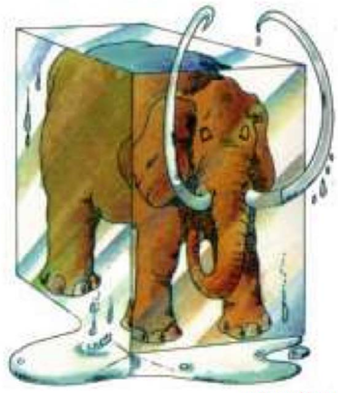
A pedestrian guide to cryogenics



credit: www.yfolire.net

credit: www.oxinst.com

Experimental Methods
in Physics
[2011-2012]

J-D Ganiere
EPFL - SB - ICMP - IPEQ
CH - 1015 Lausanne

References

Introduction to cryogenics for superconducting RF cavities
(a good introduction to refrigeration & liquefaction cycles)
by R. Bates

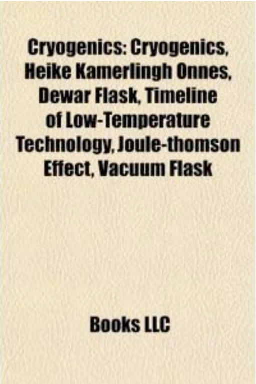
Practical Cryogenics
By N H Balshaw
Published by Oxford Instruments Superconductivity Limited

Introduction to laboratory cryogenics
by M.N. Jirmanus, Ph.D.
Janis Research Company, Inc.
(most of matter, presented here, included pictures of the cryostats, are from this reference)
This is an excellent introduction to laboratory cryogenics

An introduction to cryogenics
Ph. Lebrun
CERN

www.lakeshore.com
for the characteristics of temperature sensors

Wikipedia



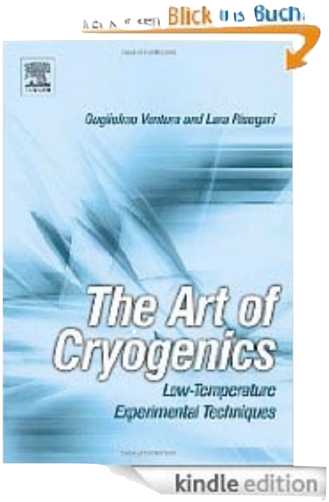
Cryogenics: Cryogenics, Heike Kamerlingh Onnes, Dewar Flask, Timeline of Low-Temperature Technology, Joule-thomson Effect, Vacuum Flask

Books LLC

Definition

Cryogenics is defined as that branch of physics which deals with the production of very low temperatures and their effect on matter.

it is also defined as the science and technology of temperatures below 120 K.



On the earth

Cryogenic Engineering is concerned with temperatures found in the range of -150°C to absolute zero –273.15°C.

The lowest natural temperature ever recorded, on the earth, was in Antarctica -89°C or 184K

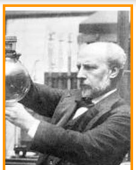
Vostok, Antarctica

	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Year
°C	-32.1	-44.3	-57.9	-64.7	-65.6	-65.2	-66.9	-67.6	-66.0	-57.1	-43.3	-32.1	-55.1
°F	-25.7	-47.6	-72.1	-84.4	-86.0	-85.3	-88.3	-89.6	-86.7	-70.7	-45.8	-25.7	-67.1

the history of the liquefaction of gases

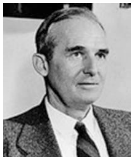
Cailletet & Pictet
1877 - liquid O2

Olszewski
1883 - Liquid N2

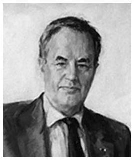


Dewar
1897 - Liquid H2

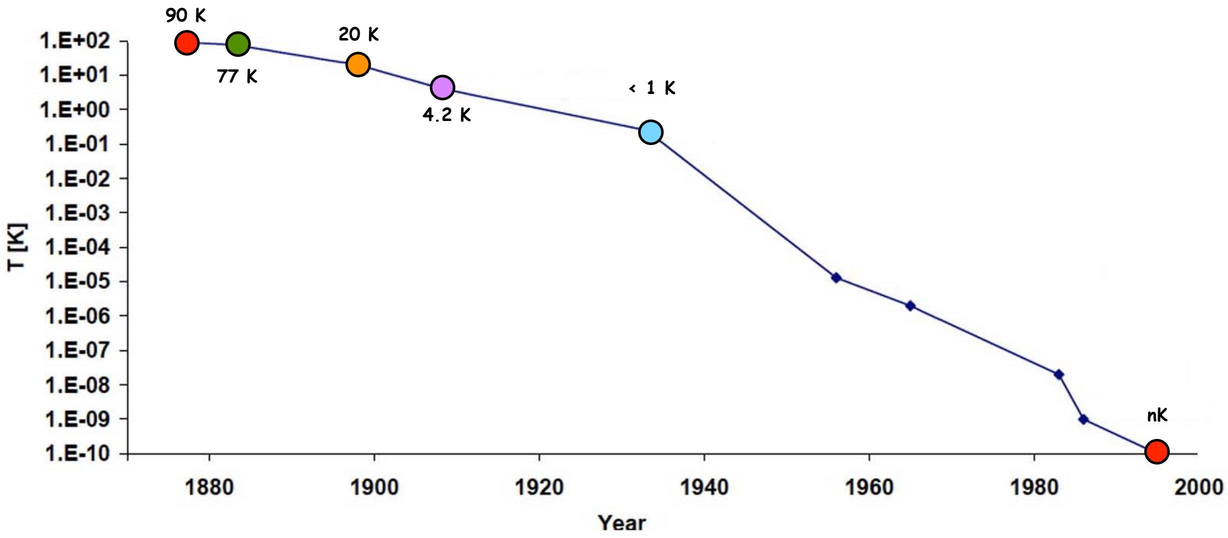
Kamerlingh Onnes
1908 - Liquid He



Giauque
1933 -Magnetic refrigeration



Lounasmaa
1980 - < 1 nK



Definition

the limit temperature of 120 K comprehensively includes the normal boiling points of the main atmospheric gases, as well as of methane which constitutes the principal component of natural gas.

Characteristic temperatures of cryogenic fluids [K]

Cryogen	Triple point	Normal boiling point	Critical point
Methane	90.7	11.6	190.5
Oxygen	54.4	90.19	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

Back to the basics

The quest for low temperatures however finds its origin in early thermodynamics.

The entropy S of the system was postulated by Boltzmann as:

$$S = k_B \cdot \ln w \qquad k_B = 1.38 \cdot 10^{-23}\ J/K$$

Adding reversibly heat δQ to the system produces a change of its entropy dS, with a proportionality factor T which is precisely temperature :

$$\delta Q = T \cdot dS$$

Thus, a low-temperature system can be defined as one to which a minute addition of heat produces a large change in entropy, i.e. a large change in its range of possible microscopic configurations.

Back to the basics

Boltzmann also found that the average thermal energy of a particle in a system in equilibrium, at temperature T, is:

$$E = k_B T$$

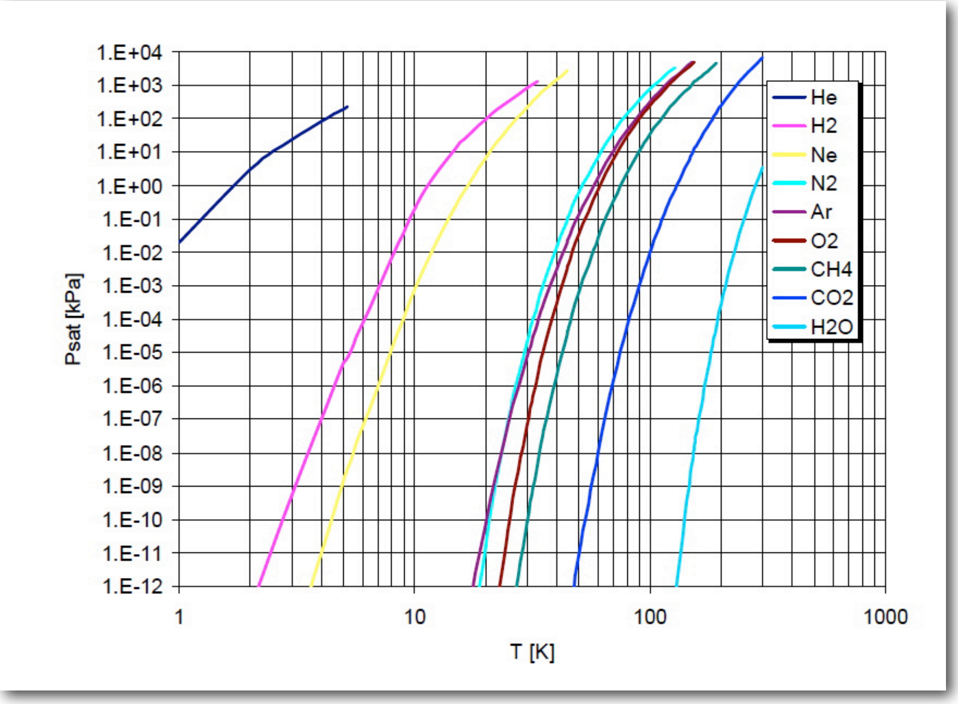
Consequently, a temperature of 1 K is equivalent to a thermal energy of 10^{-4} eV or 10^{-23} J per particle.

A temperature is therefore low for a given physical process when $k_B T$ is small compared to the characteristic energy of the process considered.

