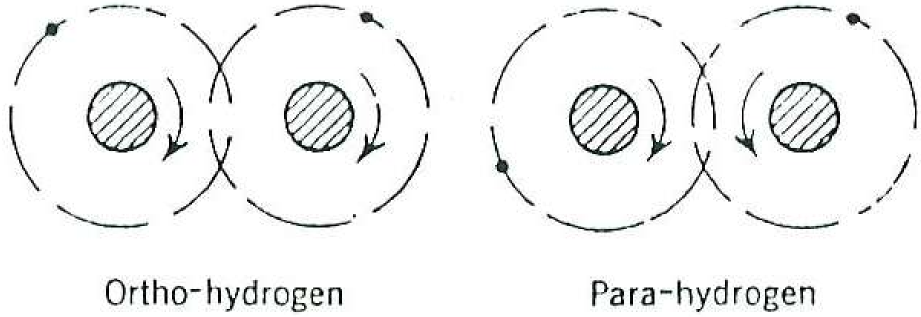


ITER

Hydrogen

- **Exists in two molecular states depending on nuclear spins**:

  orthohydrogen – nuclear spins parallel (spin1)
  parahydrogen – nuclear spins antiparallel (spin 0)



- At 300 K: Hydrogen 75% ortho and 25 % para
- At NBP = 20.4 K equilibrium concentration is ≈99.8% para and ≈0.2% ortho
- Parahydrogen is the lowest energy state and equilibrium depends on temperature
- Conversion from ortho to para is slow and exothermic
- H_2 liquefiers typically include a catalyst (e.g. nickel silicate) to speed up conversion
- Thermodynamic properties of ortho and para hydrogen are significantly different
- Deuterium has also O-P conversion, but 2/3 ortho and 1/3 para, but in this case para gets converted to orto.

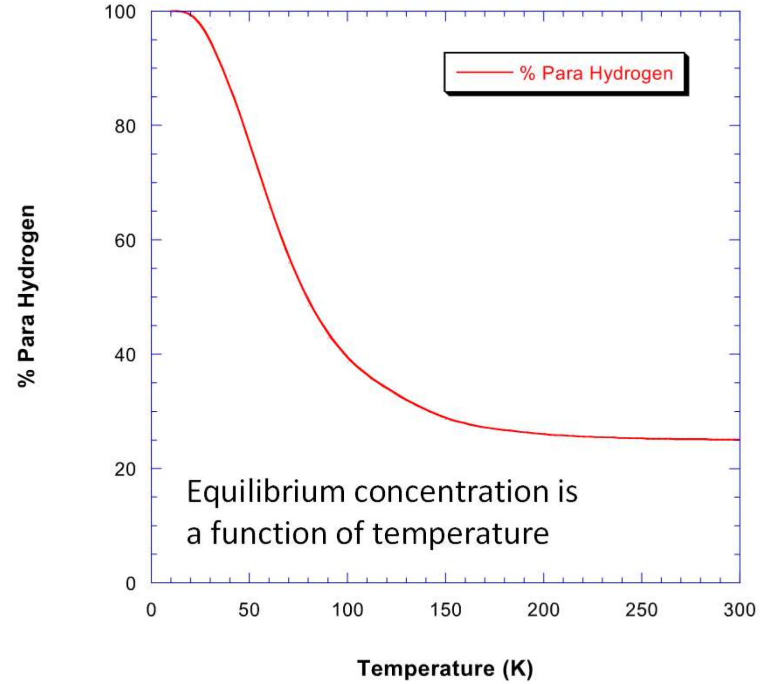
Ortho-Para Conversion of H_2

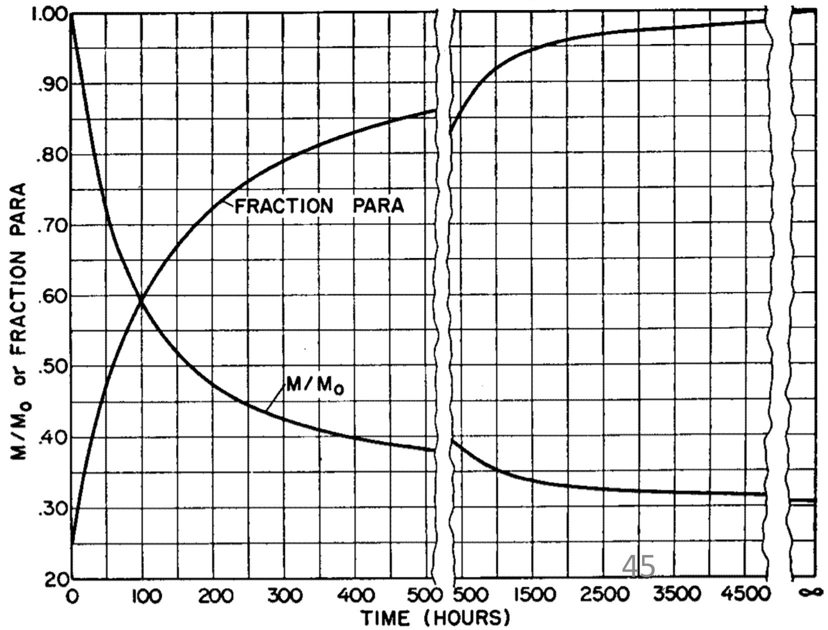
- Ortho-para conversion is an exothermic process
  - $\Delta E_{op}(20K) \approx 700$ kJ/kg
  - >> compared to heat of vaporization ≈ 445 kJ/kg
- Equilibrium concentration is a function of temperature
  - 25% Para @ 300 K
  - ~ 50% Para @ 80 K
  - 99+% @ 20 K
- For long term storage of LH_2, need to complete O-P conversion
  - The fraction loss is due to heating and vaporizing the liquid
  - See plot: M_0 storage mass at t=0
  - Reaction rate (X_{orto} fraction, C_2 = 0.0114h-1)

$$\frac{dx_o}{dt} = -C_2 x_o^2$$

- Use of catalizer (chemical reaction): $$\frac{dx_{orto}}{dt} = -C_1 x_{orto}$$
  - Ni powder, Ru/Al_2O_3, $Fe(OH)_3$
  - Partial conversion process at 80 K

C_1 rate constant depending on catalyst





Hydrogen

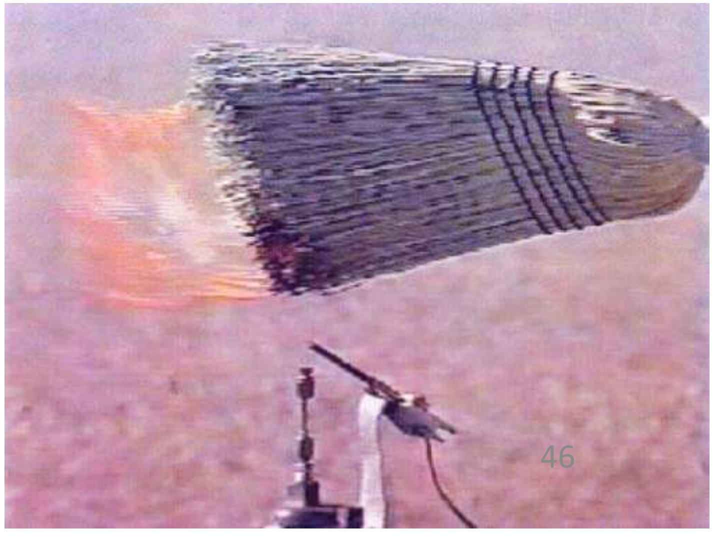
- Easily oxidized to produce energy

$$H_2 + O_2 \text{ -> } H_2O + \text{energy (286 kJ/mol)}$$

Safety issues

- Colorless, odorless
- Highly flammable
- Hydrogen gas forms explosive mixtures with air in concentrations from 4–75% and with chlorine at 5–95%. The explosive reactions may be triggered by spark, heat, or sunlight
  - Flammability limits H_2: 4.0-75.0 (vol %)
  - Detonability limits H_2: 18.3-59.0 (vol %)
  - Minimum ingnition energy: 0.02 mJ
- The hydrogen autoignition temperature, the temperature of spontaneous ignition in air, is 500 °C
- Pure hydrogen-oxygen flames emit ultraviolet light and with high oxygen mix are nearly invisible to the naked eye.

LZ 129 *Hindenburg (1937)*
V = 211.890 m³



Helium

- Inert, colorless, spherical molecule is the closest approximation to an ideal gas.
- Helium is about 5 ppm in the atmosphere
- Obtained in separation process in natural gas (~ 0.2% concentration)
- Two stable isotopes: He^4 and He^3 (which is 0.1 ppm of natural helium):
    - He^4 (He-4): 2 p and 2 n in the nucleus
    - He^3 (He-3): 2 p and 1 n in the nucleus
- Quantum effects are important at low temperatures (T < 20 K)
    - He^4 obeys Bose-Einstein statistics (nuclear spin = 0)
    - He^3 obeys Fermi-Dirac statistics (nuclear spin = ½)
- Approximate forms of the equation of state
    - Van der Waal's gas model not suitable at low temperature due to quantum effects
    - Virial expansion including quantum phenomena
- Tabulated properties for He 4 (Refprop®or HEPAK®)
- Helium is not well described by the "Law of Corresponding States" because at low temperature it is not a classical fluid
- In the following "Helium" is "Helium 4"

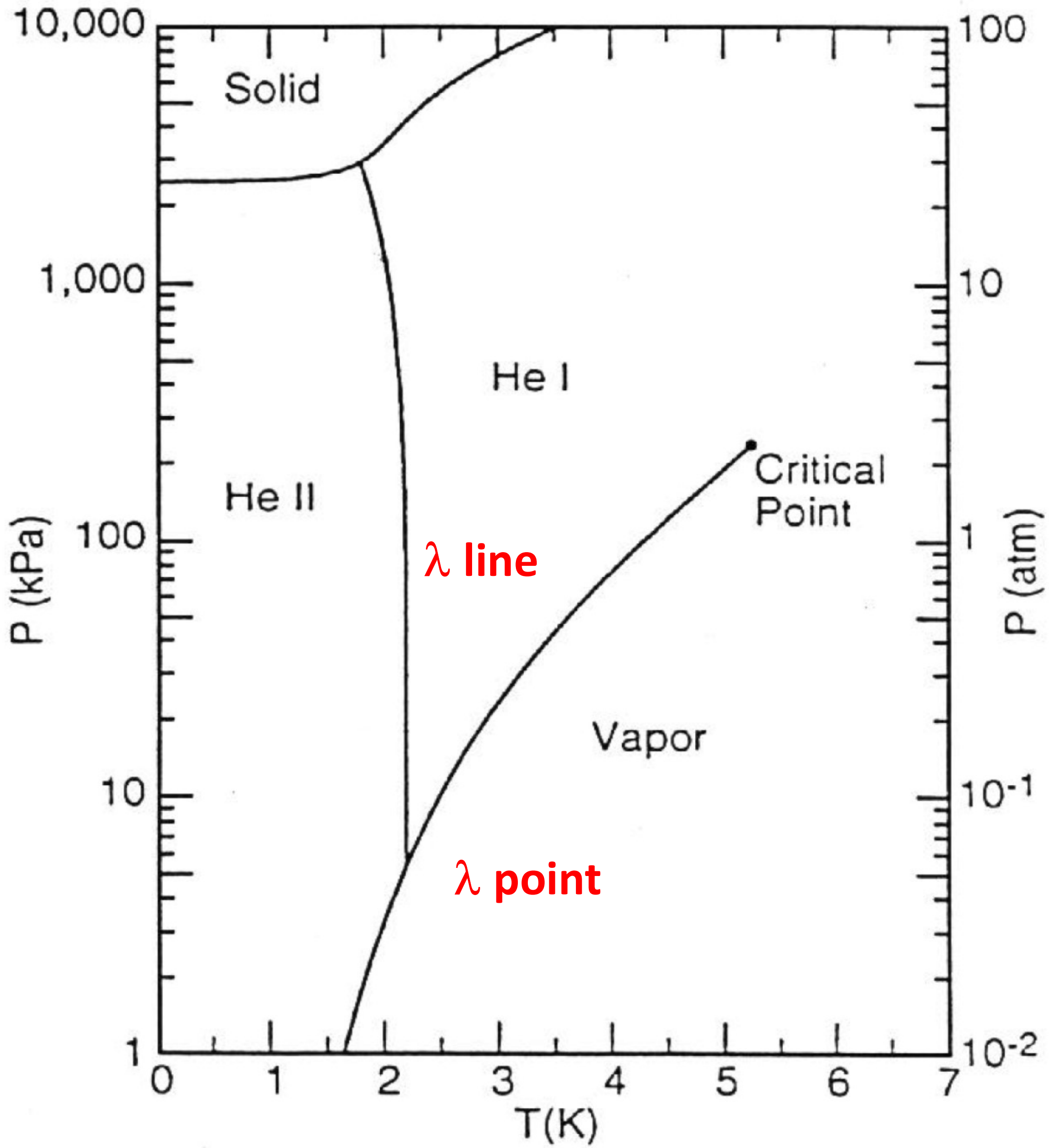
Helium use

- Widely used in cryogenics, as coolant and for low temperature superconductivitY: ≈ 1/3 helium use.
- It is also used as cooling fluid for the MRI, NMR or EPR magnets under liquid state.
- Space: liquid helium subsystem allows pressurization of the oxygen tank of Ariane 5 launch vehicle.
- Automotive: Helium is used to inflate car airbags.
- Laser welding and arc welding.
- Leaks detectors
- Electronic components: Helium allows temperature and uniformity control during etching and anneal process. It is also used as carrier gas for some Chemical Vapor Deposition process using liquid precursors.
- Helium is used to assist oxygen flow in case of respiratory obstruction. It is a component of inhaled gaseous mixtures such as for pulmonary function test. Medical device: helium is used during cryoablation procedures.
- Laboratories & Research Centers: carrier gas in gas chromatography.
- Helium is used in diving and IMR (Inspection, Maintenance & Repair) work achieved subsea for oil & gas offshore operations.
- Helium is used to inflate balloons for parties
- It is used in helium-neon lasers and in mixtures for carbon dioxide lasers.
- Helium allows to exclude air from fabrication processes (blanket gas).
- It is also used to transfer heat in some processes.

Helium 4 properties

- **Liquid Helium exhibits quantum properties**
- Requires very very high pressure for solidification at NBP
  - The zero point energy associated with the Heisenberg Uncertainty Principal ($\Delta P\ \Delta X \sim h$) for helium at room pressures is greater than the energy required to melt helium. Thus, it won't solidify.
  - The fact that Helium remains a liquid all the way down to 0 K has significant technological advantages
- Helium has a second liquid phase (He II)
  - This come about as a result of some of the atoms condensing into the lowest ground state (very similar to Bose-Einstein Condensation)
  - Is a second order phase transition: thus no latent heat is required
  - He II (called "superfluid" helium) has many unique & useful properties

P-T Diagram for Helium



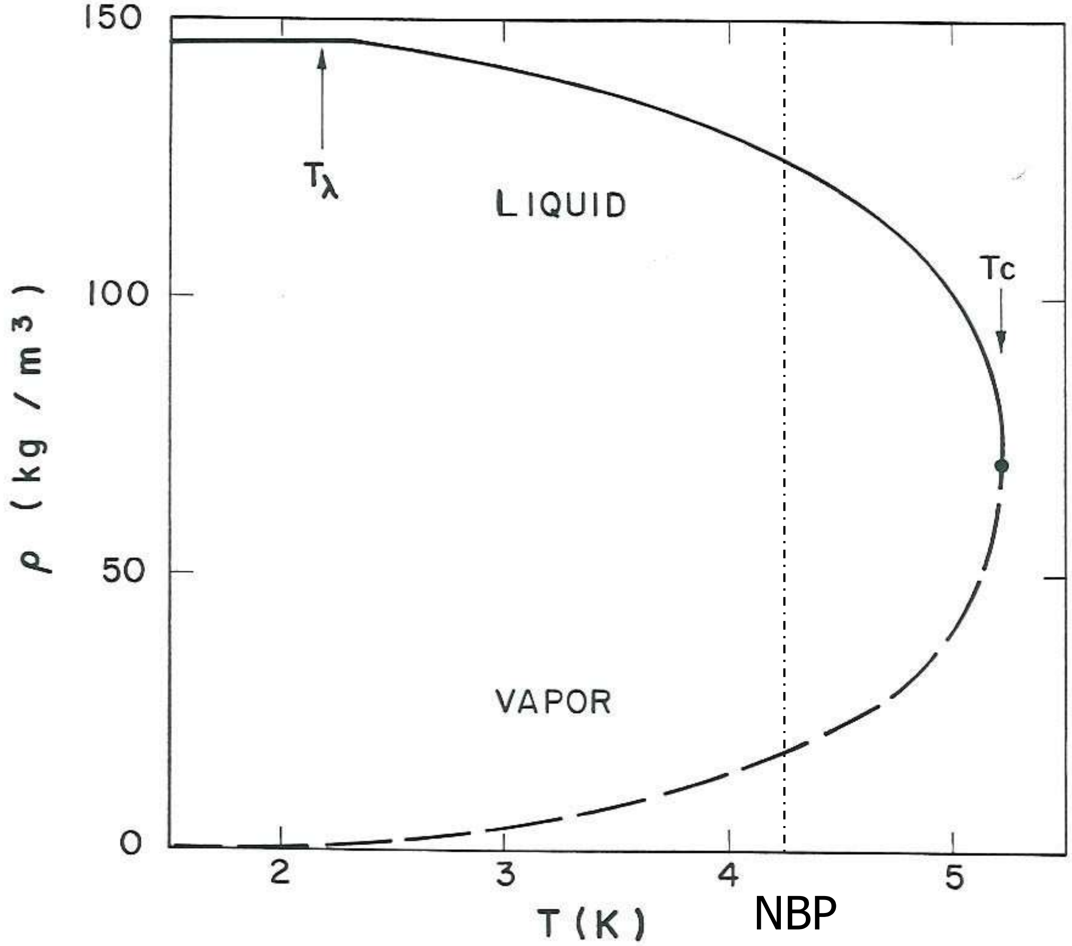
- P-T diagram for He4
- No triple point

- **T_λ = 2.176 K, 50 mbar**

- **Two liquid phases**
  - **He I: normal fluid**
    - $T_\lambda < T < T_c$, P_C = 0.226 Mpa
  - **He II: quantum fluid**
    - $T < T_\lambda$
    - T_λ (solid line, 3 MPa) = 1.76 K

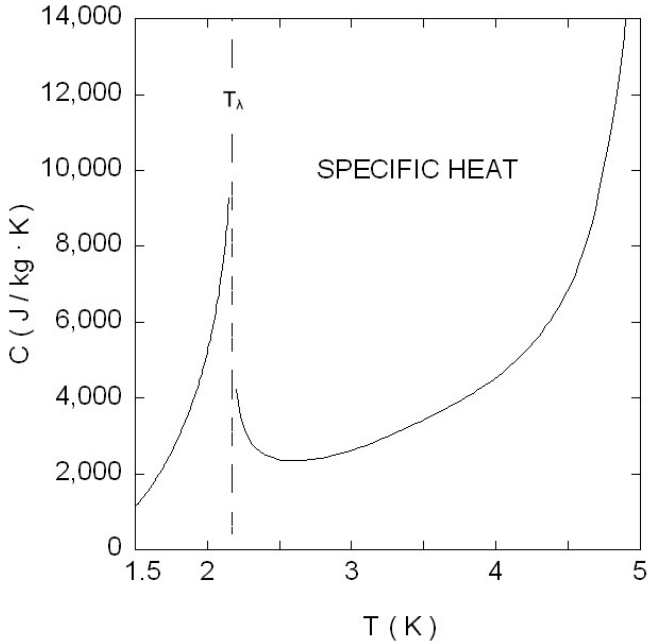
He II (called "**superfluid**" helium) has many unique & useful properties

Density of Liquid Helium



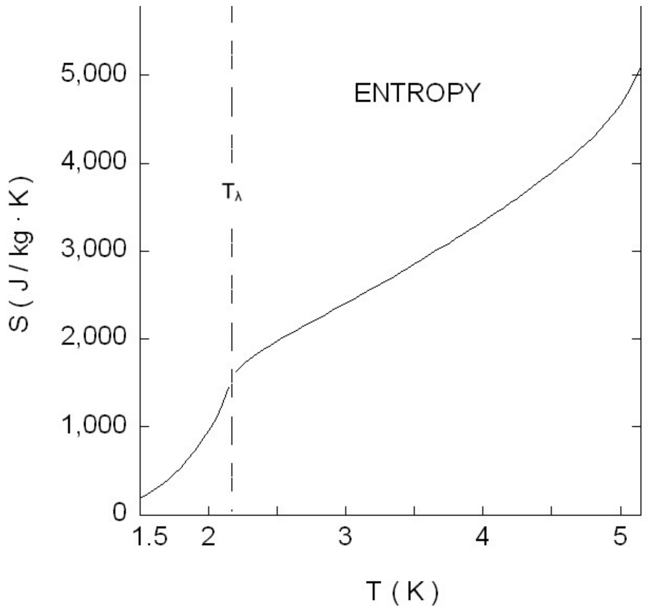
- Density (ρ)
  - $\rho_{critical} = 70\,kg/m^3$
  - $\rho_{NBP,\ 4.2\ K} = 125\,kg/m^3$
  - $\rho_{max,\ 2.2K} = 146\,kg/m^3$
  - ρ ~ constant (T < 2.2 K)
- Co-existing vapor density
  - The density of helium vapor is high compared to co-existing vapor for other fluids.
  - $\rho_{vapor}(4.2\ K) \sim 15\ kg/m^3$

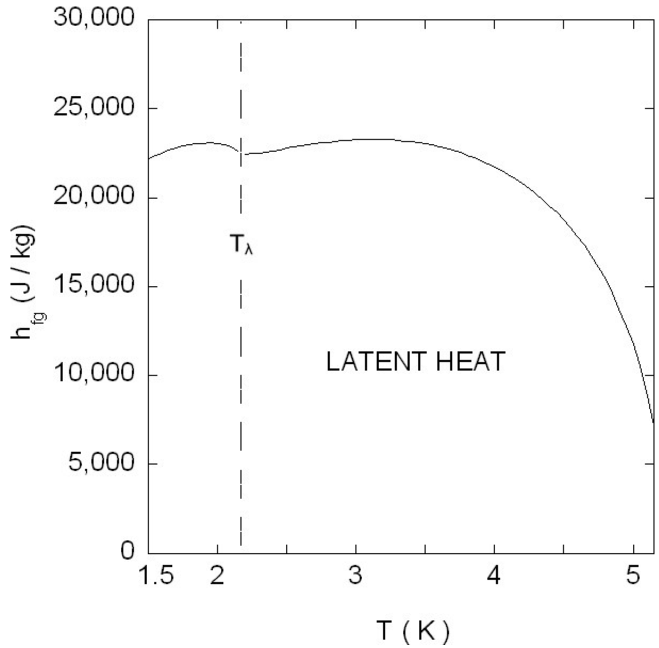
Thermal Properties of Helium



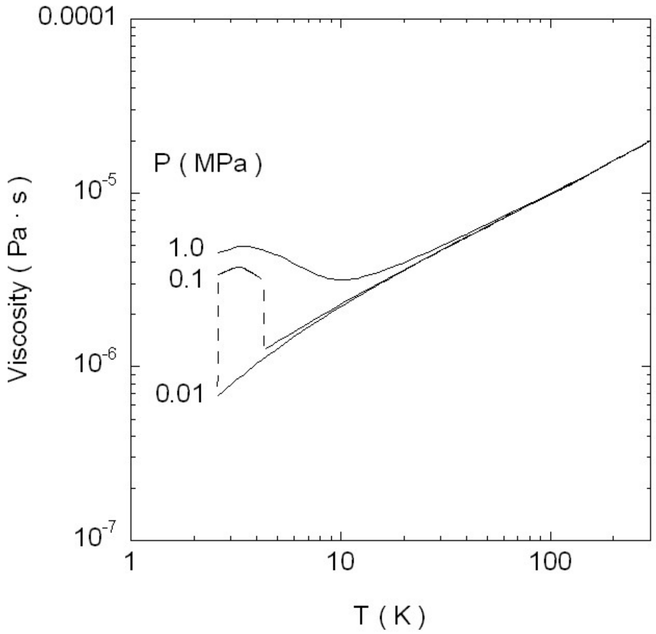
- There is no energy involved in lambda transition.
- Specific heat is infinite at λ point and it is called as 2° Order Transition.

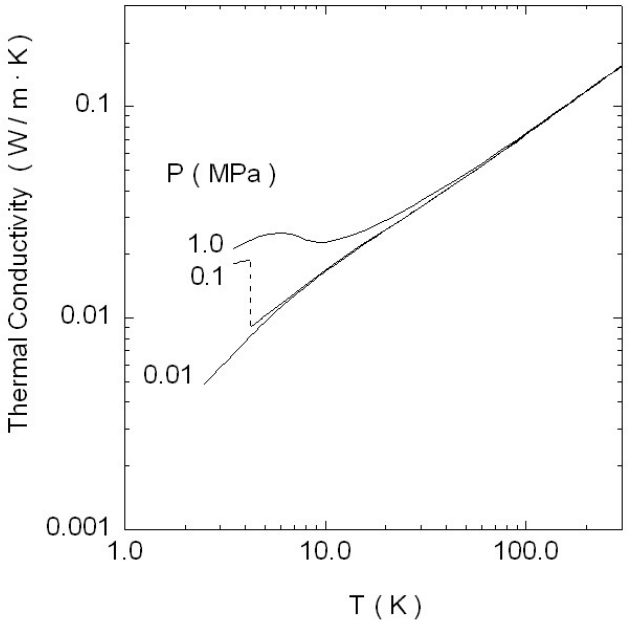
  - Specific heat
  - Entropy
  - Heat of Vaporization (h_{fg})



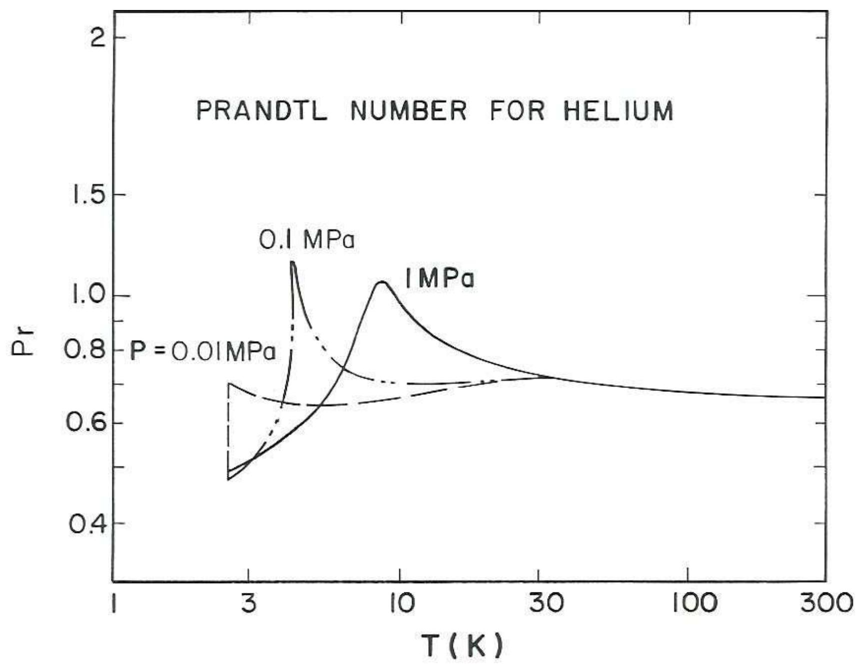


Transport properties of normal Helium





- Viscosity, (μ)
- Thermal conductivity (k)
- Prandtl number, $Pr = \mu C_p/k$



- Transport properties much different in He II regime

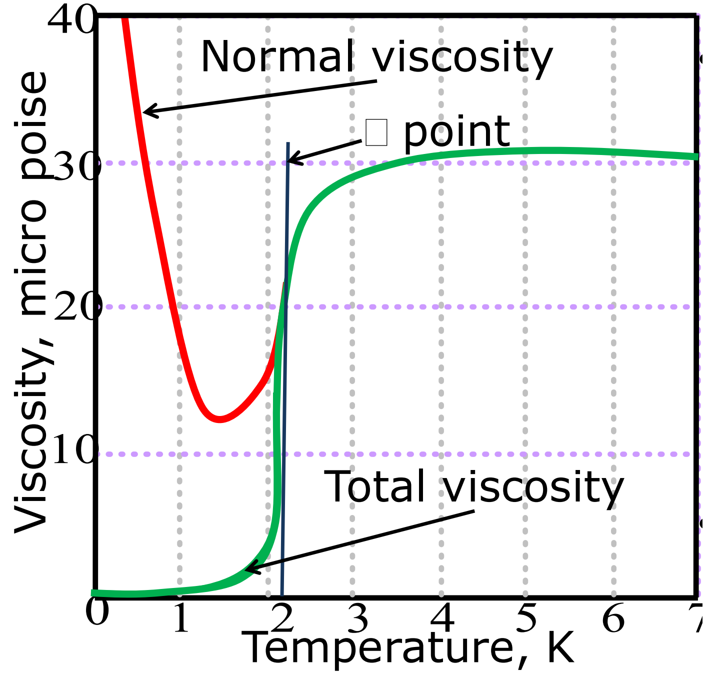
Unique properties of superfluid Helium (He II)

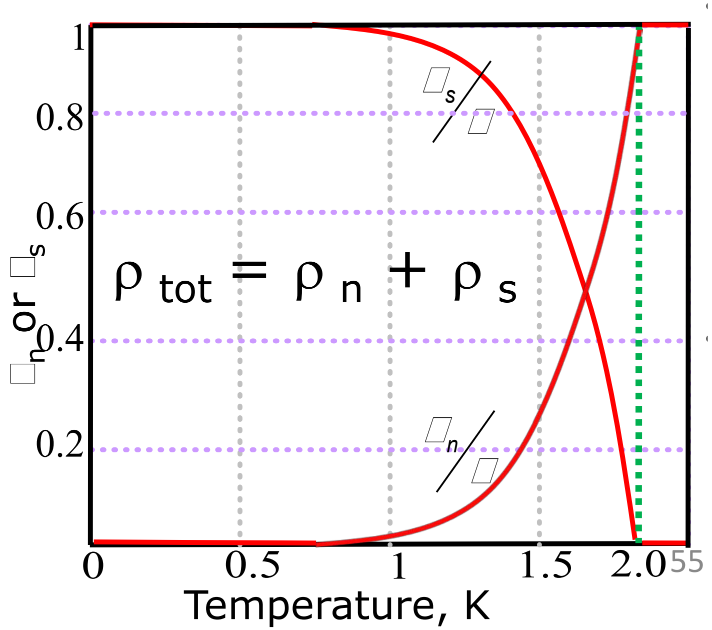
Unique behavior in the transport peoperties:

- Viscosity
- Heat conductivity
- Sound propagation
- Film flow
- Fountain effect

Viscosity of He II

- Kapitza et al. stated that viscosity for flow through thin channels is independent of pressure drop and is only a function of temperature.
- To explain this anomaly, a two fluid model is used.

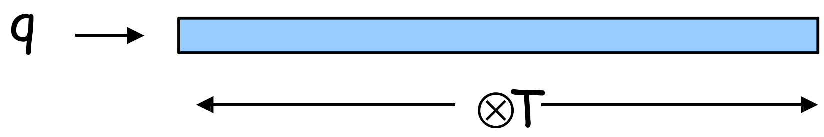


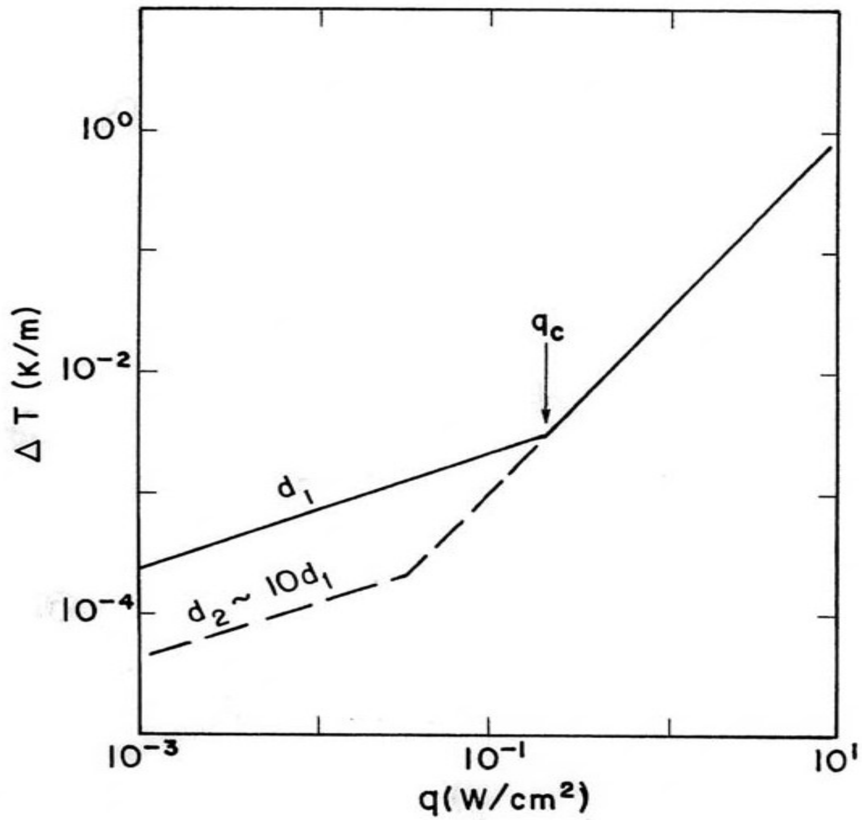


Heat Conductivity of He II

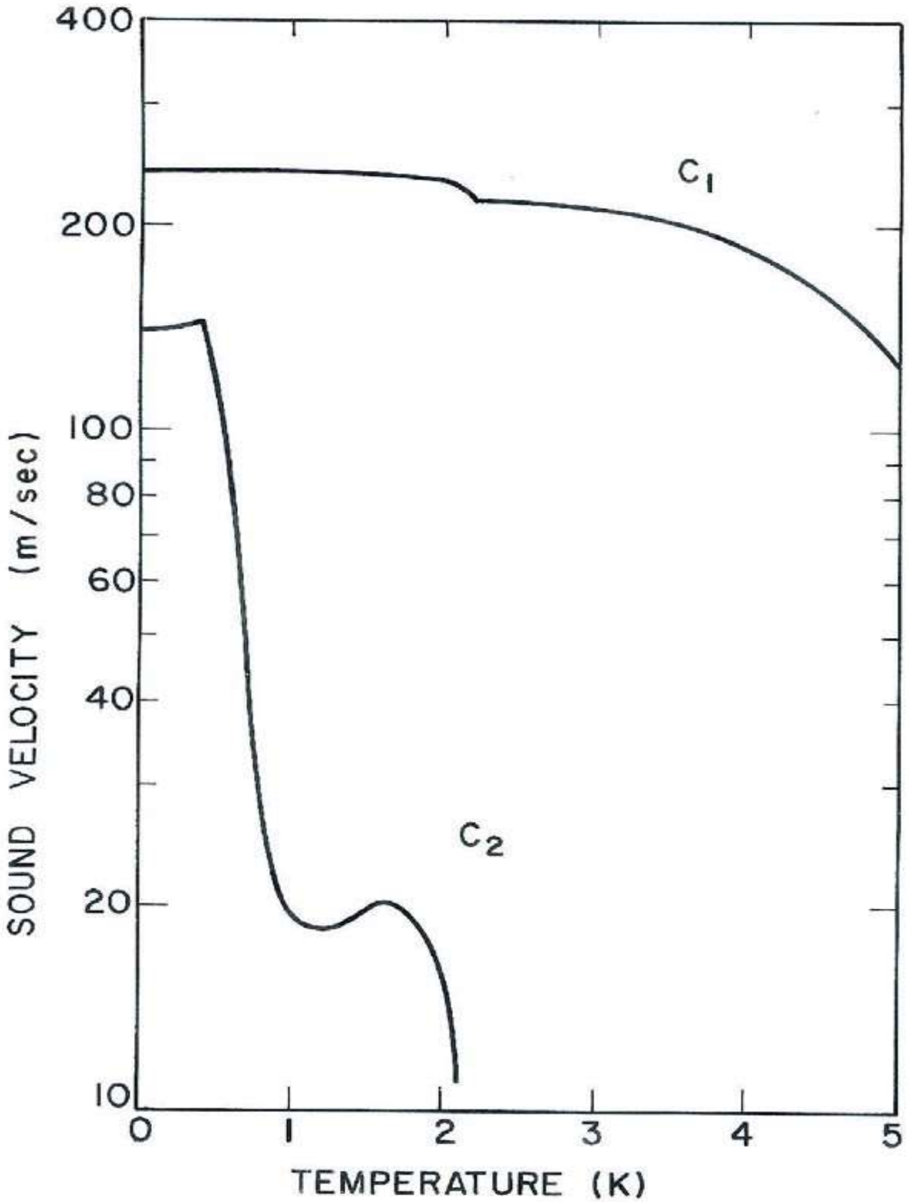
- Anomalous heat transport
  - Effective heat conductivity comparable to that of high purity metals
  - Low flux regime dT/dx ~ q
  - High flux regime dT/dx ~ q^3
  - Transition between two regimes depends on diameter of channel

➢ Heat transport in He II can be understood in terms of the motion of two interpenetrating fluids. This "Two Fluid" model effectively describes the transport properties

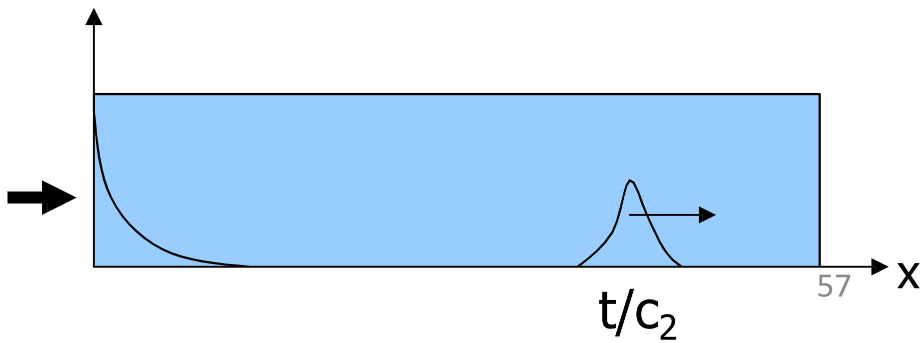




Sound Propagation in He II



- First sound (ordinary sound) propagates in liquid helium ~ 200 m/s
- Second sound (thermal wave) propagates ~ 20 m/s
- Unique quantum mechanism
- Sound propagation associated with variations in the two fluid components



He II Film Flow

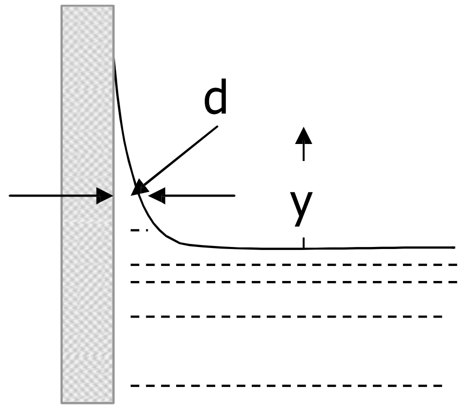
Rollin film (1937):

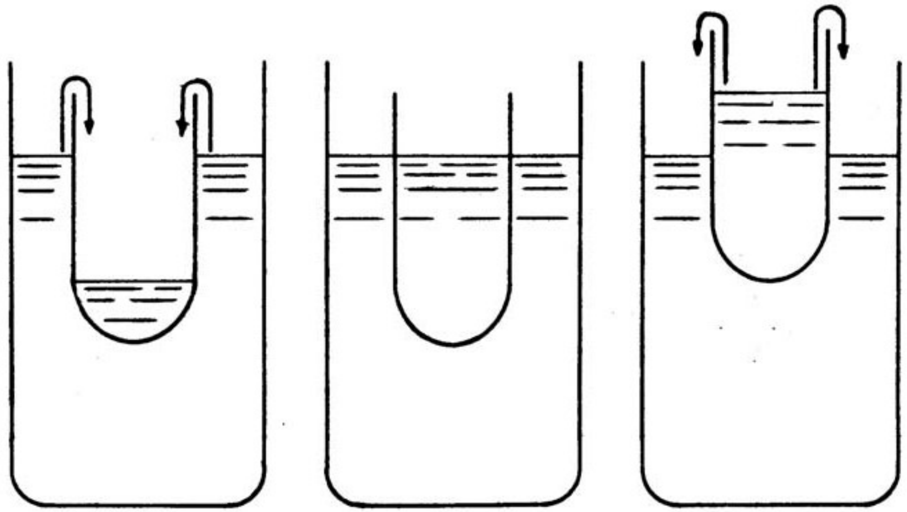
- Saturated film exists on all surfaces above the liquid
- Unique aspect of He II is that the film is mobile (flows)
- Siphon driven by hydrostatic head difference

Film thickness

$$d \sim \frac{K}{y^n}$$

$n \sim 0.4$
$K \sim 3 \times 10^{-6}\ \mathrm{cm}^{0.6}$



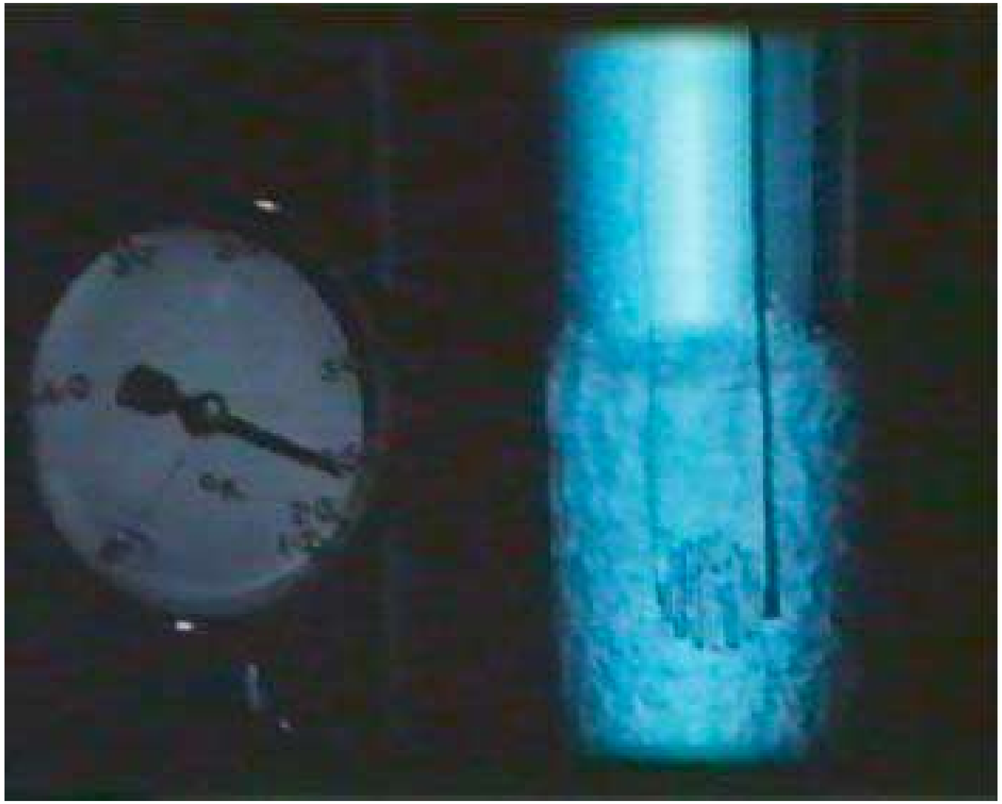


➢ Two containers with different levels will tend to equalize

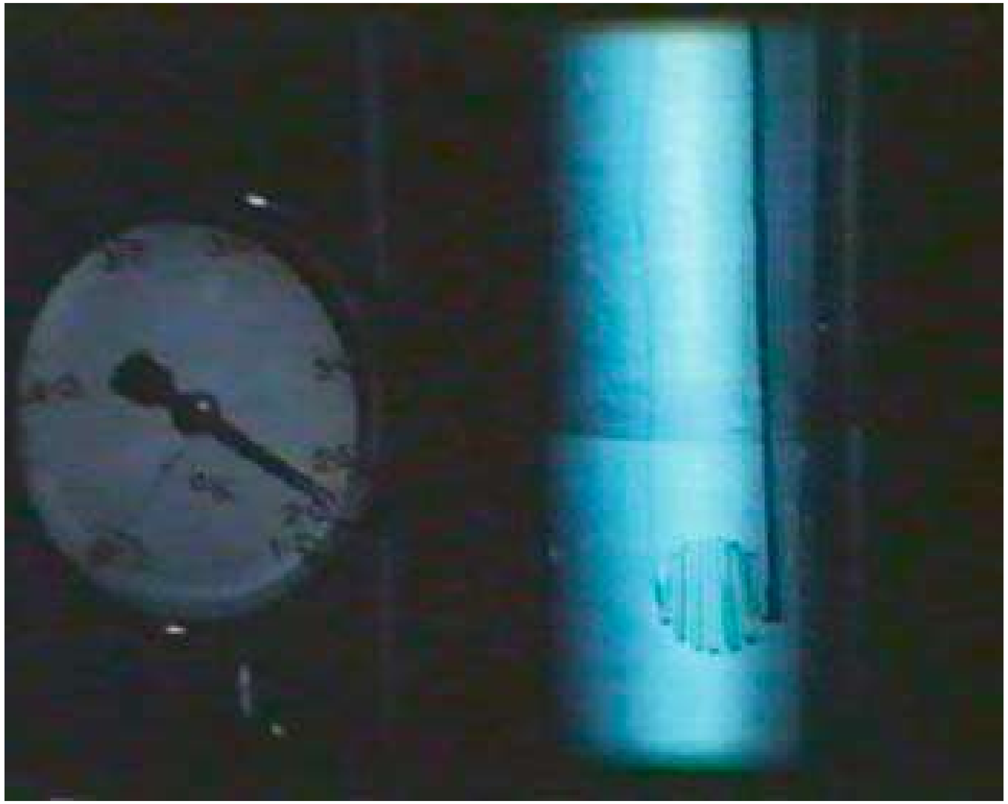
He II heat transfer

- High thermal conductivity of the liquid suppresses boiling

Electrical heaterin saturated liquid helium



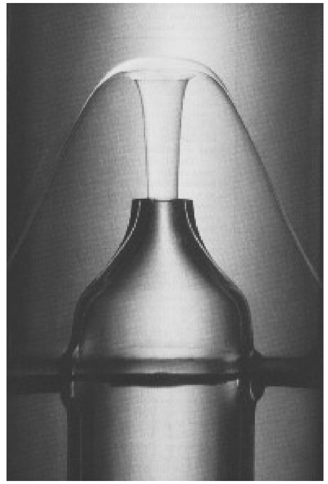
He I (T=2.4 K)

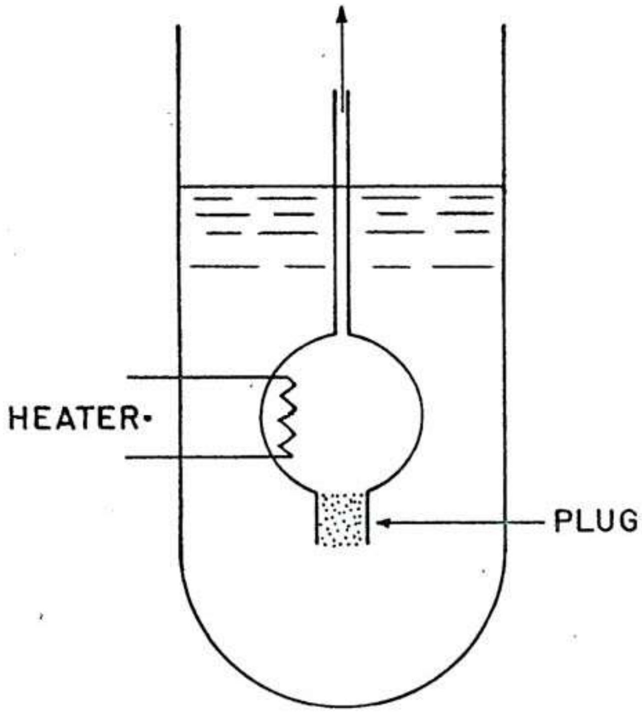


He II (T=2.1 K)

Fountain Effect

- Due to the inviscid nature of the superfluid, it can flow through microscopic channels without friction
- Ideal "superflow" conserves chemical potential, $\Delta p = \rho s \Delta T$
- Components to Fountain pump:
  - Heater provides chemical potential difference
  - Porous plug allows only inviscid superfluid component to flow
  - With the heater on, the superfluid flows into the bulb through the porous plug
  - Normal fluid can not flow out through plug since it is viscous
  - Normal fluid builds pressure and superfluid leaves the top





He II heat transfer

- Low viscosity ⇒ permeation
- Very high specific heat ⇒ stabilization
  - 10^5 times that of the conductor per unit mass
  - 2×10^3 times that of the conductor per unit volume
- Very high thermal conductivity ⇒ heat transport
  - 10^3 times that of cryogenic-grade OFHC copper
  - peaking at 1.9 K

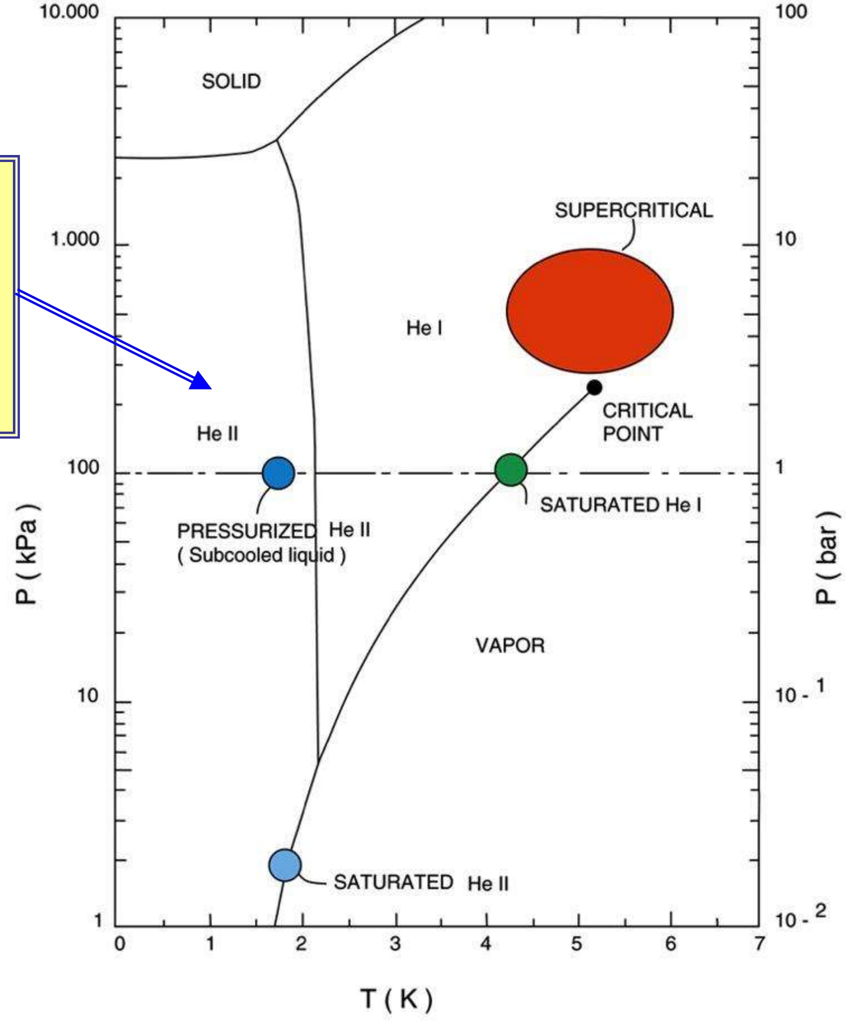
➢ Heat transfer enhancement

➢ Application to superconductors

Helium phase diagram

Superfluid Helium:

- Lower viscosity
- Larger heat transfer capacity

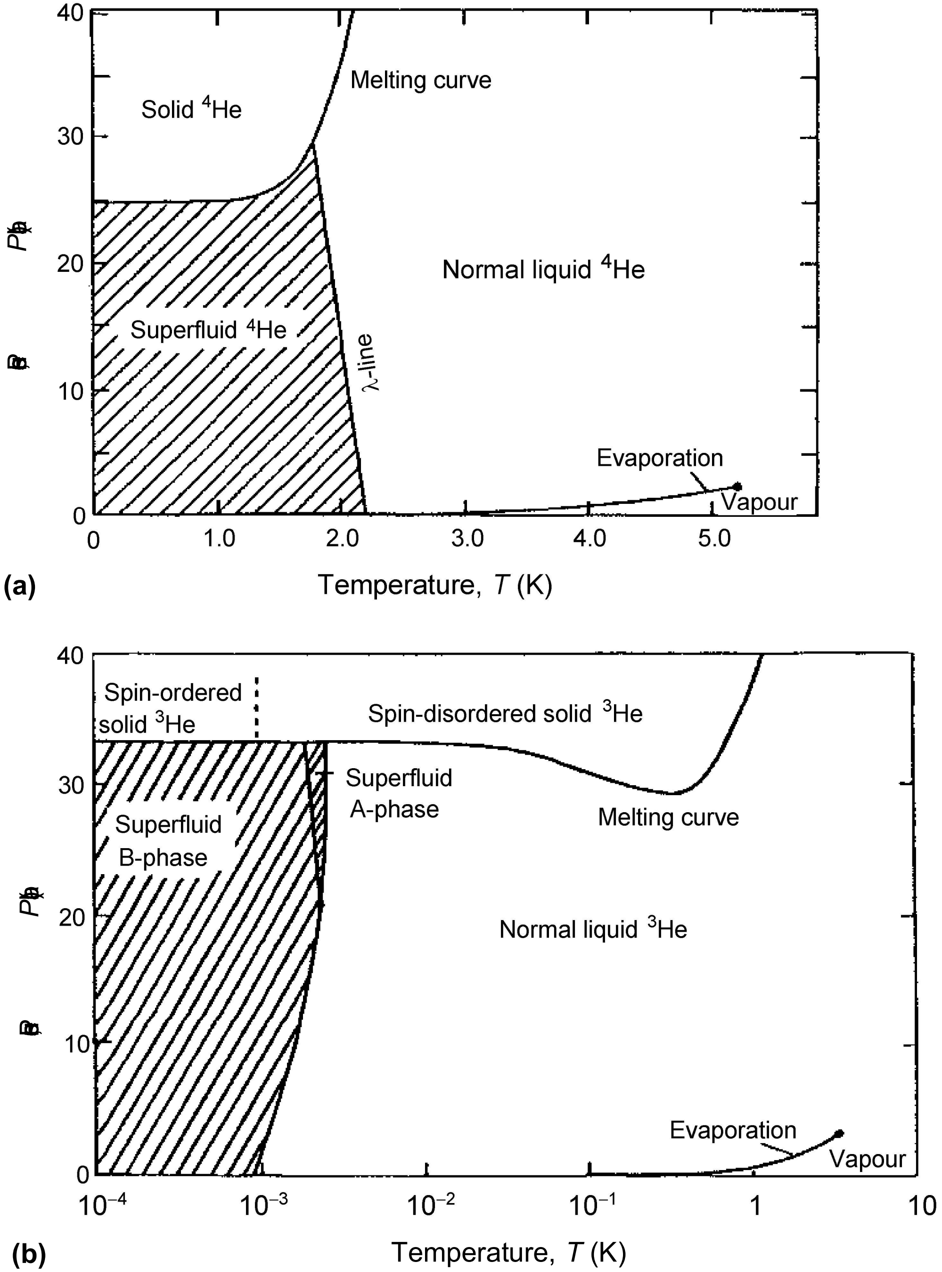


Phase domain	Advantages	Drawbacks
Saturated He I	Fixed temperature High heat transfer	Two-phase flow Boiling crisis
Supercritical	Monophase Negative J-T effect	Non-isothermal Density wave instability
He II	Low temperature High conductivity Low viscosity	Second-law cost Subatmospheric

Helium 3

- **Helium-3** (**^{3}He**, **tralphium**) is a light, non-radioactive isotope of helium with two protons and one neutron (common helium having two protons and two neutrons).
- ^{3}He is 1.3 x 10^{-4} % w.r.t ^{4}He
- It is very rare and difficult to be isolated from ^{4}He. Very expensive.
- The properties are of interest in relation to the theories of quantum statistical mechanics.
- An important property of helium-3, which distinguishes it from the more common helium-4, is that its nucleus is a fermion since it contains an odd number of spin $^1/_2$ particles. Helium-4 nuclei are bosons, containing an even number of spin $^1/_2$ particles.
- superfluid at 2.5mK, formation of weakly bound fermions: Cooper pairs
- It is an important isotope in instrumentation for neutron detection.
- ^{3}He refrigerator achieves 0.2-0.3 K
- ^{3}He ^{4}He dilution refrigerators to achieve very low temperatures (few mK).
- It is also used as working fluid in Cryocoolers. Temperature close to 1 K are reported with Pulse Tube Cryocooler.
- **Normal Boiling Point 3.19 K**
- **Normal Freezing Point -**
- **Critical Pressure 0.117 MPa**
- **Critical Temperature 3.32 K**
- **Liquid He –3 Density 58.9 kg/m3**
- **Latent Heat 8.49 kJ/kg**

Helium 3 and 4



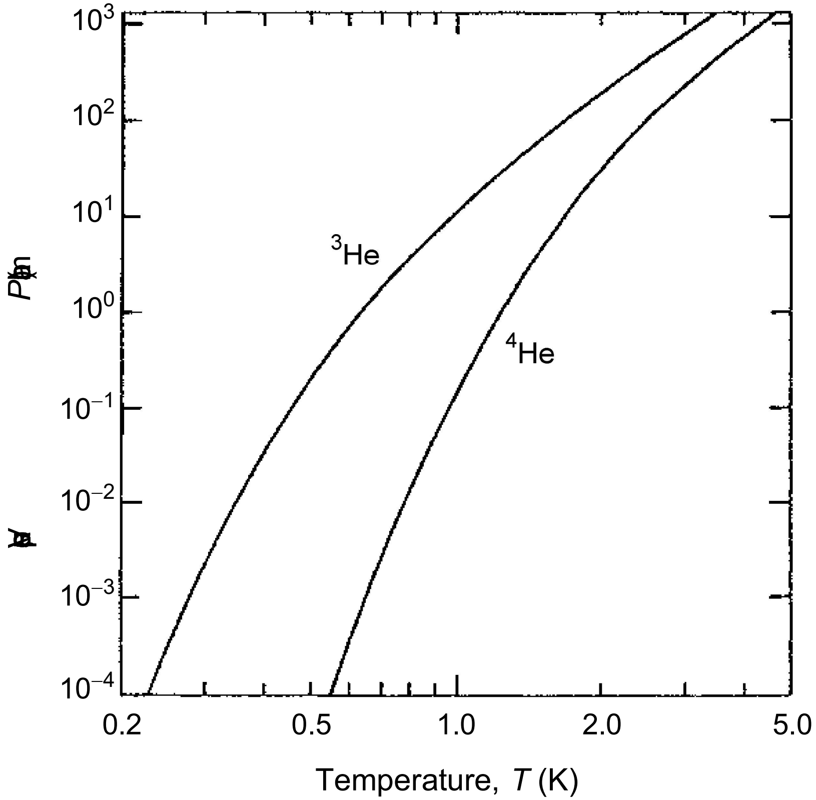


Fig. 2.7. Vapour pressures of liquid ^{3}He and liquid ^{4}He

- LHe -3 (like LHe -4) remains liquid under its vapor pressure up to absolute zero.
- No triple point.
- It must be compressed to 28.9 bar at 0.32 K to solidify.
- Liquid He -3 undergoes a different type of super fluid transition (A and B) at about 3.2 mK.

