T- and p-Measurement

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Content:

T	**P**
Resistance T-detectors	Direct: Mechanical force
Thermocouple	Indirect: Heat conductivity
Pyrometer	Indirect: Gas ionization
Comparison	gauge combinations
	QMS

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Literature - T-measurement:

F.X. Eder, Arbeitsmethoden der Thermodynamik, Bd. I, Springer, Berlin 1981.

L. Weichert et al., Temperaturmessung in der Technik, Lexika-Verlag, Grafenau, 1976.

F. Henning, Temperaturmessung, H. Moser ed., Springer, Berlin 1977.

G. Heyne, Einführung in die elektronische Messtechnik, 1997.

M. v. Ardenne, G. Musiol, S. Reball, Effekte der Physik und ihre Anwendungen, Deutsch, Thun, 1997.

Catalogs from Heraeus Sensor GmbH, Hanau:

Temperaturmessung mit Thermoelementen, Ausgleichsleitungen..., Mantel-Thermoelemente,
Temperaturmessung mit Widerstandsthermometern, Messwiderstände

Pyrometerhandbuch, IMPAC Infrared GmbH, Frankfurt 2004.

http://www.ir-impac.com/deutsch/Pyrometerhandbuch.pdf

Wikipedia English: http://en.wikipedia.org
German: http://de.wikipedia.org

Literature - p-measurement:

W. Pupp, H.K. Hartmann, Vakuumtechnik, Grundlagen und Anwendungen, Carl Hanser, München (1991).

M. Wutz, H. Adam, W. Walcher, Theorie und Praxis der Vakuumtechnik, Vieweg, Braunschweig (1982). (*New edition available*).

Leybold-Heraeus GmbH, Grundlagen der Vakuumtechnik, Berechnungen und Tabellen.

A. Roth, Vacuum Technology, North Holland, Amsterdam (1976).

J.F. O'Hanlon, A User's Guide to Vacuum Technology, 2nd ed. Wiley, New York (1989).

N.S. Harris, Modern Vacuum Practice, McGraw-Hill, Maidenhead (1989).

Temperature

Temperature measurement

Simplest definition of temperature:

$$p \ V = n \ R \ T.$$

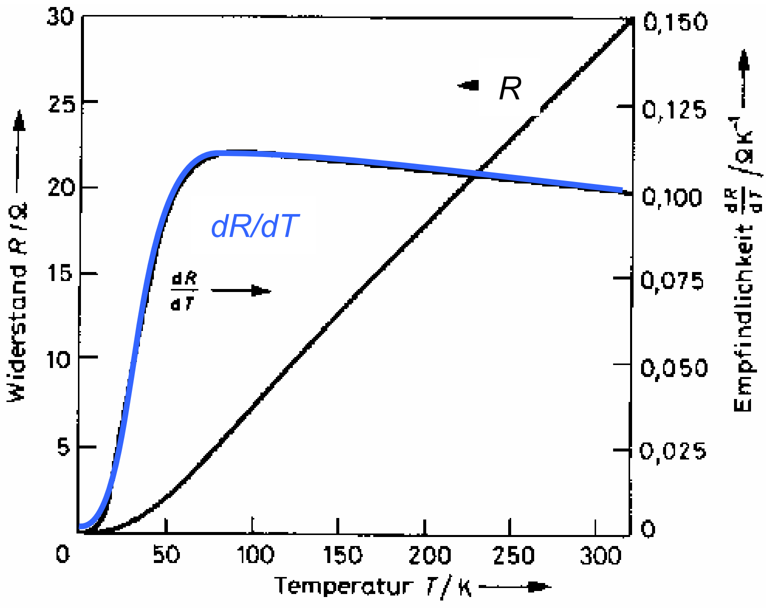
(ideal gas law, „zeroth law“ of thermodynamics)
For a given amount of gas (n moles), T is simply given by p and V
→ gas thermometer.

Practical T-measurement uses:

Thermal expansion of	gases	gas thermometer
	liquids	„normal“ thermometers
	solids	bimetal thermometers
T-dependent electrical resistance	metals	resistance T-detectors
	semicond.	diodes, thermistors
T-dependence of work function (Seebeck-effect)	metals	thermocouple
Radiation detectors	via heat	bolometer, thermopile
	direct	pyrometer