Back to the basics

Characteristic temperatures of low-energy phenomena	
Phenomenon	**Temperature [K]**
Debye temperature of metals	few 100
High-temperature superconductors	~ 100
Low-temperature superconductors	~ 10
Intrinsic transport properties of metals	< 10
Cryopumping	few
Excitons binding energy ib semiconductors	few
Cosmic microwave background	2.7
Superfluid helium 4	2.2
Bolometers for cosmic radiation	< 1
Low-density atomic Bose-Einstein condensates	~ 10^{-6}

Thermophysical properties of fluids



Vapour pressure of common gases at cryogenic temperature

Thermophysical properties of fluids

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
Liquid viscosity* [µPl]	3.3	152	278

* at normal boiling point

Properties of helium and nitrogen compared to water

Liquid boil-off

The factor of ten in latent heat of vaporization between helium and nitrogen, combined with the lower density of the former, induces a large difference in vaporization rates under the same applied heat load.

Cryogen	[mg/s]	[l/h liquid]	[l/min gas NTP]
Helium	48	1.38	16.4
Nitrogen	5	0.02	0.24

Vaporization of liquid helium and liquid nitrogen at normal boiling point under 1 W applied heat load

Liquid boil-off

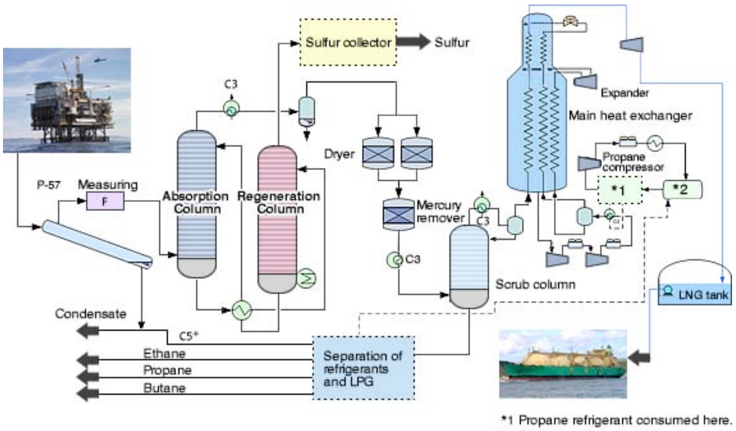
Boil-off measurements constitute a practical method for measuring the heat load of a cryostat holding a saturated cryogen bath. In steady conditions, i.e. provided the liquid level in the bath is maintained constant, the boil-off $\dot{m}_{vap}$ precisely equals the vapor flow $\dot{m}_{out}$ escaping the cryostat, which can be warmed up to room temperature and measured in a conventional gas flow-meter.

$$\dot{m}_{out} = \dot{m}_{vap}\left[1-\frac{\rho_v}{\rho_l}\right] < \dot{m}_{vap}$$

Regfigeration

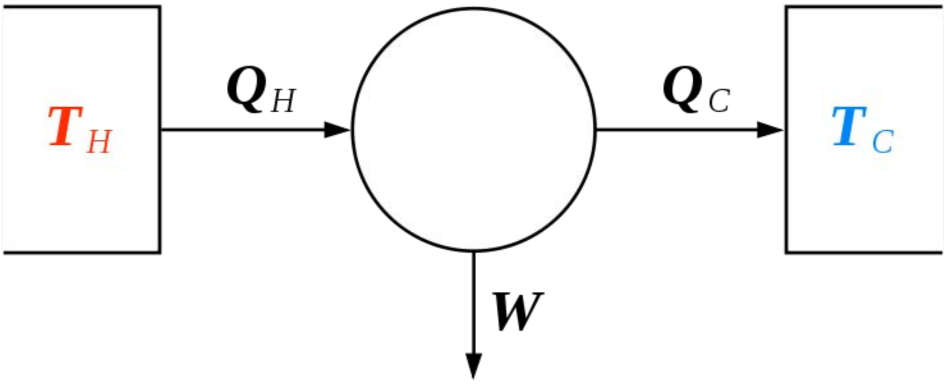
Refrigeration is not an efficient process, work is required to cool and maintain the low temperature.
The lower the temperature required the more costly refrigeration becomes.

The Ratio of work (W) done to the heat (Q) extracted is known as the Figure of Merit.



*1 Propane refrigerant consumed here.
*2 Propane refrigerant tank

Thermodynamics still applies ...



T_H

Q_H

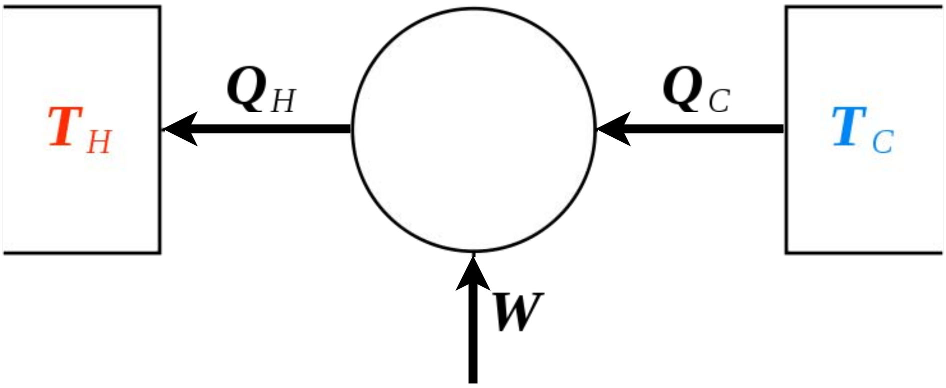
Q_C

T_C

W

$$\eta = \left| \frac{W}{Q_C} \right| = \frac{T_h}{T_c} - 1$$

Thermodynamics still applies ...



T_H

Q_H

Q_C

T_C

W

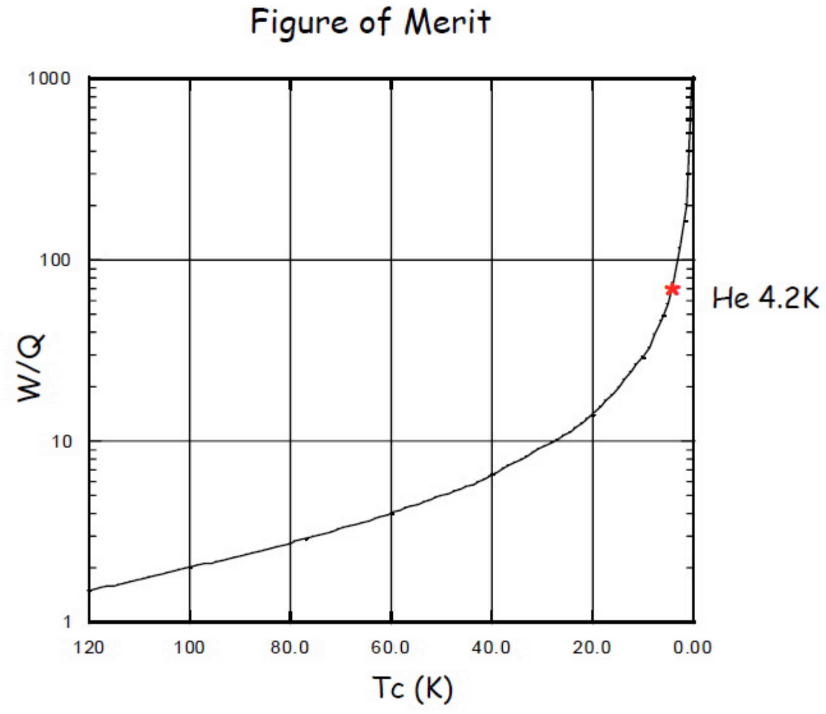
$$\eta = \left| \frac{W}{Q_C} \right| = \frac{T_h}{T_c} - 1$$

Figure of Merit

The figure of merit for various temperatures:

Tc(K)	120	77	20	4.2	1.8	1
W/Q	1.5	2.9	14	70	165	299

Figure of Merit



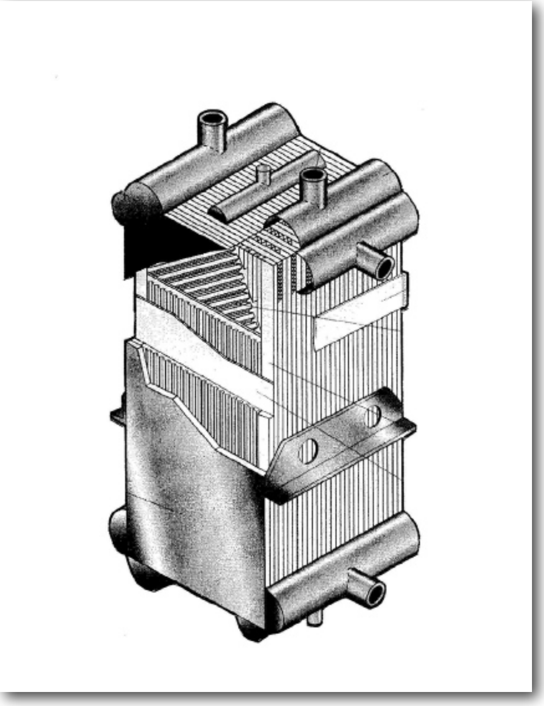
Methods to achieve cryogenics T

There are three main cooling methods that the engineer employs to achieve Cryogenic temperatures:

- Transfer of Heat.
- External work.
- Isenthalpic Expansion.

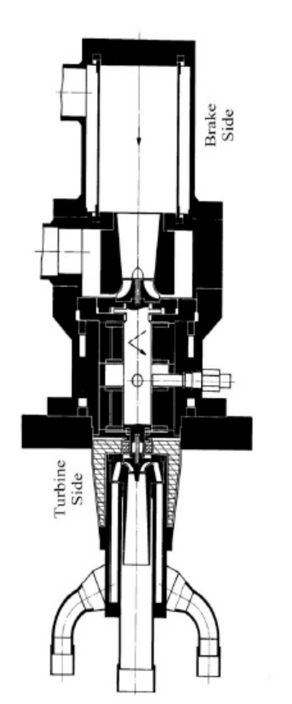
Heat Transfer

The transfer of heat to a cold gas stream using counterflow Heat Exchangers.
A matrix of flat plate & corrugated fins in a sandwich construction.