Helium 3 and 4

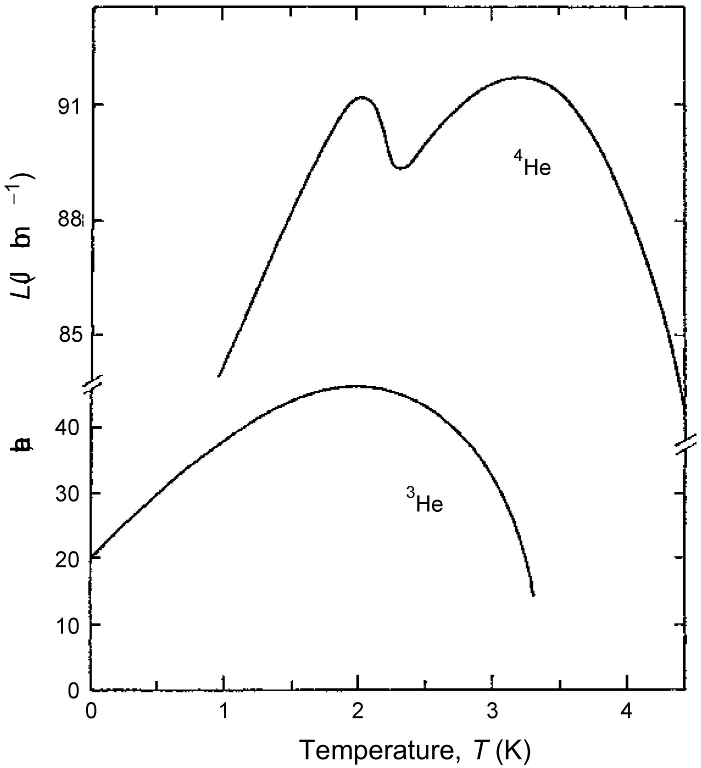


Fig. 2.6. Latent heats of evaporation of ^{3}He and ^{4}He. Note the change of vertical scale

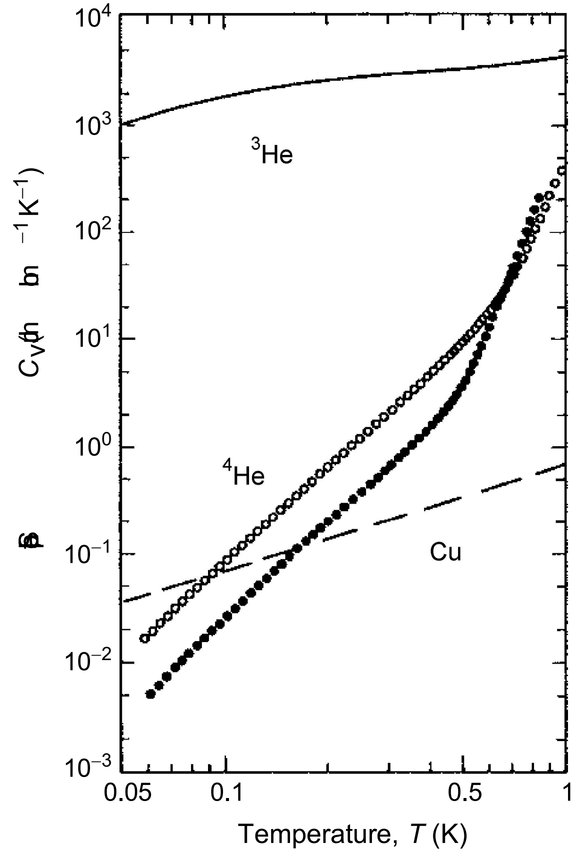
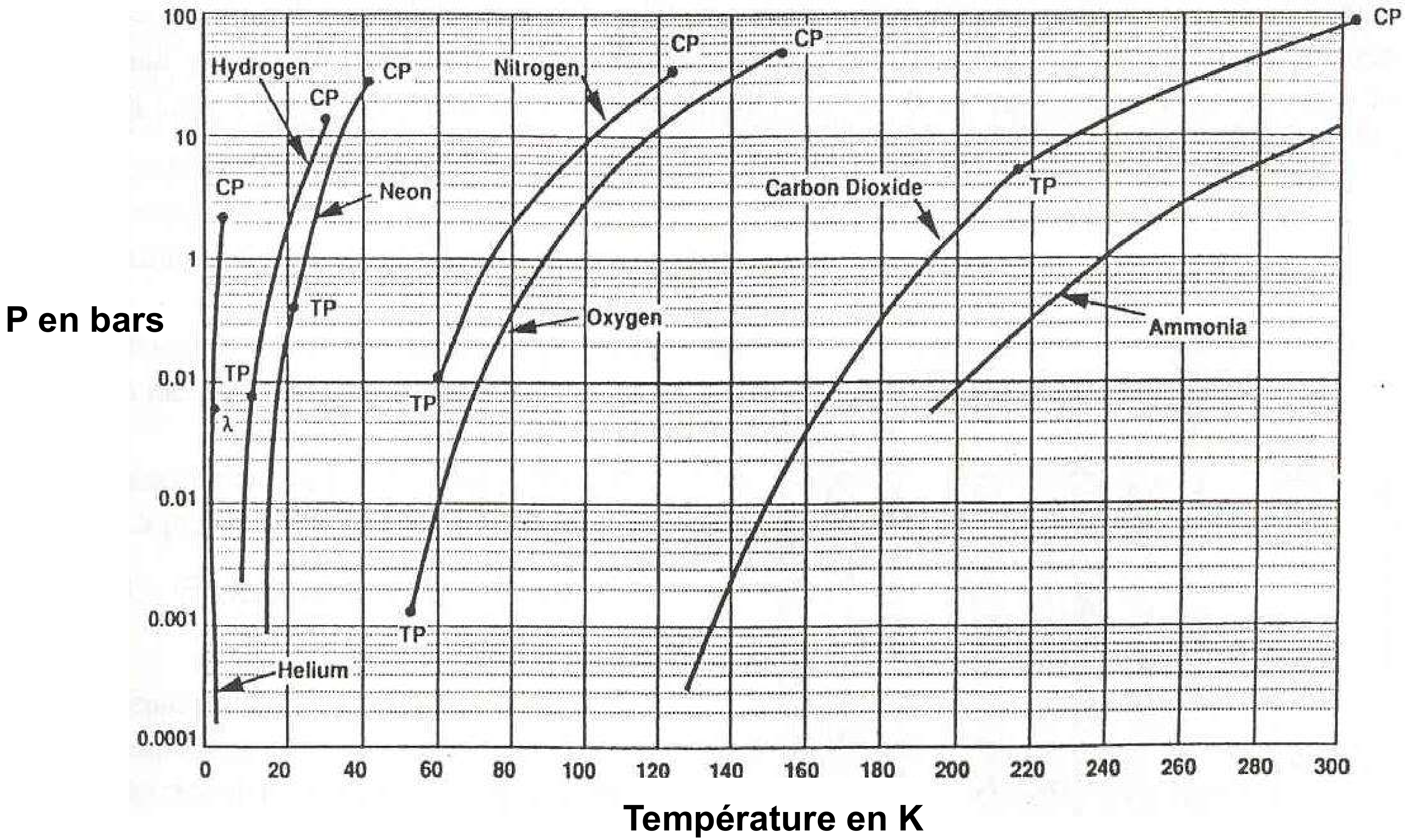


Fig. 2.9. Specific heat of liquid ^{4}He at vapour pressure (27.58 cm^3 mol^{-1}, ○) and at about 22 bar (23.55 cm^3 mol^{-1}, ●) [2.25] compared to the specific heats of liquid ^{3}He at vapour pressure [2.26] and of Cu

Vapour pressure of fluids



LHe
$\Delta T/\Delta P \approx$ 1mK/mbar
@ 4.2 K

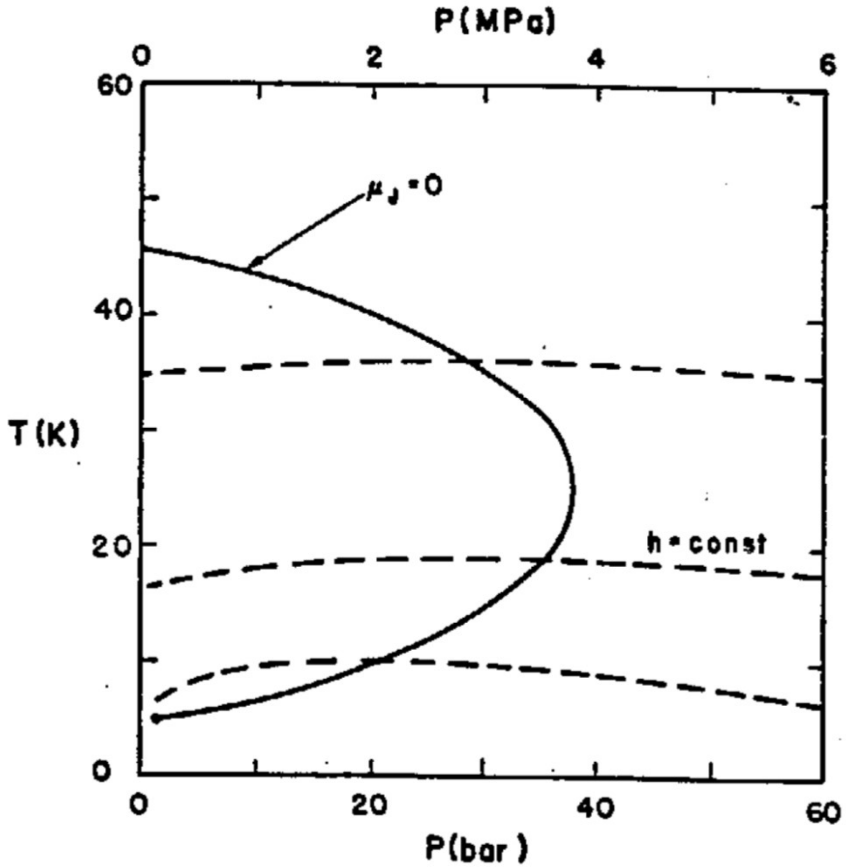
LH_2
$\Delta T/\Delta P \approx$ 2,5 mK/mbar
@ 20.4 K

LNe
$\Delta T/\Delta P \approx$ 5,5 mK/mbar
@ 27,1 K

LN_2
$\Delta T/\Delta P \approx$ 50 mK/mbar
@ 77 K

Important for crypumping application

JT Inversion Curve & Maximum Inversion Temperatures



Inversion curve for Helium

Fluid	Max Inversion Temperature (K)
Nitrogen	623
Argon	723
Hydrogen	202
Helium	43

- Maximum inversion temperature for helium is 43 K
- Note that below ~ 2 K He again warms on JT expansion
- Many fluids, such as N_2 can be liquefied using JT expansion – JT cycle

JT Inversion Curve & Maximum Inversion Temperatures

Maximum Joule-Thomson inversion temperatures

Cryogen	Maximum inversion temperature [K]
Helium	43
Hydrogen	202
Neon	260
Air	603
Nitrogen	623
Oxygen	761

While air can be cooled down and liquefied by JT expansion from room temperature, helium and hydrogen need precooling down to below inversion temperature by heat exchange or work-extracting expansion (e.g. in turbines)

48

- More in the following lessons

Introduction to cryogenics

Chiara Vignoli

Corso Nazionale INFN “Introduzione alla criogenia”

Bologna, 28-30 ottobre 2019

What is “cryogenics”

Greek:

- “Kryo”: Very cold (frost)
- “Genics”: to produce

Definition

- **cryogenics,** that branch of physics which deals with the production of very low temperatures and their effects on matter

  Oxford English Dictionary

  2nd edition, Oxford University Press (1989)

- **cryogenics**, the science and technology of temperatures **below 120 K**

  New International Dictionary of Refrigeration

  3rd edition, IIF-IIR Paris (1975)

Why 120 K?

It’s the temperature below which “permanent gases” start to condense (LXe is not “cryogenic”)

Fluid	Normal Boiling Point (K)
(Xenon)	(165.04)
Krypton	119.8
Methane	111.6
Oxygen	90.2
Argon	87.3
Nitrogen	77.4
Neon	27.1
Hydrogen	20.3
Helium	4.2

“Normal” means at ambient pressure 1 atm = 1.013 bar

Refrigeration

0 K **120 K** **273.15 K**

Cryogenics	Other kind of industrial refrigeration
Krypton	R134a (246.8 K)
Methane	R12 (243.3 K)
Oxygen	R22 (233 K)
Argon	Propane (231.1 K)
Nitrogen	Ethane (184 K)
Neon	CO_2 (195 K- solid)
Hydrogen	Ammonia NH3 (239.8)
Helium	...

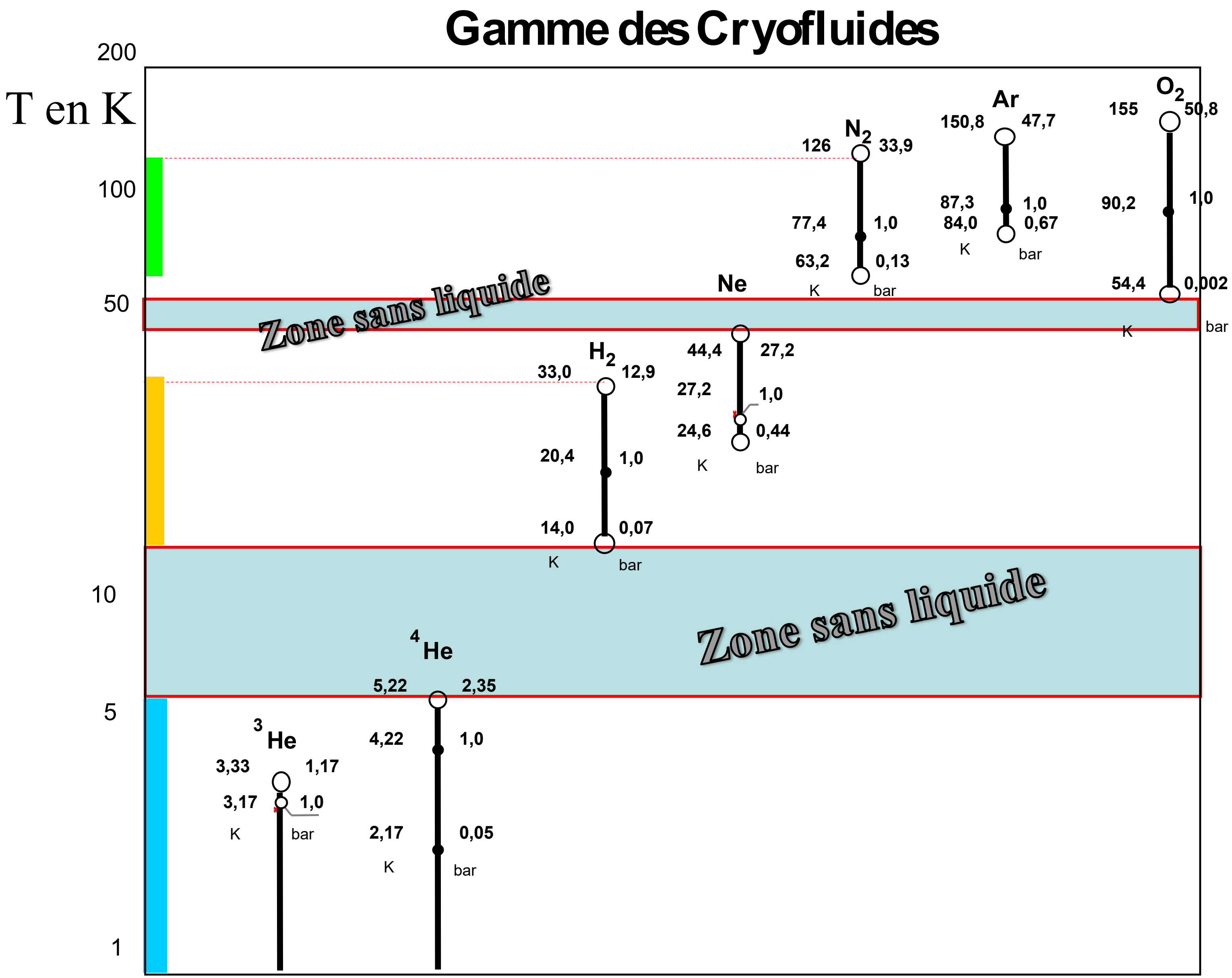
Characteristic temperatures of cryogenic fluids [K]

Cryogen	Triple Point	Normal boiling point	Critical Point
Methane	90.7	111.6	190.5
Oxygen	54.4	90.2	154.6
Argon	83.8	87.3	150.9
Nitrogen	63.1	77.3	126.2
Neon	24.6	27.1	44.4
Hydrogen	13.8	20.4	33.2
Helium	2.2*	4.2	5.2

* λ point

The simplest way of cooling equipment with a cryogenic fluid is to make use of its latent heat of vaporization, e.g. by immersion in a bath of boiling liquid. As a consequence, **the useful temperature range** of cryogenic fluids is that in which there exists latent heat of vaporization, i.e. **between the triple point and the critical point**, <u>with a particular interest in the normal boiling point,</u> i.e. the saturation temperature at atmospheric pressure.

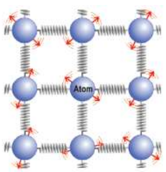
Cryofluid temperature inventory

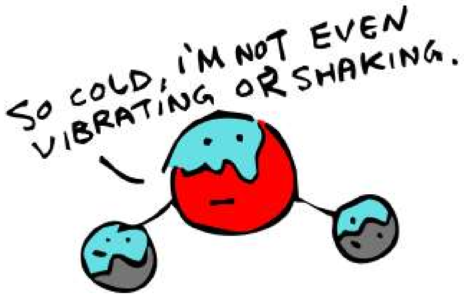


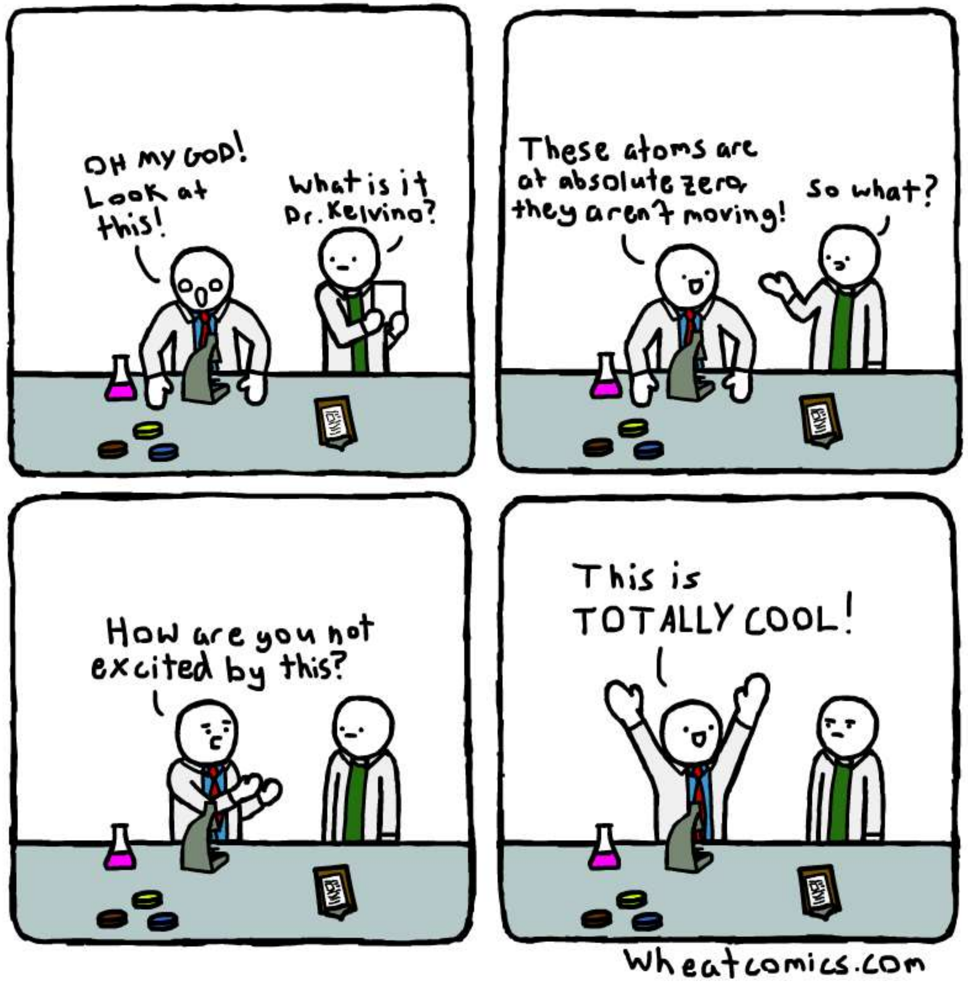
Absolute Zero Temperature

$0\ K = -273{,}15\ °C$

$T_K = T_C + 273{,}15$



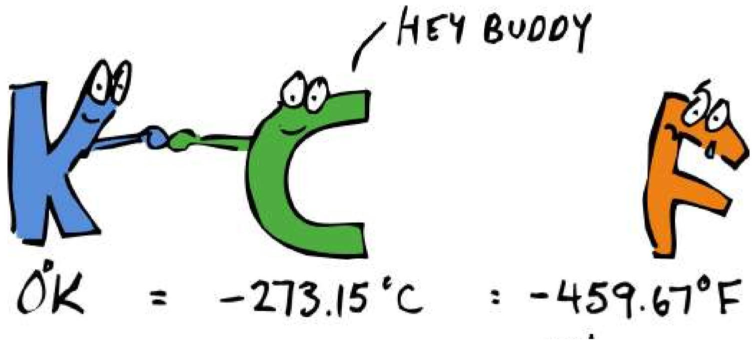


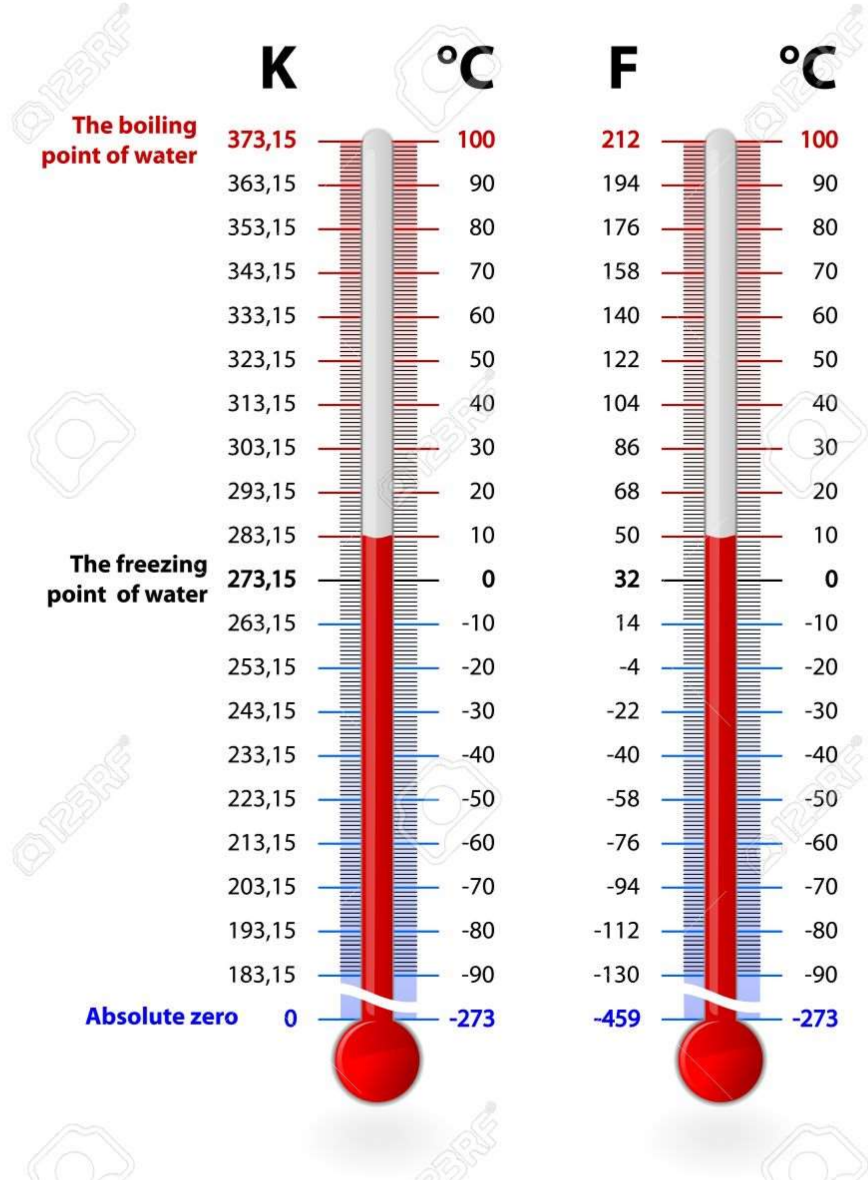


Absolute Temperature Scale

- The SI temperature scale is the Kelvin scale.
- It defines the triple point of water as the numerical value of 273.16, i.e., 273.16 K. The unit of temperature in this scale is the Kelvin (K).

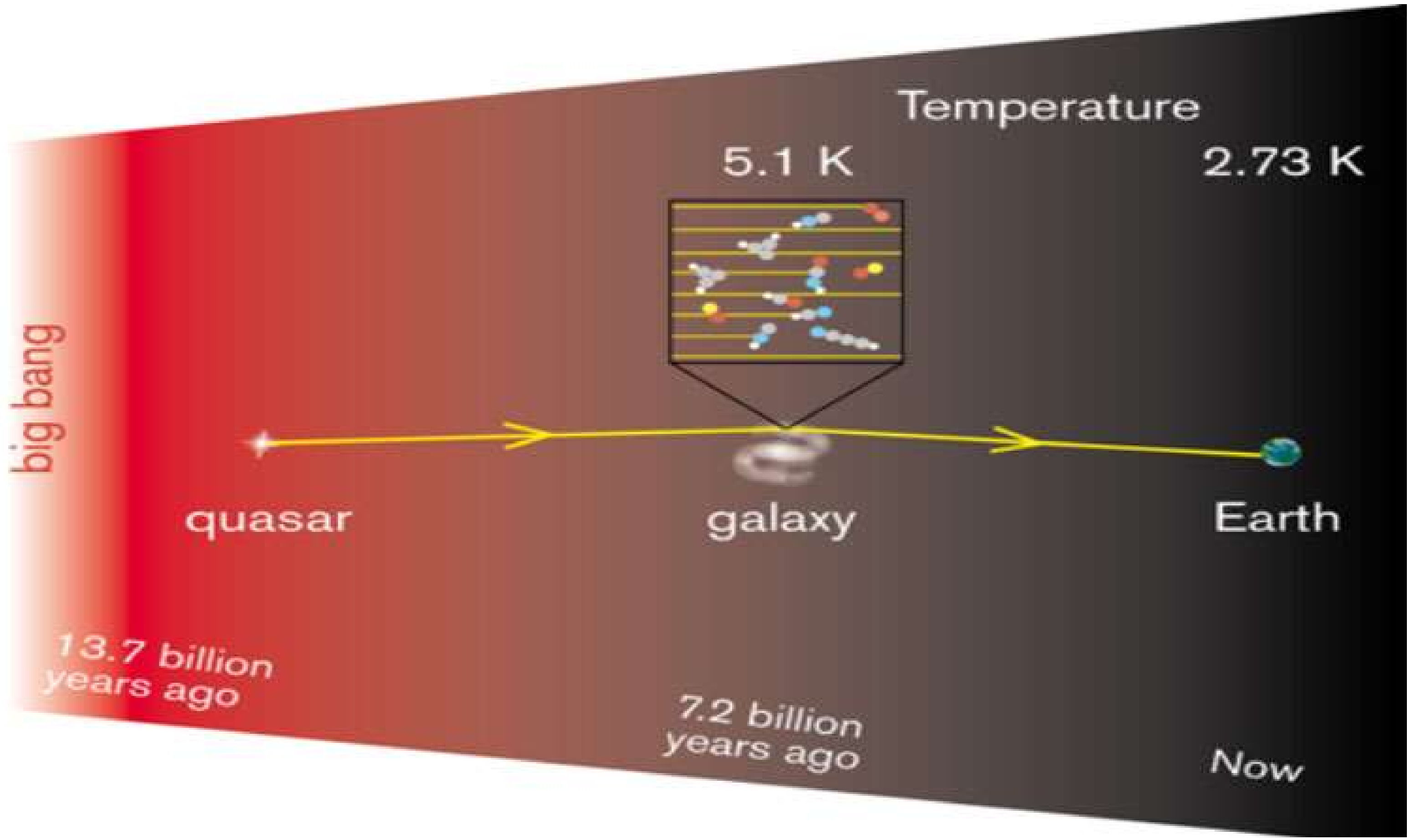
$$T_K = T_C + 273{,}15$$

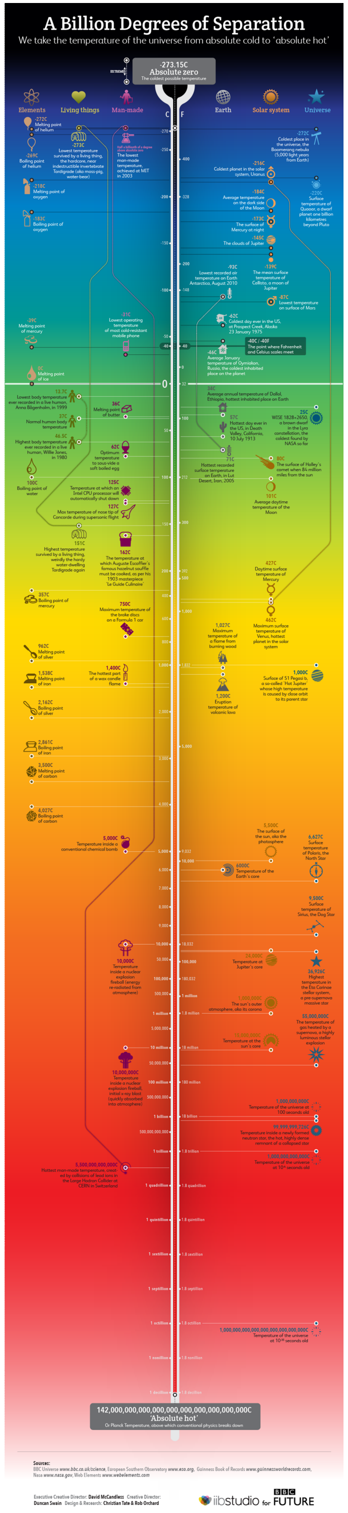


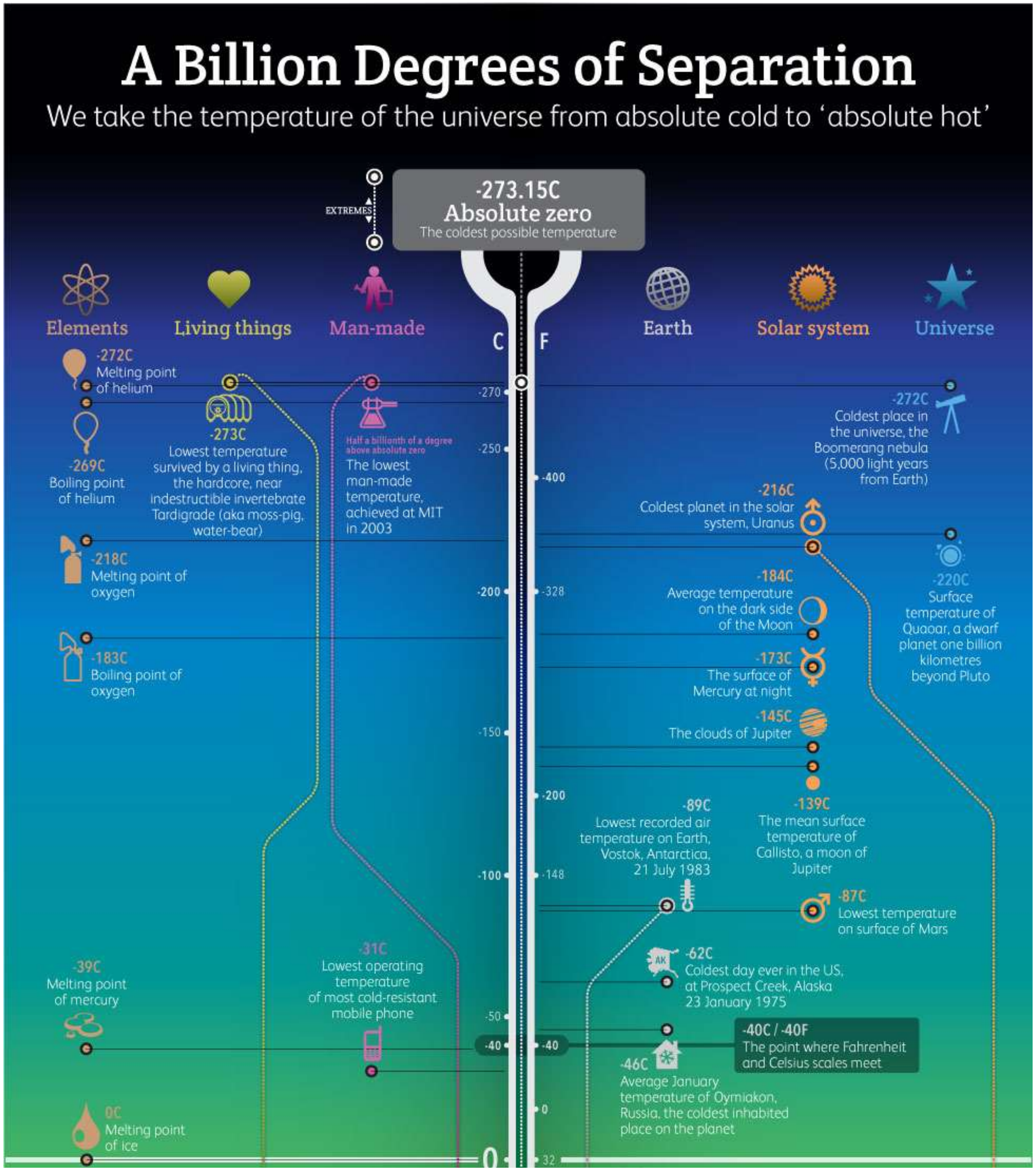


Universe Temperature

- Cosmic microwave background is at 2.73 K (cool!)




A Billion Degrees of Separation
We take the temperature of the universe from absolute cold to 'absolute hot'


A Billion Degrees of Separation
We take the temperature of the universe from absolute cold to 'absolute hot'
EXTREMES
-273.15C
Absolute zero
The coldest possible temperature
Elements
Living things
Man-made
C
F
Earth
Solar system
Universe
-272C Melting point of helium
-269C Boiling point of helium
-273C Lowest temperature survived by a living thing, the hardcore, near indestructible invertebrate Tardigrade (aka moss-pig, water-bear)
Half a billionth of a degree above absolute zero
The lowest man-made temperature, achieved at MIT in 2003
-272C Coldest place in the universe, the Boomerang nebula (5,000 light years from Earth)
-216C Coldest planet in the solar system, Uranus
-218C Melting point of oxygen
-183C Boiling point of oxygen
-184C Average temperature on the dark side of the Moon
-220C Surface temperature of Quaoar, a dwarf planet one billion kilometres beyond Pluto
-173C The surface of Mercury at night
-145C The clouds of Jupiter
-89C Lowest recorded air temperature on Earth, Vostok, Antarctica, 21 July 1983
-139C The mean surface temperature of Callisto, a moon of Jupiter
-87C Lowest temperature on surface of Mars
-62C Coldest day ever in the US, at Prospect Creek, Alaska 23 January 1975
-39C Melting point of mercury
-31C Lowest operating temperature of most cold-resistant mobile phone
-40C / -40F The point where Fahrenheit and Celsius scales meet
-46C Average January temperature of Oymiakon, Russia, the coldest inhabited place on the planet
0C Melting point of ice
-270
-250
-200
-150
-100
-50
-40
0
-400
-328
-200
-148
-40
0
32

10

Generation of low temperatures

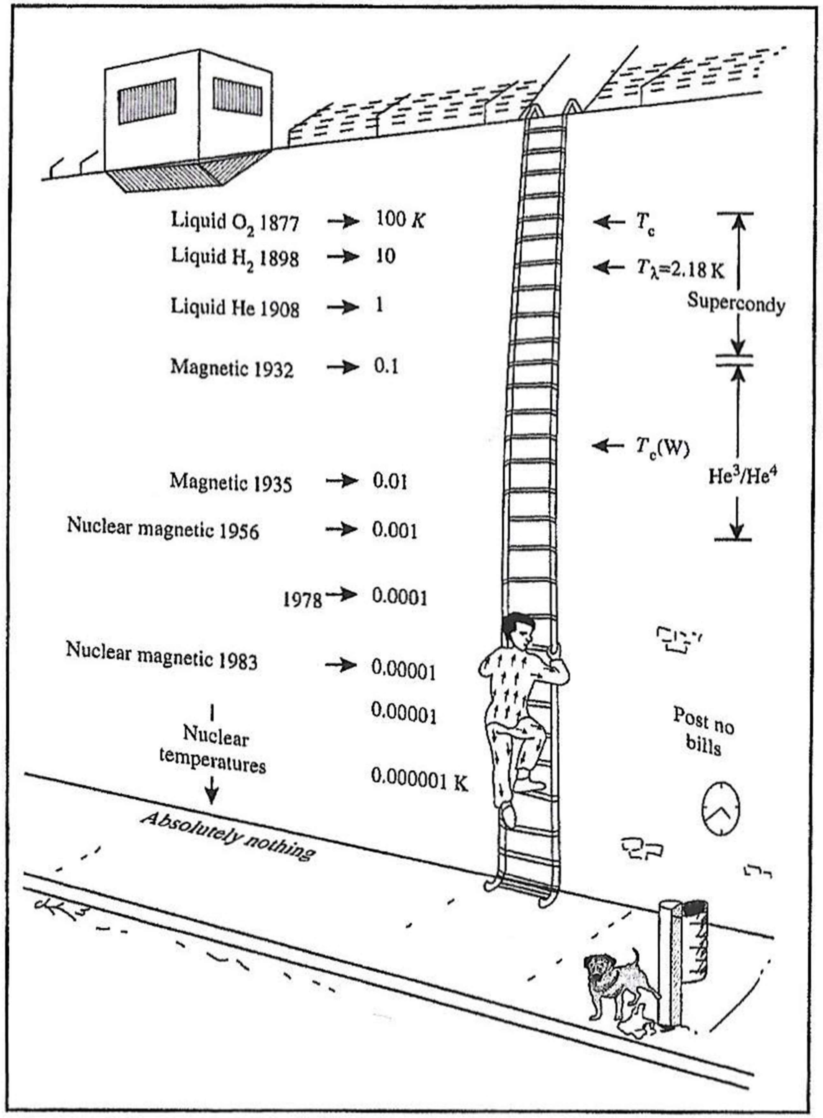
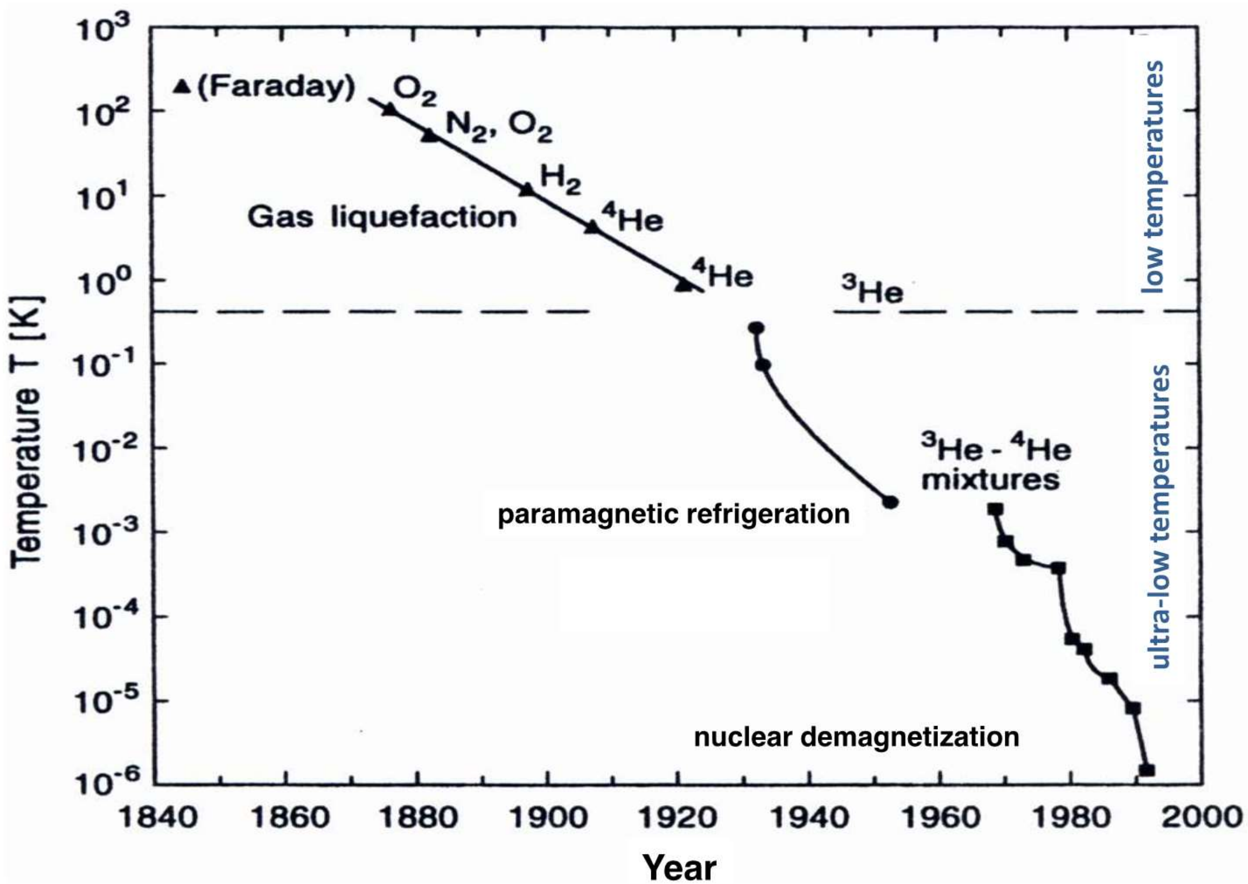
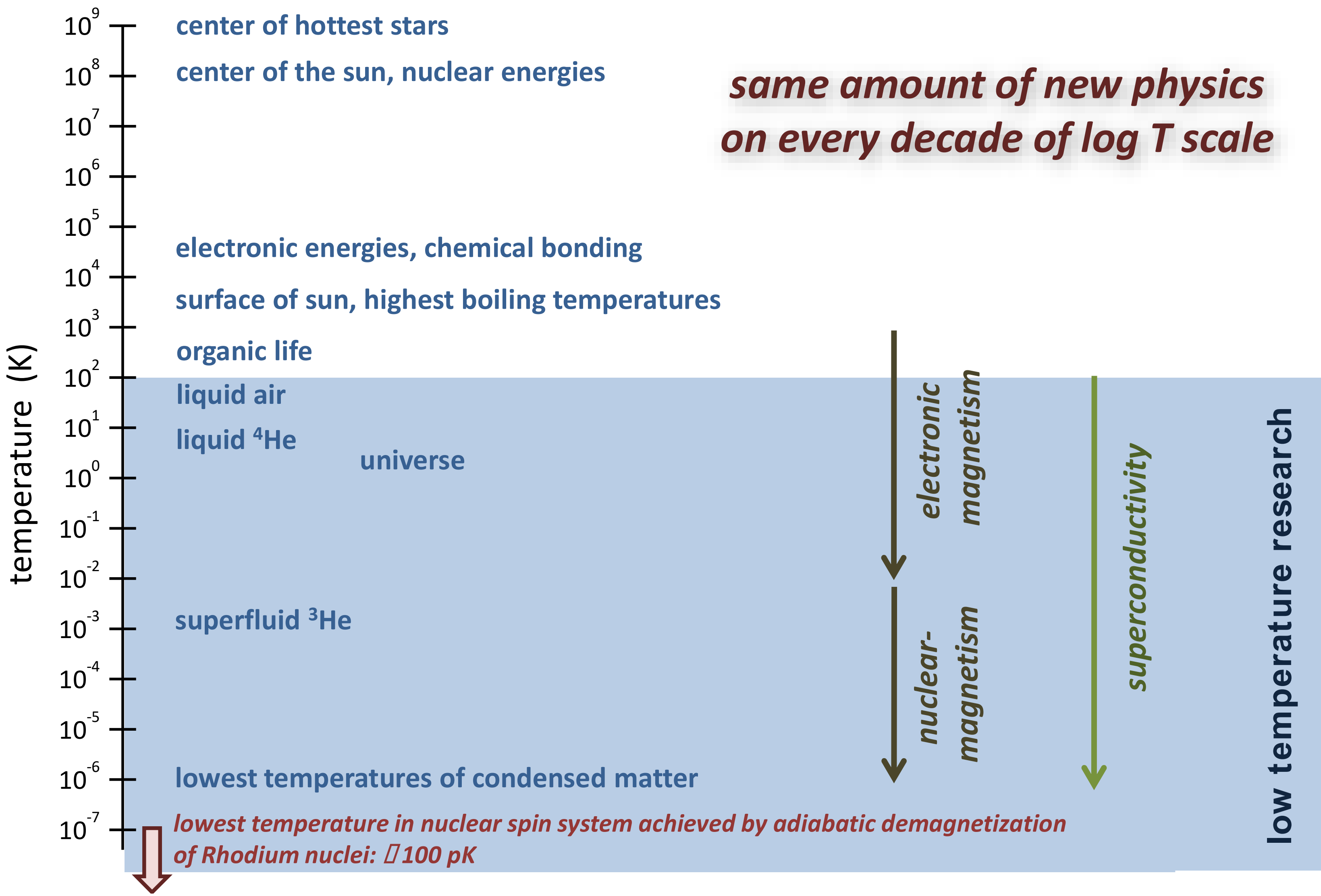


FIG. 1.2 The cooling struggle illustrated by the endless logarithmic temperature ladder.



Attempt to reach absolute zero



Refrigeration techniques

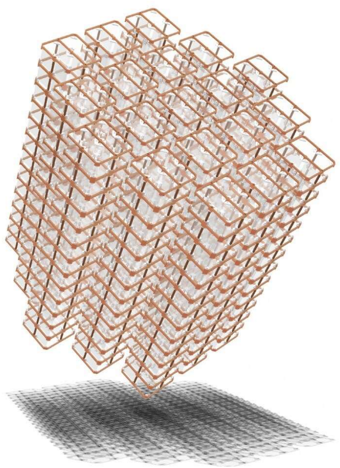
temperature range	**refrigeration technique**	**available since**	**typical T_{min}**	**record T_{min}**
Kelvin	universe			2.73 K
	^{4}He evaporation	1908	1.3 K	0.7 K
	^{3}He evaporation	1950	0.3 K	0.25 K
Millikelvin	^{3}He-^{4}He dilution	1965	10 mK	2 mK
	Pomeranchuk cooling	1965	3 mK	2 mK
	electron spin demagnetization	1934	3 mK	1 mK
Microkelvin	nuclear spin demagnetization	1956	50 µK	100 pK

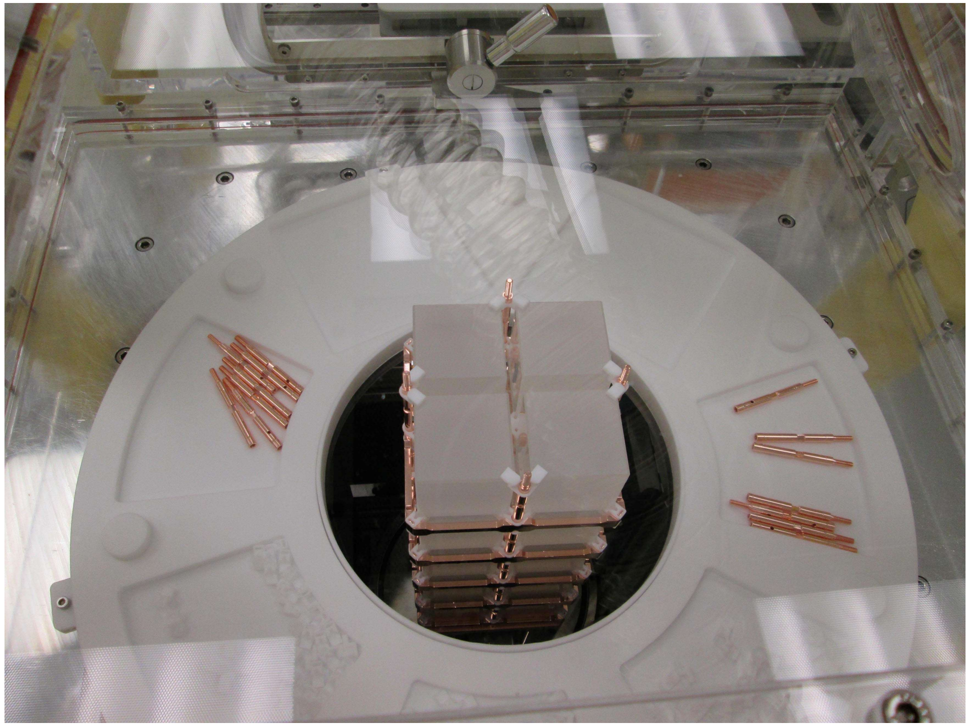
Few milli-Kelvin

CUORE @ Laboratori Nazionali del Gran Sasso, INFN, Italy

World record:

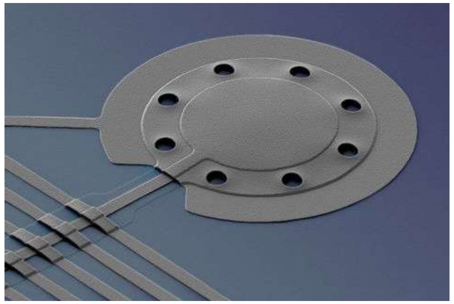
≈ 400 kg of Cu inside a 1 m^3 @ 6 mK for 15 days





Few hundreds of micro-Kelvin

- When atoms gain energy through any means possible, they vibrate faster. Unless they are at absolute zero (0 Kelvin), atoms are always vibrating. In fact, temperature is just a measure of the average rate of vibration of a collection of molecules.
- Lasers can confer energy upon atoms, heating them up and causing them to get a bit more jiggly. As it turns out, **lasers can also organize light energy in such a way as to slow down these atomic vibrations**. Thus, these **focused beams of light can cool a substance down**.
- A team of researchers at the National Institute of Standards and Technology (NIST) in Boulder, Colorado, wanted to know just how cold they could get a substance using this laser cooling method. Organizing light as best they could, they focused it at a rather small sample and began to cool it.



- The sample, an **aluminum membrane – 20 micrometers wide and 100 nanometers thick,** was lowered to a temperature of 0.000 36 Kelvin. As reported in the journal Nature, this little piece of metal was 10,000 times colder than the deep, dark vacuum of space.

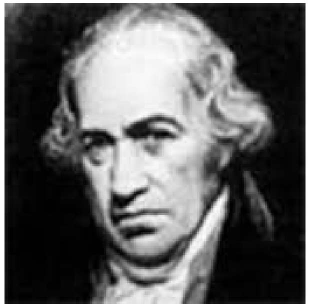
Lowest T ever reached: hundreds of pico-Kelvin

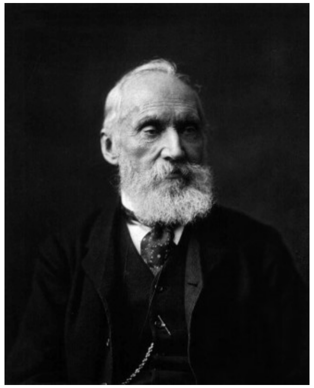
- Work carried out in the Low Temperature Laboratory @ Aalto University, in Finland
- **The record-low temperature was produced in about two-gram piece of rhodium metal, which was cooled to 100 pK, or 0.000 000 000 1 degrees above the absolute zero.**

Too low…

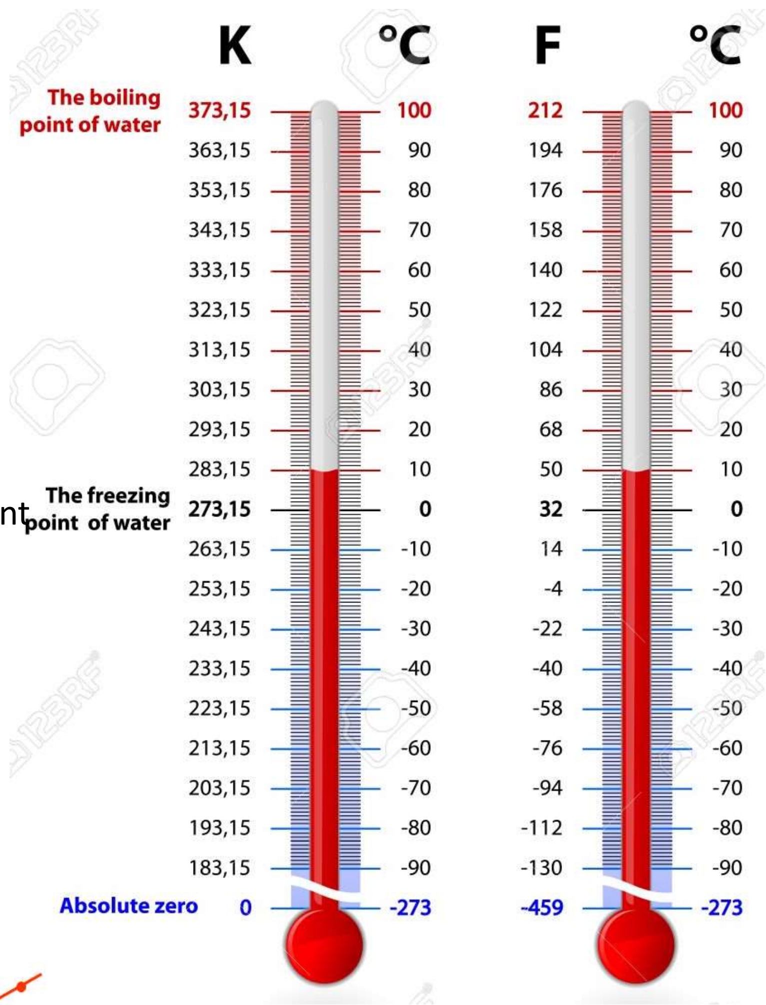
- Cooling of molecules (down to hundreds of nano Kelvins) may be used to form exotic states of matter.
- The Cold Atom Laboratory (CAL) is an instrument designed for use in microgravity to form Bose Einstein condensates (around 1 pico Kelvin temperature) and test laws of quantum mechanics and other physics principles.
- **In 2013 Ulrich Schneider at the University of Munich (Germany) cooled gas below absolute zero, which reportedly made it hotter instead of colder!**
- Sources: Braun, S., Ronzheimer, J. P., Schreiber, M., Hodgman, S. S., Rom, T., Bloch, I., Schneider, U. (2013) "Negative Absolute Temperature for Motional Degrees of Freedom". Science 339, 52–55.

