



BAYERISCHE AKADEMIE DER WISSENSCHAFTEN Technische Universität München

Superconductivity and Low Temperature Physics II



Lecture Notes Summer Semester 2022

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Chapter 3

Cryogenic Techniques Generation and Measurement of Low Temperatures



Contents Part II: Quantum Transport in Nanostructures

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- III.2.3 Secondary Thermometers



Chapter III: Cryogenic Techniques

Literature:

- 1. Tieftemperaturphysik Enss, Hunklinger Springer (2000)
- Matter and Methods at Low Temperatures
 F. Pobell
 Springer, 2nd edition (1996)
- **3. Experimental Low-Temperature Physics Anthony Kent** American Institute of Physics (1993)
- Cryogenic Systems
 Randall F. Barron
 Oxford University Press, Oxford (1985)

III.1 Generation of Low Temperatures





lowest temperatures (in solid-state systems)

low temperature record for nuclear spin system:

- experimental setup according to Tauno Knuuttila (2000)
- lowest temperature: about 100 pK by demagnetization of Rhodium nuclei (*"temperature of nuclear spins"*)

PhD Thesis, Helsinki University of Technology (Espoo, Finland)

– problem:

spin temperature cannot be transferred to lattice of solid





- generation of low temperatures by using cryo-liquids
 - **19th century:** liquefaction of various gases by pressure except for "permanent gases" (O₂, H₂, He)
 - **1877:** liquefaction of O₂ by thermal expansion
 - (L. Cailletet, C.R. Acad. Sci. Paris 85, 1213 (1877); R. Pictet, C.R. Acad. Sci. Paris 85, 1214 (1877))
 - **1884:** liquefaction of H₂ (precooling with liquid O₂)

(K. Olszewski, Ann. Phys. u. Chem. 31, 58 (1887))

- **1898:** significant amounts of LH₂ for physical experiments (J. Dewar, Proc. R. Inst. Gt. Br. 15, 815 (1898))
- **1908:** liquefaction of last "permanent gas" He by Kamerlingh Onnes (H. Kammerlingh Onnes, Leiden Commun. 105, Proc. Roy. Acad. Sci. Amsterdam 11, 168 (1908))
- **1922:** Kammerlingh Onnes reaches T < 1 K

(H. Kammerlingh Onnes, Leiden Commun. 159, Trans. Faraday Soc. 18 (1922))

- **1926:** adiabatic demagnetization of electron spins in paramagnetic salts by Debye and independently (P. Debye, Ann. Phys. 81, 1154 (1926)
- **1927:** by Giauque (W.F. Giauque, J. Am. Chem. Soc. 49, 1864 (1927)
- since 1950s: ³He available ³He cryostat ³He-⁴He dilution refrigerator



Sir **James Dewar**, (1842-1923)



Peter J. Debye 1884 - 1966



Heike Kammerlingh Onnes (1853 – 1926) Nobelpreis für Physik: 1913



- **1868**offer of chair at the
Polytechnische Schule München (now TUM)
- 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)





Carl Paul Gottfried von Linde

* 11. Juni 1842 in Berndorf, Oberfranken+ 16. November 1934 in Munich

- supplementary materia

| - 2022)

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III.1.1 Introduction





III.1.1 Introduction

• 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



- cooling techniques
 - expansion of an ideal gas
 - expansion machine
 - regenerative machine
 - work against outside world
 - expansion of a real gas
 - Joule Thomson cooler
 - work against internal interactions
 - evaporation of a real gas:
 - work against internal interactions
 - dilution cooling (³He/⁴He)
 - work against internal interactions
 - adiabatic demagnetization (electronic/nuclear moments)
 - work against magnetic ordering



• liquefaction of gases

three useful methods:

- 1. direct liquefaction by *isothermal compression*
- letting the gas perform *work against external forces* at the expense of its internal energy
 - ➔ cooling and eventual liquefaction
- 3. making the gas perform *work against its own internal forces* by Joule-

Kelvin or Joule-Thomson expansion

→ cooling and eventual liquefaction

III.1.1 Introduction

- direct liquefaction of gases by isothermal compression
 - \rightarrow starting temperature must be smaller than critical temperature T_c





critical temperatures T_c in K of selected liquid cryogens

ammonia (NH ₃)	406
O ₂	154.5
N ₂	126
H ₂	33.2
⁴ He	5.2
³ He	3.32

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properties of cryogenic liquids



Substance	T _b [K]	$T_m[K]$	T _{tr} [K]	P _{tr} [bar]	T _c [K]	P _c [bar]
@ 1 bar						
H ₂ O	373.15	273.15	273.16	0.06	647.3	220
Xe	165.1	161.3	161.4	0.82	289.8	58.9
Kr	119.9	115.8	114.9	0.73	209.4	54.9
0,	90.2	54.4	54.36	0.016	154.3	50.4
Ar	87.3	83.8	83.81	0.67	150.9	48.7
N ₂	77.4	63.3	63.15	0.12	126.0	33.9
Ne	27.1	24.5	24.56	0.43	44.5	27.2
D ₂	23.7	18.7	18.72	0.17	38.3	16.6
H ₂	20.3	14.0	13.80	0.07	33.3	13.0
⁴ He	4.21				5.20	2.28
³ He	3.19				3.32	1.16



Temperature

Chapter 3/RG 14



- direct liquefaction of gases by expansion (Joule-Thomson-Effect)
 - -> starting temperature must be smaller than inversion temperature (definition follows later)

cryogen	boiling point [K]	liquefaction	latent heat [kJ/liter]	inversion temp. [K]
oxygen	90.2	1877: Cailletet and Pictet	240	762
nitrogen	77.3	1883: Wroblewski and Olszewski	160	625
hydrogen	20.4	1898: Dewar	30	203
⁴Helium	4.2	1908: Onnes	2.6	43.2
³ Helium	3.2		0.5	-

- > liquid oxygen and hydrogen have potential hazards
- liquid nitrogen and ⁴He are the most widely used cryogens
- > liquid ³He is very expensive



liquefaction of gases by performance of external work



gas molecules are reflected at the moving piston-surface:

incoming: laboratory system: v_M piston system: $v_M - v_K$ outgoing: piston system: $-(v_M - v_K)$ laboratory system: $-(v_M - v_K) + v_K = 2v_K - v_M = v'_M$

i.e.: $|v'_{\rm M}| = v_{\rm M} - 2v_{\rm K}$ molecule is slower, i.e. colder

average momentum transfer per time to piston = force, *force* · *distance* = *work*

 \rightarrow external work at the expense of internal energy \rightarrow cooling



- liquefaction of gases by performance of external work
 - in practice,
 - 1. gas is **isothermally compressed** (involves removing of heat corresponding to work done by gas)
 - 2. and then allowed to expand adiabatically (resulting in cooling)
 - 1st law of thermodynamics $dU = dQ + dW = dQ pdV = C_V dT$
 - ➤ adiabatic expansion: dQ = 0 $\Rightarrow dQ = C_V dT + p dV = 0$
 - ➢ for ideal gas: pV = RT⇒ pdV + Vdp = RdT
 - > elimination of dT $\Rightarrow 0 = C_V \left(\frac{pdV + Vdp}{R}\right) + pdV = \frac{dV}{V}(C_V + R) + C_V \frac{dp}{p}$

$$\Rightarrow \kappa \left(\frac{dV}{V}\right) + \frac{dp}{p} = 0 \qquad \text{with } \kappa = \frac{(C_V + R)}{C_V}$$

 \succ integration yields κ ln V + ln p = const. ⇒ pV^κ = const.