External work

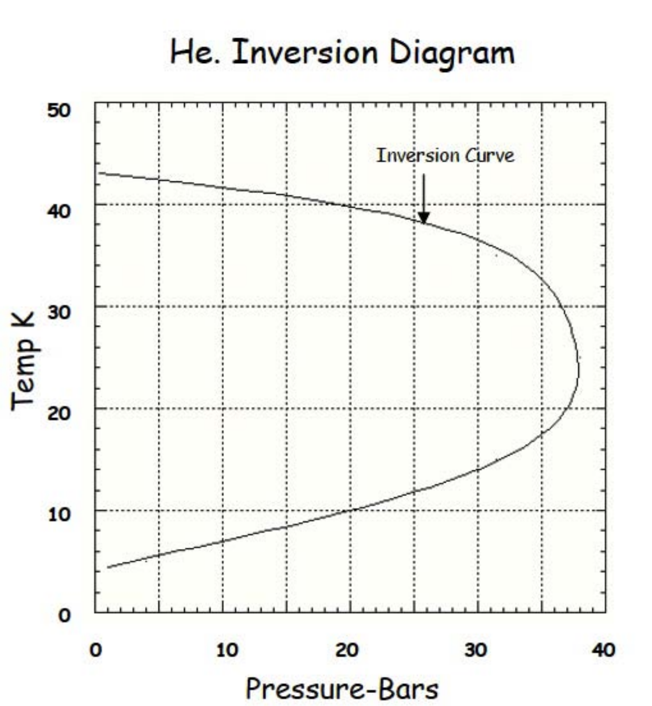
Reciprocating engine working in reverse.
More commonly a turbine expander.
Adiabatic process.



Isenthalpic Expansion

The Joules-Thomson effect.
Each gas has an inversion curve.
Starting pressure & temperature must be within the inversion curve area to achieve cooling.

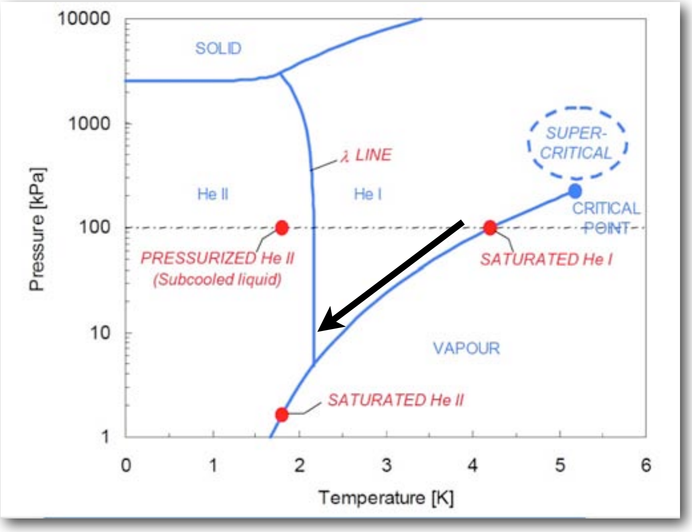
“The expansion of a previously compressed and cooled gas through a calibrated orifice valve without heat or work exchange with its surroundings”.
This process will only liquefy a gas upon expansion when it has been cooled below it’s CRITICAL TEMPERATURE.



Thermophysical properties of fluids

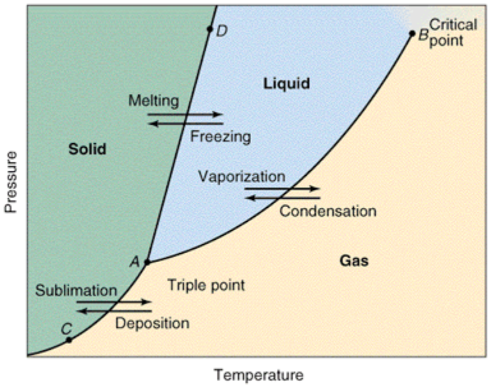
The simplest way of cooling equipment with a cryogenic fluid is to make use of its latent heat of vaporization, e.g. by immersion in a bath of boiling liquid. As a consequence, the useful temperature range of cryogenic fluids is that in which there exists latent heat of vaporization, i.e. between the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

The simplest cryostat (T ≈ 4 K !)



p ↘ ⇒ T ↘

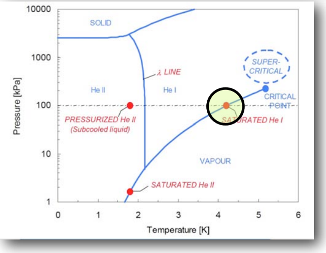
Phase diagram

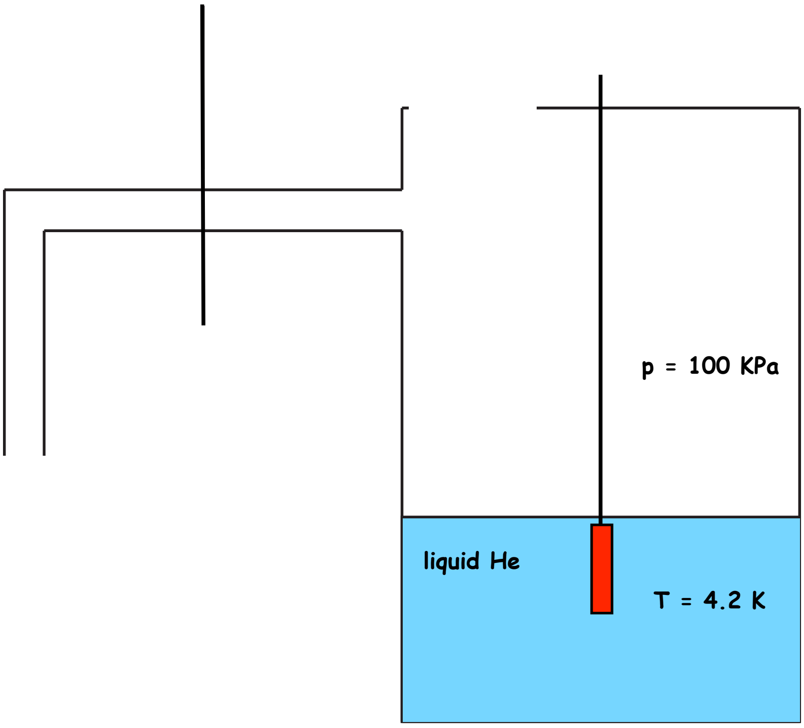


Important **Points** on a Phase Diagram:

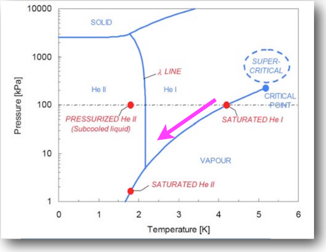
- The point where the dotted line representing 1 atm crosses the liquid-vapor line is the **normal boiling point** of the substance.
- The liquid-vapor line ends at the **critical point** (B), which is the critical temperature and critical pressure of the substance. Beyond the critical point, the liquid and gas phases become indistinguishable from one another.
- The **melting point** of a substance is identical to its **freezing point**. The two differ only in the direction from which the phase change is approached. The melting point at 1 atm is the **normal melting point**.
- Where the three lines intersect (A), is known as the **triple point**. All three phases are in equilibrium at this temperature and pressure.

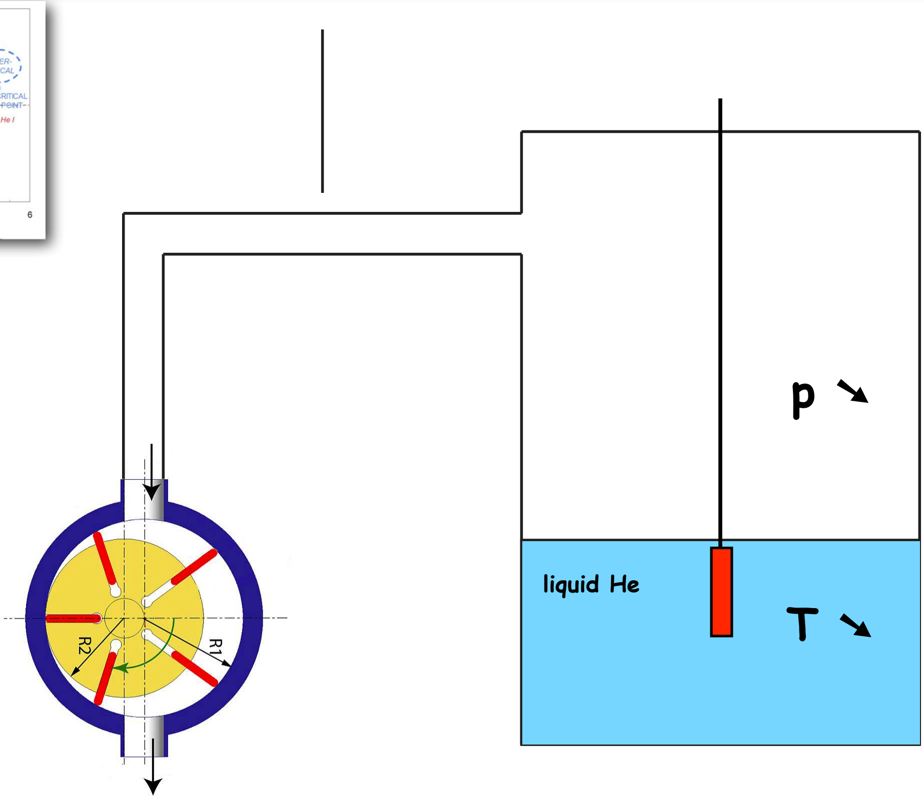
The simplest cryostat (T ≈ 4 K !)