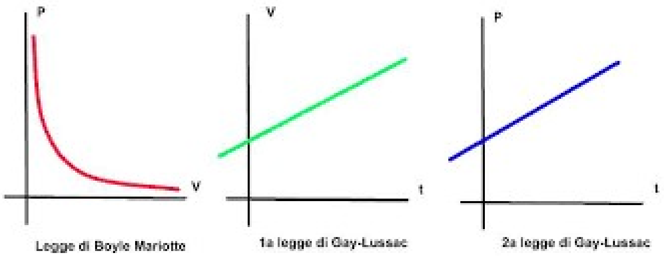
Temperature scales

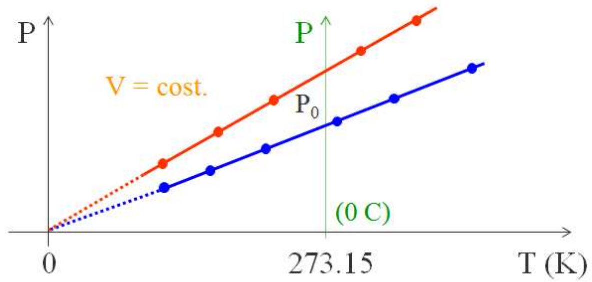




- <u>Fahrenheit (°F)</u>
  - Introduced in 1724
  - Defined by 2 fixed points
    - Ice, water & ammonium chloride mixture = 0 °F
    - Human body = 96 °F (now taken as 98.6 °F)
  - Only positive values
  - First modern thermometer (Hg)
- <u>Celsius (°C)</u>
  - Introduced 18 years later (1742)
  - Defined by setting boiling point of water to 0° and melting point to 100°.
    - Later reversed by Jean Pierre Christine (1745)
  - Absolute zero in Celsius is -273.15°
  - “Centigrade” degrees
  - Also negative values
- <u>Kelvin</u>
  - Introduced 1848
  - Zero point set to Absolute Zero







First steps towards Refrigeration

Known refrigeration methods

at the time of I. Newton (1642 -1727):

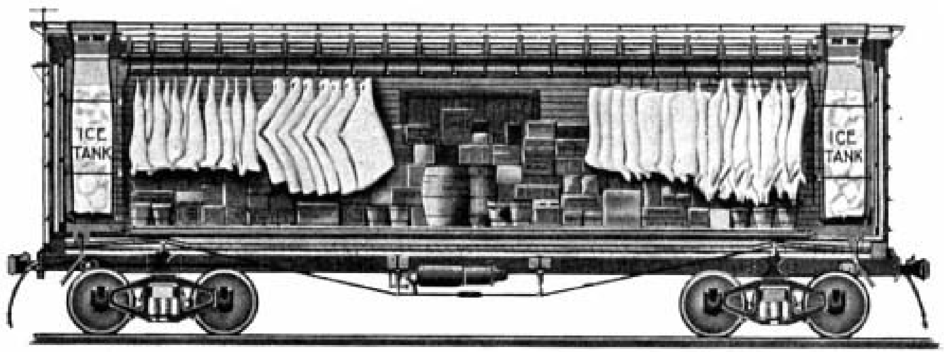
- Refrigeration by a colder object
  - e.g. ice or snow
- Refrigeration by evaporation
- Refrigeration by dissolving saltpeter in water (saltpeter = sodium nitrate $NaNO_3$ or potassium nitrate KNO_3)

Incentives for refrigeration and cryogenics

Early 19th century

- large scale refrigeration
  - only by natural ice
- Increasing demand for artificial refrigeration by
  - the butchers,
  - the brewers
  - and, later on, the industrialists

Ice harvesting



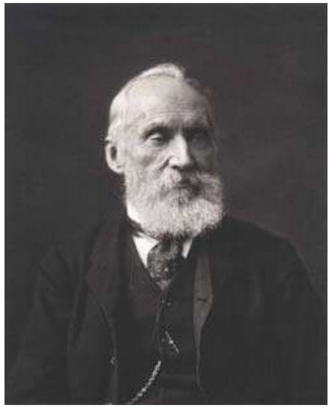
Refrigerated railroad car

Ice storage cave in Bliesdalheim

Important discoveries (1)

- **1852** Discovery of the **Joule-Thomson effect**
  Cooling of compressed gases by expansion ($T<T_i$):
  Key concept on the way to absolute zero!
  - James Prescott Joule [1818-1889],
  - Sir William Thomson [Lord Kelvin, 1824-1907]

J. P. Joule



Lord Kelvin

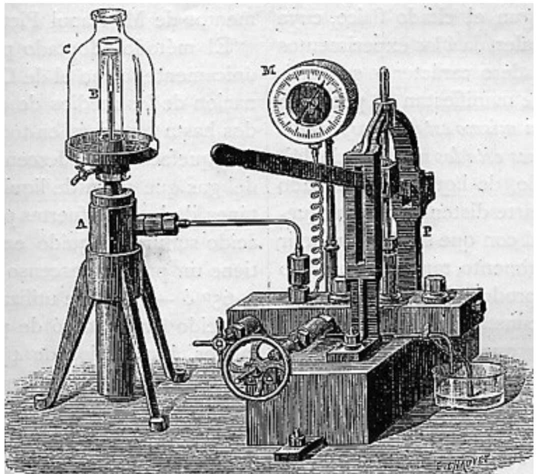
- **1877** First liquefaction of “permanent gases” in small quantities
  - oxygen: Louis Paul Cailletet [1832-1913]
  - oxygen, nitrogen: Pierre Pictet [1846-1929]
  - The successful liquefaction of Oxygen was announced at the meeting of the Académie de Sciences in Paris on December 24th, 1877 independently by the physicist Louis Paul Cailletet from Paris and the professor Raoul Pictet from Geneva.

L. P. Cailletet

P. Pictet

Cailletet and Pictet apparatus

- Cailletet's apparatus
  - compression to 200 bar in a glass tube with a hand-operated jack, using water and mercury for pressure transmission
  - pre-cooling of the glass tube with liquid ethylene to -103°C
  - expansion to atmosphere via a valve



A.—Aparato de M. Cailletet para la liquelaccion de los gases

- Pictet's apparatus
  - production of oxygen under pressure in a retort
  - two pre-cooling refrigeration cycles:

    first stage SO_2 (-10°C)
    second stage CO_2 (-78°C)
  - oxygen flow is pre-cooled by the means of heat exchangers and expands to atmosphere via a hand valve

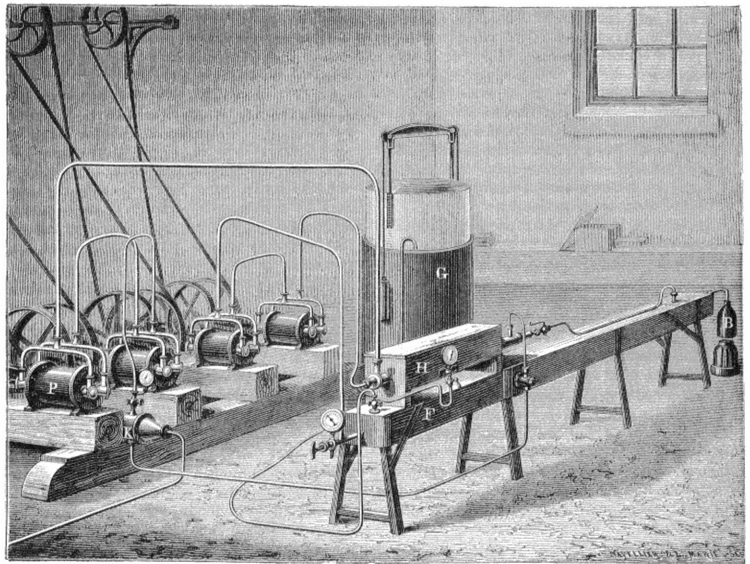


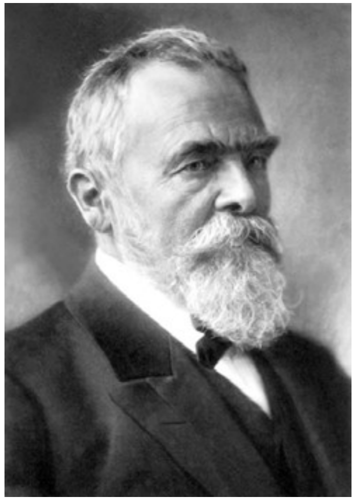
Fig. 1. — Grand appareil de M. Raoul Pictet pour la liquéfaction des gaz. (D'après une photographie.)

Important discoveries (2)

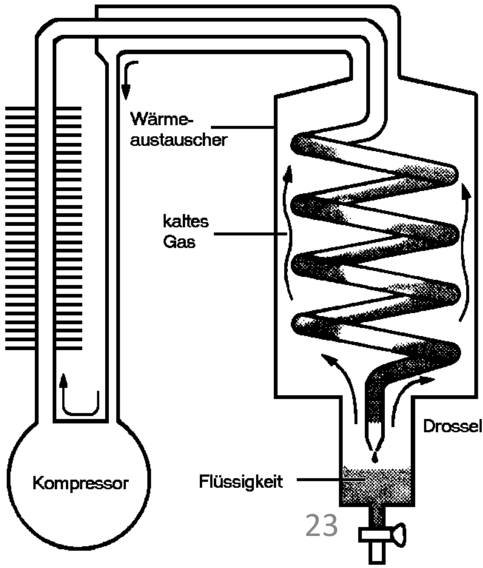
- **1883** First liquefaction of N_2 and O_2 gas in substantial quantities:
  - Zygmunt Florenty Wroblenski [1845-1888]
  - Stanislav Olszewski [1846-1915]
- **1895** First liquefactionof air:
  - Carl von Linde [1842-1934]
- **1898** First liquefaction of gaseous hydrogen using the first thermos bottle ("Dewarflask") and a cascade method:
  - James Dewar[1842-1923]

Z. Wroblenski

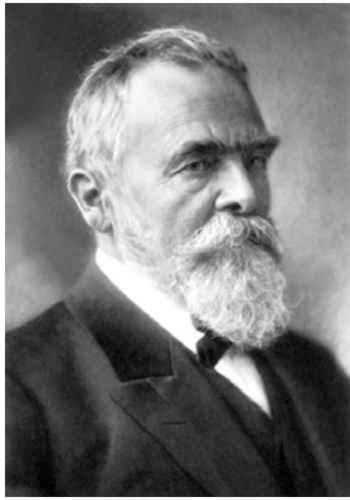
S. Olszewski



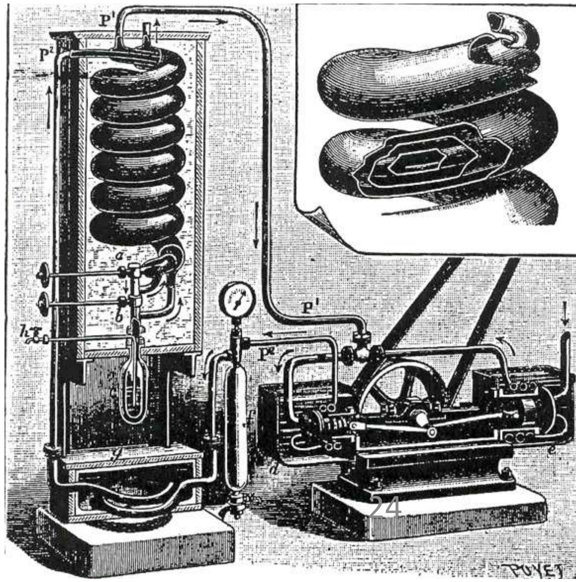
C. v Linde



C. von Linde



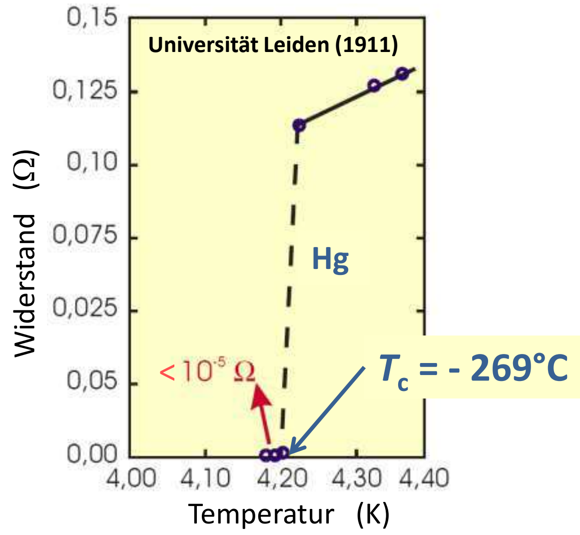
- 1873 development of cooling machine allowing the temperature stabilization in beer brewing
- 21. 6. 1879 foundation of “*Gesellschaft für Linde’s Eismaschinen AG*” together with two beer brewers and three other co-founders
- 1892 - 1910 re-establishment of professorship
- 12.5.1903 patent application: “Lindesches Gegenstromverfahren”
  liquefaction of oxygen (-182 °C = 90 K)

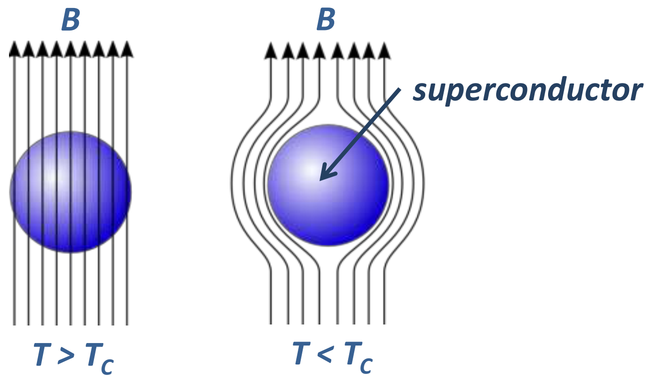


Important discoveries (3)

- **1908**: liquefaction of last "permanent gas" He (H.Kammerlingh Onnes)
- **1911** discover of vanishing resistance in mercury at 4.2 K, later referred to as superconductivity (H.Kammerlingh Onnes)
- 1922: Kammerlingh Onnes reaches T < 1K
- 1926-27: adiabatic demagnetization of electron spins in paramagnetic salts by Debye (1926) and independently by Giauque (1927)
- 1933 Meissner- Ochsenfel diamagnetism discovery
- 1937 discovery of 4He superfluidity by Kapitza and independently by Allen and Misener
- 1951 ^3He-^4He dilution refrigerator proposal
- 1956 nuclear cooling method proposal
- 1972 3He superfluid discovery at 2 mK
- 1978 laser cooling discovery
- 1995 observation of BEC in Na, Rb gas

Heike Kammerlingh Onn
(1853 – 1926)
Physics Nobel Prize: 1913





superconductors perfectly expel magnetic field

$$B_{in} = (1 + \chi)\ B_{ex} = 0$$

(χ = magnetic susceptibility)

ideal diamagnetism, $\chi = -1$

Applications

Research:

- Particle and Astro-particle Physics research
  - Accelerators
  - Detectors
- Nuclear fusion research
- Space research
- Solid state physics
- Biological, bio-molecular reseach
- Medical research
- Development of electronics, sensors and new instrumentation

Application: particle physics research

- Cryogenics plays a major role in modern particle and astroparticle physics:
  - Enables superconductivity for accelerators (LHC, RICH, HERA, TEVATRON)
    - Beam bending and focusing magnets (1.9 K – 4.5 K)
    - Magnets for particle identification in large detectors (4.2 – 4.5 K)
    - Superconducting RF cavities for particle acceleration (1.8 K – 4.2 K)
  - Allows construction of dense/large/pure liquids detectors
    - LAr calorimeters (87 K)
    - LAr /LXe TPC, both in single and double phase (87 K /165 K)
    - LH_2 targets, moderators and absorbers (20 K)
  - Provides sub-Kelvin cooling for certain types of double beta decay and dark matter searches (He_3-He_4 bolometers)

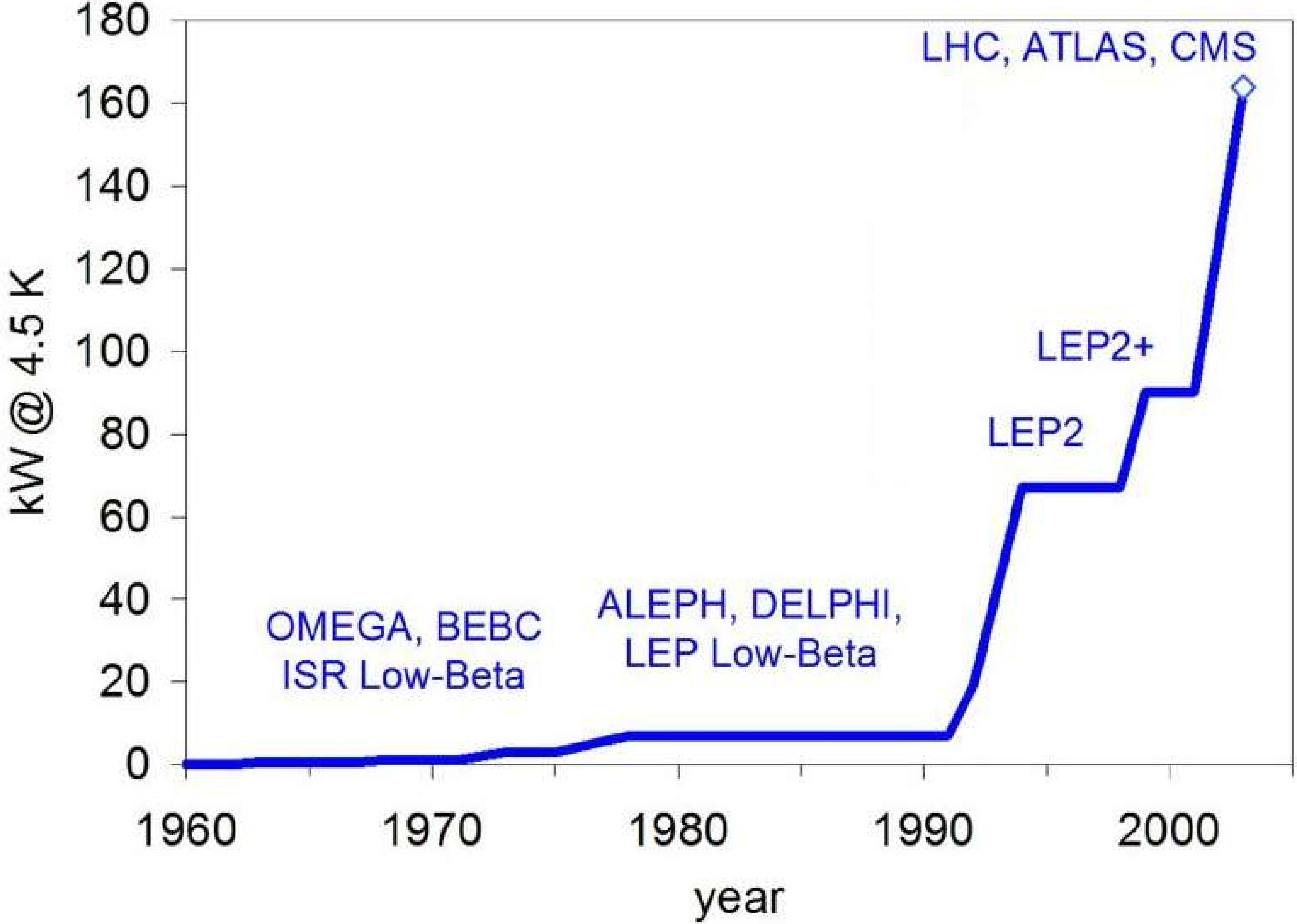

The TEVATRON at Fermilab Fermilab, USA

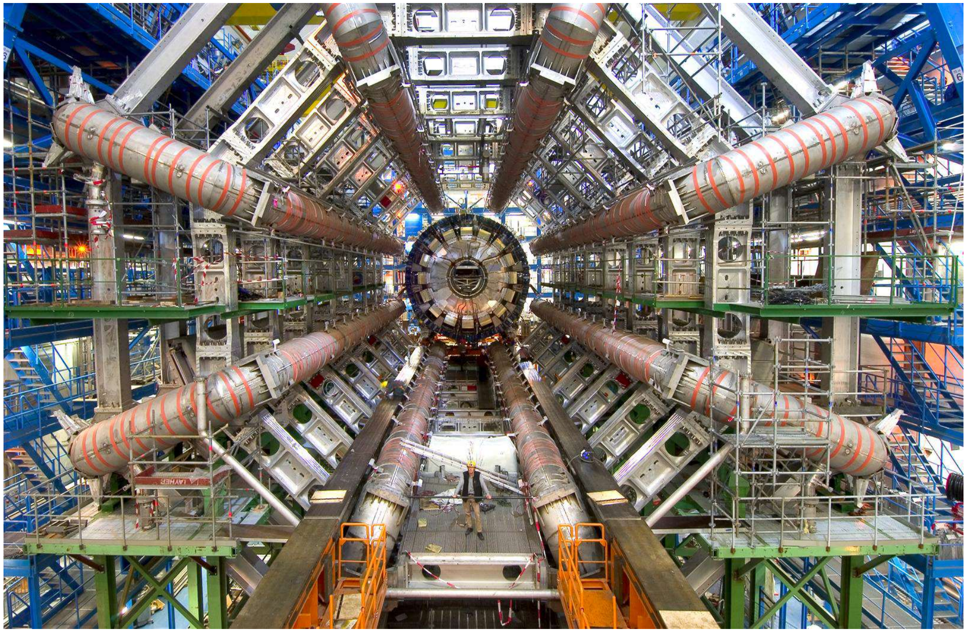
RHIC at Brookhaven National Lab, USA

HERA proton ring at DESY, Germany

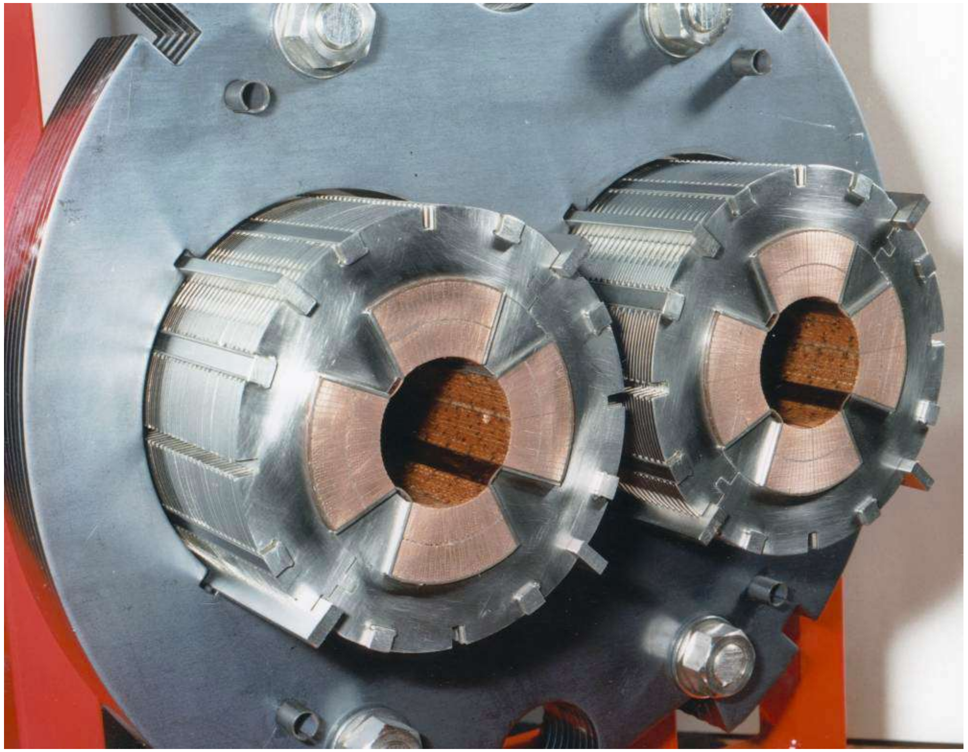

LHC at CERN

Cryogenic history @ CERN

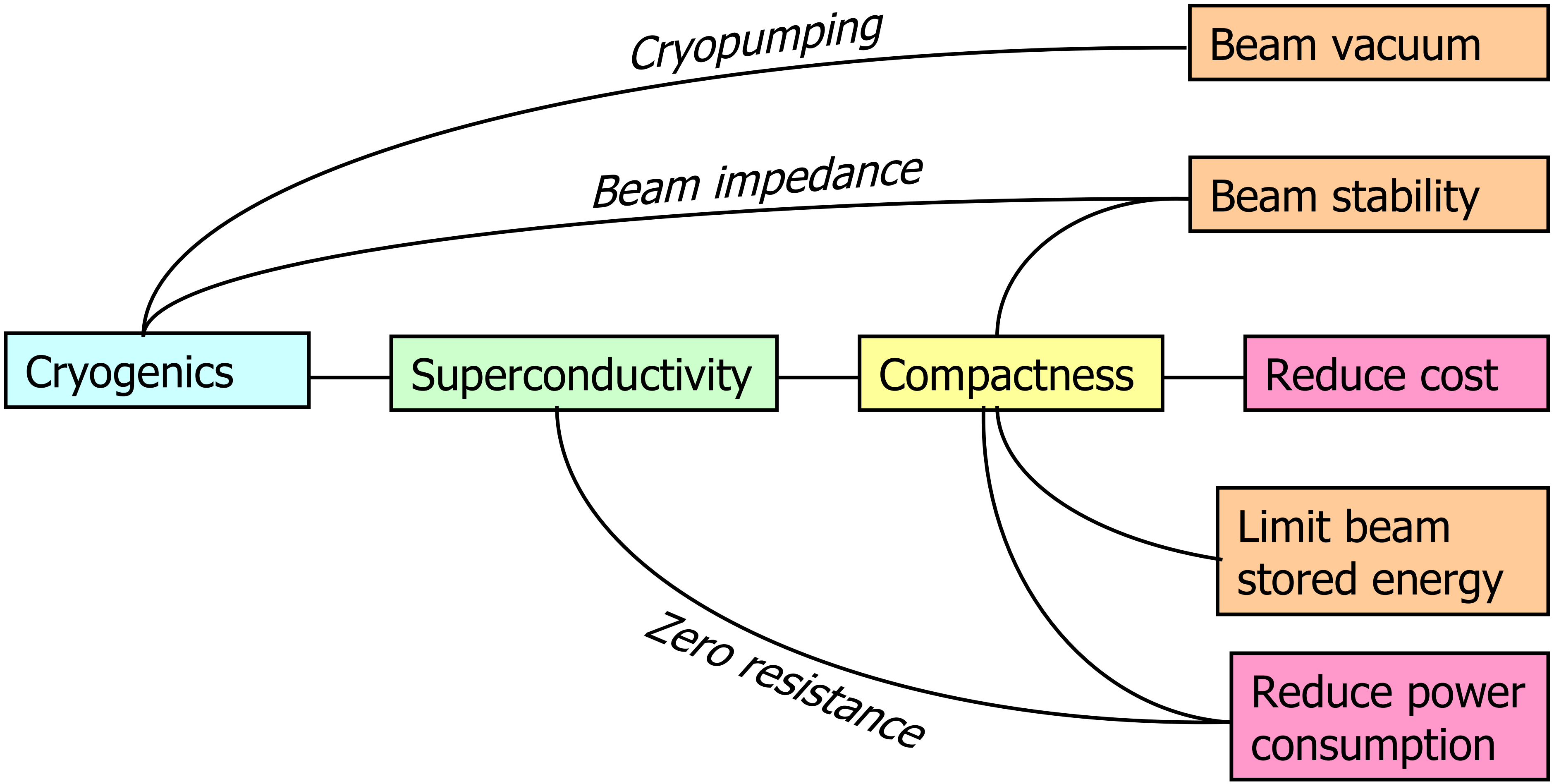




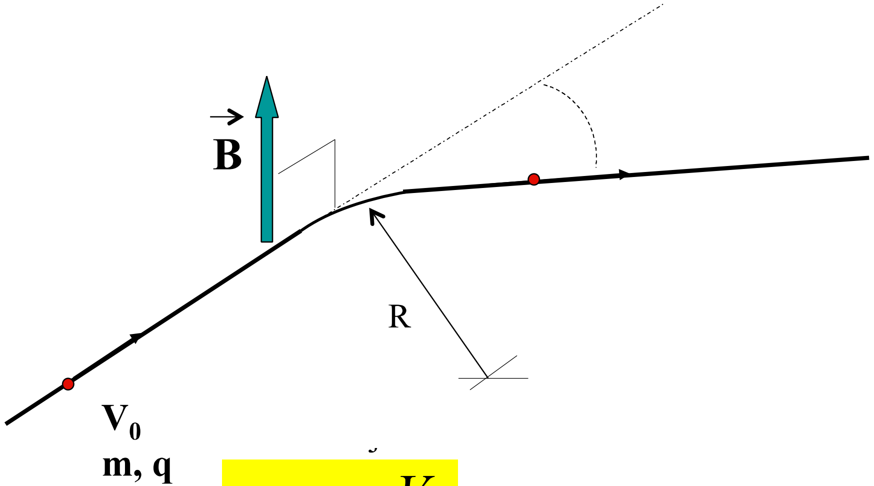
1. ATLAS Barrel Toroid – LHC 1.5 Tesla
2. CMS Solenoid -LHC 4 Tesla
3. LHC Quadrupole in LHe superfluid bath



Rational for superconductivity & cryogenics in particle accelerators

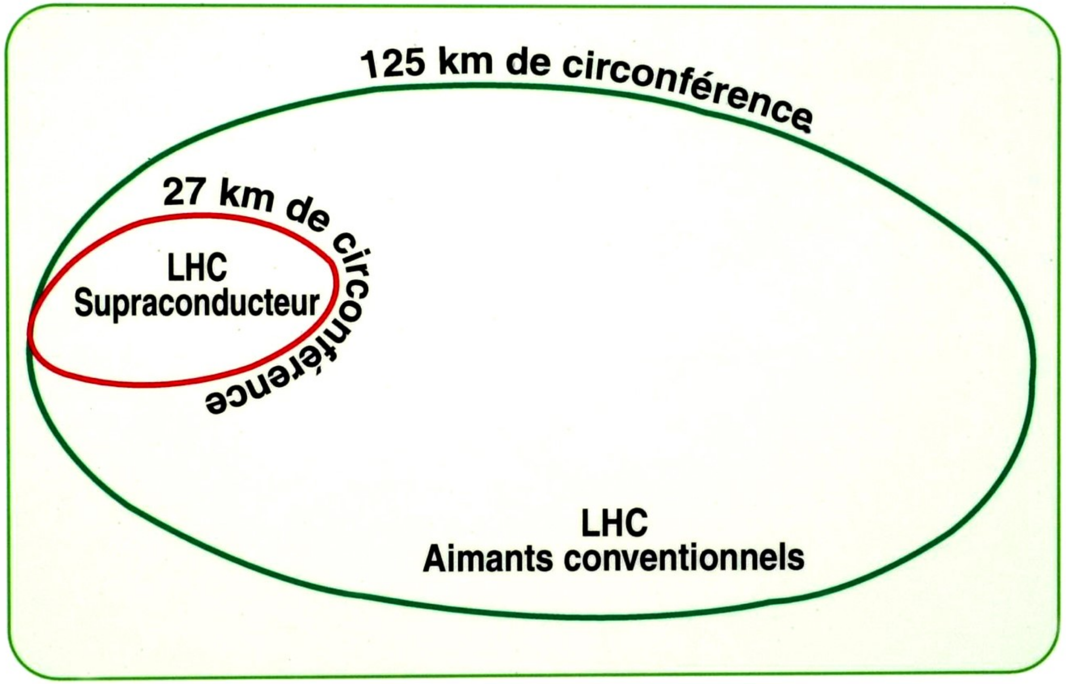


Compactness



$$R = \frac{m \cdot V_0}{q \cdot B}$$

Application: bending dipole

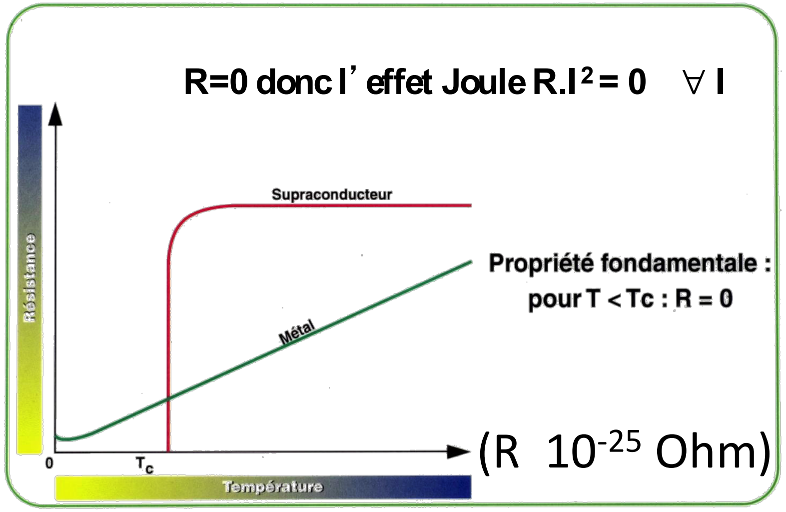


SC magnetic fields factor 5 w.r.t. normal magnets

LHC (SC NbTi): $B_{max} \approx 9$ T

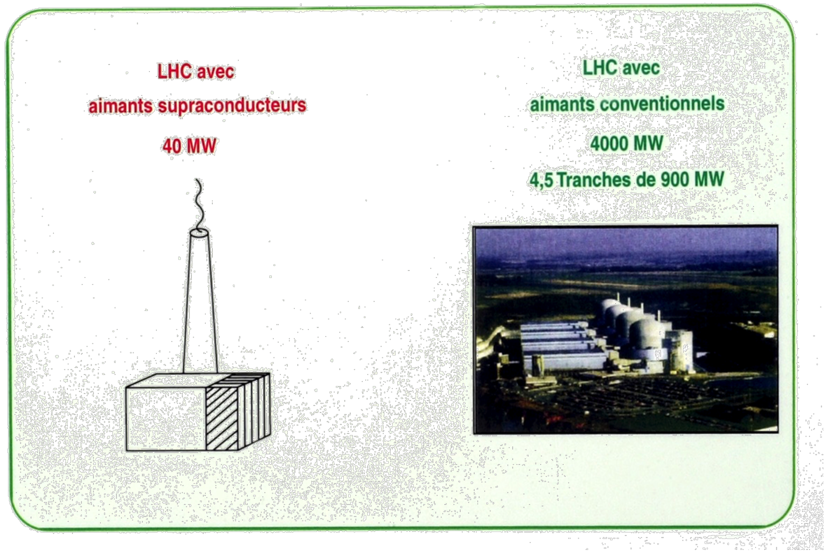
Normal magnets: $B_{max} \approx 1.8$ T (Fe saturation)

Power consumption

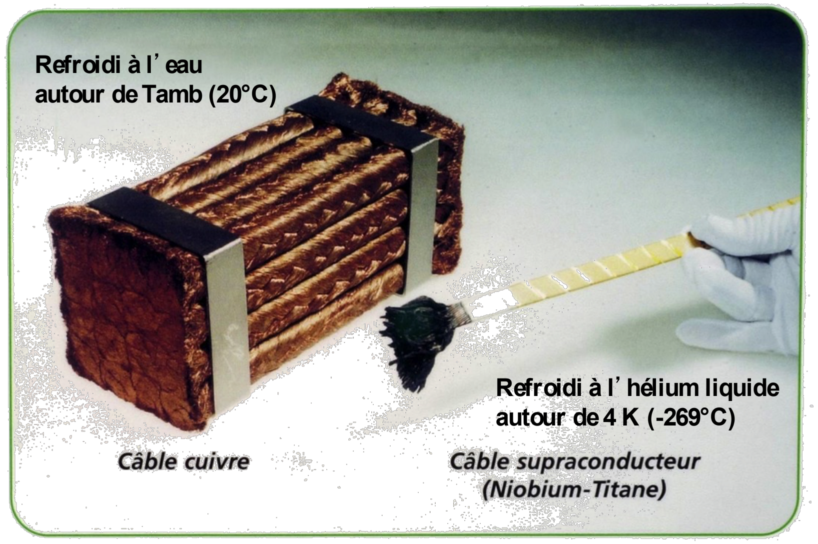


$(R\ \ 10^{-25}$ Ohm)

$P = I^2 R$



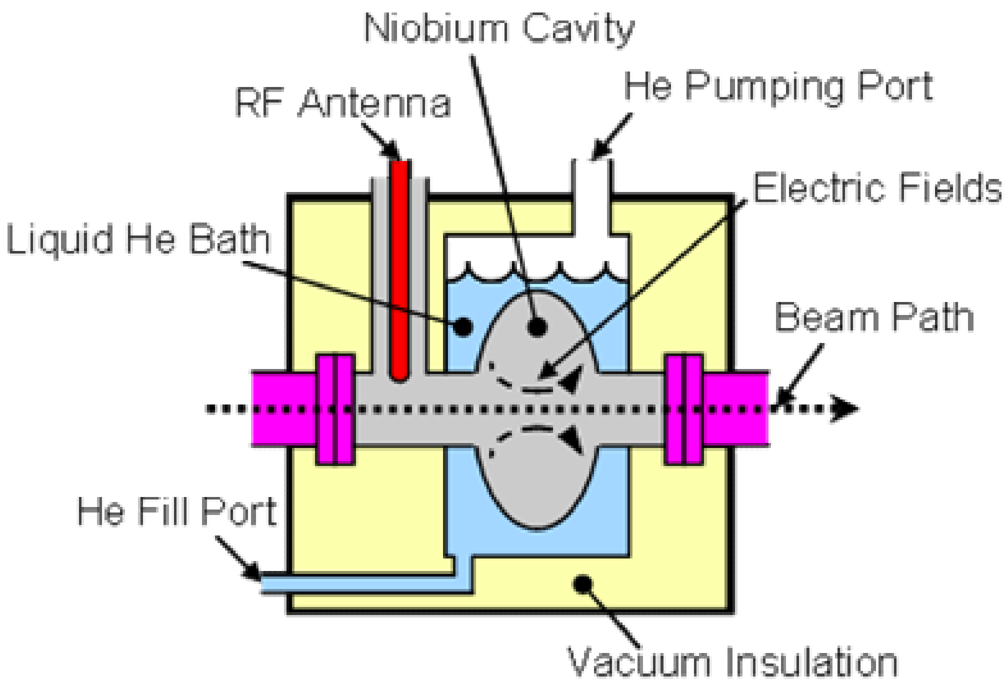
SC magnetic fields factor 5 w.r.t. normal magnets

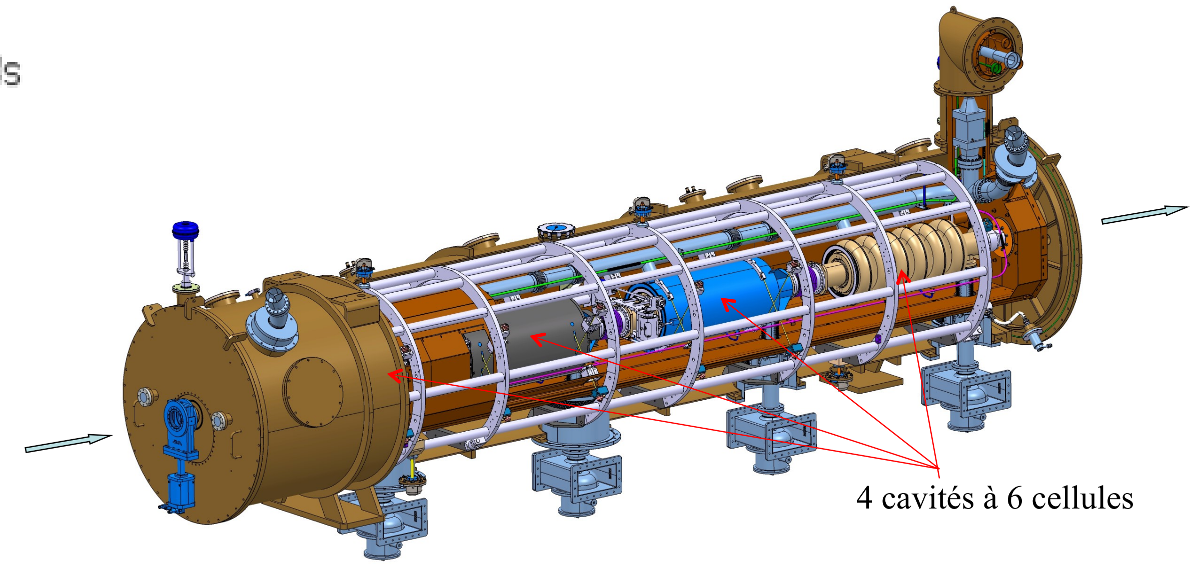


$P_{SC\ Magnet} \approx P_{refrigeration}$

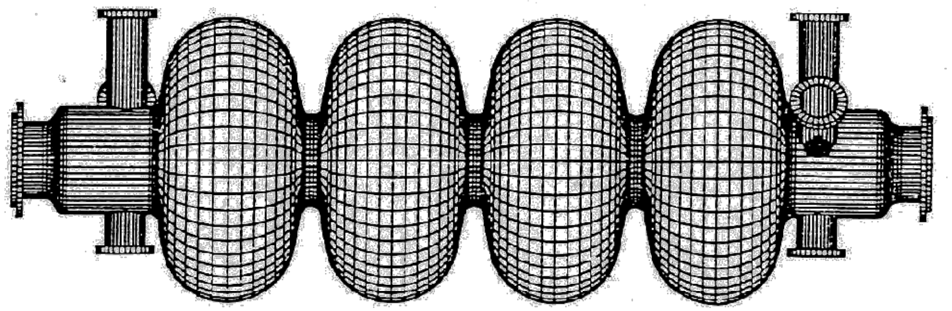
Reduced size of cables (both 15000 A)

SC cavities





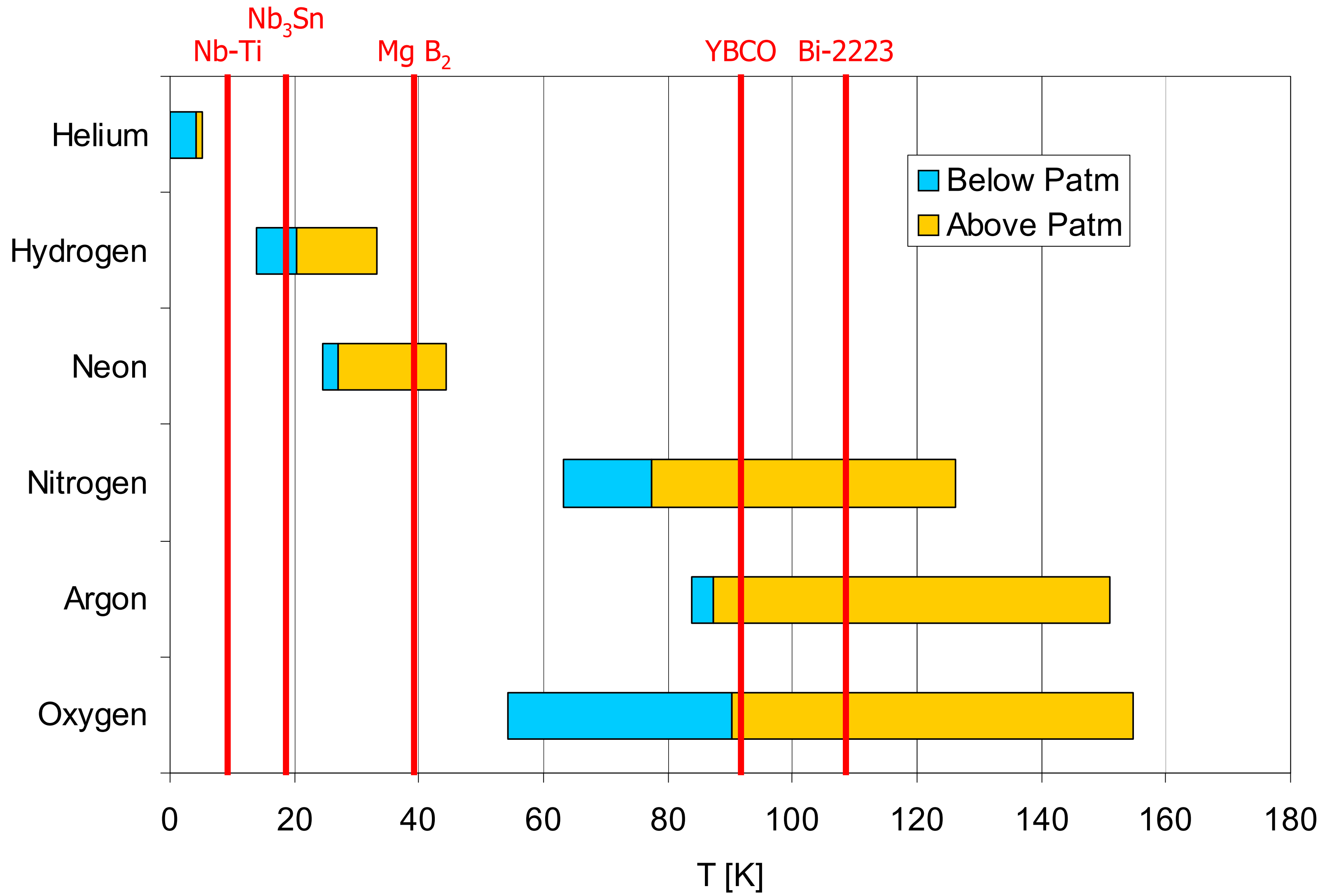
Cryomodule of ESS with 6 cells cavity
(Nb bulk SC cavities)



4 cells cavity

R in RF R doesn't go to 0

Useful range of cryogens & critical temperature of superconductors



LOW TEMPERATURE SC

HIGH TEMPERATURE SC

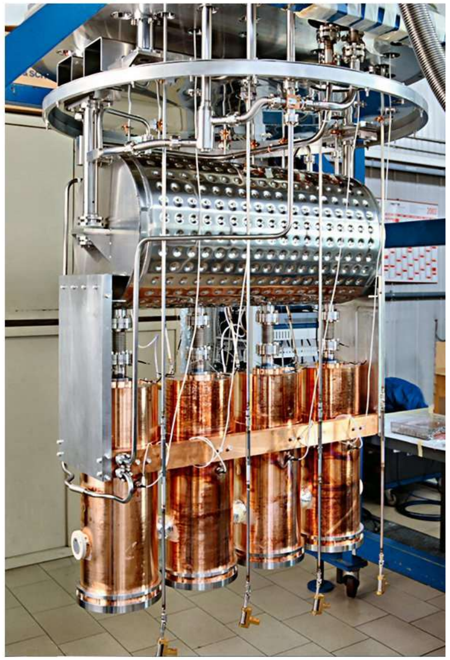
Cryogens for superconducting devices

- Helium is the only practical cryogen for LTS devices
  1. Normal LHe
  2. Superfluid LHe
- Subcooled nitrogen is applicable to HTS devices at low and moderate current density
- Thanks to its general availability and low cost, liquid nitrogen is very often used for precooling and thermal shielding of helium-cooled devices

LHe Liquid Inventory

- CERN: 130 t LHe
- The biggest LHe inventory in Italy is LNL: 2.3 t
    - ALPI linac (Acceleratore Lineare Per Ioni) @ LNL

Portion of the ALPI linac at LNL: cryostats for the superconducting cavities



Interior of a cryostat, lodging 4 accelerating, high purity copper-based, cavities (cavity inner surface is covered by a niobium layer).

LNGS inventory

LN_2 Storages

- LN_2 42000 kg (≈52000 L)

Total LN_2 Use

- 2017:

  Total 937820 Kg

  ≈8 trucks/month (ADR)

- 2018:
  - Total 1.031.649 Kg
  - ≈10 trucks/month (ADR)

Major cryogenic plants/activities (INFN sites)

- LNL (ALPI)
- LNGS
- LNS (SC cyclotron)
- LNF
- Milano LASA (XFEL, ESS, ..)
- Genova
- NA-Salerno

+ collaborations ENEA, CNR, …

- RFX: NBI (Neutral Beam Injector Facility -> ITER)

Applications: Cryogenic Detectors

- Some examples of cryogenic detectors (INFN)

  - CUORE — bolometric exp for $\beta\beta$ decay search
  - CUPID — bolometric exp for $\beta\beta$ decay search
  - CRESST — bolometric exp for Dark Matter search
  - COSINUS — bolometric exp for Dark Matter search
  - QUBIC — bolometric interferometer for cosmology
  - HOLMES — bolometric exp for neutrino mass measure

  - MEG — LXe scintillation detector
  - ATLAS — LAr calorimeter

  - GERDA — LAr cooled Ge exp for $\beta\beta$ decay search
  - Ge detectors — gamma spectroscopy

  - DARKSIDE — Dual Phase LAr TPC for Dark Matter search
  - XENON — Dual Phase LXe TPC for Dark Matter search
  - ICARUS — Single Phase LAr TPC on neutrino beam (LNGS, FNAL)
  - DUNE — Single Phase + Dual Phase LAr TPC on neutrino beam
    - PROTODUNE

Past gravitational wave cryo-antennas

NAUTILUS @ LNF
≈ 2 K

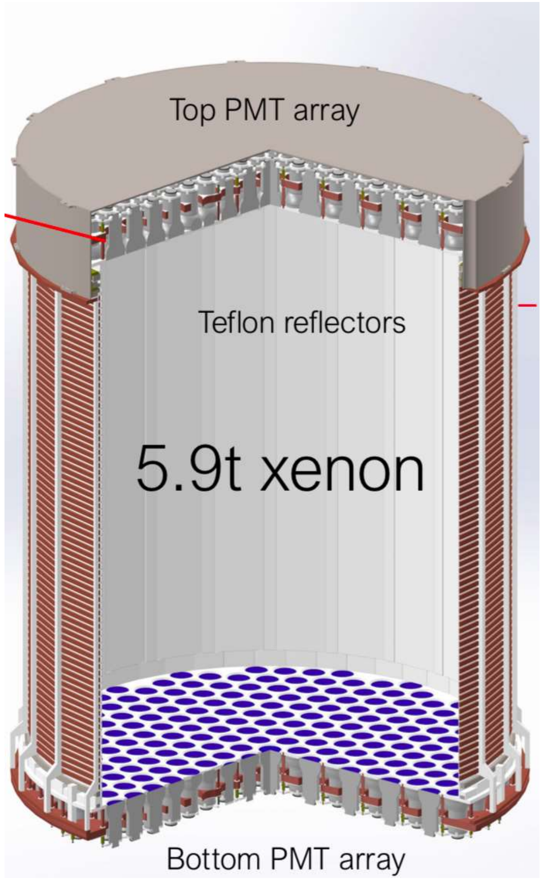
Network of 3 identical antennas :
2300 kg Al bar
(3 m long, 60 cm diameter)

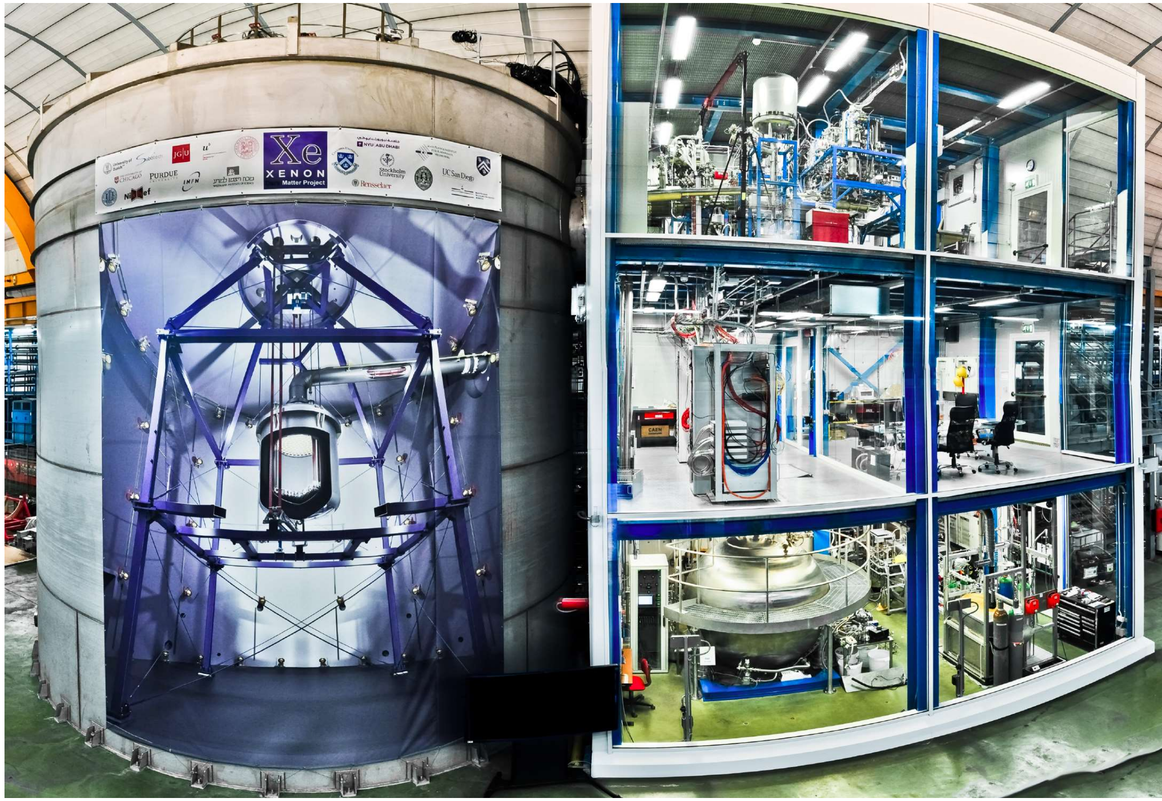
AURIGA @ LNL
≈ 50 mK

EXPLORER @ CERN
≈ 4 K

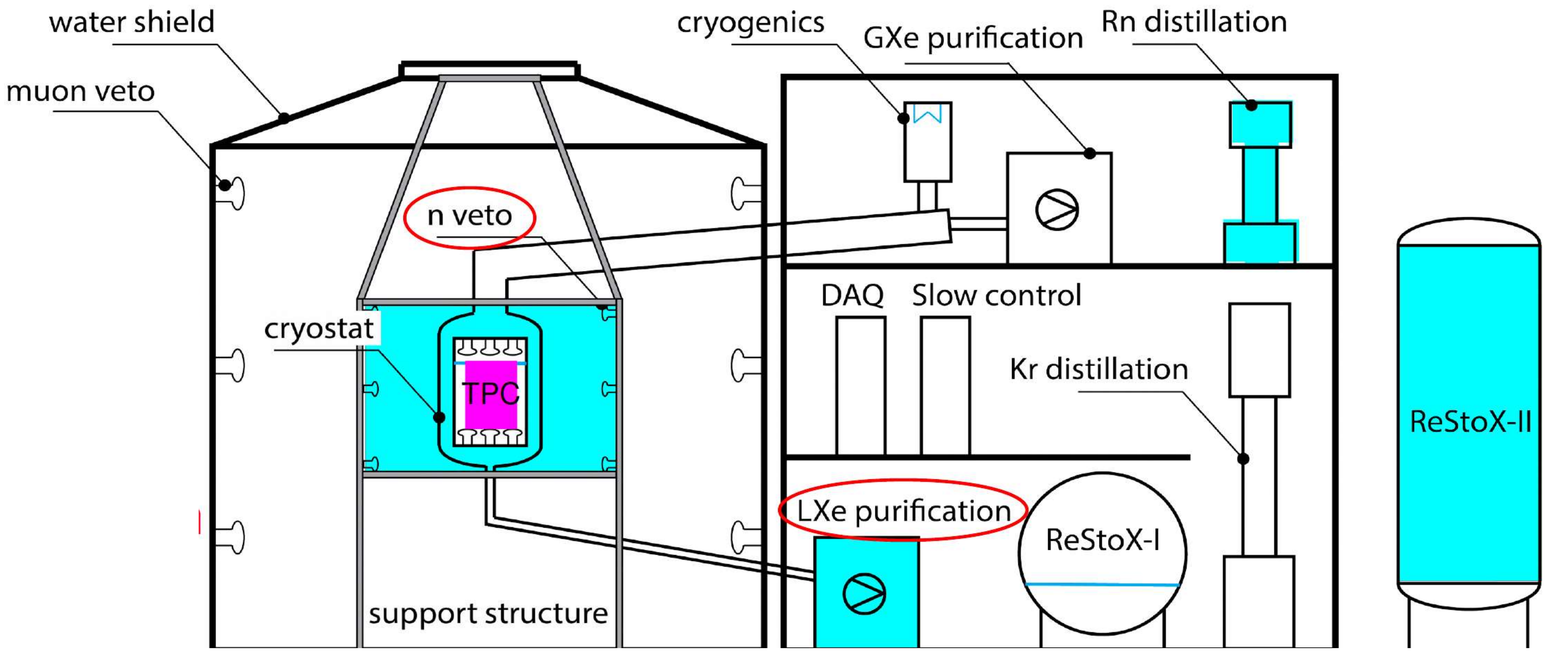
XENON 1T/nT

- Dual phase LXe TPC for dark matter
- LXe TPC stainless steel cryostat inside instrumented water tank
- Ongoing upgrade: 1 t -> n ton
- 5.9 t active Xe mass





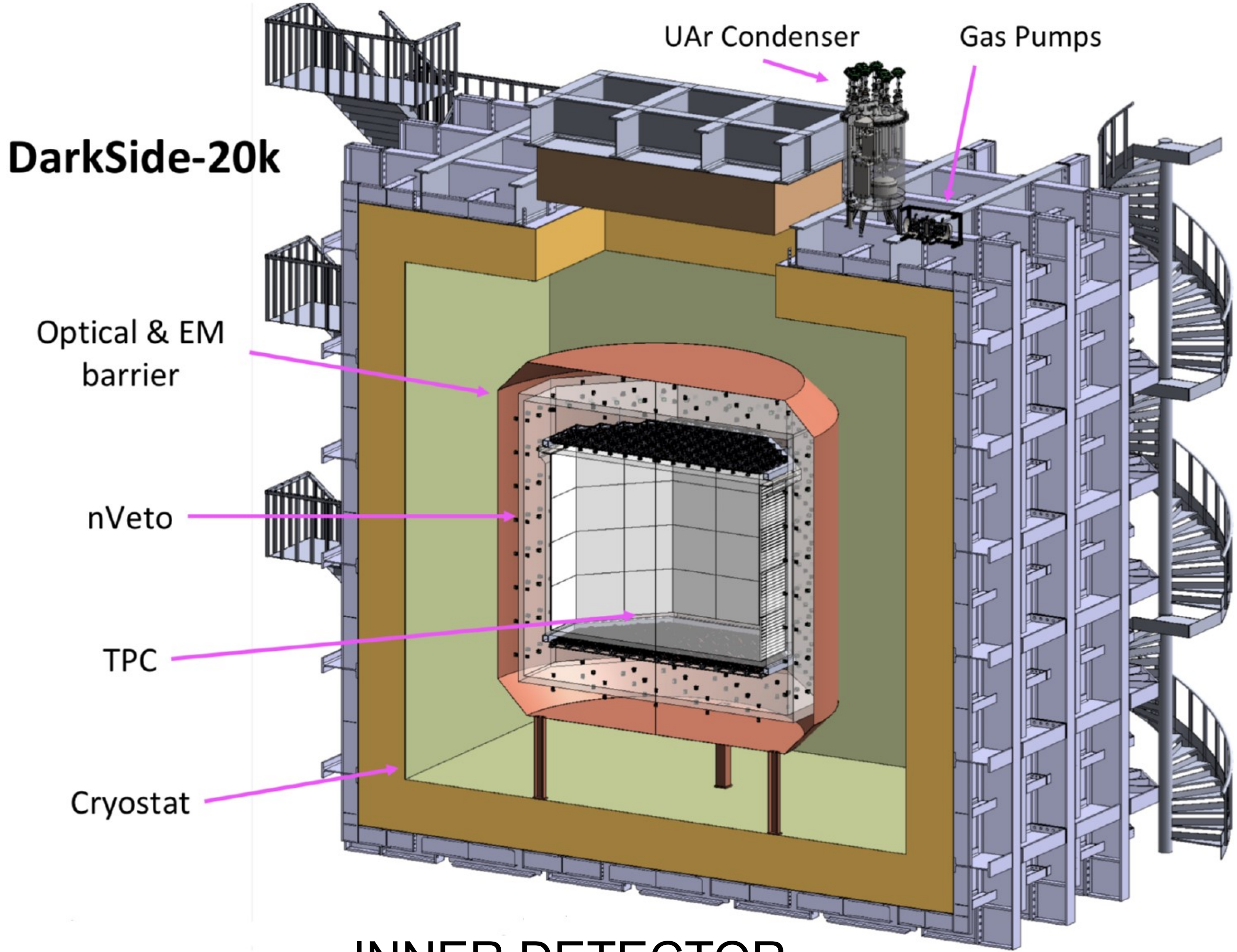
LXe: NBP 165.1 K, $T_{melting}$ 161.3 K, TP 161.4 K


water shield
muon veto
n veto
cryostat
TPC
support structure
cryogenics
GXe purification
Rn distillation
DAQ
Slow control
Kr distillation
LXe purification
ReStoX-I
ReStoX-II


