CRYOGENICS

PHY 590B F18

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History

Materials

Design

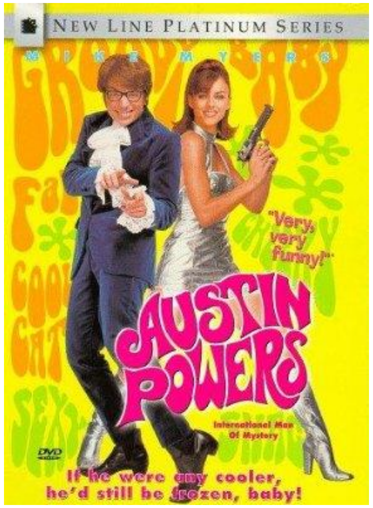
WIKIPEDIA

Cryogenics

The branches of physics and engineering that involve the study of very low temperatures, how to produce them, and how materials behave at those temperatures.

Cryonics

The emerging medical technology of cryopreserving humans and animals with the intention of future revival.



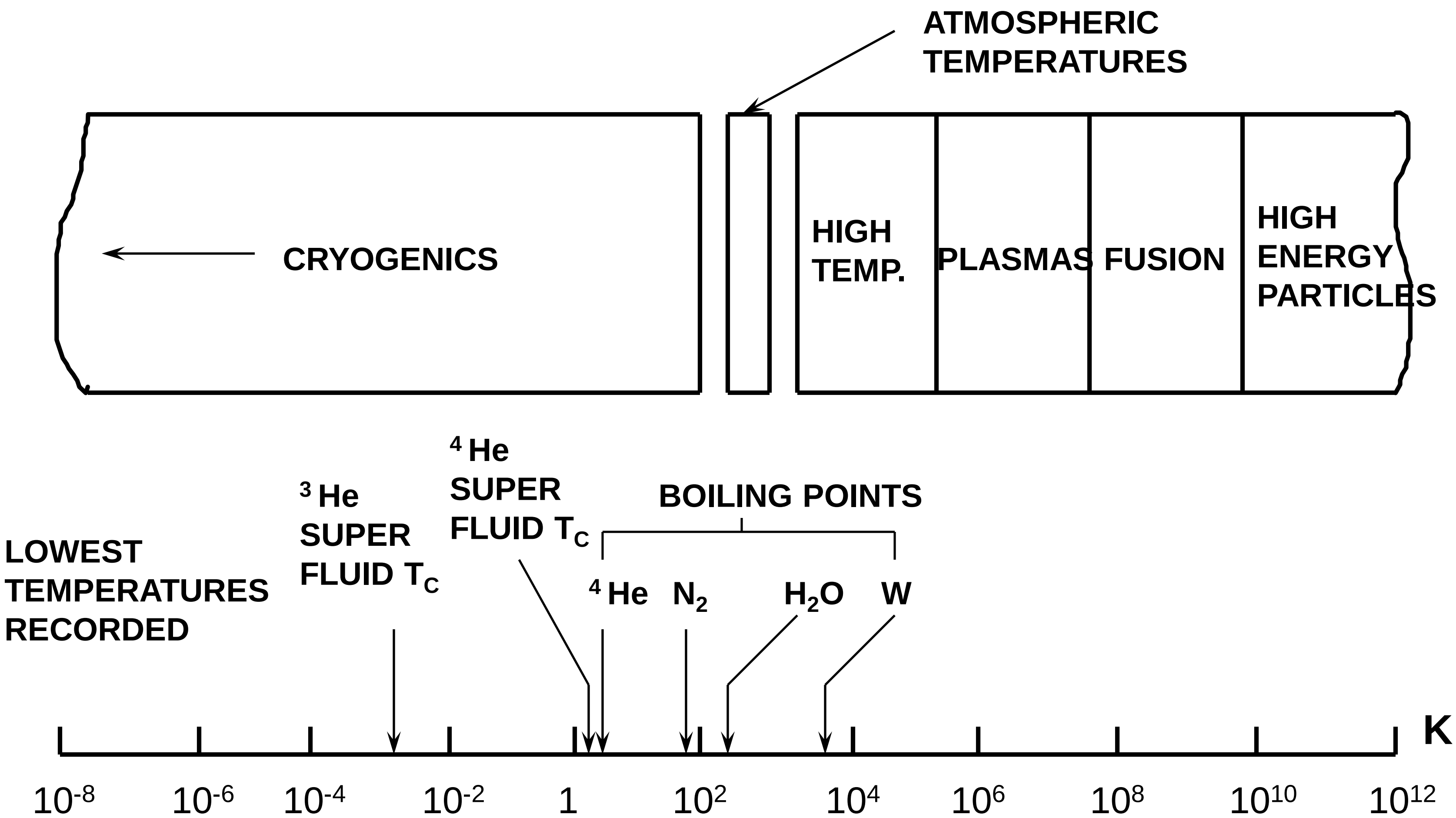
Some typical low temperatures

	Temperature Celsius (°C)	Temperature Absolute (K)
Tropics	45	318
Human body	37	310
Room temperature	20	293
Ice point	0	273
Salt + water (cryogen)	-18	255
Antarctic winter	-50	223
Solid carbon dioxide	-78	195
Liquid oxygen	-183	90
Liquid nitrogen	-196	77
Liquid helium	-269	4
Absolute zero	-273	0

Iowa record: +48°C

Iowa record: -44°C

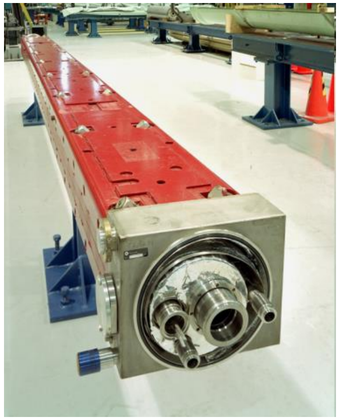
Logarithmic temperature scale



cryo-surgery and veterinary medicine

cryo-transport of natural gas

100 t LH_2 + 600 t LO_2



research - physics

medicine

space

life

Historical cryogenics events

1850   Mechanical refrigeration first applied

1877   Cailetet & Pictet liquefied O_2 (90 K).

1892   James Dewar developed the vacuum flask

1908   Kamerlingh Onnes liquefied He (4.2 K).

1911   K. O. discovered superconductivity.

1926   Giauque reached 0.25 K by magnetic cooling.

1946   Collins developed commercial He liquefier.

1995   Connell & Wiemann achieved Bose-Einstein condensation at $2x10^{-8}$ K.

Characteristic temperatures of low-energy phenomena

Phenomenon	Temperature
Debye temperature of metals	few 100 K
High-temperature superconductors	~ 100 K
Low-temperature superconductors	~ 10 K
Intrinsic transport properties of metals	< 10 K
Cryopumping	few K
Cosmic microwave background	2.7 K
Superfluid helium 4	2.2 K
Bolometers for cosmic radiation	< 1 K
Low-density atomic Bose-Einstein condensates	~ μK

record 260 K @ ~2 Mbar?

Materials change at low temperatures

Structural phase transition (brittle-ductile – Ti alloys)

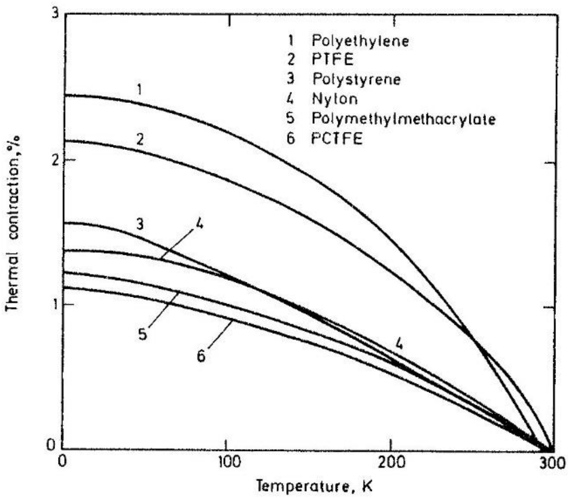
CW paramagnetism, long range magnetic order, spin glass

Superconductivity (annoying traces of it – phosphor bronze, Ti-alloys, many solders)

Freezing/glassing (liquids, oils, greases)

Condensation/liquefaction (gases)

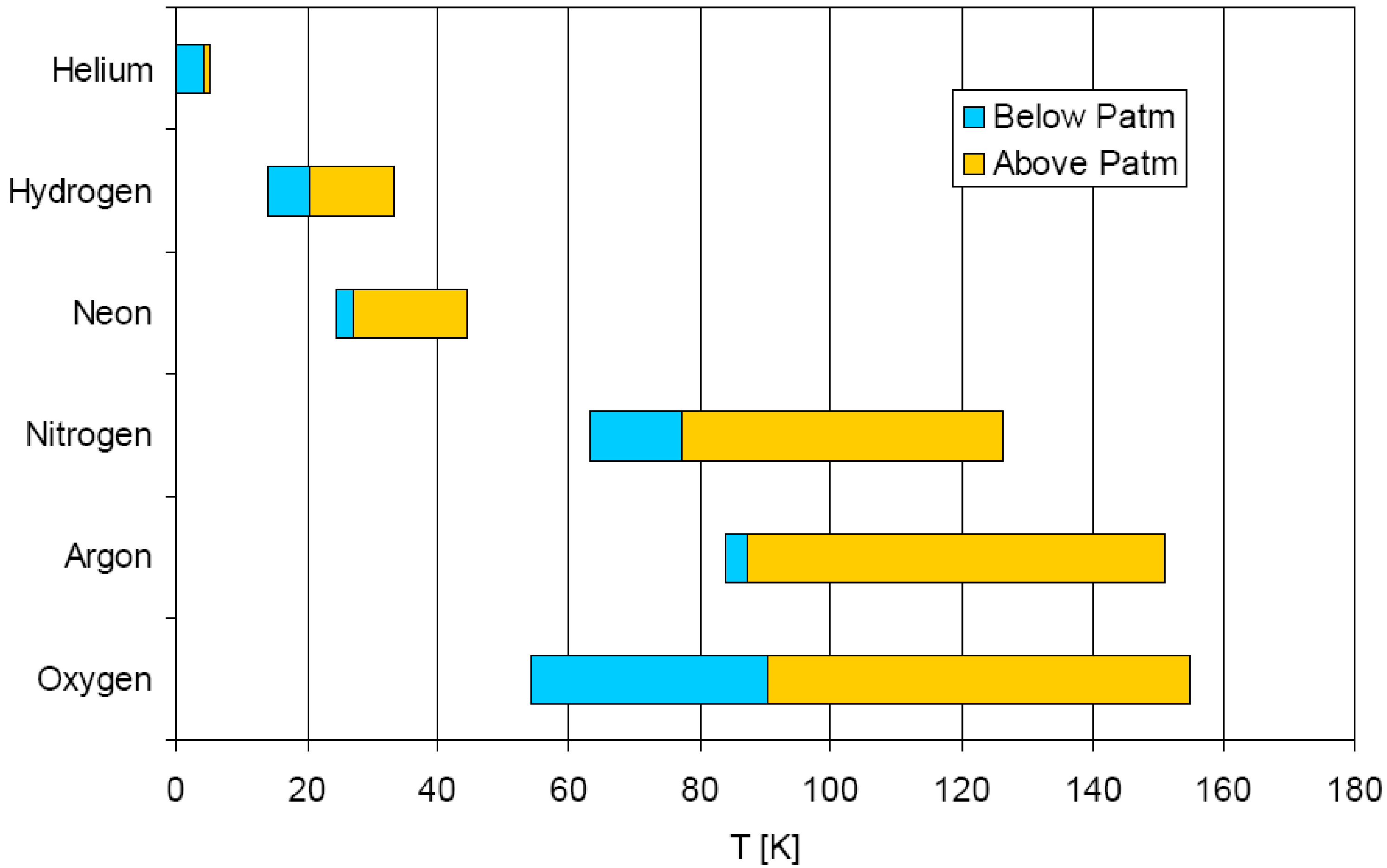
Change of mechanical properties (rubber, plastics)