The simplest cryostat (T ≈ 4 K !)





Does this cryostat work ?

The answer is NO !!!

Problems ... the thermal losses

The origins of the losses

- via conduction (walls of the cryostat, wires)
- via convection
- via radiation

Heat transfer and thermal design

solid conduction

Heat conduction in solids is represented by Fourier’s law, expressing proportionality of heat flux with thermal gradient.

$$Q = k(T)\,A\frac{dT}{dx}$$

This equation also defines the thermal conductivity k(T) of the material, which varies with temperature. Conduction along a solid rod of length L, cross section A spanning a temperature range [T1, T2], e.g. the support strut of a cryogenic vessel, is then given by the integral form

$$Q = \frac{A}{L}\int_{T_1}^{T_2} k(T)\,dT$$

Thermal conductivity integrals of standard materials are tabulated in the literature.

Heat transfer and thermal design

From vanishingly low temperature up to	20 K	80 K	290 K
OFHC copper	11000	60600	152000
DHP copper	395	5890	46100
Aluminum 1100	2740	23300	72100
2024 aluminum alloy	160	2420	22900
AISI 304 stainless steel	16.3	349	3060
G-10 glass-epoxy composite	2	18	153

Thermal conductivity integrals of selected materials [W/m]

Radiation

Blackbody radiation strongly and only depends on the temperature of the emitting body, with the maximum of the power spectrum given by Wien's law:

λ max T = 2898 [μm K]

and the total power radiated given by Stefan-Boltzmann's law

$Q = \sigma\ A\ T^4$

with Stefan-Boltzmann's constant $\sigma \simeq$ 5.67 10-8 W m-2 K-4. The dependence of the radiative heat flux on the fourth power of temperature makes a strong plea for radiation shielding of low-temperature vessels with one or several shields cooled by liquid nitrogen or cold helium vapor.

Conversely, it makes it very difficult to cool equipment down to low temperature by radiation only

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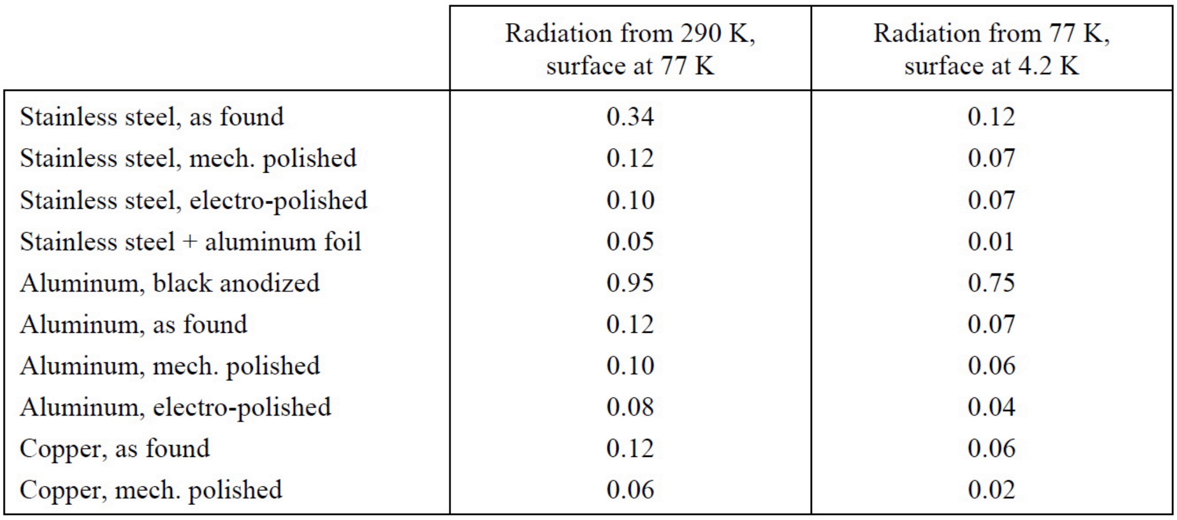
Radiation

Technical radiating surfaces are usually described as "gray" bodies, characterized by an emissivity $\varepsilon < 1$

$Q = \varepsilon\ \sigma\ A\ T^4$

Emissivity of some technical materials at low temperature



	Radiation from 290 K, surface at 77 K	Radiation from 77 K, surface at 4.2 K
Stainless steel, as found	0.34	0.12
Stainless steel, mech. polished	0.12	0.07
Stainless steel, electro-polished	0.10	0.07
Stainless steel + aluminum foil	0.05	0.01
Aluminum, black anodized	0.95	0.75
Aluminum, as found	0.12	0.07
Aluminum, mech. polished	0.10	0.06
Aluminum, electro-polished	0.08	0.04
Copper, as found	0.12	0.06
Copper, mech. polished	0.06	0.02

Radiation

The net heat flux between two “gray” surfaces at temperature T_1 and T_2 is similarly given by:

$$Q = E\ \sigma\ A\ (T_1^4 - T_2^4)$$

with the emissivity factor E being a function of the emissivities ε_1 and ε_2 of the surfaces, of the geometrical configuration and of the type of reflection (specular or diffuse) between the surfaces. Its precise determination can be quite tedious, apart from the few simple geometrical cases of flat plates, nested cylinders and nested spheres.

Radiation

If an uncooled shield with the same emissivity factor E is inserted between the two surfaces, it will “float” at temperature T_s given by the energy balance equation:

$$Qs = E\ \sigma\ A\ (T_1^4 - T_S^4) = E\ \sigma\ A\ (T_S^4 - T_2^4)$$

Solving for Ts yields the value of $Q_s = Q / 2$: the heat flux is halved in presence of the floating shield.
More generally, if n floating shields of equal emissivity factor are inserted between the two surfaces, the radiative heat flux is divided by n + 1.

Convection and gaz conduction

The mean free path of gas molecules, as predicted by kinetic theory, scales with the square root of temperature and inversely with pressure and the square root of molar mass. It therefore becomes large at low pressure, high temperature and for light gas species.
When ℓ << d corresponding to higher residual pressure, the probability of interaction of a given molecule with others before it travels distance d is high (viscous regime), and heat diffuses as in any continuous medium:

$$Q = k(T)\, A\, \frac{dT}{dx}$$

Note that the thermal conductivity k(T) of the gas is independent of pressure.

Thermal expansion

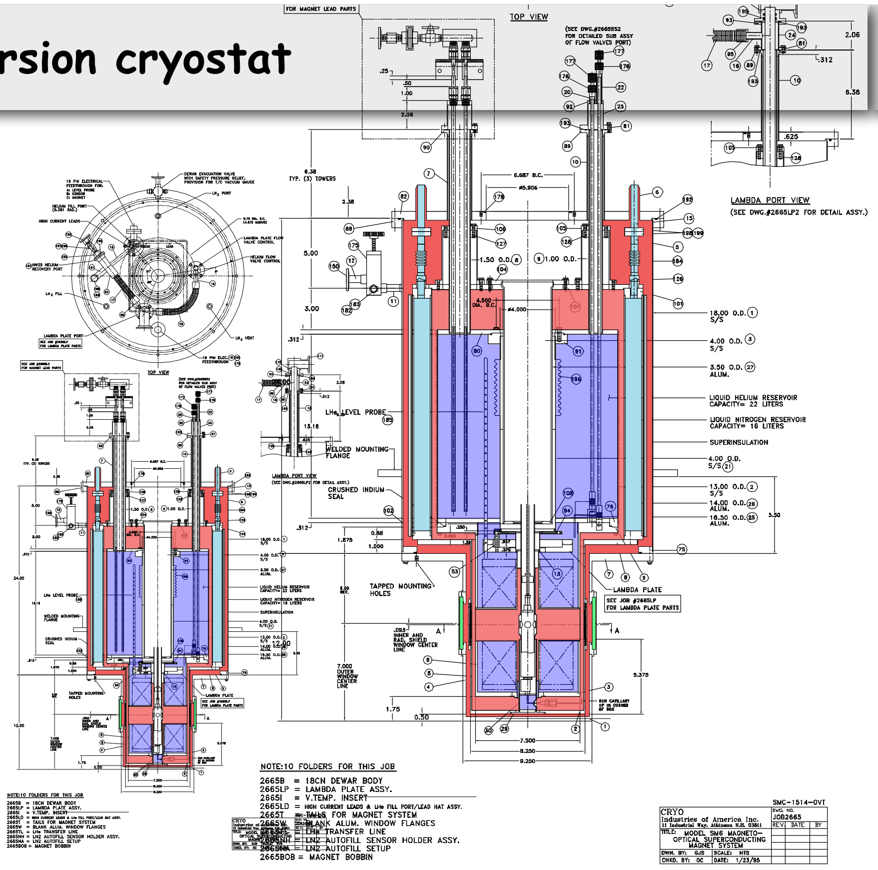
When a cryostat is cooled down to helium temperatures, the various components will contract, and it is useful to know the relative contraction for the various materials that are typically used in such cryostats. Most of the contraction usually occurs when the cryostat has been cooled to liquid nitrogen temperatures. Thus most leaks that occur, when a cryostat is cooled, will show up at liquid nitrogen temperature. Knowledge of the relative contraction is also helpful in designing joints or mechanical contacts, thermal contacts or standoffs at low temperature.