Linde

DARKSIDE 20K PROJECT

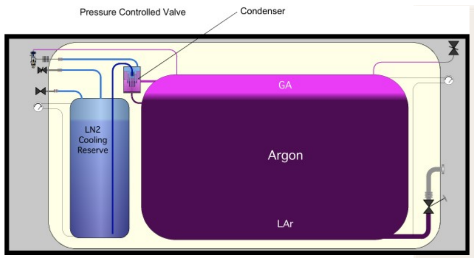
- Dual phase LAr TPC for dark matter
- Use of low radioctivity argon (UAr not AAr)
  - 50 t UAr in the TPC, 20 t FV
  - 700 t AAr
- URANIA Project (Colorado USA)
  - extraction of 50 t UAr from CO_2 deep wells (no cosmogenic ^{39}Ar)
  - Starting from 95% CO_2, 440 ppm of UAr
- ARIA Project (CarboSulcis mine, Sardinia)
  - ^{39}Ar isotopic separation with cryogenic distillation —> factor 10 suppression per pass (from UAr to DAr)
  - Seruci I 350m height, 30cm diameter column: removal of chemical impurities to make the UAr detector grade with 2 passes at 1t/day with 85% recovery—> inlet purity required by DS20k getters of order 0.25-1ppm

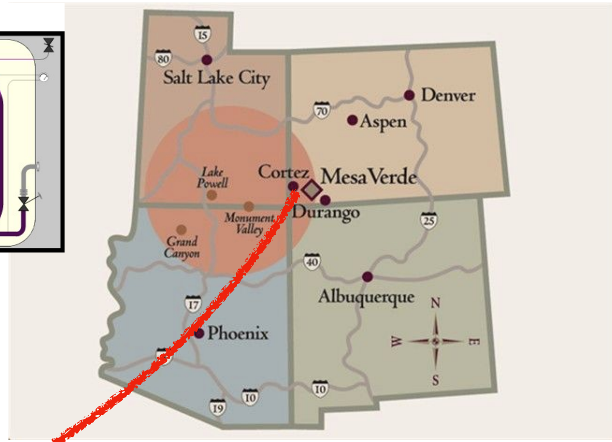


INNER DETECTOR
Acrylic vessel (no TPC cryostat)
50 t UAr (20 t fiducial volume)
Lar scintillation light readout: SiPM (30 m^2)

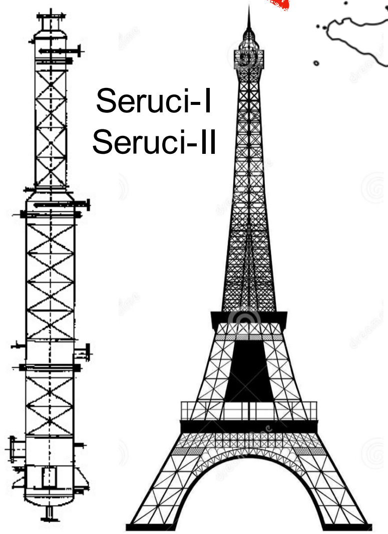
Underground Ar for DS-20k

- URANIA
- Procurement of 50 tonnes of UAr from same Colorado source as for DS-50
- Extraction of 250 kg/day, with 99.9% purity, 90 tonnes / year for the longer term
- UAr transported to Sardinia for final chemical purification at Aria





Seruci-0
Picture was taken during the installation

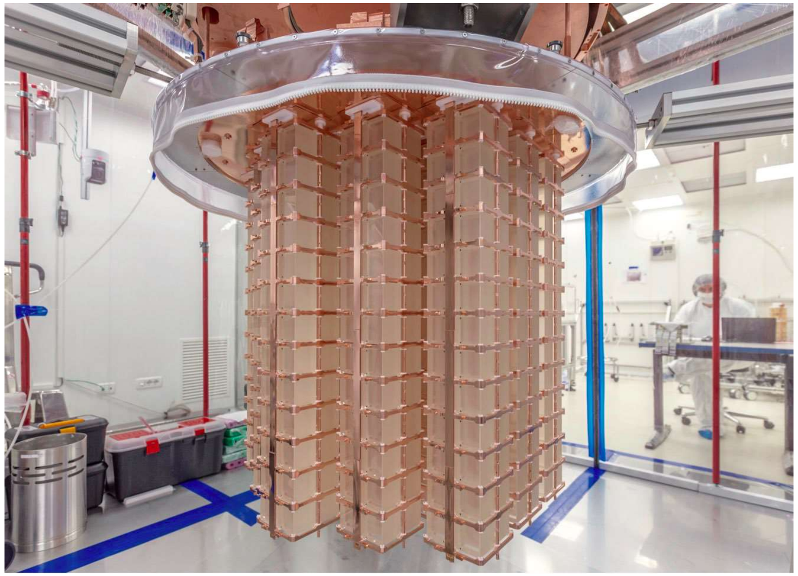


Seruci-I
Seruci-II

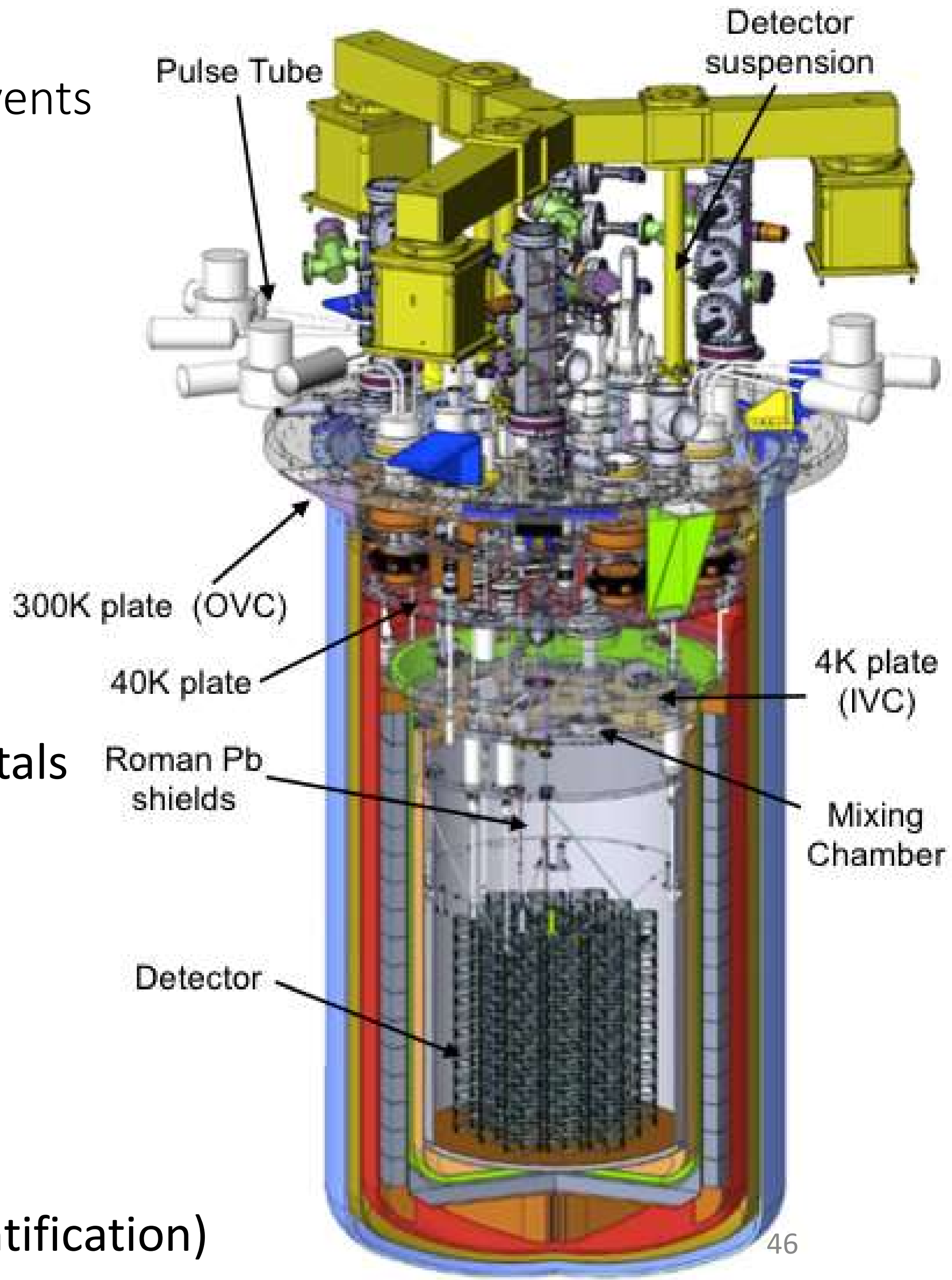
- ARIA
- Big cryogenic distillation column in Seruci, Sardinia, Italy
- Final chemical purification of the UAr
- Process O (1 tonne / day) with 10^3 reduction of all chemical impurities
- Isotopically separate ^{39}Ar from ^{40}Ar

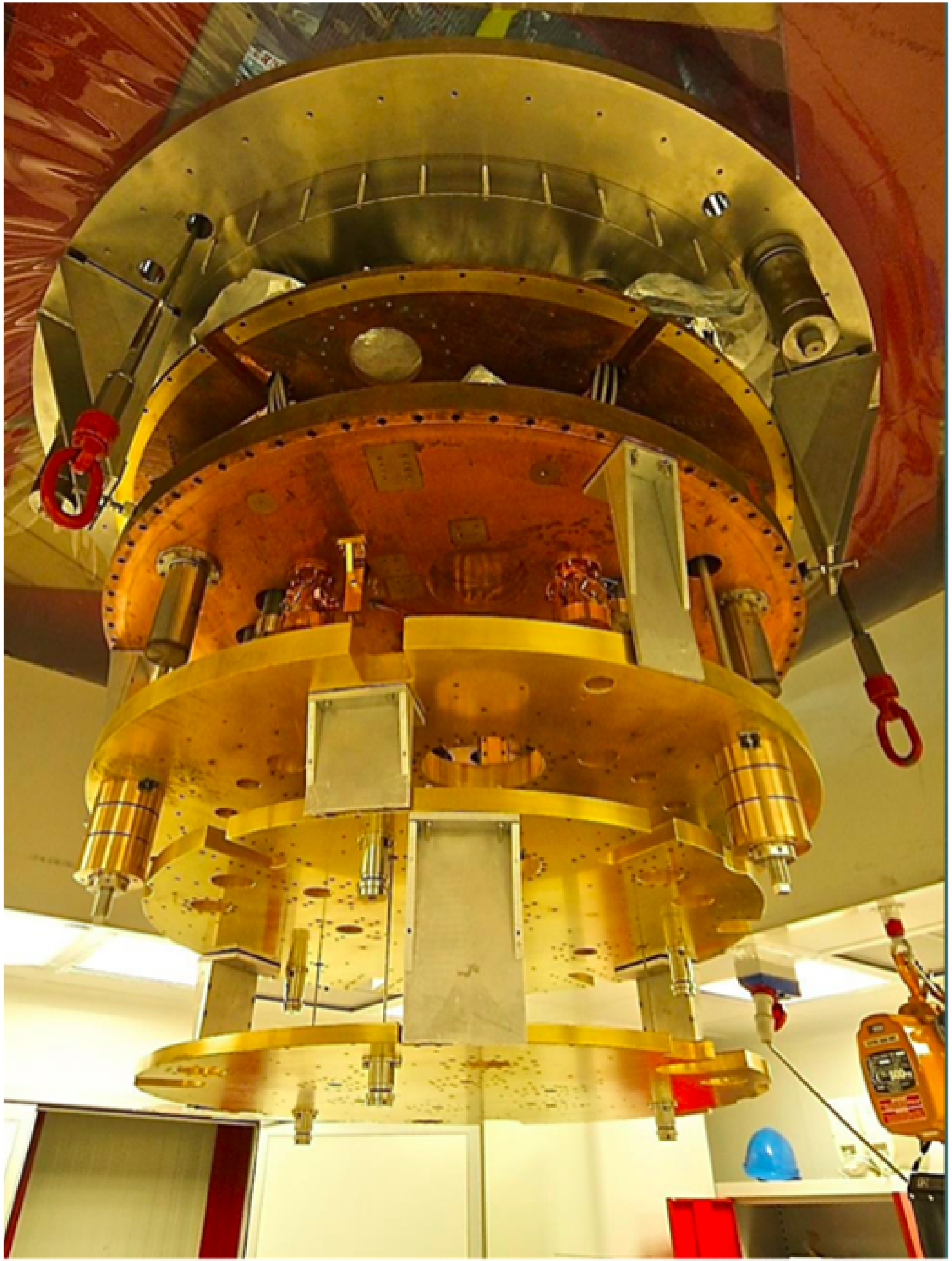
CUORE

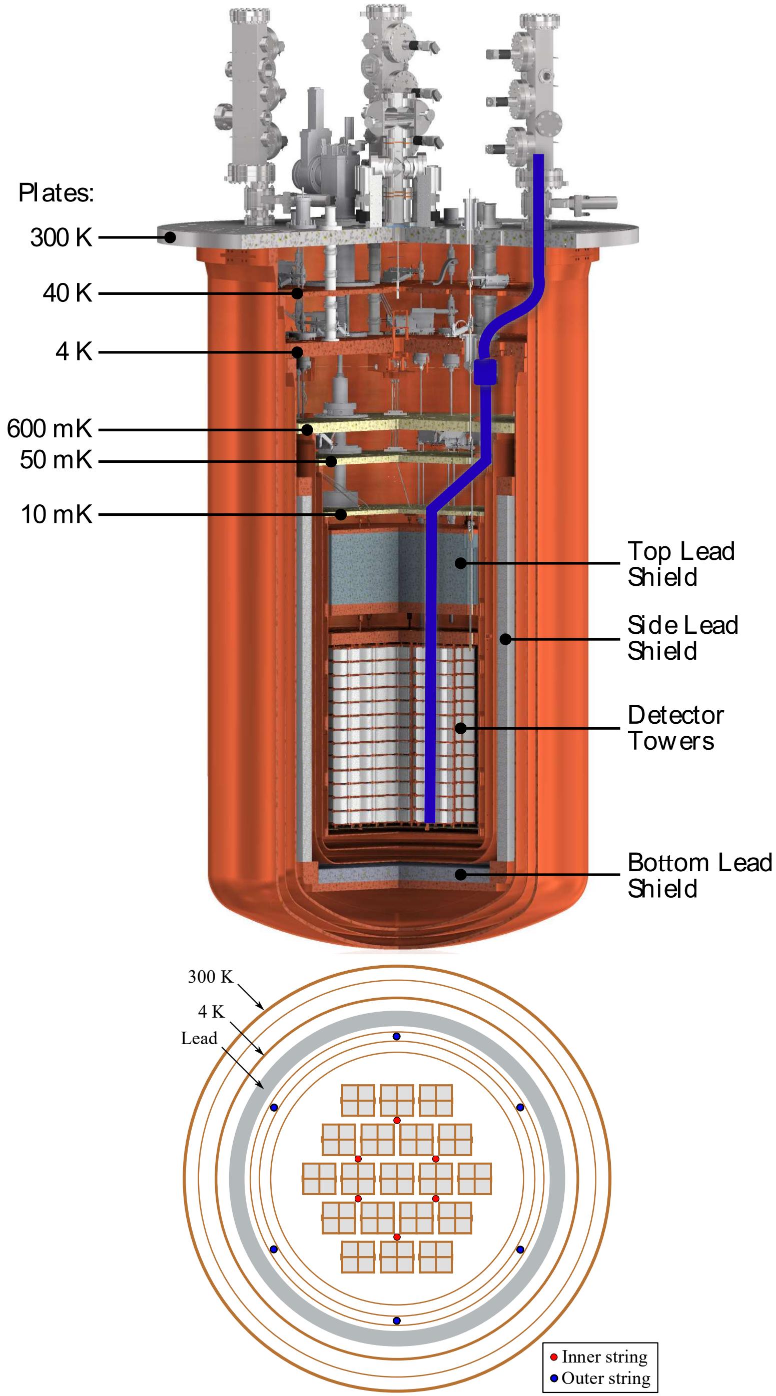
Cryogenic Uinderground Observatory for Rare Events



- Array of 988 TeO2 crystals (19 towers of 52 crystals 5~5~5 cm^3, 0.75 kg each)
- Mass of TeO_2: 742 kg (~206 kg of 130Te)
- Operating temperature: ~ 10 mK
- Mass to be cooled down: ~ 15 t (Pb, Cu, TeO_2)
- Background aim: 10^{-2} c/keV/kg/year
- Next: CUPID (CUORE Upgrade with Particle Identification)



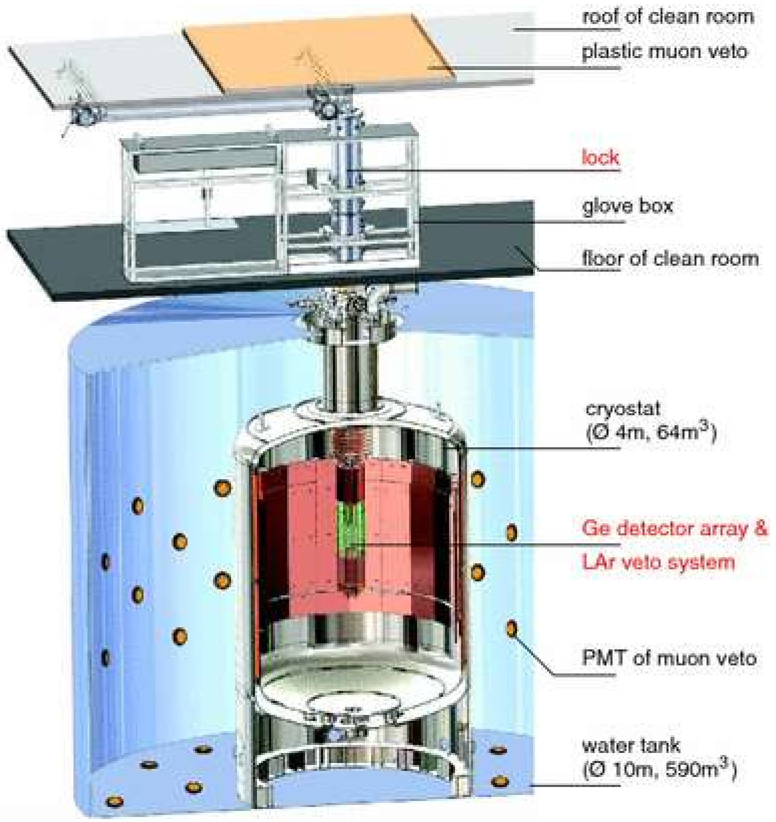


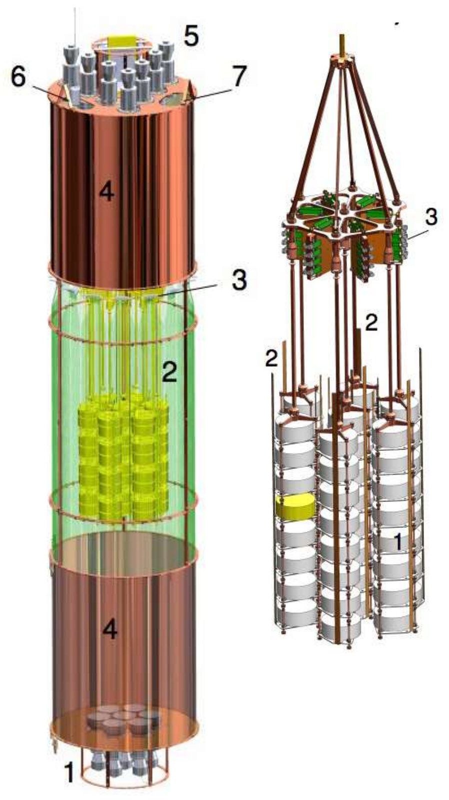

Plates:
300 K
40 K
4 K
600 mK
50 mK
10 mK
Top Lead Shield
Side Lead Shield
Detector Towers
Bottom Lead Shield
300 K
4 K
Lead
Inner string
Outer string

GERDA

GERmanium Detector Array

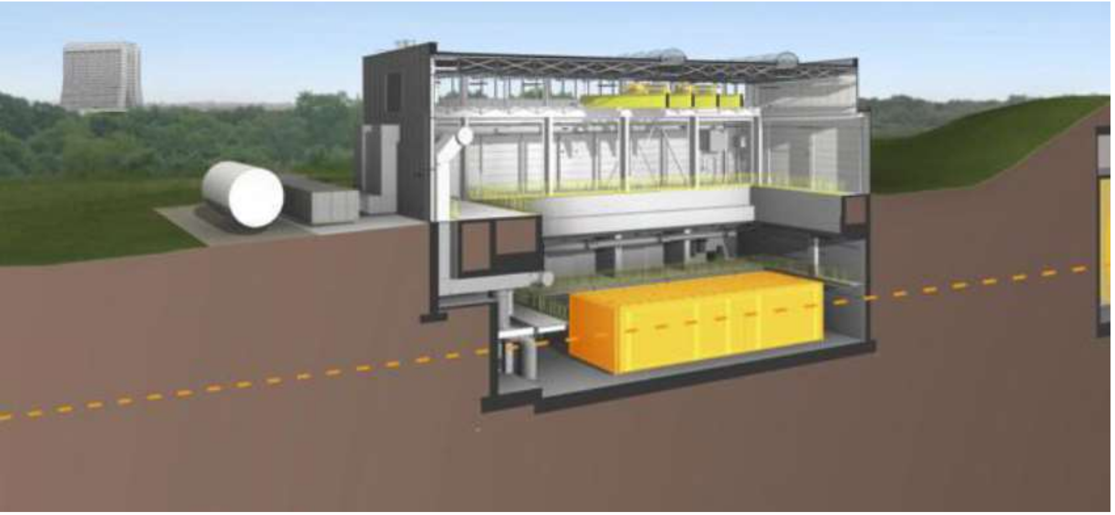
- High Purity Ge detectors enriched up to 88% in ^{76}Ge
- Inner detector in 64 m^3 LAr cryostat
- Active veto: 590 m^3 water tank





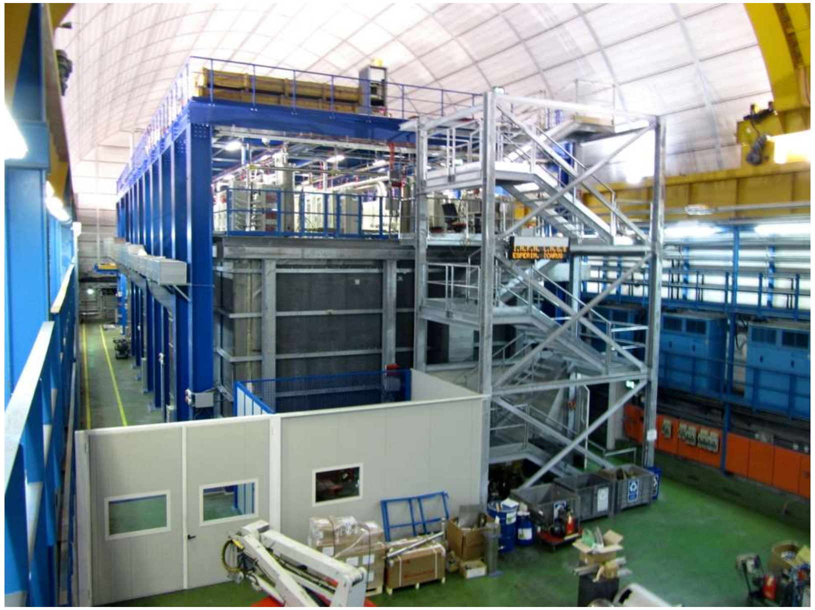
Next: LEGEND-200 Large Enriched Germanium Experiment for Neutrinoless bb Decay (200 kg)

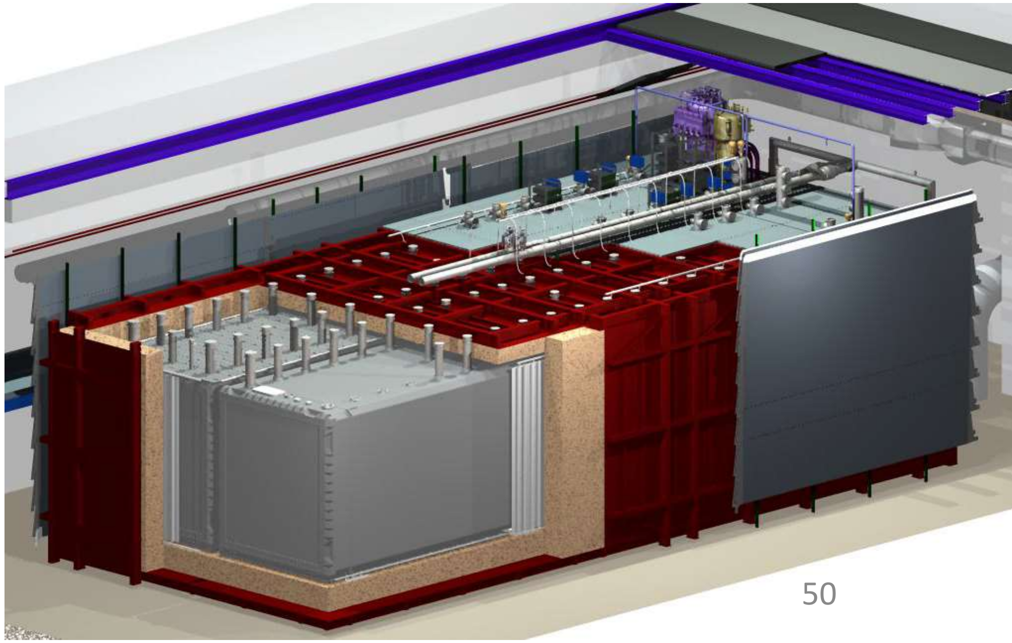
Short Baseline Neutrino Program @ FNAL



- 3 LAr detectors
- Far Detector: ICARUS T600
  - LAr TPC single phase
  - 4 volmues, 1.5 m drift
  - 55000 wires in total
- ➢ Redy for cooling

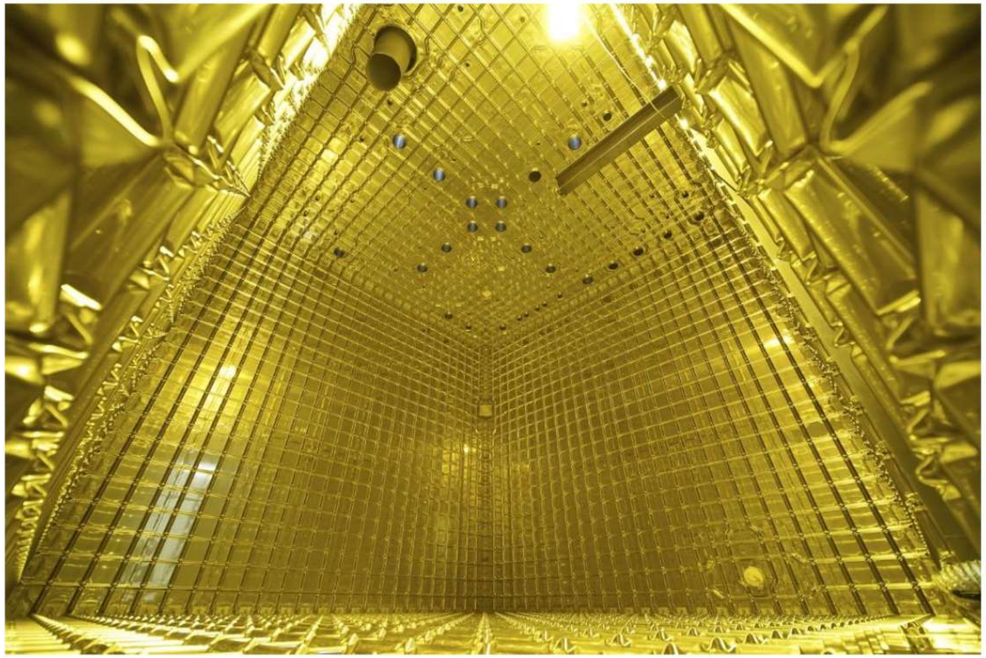
The ICARUS T600 trip





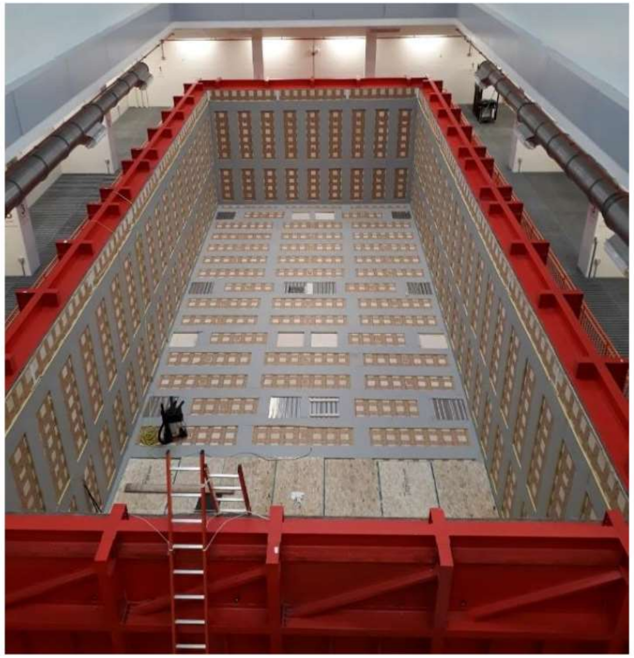
From Pavia to LNGS to CERN to FNAL

Membrane cryostat technology

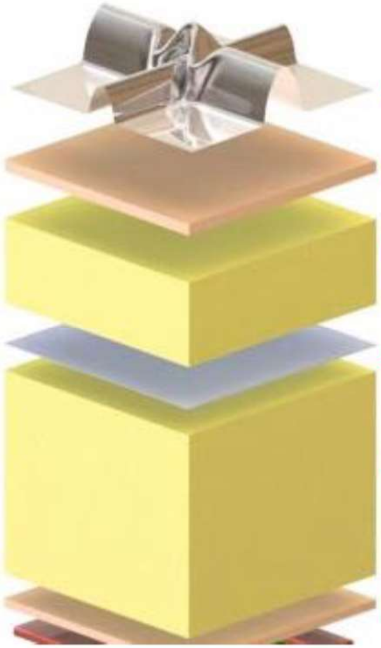


Close collaboration with industry (GTT). Membrane cryostat tech. developed for LNG transport ships

Re-engineered for LAr-TPC detectors



ICARUS: no membrane
Vacuum-pumped cryostats



SS primary membrane in contact with the LAr

Plywood

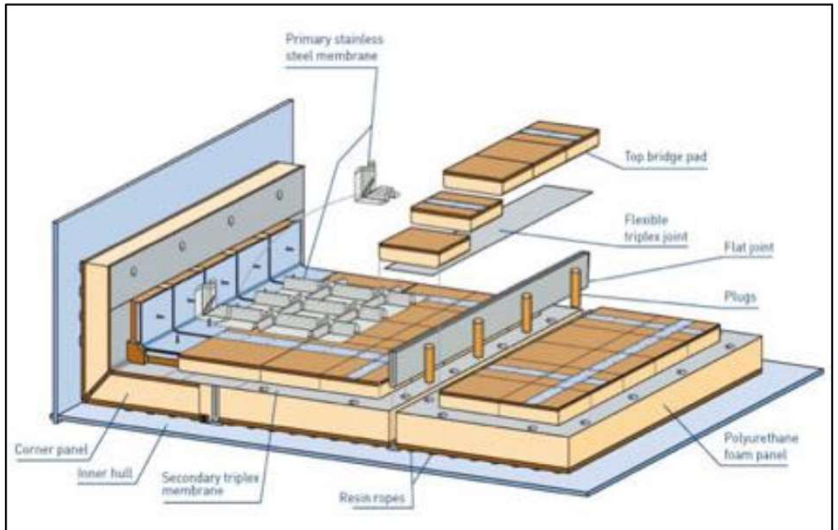
Insulation: reinforced polyurethane foam (LNG tech)

Secondary membrane for gas containment

Insulation

Plywood

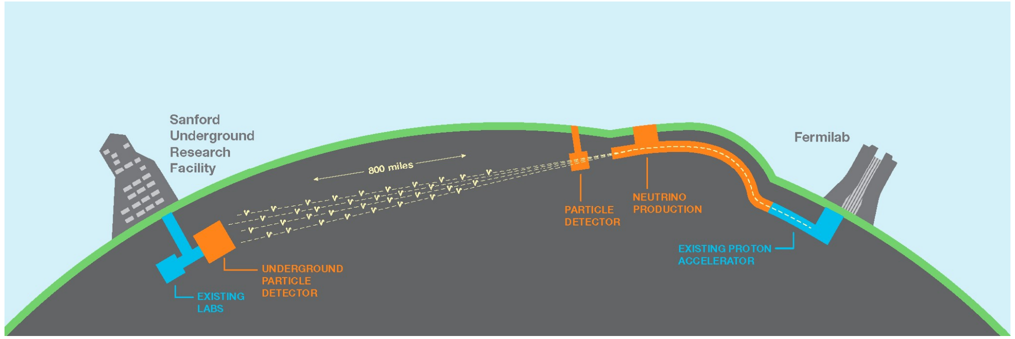
Membrane Cryostats:
No vacuum – Argon purge

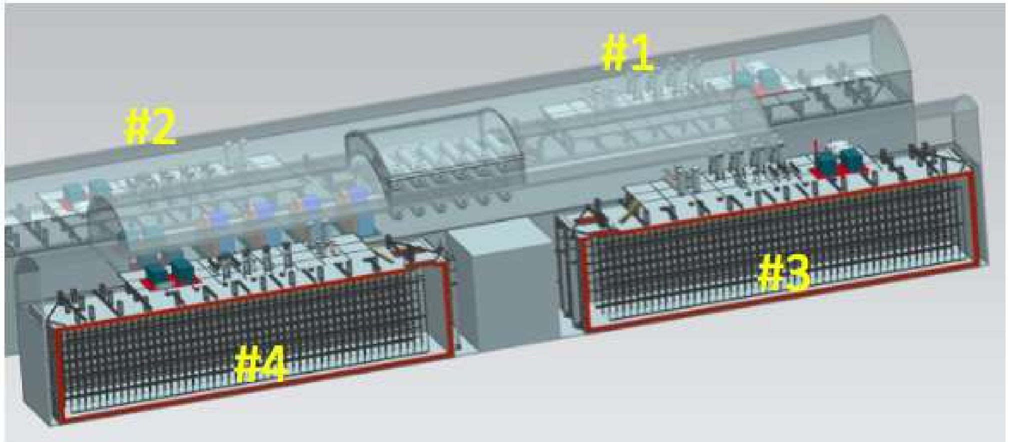


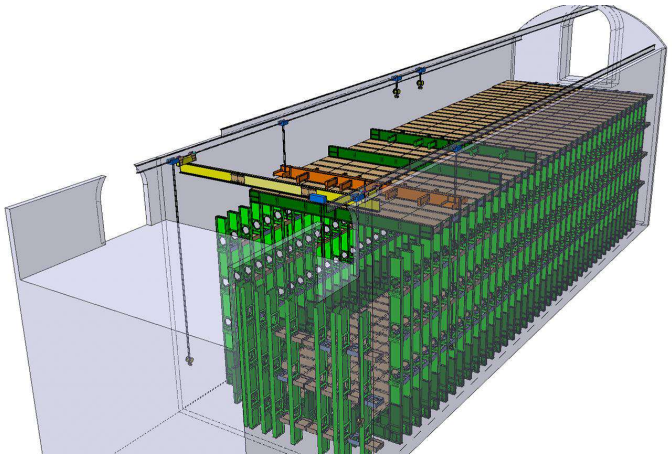
600 mm (bottom, sides), 400 mm top

DUNE

Deep Underground Neutrino Experiment

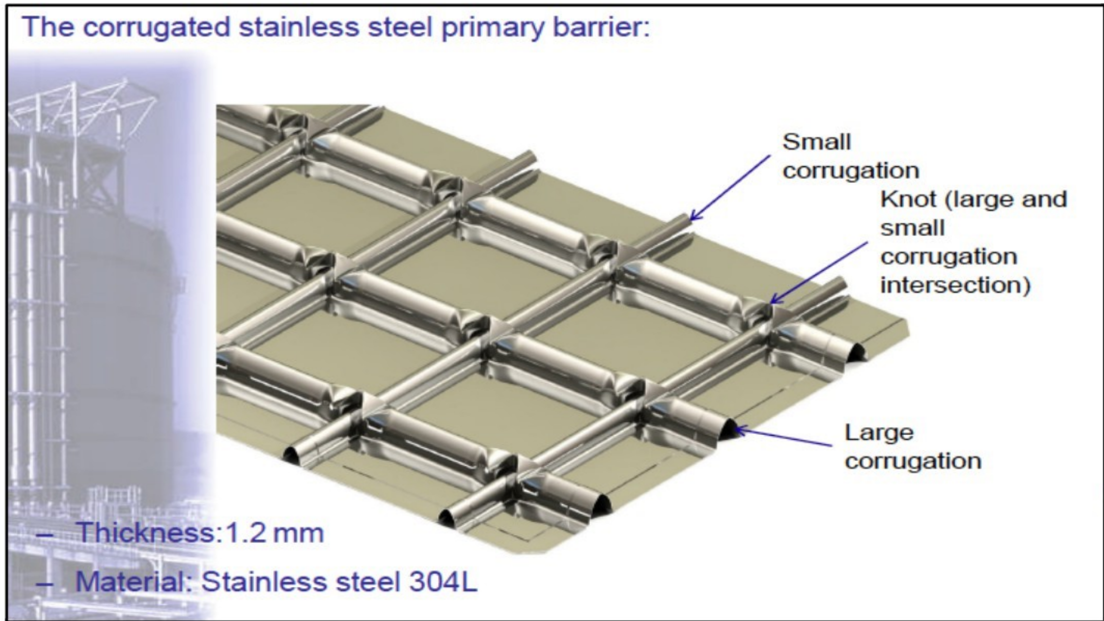


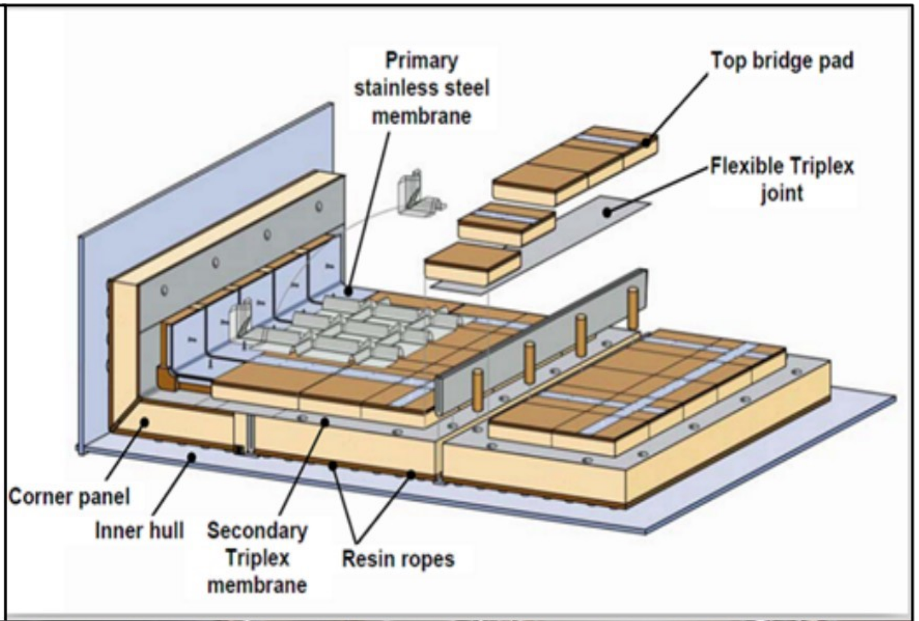


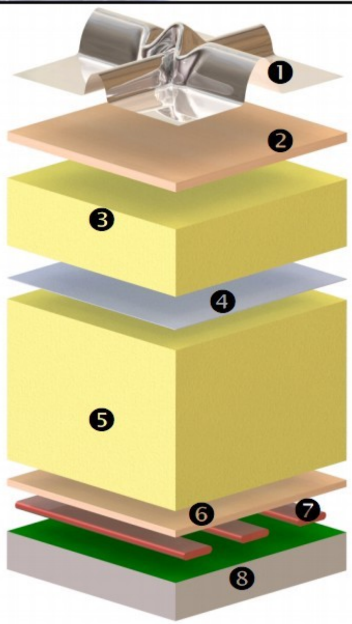


- Neutrino beam from FNAL to SURF (Sanford Underground Research Facility, South Dakota), 1300 km apart
- 4 x ≈ 10 kton DUNE far detector LAr TPC, both Single and Dual Phase (the first one Single Phase)
- Module outer (inner) dimensions: 19.1(15.1)x66(62)x18h(14 h) m^3
- TPC dimensions:
  (3.6+3.6+3.6+3.6) x 58 x 12 h m^3
  - total mass 17.1 kt
  - active mass 13.8 kt
  - fiducial mass 11.6 kt
- Membrane cryostat technology

DUNE Membrane cryostat



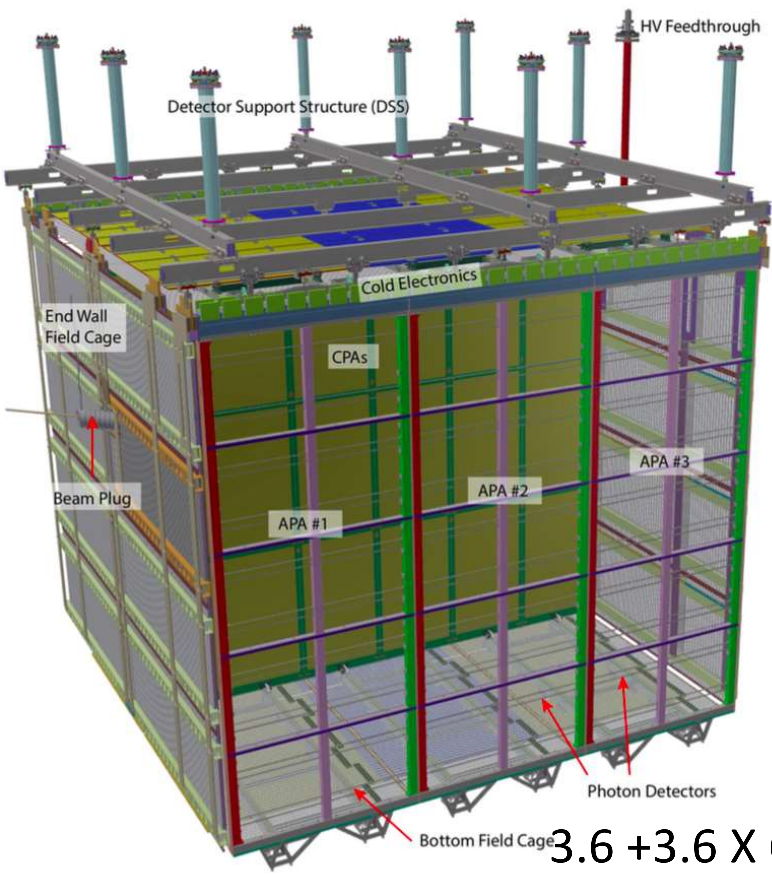




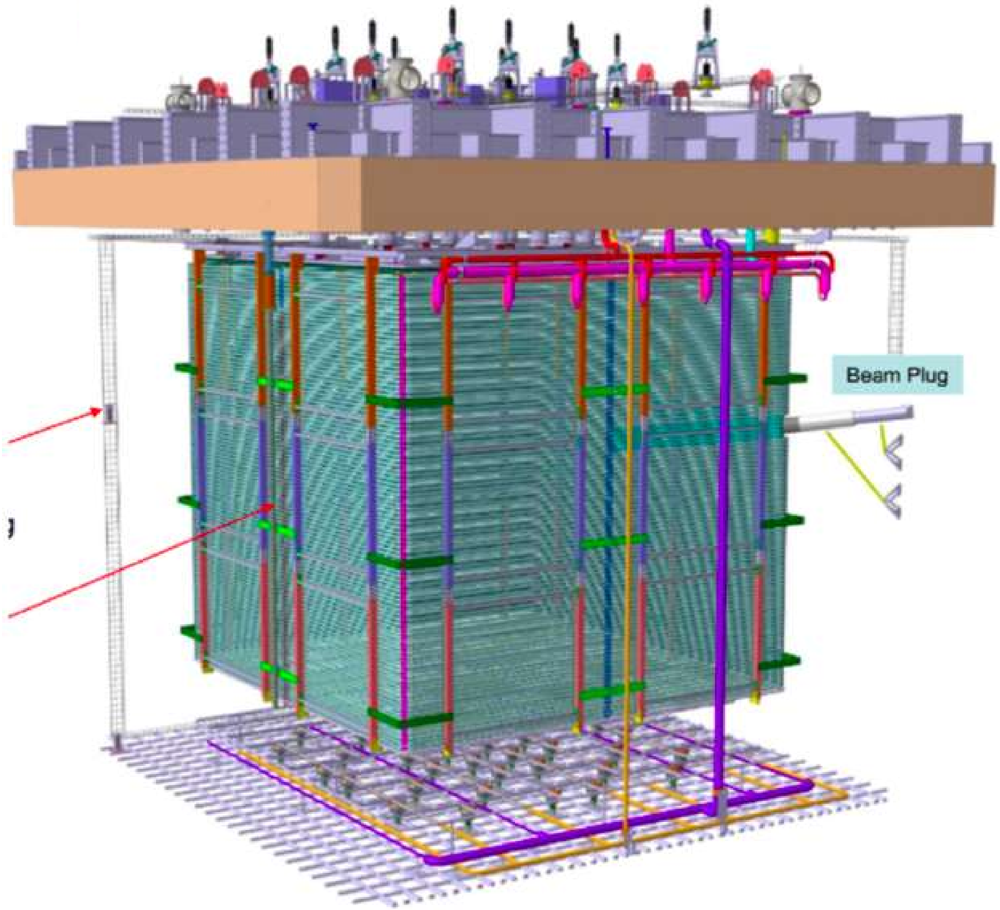
1. Stainless steel primary membrane
2. Plywood board
3. Reinforced polyurethane foam
4. Secondary barrier
5. Reinforced polyurethane foam
6. Plywood board
7. Bearing mastic
8. Steel structure with moisture barrier

PROTODUNE(S)

770 (420) ton total (active) LAr mass each



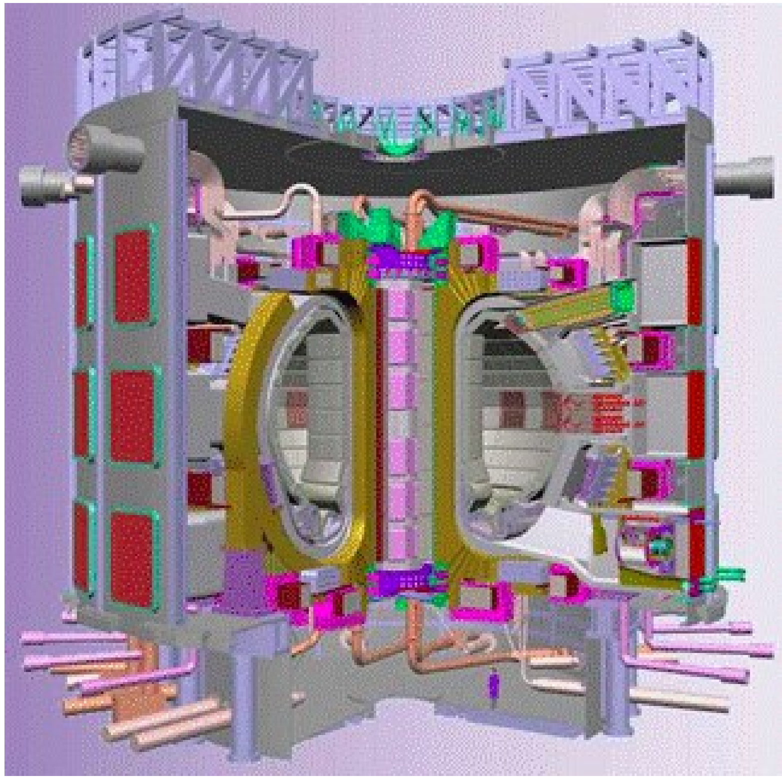
3.6 +3.6 X 6.9 X 6 H m^3



- Two large scale prototypes for the DUNE Far Detector (760 tons of LAr).
- Designed and constructed to perfectly reproduce the elements of the final DUNE FD.
- Full characterization of detector performance on charged particles beams at CERN


Protodune DP
Protodune SP

Application: Nuclear fusion



- ITER (International Thermonuclear Experimental Reactor) for nuclear fusion
- Cryogenics is involved in
  1. Cryopumping
  2. NbTi/Nb3Sn SC magnets cooling

Applications: Space

- Rocket propulsion
  - Cryogenic engines are powered by cryogenic propellants.
  - Liquid Hydrogen is used as a fuel to propel the rocket.
  - Liquid Oxygen is used as an oxidizer.
- Space instrumentation
  - Cooling of IR detectors, Telescopes, Cold probes, etc. are some of the major applications of cryogenics.
  - Development of miniature and small cryocoolers for satellites for an improved accuracy and reliability of earth observation.
- Space simulation chambers
  - Space simulations chambers are realistic environment for space craft. The cold space is simulated at cryogenic temperatures by use of LN2.
  - The levels of vacuum required in space simulation chambers are very high. This is achieved by the use of cryo pumps and turbo molecular pumps.

Ariane 5
(25 t liquid hydrogen, 130 t liquid oxygen)

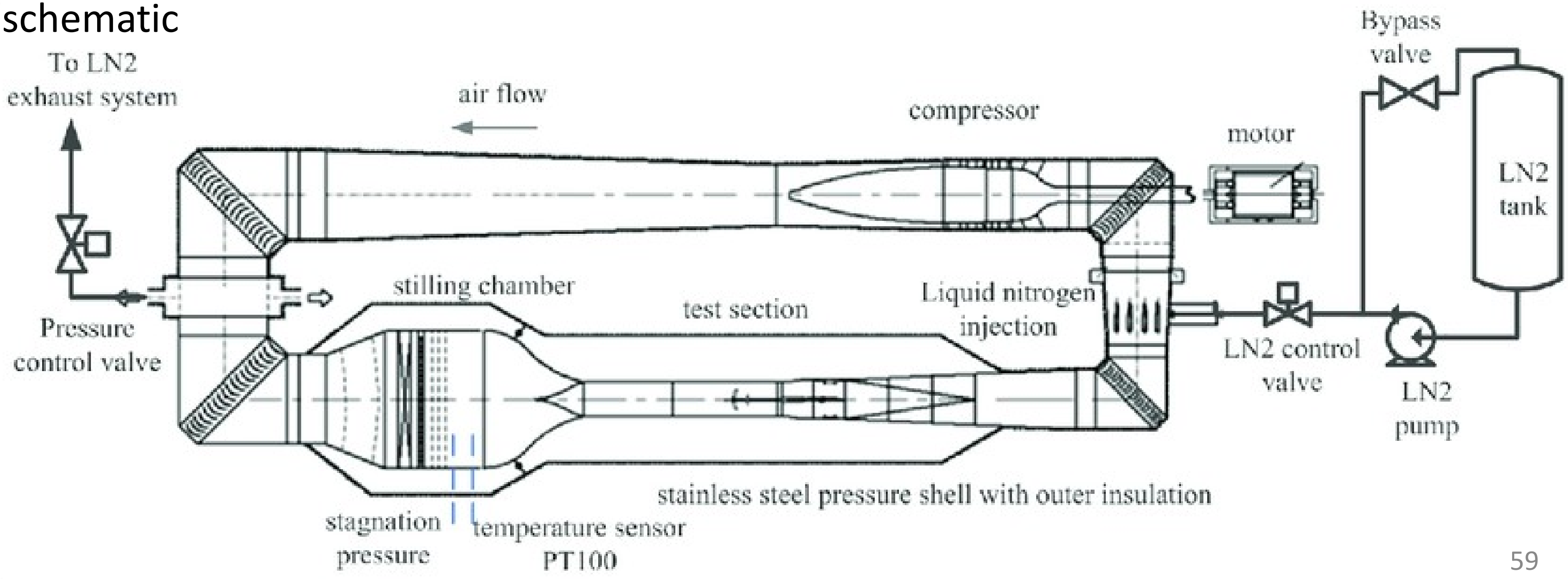
Space Shuttle
(100 t liquid hydrogen, 600 t liquid oxygen)

Cryogenic Wind Tunnels

- Fluidodynamic tests carried out in “cryogenic-driven” wind tunnels
  - LN_2 panels acting as a giagantic cryopump

- Picture: Cryogenic ROHR WIND TUNNEL (KRG) in Gottingen: research and development facility for aerodynamic experiments

schematic



Applications: Gas Industry

- **Air Liquefaction, Air Separation and Storage (liquid and gas)**
- The transportation of gases across the world is done in liquid state. This is done by storing the liquid at cryogenic temperature.
- Liquid nitrogen is used as precoolant in most of the cryogenic systems. It is mainly a by-product of the LO_2 production.
- **Steel industry**: Oxygen is used in the production of steel. Basic Oxygen Furnace (BOF) uses oxygen instead of air.
- Nitrogen and argon are primarily used to provide an inert atmosphere in chemical, metallurgical and welding industries.
- Food preservation
- The use of inert gases in **welding industry** has initiated higher demand for gas production in the recent past.
- Cryogens like LO_2, LH_2 are used in **rocket propulsion** while LH_2 has been considered in the past for **automobile**.

Applications: LH_2 as a fuel

Attempt to use liquid hydrogen fuel in automotive

Air liquefaction and separation

Two options for air separation:

1. Cryogenic air separation

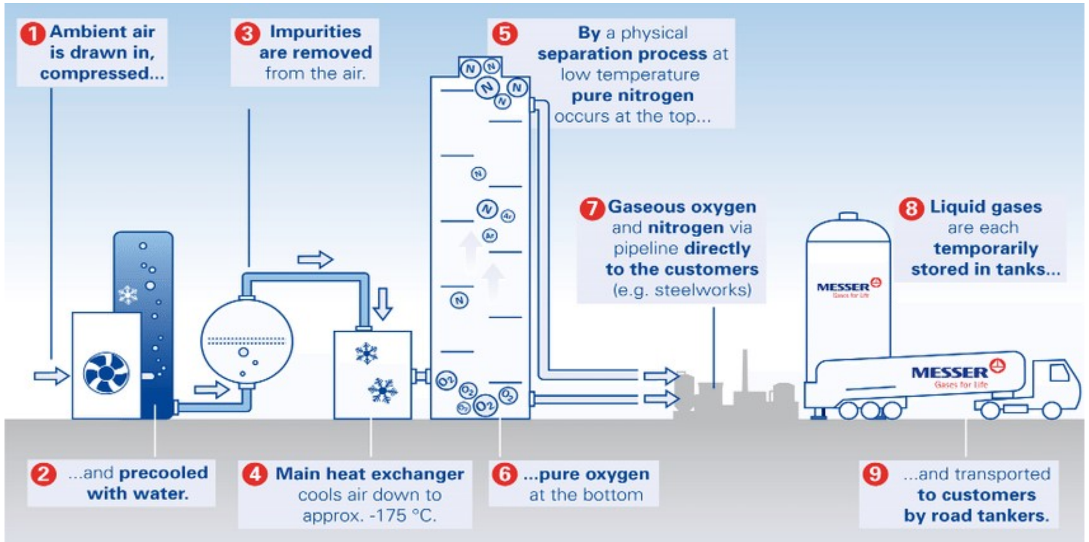
   uses the difference in the boiling points of nitrogen, oxygen and argon to separate and purify those products.

   LHe is not produced by air.

2. Non-cryogenic air separation

   uses physical property differences such as molecular size and mass, to produce nitrogen and oxygen at sufficient purity to meet the needs of users.

   Less pure than cryogenic separation.



LIN & LOX
Cryogenic air separation plant with heat exchanger and distillation column towers (source Air Products)
Up to 4500 t/day LOX

LNG transport

130 000 m3 LNG carrier with integrated Invar tanks - double hull (source Gaztransport)

266,000 m3 MOZAH (biggest tank in the world) Q-MAX LNG CARRIER

- LNG gasification plants can be located on land as well as on floating barges.
- In a conventional regasification plant, LNG is heated by sea water to convert it to natural gas / methane gas (open or closed circuit).
- Only 3 LNG terminals in Italy (7 in Spain).
- LNG started to be used as fuel on boats

Use of methane

Nave metaniera, la Umm Bab, in arrivo dal Qatar (12 giorni di navigazione), il terminale di rigassificazione offshore di Porto Levante, in provincia di Rovigo, situato a 15 chilometri dalla costa veneta.

Carico di circa 145.000 metri cubi di gas naturale liquefatto (GNL), corrispondenti a quasi 90 milioni di metri cubi di gas che rappresentano il consumo medio annuo di oltre 60.000 famiglie italiane (indicativamente il fabbisogno nel 2012 di una regione come la Valle d'Aosta).

Inaugurato nell'ottobre del 2009, il terminale gestito da Adriatic LNG ha segnato l'inizio di una nuova fase nel sistema di approvvigionamento energetico nazionale, garantendo all'Italia non solo i rifornimenti di GNL dal Qatar ma anche dall'Egitto, da Trinidad & Tobago, dalla Guinea Equatoriale e dalla Norvegia.