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III.1.1 Introduction

- Carnot process:
 - counterclockwise:
 - clockwise:

heat pump (conversion of mechanical work into heat)
heat engine (conversion of heat into mechanical work)

• *pV* diagram: expansion cooling: adiabats $(pV^{\kappa} = const, \ dQ = 0$ $\kappa = \frac{c_p}{c_V} > 1)$ heat exchange: isotherms $(pV = const, \ dT = 0)$ work per cycle: $W = \oint pdV = area$ • *efficiency:* $\frac{W}{\Delta Q} \equiv \eta = \frac{\Delta T}{T_{warm}}$



thermodynamic definition of temperature

• Carnot process: technologically difficult to realize

→ better: gas circulation, compressor and expansion machine are spatially separated



- e.g. liquefaction of air:
 - condensation on cold head
 - distillation in separation columns

$\geq N_2$	(77.4 K)	cooling
≻Ar	(87.3 K)	inert gas
≻ O ₂	(90.2 K)	welding

• temperature reduction:

$$T_2 = T_1 \left(\frac{p_2}{p_1}\right)^{(\kappa-1)/\kappa} \qquad \kappa = C_p / C_V \ (= 5/3 \text{ for He})$$

ideal, single-atomic gas: $\kappa = 1 + \left(\frac{2}{f}\right)$



• efficiency:

$$\eta = 1 - \frac{T_2}{T_1} = 1 - \left(\frac{p_2}{p_1}\right)^{(\kappa-1)/\kappa}$$

→ expansion from $p_1 = 100$ bar to $p_2 = 1$ bar results in $T_2 = 50$ K @ $T_1 = 300$ K $T_2 = 8$ K can be reached in a two-stage cycle

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Vq out

P-v Diagram

• heat pumps: heating and refrigerating machines



- heat pump: heat is generated by mechanical work

- efficiency:

$$h = \frac{\text{generated heat } @ T = T_1}{\text{performed work}} = \frac{-\Delta Q_1}{\Delta W}$$

- refrigerating machine: removing heat (generating cold) by mechanical work

- efficiency:

$$k = \frac{\text{removed heat } @ T = T_2}{\text{performed work}} = \frac{\Delta Q_2}{\Delta W} = \frac{\Delta W - \Delta Q_1}{\Delta W} = 1 - h$$

- ideal efficiency for reversible Carnot process:

$$h_c = \frac{1}{\eta_c} = \frac{T_1}{T_1 - T_2} \ge 1$$

(increases with decreasing temperature difference
$$T_1 - T_2$$
)

$$k_{c} = 1 - h_{c} = \frac{T_{2}}{T_{1} - T_{2}} \ge 0$$

(decreases with increasing temperature difference $T_1 - T_2$)

• application examples: heating and refrigerating machines



schematic diagram of a heat pump's vapor-compression refrigeration cycle: 1) condenser, 2) expansion valve, 3) evaporator, 4) compressor

- realizations of expansion machines:
 - piston-cylinder machine similar to automobile engine crankshaft, camshaft, valve
 - cooling turbine ⇒ commercially relevant higher efficiency for larger throughput



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- conclusions
 - expansion machines are technologically simple
 - multi-stage arrangements for lower temperatures

 \Rightarrow almost down to 4.2 K

• but:

 \Rightarrow *efficiency* only acceptable for cooling turbines

• no direct liquefaction of gas (mechanical problems)

 \Rightarrow liquefaction by Joule-Thomson stage

- for small-scale facilities:
 - ⇒ *regenerative machines* better suited





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- regenerator replaces heat exchanger
- \Rightarrow column with staple of fine metal meshes (Cu, Pb)
- → low flow resistance
 → high heat capacity
 → low longitudinal heat conductivity

- alternating gas flow:
 cold gas upward
 warm gas downward
- $\Rightarrow cooling of meshes \\\Rightarrow cooling of gas$
- used in *Stirling process*



cold gained in step 2 \rightarrow 3 has to be stored and provided in step 4 \rightarrow 1

Stirling machine (clockwise, heat engine)

Kompression



3 - 4Wärmebad der höheren 4 - 11 - 22 - 3Temperatur | Verdrängerkolben Regenerator Arbeitsmedium Wärmebad der niederen Temperatur, Arbeitskolben isochore isotherme isotherme isochore

Expansion

Erwärmung

periodic expansion and compression of gas along two isotherms and two isochors

- − 1 → 2: isothermal expansion, Q_{12} is added
- $2 \rightarrow 3$: isochoric cooling, Q_{23} is removed
- $3 \rightarrow 4$: isothermal compression, Q_{34} is removed
- $4 \rightarrow 1$: isochoric warming, Q_{41} is added
- for isochoric steps there is no mechanical work $Q_{23} = -Q_{41} = C_V \Delta T$
- goal: intermediate storage of Q₂₃ in regenerator to be able to add it again in step 4 → 1
 → use of two pistons with phase shift



Abkühlung

• (beta) Stirling machine



power piston (dark grey) has compressed the gas, the displacer piston (light grey) has moved so that most of the gas is adjacent to the hot heat exchanger



displacer piston now moves, shunting the gas to the cold end of the cylinder.



cooled gas is now compressed by the flywheel momentum. This takes less energy, since when it is cooled its pressure dropped.

heated gas

increases in

pressure and

pushes the power

piston to the

farthest limit of the

power stroke.

• (alpha) Stirling machine











Gifford-McMahon machine

uses compressor with switching valve instead of piston

(counterclockwise, heat pump)



heat pump (conversion of mechanical work into heat)





- isochoric cooling: 2.
- expansion in cylinder: $4 \rightarrow 3$ 3.
- isochoric regeneration: $3 \rightarrow 2$ 4.
- warm compression: 5.

• Gifford-McMahon cycle





two-stage cryocooler



- Pulse Tube Refrigerator
 - the pulse tube refrigerator (PTR) or pulse tube cryocooler is based on the operation principle of Stirling coolers
 - PTR is made without moving parts in the low temperature part (in contrast with other cryocoolers, e.g. Stirling cryocooler and Gifford-McMahon cooler)
 - compact design possible \rightarrow suitable for a wide variety of applications
 - minimum temperature about 2.5 K (with ⁴He) and 1.3 K (with ³He)

applications:

- industrial applications such as semiconductor fabrication (e.g. cryopumps)
- cooling of infrared sensors
- cooling of astronomical detectors (e.g. Atacama Cosmology Telescope or the QUBIC experiment an interferometer for cosmology studies)
- precoolers of dilution refrigerators
 - Kurt Uhlig (WMI), "Dry" dilution refrigerator with pulse-tube precooling, Cryogenics 44, (2004), pp. 53–57
- suggested to be used to liquefy oxygen on Mars







motion of gas volume element equivalent to motion of "displacer piston"

90° phase shift between motion of "displacer piston" and "work piston" realized by buffer volume

90° phase shift required for finite heat transport

coldest spot between regenerator and pulse tube

• Pulse – Tube – Refrigerator (PTR) (realizations)





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pulse tube refrigerator for studies of liquefying oxygen on Mars (580 mm total length)

- supplementary material

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III.1.3 Regenerative Machines

- Pulse Tube Refrigerator: summary
 - presently very active development
 - no moving parts at low temperatures

 \Rightarrow long endurance

mobile base stations and satellite applications

(e.g. for superconductive microwave filters)

- almost no vibrations
- efficiency lower than for displacer system
- only one simpler method:
 - \Rightarrow *Joule-Thomson* cooling









William Thomson (Lord Kelvin)

Born: 26 June 1824, Belfast, Northern Ireland Died: 17 December 1907, Netherhall, Largs Ayrshire, Scotland

- Joule-Thompson process
 - gas performs work against its own internal attractive forces
 - working medium/gas (V_1) flows through impedance and expands to V_2



$$U_2 - U_1 = 0 \text{ (adiabatic)}$$
1st law of thermodynamics:
$$\Delta U = \Delta Q + \Delta W - \int_0^{V_2} p_2 dV_2 - \int_{V_1}^0 p_1 dV_1 = -p_2 V_2 + p_1 V_1$$

 $\Rightarrow U_2 + p_2 V_2 = U_1 + p_1 V_1$

this means: process with **constant enthapy**: $H \equiv U + pV = const$.

- for ideal gas: $p_1V_1 = p_2V_2$ and hence $U_1 = U_2$, respectively $T_1 = T_2 \rightarrow no$ cooling !!