see appendix

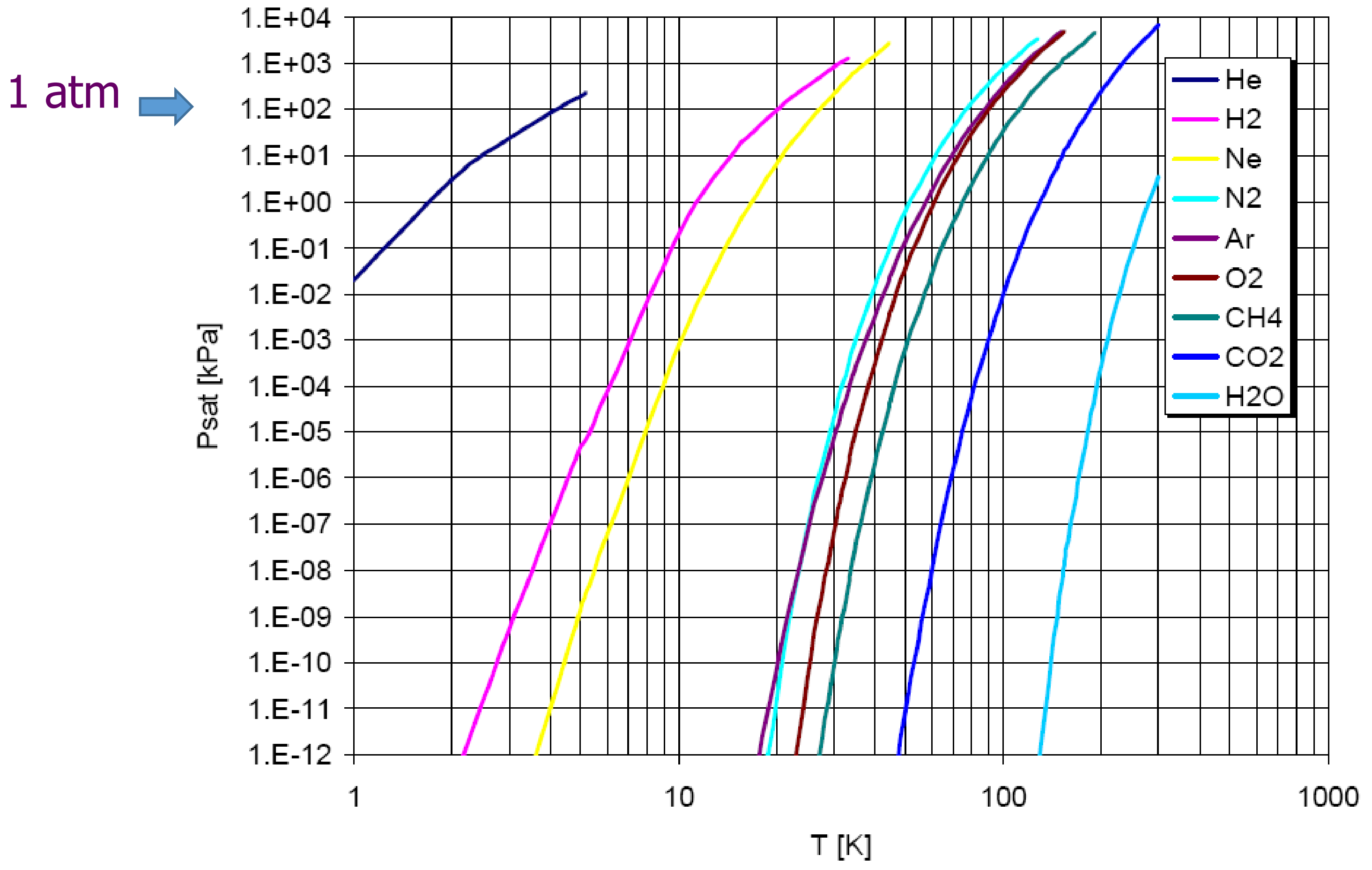
Cooling with cryogenic liquids

Useful range of cryogens



Cooling with cryogenic liquids

Vapour pressure at cryogenic temperatures



Cooling with cryogenic liquids

Normal boiling point and latent heat of fluids

Water	: 373 K	2256 kJ/kg
Ethylene	: 169 K	481 kJ/kg
Krypton	: 120 K	116 kJ/kg
Methane	: 111 K	512 kJ/kg
Xenon	: 110 K	99 kJ/kg
Oxygen	: 90 K	213 kJ/kg
Argon	: 87 K	162 kJ/kg
Nitrogen	: 77 K	199 kJ/kg
Neon	: 27 K	86 kJ/kg
Hydrogen	: 20 K	443 kJ/kg
Helium	: 4.2 K	21 kJ/kg

Cooling with cryogenic liquids

Amount of cryoliquids (in liters) to cool 1 kg of metal if only latent heat (latent heat + enthalpy of the gas) is used.

Cryoliquid	Temperature change (K)	Al	SS	Cu
N_2	300 - 77	1.0 (0.63)	0.53 (0.33)	0.46 (0.28)
^4He	77 – 4.2	3.2 (0.2)	1.4 (0.1)	2.2 (0.16)
^4He	300 – 4.2	66 (1.6)	34 (0.8)	32 (0.8)

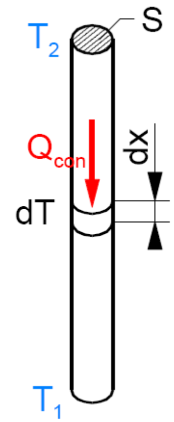
Use LN_2 to precool the equipment (caution and patience needed)

Make use of the enthalpy of the cold He gas (very slow initial transfer, end of the transfer tube close to the bottom)

Heat sources

(i) Heat conduction

Heat conduction in solids



Fourier's law: $Q_{con} = k(T) \cdot S \cdot \frac{dT}{dx}$

k(T): thermal conductivity [W/m.K]

Integral form: $Q_{con} = \frac{S}{L} \cdot \int_{T1}^{T2} k(T) \cdot dT$

$\int k(T) \cdot dT$: thermal conductivity integral [W/m]

Thermal conductivity integrals for standard construction materials are tabulated

Thermal conductivity integrals of selected materials [W/m]

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

stainless steel

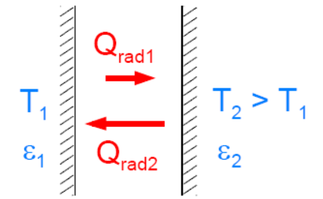
thin walls

G10

Heat sources

Thermal radiation

(ii) Heat radiation



- Wien's law
  - Maximum of black body power spectrum
    $\lambda_{max} \cdot T = 2898$ [μm.K]
- Stefan-Boltzmann's law
  - Black body: $Q_{rad} = \sigma A T^4$
    $\sigma = 5.67 \times 10^{-8}$ W/m^2.K^4
    (Stefan Boltzmann's constant)
  - "Gray"body: $Q_{rad} = \varepsilon \sigma A T^4$
    ε emissivity of surface
  - "Gray" surfaces at T_1 and T_2: $Q_{rad} = E \sigma A (T_1^4 - T_2^4)$
    E function of ε_1, ε_2, geometry

Emissivity of technical materials at low temperatures

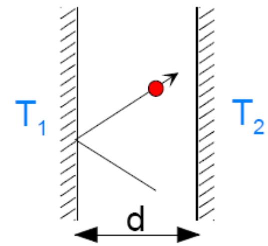
	Radiation from 290 K Surface at 77 K	Radiation from 77 K Surface at 4.2 K
Stainless steel, as found	0.34	0.12
Stainless steel, mech. polished	0.12	0.07
Stainless steel, electropolished	0.10	0.07
Stainless steel + Al foil	0.05	0.01
Aluminium, as found	0.12	0.07
Aluminium, mech. polished	0.10	0.06
Aluminium, electropolished	0.08	0.04
Copper, as found	0.12	0.06
Copper, mech. Polished	0.06	0.02

polished surfaces

radiation shields

Heat sources

(iii) Residual gas conduction



Residual gas conduction

$\lambda_{molecule}$: mean free path of gas molecules

getters on cold walls

- Molecular regime
  - At low gas pressure $\lambda_{molecule} >> d$
  - Kennard's law $Q_{res} = A\,\alpha(T)\,\Omega\,P\,(T_2 - T_1)$
  - Conduction heat transfer proportional to pressure, independant of spacing between surfaces
    Ω depends on gas species
  - Accommodation coefficient $\alpha(T)$ depends on gas species, T_1, T_2, and geometry of facing surfaces

(iv) Thermoacoustic (Taconis) oscillations

standing waves in gas filled tubes with temperature gradient and closed warm end

change in geometry

introduction of damping element

use of Taconis oscillations

Superinsulation

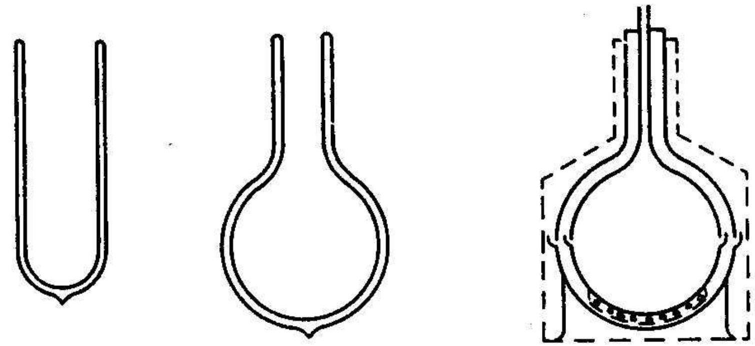
Multi-layer insulation (MLI)

- Complex system involving three heat transfer processes
  - $Q_{MLI} = Q_{rad} + Q_{sol} + Q_{res}$
  - With n reflective layers of equal emissivity, $Q_{rad} \sim 1/(n+1)$
  - Due to parasitic contacts between layers, Q_{sol} increases with layer density
  - Q_{res} due to residual gas trapped between layers, scales as 1/n in molecular regime