LNG market

- LNG has played an important role in the global energy system over the last few decades, as an increasing number of countries have turned to natural gas to meet their growing energy needs. **LNG trade increased from 100 million tonnes in 2000 to 319 million tonnes in 2018.**
- **Strong demand for cleaner-burning fuel in Asia continued to drive rapid growth in liquefied natural gas (LNG)** use in 2018, with global demand rising by 27 million tonnes to 319 million tonnes, according to Shell's latest annual LNG Outlook. Shell expects demand to reach **about 384 million tonnes in 2020** (growth expected to be absorbed by Europe and Asia).
- **China became the world's largest gas importer**, with LNG imports doubling over two years.
- Coal-to-gas switching led to 78% improvement in Beijing winter air quality over the last five years

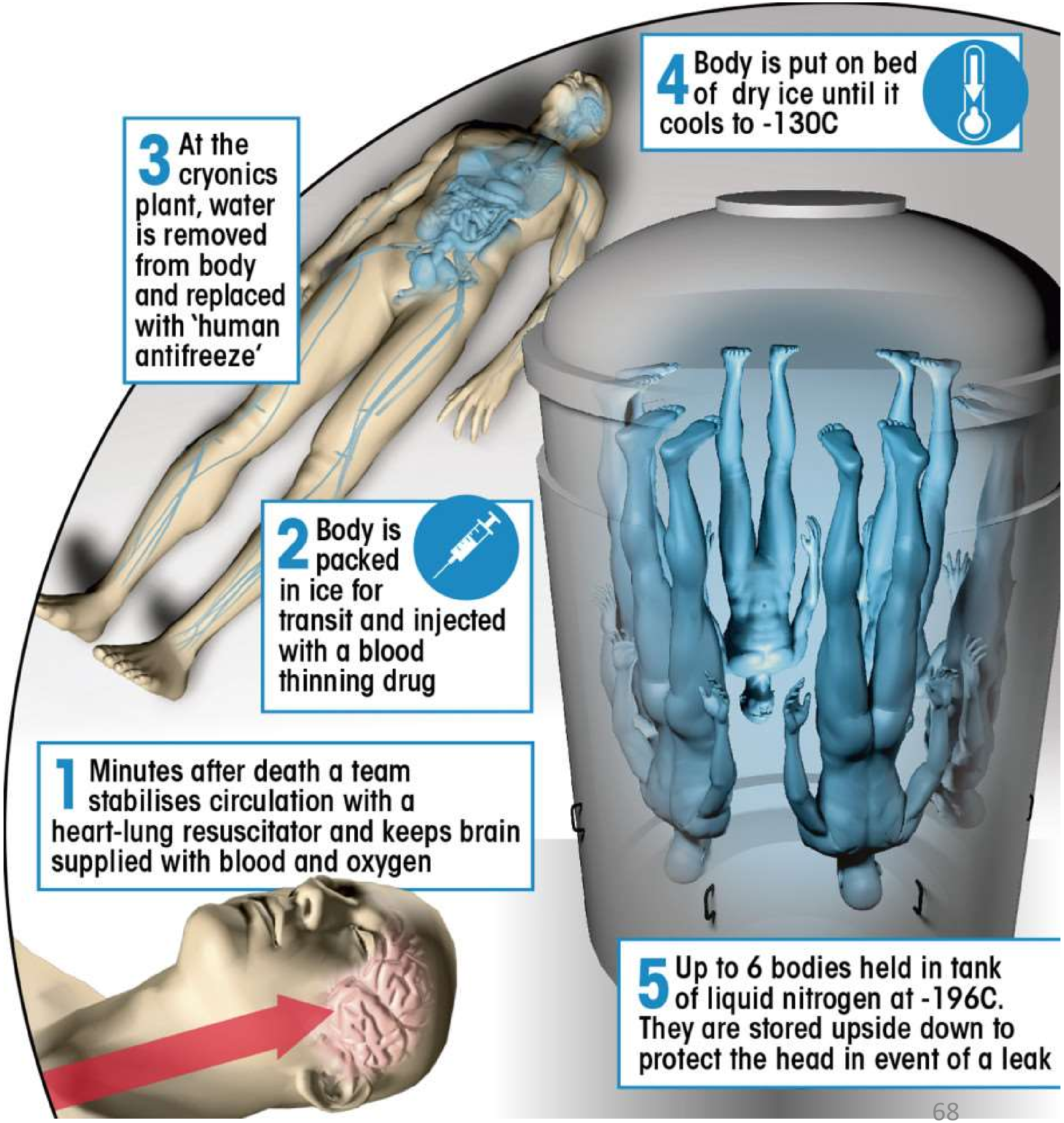
Applications: Mechanical

- Material Heat treatment
  - The lives of the tools, die castings & their dies, forgings, jigs & fixtures etc increase when subjected to cryogenic heat treatment.
  - The life of guitar strings increases by 4 to 5 times with no need for tuning.
- Recycling
  - Cryogenic recycling turns the scrap into raw material by subjecting it to cryogenic temperatures. This is mostly used for PVC, rubbers.
- Magnetic Separation
  - Magnetic separation technique is used in variety of applications like enhancing the brightness of kaolin, improving the quality of ultra-high purity quartz etc.
  - Superconducting Magnet ensures proper separation.

Applications: Medicine & Biology

- Cell preservation
  - Systems are developed to preserve blood cells, plasma cells, human organs and animal organs at cryogenic temperatures.
  - As an example: Use of LN_2 in artificial insemination in cows
- Cryosurgery
  - Cryosurgery is a novel technique in which the harmful tissues are destroyed by freezing them to cryogenic temperature.
  - Cryosurgery has shorter hospital stay, less blood loss, and small recovery time.
  - It is generally used in patients with localized prostate and kidney cancer, skin disorders, retinal problems, etc.
- Cryotherapy for athlets
- (Cryonics… non recognized by "cryogenic technology)

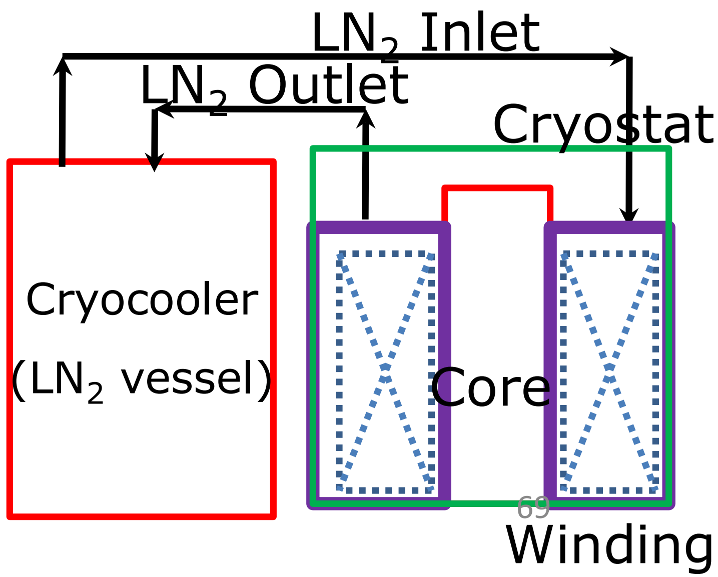
Cryonics.....



3 At the cryonics plant, water is removed from body and replaced with 'human antifreeze'
4 Body is put on bed of dry ice until it cools to -130C
2 Body is packed in ice for transit and injected with a blood thinning drug
1 Minutes after death a team stabilises circulation with a heart-lung resuscitator and keeps brain supplied with blood and oxygen
5 Up to 6 bodies held in tank of liquid nitrogen at -196C. They are stored upside down to protect the head in event of a leak

Appilications: superconductivity (SC)

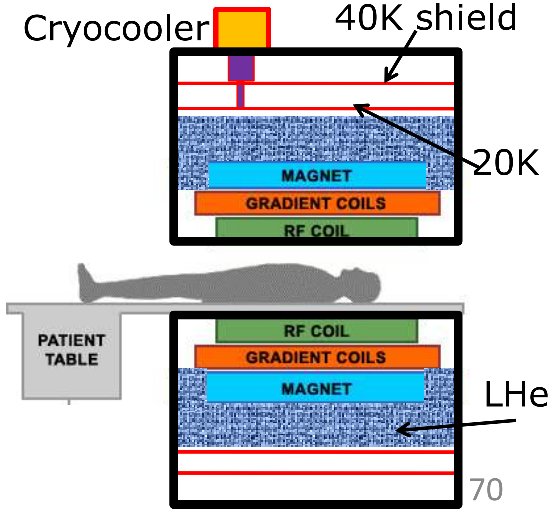
- SC Cables
- Electrotechnique (transformer, generators, ...)
  - Superconducting transformers and generators have coils (and cores sometimes) maintained at low temperature to minimize the I2R (copper) losses.
- Electronics
- NMR, MRI
- R&D on transport
- …



SC Applications: Magnetic Resonance

- The MRI (Magnetic Resonance Imaging) machines are used for body scanning.
- The SC magnets for both NMR (Nuclear Magnetic Resonance used by the pharmaceutical industry to study the molecular structure -the accuracy of measurement increases with field strength) and MRI machines are cooled by liquid Helium.
- Typical 1.5 T, max 3 T (high!).
    - Minneapolis Univ. 10.5 T!!!

Helium-cooled superconducting devices: whole-body MRI system (Bruker)

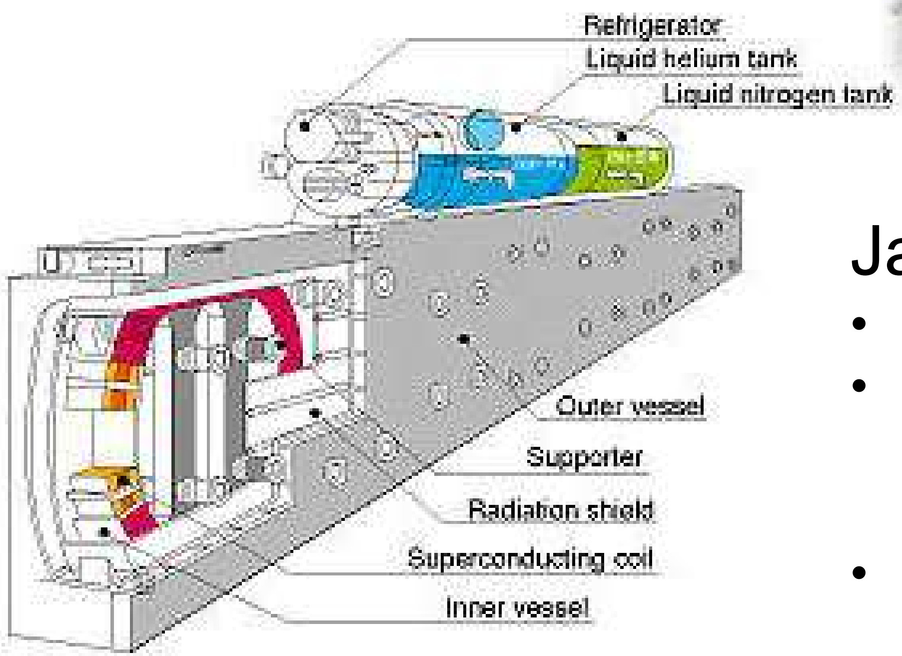


SC applications: transport (R&D)

- Ongoing R&D

42.8 km long test track between Sakaigawa and Akiyama, Japan

maximum velocity:
581 km/h (02. 12. 2003)

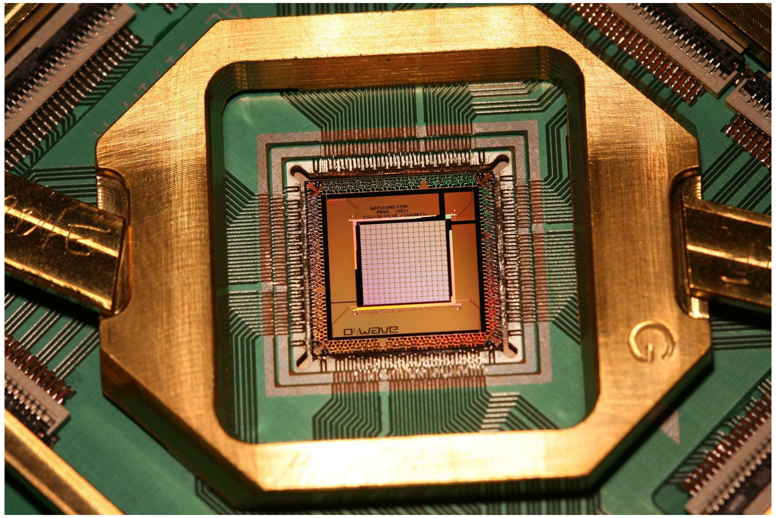


Jap. Yamanashi MAGLEV-System MLX01

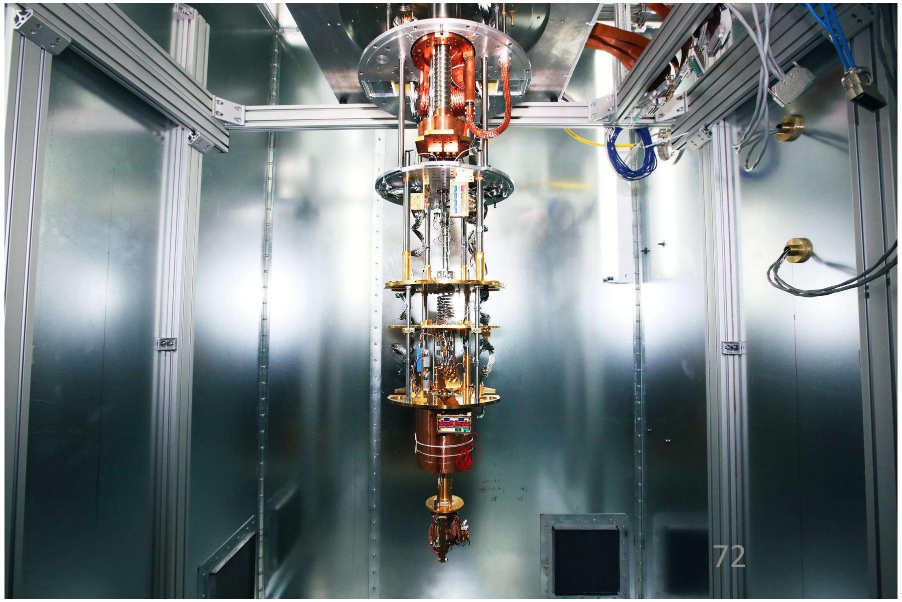
- Maglev Train runs on the principle of Magnetic Levitation.
- The train gets levitated from the guide way by using electromagnetic forces between superconducting magnets on the vehicle and coils on the ground.
- This results in no contact motion and therefore no friction.

SC Applications: Quantum computer

- A qubit is a two-state (or two-level) quantum-mechanical system.
- Quantum mechanics allows the qubit to be in a coherent superposition of both states/levels simultaneously, a property which is fundamental to quantum mechanics and quantum computing.
- An important distinguishing feature between qubits and classical bits is that multiple qubits can exhibit quantum entanglement.
- Quantum entanglement is a nonlocal property of two or more qubits that allows a set of qubits to express higher correlation than is possible in classical systems.
- Cryogenics:
  - ^{3}He and ^{4}He dilution refrigerator
  - SC electronic components (Josephson junction)
- In January 2019, IBM launched IBM Q System One, its first integrated quantum computing system for commercial use.
- Almost dayly news on quantum computers and qunatum supremacy.

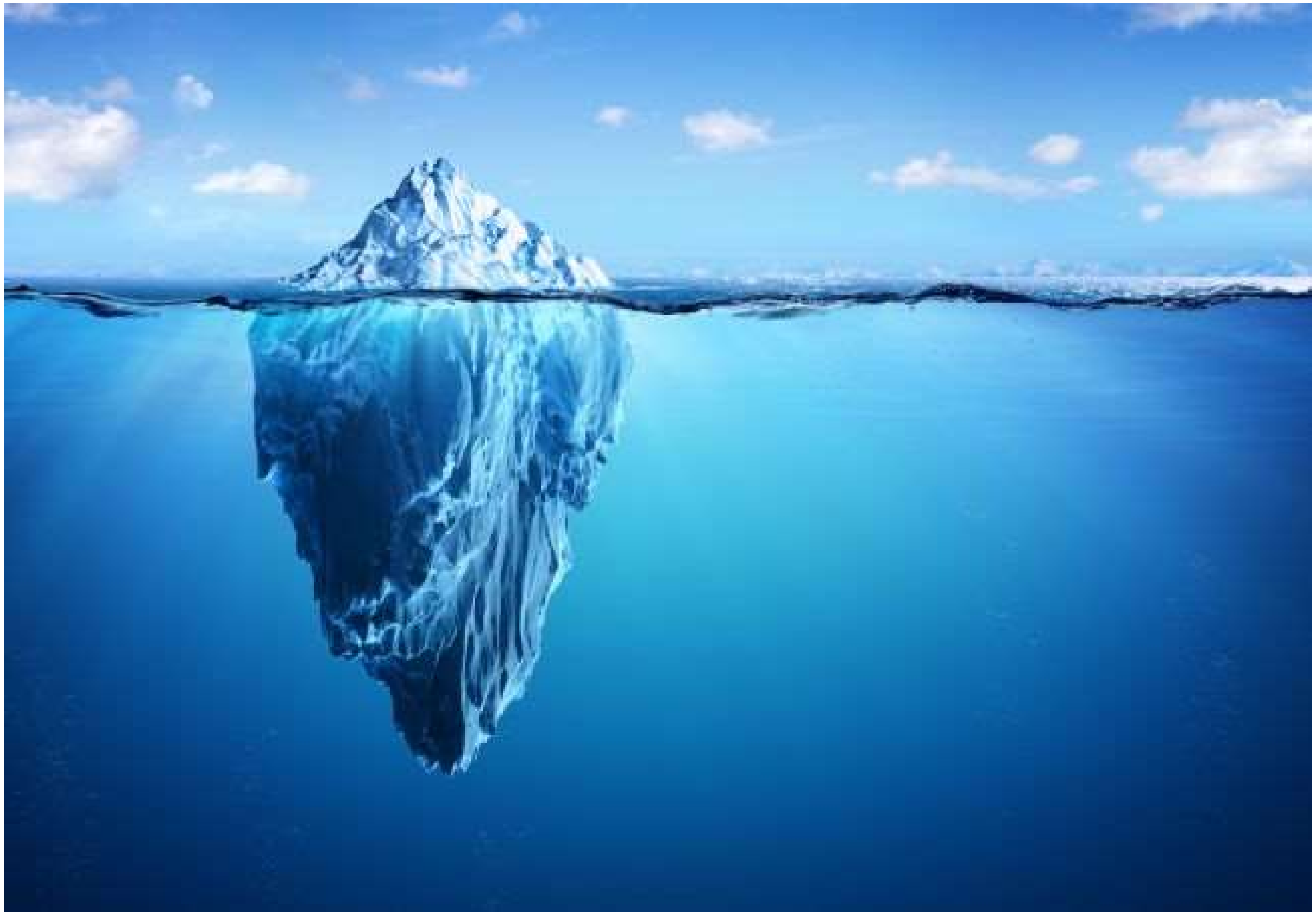


superconducting Qubit



Nice TED talk on Quantum Computer
https://www.youtube.com/watch?v=QuR969uMICM

Conclusions



Programma

- Introduzione alla criogenia.
- Proprietà dei fluidi criogenici.
- Introduzione alla progettazione di criostati.
- Refrigerazione e liquefazione: cicli Joule-Thomson, Claude, Gifford-MacMahon, Stirling, pulse tube.
- Proprietà dei materiali (entalpia e temperature di Debye, proprietà meccaniche, conducibilità termica).
- Trasmissione di calore: conduzione, convezione, irraggiamento. Tipi di isolamento. Cenni di criopompaggio.
- Linee di trasferimento.
- Strumentazione criogenica.
- Aspetti riguardanti la sicurezza in criogenia.
- Cenni alla criogenia < 4 K. Cenni di superfluidità e superconduttività.
- Discussione finale.

Dedicato a…

Prof. Ivo Modena
(Univ. Tor Vergata + LNF)
1929-2017

Ing. Luigi Mazzone
(CERN)
1926-2014

Cryogenic sensors & instrumentation

Chiara Vignoli
Corso Nazionale INFN “Introduzione alla criogenia”
Bologna, 28-30 ottobre 2019

Introduction

- The correct measurement of properties such as temperature, pressure, flow, level and vacuum in cryogenic systems is a key factor in the success of cryogenic systems.
- Measurements will allow us to understand if our cryogenic components are working properly, enable us to control them and permit the collection of scientific data.
- Think about instrumentation as a complete system – sensor, wiring, feed through, DAQ rather than just the sensor itself.
- In many cases you have a slow control system that gives you specifications about signals.
- The optimum sensor actually depends upon the requirements of the specific application.

Requirements

- Define system & sensor requirements:
  - Range
  - Accuracy
  - Sensitivity
  - Time response
  - Sensor environment (e.g. presence of magnetic or vacuum or radiation fields)
  - Reproducibility
  - Stability
  - Reliability
  - Cost

Remember

- **Losses**: The heat release, for example I^2R losses, and conduction via leads should be very low.
- **Material Properties**: Thermal, mechanical and electrical properties of sensors must be in allowable limits.

Suggestions

- Don't use more accuracy & precision than required
- Use commercially produced sensors whenever possible – there is a lot available, and follow manufacturer specifications
- When possible, mount sensors outside cryostat at 300 K (e.g. pressure transducers, flow meters)
- For critical devices inside of cryostats, install redundant sensors whenever feasible
- Be sure to consider how to recalibrate sensors
- If at all possible, avoid cold instrumentation feedthroughs

Most common sensors

We will discuss the most common sensors for a cryogenic plant/experiment:

- Temperature sensors
- Pressure sensors
- Level meters
- Flow meters

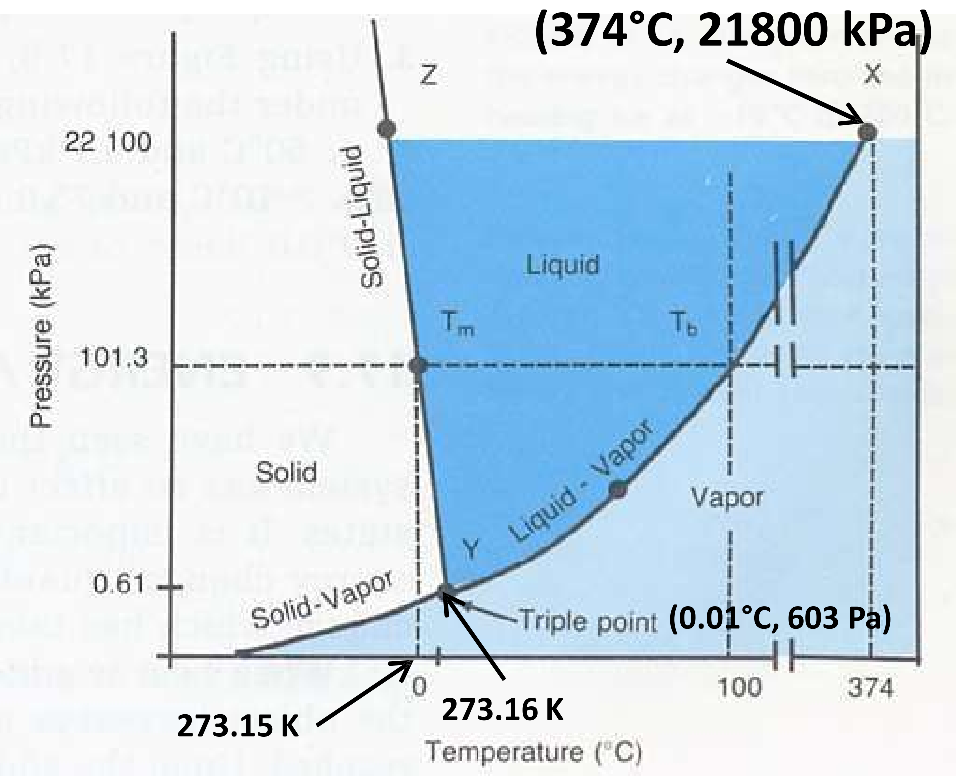
Temperature Scale

- **SI temperature scale** is the Kelvin scale. It defines the triple point of water as the numerical value of 273.16, i.e., 273.16 K. The unit of temperature in this scale is the Kelvin (K).

- **Agreement of bureaus of standards:**
  - ***ITS-90 temperature scale*** *(Comité International des Poids et Messures 1990)* **for *T* > 0.65 K** *the ITS-90 is defined by 17 fixed points and 4 defining instruments. It spans a temperature range from 0.65 K to 10 000 K. For cryogenic purposes the three defining instruments are helium vapor pressure thermometry, gas thermometry, and platinum resistance thermometry.*
  - ***PLTS-2000*** *(Provisonal Low Temperature Scale, melting curve of* ^{3}*He)* **for lower *T*** *the PLTS 2000 is defined by a polynomial, relating the melting pressure of* ^{3}*He to temperature from the range 0.9 mK to 1 K. The pressure to temperature relationship is based on primary thermometers such as Johnson noise and nuclear orientation.*

Triple point of water

The triple point of water is the most important defining thermometric fixed point used in the calibration of thermometers to the International Temperature Scale of 1990 (ITS-90).
It is the sole realizable defining fixed point common to the Kelvin Thermodynamic Temperature Scale (KTTS) and the ITS-90; the assigned value on these scales is 273.16 K (0.01°C)



ITS 90

- International Temperature Scale of 1990
- http://www.its-90.com/tables.html
- **Definition of the International Temperature Scale of 1990**
  - Between 0.65 K and 5.0 K T_{90} is defined in terms of the vapour-pressure temperature relations of ^3He and ^4He.
  - Between 3.0 K and the triple point of neon (24.5561 K) T_{90} is defined by means of a helium gas thermometer calibrated at three experimentally realizable temperatures having assigned numerical values (defining fixed points) and using specified interpolation procedures.
  - Between the triple point of equilibrium hydrogen (13.8033 K) and the freezing point of silver (961.78°C) T_{90} is defined by means of platinum resistance thermometers calibrated at specified sets of defining fixed points and using specified interpolation procedures.
  - Above the freezing point of silver (961.78°C) T_{90} is defined in terms of a defining fixed point and the Planck radiation law.
- The defining fixed points of the ITS-90 are listed in Table 1. The effects of pressure, arising from significant depths of immersion of the sensor or from other causes, on the temperature of most of these points are given in Table 2.

ITS 90 (17 fixed points)

Table 1. Defining Fixed Points of the ITS-90

Number	Temperature T_{90}/K	t_{90}/°C	Substance [a]	State [b]	W_r (T_{90})
1	3 to 5	-270.15 to -268.15	He	V	
2	13.8033	-259.3467	e-H_2	T	0.001 190 07
3	~17	~-256.15	e-H_2 (or He)	V (or G)	
4	~20.3	-252.85	e-H_2 (or He)	V (or G)	
5	24.5561	-248.5939	Ne	T	0.008 449 74
6	54.3584	-218.7916	O_2	T	0.091 718 04
7	83.8058	-189.3442	Ar	T	0.215 859 75
8	234.3156	-38.8344	Hg	T	0.844 142 11
9	273.16	0.01	H_20	T	1.000 000 00
10	302.9146	29.7646	Ga	M	1.118 138 89
11	429.7485	156.5985	In	F	1.609 801 85
12	505.078	231.928	Sn	F	1.892 797 68
13	692.677	419.527	Zn	F	2.568 917 30
14	933.473	660.323	Al	F	3.376 008 60
15	1234.93	961.78	Ag	F	4.286 420 53
16	1337.33	1064.18	Au	F	
17	1357.77	1084.62	Cu	F	

[a] All substances except ^{3}He are of natural isotopic composition, e-H_2 is hydrogen at the equilibrium concentration of the ortho- and para-molecular forms.

[b] For complete definitions and advice on the realization of these various states, see "Supplementary Information for the ITS-90". The symbols have the following meanings: V: vapour pressure point; T: Triple Point (temperature at which the solid, liquid and vapour phases are in equilibrium); G: gas thermometer point; M,F melting point, freezing point (temperature, at a pressure of 101 325 Pa, at which the solid and liquid phases are in equilibrium)

ITS 90 (cont)

Table 2. Effect of pressure on the temperatures of some defining fixed points#

Substance	Assigned Value of equilibrium temperature T_{90}/K	Temperature with pressure, p, $(dT/dp)/10^{-8} K.Pa^{-1})$*	Variation with depth, l, $(dT/dl)/10^{-3} K.m^{-1})$**
e-Hydrogen (T)	13.8033	34	0.25
Neon (T)	24.5561	16	1.9
Oxygen (T)	54.3584	12	1.5
Argon (T)	83.8058	25	3.3
Mercury (T)	234.3156	5.4	7.1
Water (T)	273.16	-7.5	-0.73
Gallium	302.9146	-2.0	-1.2
Indium	429.7485	4.9	3.3
Tin	505.078	3.3	2.2
Zinc	692.677	4.3	2.7
Aluminium	933.473	7.0	1.6
Silver	1234.93	6.0	5.4
Gold	1337.33	6.1	10
Copper	1357.77	3.3	2.6

*Equivalent to millikelvins per standard atmosphere

**Equivalent to millikelvins per metre of liquid

#The Reference Pressure for melting and freezing points is the standard atmosphere (p_0 = 101 325 Pa). For triple points (T) the pressure effect is a consequence only of the hydrostatic head of liquid in the cell

Temperature measurement

- definition of temperature via reversible Carnot process is not well suited for establishing useful measuring methods

  in practice: use of ***fixpoints*** and ***interpolation polynoms***

- ***primary thermometers:***

  → measured quantity is related directly to temperature

  (in a theoretically predictably way)

  → no calibration is required

- ***secondary thermometers:***

  → measured quantity varies with temperature in a reproducible way

  → must be calibrated using a primary thermometer

- requirements for temperature measurement:

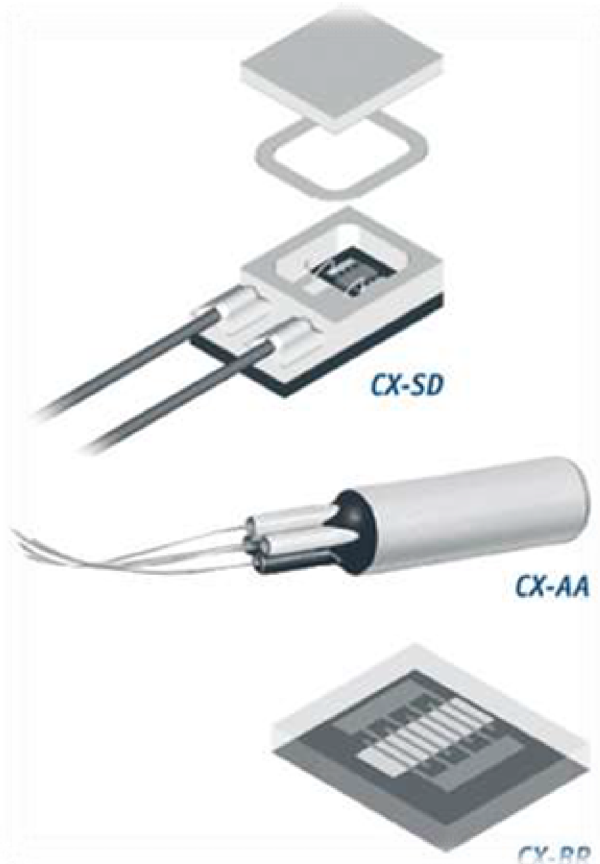
  → good thermal contact between thermometer and sample

  → low self-heating

  → sensitive to temperature changes

Thermometers for 1 K < T < 300 K

- gas thermometer: $p = p(T)$
  - Helium gas ≈ ideal gas down to 10K
- vapour pressure thermometer: $T_{liquid} = f(p_{vapor})$
  - pressure of 10 Pa corresponds to 0.4K for ^3He
- thermocouples: $V_{th} = V_{th}(T)$
- resistance thermometry: $R = R(T)$
  - 1K -300K
  - semiconductors (e.g. Ge doped with Arsenic has 100-500 Ω/K @ 4.2K, self-heating around 10µA)
  - p-n junction diode (problem with high bias current → self heating)
- capacitance thermometry: $C = C(T)$
  - based on temperature change of dielectric properties
  - virtually no magnetic field-induced errors
- noise thermometer: $S = S(T)$
  - Johnson noise in resistor: $S_V = 4k_BTR$
  - like gas thermometer, but with electrons
  - with SQUID measurements: 0.1% @ 1K



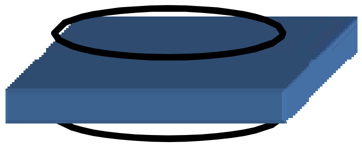
Thermometers for T < 1 K

1mK ≤ *T* ≤ 1K:

- ***magnetic suceptibility thermometer***

Curie's law: $$\chi = \mu_0 \frac{M}{B} = \frac{C}{T}$$

M: magnetization
B: applied magnetic field
C: Curie constant



mutual inductance between two coils: $m = m_0 \, f \, \chi$

→ Cerium magnesium nitrate (CMN) useful from 1K - 10mK
→ low temperature limit set by magnetic ordering at ≈ 1mK

- ***resistance thermometers***

T < 1mK:

- ***Nuclear Magnetic Resonance (NMR) thermometer***

→ temperature dependence of spin relaxation
→ platinum ideal choice for NMR thermometry

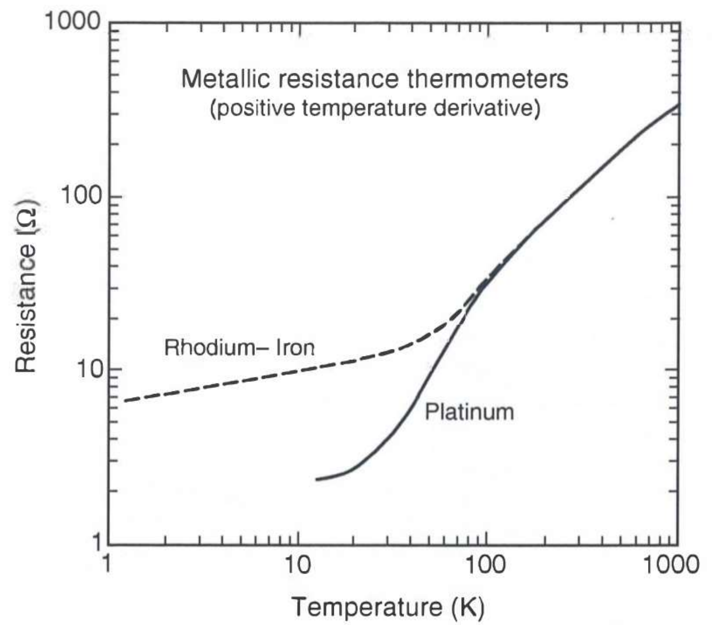
Thermometers - Considerations

- Temperature range
- Type of signal: voltage, capacitance
- Temperature sensitivity: change in signal per change in temperature
- Response time: size, thermal mass
- Mounting package
- Magnetic field sensitivity
- Strain sensitivity
- Repeatability (thermal cycling)
- Interchangeability.
- Long term stability
- Radiation resistance
- Calibration
- Excitation requirement
- Cost

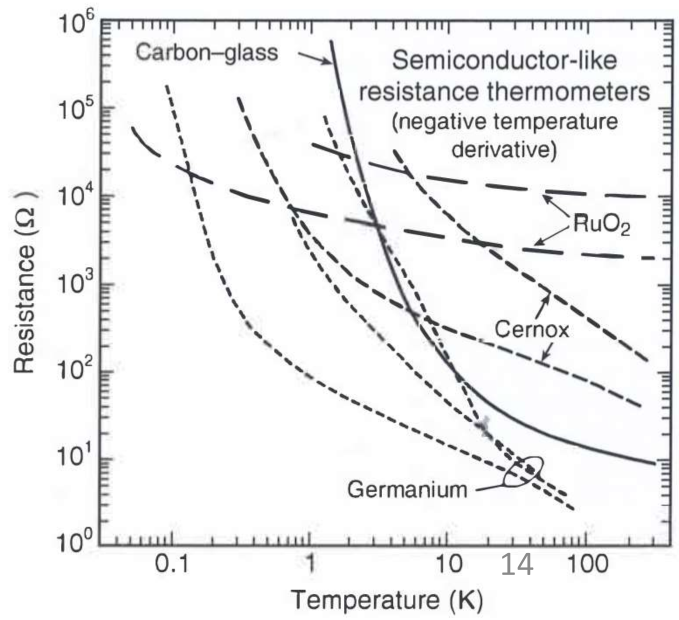
➢ NO IDEAL THERMOMETER EXIST

Resistance thermometers (RTD)

Resistance of a conductor or a semi conductor changes with the change in temperature.



- **Metallic resistance thermometers**
  - positive temperature derivative
  - highly reproducible, interchangeable
  - less sensitive at lower temperatures



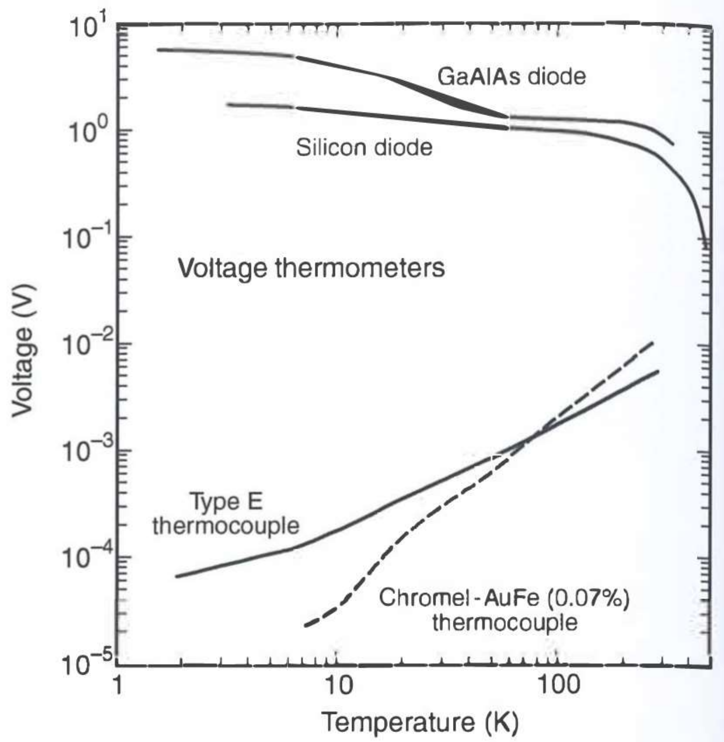
- **Semiconductor-like resistance thermometers**
  - negative temperature derivative, like semiconductors
  - variable from sensor to sensor (electrical resistivity depends on impurity doping levels)
    - they usually require individual calibration
  - optimum sensitivity at low temperatures

Voltage thermometers

Diode and thermocouple sensors have a voltage output

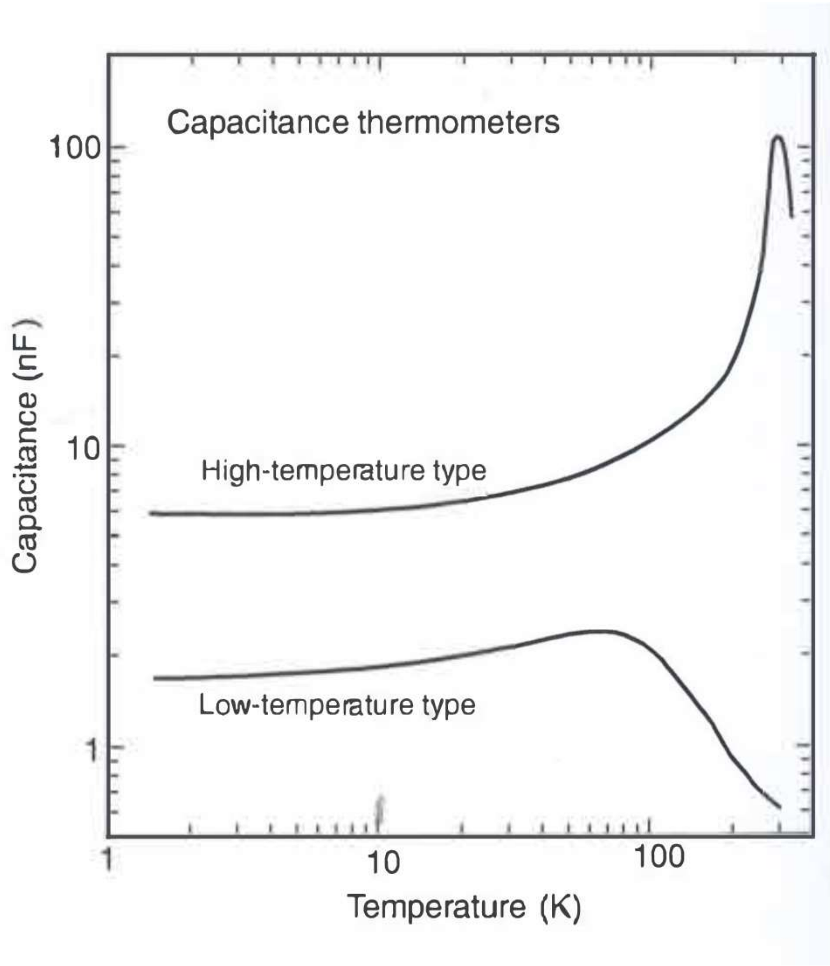
- **Diode thermometers**
  - the most easy-to-read output signal, ≈ 1 V
  - not as accurate and reproducible as semiconductor or metal sensors

- **Thermocouples**
  - output difficult to measure: mV but aso even ≈ μV
  - accuracy (except for differential measurements) not as good as that of semiconductor or metal thermometers
  - when resolution ≈ 1 degree is sufficient, thermocouples provide a small, quick-responding and economical sensor that can be used over a very wide temperature range



Capacitance thermometers

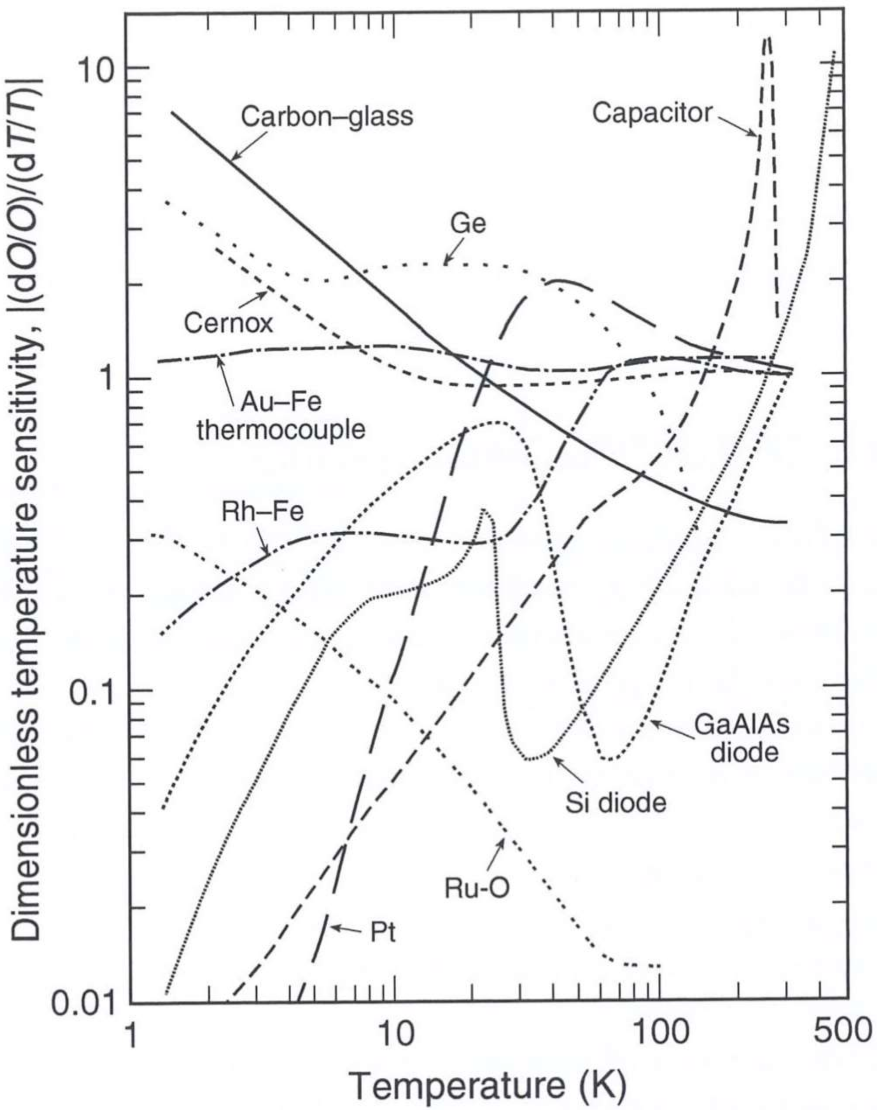
- **Capacitance thermometers**
  - The smallest magnetic-field error
  - Not reproducible upon thermal cycles
  - <u>Not at all useful thermometers,
but rather as control sensors</u> once a stable temperature is reached
    - e.g. for holding temperature constant during magnetic-field sweeps

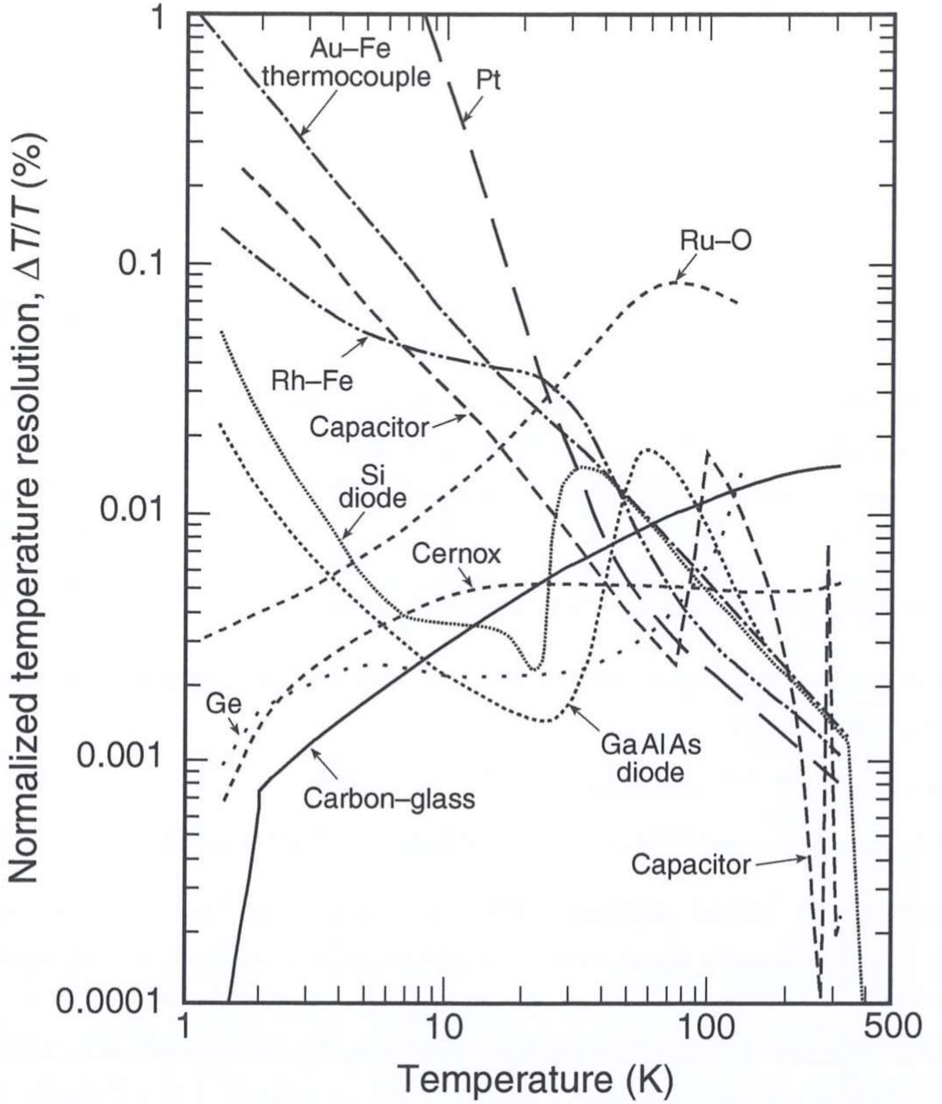


Characteristics of commercial cryogenic thermometers

This graph could be measliding if the magnitude of the signal become too low/high

ΔT smalleest T change that can be resolved under typical operating conditions of sensors



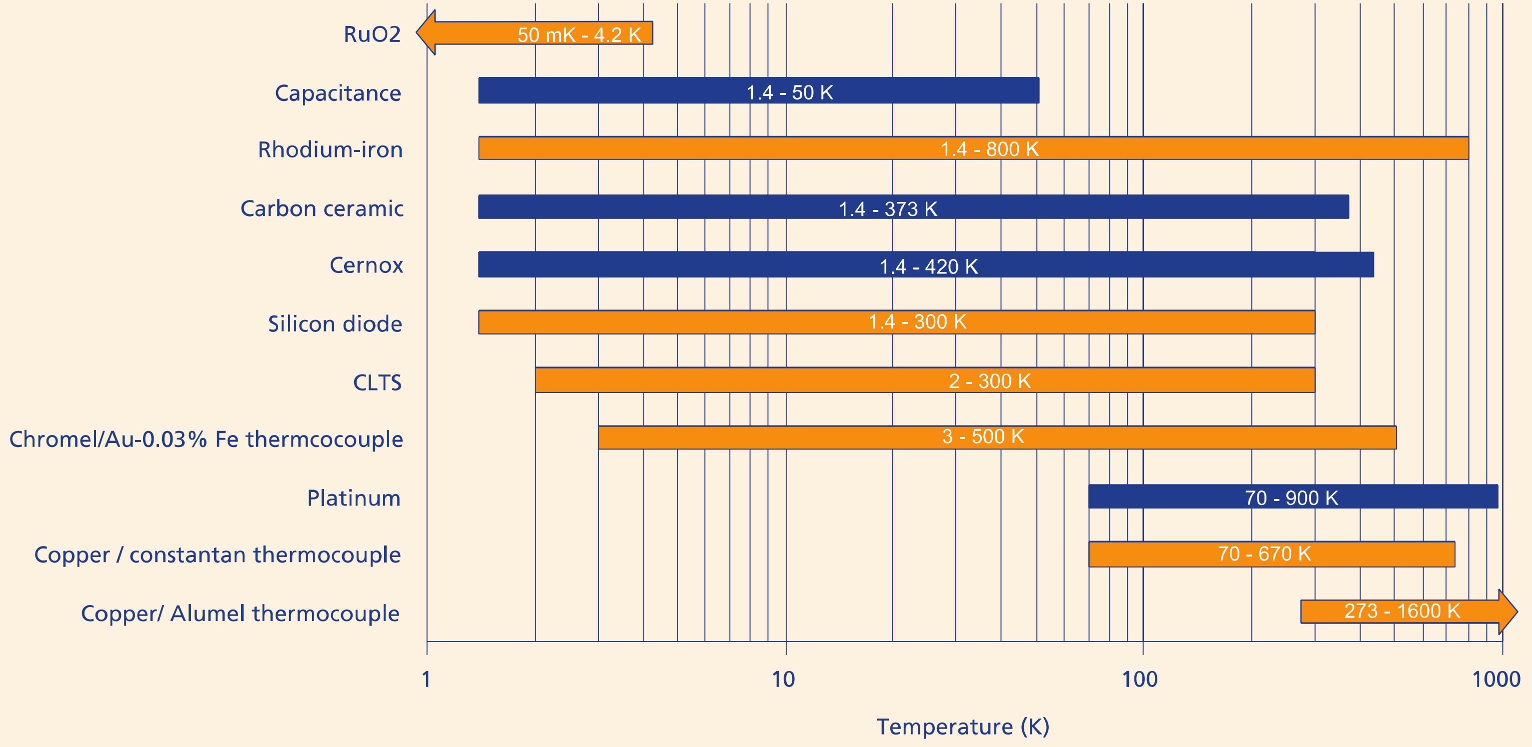


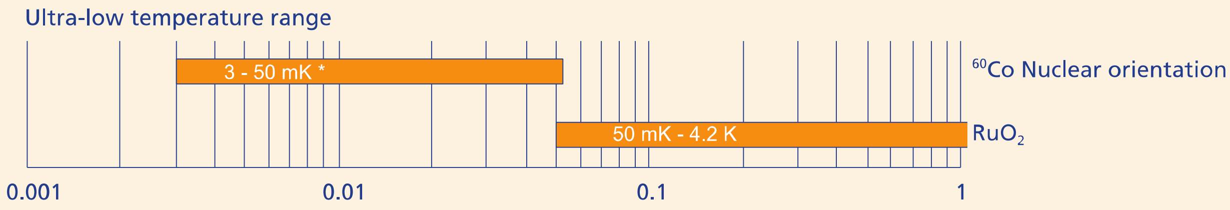
Sens

$$S_T = \frac{\%\, change\ in\ signal}{\%\ change\ in\ T} \equiv dimensionless\ sensitivity$$

S ≥ 0.1 is best

$\Delta T/T < 0.1$ % is generally acettable

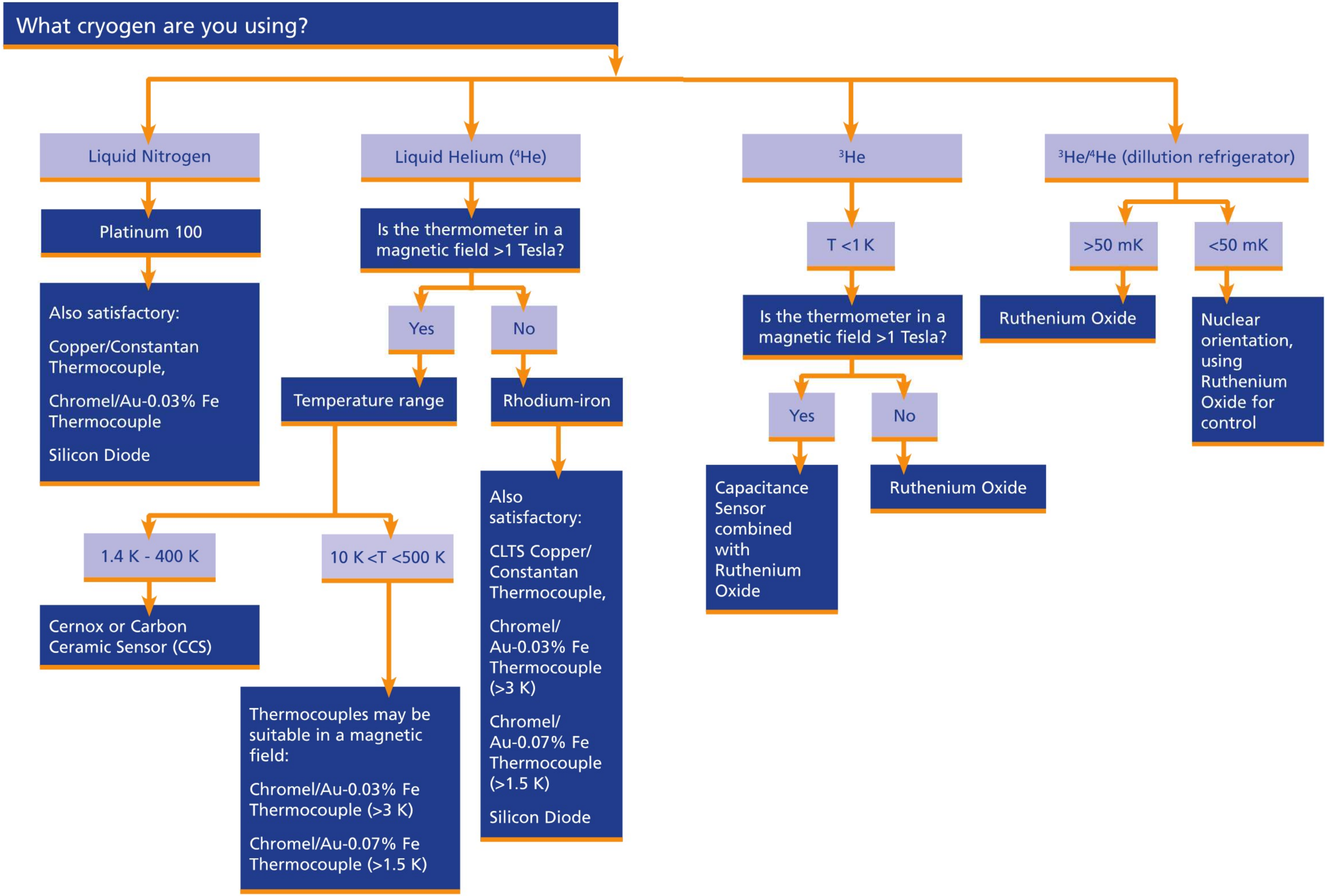




Notes

* Nuclear orientation thermometry is sensitive to external magnetic fields, and the source can be damaged by high fields. See sensor page for details.