• Joule-Thompson process: real gas

transformation of gas into liquid on decreasing T and (or) increasing p due to work against attractive interaction between the molecules

 weak long-range attraction: tends to keep molecules closer together, same effect as additional compression of the gas

$$p_{\rm eff} = p + \frac{a}{V^2}$$

a is a measure of the long-range attraction

- strong short-range repulsion:
 molecules are rigid:
 - $p \rightarrow \infty$ as soon as the molecules "touch" each other.

$$V_{\rm eff} = V - k$$

b ($\approx 4\pi\sigma^3/3$): "excluded volume" per particle

- Van der Waals equation:

$$\left(p + \frac{a}{V_m^2}\right)(V_m - b) = RT$$



expansion (decrease of pressure): low pressure: attraction costs work ⇒ cooling of gas high pressure: repulsion provides work ⇒ heating of gas





• Joule-Thompson process: more detailed analysis of isenthalpic expansion

$$\Delta H = \left(\frac{\partial H}{\partial T}\right)_p \Delta T + \left(\frac{\partial H}{\partial p}\right)_T \Delta p = 0$$

with
$$\left(\frac{\partial H}{\partial T}\right)_p = C_p \implies C_p \Delta T = -\left(\frac{\partial H}{\partial p}\right)_T \Delta p \implies -\frac{1}{C_p} \left(\frac{\partial H}{\partial p}\right)_T = \left(\frac{\Delta T}{\Delta p}\right)_H \equiv \mu_{\text{JT}}$$

Joule-Thomson coefficient

 $\mu_{\rm JT} > 0$: cooling on expansion $\mu_{\rm JT} < 0$: heating on expansion

with
$$\Delta H = T\Delta S + V\Delta p \Rightarrow \left(\frac{\partial H}{\partial p}\right)_T = T\left(\frac{\partial S}{\partial p}\right)_T + V$$

and
$$\left(\frac{\partial S}{\partial p}\right)_T = -\left(\frac{\partial V}{\partial T}\right)_p$$

$$\implies \mu_{\rm JT} = \left(\frac{\Delta T}{\Delta p}\right)_{H} = -\frac{1}{C_p} \left(\frac{\partial H}{\partial p}\right)_{T} = \frac{1}{C_p} \left(T \left(\frac{\partial V}{\partial T}\right)_{p} - V\right)$$

• Joule-Thompson process: more detailed analysis of isenthalpic expansion – ideal gas



Joule-Thompson process: more detailed analysis of isenthalpic expansion – real gas

- for real gas:
$$\left(p + \frac{a}{V_m^2}\right)(V_m - b) = RT \implies H(T, p) = U + pV = \frac{5}{2}Nk_BT + \Delta U(p, T) \neq const.$$

- at *low densities:* we can use *approximation* $p \gg \frac{a}{V^2}$, $V \gg b$ and obtain

$$pV + \frac{a}{V} - pb = RT$$
, $\left| \frac{\partial}{\partial T} (\dots)_p \right| \longrightarrow p \left(\frac{\partial V}{\partial T} \right)_p - \frac{a}{V^2} \left(\frac{\partial V}{\partial T} \right)_p = R \Rightarrow \left(\frac{\partial V}{\partial T} \right)_p = \frac{R}{p - \frac{a}{V^2}} \left(\frac{\partial V}{\partial T} \right)_p$

insert into

$$\mu_{\rm JT} = \left(\frac{\Delta T}{\Delta p}\right)_{H} = -\frac{1}{C_p} \left(\frac{\partial H}{\partial p}\right)_{T} = \frac{1}{C_p} \left(T \left(\frac{\partial V}{\partial T}\right)_{p} - V\right)$$

$$\implies \mu_{\rm JT} = \left(\frac{\Delta T}{\Delta p}\right)_{H} \approx \frac{1}{C_{P}} \left(2\frac{a}{RT} - b\right)$$

 $\mu_{\text{JT}} > 0$ for $T < 2a/bR \Rightarrow$ cooling on expansion $\mu_{\text{IT}} < 0$ for $T > 2a/bR \Rightarrow$ heating on expansion

inversion temperature:

$$T_{\rm inv} = \frac{2a}{bR}$$

• Joule-Thompson process: more detailed analysis of isenthalpic expansion – real gas



- Joule-Thompson process: more detailed analysis of isenthalpic expansion real gas
 - without "low density" approximation

$$\mu_{\rm JT} = \frac{(2a/RT)(1-b/V^2)-b}{C_p[1-(2a/VRT)(1-b/V)^2]}$$

low density **approximation** $p \gg \frac{a}{V^2}, V \gg b$: $\mu_{\text{JT}} \approx \frac{1}{C_P} \left(2 \frac{a}{RT} - b \right)$

> **inversion curve:** points where $\mu_{JT} = 0$: for vdW gas: (2a/RT)(1 - b/V)2 = b

inversion temperature:

$$T_{\rm inv} = \frac{2a}{bR} \left(1 - \frac{b}{V}\right)^2$$

equation of state gives $T_{inv}(p, T)$

maximum inversion temperature: $T_{inv} = \frac{2a}{bR}$





 $\mu_{\rm JT} \propto$



• Joule-Thompson coefficient for nitrogen





Joule-Thomson coefficients for various gases at atmospheric pressure

• Joule-Thompson cooling: inversion temperature

gas	maximum inversion temperature [K]
Helium-3	(23)
Helium-4	45
Hydrogen	205
Neon	250
Nitrogen	621
Air	603
Carbon monoxide	652
Argon	794
Oxygen	761
Methane	939
Carbon dioxide	1500
Ammonia	1994

vdW gas can be liquefied only for $T < T_{inv}$!!!

• "Linde process"



- gas is cooled by JT-expansion until liquid drops out the impedance
- patent application by Carl von Linde on May 12, 1903 (liquefaction of oxygen)





Carl von Linde (1842 – 1934)

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Helium liquefaction by Joule-Thompson cooling



schematics of a Helium liquefier

III.1.5 Summary

106 NG Air liquefaction Accelerators 105 & Fusion Mixed-gas JT Transmission, **REFRIGERATION POWER (W)** lines (Commercial) Stirling 104 Turbo-Brayton/Claude 1 TJ Large ervorape Transformers size 10³ **Turbo-Brayton** GJ (Special) Mid-size Cryopumps Gifford-10² SMES Motors McMahon GM+JT Bearings FCI 101 Maglev Cryosurgery MRI Wireless Micro-SMES 10° Low frequency Magnetic TE NbN 10-1 Electronics Elect. /Space SQUIDs **SQUIDs Pulse Tubes** radiators 10-2 He₅ H_2 10 50 100 300 JT JT N_2 TEMPERATURE (K) JT

application of cooling techniques in plane of refrigeration power versus temperature

III.1.5 Summary



efficiency of cryocoolers

WMI

III.1.5 Summary

Northrop Grumman's HEC cryocooler



specification for cryocooler:

- 1 Watt of cooling @ 80 K, rejecting heat at 300 K
- 10 year life
- 230 K to 340 K survival temperature
- survival of launch vibration (non-operating)
- low exported vibration
- high efficiency
- no maintenance possible → oil-free

Sumitomo Heavy Industries



Stirling cycle miniature cryocooler:

- lightweight cooler, ideal for cooling of sensors and other electronics when low power consumption is important
- mean time before failure of 24,000 hours
- cooling capacity of 1 W @ 80 K
- power consumption of only 55 W.

- supplementary material

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- physics of evaporation cooling
 - everyday experience: sweating, wind direction, cooling of coffee, ...
 - \Rightarrow moisten finger, evaporation cooling
 - microscopically:
 - evaporation: work required to overcome binding forces
 - \Rightarrow only the fastest molecules will do it
 - \Rightarrow high-energy particles are lost
 - \Rightarrow liquid cools down

- physics of evaporation cooling
 - limit of evaporation cooling: $k_{\rm B}T$ becomes too small compared to $\Delta H_{\rm vap}$ (heat of evaporation)
 - $-\Delta H_{\rm vap}$ should be small to reach large cooling power at low temperatures
 - numbers: about 1 K can be reached with ⁴He, about 0.3 K with ³He

 boiling point can be calculated by using the Clausius-Clapeyron equation, if heat of vaporization and the vapor pressure of the liquid at a certain temperature is known

• physics of evaporation cooling: Clausius-Clapeyron equation

$$\frac{dp}{dT} = \frac{S_{\text{gas}} - S_{\text{liquid}}}{V_{\text{gas}} - V_{\text{liquid}}} = \frac{\Delta H_{\text{vap}}}{(V_{\text{gas}} - V_{\text{liquid}})T} \simeq \frac{\Delta H_{\text{vap}}}{V_{\text{gas}}T}$$

 $\Delta H_{\rm vap}$: molar latent heat [J/mole] \approx 90 J/mole for ⁴He

- approximate expression using pV = RT (ideal gas):

$$\frac{dp}{p} = \frac{\Delta H_{\rm vap}}{RT^2} dT$$

- integration yields (assuming that ΔH_{vap} is constant over the considered T range):

$$\ln\left(\frac{p}{p_0}\right) = \frac{\Delta H_{\text{vap}}}{R} \left(\frac{1}{T_0} - \frac{1}{T}\right)$$

normal boiling temperature:

 $T_{\rm B}(p) = \left(\frac{1}{T_0} - \frac{R}{\Delta H_{\rm vap}} \ln\left(\frac{p}{p_0}\right)\right)^{-1}$ boiling temperature at p_0

pressure above liquid

boiling point corresponds to the temperature at which the vapor pressure of the liquid equals the surrounding environmental pressure







• physics of evaporation cooling: vapor pressure of some elements



Temperature °C



Pressure (Pa)

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- liquid ⁴He (cf. chapter I)
 - boson
 - liquid down to 0 K (@ 1 atm)
 - superfluid ⁴Helium at 2.17 K
 - Bose condensation: macroscopic number of atoms in ground state
 - very low viscosity
 - very high heat conduction
 - strange thermomechanical effects
 - creeping on vertical surfaces
 - vortex core with radius 0.8 Å @ 0.6K
 - explained by a two-fluid model
 - density 125 kg/m³





liquid helium cryostats

- LHe has small latent heat
 - → good thermal insulation by vacuum
 - → LHe container of poor thermal conductivity, glass or stainless steel
 - thermal radiation shield at liquid Nitrogen temperature to reduce black-body radiation
- bath cryostat sample is immersed in the LHe
 gas flow cryostat sample is located in cold He gas

• liquid helium container

narrow neck to minimize

- heating by radiation
- heating by thermal conduction

typical losses

1 liter of LHe / day







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• liquid helium bath cryostat





• liquid helium gas flow cryostat (1)





• liquid helium gas flow cryostat (2)





Possible Permanent Magnet Layout with ST-500 Microscopy Cryostat

- no liquid Nitrogen required
- radiation shield cooled by cold helium return gas

- liquid helium temperatures below 4.2 K
 - reduce the vapour pressure over bath of ⁴Helium
 - temperature down to 1.2 K
 at pumping power of 10 m³/h



\rightarrow up to 10 mW cooling power @ 1.2K

Clausius-Clapeyron equation: $\frac{dp}{dT} = \frac{\Delta H_{\text{vap}}}{(V_{\text{gas}} - V_{\text{liquid}})T} \simeq \frac{\Delta H_{\text{vap}}}{V_{\text{gas}}T} \implies T_B(p) = \left(\frac{1}{T_0} - \frac{R \ln(p/p_0)}{\Delta H_{\text{vap}}}\right)^{-1} \\ \ln \frac{p}{p_0} = \frac{\Delta H_{\text{vap}}}{R} \left(\frac{1}{T_0} - \frac{1}{T}\right)$ $\Delta H_{\text{vap}}: \text{ molar latent heat [J/mole]} \\ \simeq N_A \cdot E_{\text{binding}} \simeq 90 \text{ J/mole for }^4\text{He}$



- liquid ³He (cf. chapter I)
 - fermion
 - superfluid at 2.5mK
 - formation of weakly bound fermions: Cooper pairs
 - density 59 kg/m³
 - higher vapour pressure than ⁴He due to smaller latent heat:

 $\Delta H_{\rm vap} = 40$ J/mole \rightarrow cooling power \approx 80mW @ 1.2K and 10 m³/h pumping speed

0.3 K by pumping ³He vapour

some cm³

0.1 mW cooling power @ 0.3K

³He obtained by nuclear reactions

extremely expensive

1 liter of ³He gas costs more than US \$5.000 (2022)

• liquid ³He cryostat



latent heat of ³He: $\Delta H_{vap} = 40$ J/mole as compared to 90 J/mole for ⁴He \rightarrow larger cooling power

$$\dot{Q} = \dot{n}_{\rm gas} \Delta H_{\rm vap} \propto p \propto \exp\left(-\frac{\Delta H_{\rm vap}}{RT}\right)$$

≈ 80 mW @ 1.2 K for ³He as compared to ≈ 10 mW @ 1.2 K for ⁴He

⁴He impedance

- \leftarrow condensation of backflowing ³He gas
- flow restriction for condensed ³He

minimum temperature ≈ 300 mK cooling power ≈ 0.1 mW

III.1.7 ³He/⁴He Dilution Cooling

- ³He/⁴He dilution refrigeration (cf chapter I.6)
 - operation principle:
 - ightarrow remove ³He atoms from the dilute phase below $T_{
 m tri}~=~0.87~
 m K$
 - \rightarrow transport of ³He atoms across phase boundary to maintain equilibrium concentration
 - \rightarrow corresponds to evaporation of ³He from concentrated phase
 - \rightarrow cooling effect as the latent heat of evaporation is removed
 - for Fermi liquid:
 - $\rightarrow C_{V,\text{concentrated}} < C_{V,\text{diluted}} (x_3 = 0.065) \qquad (C_V \propto T/T_F, \ T_F \propto n_{3\text{He}}^{2/3})$

- with
$$U = Q = \int_0^T C_V(T') dT' = \frac{\gamma}{2} T^2$$
 we therefore obtain ($C_V = \gamma T, \gamma =$ Sommerfeld coefficient)

 $U_{\text{concentrated}}(T) < U_{\text{diluted}}(T)$

on transition across phase boundary:

$$dG = 0 = dU - TdS \Rightarrow dU = TdS = dQ$$

- → removal of heat $\Delta Q = T\Delta S = T [S_{dil}(T) S_{con}(T)]$ → cooling effect
- \rightarrow ³He/⁴He dilution refrigerator





concentrated

III.1.7 ³He/⁴He Dilution Cooling

- ³He/⁴He dilution refrigeration: cooling power (cf chapter I.6)
 - assumption: one mole of ³He crosses boundary between diluted and concentrated phase
 - removed heat: $\Delta Q = T \Delta S = T \left[S_{\text{dil}}(T) S_{\text{con}}(T) \right]$

- cooling power:
$$\Delta \dot{Q} = \left(\frac{\Delta Q}{n_{3He}}\right) \cdot \dot{n}_{3He} \Rightarrow \Delta \dot{Q} = \dot{n}_{3He} T \left[s_{dil}(T) - s_{con}(T)\right] \qquad S_{dil} = \frac{s_{dil}}{n_{3He}}, S_{con} = \frac{s_{con}}{n_{3He}}$$

since there is no volume change:

$$dQ = dU = TdS = C_V dT, \qquad U(T) = \int_0^1 C_V dT', \qquad S(T) = \int_0^1 \frac{C_V}{T'} dT'$$

$$\Rightarrow \text{ with } C_{\text{mol}} = \frac{\pi^2 N_A k_B^2 T}{2\varepsilon_F} \text{ and } \varepsilon_F = \frac{\hbar^2}{2m_{3\text{He}}^*} \left(\frac{3\pi^2 N_{3\text{He}}}{V}\right)^{2/3} = \frac{\hbar^2}{2m_{3\text{He}}^*} \left(\frac{3\pi^2 N_A}{V_{\text{mol}}}\right)^{2/3} \text{ (standard expressions for Fermi liquid)}$$

we obtain the entropy
$$S_{\text{mol}} = \frac{\pi^2 N_A k_B^2 m_{3\text{He}}^*}{\hbar^2} \left(\frac{V}{3\pi^2 N_{3\text{He}}}\right)^{2/3} T \propto \frac{T}{n_{3\text{He}}}$$

$$\implies S_{con} < S_{dil} \qquad (as n_{3,con} > n_{3,dil})$$

$$\implies \dot{Q}(T) = 84 \dot{n}_{3He} T^2[W] \qquad (cooling power \propto {}^{3}\text{He throughput } \dot{n}_{3} \text{ [mol/s]})$$

Т

Т
• ³He/⁴He dilution refrigeration: large cooling power $\dot{Q}(T) = 84 \dot{n}_{3He}T^2$ [W] requires large ³He throughput



• ³He/⁴He dilution refrigeration

—	example:	desired cooling power:	10 ⁻⁵ W
		still temperature:	0.7 K
		mixing chamber temperature:	10 mK

– what is the required pumping speed ??

$$\dot{n}_3 = \frac{10^{-5}}{84 \ (10^{-2})^2} = 0.0012 \text{ mole / s}$$

$$\dot{Q} = 84 \, \dot{n}_3 \, T^2$$

we assume that ³He is an ideal gas (R = 8.31 J / mole K)

 $\dot{V} = \dot{n}_3 RT/p$

R = 8.31 J/mole K

- numbers: vapor pressure of ³He at 0.7 K: 0.0828 mbar = 8.28 Pa \rightarrow @ 300 K we obtain:

 $\dot{V} = 0.0012 \cdot 8.31 \cdot 300/8.28 = 0.363 \text{ m}^3/\text{s} \approx 360 \text{ l/s}$

\rightarrow large ³He pump is required







III.1.8 Pomeranchuk Cooling

• Pomerantchuk cooling (cf. chapter I.5.2)



- precooling to $T < T_{\min}$
- − adiabatic compression
 → solidification and cooling
- − lowest T: ≈ 1.5 mK
 - ➔ limitation due to antiparallel spin ordering in solid ³He



principle: combination of isothermal magnetization and adiabatic demagnetization



- → switch on B_{ext} at T = const, magnetic work is done, generated heat ΔQ is dumped into heat sink → reduction of entropy if T = const.
- isenthalpic or adiabatic demagnetitation: S = const.