Material	Aluminium	copper	Stainless steel	Brass	Nylon	Teflon	Fused silicon
Thermal Expansion Data Percentage Change in Length (room temperature to 4.2 K)	0.415	0.324	0.306	0.369	1.39	2.14	0.0032

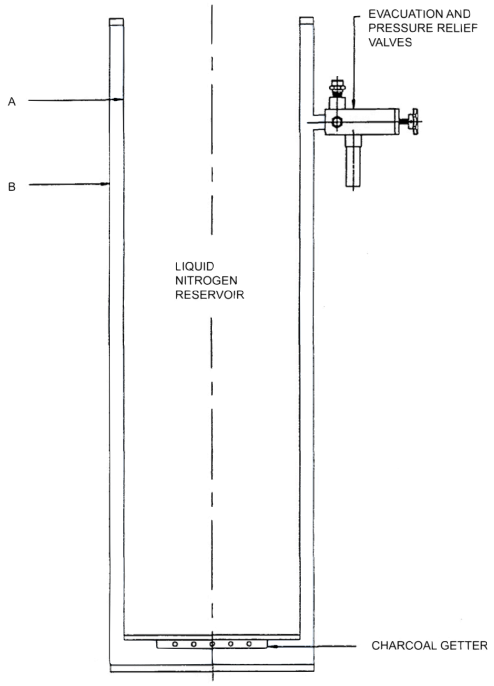
Practical design

All design is compromise

Immersion cryostat

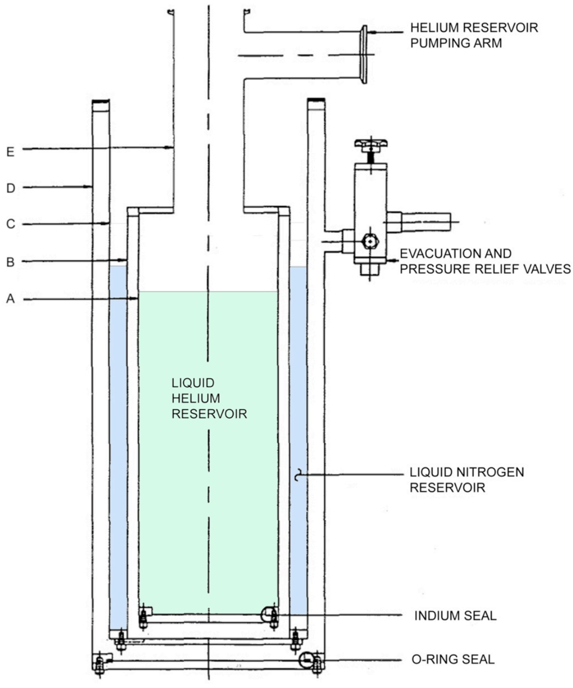


Example: Dewar



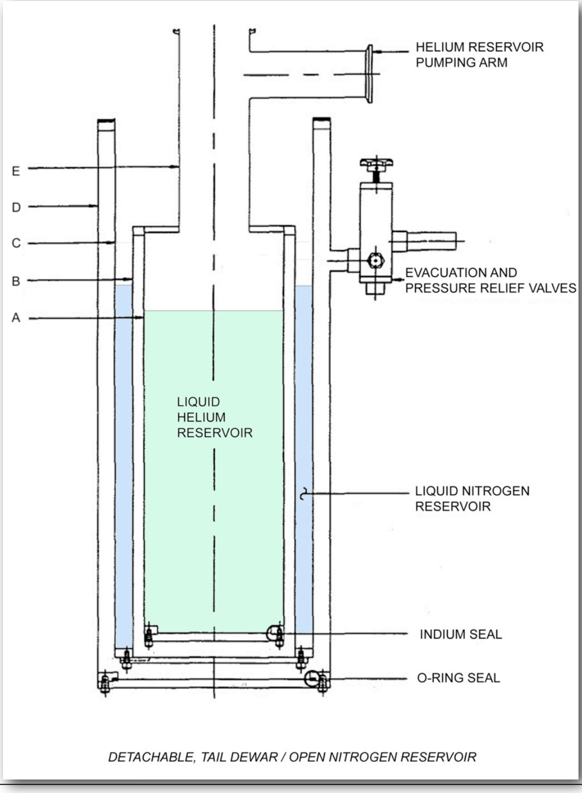
OPEN NECK LIQUID NITROGEN DEWAR

Example: Dewar

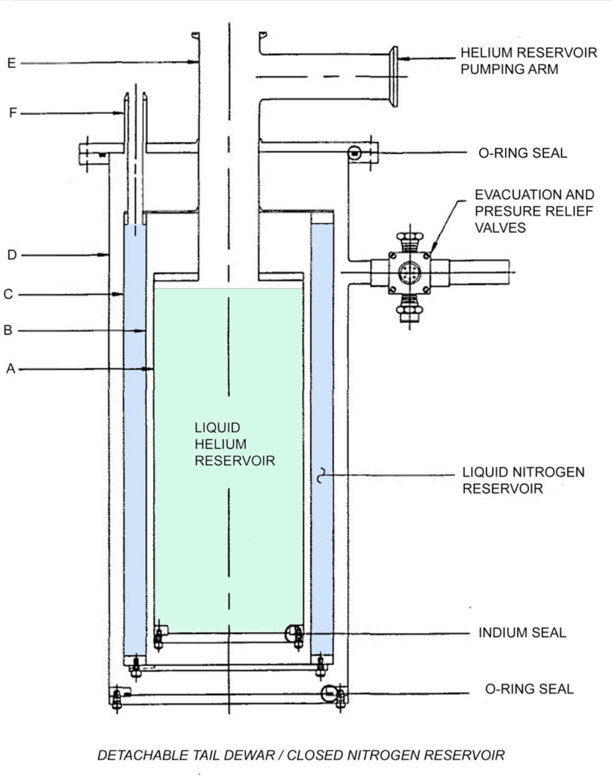


DETACHABLE, TAIL DEWAR / OPEN NITROGEN RESERVOIR

Example: Dewar



DETACHABLE, TAIL DEWAR / OPEN NITROGEN RESERVOIR



DETACHABLE TAIL DEWAR / CLOSED NITROGEN RESERVOIR

The different types of cryostats

Closed-cycle cryostats

- Continuous-flow cryostats
- Bath cryostats
- Multistage cryostats

Types

Bath cryostats

Bath cryostats are similar in construction to vacuum flasks filled with liquid helium. A coldplate is placed in thermal contact with the liquid helium bath. The liquid helium may be replenished as it boils away, at intervals between a few hours and several months, depending on the volume and construction of the cryostat. The boil-off rate is minimised by shielding the bath with either cold helium vapour, or vacuum shield with walls constructed from so-called super insulator material. The helium vapour which boils away from the bath very effectively cools thermal shields around the outside of the bath. In the older designs there may be additional liquid nitrogen bath, or several concentric layers of shielding, with gradually increasing temperatures. However, the invention of super insulator materials has obsoleted this technology.

Types

Closed-cycle cryostats

Closed-cycle cryostats consist of a chamber through which cold helium vapour is pumped. An external mechanical refrigerator extracts the warmer helium exhaust vapour, which is cooled and recycled. Closed-cycle cryostats consume a relatively large amount of electrical power, but need not be refilled with helium and can run continuously for an indefinite period. Objects may be cooled by attaching them to a metallic coldplate inside a vacuum chamber which is in thermal contact with the helium vapour chamber.

Types

Continuous-flow cryostats

Continuous-flow cryostats are cooled by liquid cryogens (typically liquid helium or nitrogen) from a storage dewar. As the cryogen boils within the cryostat, it is continuously replenished by a steady flow from the storage dewar. Temperature control of the sample within the cryostat is typically performed by controlling the flow rate of cryogen into the cryostat together with a heating wire attached to a PID temperature control loop. The length of time over which cooling may be maintained is dictated by the volume of cryogens available.

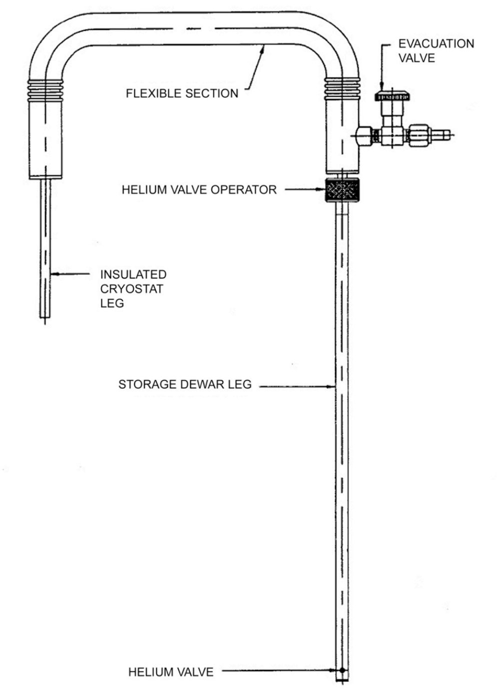
Owing to the scarcity of liquid helium, some laboratories have facilities to capture and recover helium as it escapes from the cryostat, although these facilities are also costly to operate.

Types

Multistage cryostats

In order to achieve temperature lower than liquid helium additional cooler stages may be added to the cryostat. Temperatures down to 1K can be reached by attaching the coldplate to 1-K pot, which is a container of He-4 isotope which is connected to vacuum pump. Temperatures down to 1mK can be reached by employing dilution refrigerator or dry dilution refrigerator typically in addition to the main stage and 1K pot. Temperatures below that can be reached using magnetic refrigeration.