Blue	Suitable for use in magnetic fields
Orange	**Not** Suitable for use in magnetic fields


What cryogen are you using?
Liquid Nitrogen
Platinum 100
Also satisfactory:
Copper/Constantan Thermocouple,
Chromel/Au-0.03% Fe Thermocouple
Silicon Diode
Liquid Helium (^{4}He)
Is the thermometer in a magnetic field >1 Tesla?
Yes
Temperature range
1.4 K - 400 K
Cernox or Carbon Ceramic Sensor (CCS)
10 K <T <500 K
Thermocouples may be suitable in a magnetic field:
Chromel/Au-0.03% Fe Thermocouple (>3 K)
Chromel/Au-0.07% Fe Thermocouple (>1.5 K)
No
Rhodium-iron
Also satisfactory:
CLTS Copper/Constantan Thermocouple,
Chromel/Au-0.03% Fe Thermocouple (>3 K)
Chromel/Au-0.07% Fe Thermocouple (>1.5 K)
Silicon Diode
^{3}He
T <1 K
Is the thermometer in a magnetic field >1 Tesla?
Yes
Capacitance Sensor combined with Ruthenium Oxide
No
Ruthenium Oxide
^{3}He/^{4}He (dillution refrigerator)
>50 mK
Ruthenium Oxide
<50 mK
Nuclear orientation, using Ruthenium Oxide for control

Temperature Sensor Overview

Table 4-6 Overview of cryogenic temperature sensors

Sensor	Measurement technique	Range [K]	Sensitivity	Stability	Size	Magneto-resistance	Radiation effect	Cost [$]
Carbon	Resistance	0.01–300	Good	Poor	Moderate	Moderate	–	0.1
Carbon-glass	Resistance	1.4–325	Very high	Moderate	Moderate	Moderate	–	195
Capacitance	Capacitance	0.2–250	Moderate	Poor	Moderate	None	–	300
Cernox	Resistance	0.3–325	Good	Good	Small to moderate	Small	Low	125
CLTS	Resistance	4–300	Very low	Good	Large	–	Small	–
CMN	Susceptibility	0.001–10	–	–	–	<0.02 T	–	DIY
GaAs or GaAlAs diode	Voltage	1.4–475	Low	Good	Moderate	Moderate	–	–
Germanium	Resistance	0.05–100	Good to low	Very good	Moderate	Large	–	150–2000
^{3}He melting curve	Pressure	0.001–0.32	–	–	–	Small	–	DIY
Mössbauer	Gamma detector	0.002–0.02	–	–	–	–	–	DIY
NMR	NMR	μK–mK	–	Moderate	Very large	Moderate	–	DIY
Noise	Voltage (SQUID)	μK–300	–	Moderate	–	–	–	DIY
Nuclear orientation	Gamma detector	0.004–4	–	Moderate	–	Small	–	680
Platinum	Resistance	10–800	Low to good	Very good	Moderate	Large	Small	75
Rhodium–iron	Resistance	0.1–600	Low to good	Very good	Small to large	Large	Small	360
Ruthenium oxide	Resistance	0.05–20	Good to low	Moderate	Moderate	Small	–	90
Si diode	Voltage	1.4–475	Low	Moderate	Moderate	Very large	Large	100
Superconducting fixed points	Susceptibility	0.015–7	–	Very good	Moderate	Zero field required	–	3500
Thermistor	Resistance	77–300	Very high	Good	Small	Small	–	–
Thermocouple, Au–Fe	Voltage	2–300	Low	Moderate	Small	Moderate	–	10

Note: DIY in the cost column stands for Do It Yourself and can be quite expensive.

Temperature Sensor Overview

Sensor Type	Temperature Range	Accuracy[a] (± value)	Reproducibility[b] (± value)	Long-Term Calibration Drift	Inter-changeability[c]	Magnetic Field Use	Best Use	Cost
Platinum resistance thermometer	77–800 K With impurity correction: 20–77 K (Appendix A5.3b)	Without individual calibration: 0.6 K at 70 K 0.2 K at 300 K With individual calibration: 20 mK at 70 K 35 mK at 300 K	10 mK from 77 K to 305 K	±10 mK/yr at 77 K to 237 K	Yes	Recommended above 70 K; error < 0.1% with standard correction given in Appendix A5.5	Measurements above 77 K Excellent reproducibility interchangeability, low magnetic field error Many shapes and sizes available	Low without calibration High with individual calibration
Zirconium–oxynitride resistance thermometer (Cernox™)	0.3–325 K	Must be individually calibrated 5 mK at 4.2 K < 0.1% at > 10 K	3 mK at 4.2 K	±25 mK/yr at 1 K to 100 K 0.05% of reading at 100 K to 300 K	No	Recommended Lowest error Standard correction given Appendix A5.6	One of the best sensors for use in magnetic fields Good sensitivity over a wide temperature range Fast response time as chip	High with individual calibration
Germanium resistance thermometer	0.05–100 K	Must be individually calibrated With individual calibration: 5 mK at < 10 K 0.07% at > 10 K	0.5 mK at 4.2 K	±1 mK/yr at 4.2 K ±10 mK/yr at 77 K	No	Not recommended	Secondary-standard thermometer Excellent reproducibility	High with individual calibration
Silicon diode thermometer	1.4–450 K	Without calibration: 1 K at < 100 K 1% at 100 K to 300 K With individual calibration: 20 mK at 1.4 K–10 K 50 mK at 10 K–330 K	5 mK at 4.2 K 20 mK at 77 K 15 mK at 300 K	±10 mK/yr at 4.2 K ±40 mK/yr at 77 K ±25 mK/yr at 300 K	Yes	Not recommended below ~60 K	Relatively inexpensive, interchangeable, easily measured output Small size	Medium for low accuracy High with individual calibration

* Compiled from data supplied by Lake Shore Cryotronics 2002, Besley (1993), Rubin (1999).

[a] Accuracy: the difference between the measured and true temperature value.

[b] Reproducibility: the change in apparent temperature when the sensor is subjected to repeated thermal cycling from room temperature.

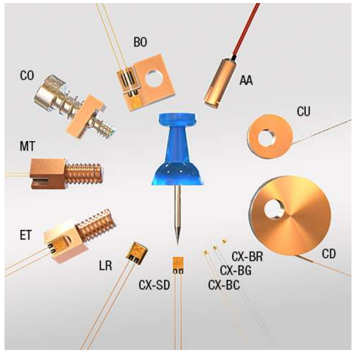
[c] Interchangeability: the ability to substitute one sensor for another with little change in calibration.

Measure of temperature

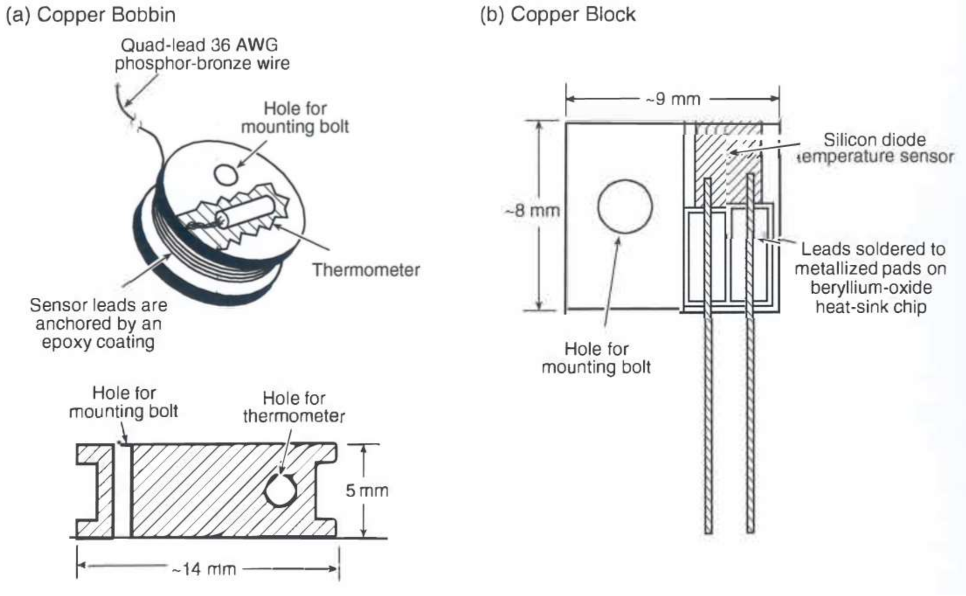
Source of errors in temperature measurements over the cryogenic range:

1. Poor thermal contact of the sensor to sample being measured
2. Errors arising from thermometer instrumentation and calibration
   - Sensor sensitivity
   - Output (V,R,C) uncertanty
   - Current source (I) uncertanty
   - Calibration uncertanty (manufactor)
   - Thermal noise
   - Electromagnetic noise
   - Thermoelectric noise
3. Sensor self-heating
4. Parasitic-heating

Sensor mounting (1)



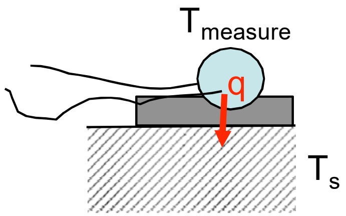
- Good sensor-sample thermal contact
  - Typically directly coupled to the sample hoder or through a sensor holder
  - Thermometers can also be soldered (In or In-3%Ag) or epoxied to thermally attach the sensor to the holder (or better piersed)
- Sensor holder
  - High thermal conductivity sensor holder (copper) to be bolted to the sample holder, using a thin (≤ 0.5 mm) coating of thermally conductive greese to reduce the interfacial thermal resistance
  - Use bolt/screw of brass so that thermal contraction on cooling will tighten the joint
  - Pierced holder to host the sensor
  - Use thermally conductive greese (Apiezon N^{TM} or Cry-Con^{TM}), or expoxy, or varnish

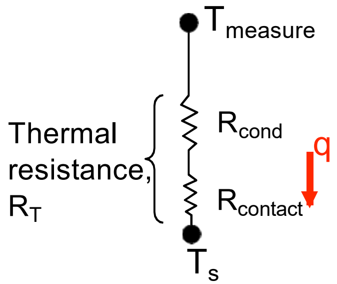


Bolt-on copper bobbin (a), and copper block (b), for thermally anchoring a thermometer and its leads. For the bobbin technique, the thermometer leads are wrapped around the bobbin and thermally attached to it with varnish or epoxy. For the copper block technique, a beryllium-oxide chip with electric-terminal metallization on top can be used to thermally anchor the thermometer leads.

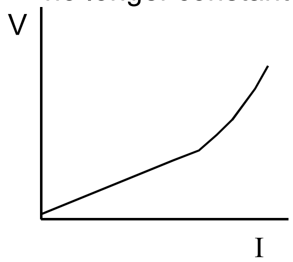
Sensor mounting (2)

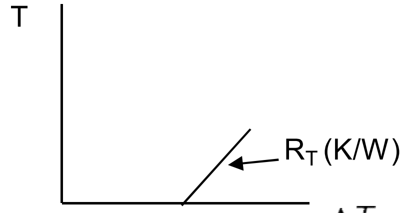
- Self heating





As the current is increased, if T is no longer constant, then ρ is no longer constant





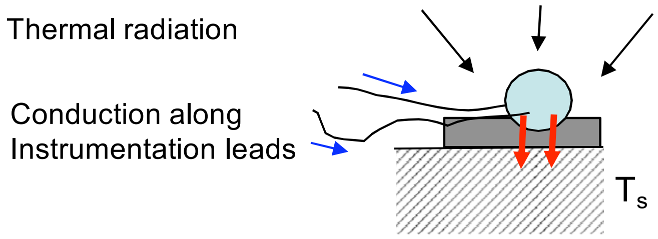
$$q(W) = I^2 R_e = \frac{\Delta T}{R_T}$$

A compromise must be made between

- signal uncertainty : excitation current is necessary to make the temperature measurement
- self-heating error : dissipation of power $q(W) = I^2 R_T$

- Parasitic heat leak

- Screen sensor to avoid radiation effect
- Reduce conduction effect
  - Use low thermal conductivity wires (manganin, constantan, phosphore bronze, nichrome, stainless steel, Evan-ohm)
  - Reduce wire cross section and increase length
  - Heat sink the wires at a temperature near the sensor operating temperature to intercept the heat leak
  - Reduce insulation thickness to improve ocntact with the heat sink. Thin enamel is better than thicker Teflon.

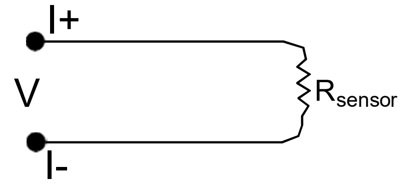


$$q_{cond} = \frac{A}{L}\int k(T)\,dT$$

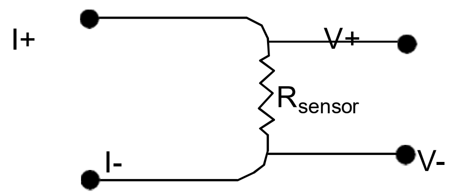
Sensor leads

- Some sensors, such as zirconium-oxynitride, carbon-glass, and germanium sensors, are packaged with four wires (two for current and two for voltage sensing).
- Other sensors, such as platinum resistors, are supplied with only two wires.
- The four-lead type should be used to eliminate the resistance of the instrumentation cables, which can be quite high since they run a long distance, from room-temperature instrumentation down through the cryostat to the thermometer. This is especially true for cryogenic thermometry.
- To minimize current-induced pickup in the voltage leads, the leads from the voltmeter have to be twisted together, separate from the pair of current-source leads.
- The reduction in error afforded by the four-lead scheme scales with the ratio of voltmeter resistance to thermometer resistance. Therefore, a voltmeter with a fairly high input impedance should be used. This is especially the case for semiconductor-like sensors, since at low temperatures their resistances can approach values even of the order ot MOhm.
- Note: the heating caused by soldering to thermometer-sensor wires may destroy the calibration of some sensors

$$V = I(R_{sensor} + R_{leads})$$



Solution: 4-wire connection



Four-lead measurement scheme. Two leads are used to connect the current source to the thermometer terminals, and two additional leads to connect the voltmeter. High-resistance phosphor-bronze "quad" leads, work well for thermometer instrumentation because the high thermal resistance along their length minimizes unwanted heat conduction down the leads to the thermometer. These are offered commercially in a convenient package as four phosphor-bronze leads in a "quad" cable that can be attached to the four terminals of a thermometer.

Wire Heat Sinking

Table 4-3 Wire heat-sinking lengths required to thermally anchor to a heat sink at temperature *T* to bring the temperature of the wire to within 1 mK of *T*

			Heat-sinking length, L_2 (mm) for wire sizes			
Material	T_1 [K]	T_s [K]	0.21 mm^2 (24 AWG)	0.032 mm^2 (32 AWG)	0.013 mm^2 (36 AWG)	0.005 mm^2 (40 AWG)
Copper	300	80	160	57	33	19
	300	4	688	233	138	80
Phosphor-Bronze	300	80	32	11	6	4
	300	4	38	13	7	4
Manganin	300	80	21	4	4	2
	300	4	20	7	4	2
304 ss	300	80	17	6	3	2
	300	4	14	5	3	2

Note: Values are calculated assuming wires are in a vacuum environment, and the thermal conductivity of the adhesive is given by the fit to the thermal conductivity of GE 7031 varnish.

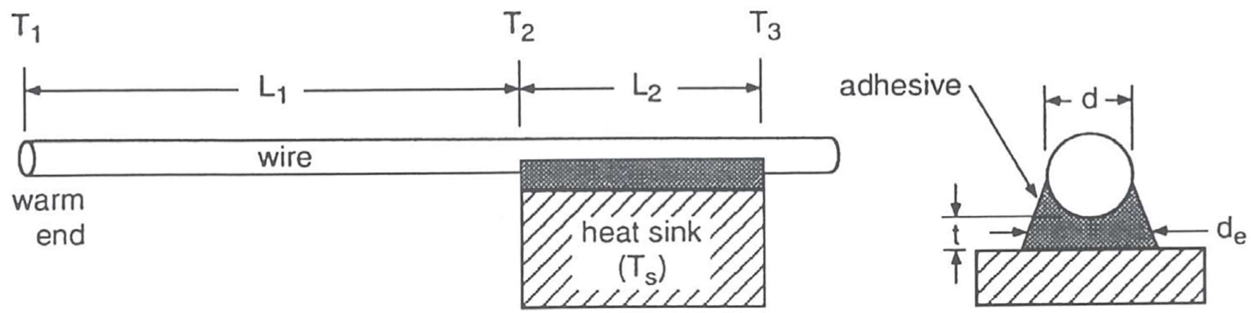


Figure 4-2 Arrangement for heat-sinking wires. The two schematic views are not to the same scale.

Measurement uncertanties

- Factors contributing to uncertainty:
  - Sensor sensitivity:

$$S_T = \frac{\%\,change\ in\ signal}{\%\ change\ in\ T} \equiv dimensionless\ sensitivity$$

  - Voltmeter uncertainty (V, or R, or C)

$$\frac{U_{T,V}}{T} = \frac{U_V/V}{S_T} = \%uncertainty\ in\ V \bullet \frac{\%change\ in\ T}{\%change\ in\ V}$$

  - Current source uncertainty

$$\frac{U_{T,I}}{T} = \frac{\left(U_I/I\right)}{S_T}$$

  - Calibration uncertainty – see mfc.
  - Thermal noise – usually negligible
  - Electromagnetic noise: $emf = \frac{dB}{dt} \bullet A$
    - •Twisted pairs
    - •Shielding – connect shield at one end only – preferably at signal source

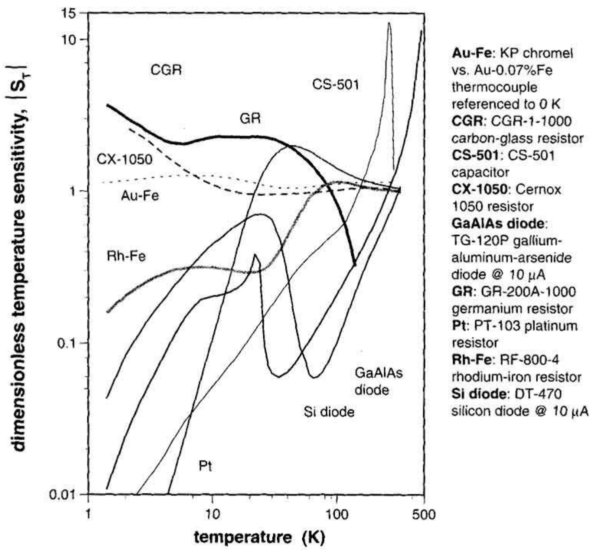
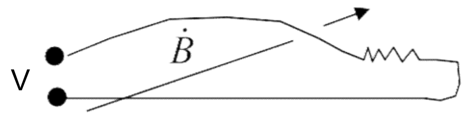
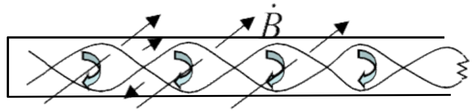


Figure 1. Absolute dimensionless temperature sensitivities of representative Lake Shore sensors.

Problem:



Solution:



- Combined total uncertainty: $U_T = \left[\left(U_{T,V}\right)^2 + \left(U_{T,I}\right)^2 + \left(U_{T,Cal}\right)^2 + \left(U_{T,therm\_noise}\right)^2\right]^{1/2}$

Measurement instrumentation

- DC measurement instrumentation
  - Offset voltages and noise in DC measurement circuits are typically on the order of microvolts. The noise floor can be lowered an order of magnitude or more by reversing the excitation current polarity and combining the two measurements. Some measurement systems place a reference resistor in series with the sensor and provide switching of the voltmeter to allow measurement of the voltage across either the sensor or reference resistor. The additional switching and data manipulation are often well worth the improvements in measurement accuracy. Averaging many readings obtained with high-stability instruments can push the noise floor down towards 1 n V.
- Pulse measurement techniques
  - Difficulties in measuring low-voltage DC signals also can be overcome by using pulse techniques thus reducing the average power dissipation.
- AC measurement instrumentation
  - Offset voltages can be eliminated by using AC measurements without requiring switching of the excitation and extra data manipulation.
- Thermoelectric and zero offset voltage
  - The Seebeck coefficient of joints of different materials, in regions of ΔT, produce a thermoelectric voltage, typically of the order of microvolts. Solution: reversing polarity
  - Thermoelectric voltages and zero offsets can be eliminated from voltage measurements on ohmic resistors by reversal of the excitation current and use of the formula $V = (V_+ + V_-)/2$

Thermometers: Do’s & Don’ts

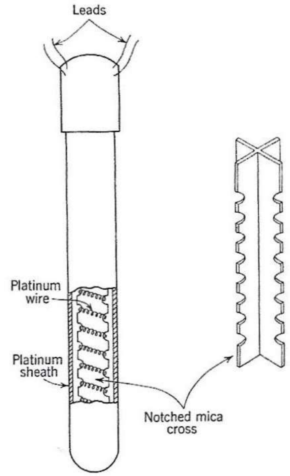
- Thermally anchor leads as close to measurement temperature as possible (5 -10 cm length)
- Use twisted, shielded leads to minimize electromagnetic noise (connect shield at one end only)
- Minimize conduction heat load by using long lengths, small diameters, low thermal conductivity materials
- Follow recommended excitation levels to avoid self heating
- Isolate low-level signal leads from high-level signal leads
- Reverse polarity to cancel thermal emf components

Platinum Resistor Thermometers

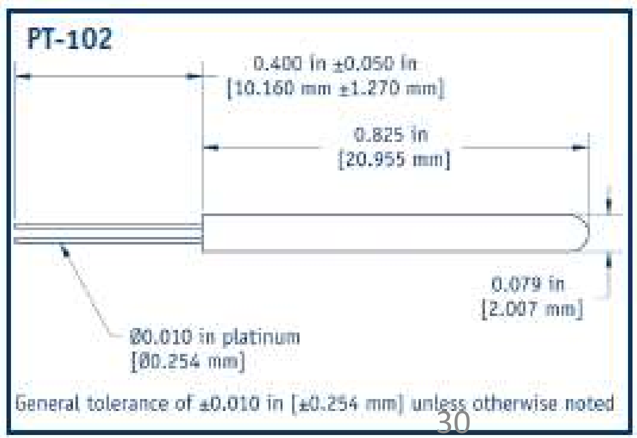
- Positive temperature coefficient
- A single excitation current can be used for a wide temperature range. resulting in decreasing power dissipation with decrasing temperature, as required to prevent self-heating.
- Good linearity with T
- Typical calibration equation (in °C) for t < 0°C (A,B,C found by calibration at 3 standard T)

$$\frac{R_t}{R_o} = 1 + At + Bt^2 + Ct^3\,(t - 100)$$

- Standard calibration with computer cards is common. Many digital multimeters and even some digital voltmeters have standard platinum calibration curves built in and can display temperature directly.
- Suffer mechanical and thermal strain
  - ➢ carefull sensing element mounting
  - ➢ encapsulated in a sealed Pt tube with GHe (30-40 torr)
- Several industrial types are encapsulated in a metal encase with diameters on the order of few mm and length on the order of 10 mm.
  - Time constant 1.3 to 1.6 s
- PT-100: 100 Ohm at 0 °C
- PT-1000: 1000 Ohm at 0 °C
- Packages and adapters for platinum RTDs allow the sensor to be soldered in place, screwed on, bolted down, inserted into a hole, or inserted through a pressure seal in the form of a thermowell



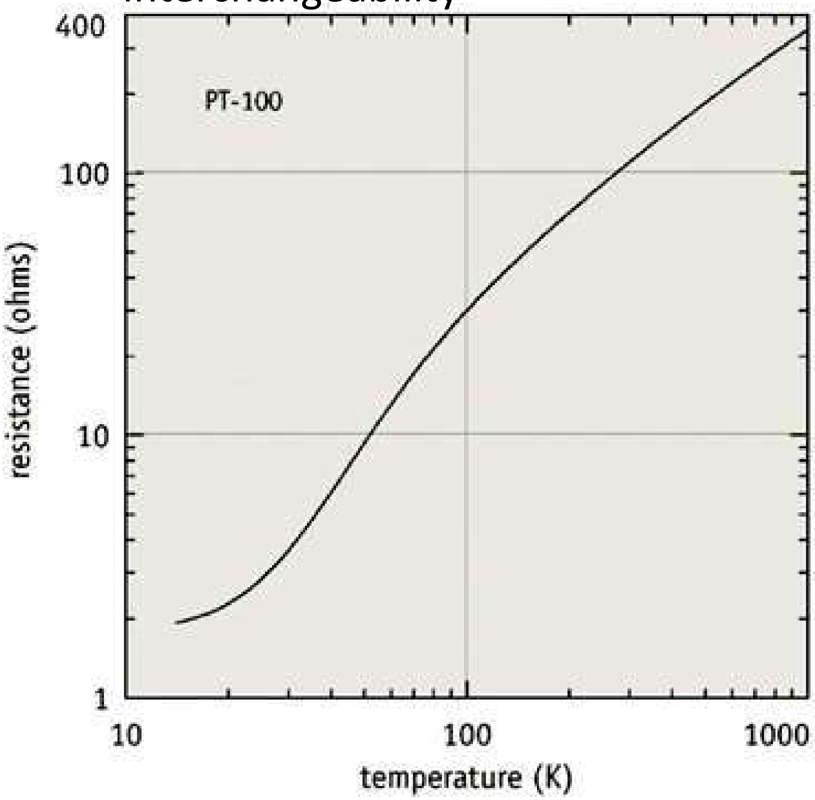
Encapsulated Platinum resistance thermometer



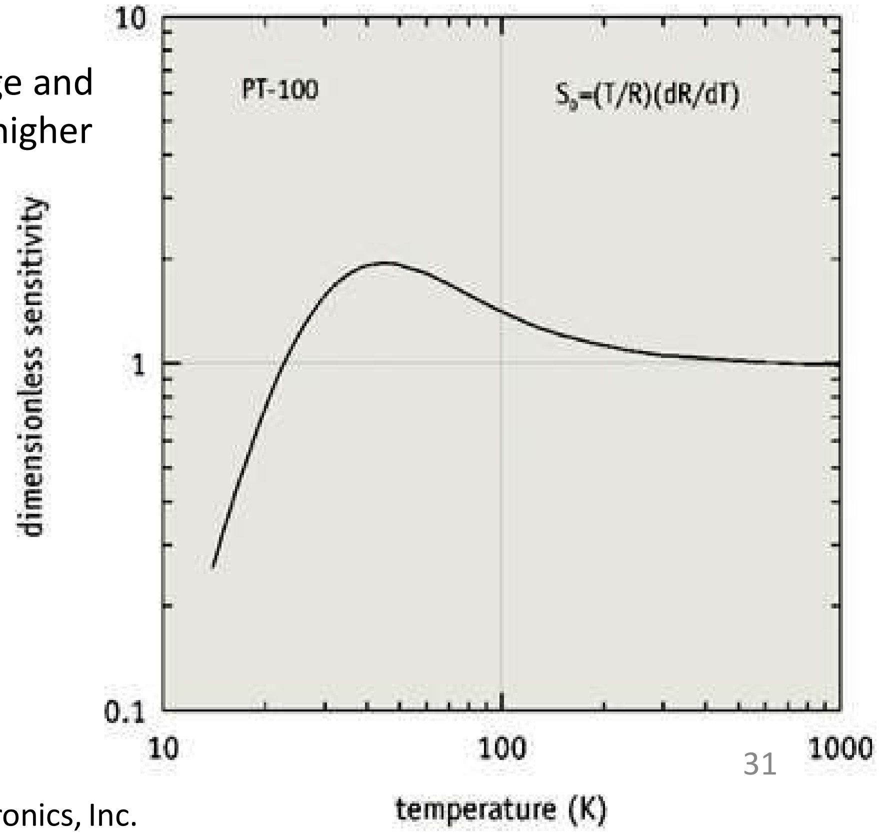
Source: Lake Shore Cryotronics, Inc.

Platinum Resistor Thermometers

- Wide range of quality available, from standard instrument (SPRT) of the ITS-90 to industrial types
- Wide Temperature range: 14 K to 873 K. Good down to ~ 30 K
- Excellent for use in ionizing radiation fields
- Low magnetic field dependence above 40 K (easily corrected)
- Non-magnetic packages available
- **Most accurate and reproducible over a wide range of T, interchangeable**
- Can be calibrated with good accuracy to common calibration curves
- Relatively low cost, most common sensors in the range 77-300 K
- Excitation is generally 1 mA DC
- Interchangeability

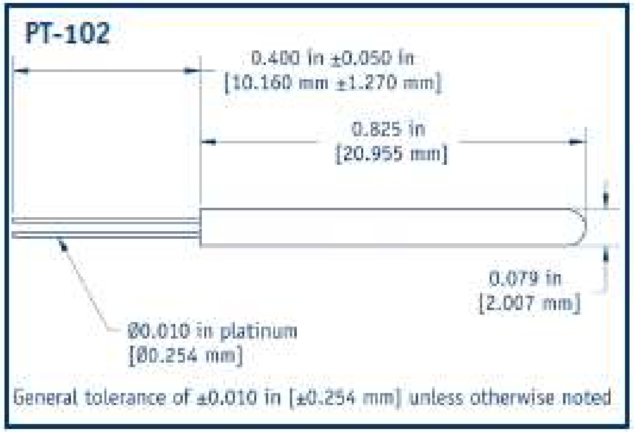


Select the linear range and where sensitivity is higher



Source: Lake Shore Cryotronics, Inc.

PT-100 (Lake Shore)
example: PT-102 model



POSSIBILE OPTION (ex. PT102):
- Adapter material: 6061 Al
- Flat aluminum block with a PT-102 mounted Inside (PT mounted to adapteR using Cotronics Durabond¨ 950 Al-based adhesive)
- Can be mounted to any flat surface with a 6-32 or M3 screw and Inconel Belleville washer
- Mass: 3.8 g
- Limitation: The aluminum alloy limits the upper useful temperature of these configurations to 800 K

Platinum Specifications

General

Standard curve IEC 751

Recommended excitation 1 mA

Dissipation at recommended excitation 100 µW at 273 K

Thermal response time PT-102 & PT-103: 1.75 s at 77 K, 12.5 s at 273 K; PT-111: 2.5 s at 77 K, 20 s at 273 K

Use in radiation Recommended for use in ionizing radiation environments — more information

Use in magnetic field Because of their relatively low magnetic field dependence above 30 K, platinum sensors are useful as control elements in magnetic field applications when some error can be tolerated — more information

Reproducibility[4] ±5 mK at 77 K

Soldering standard J-STD-001 Class 2

[1] Short-term reproducibility data is obtained by subjecting sensor to repeated thermal Cookies *cks from 305 K to 77 K*

Range of use

	Minimum limit	Maximum limit
PT-102	14 K	873 K
PT-103	14 K	873 K
PT-111	14 K	673 K

- Mass: 250 mg
- 2 platinum leads
- Internal Atmosphere: Partially filled powder
- Metrial used: Platinum winding partially supported by a high temperature alumina powder inside a ceramic tube,
- Platinum lead wires
- Vacuum Compatible:No
- Nonmagnetic packaging: Yes

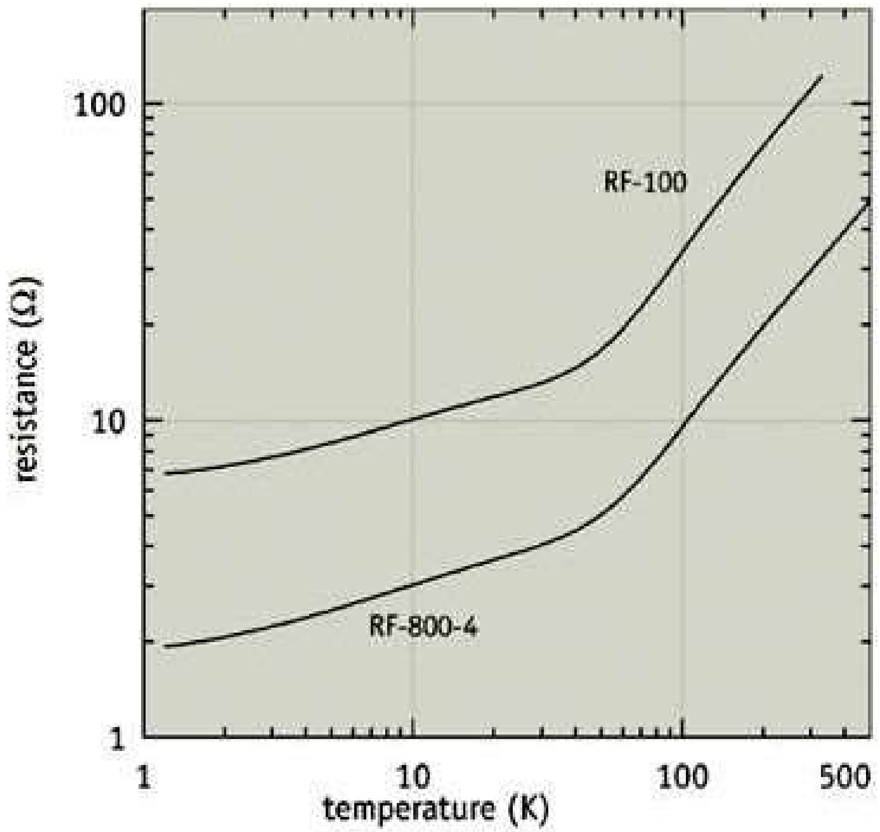
Platinum PT-100

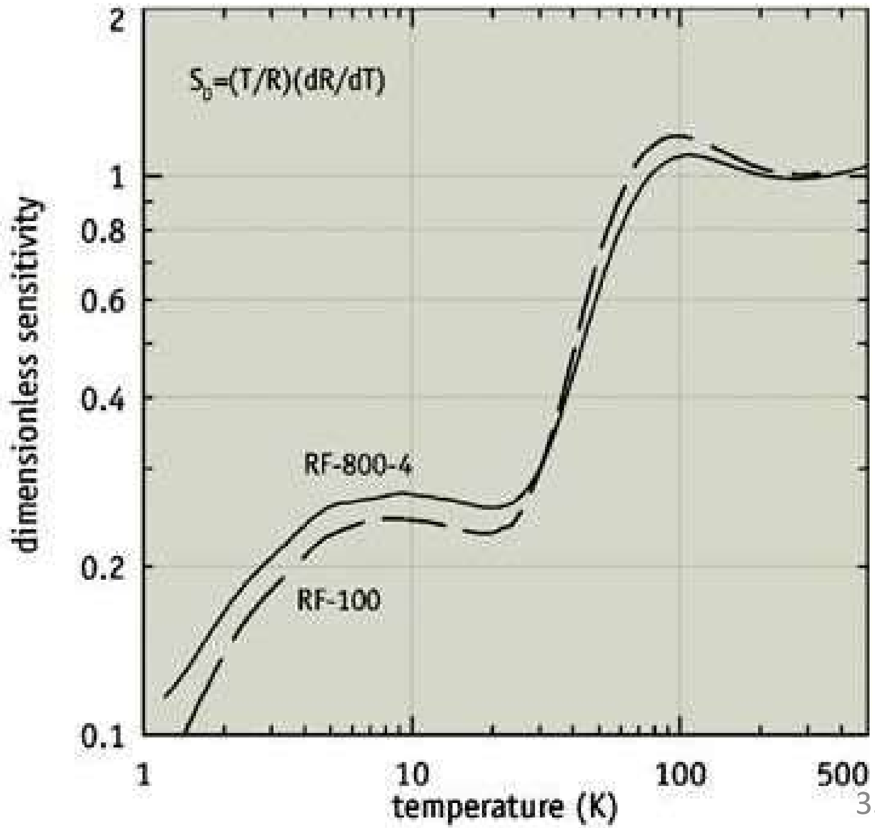
T (K)	R (Ω)	dR/dT (Ω/K)	(T/R)·(dR/dT)
20	2.2913	0.085	0.74
30	3.6596	0.191	1.60
50	9.3865	0.360	1.90
77.35	20.380	0.423	1.60
100	29.989	0.423	1.40
150	50.788	0.409	1.20
200	71.011	0.400	1.10
250	90.845	0.393	1.10
300	110.354	0.387	1.10
400	148.640	0.383	1.00
500	185.668	0.378	1.00
600	221.535	0.372	1.00
700	256.243	0.366	1.00
800	289.789	0.360	1.00

https://www.lakeshore.com/products/categories/specification/temperature-products/cryogenic-temperature-sensors/platinum

RhFe resistor thermometers

- Relatively new sensors, Rhodium with 0.5% Fe
- Behave like Platinum Resisor Thermometers at higher temperatures but more sensitive below 20 K.
- Sensitively cover an **incredible temperature range (0.5 K up to 900 K)** with a single thermometer. Among low-temperature sensors, they also have the **best reproducibility on thermal cycling**, less than 0.1 mK at 4.2 K if they are high-purity and strain-free packaged.
- They can be used as a secondary standard over a very wide range of T.

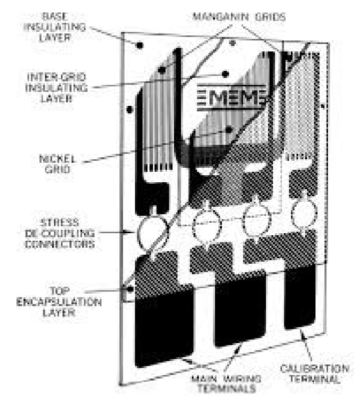


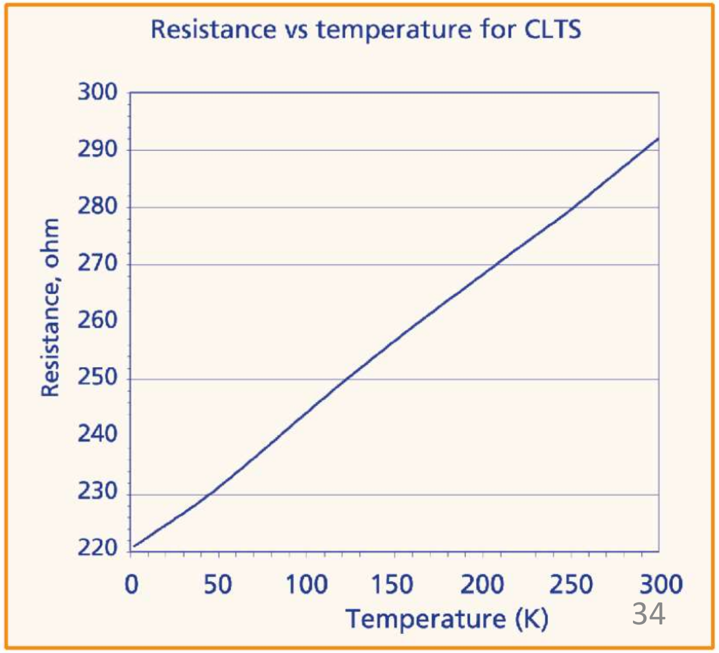


Source: Lake Shore Cryotronics, Inc.

Cryogenic Linear Temperature Sensor (CLTS)

- Flax, flexible, thin-foil sensing grids laminated into fiberglass-epoxy. The two alloys are special grades of nickel and Manganin processed to produce a combined resistance that is very nearly linear in temperature from 4 to 300 K.
- 220 Ohm at 4 K to 290 Ohm at 297 K with a temperature sensitivity of 0.2389 Ohm/K. Maximum linearity deviation about ±3 K.
- CLTS responds quickly to temperature changes and can be plunged directly into liquid nitrogen or helium without damage due to unique design features.
- Tue magnetoresistance at 4-6 K is sufficiently large that the CLTS has been used to measure magnetic fields.
- The CLTS is a useful temperature indicator where high accuracy is not necessary and requires only a 100 µA current source and a voltmeter for instrumentation.
- **Similar to strain gauges**, same readout instrumentation.
- **Linear**



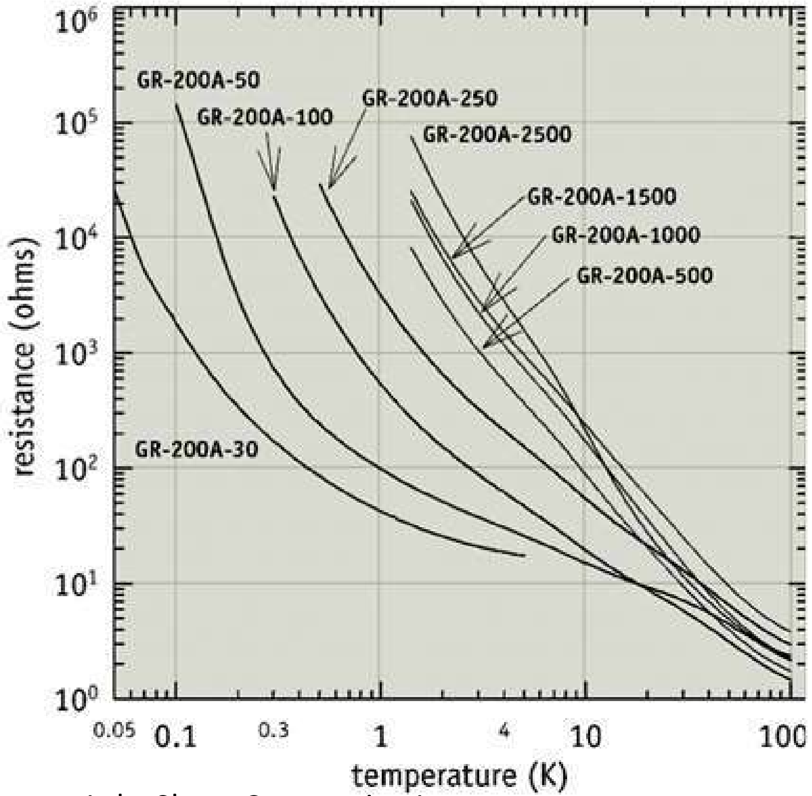


Semiconductor-like resistor thermometers

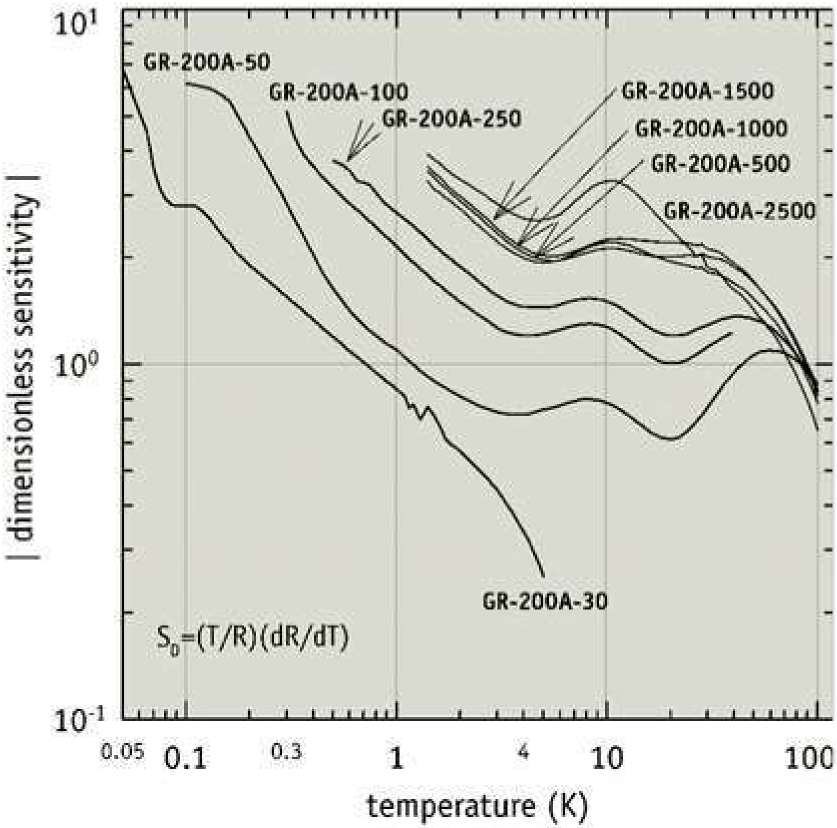
- Semiconducting resistors typically have negative temperature coefficients (NTC).
- Temperature sensitivity is often small near 300 K but increases with decreasing temperature.
- **The resistance can change by 5 orders of magnitude over the sensor's useful temperature range**.
- Semiconductors are typically **piezoresistive**, and thus bulk sensing elements must be mounted strain free, which reduces the thermal connection to the sample whose temperature is to be measured. Consequently, **self-heating can be significant** unless measurement power to the device is kept extremely small. The large resistance change coupled with thermal considerations results in a requirement for a variable current source for measurement in which the current must be varied from about 0.01 µA to 1 mA or more as well as a voltmeter capable of measuring voltages as small as 1 µV.
- The most common NTC temperature sensors are made from carbon, carbon-glass, germanium, Cernox ™, and ruthenium oxide.

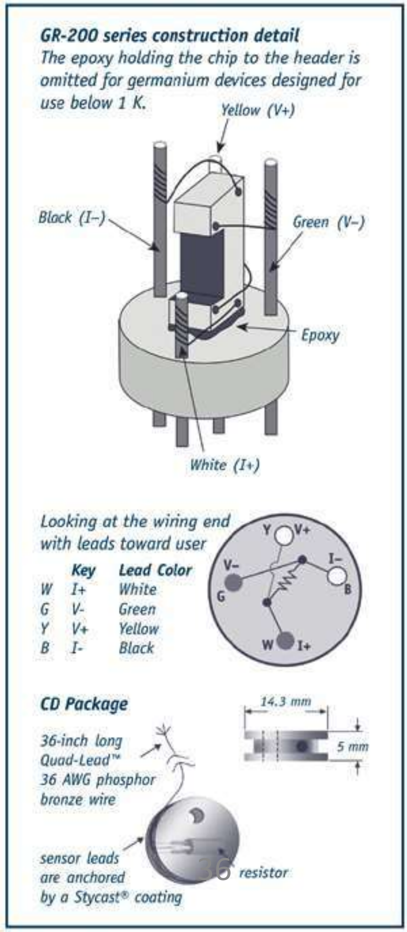
Germanium resistor thermometers

- Germanium crystal doped with arsenic, gallium, antimony.
- A single device typically has a useful T **range of about two orders of magnitude** (e.g., 0.05 to 5 K or 1 to 100 K) and can normally be used to submillikelvin contro! in the lower portion of its range.
- The main advantage**: reproducibility on thermal cycling**, **extremely good stability** (about ±0.5 mK at 4.2 K) and are probably the best choice for high-accuracy work under 30 K when magnetic fields are not present.
- Recognized as a "Secondary Standard Thermometer” .
- **Very fragile**. Must be strain-free mounted. Sensitive to self-heating thus requiring lowering of excitation current at low T, where they have relatively high resistance at low T (high inputimpedance voltmeter for readout)
- Interchangeability is poor,



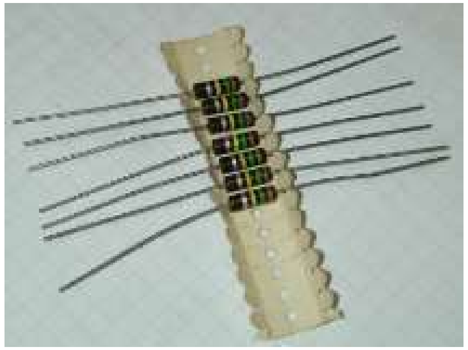
Source: Lake Shore Cryotronics, Inc.

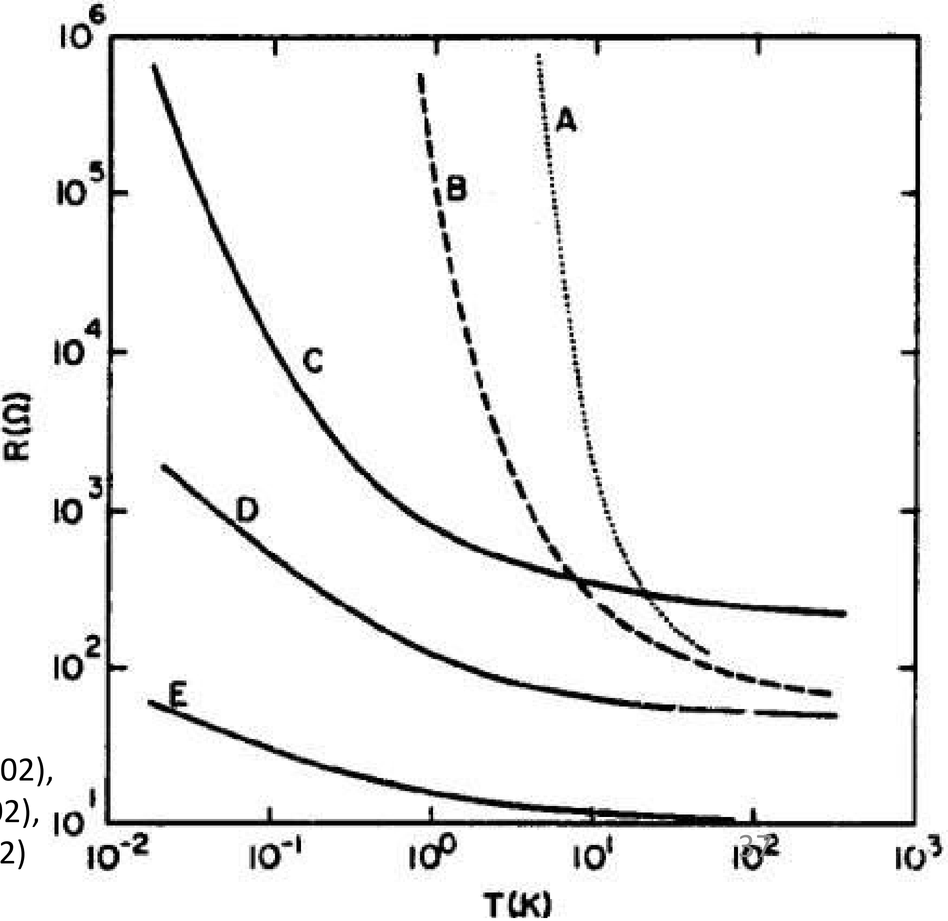




Carbon resistor thermometers

- Carbon RTDs are made from common radio resistors.
- 1/2 W Speer resistors and 1/8 W AllenBradley resistors, have been used for many years as cheap thermometers for control in magnetic fields. However, their run-to-run reproducibility is not as good as thermometers developed more recently, and they have been replaced for magnetic-field use, in large part, by zirconium-oxynitride sensors.
- Nevertheless, they are still commonly used as an inexpensive thermometer for **<u>cryogen-level sensing</u>**
- On the plus side, they are cheap, physically small with low heat capacity, have fast time response.

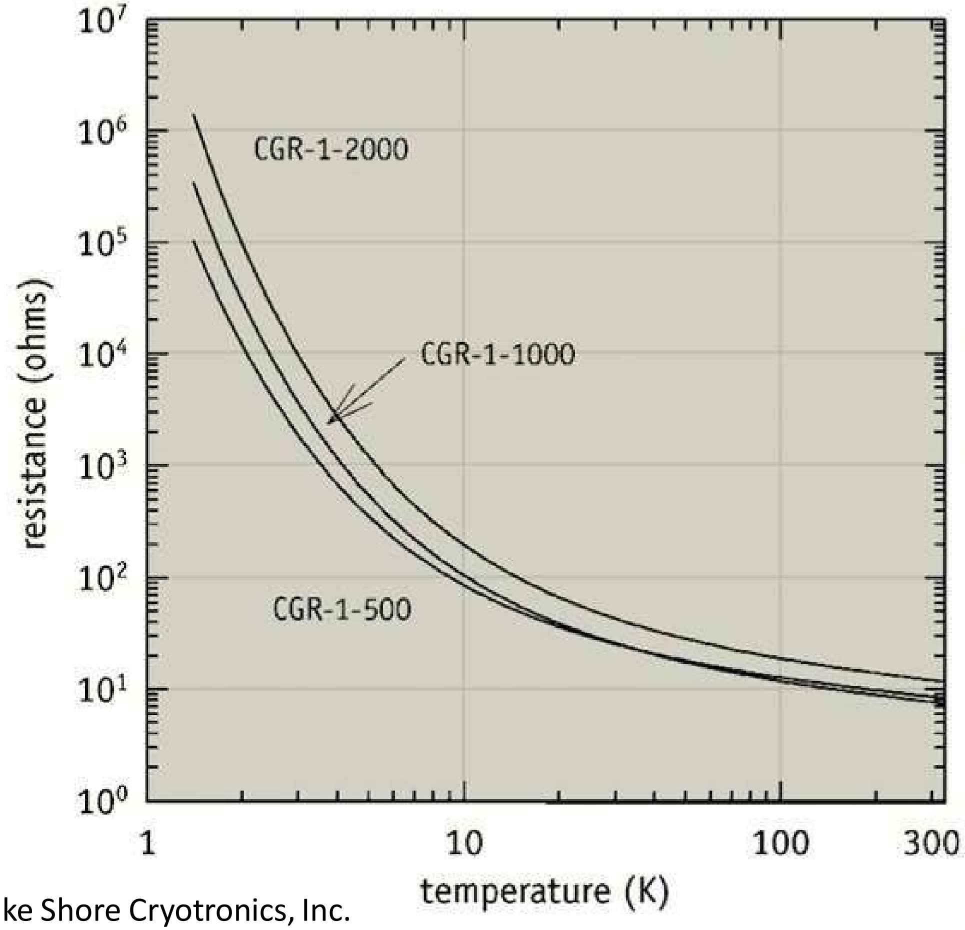


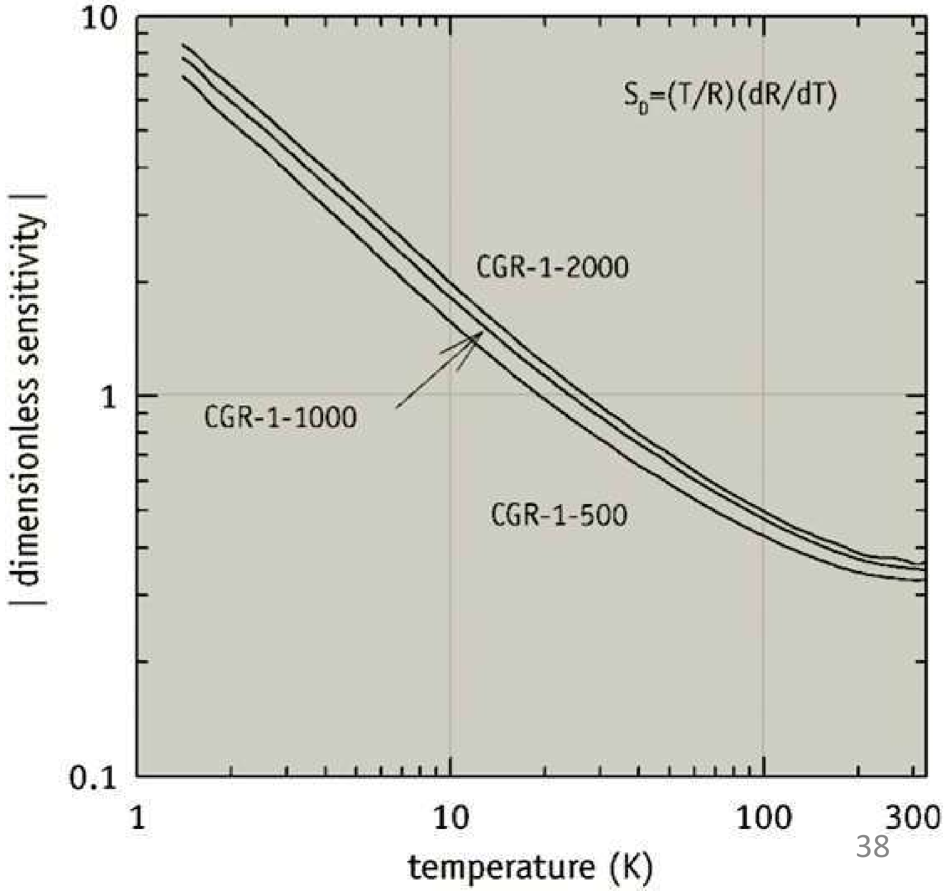


A: thermistor,
B: 68 Ω Allen-Bradley,
C: 220 Ω Speer (grade 1002),
D: 51 Ω Speer (grade 1002),
E: 10 Ω Speer (grade 1002)

Carbon glass resistor thermometers

- Carbon-glass RTDs are bulk devices manufactured from carbon impregnated glass.
- The R-T characteristic is monotonic over 1 - 325 K and very reproducible below 100 K.
- **Very high sensitivity below 10 K and useful for submillikelvin control.**
- These devices behave very well in magnetic fields in fields up to 19 T, and accurate corrections can be made to their field-induced temperature errors.
- Strain-free mounting in a canister is required. The contacts to the carbon-glass are a weak point and are prone to failure after repeated thermal cyclings.



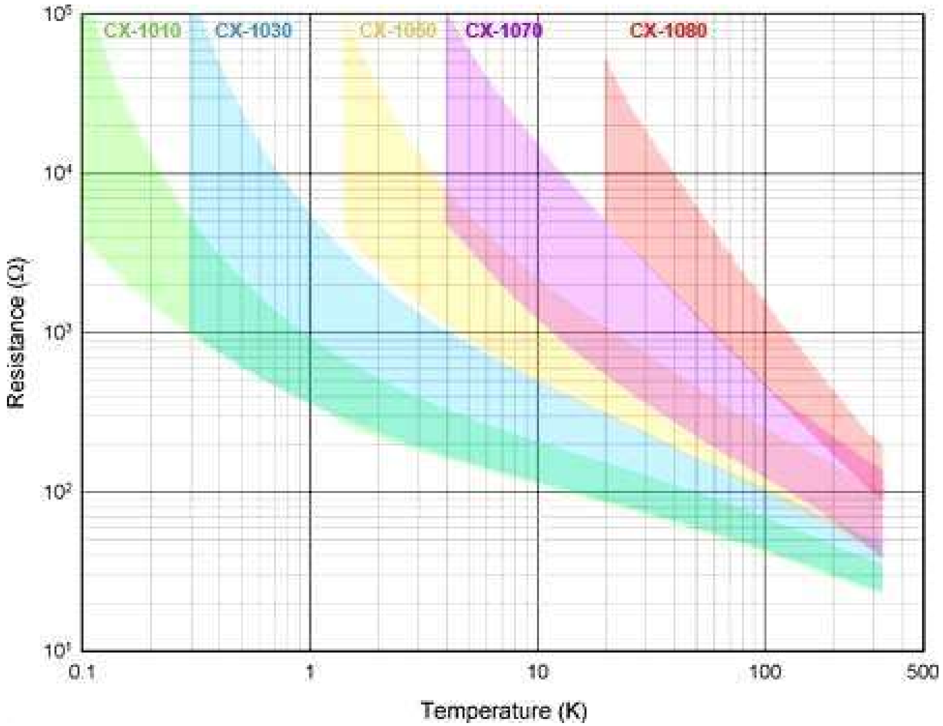


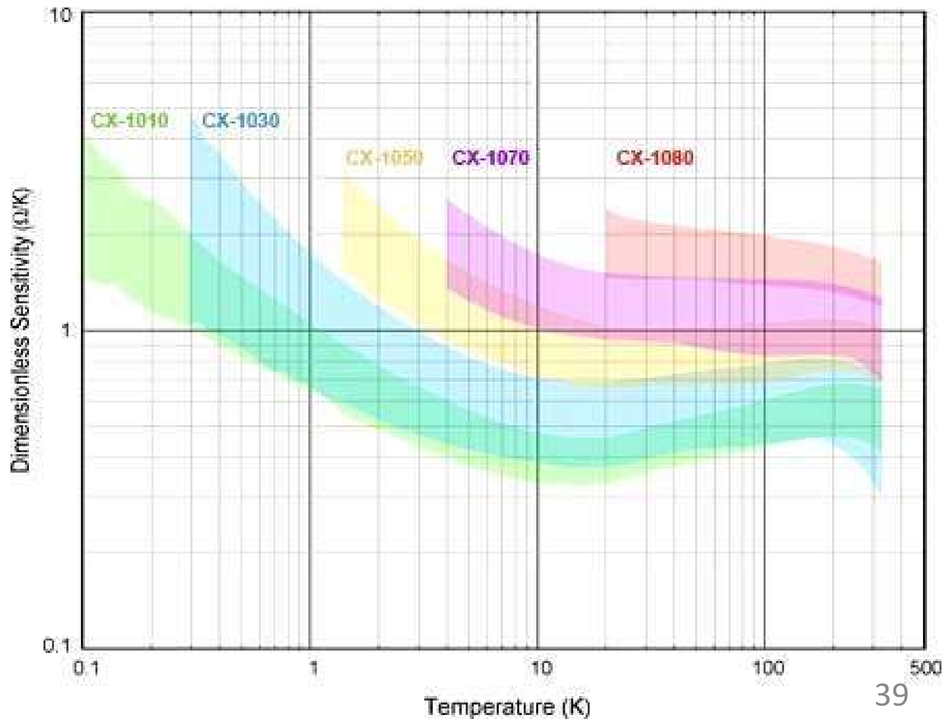
Source: Lake Shore Cryotronics, Inc.