- \rightarrow switching off B_{ext} is increasing entropy S of the magnetic system
- → to keep S = const, T has to be reduced and, as $\frac{\Delta Q}{\Delta T} = S = const$, heat ΔQ is removed from spin system

• adiabatic demagnetization



- thermodynamics of adiabatic demagnetization
 - which amount of heat $\Delta Q_{
 m spin}$ can be absorbed by the spin systems ?

$$\Delta Q_{\rm spin}(B=0) = \int_{T_f}^{T_i} C_{\rm spin} dT = \int_{T_f}^{T_i} T\left(\frac{\partial S_{\rm spin}}{\partial T}\right)_B dT \qquad (cooling \ capacity)$$

- entropy of spin system with spin quantum number J for $g\mu_{\rm B}B\ll k_{\rm B}T$:

$$S = Nk_{\rm B} \left\{ \ln(2J+1) - \frac{g^2 J (J+1) \mu_{\rm B}^2}{6k_{\rm B}^2} \quad \frac{B^2 + B_{\rm int}^2}{T^2} \right\} \qquad \text{finite } B_{\rm int} \text{ due to finite spin-spin interaction}$$

– final temperature

$$T_f = T_i \sqrt{\frac{B_f^2 + B_{\text{int}}^2}{B_i^2 + B_{\text{int}}^2}}$$

remaining internal field due to finite magnetic interactions (should be as small as possible)

• adiabatic demagnetization

paramagnetic salts:

e.g MAS = $MnSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$

- \rightarrow cooling of electron spins
- → material with large entropy S/R but large B_{int}
- \rightarrow lowest temperatures $T_f \approx 100 \text{ mK}$
- \rightarrow large cooling capacity

e.g CMN = $2Ce(NO_3)_3 \cdot 2Mg(NO_3)_2 \cdot 24H_2O$

- ightarrow cooling of electron spins
- → material with small entropy S/R but small B_{int}
- \rightarrow lowest temperatures $T_f \approx 2 \text{ mK}$
- ightarrow small cooling capacity

nuclear demagnetization:

e.g ⁶³Cu (
$$L = \frac{3}{2}$$
) or ⁶⁵Cu ($L = \frac{3}{2}$)
($B_{int} \approx 0.3$ mT, $T_i \approx 10$ mK, $B_i \approx 3$ T)

 \rightarrow cooling of nuclear spins

$$\rightarrow T_f (B_f = 0) \approx 1 \,\mu\text{K}$$

→ problem: transfer of spin temperature to lattice

long spin-lattice relaxation time

other materials: ¹⁴¹PrNi₅ (
$$L = \frac{5}{2}$$
), ¹⁹⁵Pt ($L = \frac{1}{2}$)



Cryogen-free Two Stage Adiabatic Demagnetization Refrigerator from Janis

A cryogen-free two stage adiabatic demagnetization refrigerator using a 4 K pulse tube cryocooler. Gallium Gadolinium Garnet (GGG) and Ferric Ammonium Alum (FAA) paramagnetic pills were used for the first and second stage of the ADR, with Kevlar string supports for each stage. The FAA stage reaches a base temperature below 50 mK, and remains below 100 mK for more than two days.



JANIS ADR system:

- \rightarrow pulse tube refrigerator pre-cooling
- → uses gadolinium-gallium garnet (GGG) crystal and a ferric ammonium alumina (FAA) salt pill



Continuous Adiabatic Demagnetization Refrigerator (CADR) under development at NASA's Goddard Space Flight Center

- CADR to cool from below 5K to \approx 35 mK
- advantage: no stored cryogens
 - → maximizing the lifetime/mass ratio for the instrument



Cu demagnetization stage (length: 525 mm, diameter: 78 mm)



R. Gloss et al., *J.* Low Temp. Phys. 73, 101 (1988)



μK facility of PTB Berlin:

lattice temperatures measured on the 105-mol-copper stage of the Berlin microkelvin facility with Pt-NMR. The achieved minimal temperature was 23.3 μ K. The red line depicts the calculated course of temperature for the thermodynamically optimized demagnetization function.

heat leak: below 1.5 nW.

"Bayerische Millimühle 2" @ WMI







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III.2 Thermometry

III.2.1 Thermometry - Introduction

- temperature and temperature scales
 - temperature of a system in thermodynamic equilibrium:

defined as the relation between the amount of heat δQ incident on the system during an infinitesimal quasi-static transformation, and the variation δS of its entropy during this transformation:

→ for reversible Carnot process (
$$dS = 0$$
): $0 = \oint \frac{\delta Q}{T}$

- Lord Kelvin (1854): there is an absolute zero of temperature scale

$$\Rightarrow T_0 = 0 K = -273.15^{\circ}C$$

$$f = 1 K = 1^{\circ}C$$
Kelvin scale
Celsius scale (1742)

see http://www.its-90.com

• William Thomson (Lord Kelvin)

Born	<u>26 June 1824, Belfast, Co. Antrim, Ireland</u>
Died	<u>17 December 1907</u> (aged 83) ^{[1],} Largs, <u>Ayrshire</u> , <u>Scotland</u> ^[1]
Residence	Cambridge, England Glasgow, Scotland
Nationality	United Kingdom of Great Britain and Ireland
Institutions	University of Glasgow
<u>Alma mater</u>	Glasgow University, Peterhouse, Cambridge

a variety of physical phenomena and concepts with which Thomson is associated are named Kelvin:

- Kelvin material
- > Kelvin water dropper
- Kelvin wave
- Kelvin-Helmholtz instability
- Kelvin-Helmholtz mechanism
- Kelvin-Helmholtz luminosity
- > The SI unit of temperature, Kelvin
- Kelvin transform in potential theory
- Kelvin's circulation theorem
- Kelvin-bridge (also known as Thomson-bridge)



William Thomson (Lord Kelvin)

temperature scales

SI 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).

- Celsius scale:

the Celsius scale has units of °C (degrees Celsius) with the size of the unit equal to 1 Kelvin.

 $T(^{\circ}C) = T(K) - 273.15$

agreement of bureaus of standards:

→ *ITS-90 temperature scale* for *T* > 0.65 K (Comité International des Poids et Messures 1990)

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 temperature scale for lower T

(Provisonal Low Temperature Scale, melting curve of ³He)

the PLTS-2000 is defined by a polynomial, relating the melting pressure of ³He 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.



• The Water Triple Point

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)





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• defining Fixed Points of the ITS-90

Number		Temperature	Substance ^a	State ^b	Wr (T ₉₀)
	Т ₉₀ /К	T ₉₀ /°C			
1	3 to 5	-270.15 to -268.15	Не	v	
2	13.8033	-259.3467	e-H ₂	т	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	т	0.008 449 74
6	54.3584	-218.7916	02	т	0.091 718 04
7	83.8058	-189.3442	Ar	т	0.215 859 75
8	234.3156	-38.8344	Hg	т	0.844 142 11
9	273.16	0.01	H ₂ 0	т	1.000 000 00
10	302.9146	29.7646	Ga	м	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	AI	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 ³He are of natural isotopic composition, e-H₂ is hydrogen at the equilibrium concentration of the ortho- and para-molecular forms.