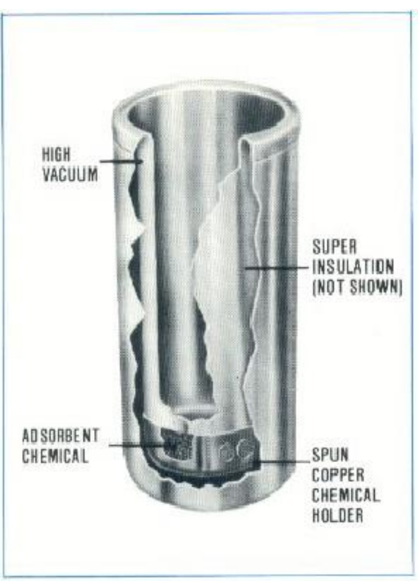
Typical heat fluxes at vanishingly low temperature between flat plates [W/m^2]

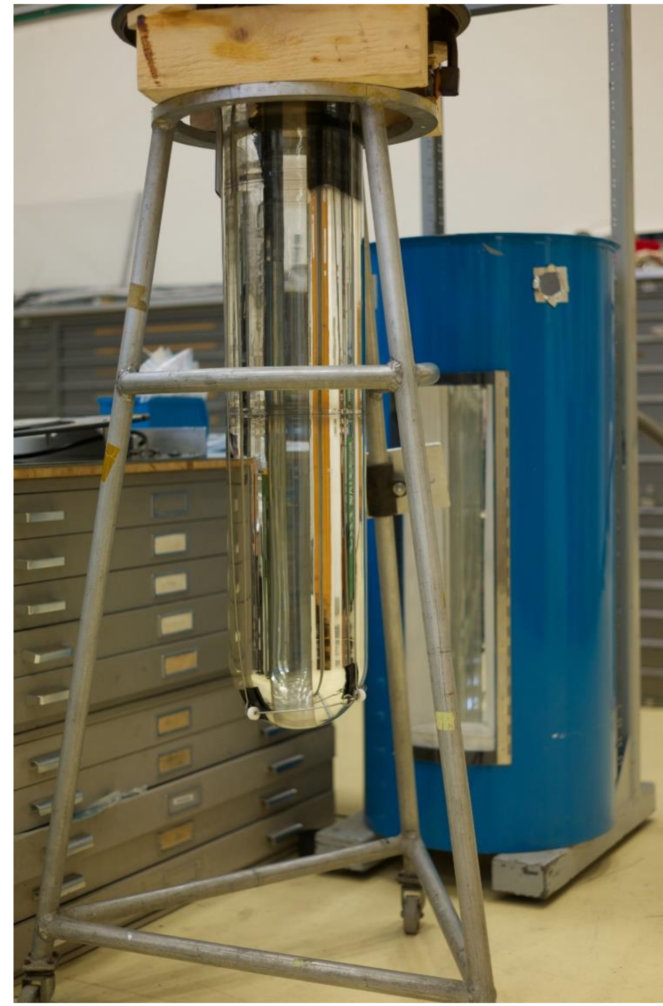
Black-body radiation from 290 K	401
Black-body radiation from 80 K	2.3
Gas conduction (100 mPa He) from 290 K	19
Gas conduction (1 mPa He) from 290 K	0.19
Gas conduction (100 mPa He) from 80 K	6.8
Gas conduction (1 mPa He) from 80 K	0.07
MLI (30 layers) from 290 K, pressure below 1 mPa	1-1.5
MLI (10 layers) from 80 K, pressure below 1 mPa	0.05
MLI (10 layers) from 80 K, pressure 100 mPa	1-2

Dewar flask



(a) Glass (b) Glass (c) Metal





Was able to engage in quite vitriolic arguments with other scientists: "…argue with Dewar was akin to being a fly in molasses…"

Lost race for He liquefaction

Lost patent case against *Thermos* in court

Was nominated many times but did not get Nobel prize

Refused to retire from Fullerian Professorship in Chemistry at the Royal Institution

Sir James Dewar, FRS (1842-1923)

Developed *Cordite* (smokeless gunpowder alternative) – with Sir Frederick Abel, 1889

Described several formulae for benzene, 1867

Studied physiological action of light, with Prof. J. G. McKendrick (conditions of retina)

Spectroscopical observations, with Prof. G.D. Living

Public liquefaction of oxygen and air, 1884

Cir. 1892 – vacuum flask

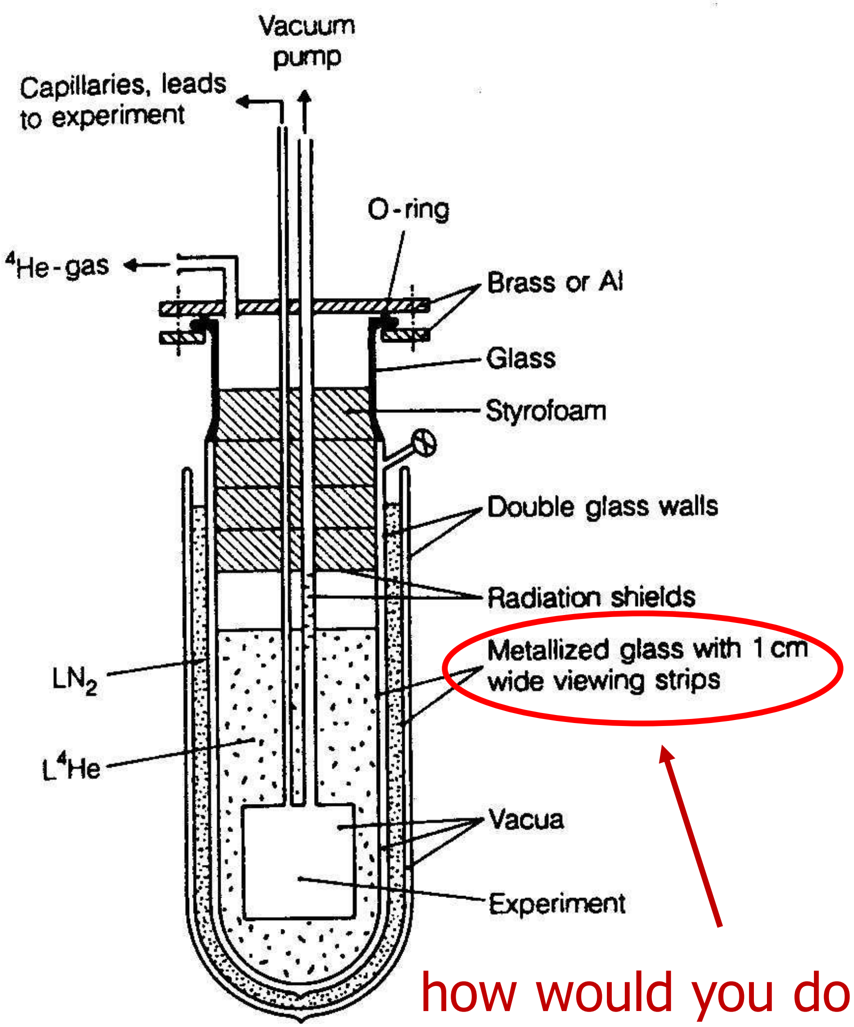
Liquid hydrogen, 1898, solid hydrogen, 1899

Gas-absorbing powers of charcoal, 1905

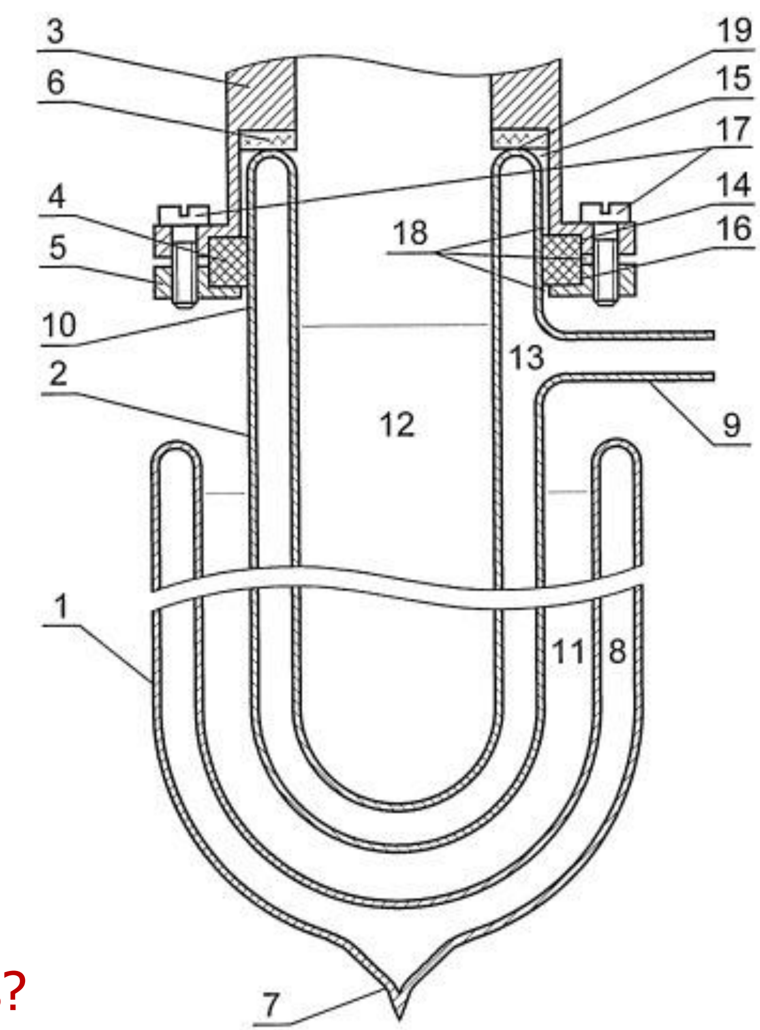
Surface tension of soap bubbles, during and after WW1

Last publication in 1923 (at 80 yrs age)

Glass cryostats – more details



how would you do this?



Фиг. 1

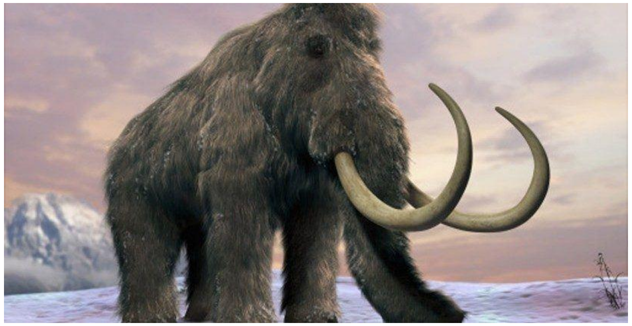
LN_2 dewar – no valve, LHe dewar - valve: why?