Cernox resistor thermometers

- Zirconium-oxynitride (Cernox™): ceramic oxynitride thin film sputtered onto a sapphire substrate, manufactured by Lake Shore, USA.
- Monotonic response curve, the best single thermometer for covering the entire cryogenic range, even in the presence of high magnetic fields
- Robust resistors mounted in packages designed for excellent heat transfer resulting in much faster thermal response times than those for bulk devices requiring strain-free mounting
- Expensive
- Non optimal reproducibility.
- Requires individual calibration for best results
- Very good in ionizing radiation environments
- **small magnetic field coefficient**:

D*T/T* typically smaller than 0.5% @ 19 T

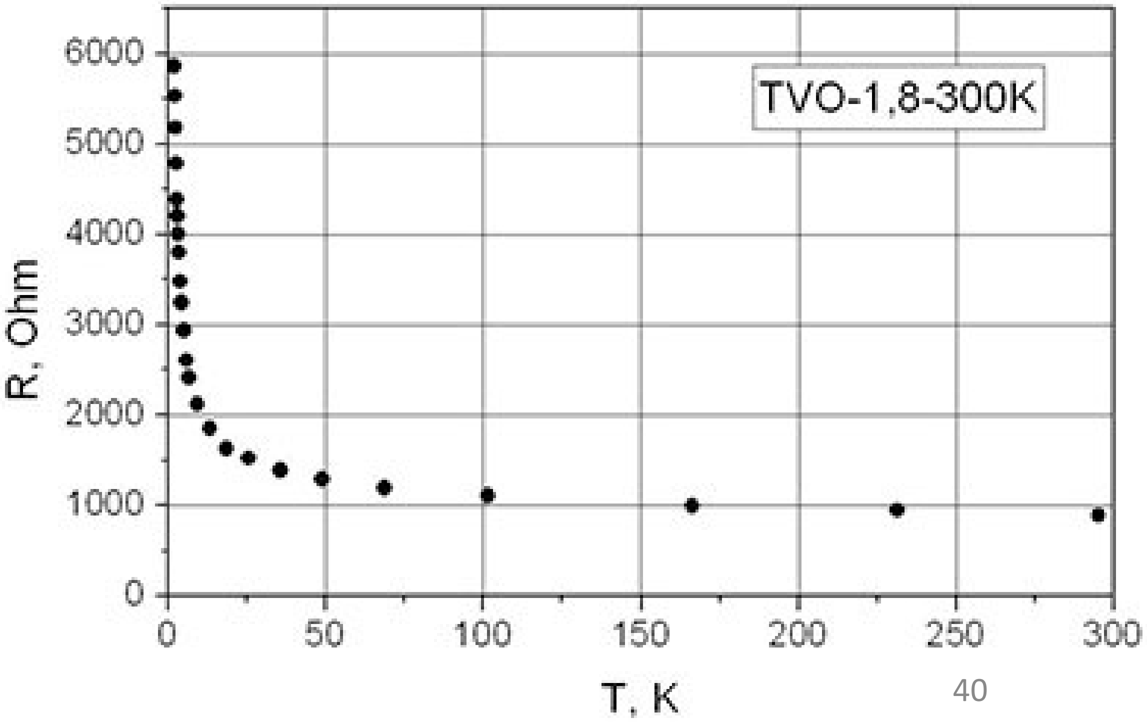




Source: Lake Shore Cryotronics, Inc.

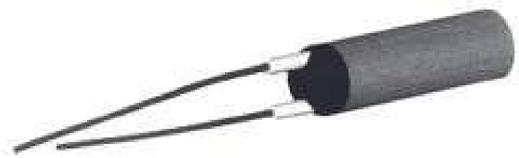
TVO resistor thermometers

- Safety control for superconducting solenoids:
  - MR imaging unit
  - ESR spectrometer
  - NMR spectrometer
- Russian Competitor of the Cernox
- Characteristics:
  - Resistance at R.T. 910 Ohm
  - Resistance spread at R.T. 5%
  - Resistance shift in 8 T magnetic field at 4.2 K: 4%
  - Repeatability after 1 year at T = 4.2 K: 0.01
  - Sensor dimensions: mm1 x 2.5 x 8
  - Whole length of resistors including current leads and potential contacts solderin: 60 mm



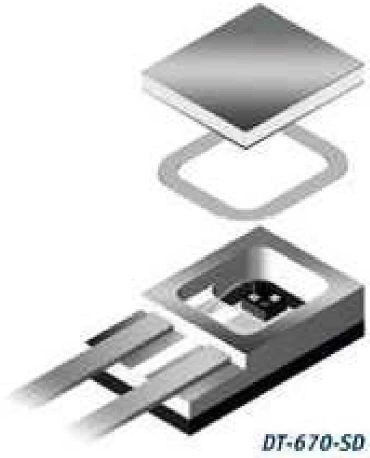
Ruthenium Oxide resistor thermometers

- Ruthenium oxide RTDs are typically thick-film sensors fab-ricated from ruthenium oxide or bismuth ruthenate pastes (proprietary).
- These thermometers are useful **down to quite low temperatures, about 50 mK**. At higher temperatures not as sensitive as other sensors, but useful over the temperature range 0.05-40 K, and especially well suited for sensitive temperature measurements below the lambda point of helium (2.2 K).
- Widely used in magnetic field applications at low T.
- Interchangeability of their calibration, but only within one manufacturer's lot.
- These sensors are more easily damaged by thermal cycling at low temperature than other thermometers.
- At temperatures below 1 K the thermal resistance is large thus low excitation power is required to avoid self-heating.



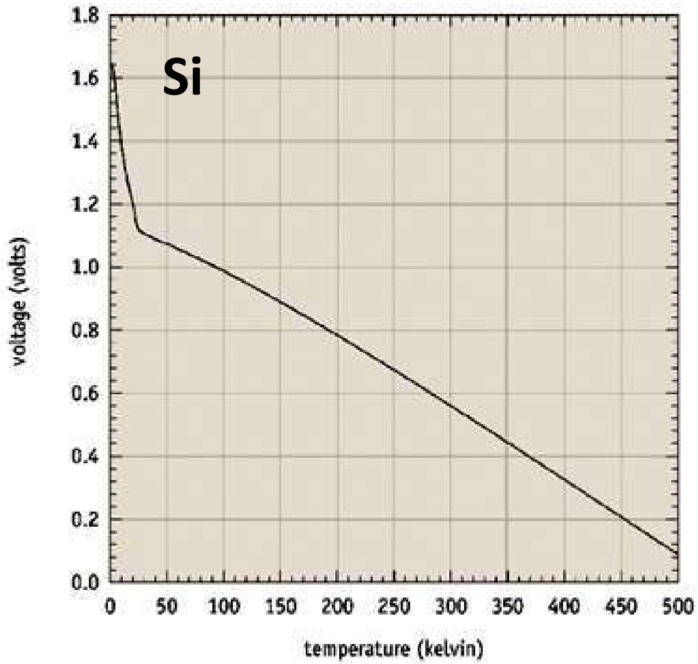
Silicon Diode Voltage Thermometers

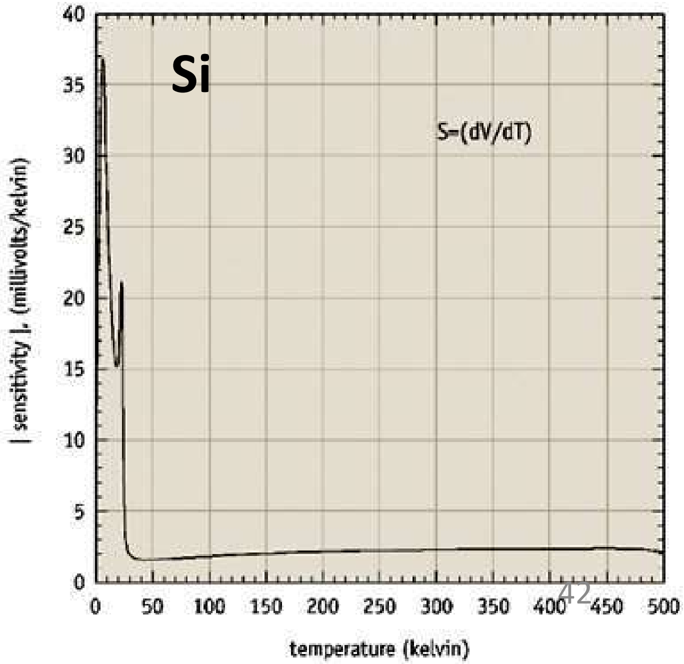
- Si diodes: choice for zero-magnetic-field "industrial" quality thermometry over the 4-300 K range. Fairly inexpensive.
- **Easy to use**. Only a fixed-current source (typically at 10 µ,A) and a voltmeter with a range of 0.1 to 6 V are required.
- **Small size** useful when small thermal mass is needed (for rapid temperature response) or where space is restricted.
- Readily interchanged, albeit with somewhat less accuracy than individually calibrated sensors. A "standard" calibration curve for a common silicon diode is given.
- Disadvantages: **no absolute accuracy**, and the reproducibility on thermal cycling is not high. For high accuracy, each silicon sensor must be measured individually.
- Voltage across the diode can be measured only in the forward direction, thus the voltmeter must now make an absolute measurement. Without current reversal, thermoelectric voltages and voltmeter offsets may be present, affecting the achievable accuracy. . Current noise through a di ode produces a shift in the temperature reading caused by the nonlinear current-voltage characteristic of the diode.



Source: Lake Shore Cryotronics, Inc.

- A transition region in conduction mechanism around 20 K makes fitting a V-T characteristic over the whole temperature range difficult for GaAlAs and impossible for Si.
- GaAlAs diodes are used with magnetic field. Voltage sensitivity is higher than that of Si diodes, but each sensor must be calibrated.



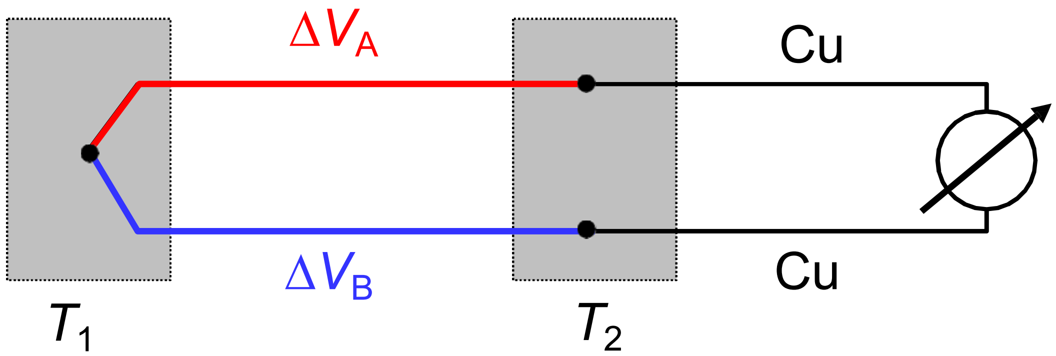


Thermocouples

- Thermocouples are pairs of dissimilar metal wires joined at least at one end, which generate a net thermoelectric voltage between the open pair according to the size of the temperature difference between the ends, the relative Seebeck coefficient of the wire pair and the uniformity of the wire-pair relative Seebeck coefficient.

- based on Seebeck effect: $$\Delta V = S \cdot \Delta T$$ S = thermopower

- measurement of difference of thermovoltages of two different materials



well-known reference temperature

➢ No need to be feeded

Types of thermocouples

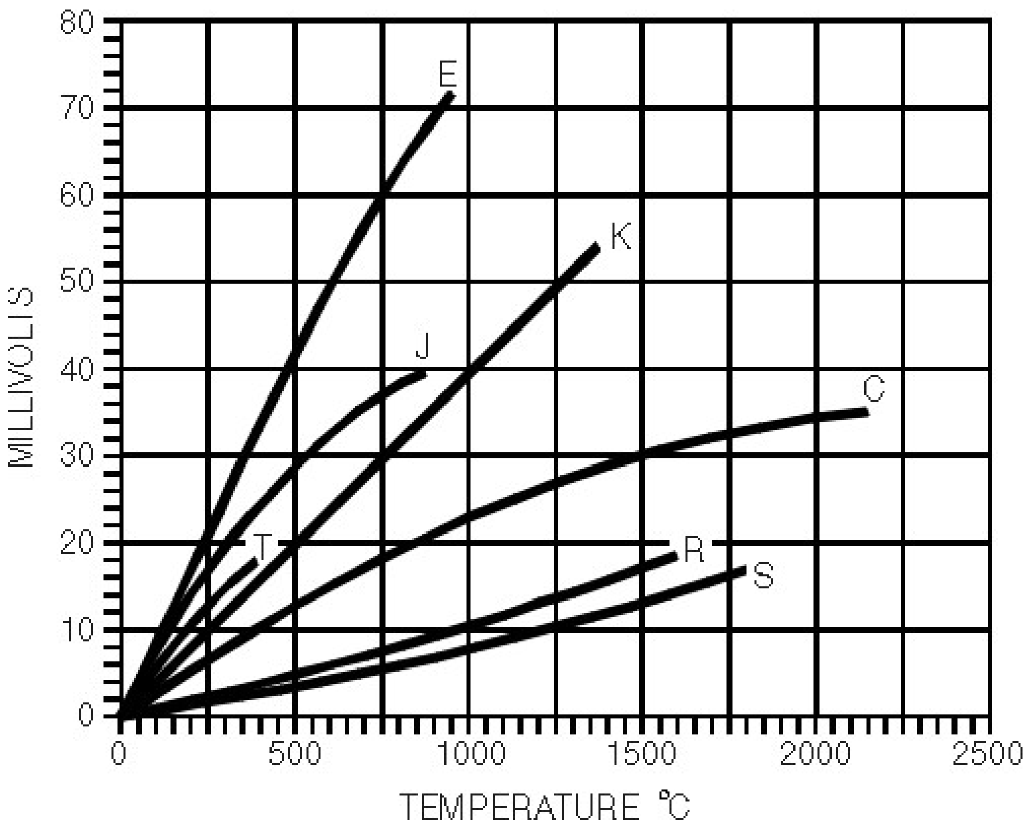
- **Chromel-Gold/Iron (0.07%)**
  consists of a Gold (Au)-0.07 at % Iron (Fe) as the negative thermoelement and a Ni-Cr alloy (Chromel) as the positive thermoelement. This thermocouple is more widely used because of ist relatively high thermoelectric sensitivity (>15 µV/K above 10 K).

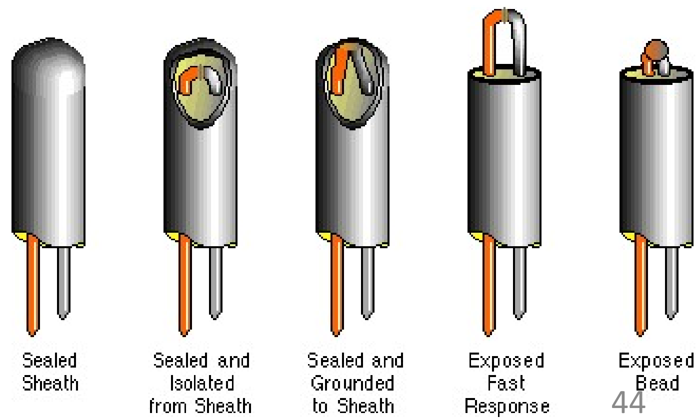
- **Type E (Chromel (Ni-Cr-alloy) / Constantan (Cu-Ni-alloy))**
  has the highest sensitivity among the three standard thermocouple types typically used at low temperatures (types E, K, and T). The best choice for temperatures down to 40 K.

- **Type K (Chromel (Ni-Cr-alloy) / Alumel (Ni-Al-alloy))**
  recommended for continuous use in inert atmospheres. Has a sensitivity of 4.1 mV/K at 20 K (about ½ of Type E).

- **Type T (Copper / Constantan)**

many more !!



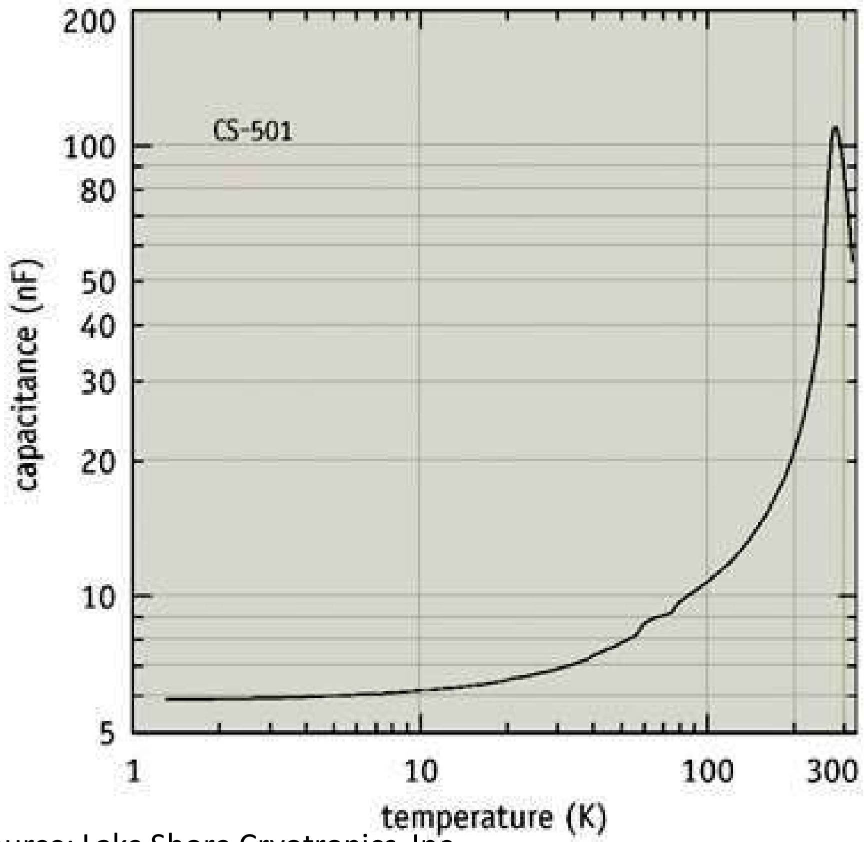


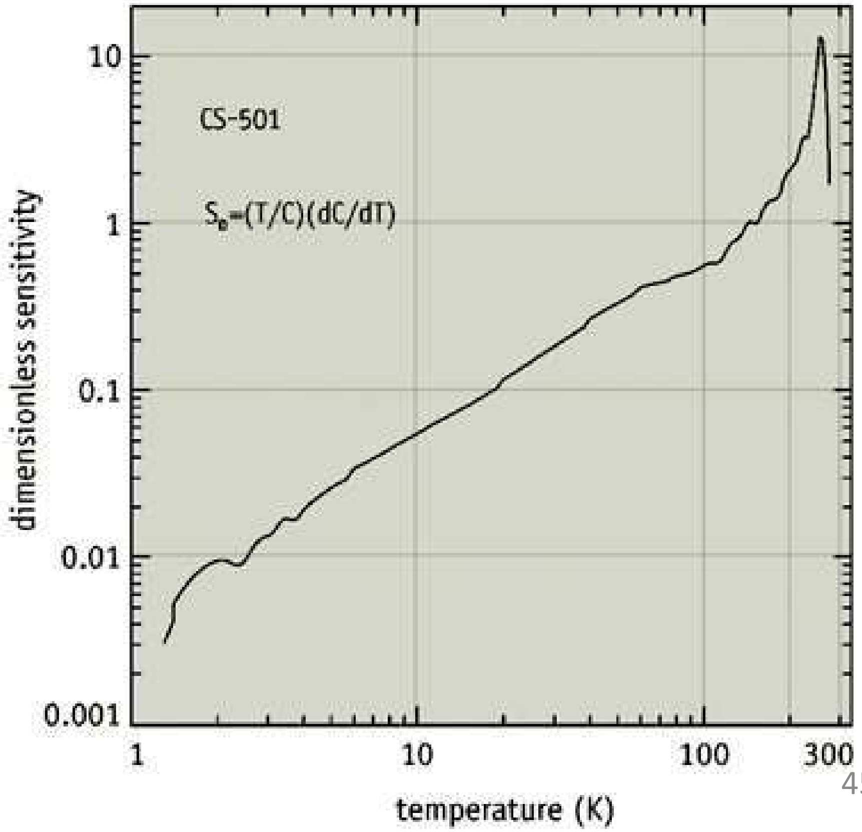
Thermocouple Sheath Options

Capacitive Thermometers

- Based on the well defined relation between the dielectric constant and temperature
- Temperature is determined via a capacitance measurement
- Advantage: virtually no magnetic field dependence
- Due to high reproducibility error, the SrTiO3 capacitance thermometer is useful **only for control of temperatures** in high magnetic fields. Fluctuations are kept to a minimum when sweeping magnetic field or when changing field values under constant temperature operation.

CS-501GR





Source: Lake Shore Cryotronics, Inc.

Practical choice (general applications)

- WITHOUT magnetic field
  - Platinum resistance thermometers: 77-300 K
  - Germanium or Zirconium-oxynitride resistance (Cernox): 0.5 – 77 K (full range)
  - Silicon diode thermometer: 1.5 – 300 K (full range)
  - As level meters: Carbon-composition resistors, Silicon diodes
- WITH magnetic field
  - Zirconium-oxynitride resistance (Cernox) thermometers: 0.5 – 77 K (full range)
  - Platinum resistance thermometers: 77-300 K
  - For T control in changing magnetic field: Capacitive sensors 1-290 K
- RADIATION (GAMMA, NEUTRONS)
  - Germanium, Zirconium-oxynitride resistance (Cernox) or Rhodium-Iron

Specific applications

For some particular applications:

- Limited space
- Specific measurements
- Calorimetry
- Thermal response time
  - correlated to sensor mass (mainly due to packaging mass)

➢ Small sensing elements, for instance:

- Thermocouples (gold-iron vs Chromel)
- Silicon bolometers

Temperature Sensor Overview

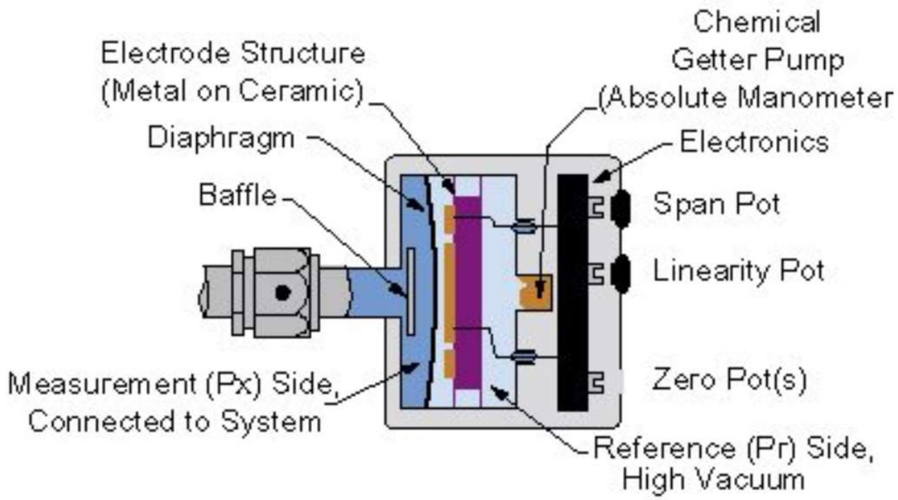
Sensor	T range (K)	Individual calibration	Reproducibility	Interchange-ability	Magnetic field use	Best Use	Cost
Platinum Resistance Thermometer	77-800 (20-77 special)	Not necessary	Excellent	Excellent	Yes	Reccomended >70 K Low cost	Low (High with individual calibration)
Cernox Resistance Thermometer	0.3-325	Yes	Yes	No	Excellent	Magnetic field Full range	High with individual calibration
Germaniun Resistance Thermometer	0.05-100	Yes	Excellent	No	No		High with individual calibration
Silicon diode Thermometer	1.4-450	Not necessary	Good	Good	No	Small Easy Full range	Medium ()High with individual calibration

Pressure Measurement

- Carry out at room temperature where possible (using a capillary tube)
- Problems with room temperature pressure measurement
  - Thermal Acoustic Oscillations
  - Time response will be too slow for high speed transients but for most operations this is not an issues
    - High speed pressure pulse due to magnet quenching is an exception
- Some cold pressure transducers exist that solve these problems
- There are a wide range of 300 K commercial pressure transducers that exist
  - Many are based on **capacitive sensors or strain gauge ($\Delta L/L$) bridges mounted on diaphragms that change shape with pressure**

Pressure Measurements

- <u>Absolute measurement</u>:
  - one port connected to the pressure of interest. A pressure reference cavity is built into the sensor. A vacuµm reference should be used for cryogenic measurements, or the reference pressure will change with temperature, especially if the reference atmosphere condenses and freezes.
- <u>Relative/gauge measurement</u>:
  - consists of one port connected to the pressure of interest; a hole in the pressure sensor body allows measurement of pressure relative to the ambient.
- <u>Differential measurement</u>:
  - two ports connected to two pressure sources. Common applications include Venturi flowmeters or measurement of pressure drops due to flows through piping. Differential ports are typically capable of measuring small pressure differences.



Cryogenic pressure sensors

Table 4-14 Properties of some commercially available cryogenic pressure sensors

Type[a]	Manufacturer	Model	P range FS [kPa]	Style[b]	T range [K]	Excitation	Span [mV]	Linearity and hysteresis	Mass [g]	Cost [$]
BSG	Precise Sensors	111-3	1400 to 105 000	A	77–422	10 Vdc, 286 mW	20	±0.5%	170	770
BSG	Sensotec	Cryo Series	170 to 70 000	A, D	77–300[c]	10 Vdc, 286 mW	10–30	±0.25%	370	485
PE	Kistler	601B1	1000 to 100 000	Y	6–533	[d]	[d]	±1%	7	400
PE	PCB Piezotronics	102A	700 to 70 000	Y	20–373	2mA	5000	1%	11	660
PR Si	Endevco	8510B/C	7 to 14 000	D	219–394[c]	10 Vdc, 37 mW	300/220	±0.25%	2.3	610
PR Si	Endevco	8515C	100 to 34 000	A	219–394[c]	10 Vdc, 37 mW	200	±0.2%	0.08	590
PR Si	Kulite	CCQ-093	700 to 3500	A, D	77–393	10 Vdc, 125 mW	100	±0.1%	0.4	510
PR Si	Kulite	CT-190	350 to 14 000	A, D	77–393	10 Vdc, 125 mW	100	±0.1%	4	650
PR Si	Kulite	CT-375	350 to 14 000	A, D	77–393	10 Vdc, 125 mW	100	±0.1%	25	680
PR Si	Pressure Systems	4100,4200	7–3500	D, A	1.5–400	1 mA, 6 mW	150–350	±0.5%	75	695
PR Si	Siemens[e]	KPY-10,12	200	A, D	1.4–400	500 μA,1.5 mW	51	±0.7%	3	25
PR Si	Siemens[e]	KPY-14,16	1000	A, D	1.4–400	500 μA,1.5 mW	120	±0.7%	3	25
PR Si	Siemens	KPY-32R, 33R	5,10	D	1.4–400	500 μA,1.5 mW	21,24	±1.0%	5	25
PR Si	Siemens	KPY-43A, R	160	A, D	1.4–400	500 μA,1.5 mW	42	±0.4%	5	25
PR Si	Siemens	KPY-47A, R	6000	A, D	1.4–400	500 μA, 1.5 mW	120	±0.4%	5	25
PR Si	Toyoda Machine	PD116S	3000	G	4–300	100 μA, 8.5 μW	2	±2%		
PR ss	Omegadyne	PX1005	100 to 70 000	A	113–422[c]	10 Vdc, 250 mW	30	±0.25%	145	1500
PR SOS	Sensotron	SEN-202B-30	202	A	4–300	1 mA, 4.6 mW	15	±0.25%	170	225
VR	Validyne	DP10	0.5 to 22 000	D	1.4–390	5 Vac, 3 kHz	150	±0.7%	330	640

Note: Specifications are for operation at 298 K.

[a] BSG = bonded strain gage; PE = piezoelectric (dynamic pressure); PR Si = peizoresistive, Si diaphragm; PR ss = piezoresistive, stainless steel diaphragm; SOS = silicon on sapphire; VR = variable reluctance.

[b] A: absolute, D: differential, G: gauge, Y: dynamic.

[c] Operation possible at lower temperatures.

[d] Requires a charge amplifier for voltage output; prices vary from $400 to $1200.

[e] No longer in production.

Flow Measurements

- Many types of flowmeters have been used successfully at cryogenic temperatures.
- A variety of techniques are available mostly the same ones as used in standard fluid mechanics including:
  - Venturi Flowmeters
  - Orifice plate Flowmeters
  - Turbine Flowmeters
  - Coriolis Flowmeters
- Measure:
  - Mass flowmeters
  - Volumetric flowmeters
  - Differential pressure flowmeters
- Comments
  - Insure that the devices are calibrated for operation at the temperatures and pressures that you are expecting (use appropriate fluid properties)
  - Beware of situations that can result in unplanned two-phase flow
  - Allow sufficient length for flow straightening if required (e.g. Venturi)
  - If possible install the flow meters in the 300 K portions of the flow

Cryogenic flow metering techniques

- Single phase flows
  1. Pressure drop devices based on Bernoulli Principle
     a) Venturi
     b) Orifice plate

$$\Delta p = p_1 - p_2 = 1/2 \ \rho \ (v_2^2 - v_1^2)$$

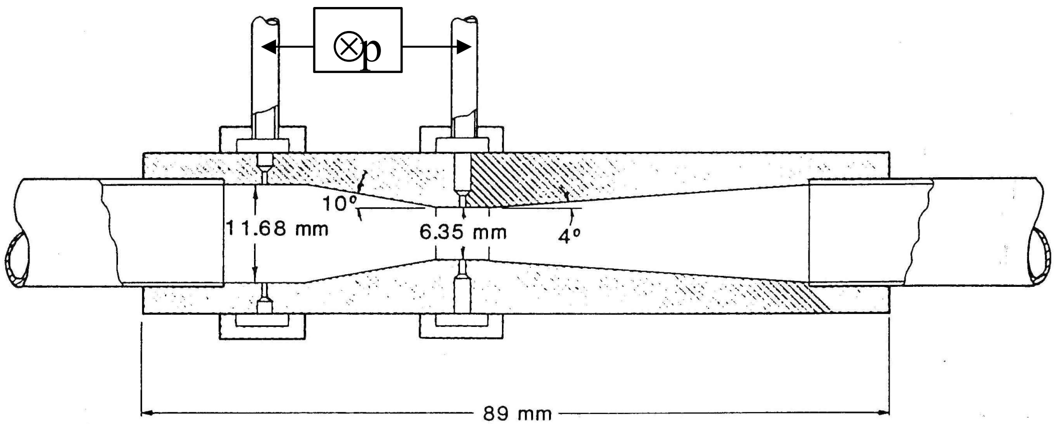
  2. Coriolis
  3. Turbine flow meters where frequency ~ velocity

- Two phase flows
  1. Coriolis
  2. Void fraction measurement (A_v/A)
     a) Capacitance measurement
     b) Optical characterization
  3. Quality measurement (m_v/m)

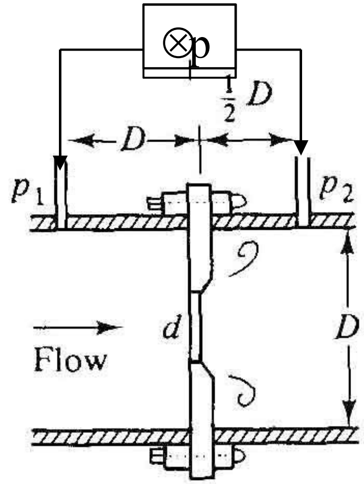
These techniques are for the most part all used in classical fluid flows.

The unique "cryogenic" features have to do with instrumentation used to detect signal and need for low heat leak.

Differential pressure flowmeters



Venturi



Orifice

$$\dot{m} = k_d\, E\, \varepsilon\, A_{or}\, (2\rho\, \Delta p)^{1/2}$$

$$\Delta P = \frac{\dot{m}^2}{2\rho\ (k_d\, E\, \varepsilon\, A_{or})^2}$$

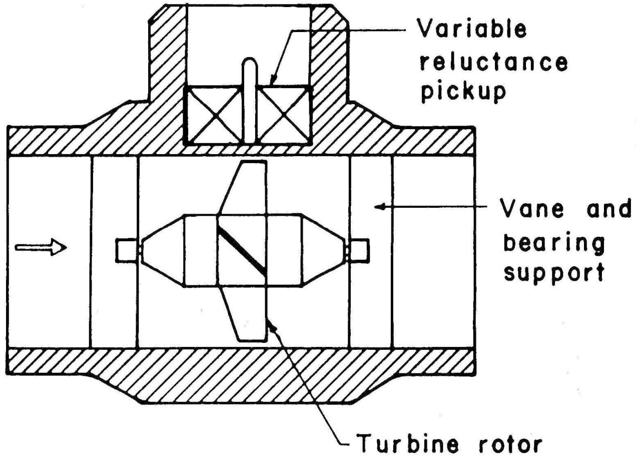
$E = 1/(1-\beta^4)^{1/2}$ $\beta = d/D$ $\varepsilon = 1$

K_d = 0.6 (orifice), 1 (venturi)

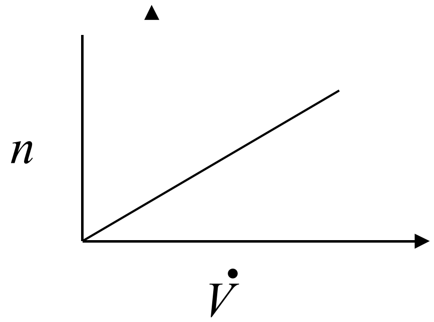
- Pressure drop across some flow element is a function of the flow rate.
- They have **no moving parts** and can be **highly reliable**.
- Venturi flow meters have advantage over orifice plate due to low loss coefficient (turbolence and pressure drop are reduced). The throat diameter is usually about one-half the tube diameter.
- C_d is the discharge coefficient (~ 1 for venturi & 0.6 for orifice)
- Pressure transducer should be located at low temperature, if possible
- A more common approach is to use capillary tubes and a warm transducer
- Requires determination of density at meter inlet

Turbine flowmeters

- Turbine flowmeters indicate flow rate by measurement of the rotational speed of a freely spinning turbine wheel placed in the center of the flowing stream.
- A permanent magnet is placed in the meter body. Each time a turbine blade passes the face of the magnet, the change in the permeability of the magnetic circuit produces a voltage pulse at the output terminal.
- Rotation speed is proportional to volumetric flow rate
- Linear response function allows a wide range of operation
- Turbine flowmeters are true volumetric-flow measurement instruments and are unaffected by changes in density of a single-phase fluid, but in case of 2-phase flow they have to be combined to a quality measure meter.



$$\dot{V} = \frac{\pi D_b A_f}{\tan \theta_b} n = Kn$$



Coriolis Flowmeters

- Coriolis flowmeter has one or more measuring tubes which an exciter causes to oscillate artificially. As soon as the fluid starts to flow in the measuring tube, additional twisting is imposed on this oscillation due to the fluid's inertia. Two sensors detect this change of the tube oscillation in time and space as the "phase difference." This difference is a direct measure of the mass flow.

Figure 1 Coriolis meter sensor and transmitter

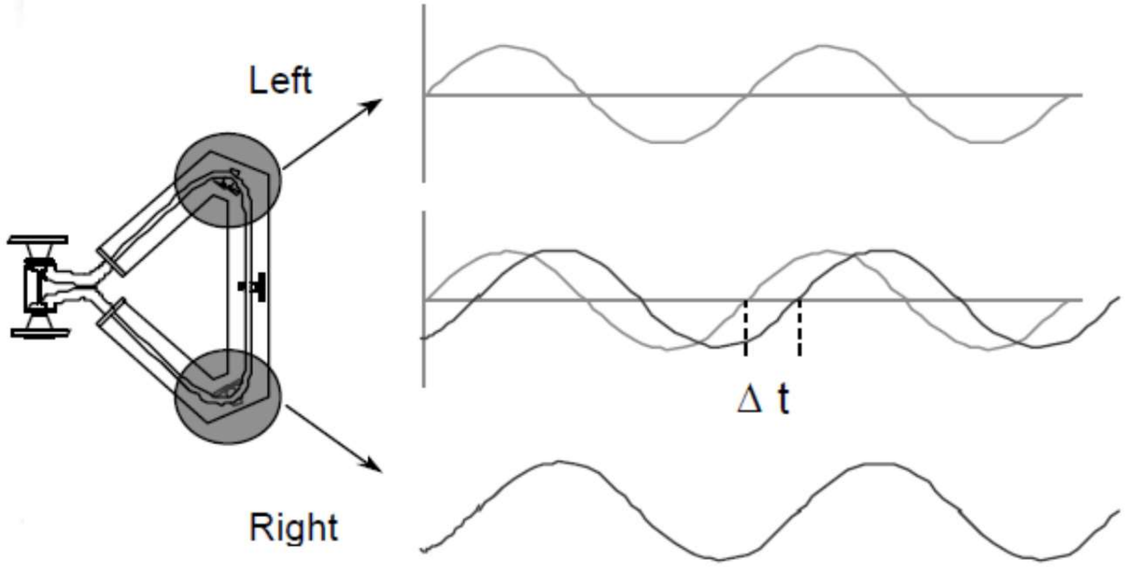


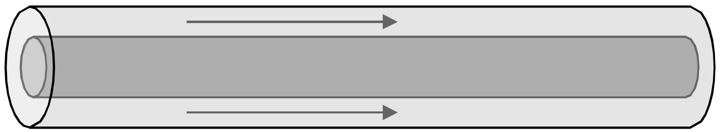
Figure 2 Coriolis flow meter operation

Tested down to 1.7 K at CERN LHC
Flow produces vibrations in the flow tubes that have a phase offset directly related to mass flow

From *Development of a mass flowmeter based on the Coriolis acceleration for liquid, supercritical and superfluid helium*
de Jonge T. et al. Adv. Cryo. Engr. Vol 39 (1994)

Two phase flow measurement

- Measurement of flow quality (m_v/m) in a two phase mixture (liquid + vapor) is difficult.
  - Vapor velocity and liquid velocity may be different
  - Flow regime is not known
- Measurement of void fraction (A_v/A) is more straightforward
  - Capacitive meter based on different dielectric constant



Co-axial capacitor

  - Optical techniques

- Total mass flow rate can be determined in some part of the circuit where the fluid is single phase using a conventional flow meter

RF Void Fraction Measurement

Fluid quality measurement

Quality-measuring probe based on the variation of the fluid dielectric constant with quality (probe capacitance may be related to the fluid quality).

It consists of a coaxial capacitor inserted in an oscillating resonant circuit. Due to the fact that the dielectric constant differs of about 4% from the pure vapor to the pure liquid for He at 4.5 K,the resonant frequency of the circuit changes accordingly.

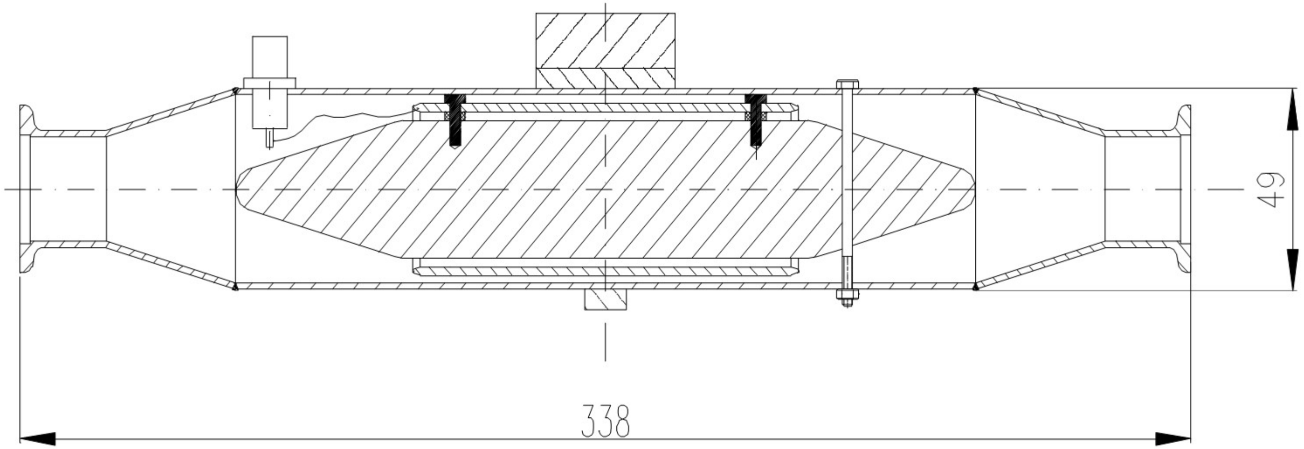


Fig.3. The cross section of the Void Fraction Meter. Dimensions are in mm. On the top centre part is shown the location of the cold oscillator

$$\varepsilon = \varepsilon_g \varphi + \varepsilon_l (1 - \varphi)$$

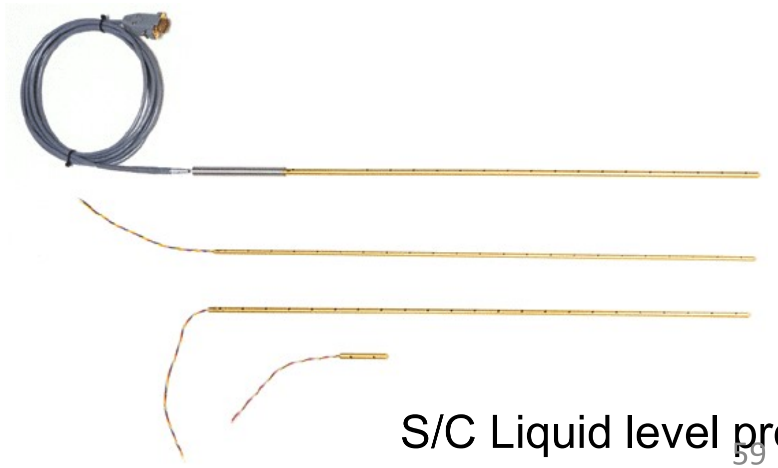
$$\varphi = \frac{A_g}{A_g + A_l}$$

For He $\varepsilon_{gas}/\varepsilon_{liq} \approx 4\%$

R. Pengo et al. (CERN/AT 2004-72014)

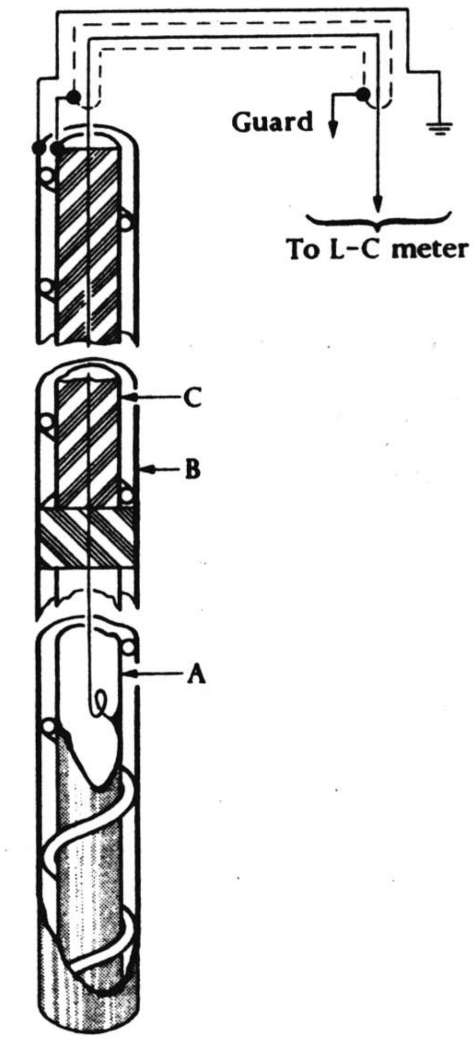
Level Measurements

- Measuring the level of cryogenic baths is important to proper operations
- Options include:
  - Differential pressure techniques
  - Capacitance gauges (LN_2, LOX, LH_2)
  - Superconducting level probes (LHe)
  - Acoustic “Dip stick” method
  - Discrete level meter (temperature sensors array)



S/C Liquid level probes
American Magnetics Inc

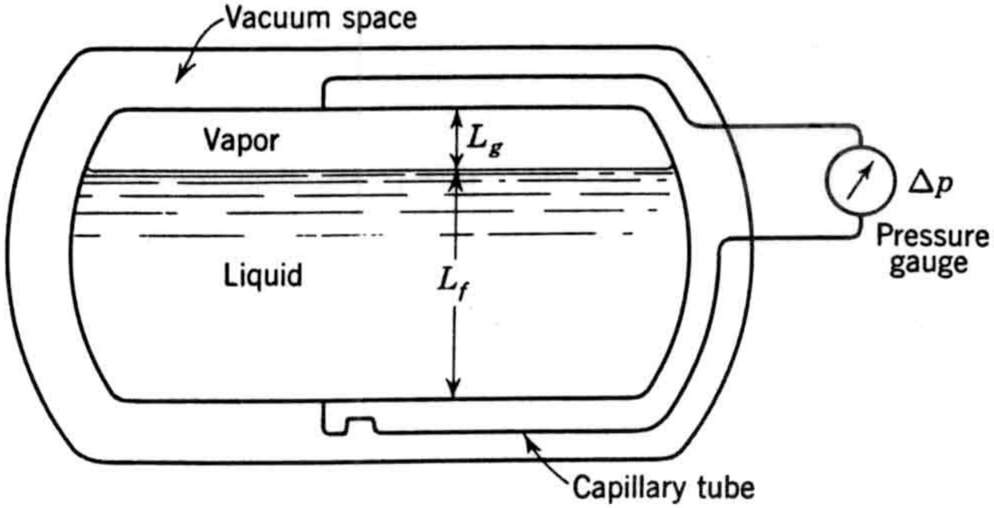
Capacitive Level Gauges



- Most are custom, some are available as a prototype commercial units, particularly for high dielectric constant fluids (e.g. LN_2)
- <u>Measurement Methods:</u>
  - AC Bridge
  - High frequency oscillator
  - Time constant method
  - Phase-lock loop technique
- *In-situ* calibration necessary
- Sensitivity is independent of the length of the probe or the liquid level. The gauge can be made more sensitive by making the annular space between the capacitance elements (and, therefore, the diameter ratio) as small as possible without causing arcing across the capacitor surfaces.

$$L_f = C \ln(D_o/D_i)/[2\pi(\varepsilon_f-\varepsilon_g)\varepsilon_0] - \varepsilon_g L/(\varepsilon_f-\varepsilon_g)$$

Differential pressure (head) gauge



Requirements :

- No liquid in vertical leg of lower capillary tube

□ ⊗p/⊗L = ⊗ρ g

= 1.06 (Pa/mm)$_{helium}$

- Heat load may be large to keep vapor line dry

$$\Delta p = \rho_f L_f g/g_c + \rho_g L_g g/g_c$$

$$\Delta p = (\rho_f - \rho_g) L_f g/g_c + \rho_g L g/g_c$$

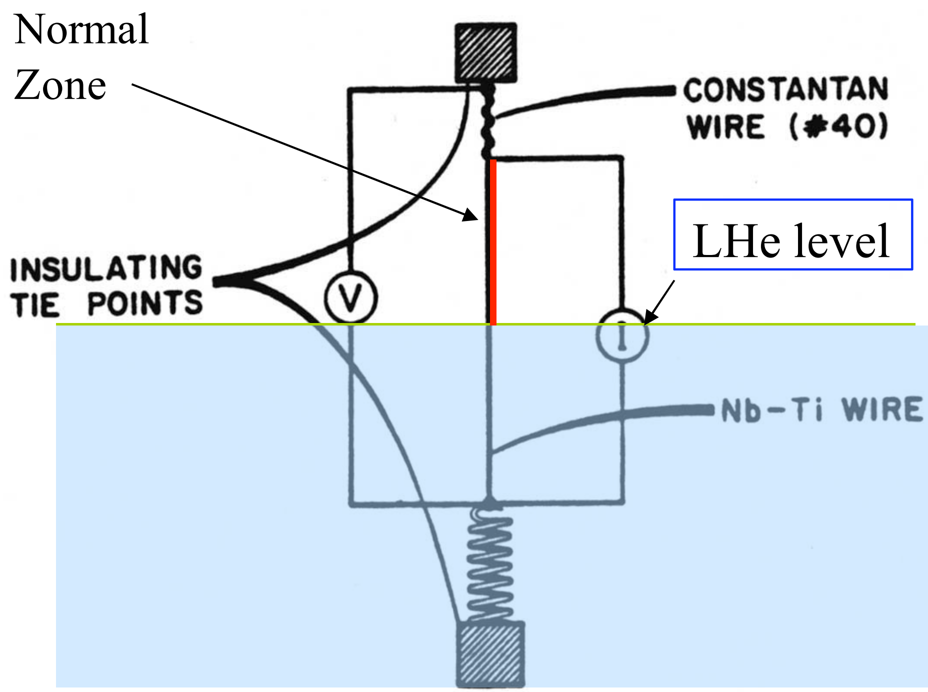
$$\Delta p \doteq \rho_f L_f g/g_c$$

(approximation not for He and H2)

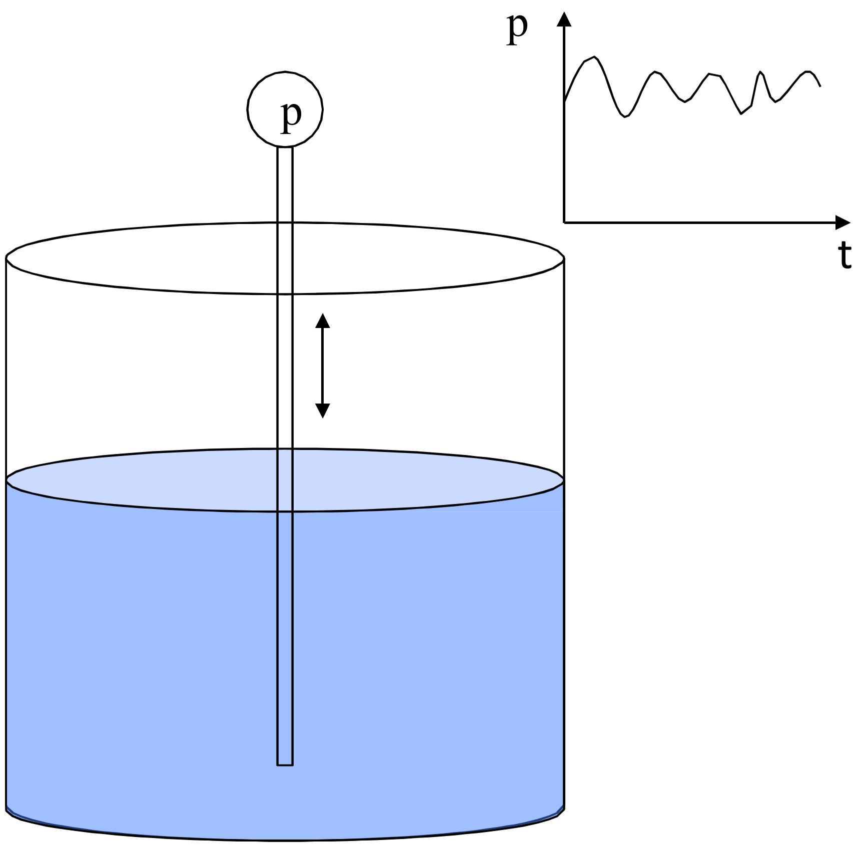
	He	H_2	Ne	N_2	O_2	Ar
ρ_l	125	70.8	1240	807	1141	1394
ρ_g	16.7	1.33	9.4	4.6	4.47	5.77

Superconducting wire level meters

- Now a commercial product
- Heater drives the normal zone of SC wire to the liquid interface, where it stops due to improved heat transfer
- Units are most often calibrated in LHe at 4.2 K
- Variable performance in He II due to improved heat transfer
- Some SC level meters based on HTS materials have been developed for LN_2



"Dip Stick" level measurement



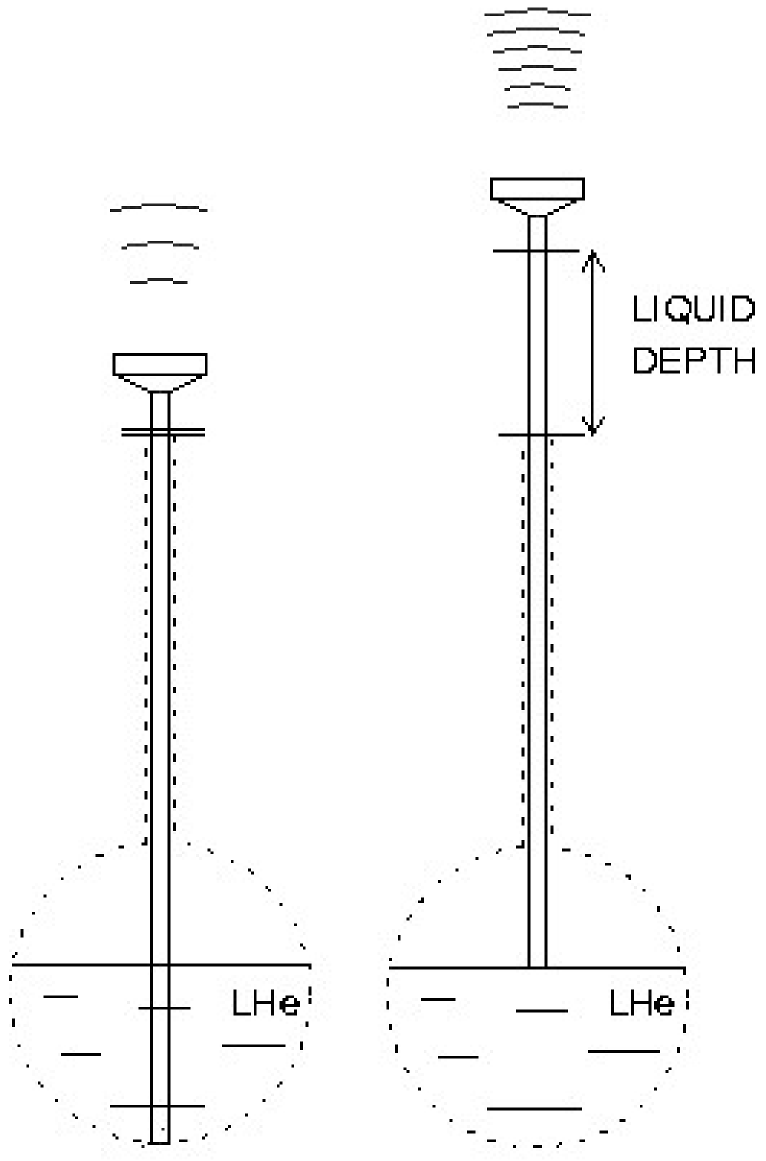


Figure 3

Acoustic oscillation changes frequency & amplitude when capillary leaves liquid

Commercial Instrumentation

- Cryogenic LTD
- Oxford Instruments
- Lakeshore Cryogenics
- Scientific Instruments
- American Magnetics
- ….
- Possible Cold Pressure Transducers
  - http://www.omega.com/
  - http://www.gp50.com/
- Cryogenic Society of America Buyer's Guide
  - http://www.cryogenicsociety.org/buyers_guide/

Safety issues in cryogenics

Chiara Vignoli

Corso Nazionale INFN “Introduzione alla criogenia”

Bologna, 28-30 ottobre 2019

Introduction

- Cryogens are stored or transported at very low temperature.
- Materials may change their properties and may become brittle at low temperature.
- They shrink at low temperatures. This may lead to material failure or may cause leaks in the system.
- Sometimes due to the excessive boil –off, sudden pressure rise may occur. This may lead to accidents.
- Therefore, while handling cryogens, important precautions have to be taken.

Fluid	^4He	H_2 para	Ne	N_2	Air	Ar	O_2	Kr	*Water*
Boiling temperature NBP in (K) at 1.013 bar	4.2	20.3	27.1	77.3	78.8	87.3	90.2	119.8	*373*
Latent heat of evaporation at NBP in kJ/kg	21	448	87.2	199.1	205.2	163.2	213.1	107.7	*2260*
Ratio volume gas (273 K) /liquid	709	798	1356	652	685	795	808	653	*…..*
Density at NBP in kg/m^3	125	71	1204	804	874	1400	1140	2413	*960*

1 liter of cryogenic fluid expands to about 700 liters (0.7 m^3) of gas when warmed to ambient temperature(at constant pressure)

Characteristics	Krypton	Argon	Nitrogen	Neon	Helium
Boiling point at 1 bar	-153.4 ℃ (120 K)	-185.8 ℃ (87.3 K)	-195.8 ℃ (77.3 K)	-246 ℃ (27.1 K)	-268.9 ℃ (4.2 K)
Density of liquid at boiling point [kg/m^3]	2413	1400	810	1210	125
Litres of gas at 20 ℃ produced by 1 litre of liquid, 1 bar	700	841	693	1454	751
Density at 20 ℃ compared to density of air	2.9	1.4	1.0	0.7	0.14
Latent heat [kJ] of evaporation for 1 litre of liquid	260	220	160	104	2.6
Ratio of enthalpy of vapour at 20 ℃ and latent heat of evaporation	0.67	0.7	1.14	3.2	72

Characteristics	Helium	Nitrogen	Argon	*(Krypton) not used*
Boiling point at 1 bar	4.2 K	77.3 K	87.3 K	*120 K*
Density of liquid at boiling point [kg/m3]	125	810	1400	*2413*
Liters of gas at 20 degr. and 1 bar produced by 1 liter of liquid	751	693	841	*700*
Density at 20 degr. compared to density of air	0.14	1.0	1.4	*2.9*
Latent heat [kJ] of evaporation for 1 liter of liquid	2.6	160	220	*260*
Ratio of enthalpy of vapor warmed to 20 degr. and latent heat of evaporation	72	1.14	0.7	*0.67*

Definitions

- A **hazard** is something that can cause **harm**, e.g. electricity, chemicals, working up a ladder, noise, a keyboard, a bully at work, stress, etc.
- A **risk** is the chance, high or low, that any hazard will actually cause an harm.