^b 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)

see http://www.its-90.com

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:
 - \rightarrow measured quantity is related directly to temperature (in a theoretically predictably way)
 - ightarrow no calibration is required

- secondary thermometers:

- \rightarrow measured quantity varies with temperature in a reproducible way
- \rightarrow must be calibrated using a primary thermometer
- requirements for temperature measurement:
 - \rightarrow good thermal contact between thermometer and sample
 - \rightarrow low self-heating
 - \rightarrow fast response to temperature changes

typical temperature range of some thermometers



- most common thermometers for 1K < 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 0.4 K for ³He
 - thermocouples: $V_{\rm th} = V_{\rm th}(T)$
 - resistance thermometry: $\mathbf{R} = \mathbf{R}(\mathbf{T})$
 - 1K 300K
 - > semiconductors (e.g. Ge doped with Arsenic has 100-500 Ω/K @ 4.2K, self-heating around 10 μ A)
 - ▶ 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



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• most common thermometers for T < 1 K

$1 \text{ mK} \leq T \leq 1 \text{ K}$:

• 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:

 $m = m_0 f \chi$

- \rightarrow Cerium magnesium nitrate (CMN) useful from 1 K 10 mK
- \rightarrow low temperature limit set by magnetic ordering at \approx 1 mK
- resistance thermometers
- *T* < 1 mK:
 - nuclear Magnetic Resonance (NMR) thermometer
 - \rightarrow temperature dependence of spin relaxation
 - \rightarrow platinum ideal choice for NMR thermometry



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- gas thermometers
 - ideal gas would be a perfect thermometer:

pV = nRT \rightarrow measure pressure at constant volume

for real gases life is more complicated → deviations from ideal behavior

$$pV = n\{RT + b(T)p + c(T)T^{2} + d(T)T^{3} + \cdots\}$$

$$irial coefficients (tabulated ITS-90 values)$$

- systematic errors:
 - \rightarrow dead volumes
 - \rightarrow thermal expansion of cell, elastic deformation of cell
 - \rightarrow adsorption and desorption from walls

→ mainly used in calibration laboratories !

- vapour pressure thermometry
 - Clausius-Clapeyron equation:

$$\frac{dp}{dT} = \frac{L(T)}{(V_{\text{gas}} - V_{\text{liquid}})T} \approx \frac{\Delta H_{\text{vap}}(T)}{V_{\text{gas}}T}$$

- for ideal gas (
$$pV = RT$$
): $R \ln p + const. = \int \frac{\Delta H_{vap}(T)}{T^2} dT$

- if $\Delta H_{\text{vap}}(T)$ is known \rightarrow determine T of liquid (e.g. He) via measurement of He pressure above liquid
- in practice, a set of secondary vapour pressure scales is used:

ITS-90:
$$T = \sum_{i} A_{i} \left\{ \frac{(\ln p + B)}{C} \right\}^{i}$$

with ³He and ⁴He: ITS defined down to 0.65 K

(in principle no primary thermometer !!)





• helium vapor pressure thermometer

values of the constants for the helium vapour-pressure, and the temperature range for which each equation, identified by its set of constants, is valid (see http://www.its-90.com).

	³ He 0.65 K to 3.2 K	⁴ He 1.25 K to 2.1768 K	⁴ He 2.1768 K to 5.0 K	
A ₀	1.053 447	1.392 408	3.146 631	
A ₁	0.980 106	0.527 153	1.357 655	
A ₂	0.676 380	0.166 756	0.413 923	
A ₃	0.372 692	0.050 988	0.091 159	∇ ((ln n +
A ₄	0.151 656	0.026 514	0.016 349 7	$T = \sum A_i \left\{ \frac{(mp+1)}{C} \right\}$
A ₅	-0.002 263	0.001 975	0.001 826	$\frac{1}{i}$ (\overline{c}
A ₆	0.006 596	-0.017 976	-0.004 325	
A ₇	0.088 966	0.005 409	-0.004 973	
A ₈	-0.004 770	0.013 259	0	
A ₉	-0.054 943	0	0	
В	7.3	5.6	10.3	
С	4.3	2.9	1.9	







vapor pressure thermometry



vapor pressure thermometry



www.bm-industries.com

Scale in °C	Range of
	measure in
	°C
- 40+25	-13+15
- 25+40	+5+30
- 10+60	+25+50
0+100	+45+85
+25+125	+70 + 110
+40+160	+100+145
+50+200	+120+185
+60+250	+165+230
+100+320	+230+300
+160+400	+280+375



• ³He melting curve thermometry

- use of melting curve of ³He to define PLTS-2000 temperature scale *down to 0.9 mK*
- polynom for melting curve:

$$p = \sum_{i=-3}^{9} \alpha_i T^i$$

- \rightarrow coefficients given by PLTS-2000
- \rightarrow also use of 4 fix points

minimum of melting curve, transition temperatures to A and B phase and afm order of nuclear spins in solid ³He

• ³He melting curve thermometry

$$p/MPa = \sum_{i=-3}^{+9} a_i (T_{2000}/K)^i$$

$$a_{-3} = -1.385\ 544\ 2 \cdot 10^{-12}$$

$$a_{-2} = 4.555\ 702\ 6 \cdot 10^{-9}$$

$$a_{-1} = -6.443\ 086\ 9 \cdot 10^{-6}$$

$$a_0 = 3.446\ 743\ 4 \cdot 10^0$$

$$a_1 = -4.417\ 643\ 8 \cdot 10^0$$

$$a_2 = 1.541\ 743\ 7 \cdot 10^1$$

$$a_3 = -3.578\ 985\ 3 \cdot 10^1$$

$$a_4 = 7.149\ 912\ 5 \cdot 10^1$$

$$a_5 = -1.041\ 437\ 9 \cdot 10^2$$

$$a_6 = 1.051\ 853\ 8 \cdot 10^2$$

$$a_7 = -6.944\ 376\ 7 \cdot 10^1$$

$$a_8 = 2.683\ 308\ 7 \cdot 10^1$$

$$a_9 = -4.587\ 570\ 9 \cdot 10^0$$



source: R.L. Rusby et al. (2001)

noise thermometry



valid only in the low frequency limit $f \ll k_{\rm B}T$ / h~ (pprox 20 GHz @ 1K)

- temperature determined by measurement of S_V and R

- **example**: $R = 10 \text{ k}\Omega$, T = 1 K, band width $\Delta f = 10^5 \text{ Hz}$

 $\rightarrow (S_V \Delta f)^{1/2} \approx 2 \cdot 10^{-7} \text{ V} \rightarrow \text{sensitive amplifier required} \rightarrow \text{SQUID preamplifier}$

 $\rightarrow P \approx 10^{-18} \,\mathrm{W}$

- superconducting fix point thermometers
 - based on the precise measurement of the transition temperatures of superconductors
 - available from NIST at Boulder

NIST fixpoint device

0.6-2.0

		•Tc	Substance	Width	Reproducibility
I	TS-90	(K)		(mK)	(1 standard deviation) (mK)
Element	TC90/ K	0.0160	°W	0.7	± 0.2
Cd	0.5200 ± 0.0030	0.0230	°Be	0.2	0.1
Zn	0.8500 + 0.0030	0.0990	°lr	0.8	0.1
	1 1010 . 0 0005	0.1605	°AuAl2	0.3	0.1
AI	1.1810 ± 0.0025	0.2065	°Auln2	0.4	0.15
In	3.4145 ± 0.0025	0.5190	+Cd	0.5-8.0	0.3
Pb	7.1997 ± 0.0025	0.8510	+Zn	2.5-10	0.4
Nb	9 2880 + 0 0025	1.1796	+AI	1.5-4.0	0.4
112	5.2000 I 0.0025	3.4145	+In	0.5-2.5	0.2

7.1996

°SRM 768 [Schooley and Soulen (1982)]

+Pb

+SRM 767 [Schooley et al. (1980)] * T₉₀ above 0.5 K.