“silver mirror reaction” or Tollens’ test

qualitative laboratory test used to distinguish between an aldehyde and a ketone

you can try to see demonstration at one of the ISU’s GenChem or Organic Chemistry courses

Glass research cryostats



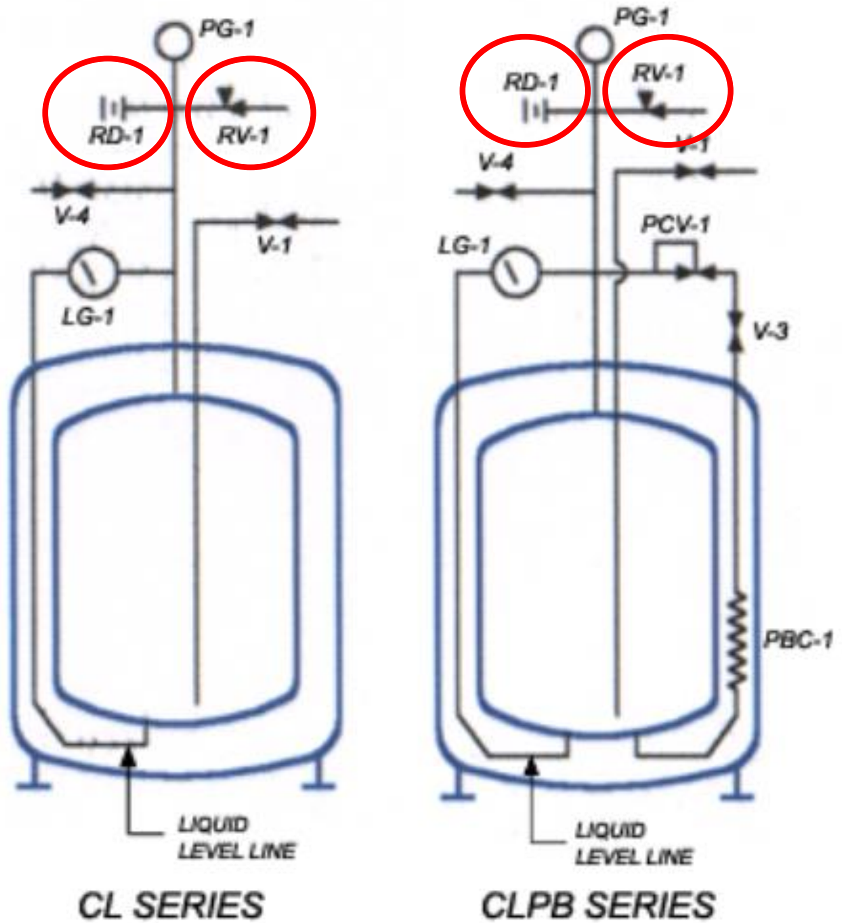
Basically extinct by now

At a time:
- **Were cheap**
- **Could be fabricated and repaired in a good glass shop**
- **Offer visual control of cryogens**
- **Small amount of cryogens**

However
- **Not particularly safe (break in multiple glass pieces flying around – unless you use nylon stockings)**
- **Need to take particular care of overpressure safety features**
- **Not recommended to leave unattended**

Storage dewars

Storage dewars – LN_2



Symbol	Description
V-1	Liquid Fill/Withdrawal
V-2	Fill/Wlithdrawal Valve
V-3	Pressure Building
V-4	Vent Valve
RD-1	Rupture Disk
RV-1	Relief Device
Pg-1	Pressure Guage
LG-1	Liquid Level Buliding
PCV-1	Pressure Building Regulator
PBC-1	Pressure Building Coil

Storage dewars - LHe

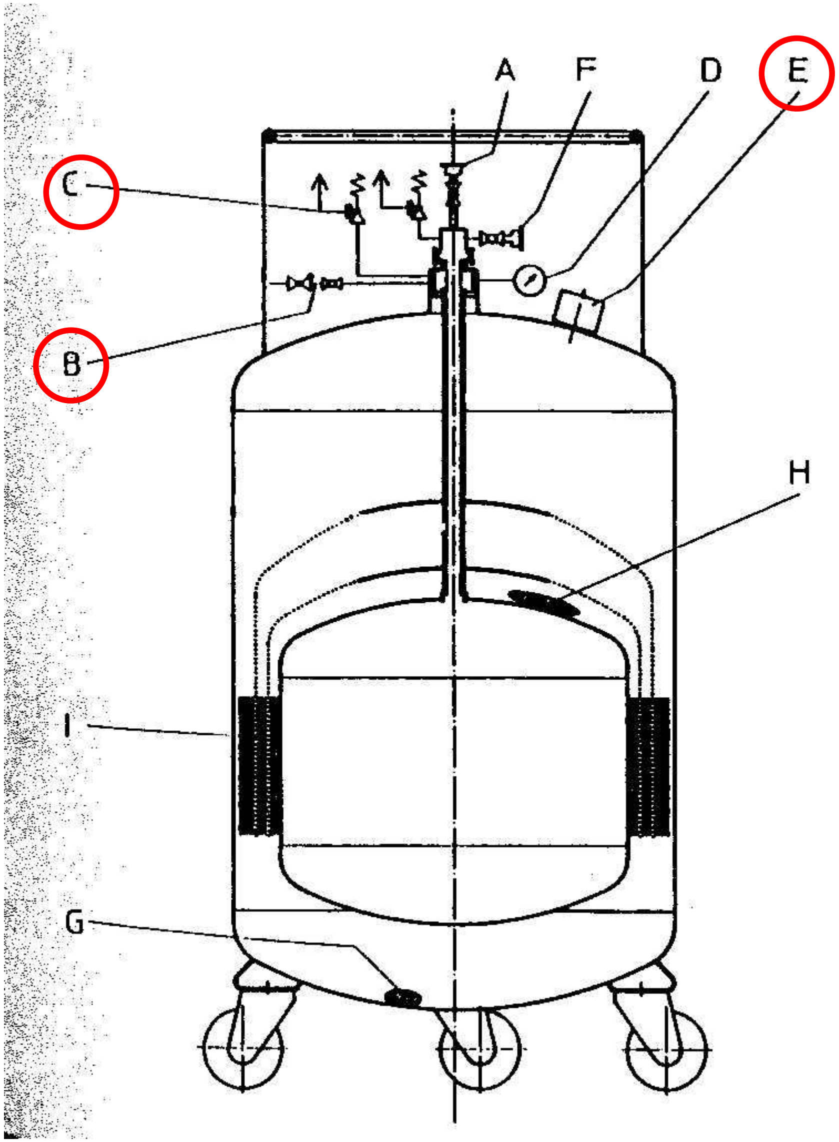
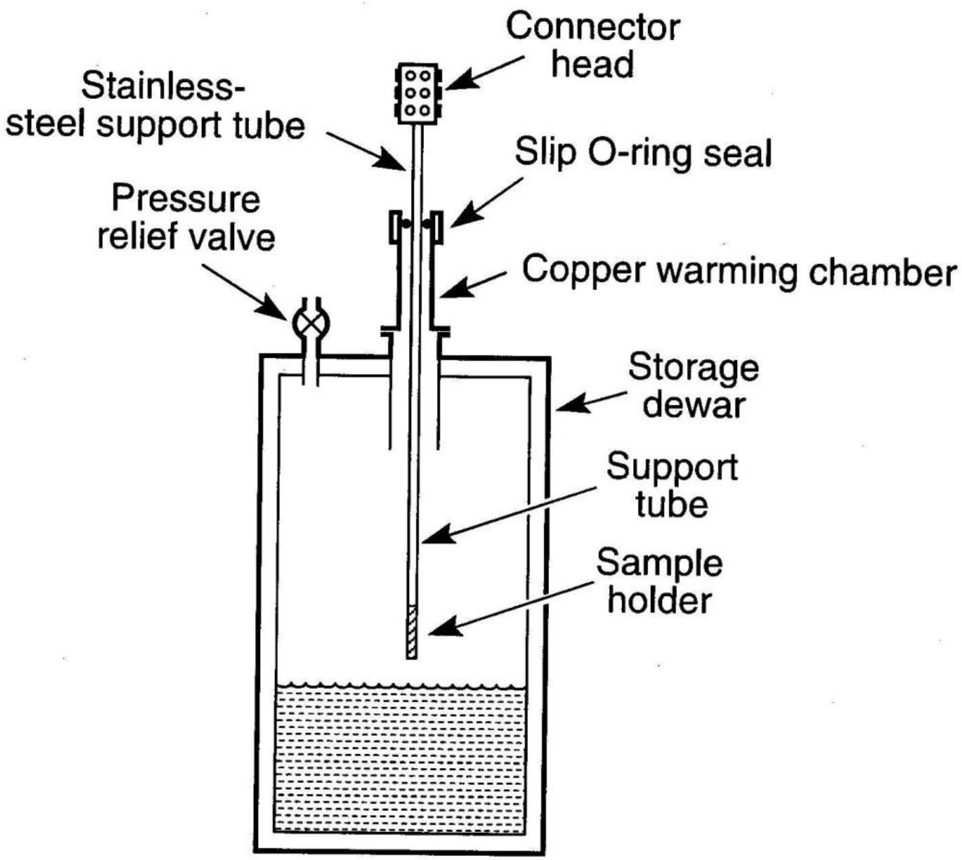


Fig.5.7. Commercial storage vessel for liquid ^{4}He (*A*: connection for transfer tube, *B*: overflow valve, *C*: safety valve, *D*: manometer, *E*: vacuum and safety valves, *F*: gas valve, *G*: getter material, *H*: adsorbent material, *I*: superinsulation)