Safety

Cryogenic Hazards

- Human
- Technical plants, Infrastructures, Places

Technical and Safety Practices and Instructions

- Preventive and protective measures
- Plant design and realization
- Plant reliability
- Procedures
- Training
- Good cryogenic handling practices

Basic hazards

- **Extreme cold or low temperature**
  - Burns, Hypotermia
  - Embrittlement of material, Thermal contraction effects
- **Pressure, liquid/gas spill, explosion**
  - Expansion in a closed volume
  - Explosion with formation of dust, debris, force of blast, propelled objects
- **Oxygen Deficiency**
- **Reduced visibility**
- **Fire**
- **Magnetic field**

Extreme cold: human exposure

- Cryogenic liquids and cold vapors can cause thermal burn and frost bites and severe problems if inhalated.
- Brief contact with liquid: small effects due to gas film formation that reduce thermal contact. Cold vapour may cause worse effects.
- Prolonged contact with liquid and cold vapour severely damage delicate tissues (eyes, skin, ears, etc). The same if hair or dresses are soaked with fluid.
- Liquid and vapour driven by pressure can instantly freeze the skin.
- Unprotected skin can stick to cold metal surfaces. Even non-metallic materials are very dangerous to touch at cryogenic temperatures.
- Inhaling of extremely cold air or vapors can damage lungs.
- Hazard of hypotermia to prolonged exposure to cold ambient that may lead to generalized organ dysfunction and central nervous system, cardiac, and respiratory depression occur.

Cryogenic Hazardous Events
Cold Temperature related Physiological Issues

Contact burns

Similar to hot burns

Frost bite → freezing of skin and body party

Permanent damage and discoloration

Exposure time on the order of **seconds**, not minutes!

Inhalation

Inhalation of cold vapour can cause damage to the lungs and may trigger and asthma attack

Due to the low viscosity and surface tension of cryogens, it will flow through clothing much faster than water.

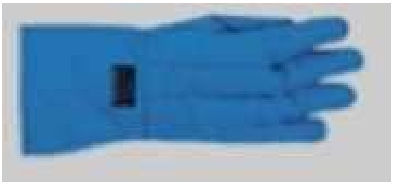
Jewelry, watches, rings etc. should not be worn, as metals can get frozen onto the skin.

General Safety Practices
PPE

Wear Personal Protective Equipment (PPE)

- Safety glasses (or face shield)
- Cryogenic gloves → loosely fitting
- Cryogenic apron
- Full length pants that extend over shoe tops
- Closed-toed shoes that are impermeable to liquids, such as leather, or covered with liquid proof shoe covers

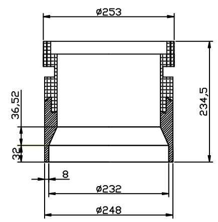
PPE must be clean, free of oil and grease



Extreme Cold: materials

Properties of materials change at low temperature

- Some materials become brittle at low temperature:
    - F.i. piping support structures or tank feet break when subjected to small loads.
    - Rubbers, plastics, …
- Materials undergo a thermal contraction.
- Thermal gradients are also important.
- Differential contractions of materials could arise problems too.
    - Localized stresses, deformations, ruptures, leaks, …
    - With big set-up this could be an issue
        - ICARUS
            - Aluminum cold vessel ≈ 20 m long -> ≈ 8 cm reduction
            - Stainless steel wire chamber frame ≈ 18 m long -> ≈ 5.8 cm reduction
            - Wire lenght ≈ 3.7 m/9 m -> ≈ 1 cm/1.8 cm reduction
            - NO anchoring fixed points
            - Bimetallic joint transition from Al to SS for CF flanges on the cold vessel top



Cryogenic Hazardous Events

Cold Temperature related Technical Issues

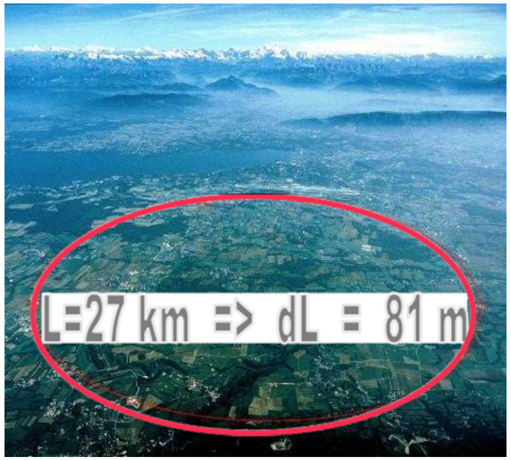
Embrittlement

- Some materials become brittle at low temperature and rupture when subjected to loads

- Protect surrounding equipment/structures from crogens discharge.

Thermal contraction (293 K to 80 K)

- Stainless steel: 3 mm/m
- Aluminium: 4 mm/m
- Polymers: 10 mm/m



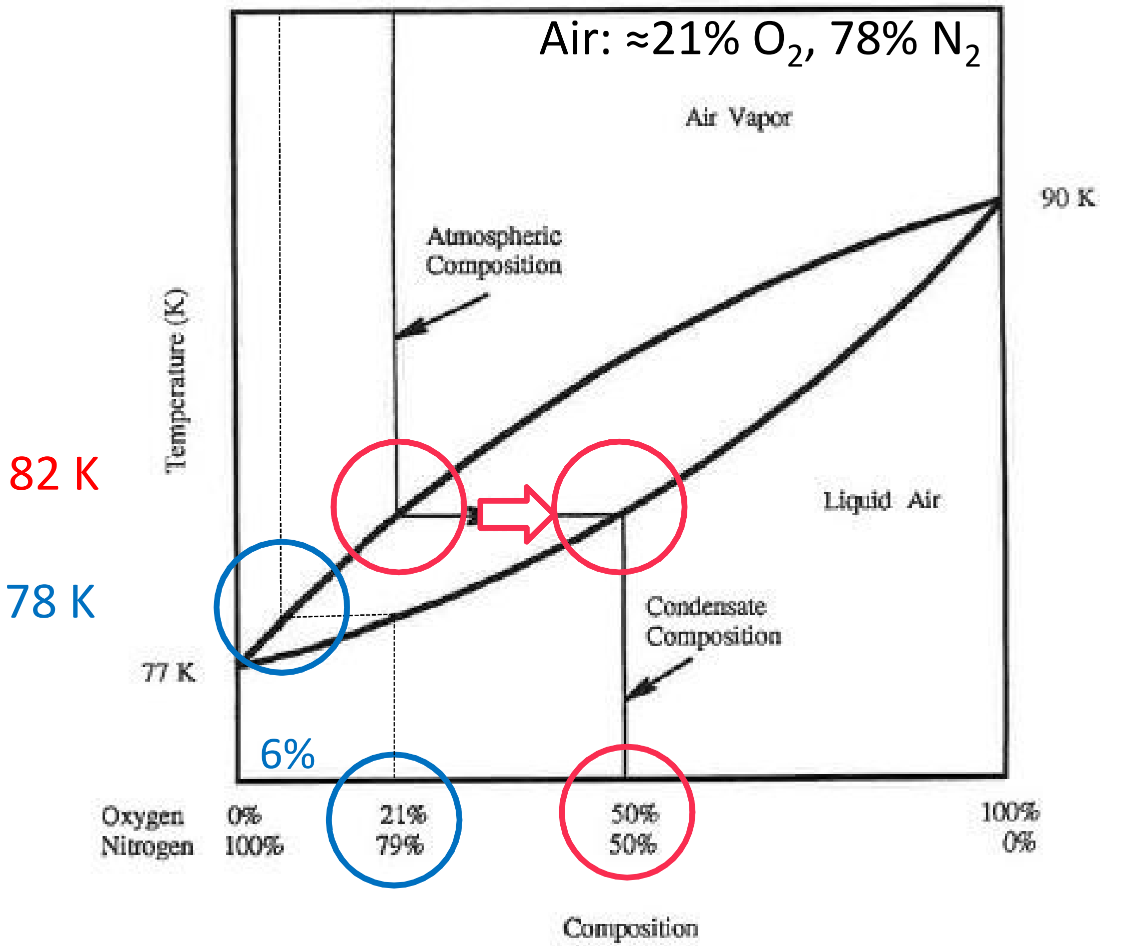
Thermal gradients

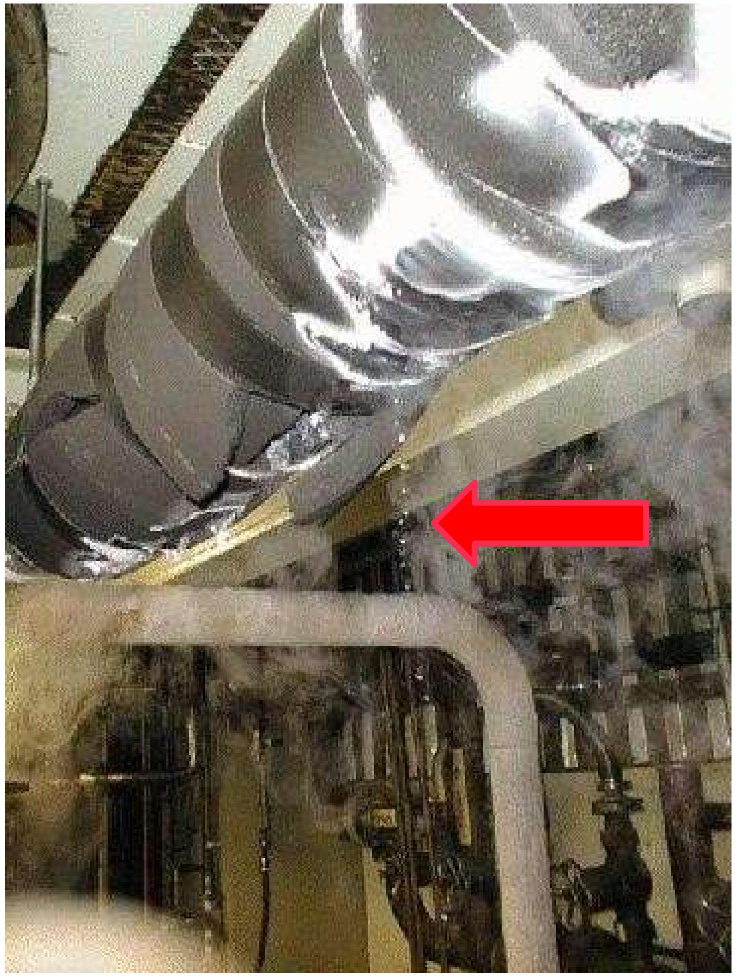
Extreme cold: water condensation hazard

- Condensation of water on power cabinets, switches, electrical plugs, etc.
- Ice formation on safety valves or instrumentation can cause blocks.
- Ice formation on open necks of dewars and cryostats.
- Material enbrittlement for ice formation (for instance open cell insulation)

Extreme cold:Oxygen condensation hazard

Condensation of atmospheric gases on cold surfaces – Oxygen enrichment





Poor insulated pipe

Cryogenic Hazardous Events

Cold Temperature

Condensation of air

- Innappropriate insulation or discharge of cryogens can lead to oxygen enrichment
- Mainly observed at tranfer lines and during filling operations
- (liquid air → **50% O_2** instead of 21% in atmospheric air)

Combustion / Fire

- Liquid oxygen can cause spontaneous combustion.
- Adheres to clothing and presents an acute fire hazard.
- Normal combustion could become explosve in enrixhed air.
- Almost all substances that are not completely oxidized can burn in oxygen, so any source of heat could rise any part of the system to its ignition temperature.

Cryogenic Hazardous Events

Build-up: Technical

Build-up of pressure

- Pressure can be released when thermal loads are beyond normal operation due to:
  - Fire
  - Loss of insulation vacuum
  - SC magnet resistive transition (quench)
  - Return line blocked

- Release of cryo-pumped gases during warm-up (air leaks)

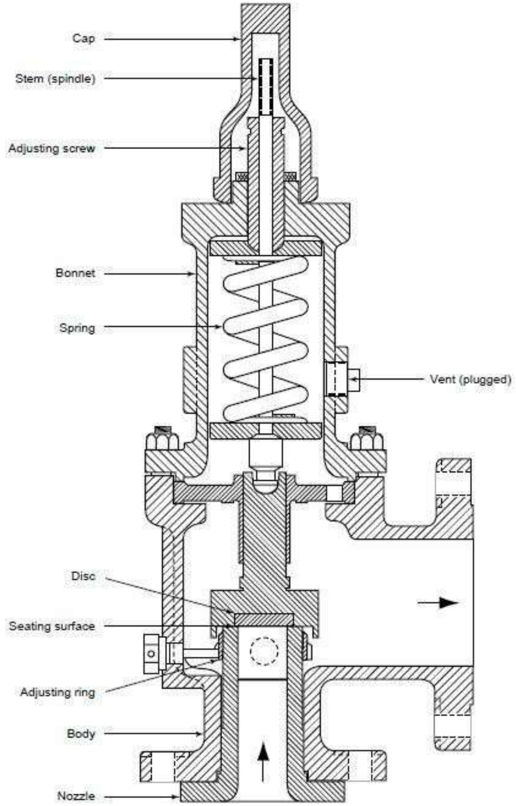
- <u>Use pressure-relief devices to protect both the fluid volume and vacuum vessel against overpressure is mandatory.</u>

Overpressure hazard

- Unusual or accidental conditions such as an external fire, or a break in the thermal insulation vacuum, stop of cooling, magnet quenching, may cause a very rapid increase of vaporization -> pressure rise.
- Without adequate venting or pressure-relief devices on the containers, pressures can build up and may cause an explosion, with all the consequent effects (dust, debris, force of blast, propelled objects, …).
- Human error can be source of overpressurization.
- Increasure of pressure could be due to different working conditions (keep in mind variation of external pressure f.i. height over the see level),
- It is necessary to protect any cryogenic plant and subsystem (f.i. a transfer line) with pressure relief valves.
- Protective devisec: pressure relief valves, bursting disks and magnetic disks.
- The protective decices must be properly dimensioned and installed and free from obstruction. Usually doubling of protective devices is highly recommended. Mounting them on a three-way valve allow maintenance of one per time.
- Relief valves shall be mounted vertically (preferably with the exhaust side down) and with sufficient stand-off distance to prevent valve obstruction by freezing of the water vapour present in the air.

Pressure relief valves

Image from Rockwood Swendeman brochure



- Conventional safety relief valve: spring-loaded. Pressure operates against a spring. Set pressure may be adjusted via spring compression.
- Even though valve at room temperature, will cool upon relieving, so need cold-tolerant material and design

Rupture disks

The "granddaddy" of all metal disks is the solid design shown in Figure 11. This disk design has been around for over sixty years and has maintained a position of leadership because it is available in a greater range of sizes and pressure ratings than are disks in other designs.

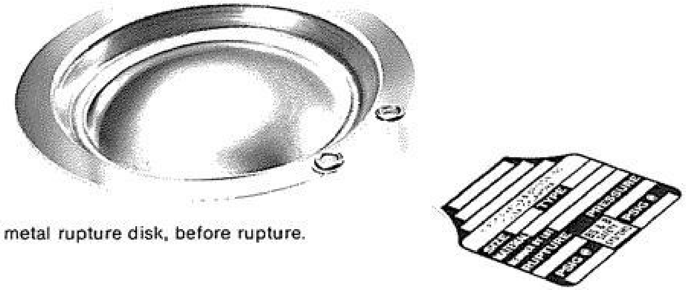


Figure 11: Solid metal rupture disk, before rupture.

A solid metal disk should retain its initial contour during exposure to the normal system pressure. An overpressure buildup to the rating of the disk will cause a thinning out of the metal. Failure will then take place at the center of the crown. When the flowing media is a gas, the opening pattern will be as shown in Figure 12.

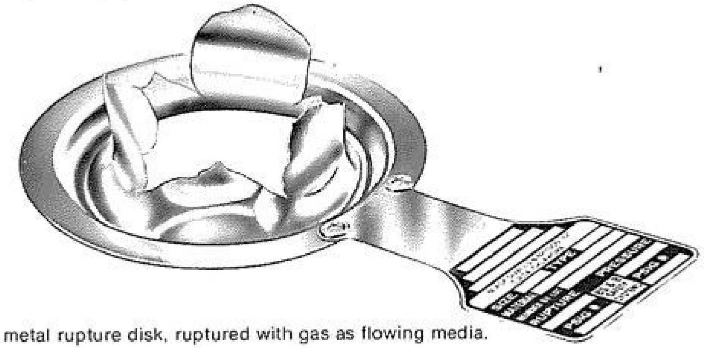


Figure 12: Solid metal rupture disk, ruptured with gas as flowing media.

- Various types, some pre-etched or with knife edge, or failure in collapse (pressure on the dome) and other designs and materials
  - Difficult to set a precise opening pressure
- A last resort device since they do not close
  - You don't want these opening in normal operations
  - Switching valves available for dual disks such that one can be replaced while the other holds pressure and provides protection
- Inexpensively provide very large capacity, so typical for the worst-case loss of vacuum
  - Operational reclosing relief valves set at a safely lower pressure (80% of RD or less) prevent accidental opening of the rupture disk

Magnetic disks

- Overpressure device using permanent magnets
- Fully open after triggering
- Automatic re-closure after circuit discharge
- High conductance
- Quite expensive

Velan (Adareg)

Vent line and exhaust point

- Collect all the possible points of release into a vent line to be driven outside the building, or in a storage device (balloon or gazometer for He), or, taking into account the natural or forced ventilation system performance, adequately define the exhaust position, to avoid gas pockets formation.
- The height of discharge may be selected on the basis of the cryogen used and the presence of workers. The exhaust shall be directly towards the top for helium and towards the bottom for nitrogen and argon.
- Design the line taking into account all the conditions (material, temperature, pressure drop, back pressure, discharge of other relief devices into the same manifold, …) and to avoid overpressure during the worst emergency discharge case (don’t reduce the cross section).
- In general it is a good practise to insulate the vent line to warm-up the cold vapour to room temperature before discharge in the ambient or in the air extraction system and to avoid water and air condensation and possible ice blocks.
- To avoid back flow flap valves can be used.

Cryogenic fluid spillages

They may happen in various situations:

- Emergency situation:
  - Pressure relief valve opening, leak, tank overfilling, accidental cryopump warm-up, tank explosion, …
- Transitory phases
  - for instance: open circuit liquid transfer, test of plants, commissioning, cooling down, warming/up, maintenance, …
- Normal operation
  - for instance: evaporation from a cryogenic reservoir due to heat losses
- Hazard:
  - Cold fluid and cold temperature related problems
  - Oxygen displacement
  - Oxygen deficiency
  - Dangerous (toxic, flammable, reactive) fluid spill
  - ….

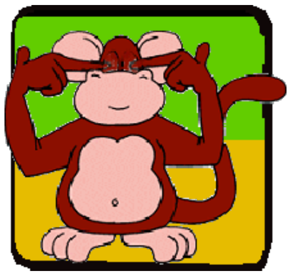
Cryogenic fluid release

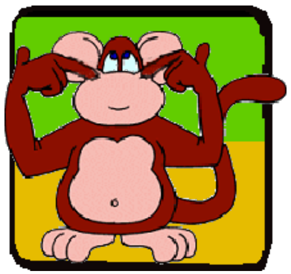
- Large increase of volume of vapour from normal boiling point to ambient temperature (300 K) at 1 bar

Fluid	(Volume of gas at 1 bar, 300 K) / (Volume of liquid at normal boiling point)
Xenon	556
Krypton	711
Methane	660
Argon	861
Oxygen	879
Nitrogen	720
Neon	1488
Hydrogen (Para)	875
Helium	783

Cryogenic Hazardous Events

Cryogens – Warning signs





Eyes

Ears

Nose

Liquid or gaseous cryogens are odourless and colourless.

Surface temperatures are not obvious

The human senses do not warn!

OFTEN ONLY secondary signs:

- Ice, water, air condensation — indicates cold surfaces
- Fog — may indicate a leak of liquid or gazeous cryogens

Characteristics	Krypton	Argon	Nitrogen	Neon	Helium
Boiling point at 1 bar	-153.4 ℃ (120 K)	-185.8 ℃ (87.3 K)	-195.8 ℃ (77.3 K)	-246 ℃ (27.1 K)	-268.9 ℃ (4.2 K)
Density of liquid at boiling point [kg/m^3]	2413	1400	810	1210	125
Litres of gas at 20 ℃ produced by 1 litre of liquid, 1 bar	700	841	693	1454	751
Density at 20 ℃ compared to density of air	2.9	1.4	1.0	0.7	0.14
Latent heat [kJ] of evaporation for 1 litre of liquid	260	220	160	104	2.6
Ratio of enthalpy of vapour at 20 ℃ and latent heat of evaporation	0.67	0.7	1.14	3.2	72

Cryogenic fluid release

Release of liquid, cold vapour, gas

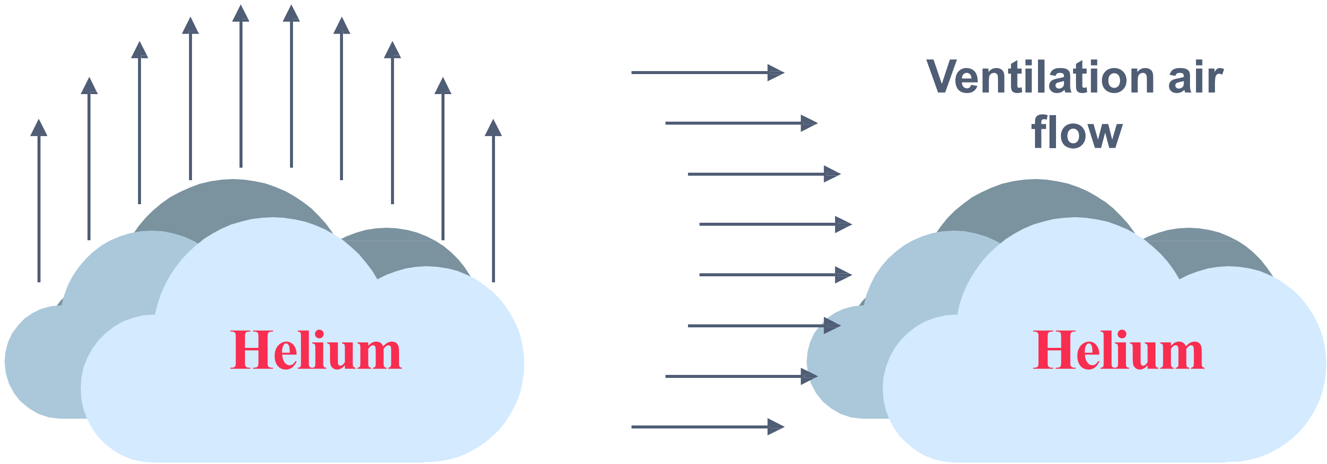
- LIQUID
  - Depending on the liquid it accumulates at ground level until it is completely evaporated (insulated catch basins may be required)
- COLD VAPOUR
  - He mixes with air and the cloud formed with air moisture rises
  - Nitrogen falls towards ground forming mist with air moisture.
  - Argon falls towards ground forming mist with air moisture.
- GAS
  - He rises
  - Nitrogen mixes with air
  - Argon accumulates at ground level

➢ **Depending on ventilation performance, <u>fluid stratification and oxygen displacement</u> may happen**

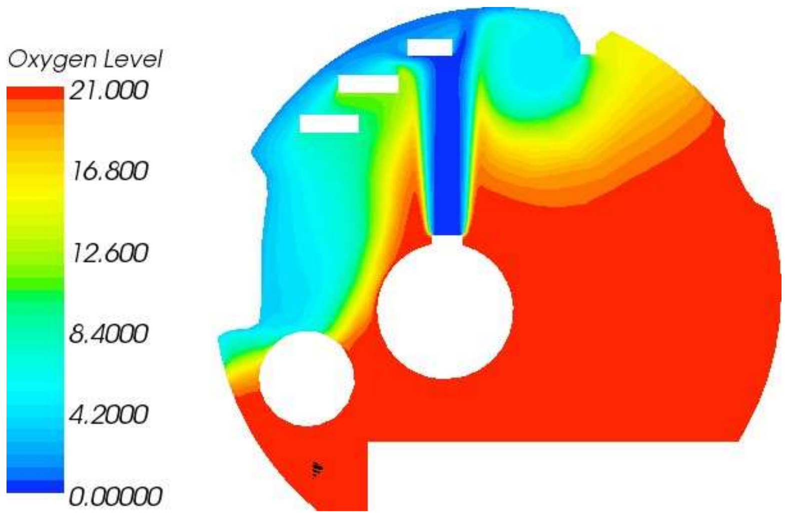
➢ The formation of mist due to the condensation of water vapour present in the air may severely **<u>reduce visibility</u>**

Cryogenic Hazardous Events

Cryogens - Discharge



Safer location at the floor

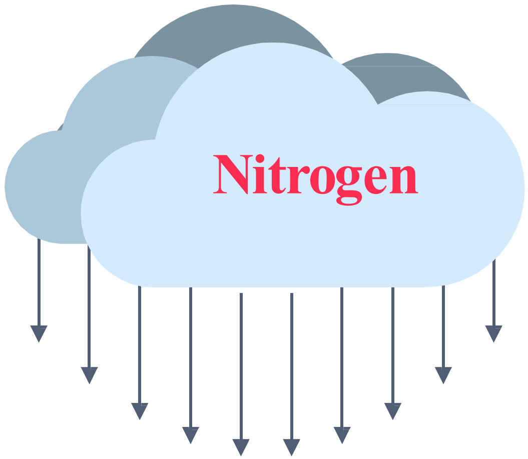


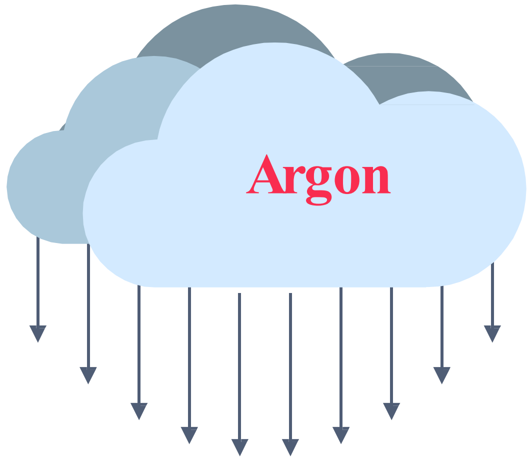
Simulated blow out in the LHC tunnel cross section

- Helium forms clouds while evaporating that move up, mixing rapidly with air
- Helium gas accumulates on the top T>40 K
- **Displacement of Oxygen!**

Cryogenic Hazardous Events

Cryogens - discharge





Safer location at the top

- Argon and nitrogen fall downwards when discharged, forming clouds
- Avoid confined spaces in pits, underground channels etc.
- **Displacement of Oxygen!**
- When warmed up nitrogen mixes with air

Oxygen Deficiency Hazards

- Gases used in cryogenic systems such as He, N_2, Ar, H_2 can displace oxygen in an area causing the area to be unsafe for human life
  - Any oxygen concentration less that 19.5 % is considered oxygen deficient
- There are several aspects to this problem
  - Large volume changes from cryogenic liquids to room temperatures gases
  - Even small amounts of liquid can be a hazard if released into a small enough volume e.g. small rooms, elevators or cars
  - Little or no warning of the hazard at sufficiently low O_2 concentrations
  - Consequences can easily be fatal
- <u>This is not just a problem in large cryogenic installations</u>
  - It can easily be a problem in small labs and university settings – in fact, complacency in smaller settings may be an added risk factor
- <u>Confined places</u>
  - Tunnel or below-ground installations, pits, vessels, …
  - Warning entering in a confined/not vented place where cryogens are present or in a vessel that was previously in contact with cryogenic fluids

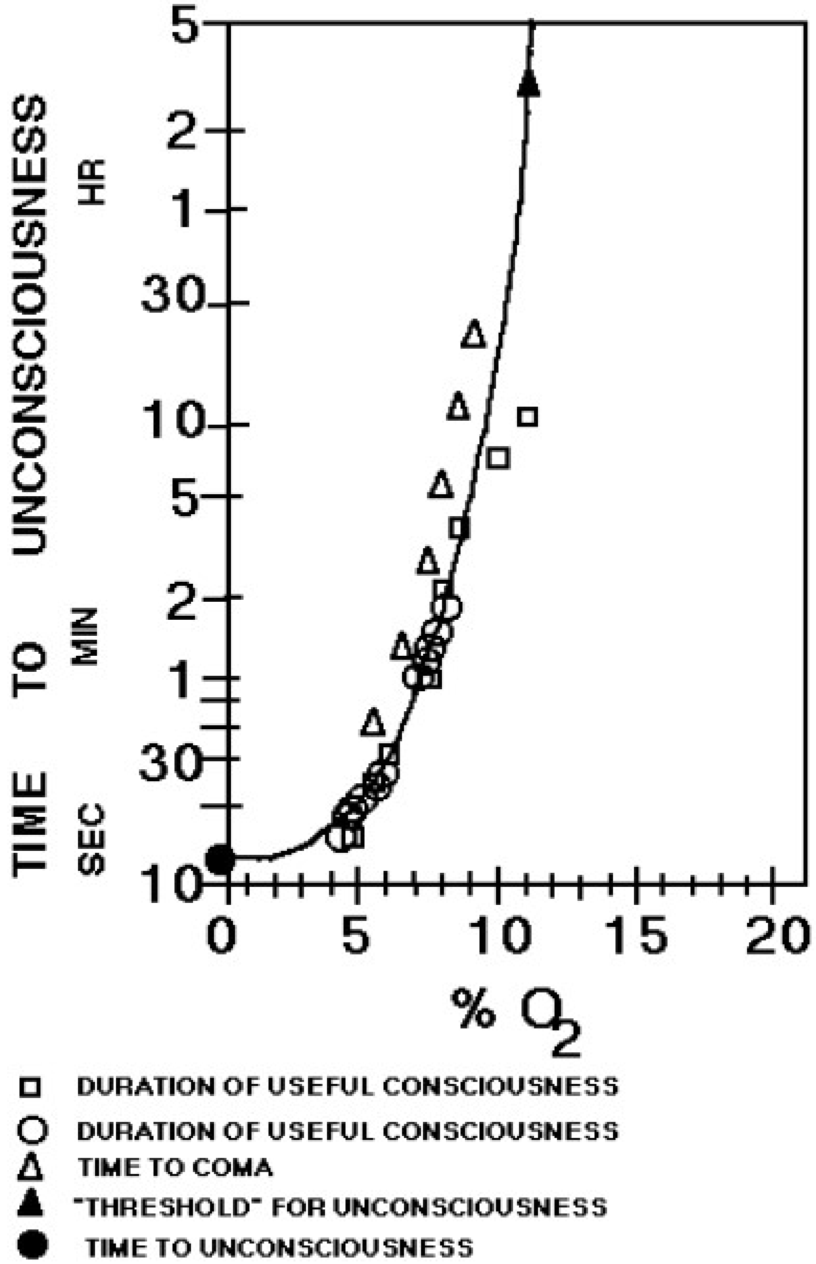
Oxygen Deficiency Hazards (ODH)

- Human body needs oxygen for survival.
- The minimum permissible oxygen content in breathing atmosphere for a normal human survival is around **19.5%**.
- If human body is deprived of this minimum percentage for more than a few minutes, it may lead to choking/unconsciousness. In certain cases, it may also lead to death.
- This condition is called as **Asphyxiation**.

OXYGEN CONCENTRATION	EFFECTS
21%	Normal level in atmosphere at see level
18%	Oxygen deficiency alarm level
17%	Accelerated heartbeat Increased breathing volume Night vision reduced
16%	Dizziness Reaction time for new tasks is doubled
15%	Poor judgment, attention, coordination Intermittent breathing, rapide fatigue, loss of muscle control
12%	Vary faulty judgment, very poor muscolar coordination, loss of consciusness, permanent brain damage
10%	Inability to move Nausea, vomiting
6%	Spasmodic breathing Convulsive movements
5%	Permanent brain damage
4%	Coma after 40 seconds, respiratory failure, death

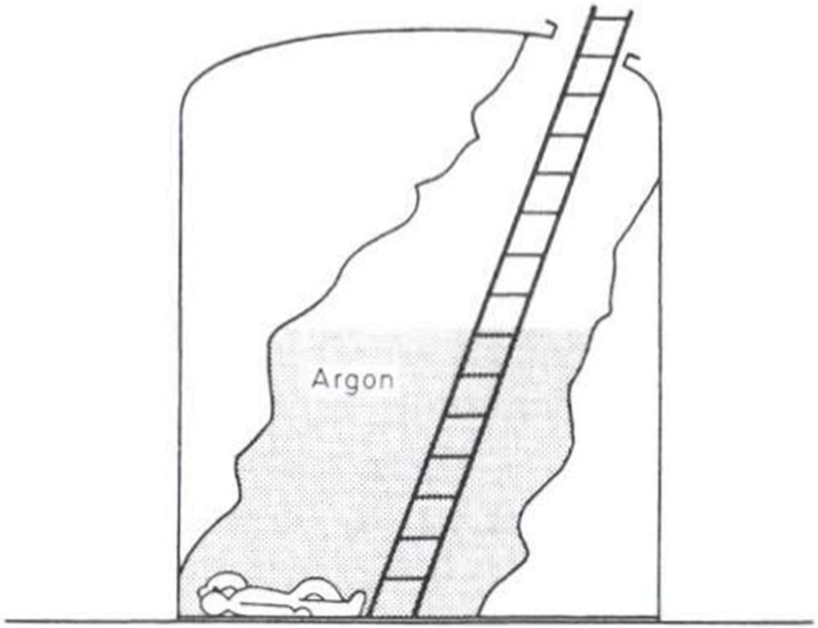
Note that in sudden or acute asphyxiation, such as inhalation of pure nitrogen or helium gas, unconsciousness is immediate. The person falls as if struck down and may die in a few minutes.

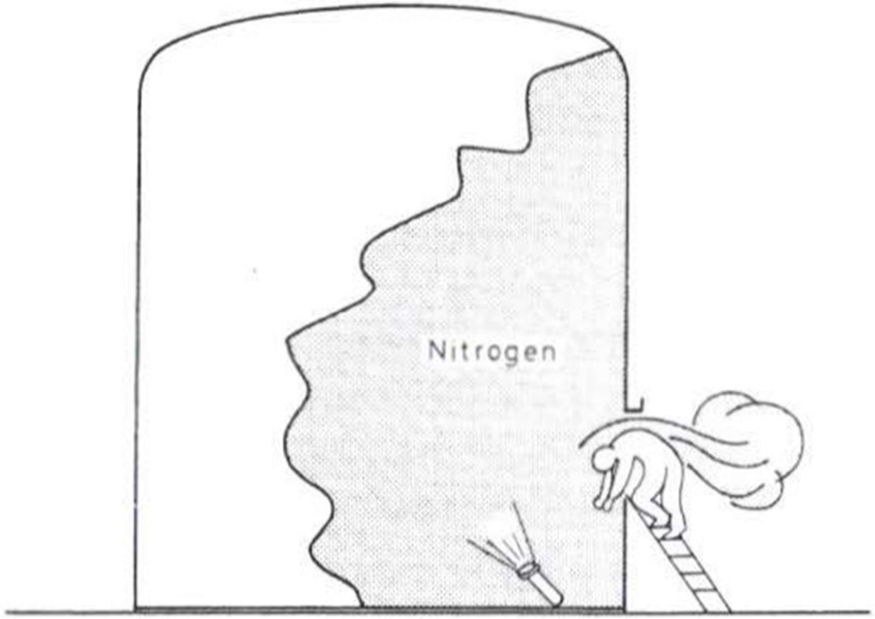
Approximate Time of Useful Consciousness for a seated subject at sea level vs %O_2

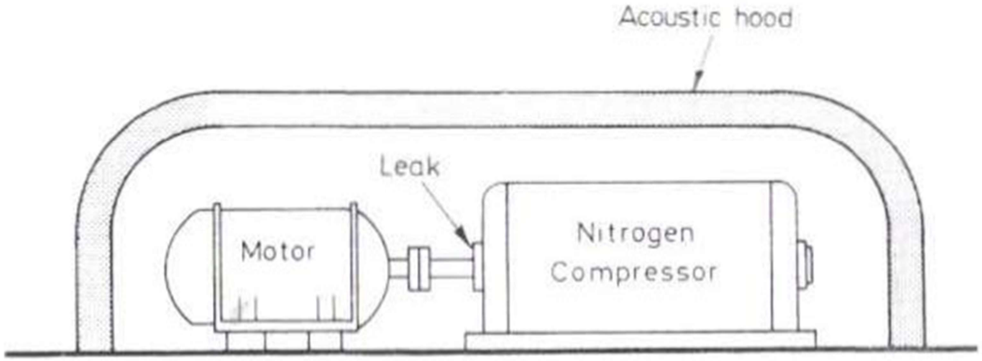


- At low enough concentrations you can be unconscious in less that a minute with NO warning
- This is one of the things that makes ODH so dangerous & frequently results in multiple fatalities

Confined places







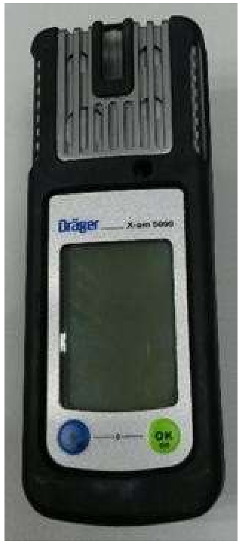
Pay attention: Not only tunnels, pits, tanks, but all the non-properly vented places

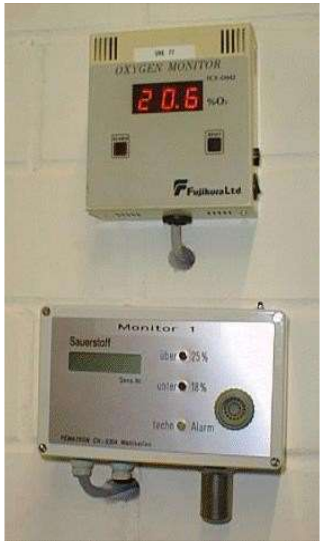

KAESER
RH100

Cryogenic Hazardous Events

Oxygen Deficiency Hazard (ODH)

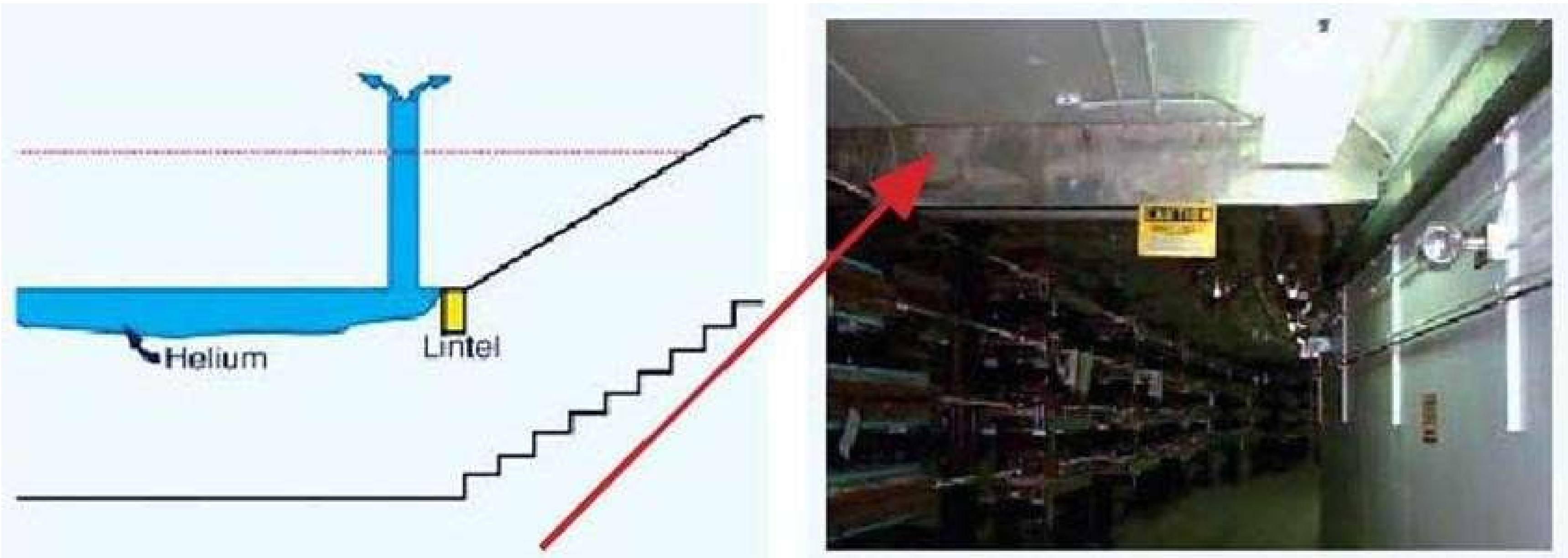
- Vent discharged cryogenic fluids to safe locations outdoors (use of relief lines)
- Equipment/system leak tightness
- Ventilation/extraction systems
- Oxygen deficiency hazard monitoring (ODH detectors), also portable devices for exposed people
- Emergency procedures & evacuation plan (keep in mind movement of clouds)
- Use self-rescuing mask (PPE for long exposure to lack of oxygen)
- Evacuation path visible even with clouds
- Evacuatinon/exit panels





ODH Mitigations

- Work Rules
  - Prohibit activities that increase risk of an accident
    - ESS and CERN: No tunnel entry during cool down and warm up of accelerator
    - Fermilab: No pit entry during LAr detector cooling and filling
  - Two Person Rule
  - Three Person Rule (unexposed observer)
- Use of lintels and vents to keep helium away from escape routes
  - For example at Jlab



Cryogenic Hazardous Events

Technical

Combustion / Fire

- Use of flammable cryogens: H_2, CH_4
  - Flammability limits H_2: 4.0-75.0 (vol %)
  - Detonability limits H_2: 18.3-59.0 (vol %)
  - Minimum ingnition energy: 0.02 mJ

  - Flammability limits CH_4: 5.3-15.0 (vol %)
  - Detonability limits CH_4: 6.3-13.5 (vol %)
  - Minimum ingnition energy: 0.29 mJ

- Presence of O_2 :
  - strong oxidizer, vigorously support combustion

Hydrogen use hazards

- The use of moderate quantities of LH_2 requires special safety considerations
- Ignition temperature in Air:
  - $T(H_2)$ = 858 K
  - $T(CH_4)$ = 813 K
  - T(kerosene) = 523 K
- Low ignition energy makes hydrogen more flammable than kerosene

- Flammability limits
  - In Air: 4 to 75%
  - In pure O2: 4 to 96%

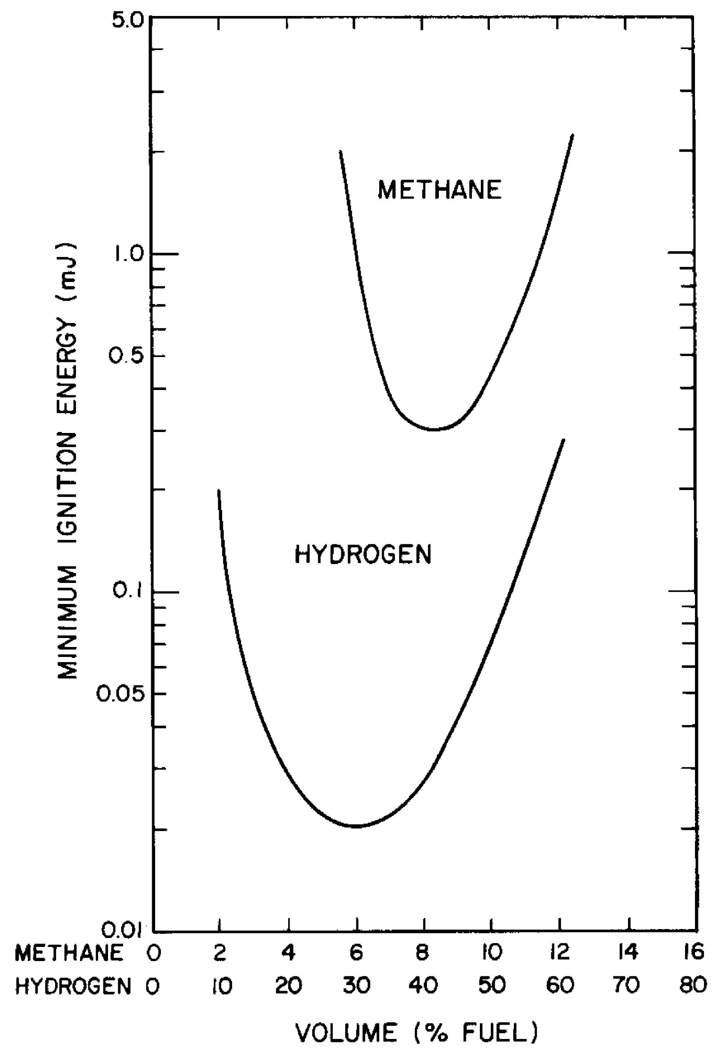


FIGURE 3. Electric spark ignition energy requirements for methane-air and hydrogen-air mixtures as a function of composition.[30]

Fire related differences: O_2 vs H_2

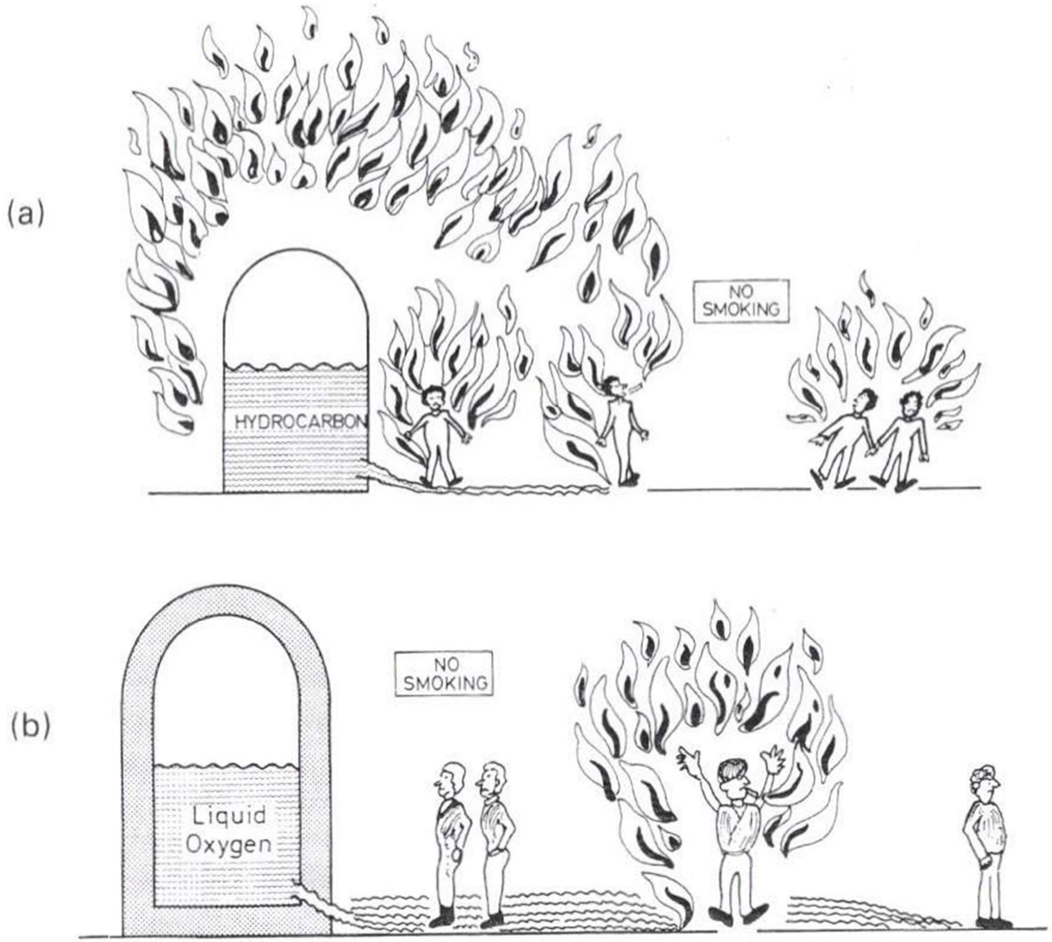
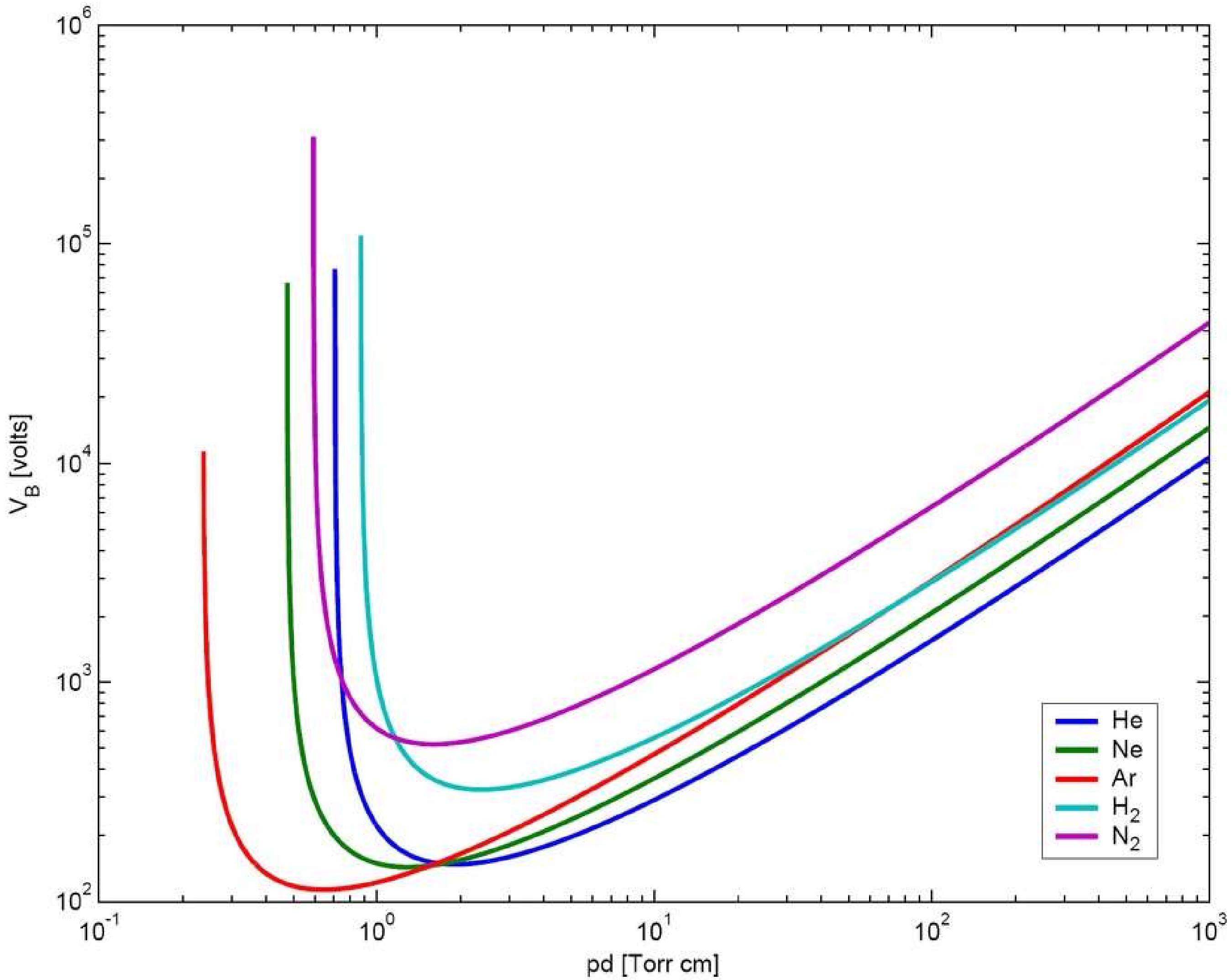
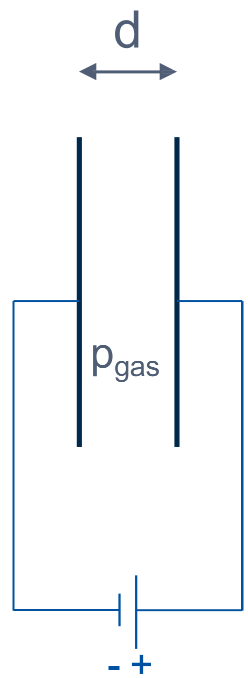


Fig. 3.5 (a) A leak from a tank containing an inflammable gas may spread a considerable distance and on ignition will engulf anybody in the area. (b) A fire in the area of a leak of oxygen will not spread through the spill.

Other Technical Hazards

Electric breakdown





General Safety Approach

- Use only containers and systems specifically designed for these products!
- Always wear personal protective equipment while handling cryogenic liquids.
- Use and transfer of cryo-liquids only in well ventilated areas => ODH!
- Materials must be compatible with the cryogen and the low temperatures.
- Keep in mind stresses caused by (differential) thermal contraction.
- Select the proper insulation
- All cryogenic systems, including piping and vacuum space must be equipped with pressure-relief devices to prevent excessive overpressures.
- Only trained and qualified personal should be allowed to handle, transport or storing liquefied gases.

General Safety Approach

PREVENTION! PREVENTION! PREVENTION!

- At the system design level it has to be as much intrinsicly safe as possible
- Equipment design, safety margins, material choices, quality assurance, standardization,…
- Reliable service supply + redundancy
  - Electrical network, diesel generator, UPS (PLC and sensors), cooling water, compressed air back-up
- Cryogenic systems process design and logic in implementation of active measures have to cope with potential failures (+redundancy) + off nominal operation mode (automized execution without operator intervention)
- Phylosophy: do not wait for accident to occur!

Accident Scenarios

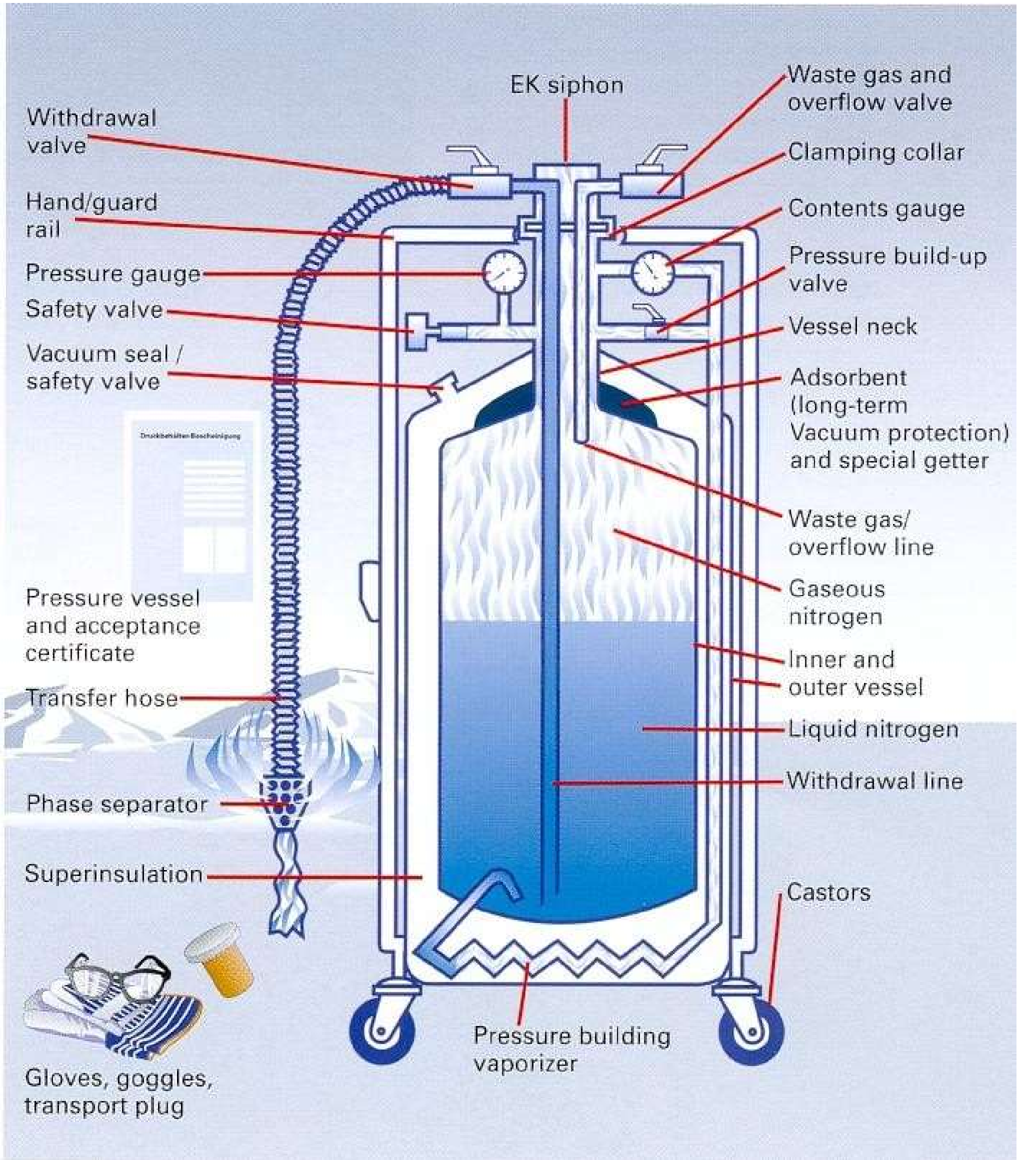
Frozen or blocked Dewar

Risks:

- Pressure rise in the Dewar
- Bursting of outer or inner shell

In case of a blocked Dewar

Evacuate the zone



Check of Equipment

Control of experimental set-up

- Check safety equipment before cooldown!
  Respect Pressure Equipment Directive (PED)
  PS>0.5 barg.

- Check vacuum insulation space
  $p<10^{-3}$ mbar at warm conditions.

- **Nitrogen**: vent line with non return valve to ambient.

- **Helium**: vent line with non return valve to recovery line!

- Equipment should be kept clean and dry.
  Before cool-down the cryostat should be purged with the gas intended to be filled in.

- Check leak tightness during purge procedure