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0.2


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• adiabatic demagnetization





resistance thermometers

- required: well established relation between resistance and temperature, sufficiently large dR/dT
- advantage: resistance easy to measure \rightarrow resistance thermometry very popular
- fact: temperature variation of resistance may have very different physical origin
- commonly used:
 - \rightarrow Pt resistors (PT-100, PT-1000)
 - \rightarrow RhFe resistors
 - \rightarrow carbon resistors (Speer, Allen-Bradley)
 - \rightarrow carbon glass resistors
 - \rightarrow Ge resistors
 - \rightarrow RuO₂ resistors



• Platinum resistors

The platinum resistance thermometer (PRT) is very widely used below 500 °C as a thermometric sensor. There is a wide range of quality of PRT available, from the standard instrument (SPRT) of the ITS-90 to some industrial types (IPRT) that are accurate only to within a few tenths of a kelvin or, perhaps, even a kelvin or more. The major difference of the industrial type of fabrication from the standard type is not just the purity of platinum, but also the less strain-free mounting of the film or wire which is embedded (partially or totally) in a cement (glass or refractory). Furthermore, in most cases, the thermometer body is not hermetically sealed.



Source: Lake Shore Cryotronics, Inc.

- Platinum resistors
 - ITS-90 requirement for Pt resistance thermometer (PRT)

temperatures are determined in terms of the ratio of the resistance $R(T_{90})$ at a temperature T_{90} and the resistance R(273.16 K) at the triple point of water:

$$W(T_{90}) = R(T_{90})/R(0.01 \text{ °C})$$

 $W(29.7646 \ ^{\circ}C) \ge 1.118 \ 07$ $W(-38.8344 \ ^{\circ}C) \le 0.844 \ 235$

– industrial PRT

for $0 < T < 100^{\circ}C$ $R = R_0 \cdot (1 + a \cdot T)$ $a = 3.85 \cdot 10^{-3} / K$

allowed errors in °C: Class A: $dT = \pm (0.15 \text{ °C} + 0,002 \cdot T)$ Class B: $dT = \pm (0.30 \text{ °C} + 0,005 \cdot T)$ 1/3 Class B: $dT = \pm 1/3 \cdot (0.30 \text{ °C} + 0.005 \cdot T)$



• RhFe resistor thermometer



Rhodium with 0.5% Fe

Source: Lake Shore Cryotronics, Inc.

RF-100-AA

RF-800



• carbon resistors

commercial carbon-composition resistors manufactured by Allen-Bradley were introduced as temperature sensors by Clement and Quinnell in 1952. The carbon composition resistor is a small cylinder consisting of graphite with a binder encased in an outer phenolic shell.

The carbon resistors used as thermometers are generally characterized by their room temperature resistance and their wattage [see Rubin (1980)], and have come largely from the following manufacturers:

- Allen-Bradley
- > Airco Speer (usually referred to simply as Speer)
- Ohmite
- Matsushita,
- CryoCal

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 resistors

A porous glass is prepared by removing the boron-rich phase from a borosilicate alkaline glass to leave a material having the appearance of silicate spheres of about 30 nm diameter, randomly distributed and separated by 3 to 4 nm pores. The spaces are then partially filled with high-purity carbon to form amorphous fibres \rightarrow better stability than carbon resistors.



Source: Lake Shore Cryotronics, Inc.



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III.2.3 Secondary Thermometers

• Cernox resistors

Patents:

#5,363,084, Nov. 1994, "Film Resistors Having Trimmable Electrodes"

#5,367,285, Nov. 1994, "Cernox[™]", "Metal Oxy-nitride Resistance Films and Methods of Making the Same,"

→ small magnetic field coefficient: $\Delta T/T$ typically smaller than 0.5% @ 19 T









100

500

• Cernox resistors



Low temperature thermometry in high magnetic fields VII. Cernox sensors to 32 T, B.L. Brandt *et al, Rev. Sci. Instrum., vol 70, No 1, 1999, pp 104-110.*

• Germanium resistors



Source: Lake Shore Cryotronics, Inc.

• Germanium resistors





Source: Lake Shore Cryotronics, Inc.



• Thermocouples

Thermocouples are pairs of dissimilar metal wires joined at least at one end, which generate a net thermoelectric voltage between the 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



• 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 μ V/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 !!



Thermocouples

Thermocouple Type	Names of Materials	Useful Application Range		
В	Platinum30% Rhodium (+) Platinum 6% Rhodium (-)	2500 -3100F 1370-1700°C		
С	W5Re Tungsten 5% Rhenium (+) W26Re Tungsten 26% Rhenium (-)	3000-4200F 1650-2315°C		
E	Chromel (+) Constantan (-)	200-1650F 95-900°C		
J	Iron (+) Constantan (-)	200-1400F 95-760°C		
К	Chromel (+) Alumel (-)	200-2300F 95-1260°C		
Ν	Nicrosil (+) Nisil (-)	1200-2300F 650-1260°C		
R	Platinum 13% Rhodium (+) Platinum (-)	1600-2640F 870-1450C		
S	Platinum 10% Rhodium (+) Platinum (-)	1800-2640F 980-1450°C		
Т	Copper (+) Constantan (-)	-330-660F -200-350°C		

• Thermocouples









• Diode Thermometers

The temperature-indicating parameter is the forward-biased junction voltage, which decreases approximately linearly with increasing temperature when the current is kept constant, since $I \propto \exp(eV/k_{\rm B}T)$

- → The typical I-V characteristic is such as to make the internal impedance of the device very high (easily greater than 100 k Ω) at small currents; or else - using a larger current one encounters unacceptably high power dissipation at low temperatures.
- → There is a transition region in the conduction mechanism around 20 K that makes fitting a V-T characteristic over the whole temperature range difficult for GaAs and impossible for Si







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

Capacitive Thermometers



100

300





Type of Sensor	T(K)	1.T	Magnet 2.5 T	ic Flux Den 8 T	nsity, B 14 T	19 T	Notes	References
Carbon and a surface							2	Description of District
Carbon radio resistors			~ .		7.00			Sample and Rubin
Allen-Bradley (2.7, 3.9,	0.5		2.4	5-13	7-20			(10//)
5.6, 10 C)	1.0		2-9	0-15	8-20			
	2.5		1-5	8-18	10-30			
	9.2		1-0	5-20	10-35			
Allen-Bradley (47, 100,	4.2		<1	5	10		a	ibidem
220 D)	10		<1	3	5			
	20		<1	1	2			
Speer, Grade 1002 (100,	0.5		0-2	0-1	0-6		ь	ibidem
220, 470 C)	1.0		1-2	2-4	3-9			
ACASI ANG SAINA	2.5		3-5	1-4	7-14			
	4.2		4-9	2-5	4-13			
Matsushita (68, 200	15		1-2	10-15				ibidem
510 C)	21			10-15				- Andrews
	4.2		2-3	4-8				
K) (ht each on composite					2		α.	Astrony at al
Kvm carbon composite	4.9		3	8 (51)			d	Astrov et al.
resistors	4.2		1.0					(1877)
	10		0.4	14				
	20		0.1	0.4				
	80		<0.01	<0.01				
Carbon-Glass Resistors	2.2		0.1	1.5	3	4		Rubin and Brandt
	4.2		0.5	2	5	7		(1986)
	10		0.2	1.1	3	4		
	20		< 0.01	0.02	0.03	0.13		
	45		0.07	0.5	1.3	2		
	88		0.06	0.5	1.3	2		
	190		0.04	0.3	1.0	1.7		
	310		< 0.01	0.2	0.6	1.1		
Thermistors	4.2		<0.05	1	3		4	Sample and Rubin
	10		<0.05	0.3	1		12	(1977)
	20		<0.05	0.1	0.5			1. C.
	40		<0.05	0.1	0.5			
	60		<0.05	0.1	0.3			
Germanium Resistors	2.0		8.10	80				ibidam
Germanion Resistors	4.2		5.20	30.65	60.70			ibideni
	10		4.15	35-00	60.75			
	20		3.20	15.35	50-80			
	70		3-10	15-30	25-50			
-			-				22	100000000000000000000000000000000000000
Besisters TSC 2	9.2		30	120			9	Astrov et al.
Resistors 150-2	20		2,5	0				(19/7)
Specially doped	4.2		<0.2.	0.5 (6T))		h	ibidem;
Ge resistors KG	10		<0.2	<0.5				Matacotta et al.
	20		<0.5	2-3 "				(1984)
	30		<0.5	5 .				
	80		0.15	0.5 *				
Platinum Resistors	10		100	250	34	1	i i	Pavese & Cresto
	20		2-8(20	25 100	250	1		(1984):
	40		0.5/<1	315	6110	90		Neuringer et al
	66		0.11<0.5	0.812	215	41		(1971);
	87		0.041<0.5	0.411	112	21		Rubin & Brandt
	110		0.02	0.2	0.6	11		(1986)
	190		<0.011	0.06	0.2	0.3		10071120
	300		<0.011	0.021	0.071	0.13		