Dipper



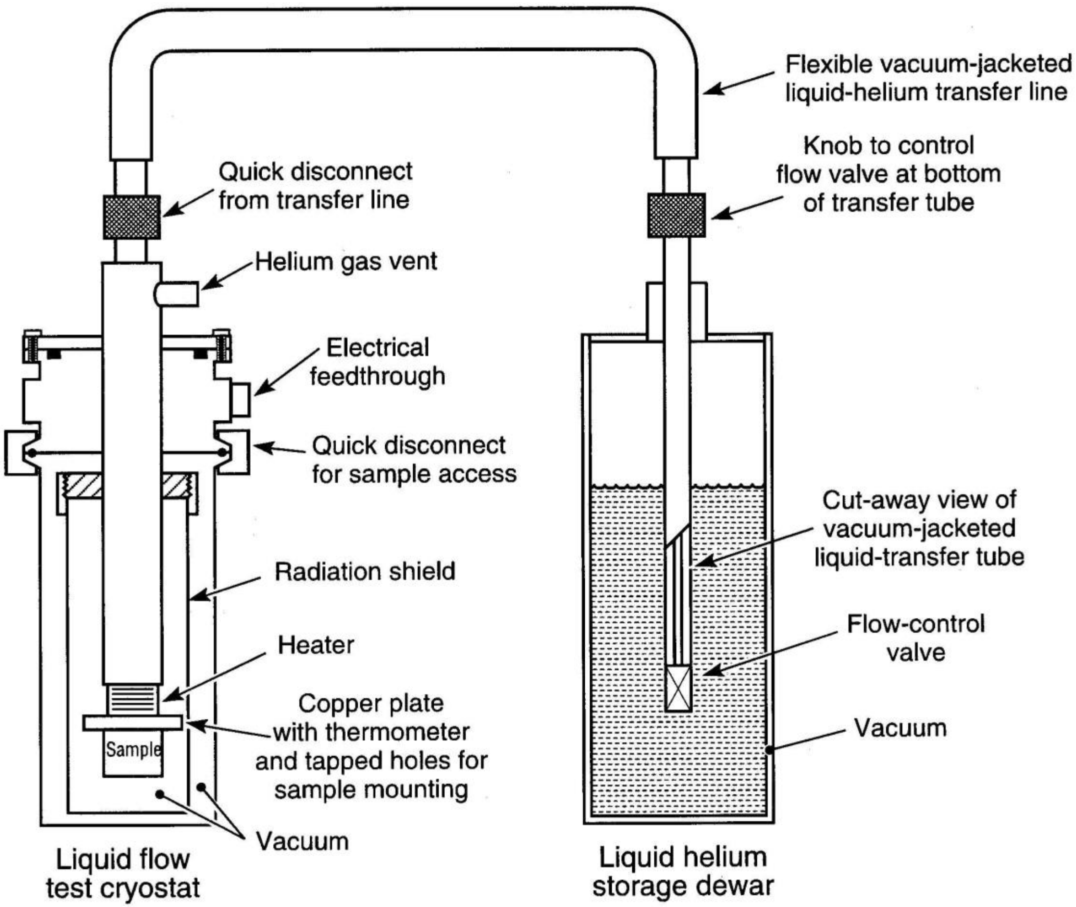
Dipper cryostat

Fast, cheap (and dirty)

Can use storage dewar

Poor temperature control (easy to improve somewhat)

Continuous flow cryostat



Cheap

Fast cooling

Relatively low He consumption

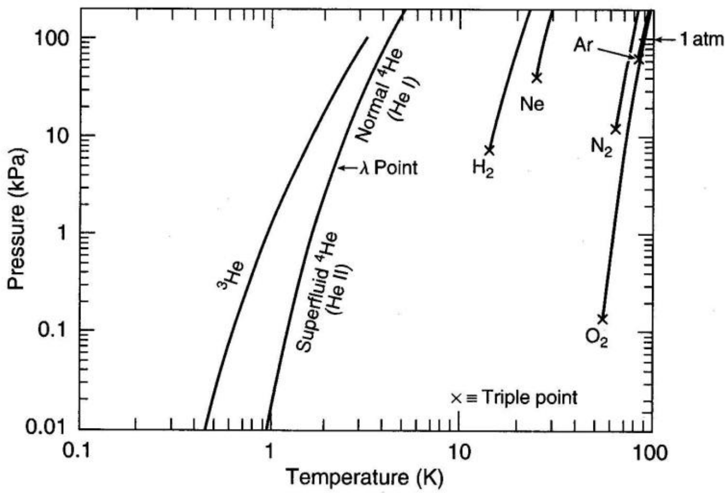
Relatively easy optical access

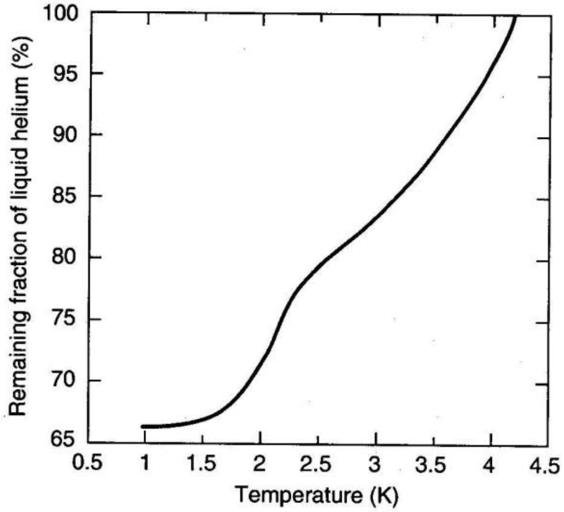
Base temperature?

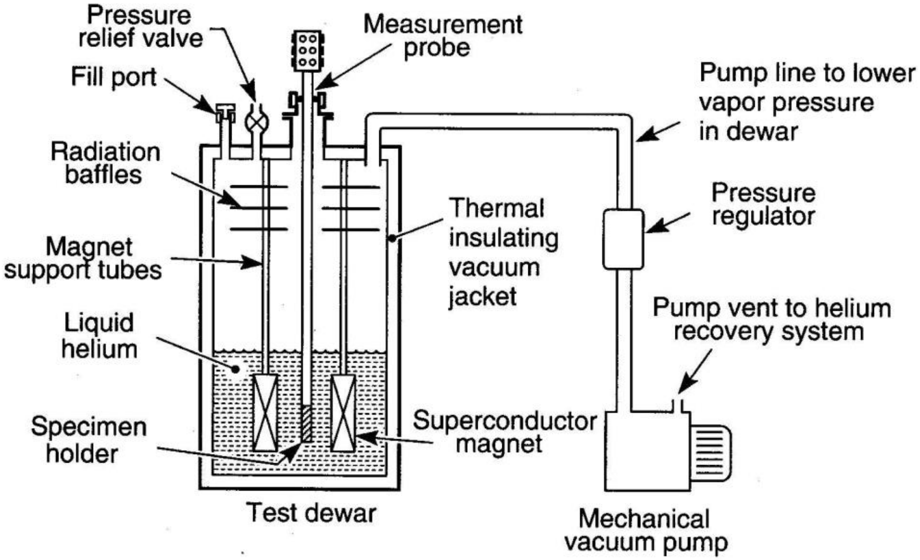
Mediocre temperature control

Hard to incorporate high field

Going below 4.2 K – ^{4}He



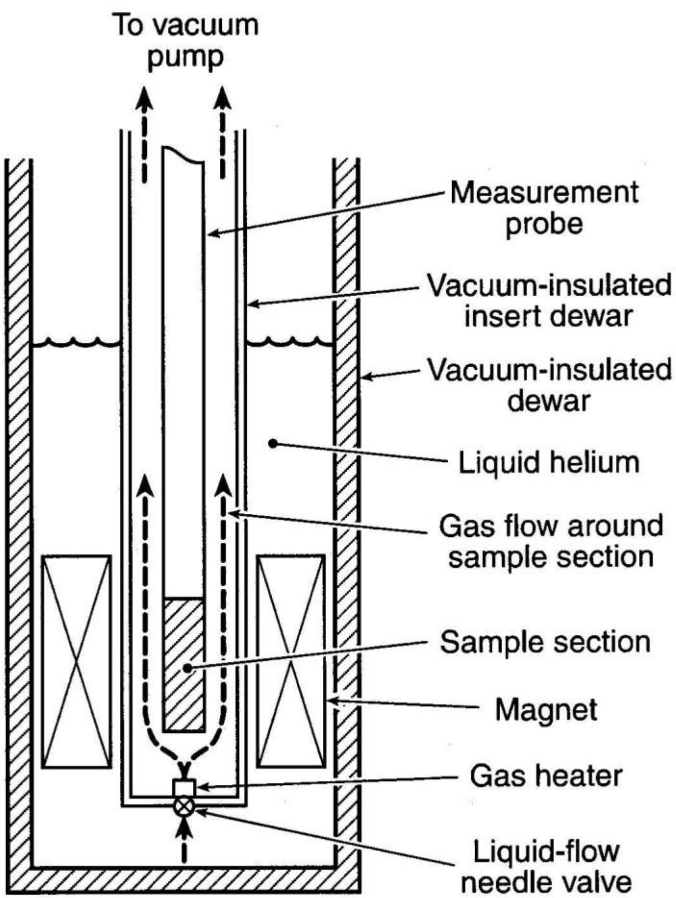




Usually not done like this nowadays.

Still might be considered for objects with large thermal mass or if experiment generates a lot of heat.