Transfer line



FLEXIBLE TRANSFER LINE FOR CONTINUOUS TRANSFER

Continuous flow cryostat

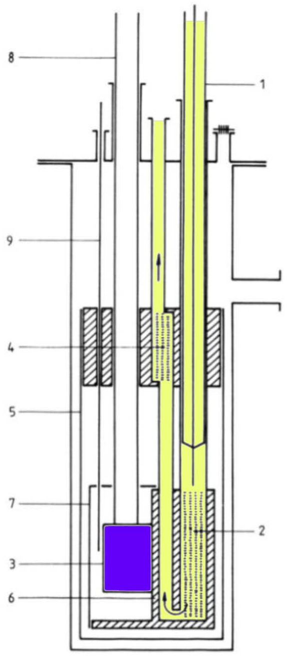
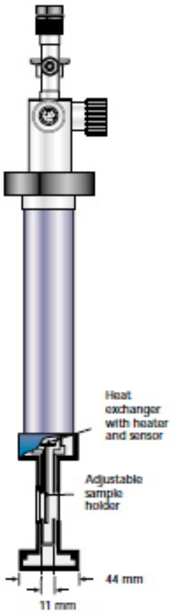
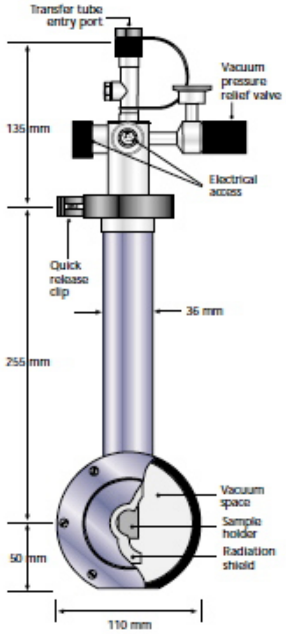
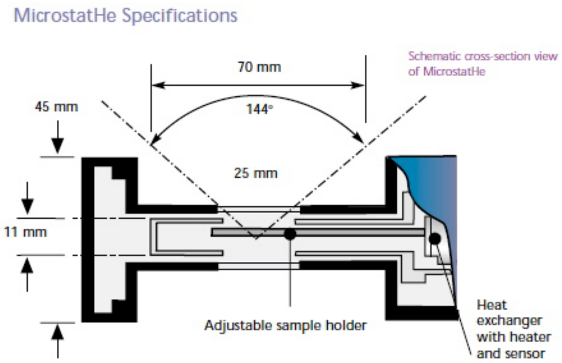


Figure 1. Schematic drawing of the continuous-flow cryostat. (1), Transfer tube; (2), main heat exchanger; (3), sample holder; (4), second heat exchanger; (5), radiation shield; (6), cold copper block; (7), shield; (8), waveguide; (9), rod.

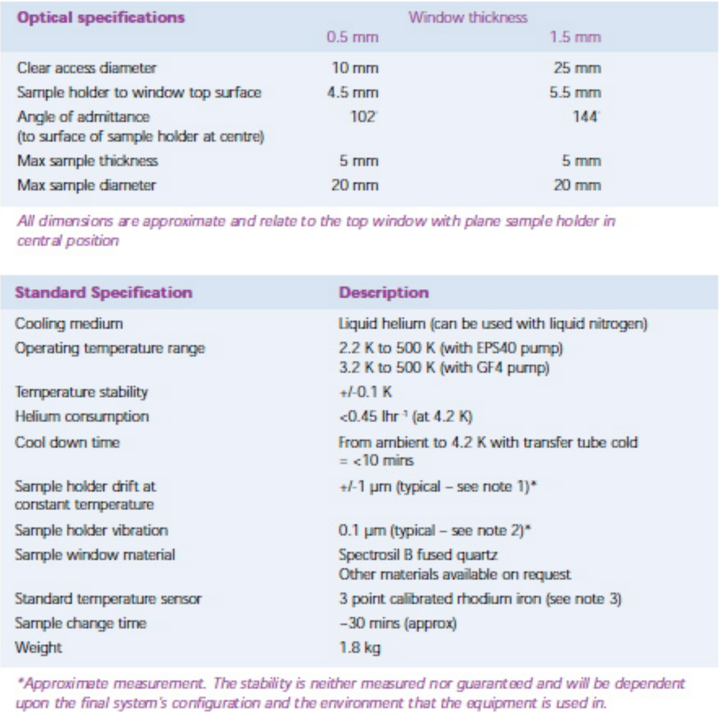
Continuous flow optical cryostat






MicrostatHe Specifications

Continuous flow optical cryostat



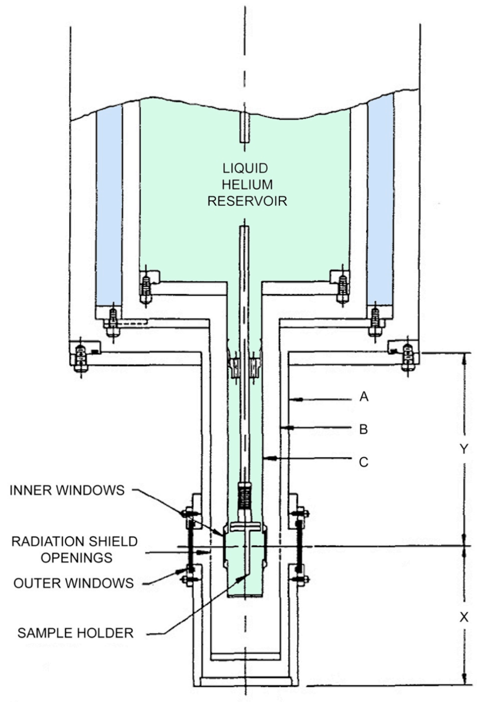
Optical specifications	Window thickness 0.5 mm	 1.5 mm
Clear access diameter	10 mm	25 mm
Sample holder to window top surface	4.5 mm	5.5 mm
Angle of admittance (to surface of sample holder at centre)	102°	144°
Max sample thickness	5 mm	5 mm
Max sample diameter	20 mm	20 mm

All dimensions are approximate and relate to the top window with plane sample holder in central position

Standard Specification	**Description**
Cooling medium	Liquid helium (can be used with liquid nitrogen)
Operating temperature range	2.2 K to 500 K (with EPS40 pump) 3.2 K to 500 K (with GF4 pump)
Temperature stability	+/-0.1 K
Helium consumption	<0.45 lhr^{-1} (at 4.2 K)
Cool down time	From ambient to 4.2 K with transfer tube cold = <10 mins
Sample holder drift at constant temperature	+/-1 µm (typical – see note 1)*
Sample holder vibration	0.1 µm (typical – see note 2)*
Sample window material	Spectrosil B fused quartz Other materials available on request
Standard temperature sensor	3 point calibrated rhodium iron (see note 3)
Sample change time	~30 mins (approx)
Weight	1.8 kg

**Approximate measurement. The stability is neither measured nor guaranteed and will be dependent upon the final system's configuration and the environment that the equipment is used in.*

Bath cryostat



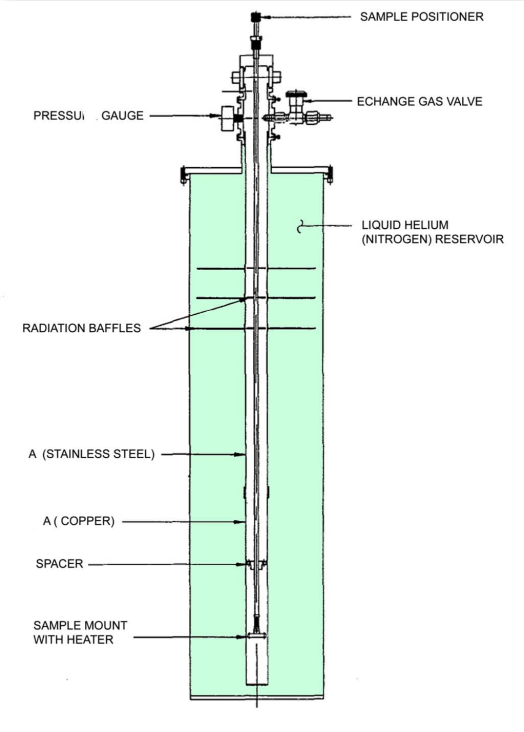
IMMERSION OPTICAL TAIL

Helium cryostat

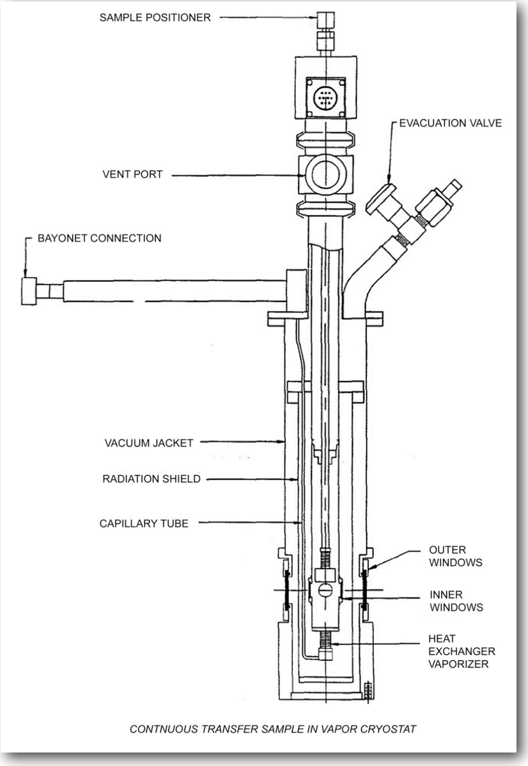
the specimen can be:

- in contact with liquid Helium
- in contact with cold gas Helium
- in vacuum

Exchange gas cryostat

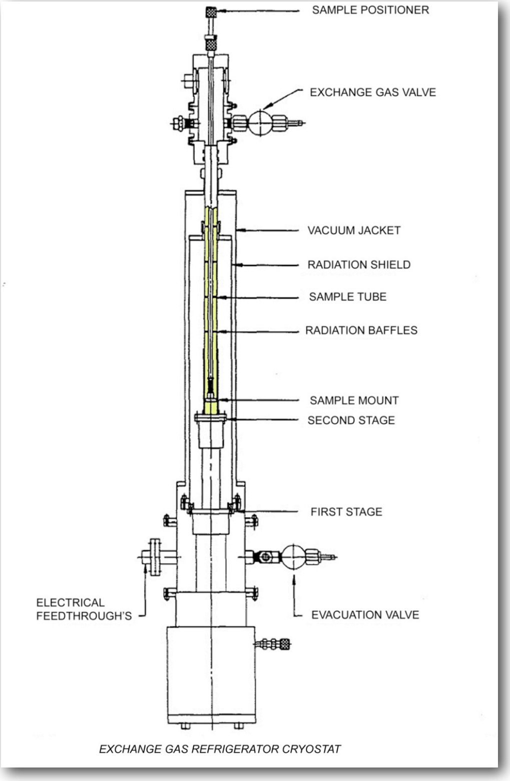


EXCHANGE GAS INSERT



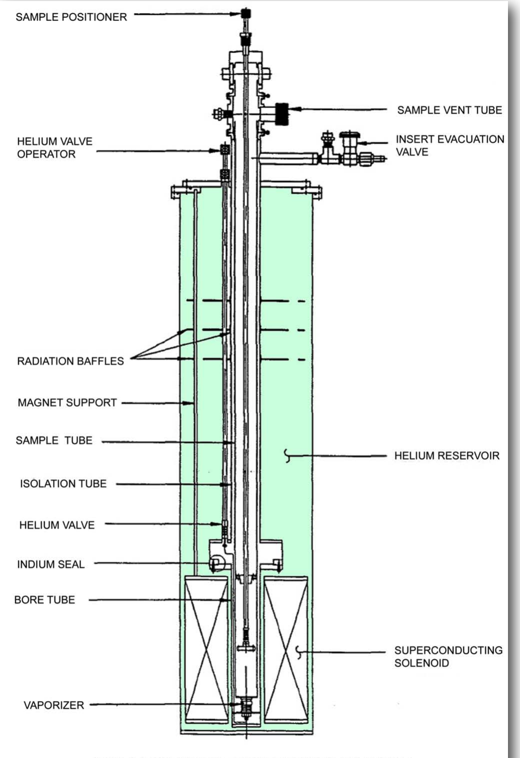
CONTNUOUS TRANSFER SAMPLE IN VAPOR CRYOSTAT

Closed cycle - exchange gas cryostat



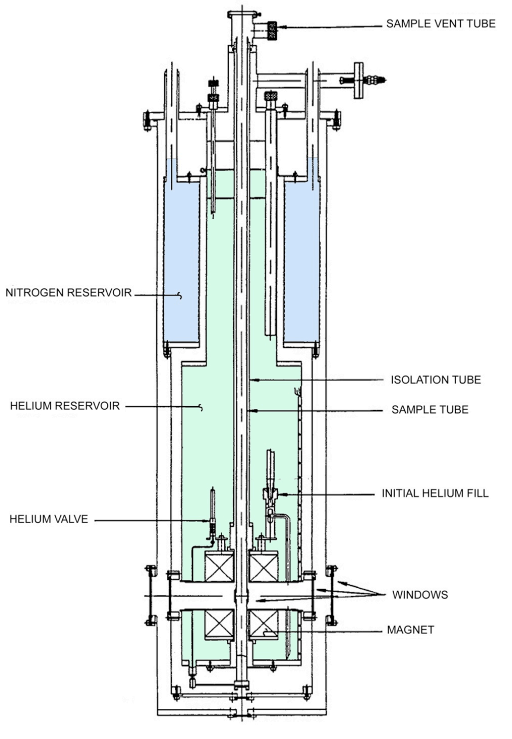
EXCHANGE GAS REFRIGERATOR CRYOSTAT

Helium cryostat with superconducting magnet

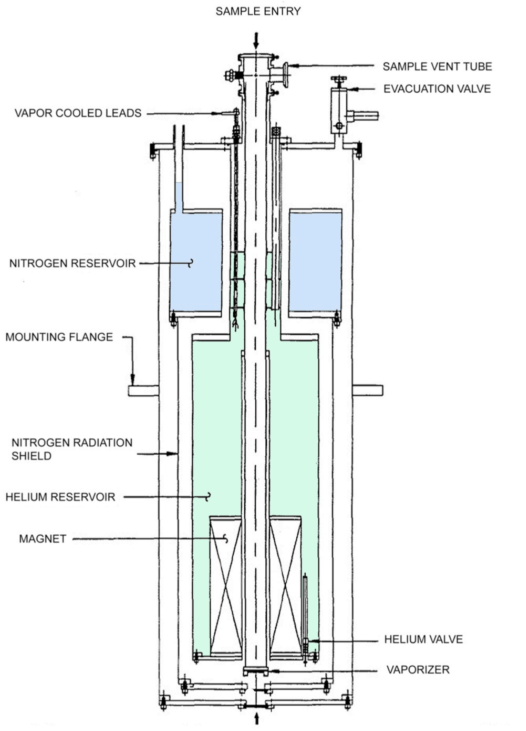


FLOW CRYOSTAT INSERT FOR SUPERCONDUCTING MAGNET

Helium cryostat with superconducting magnet



MAGNET CRYOSTAT FOR MAGNETO-OPTICAL EXPERIMENTS

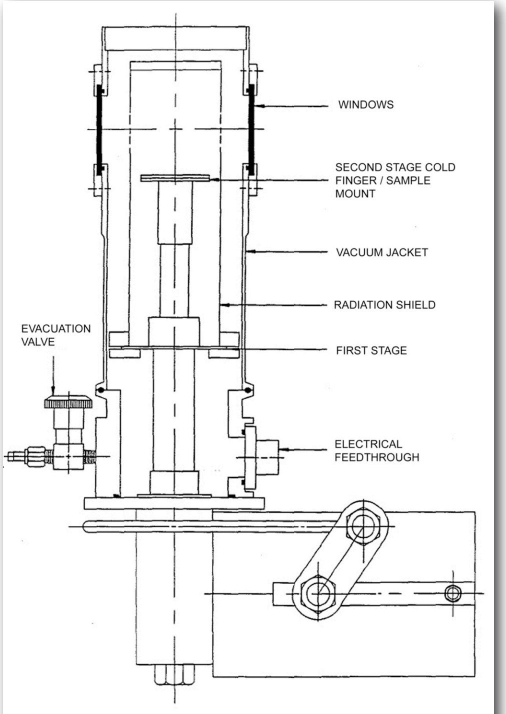


MAGNET CRYOSTAT WITH BOTTOM OPTICAL ACCESS

Lambda point refrigerator (from wikipedia)

A lambda point refrigerator is a device used to cool liquid helium, typically around a superconducting magnet or for low temperature measurements, from approximately 4.2 K to temperatures near the lambda point of helium (approximately 2.17 K), the temperature at which normal fluid helium (helium I) transitions to the superfluid helium II. Cooling is achieved by pumping the liquid helium in the bath through a cooling coil via a needle valve and vacuum pump. The reduced pressure in the coil causes some of the helium to evaporate, creating a two-phase system within the cooling coil. The heat removed via evaporation lowers the temperature of the cooling coil closer to the lambda point. Since the cooling coil is immersed in the liquid helium bath, liquid surrounding the coil is also cooled. The colder, higher density liquid sinks away from the coil toward the bottom of the bath while the warmer, lower density liquid helium rises to the top. Liquid helium typically has poor thermal conductivity, so convective currents associated with a temperature gradient in the bath provide a constant flow of this colder liquid helium toward the bottom of the bath, allowing temperatures below 4.2 K to be realized in the helium bath, typically close to 2.2 K.

Cryostat - closed cycle



CLOSED CYCLE REFRIGERATOR CRYOSTAT

How it works ?

sterling cycle

- two stages

advantages:

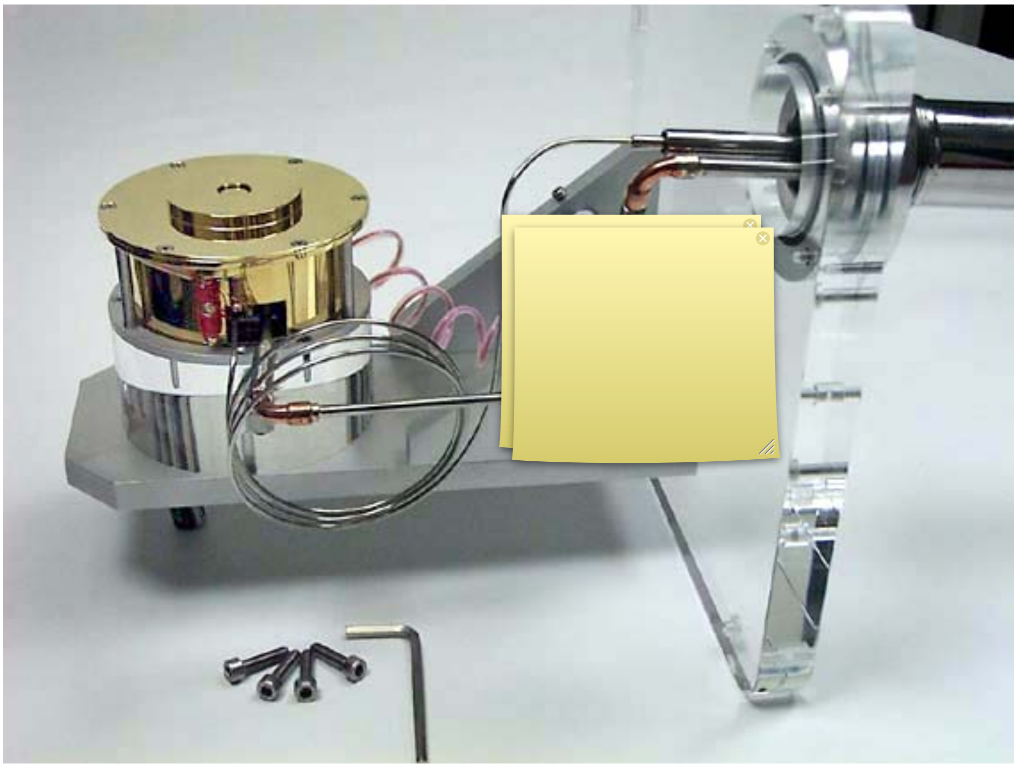
- little or no consumption of helium
- low temperature <8K

disadvantages:

- maintenance (moving parts, ...)
- vibrations

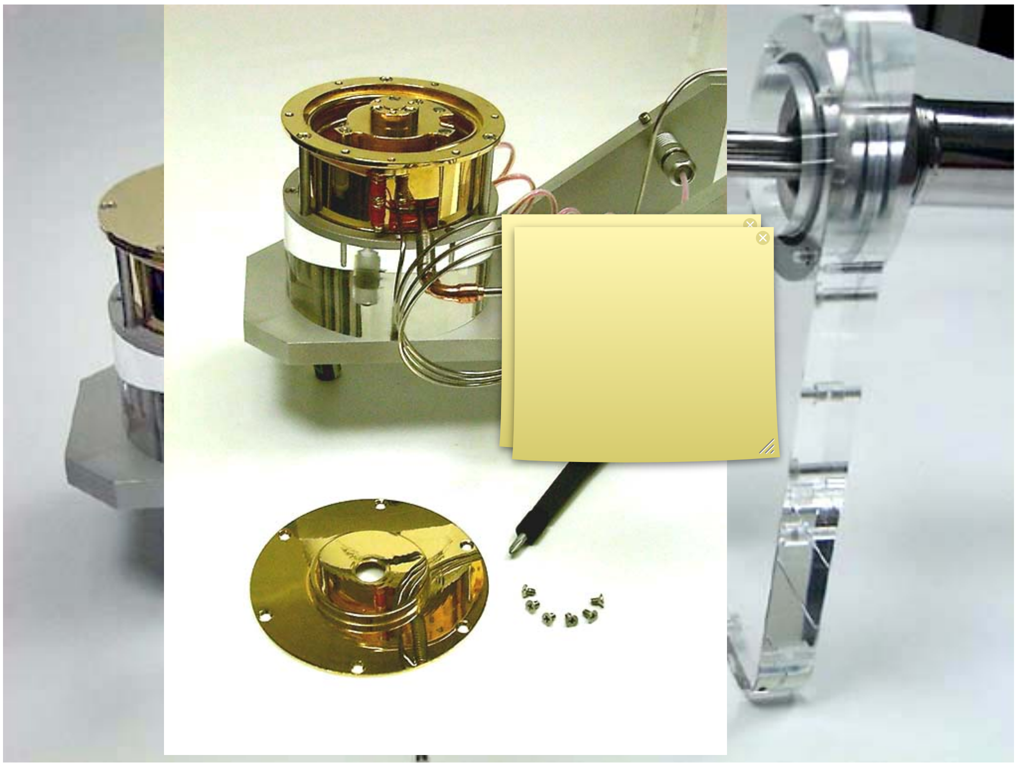
SEM cryostat

Cryogenics



SEM cryostat

Cryogenics



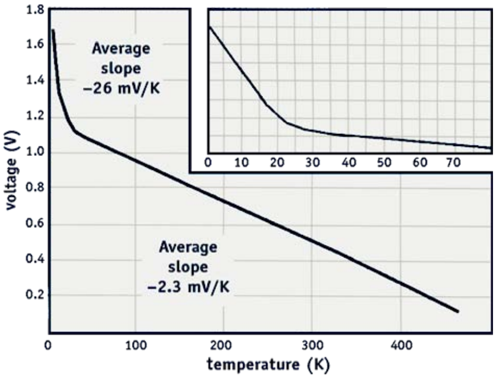
Temperature sensors (from lakeshore.com)

There are many types of commercially available temperature sensors that can be used at low temperatures. The ones that are most commonly used are silicon diodes, germanium resistors, carbon glass resistors, platinum resistors, Gallium (and Gallium-Aluminum) Arsenide diodes, and rhodiumiron resistors. These thermometers tend to retain their characteristics with repeated thermal cycling, and thus can be reliably calibrated. Silicon, gallium arsenide and gallium-aluminum arsenide.

Silicon diodes (from lakeshore.com)

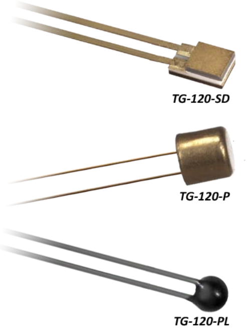
Silicon diodes have become the most common type of thermometers used in the temperature range of 1.5 K to 300 K (or 475 K). When activated with a 10 micro-ampere constant current source, they offer a voltage of about 1.7 volts at low temperatures, dropping down to about 0.5 volts at room temperature. Their sensitivity ranges between approximately 25 mV/ K below 20 K to 2.3 mV/ K above 70 K. Because they are activated with a constant current, they are the obvious choice for use with an automatic temperature controller.

DT-470/471-SD



Gallium Arsenide Diodes (from lakeshore.com)

GaAs and GaAIAs diodes are also excited with a constant current (10 or 100 micro-Amps), but they cannot be mass produced with temperature characteristics which conform with any standard curve. They can still be calibrated and used with a simple (constant current) temperature controller. Their only advantage over si diodes, is that they can be used in low magnetic fields (1 to 2 Tesla) since their voltage does not vary significantly with the applied field.



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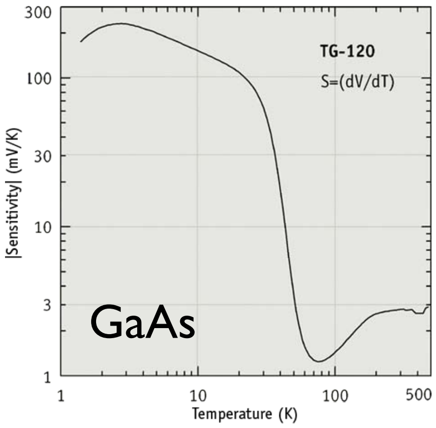
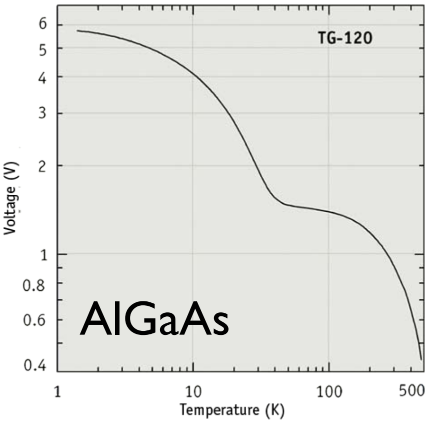
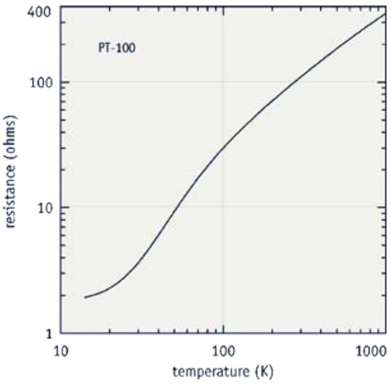
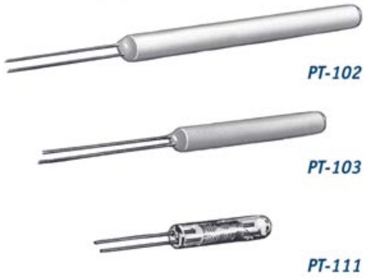




Platinum resistors (from lakeshore.com)

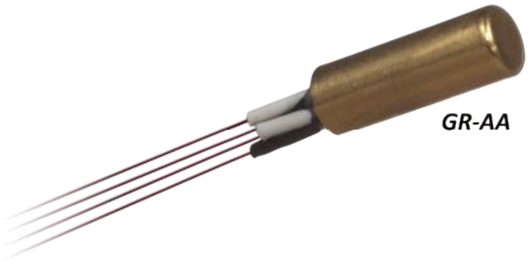
Platinum resistance thermometers are generally used between 60 K and 300 K, where the resistance increases at a fairly constant rate of about 0.4 ohms/ K. They are occasionally used down to about 20 K, but their sensitivity drops to about 0.08 ohm/ K. Their ceramic outer case, allows these sensors to be used up to 600·C, however it is difficult to obtain an epoxy that will thermally anchor the thermometer and its leads at the lower temperatures, and also withstand the higher temperatures.

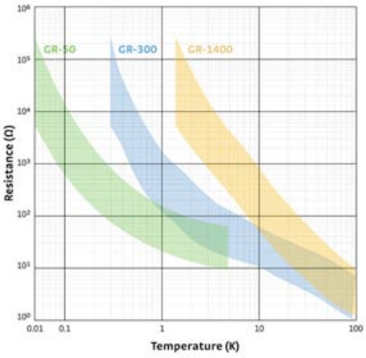




Germanium resistors (from lakeshore.com)

These thermometers are always supplied in a four lead configuration, labeled for current and voltage.
Installation of these sensors requires careful thermal anchoring of these leads as discussed earlier. The resistance of these thermometers decreases with increasing temperature, up to 100 K, where the curve starts slowly turning -- thus limiting the usefulness of these sensors to 100 K or less. One class of sensors is commercially available for use between 1.5 K and 100 K, while another class is used for lower temperature ranges (6 K to 0.3 K or 0.05 K). Their resistance changes by several orders





The movie ! (Uni. Joseph Fourier -Grenoble)