(a) (b) (a) (b)

(a) (b) (a) (b)

Magnitude of relative temperature error (ΔT)/T (%) for values of B

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magnetic field effects

Type of Sensor	T(K)	1 T	Magneti 2.5 T	e Flux Den 8 T	sity, B 14 T	19 T	Notes	References
Rhodium-Iron realistors	2.0 4.2 40 66 87 110 190 300	2 0.8	22 11 4 1.5 0.3 0.2 0.1 0.03 <0.01	40(8T) 10(5T) 12 2.5 1.5 0.9 0.3 0.1	30 6 4 2.4 0.9 0.4	40 9 6	I	Pavese & Cresto (1984); Rusby (1972); Rubin & Brandt (1986)
Platinum-Cobalt resistors	2 4.2 10 20 30	25 8 1 0.2 0.2	30 3 <0.1 1 0.3	40 (5T) 12 * 3.5 * 1.5 *			k	Shiratori et al. (1982); Pavese & Cresto (1984)
Cryogenic linear resistance sensor	4 10 20 30	20 17 8 5	250 100 50 30				3	McDonald (1973)
KELTIP resistors (AuMn)	4.2 40 87 110 190 300		4 0.4 0.15 0.03 0.02 0.02	13 30 4 1.5 0.25 0.2 0.1	20 70 12 5 1 0.5 0.4	110 20 10	m	Rubin & Brandt (1986)
SrTi03 capacitors	2.2 4.2 20 50 88 110 190		<0.02 <0.01 <0.05 <0.05 <0.01 <0.01 <0.01	<0.02 <0.01 <0.05 <0.05 <0.01 <0.01 <0.01	0.02 0.01 <0.05 <0.05 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01	n	ibidem
Si Diodes	4.2 10 20 30 77		75 20 4 3 0.2	30 7 4 0.5	50 10 5 0.5		0	Sample & Rubin (1977)
GaAs Diodes	4.2 10 20 40 80		2-3 1.5-2 0.5-1 0.2-0.3 0.1-0.2	30-50 25-40 20-30 4-8 0.5-1	100-250 75-200 60-150 15-30 2-5		р	ibidem
Au + 0.07 % Fe/ Chromel P thermocouple	5 10 20 45 100		2 3 2 1 0.1	10 20 15 5 0.8	15 30 20 7		q	Sample et al. (1974)
Chromel P/Constantan thermocouple (Type E)	10 20 45		1 <1 <1	3 2 <1	7 4 2		1	lbidem
Cu + 0.01 % Fe/Cu thermocouple	5 10 20 50		2 0.8 0.6 0.3	3.5 (5T) 2 - 1.5 - 0.6 -			q,f	Astrov et al. (1977)
Vapour pressure thermometers	no intrinsio except with	c error h 03						
Helium gas thermometer	no intrinsia	s error						Van Degrift et al. (1980)

W MI

Table 1.1: Summary of Some Properties of Most-Commonly-Used Thermometers

hermometer	Usual Temperature Range	Thermometric Quantity	Typical Uncertainty
Sermanium	1 K to 100 K	elec. resist.*	ΔT / T < 2x10-4
Rhodium-Iron	0.5 K to 30 K	elec. resist.*	0.3 mK
Platinum-Cobalt	2 K to 20 K	elec. resist.*	10 mK
(industrial type)			
Carbon	0.5 K to 30 K	elec. resist.*	ΔT / T < 5x10-3
Carbon-glass	0.5 K to 100 K	elec. resist.*	ΔT / T < 1 x1 0-3
Diode	4 K to 300 K	junction voltage	-50 mK
/apour-pressure	various subranges between 0.5 K and 100 K	pressure	-1 mK
Aercury-in-glass	-50 °C to 250 °C	thermal expansion of mercury	0.1 K
Thermocouples	4 K to 2500 °C	thermoelectromotive force	
Type S	-50 °C to 1600 °C		0.3 K < 1000 °C 1 K > 1000 °C
Type R	-50 °C to 1600 °C		0.3 K < 1000°C 1 K > 1000 °C
Type B	300 °C to 1800 °C		0.5 K to 2 K
Type T	-200 °C to 350 °C		0.1 K
Type E	-200 °C to 870 °C		0.1 K < 300 K 1 K > 300 K
Type J	0 °C to 760 °C		0.5 K < 300 °C 2 K > 300 °C
Type K	-200 °C to 1260 °C		0.1 K < 200 °C 1 K 200-1000 °C 3 K > 1000 °C
Type N	0 °C to 1300 °C		0.1 K < 200 °C 0.5 K 200-1000 °C 3 K > 1000 °C
W/Re	1000 °C to 2400 °C		3-10 K

III.2.3 Secondary Thermometers

Thermometer	Usual Temperature Range	Thermometric Quantity	Typical Uncertainty
Thermistor	-80 °C to 250 °C	elec. resist.*	0.1 K (much better if use confined to very small temperature range)
Platinum: SPRT IPRT	14 K to 630 °C 20 K to 600 °C	elec. resist.*	0.5 mK 50 mK
Radiation	100 °C to 3000 °C	spectral radiance of source	1 K < 1000 °C 5 K > 1000 °C

Magnetic Susceptibility Thermometers

- Curie-Weiss law:
$$\chi = \frac{C}{T - TC}$$

 \rightarrow measure χ as a function of *T*, calibaration requires determination of *C* and *T*_C

- experimental techniques:
 - → inductance bridge: compare inductance of coil containing the magnetic material to empty reference coil
 - → SQUID magnetometer: resolution: $\rightarrow \Delta T/T \approx 10^{-10} @ 1 K$ $\rightarrow increases \approx 1/T$ First order gradiometer reference coil First order gradiometer First order First

T.C.P. Chui et al., Phys. Rev. Lett. 69, 3005 (1992)

- Magnetic Susceptibility Thermometers
 - materials (use of electronic magnetic susceptibility):
 - \rightarrow paramagnetic salts:
- e.g. Ce-Mg-nitrate (CMN) ++ very low ordering temperature: $T_c \sim 2 \text{ mK}$ ++ large Curie constant
- - long time constant at low $T (\sim 100 \text{ s})$
- -- cannot be used in vacuum (instable)



- Magnetic Susceptibility Thermometers
 - materials (use of nuclear susceptibility):

 \rightarrow use of nonmagnetic metals: e.g. Cu

- - nuclear moments much smaller \rightarrow sensitive SQUID magnetometer
- - perturbing magnetic impurities \rightarrow very pure materials

(e.g. 1 ppm Fe in Cu gives similar signal as all Cu nuclear spins)

++ can be used below 1 mK

- **Nuclear Magnetic Resonance Thermometers**
 - are not based on static orientation of nuclear moments as in susceptibility thermometers
 - have selectivity to specific nuclear moments (less sensitive to magnetic impurities)
 - usable at temperatures below 1 mK
 - materials: predominantly ¹⁹⁵Pt
 - experimental techniques: stationary and pulsed NMR
 - \rightarrow measures resonance absorption of high frequency signal \rightarrow allows to determine induced
 - $M_{\gamma} \propto M_0(T) \propto 1/T_N$ at resonance
 - \rightarrow requires small $B_{\nu} \approx 1 \, \mu T$ to avoid saturation effects

- \rightarrow measures decay of induced $M_{v}(t)$ after 90° pulse \rightarrow amplitude $dM_{\gamma}(t = 0)$ or integral of decay curve $\propto M_0(T) \propto 1/T_N$
- \rightarrow general problem: one measures temperature T_N of nuclear spins \rightarrow are they in thermal equilibrium with lattice ??

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 B_z

 $B_v \cos \omega t$

Nuclear Magnetic Resonance Thermometers



comparison of the Curie and Korringa temperatures of a Pt sample:

the inverse nuclear susceptibility $1/\chi_N$ (arb. units) is plotted against the electron temperature

$$T_e = \left(\frac{1}{t_1}\right) \cdot 29.9 \times 10^{-3} \text{ K sec.}$$

A.I. Ahonen et al., J. Low Temp. Phys. **25**, 421 (1976)