^{4}He cryostat with variable temperature insert



Workhorse for LT measurements

Good temperature control

Easy magnet accommodation

Allows for variety of sample holders

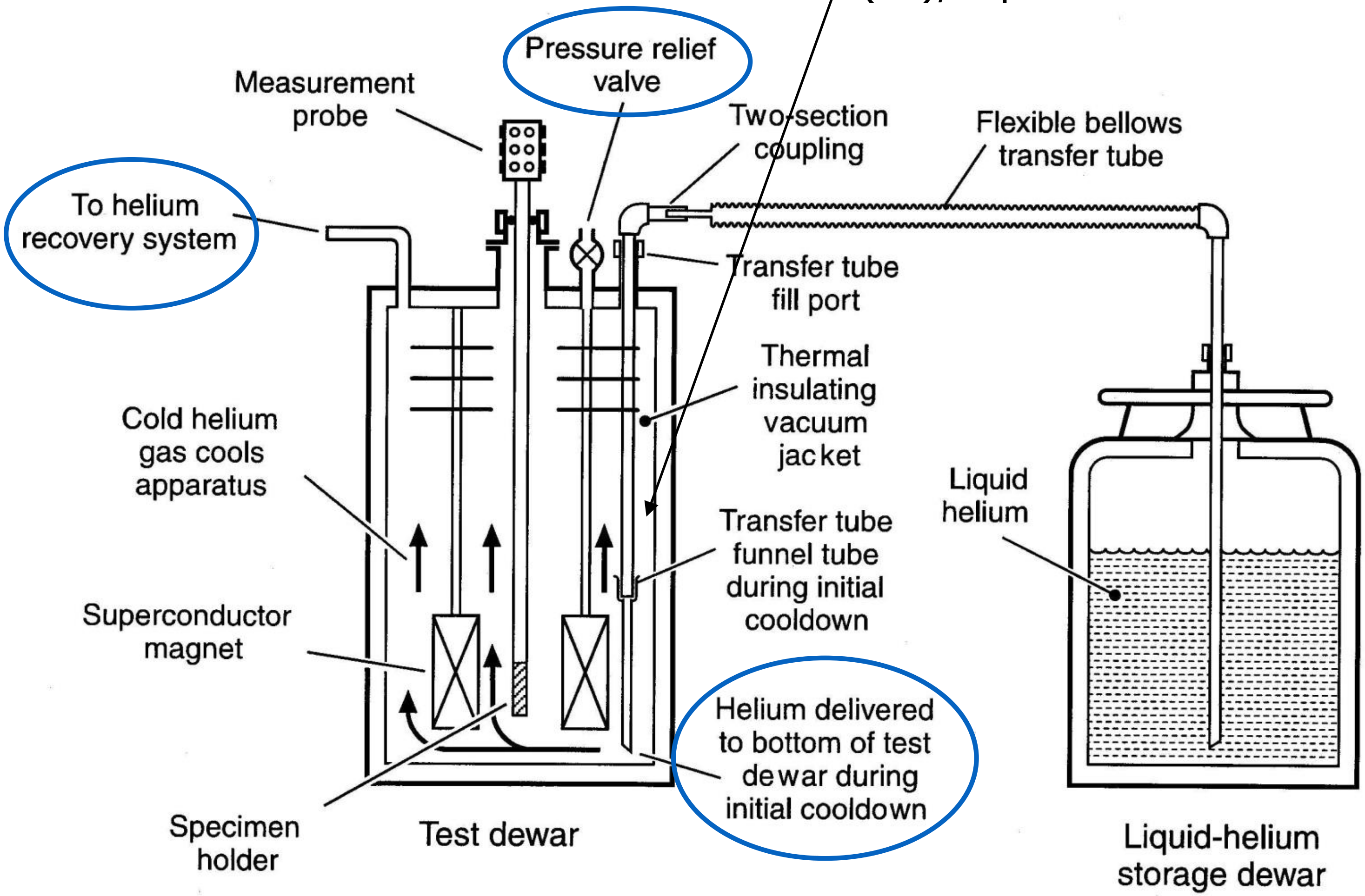
Not so cheap

Uses fair amount of cryogens

Needle valves and small impedances could be tricky

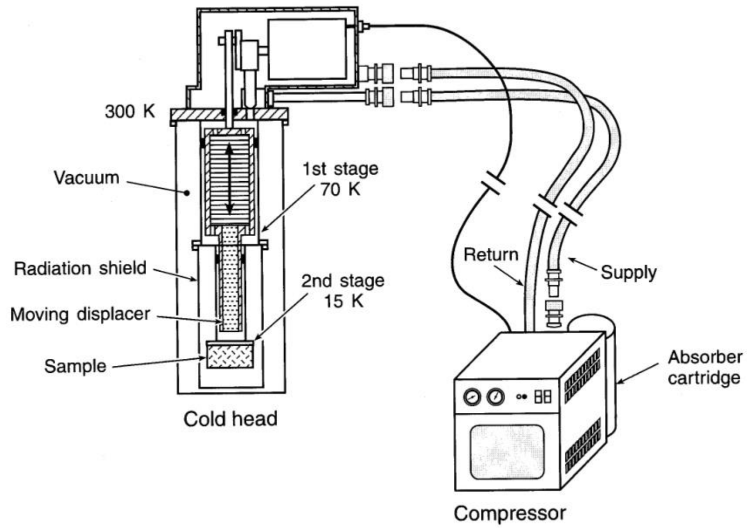
Helium transfer

He level detectors: acoustic, resistive (SC), capacitive.

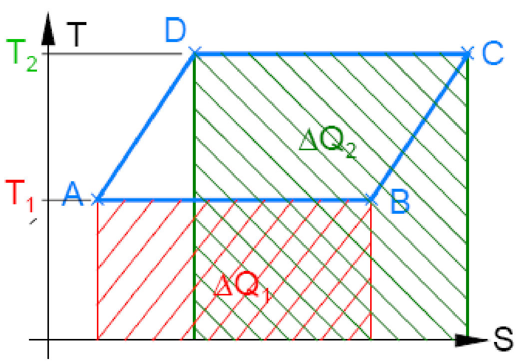


How to get there? Closed cycle refrigerators…

Gifford-McMahon (GM) or Pulse Tube (PT) cycle



To make a refrigeration cycle, need a substance, the entropy of which depends on some other variable than temperature



Pressure of gas: Compression/expansion cycle
Magnetization of solid: magnetic refr. cycle

ΔQ_1: heat absorbed at T_1
ΔQ_2: heat rejected at T_2

→ Refrigeration cycle A B C D

Closed cycle refrigerators GM vs PT

1) Price

In general, GM cryocoolers are less expensive than PT cryocoolers of similar cooling power.

2) Vibration

Both GM and PT cryocoolers are mechanical refrigerators that do have some level of vibration. Two locations exist on the cold head where vibration is important: The room temperature mounting flange and the 2nd stage.

3) Orientation

The performance of PT cryocoolers is orientation-dependent. PT cryocoolers only function properly when they are operated in a purely vertical orientation with the 2nd stage pointing down. GM cryocoolers will lose some cooling power when the cold head is not operated vertically, but the base temperature will not be affected. GM cryocoolers can operate in any orientation.

Closed cycle refrigerators

GOOD

Turn-key

Easy optical (etc…) access

No liquid cryogens

Small footprint (but remember compressor)

Sometimes reasonably priced

NOT SO GOOD

"Usually" base T > 4 K

Noise, vibrations

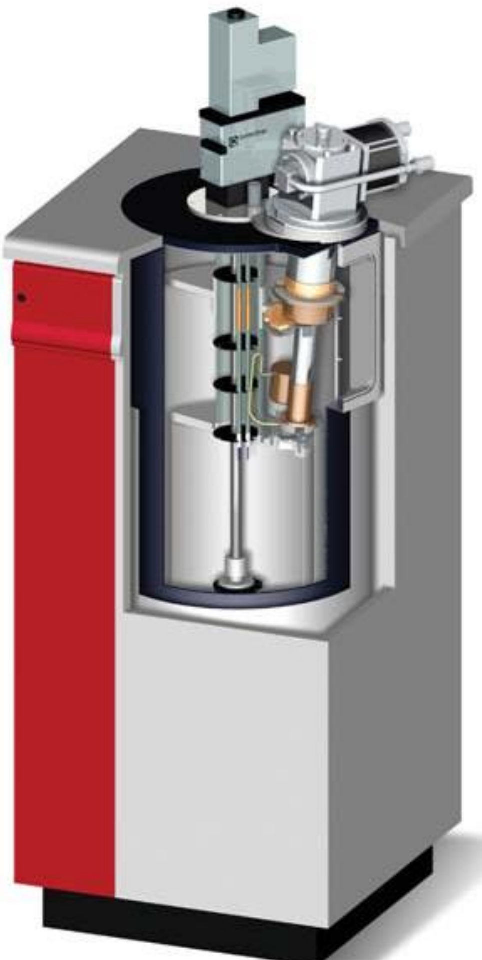
"Small" cooling power at low temperatures

Long term maintenance cost?

Use in high fields?

Somewhat difficult to tweak

Cryogen free systems



One can buy

14T/100mK measurements platform (combination of PT cryocooler and magnetic refrigeration stage)

SQUID magnetometer with installed liquefaction unit.

Presented as example only. We are not endorsing any particular manufacturer.

Cryogen free systems

One can buy

DRYICEEDEN

Cryogen Free Dilution Refrigerator Systems

	DRYICEEDEN 200	DRYICEEDEN 400
Cryostat and radiation shields	✓	✓
Insert and dilution refrigerator	✓	✓
Control wiring, temperature sensors and heaters	✓	✓
Pulse Tube cold head	✓	✓
Compressor, lines and tool kit	✓	✓
Vacuum lines	✓	✓
ICECUBE Automatic Gas Handling system	✓	✓
Integrated Temperature controller	✓	✓
Oil free Sealed pumps for circulation of mixture	✓	✓
Internal cryogenic cold trap	✓	✓
Optimised He3 / He4 mixture	✓	✓
PERFORMANCE		
Base Temperature	<10mK	<10mK
Cooling power at 100mK	200μW	400μW
Temperature stability	<±1mK	<±1mK
Initial Cool down to Base Temperature	24 hours	24 hours
Experimental Access: Line of Sight	3 x 50mm	3 x 50mm
Experimental Access: Non Line of Sight	3 x 50mm	3 x 50mm
Sample space size	300mm	300mm
Sample environment	Vacuum	Vacuum
Labview drivers and sequence controls	✓	✓
One common vacuum, no need for exchange gas	✓	✓
Vibration	100nm	100nm
Radiation Shield	50k al	50k al
	4k cu	4k cu
	Still cu	Still cu
Cold Head	1.0w	
Compressor	Water	
Circulation pumps	Turbo	Turbo
	ACP 20	ACP 40
He3 gas requirement	16 litres	18 litres
Integrated Lakeshore	Included	Included
Support Stand for the cryostat	Included	Included
OPTIONS		
Experimental wiring:	✓	✓
Flexible coaxial cables	✓	✓
Semi Rigid coaxial	✓	✓
Cryogenic Ribbon cables	✓	✓
Optical Fibres	✓	✓
Special, customer selected options	✓	✓
Superconducting magnet with HTS leads	✓	✓
Superconducting magnet power supply	✓	✓
Remote motor for PTR head	✓	✓
Sample rotators	✓	✓
Turbo molecular pump and lines for the OVC	✓	✓
Calibrated RuO2 sensor for the mixing chamber	✓	✓
Adaptions to fit specific room layouts	✓	✓
On-site installation and end user training	✓	✓
Additional sample access ports	✓	✓
Software / Single push button cool down	✓	✓
Circulating pumps mounted remotely from cryostat	If required	If required
Special support stands, including ceiling mounted	Available	Available

Presented as example only. We are not endorsing any particular manufacturer.

Safety

Temperature

Pressure

Energy (SC magnets)

Ice blocks

Relief valves

He level – magnet quench

Glass dewars

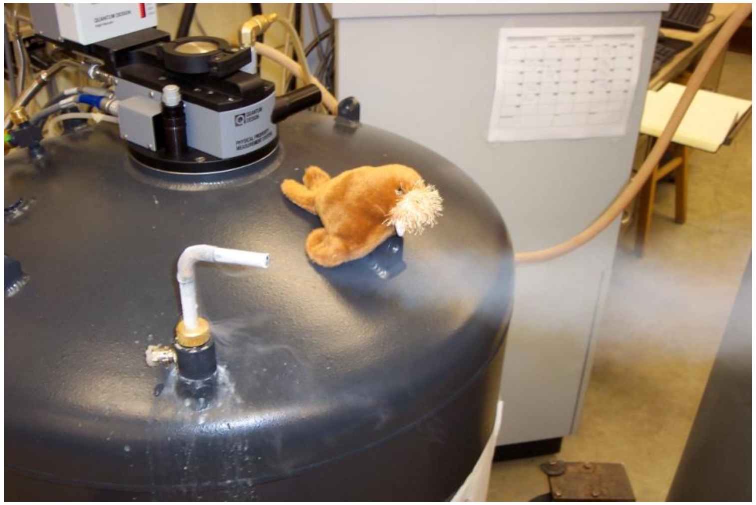
Liquid oxygen

Think

Learn

Use PPE (!?)

RECOVER!



Take Ames Laboratory "Safe use of cryogens" training

Reading materials

Matter and methods at low temperatures
Author: Frank Pobell; Springer, 2007

Experimental techniques in low-temperature physics
Author: Guy K. White; Clarendon Press, 1979

Experimental low-temperature physics
Author: Anthony Kent; American Institute of Physics, 1993

Experimental techniques in condensed matter physics at low temperatures
Author: Robert C Richardson; Eric N Smith Addison-Wesley Pub. Co., 1988

Experimental techniques for low-temperature measurements : cryostat design, material properties, and superconductor critical-current testing
Author: J. W. Ekin; Oxford University Press, 2006

Electrical resistivity and heat capacity

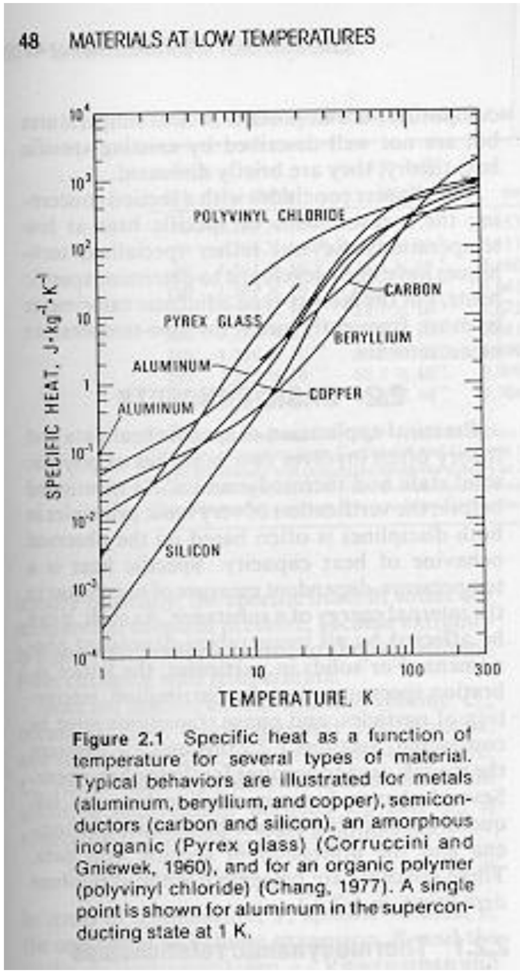
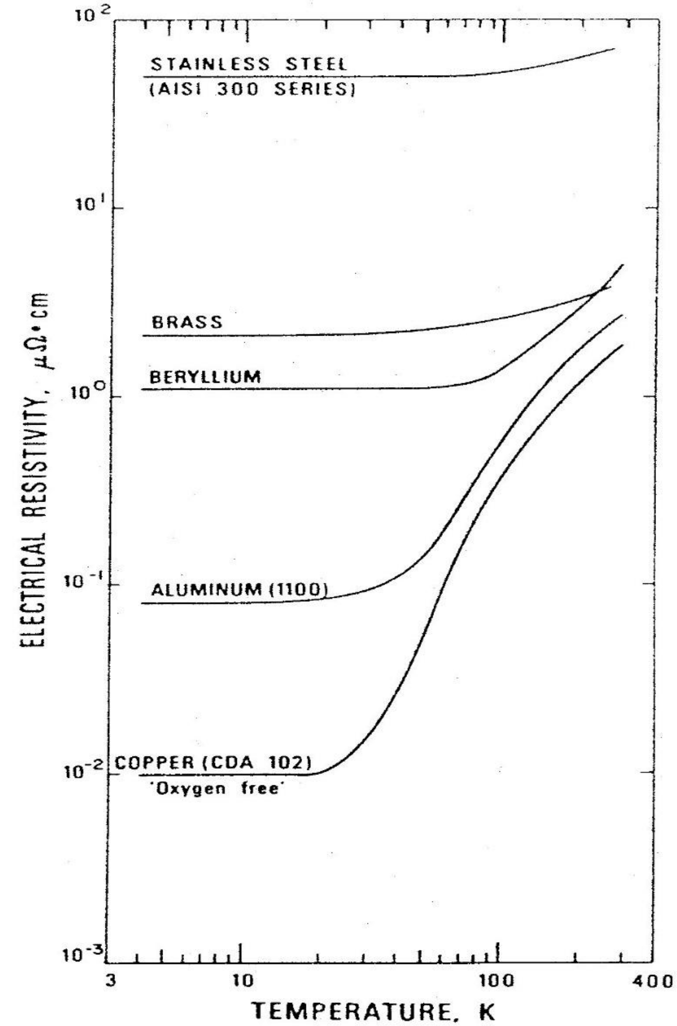


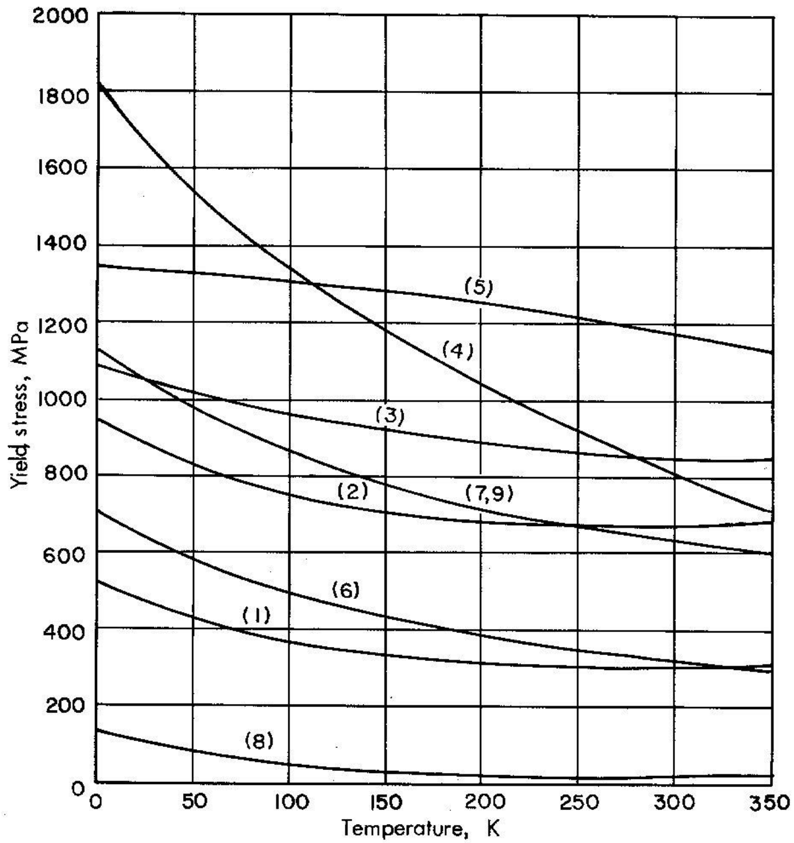
Figure 2.1 Specific heat as a function of temperature for several types of material. Typical behaviors are illustrated for metals (aluminum, beryllium, and copper), semiconductors (carbon and silicon), an amorphous inorganic (Pyrex glass) (Corruccini and Gniewek, 1960), and for an organic polymer (polyvinyl chloride) (Chang, 1977). A single point is shown for aluminum in the superconducting state at 1 K.



appendix

Yield strength of engineering materials

(1) 2024-T4 Aluminum
(2) Beryllium copper
(3) K-monel
(4) Titanium
(5) 304 Stainless steel
(6) C1020 Carbon steel
(7) 9% Nickel alloy steel
(8) Teflon
(9) Invar-36



appendix

Variation of Young's modulus with temperature

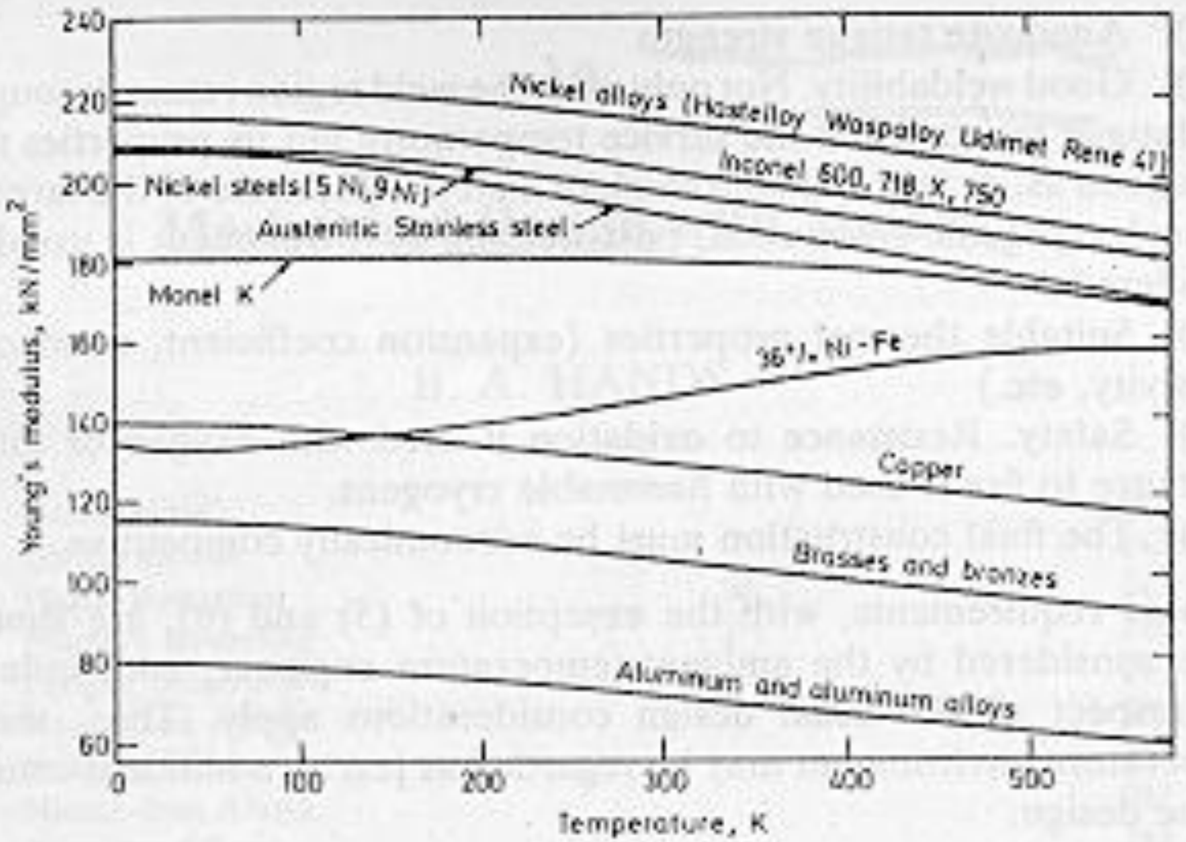
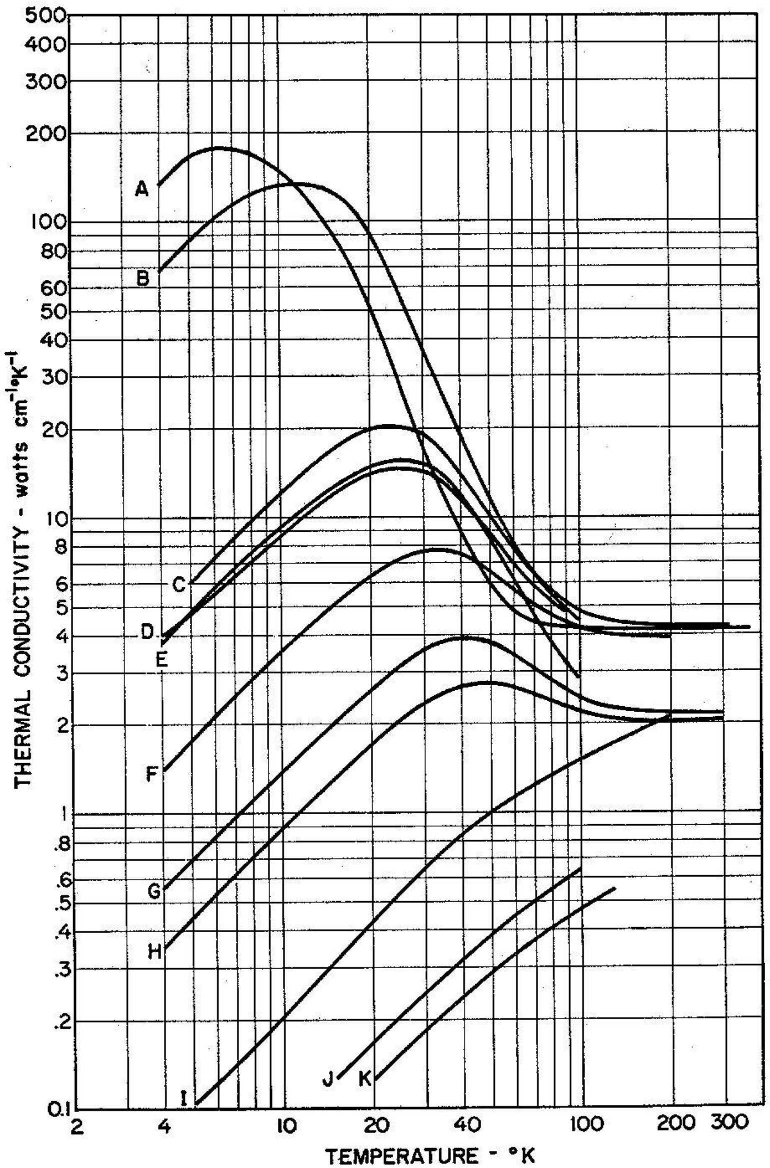


Fig. 10.1 Variation of Young's modulus with temperature. (From Ledbetter [10.1].)

appendix

Thermal conductivity - high

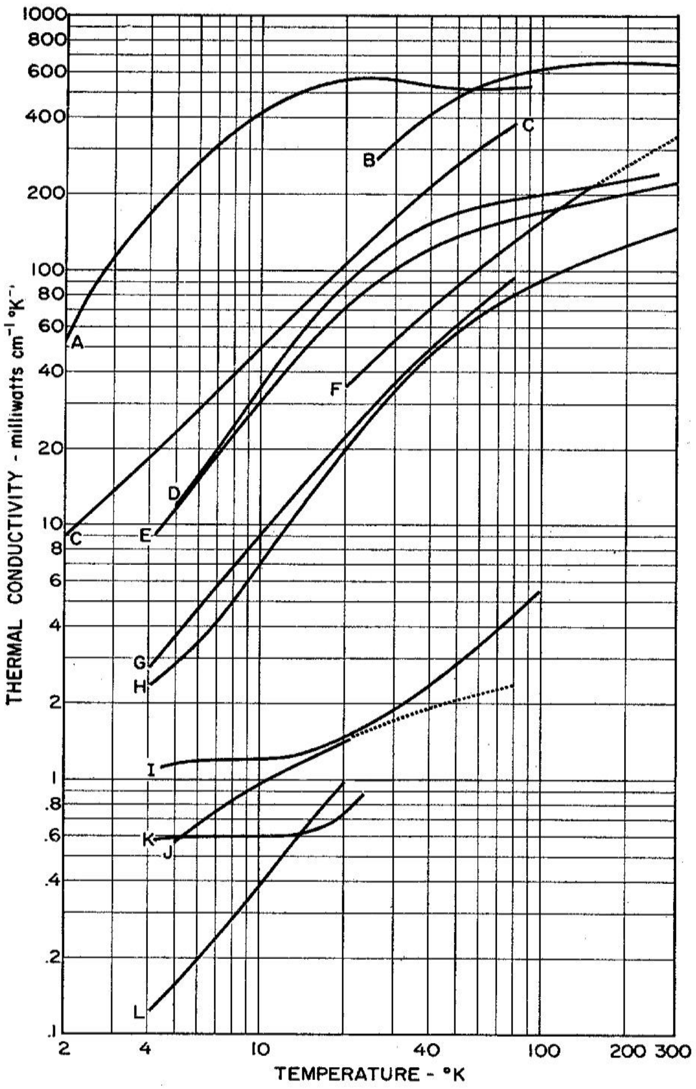


A: silver 99.999% pure,
B: copper (OFHC),
C: coalesced copper,
D: electrolytic tough pitch,
E: single crystal aluminum,
F: machining copper,
G: aluminum 1100 F,
H: aluminum 6063-T5,
I: phosphorus deoxidized copper,
J: aluminum 2024-T4,
K: brass

appendix

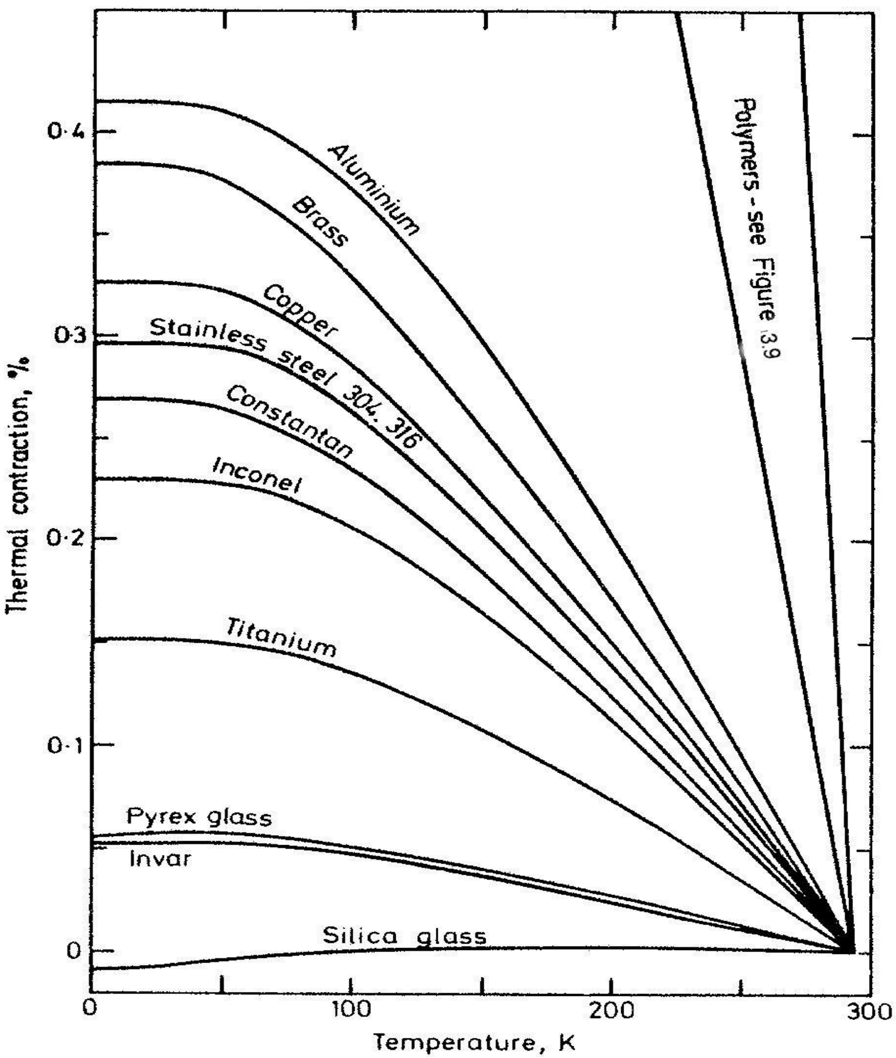
Thermal conductivity - low

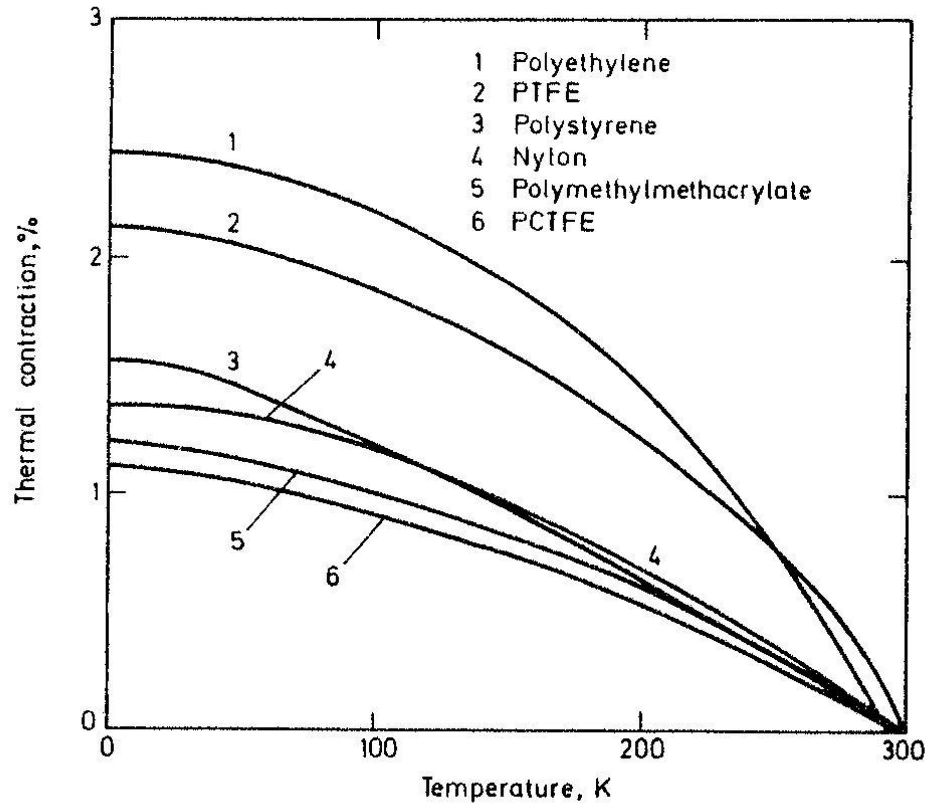
A; 50-50 Pb-Sn solder
B; steel, SAE 1020
C; beryllum copper
D; constantan
E; monel
F; silicon bronze
G; inconnel
H; 347 stainless steel
I; fused quartz
J; teflon (PTFE)
K; polymethylmethacrylate (PMMA)
L; nylon



appendix

Thermal contraction





appendix