More information: => separate sheet

T-measurement - Resistance T-detectors



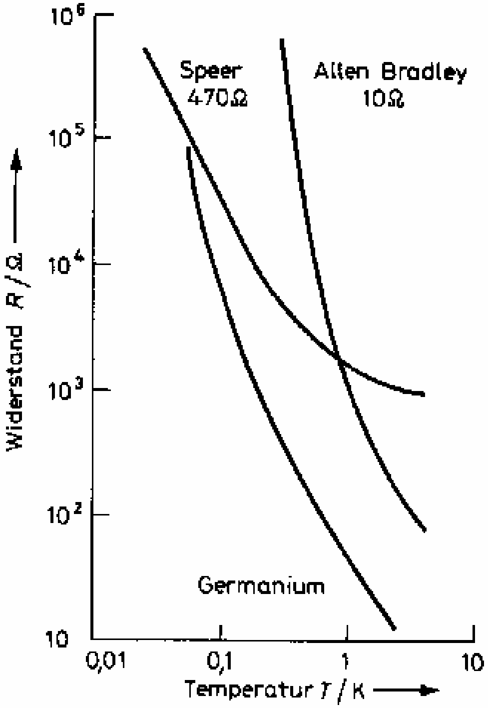
Metals (Pt): R almost linear with T

Empirically
for standard Pt resistors, $R_{0°C}=100\Omega$):

0 < t < 850°C:
$R_t = 100\,(1+3.90802\times10^{-3}\,t - 0.5802\times10^{-6}\,t^2)$

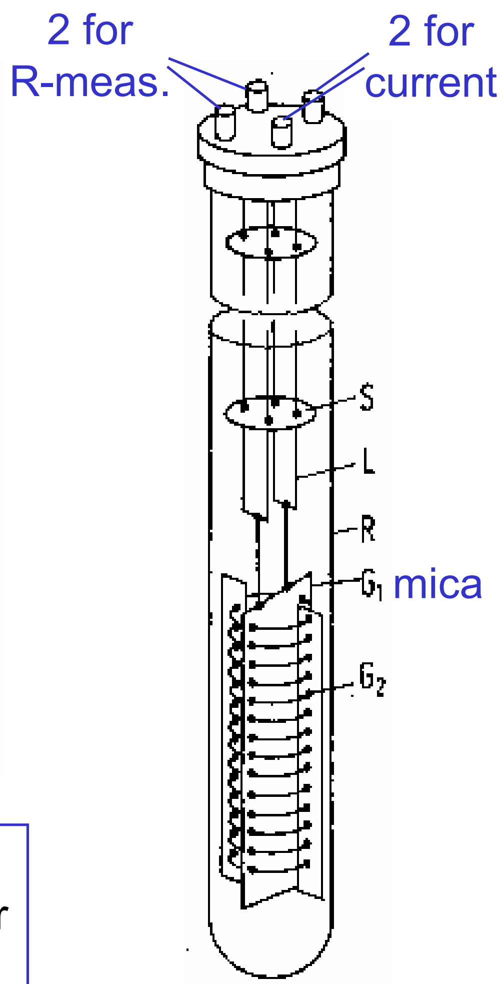
-200 < t < 0°C
$R_t = 100\,(1+3.90802\times10^{-3}\,t - 0.5802\times10^{-6}\,t^2$
$+0.42735\times10^{-9}\,t^3 - 4.2735\times10^{-12}\,t^4)$

Max deviation:
+-(0.3+0.005 |t|) (class B)



Semiconductors:
Strong but nonlinear T-dependence of R, used at low T.

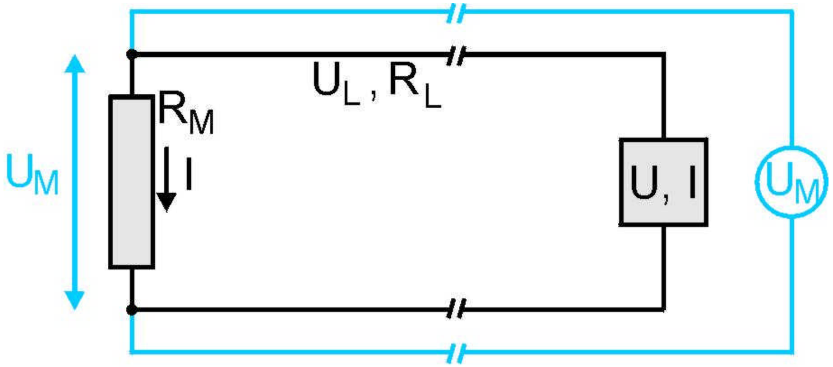
Thermistor:
T-dependence of transistor properties



Pt-Resistance thermometer for calibration (NBS)
secondary standard

T-measurement - Resistance T-detectors

How to measure R precisely? – Don‘t forget the resistance of the wiring!



$$R_M + R_L = U / I$$

$$R_M = U_M / I$$

In order to measure a resistance R_M, always a <u>current</u> has to be passed through it. Then, according to Ohm‘s law,

$$R_{tot}=U/I.$$

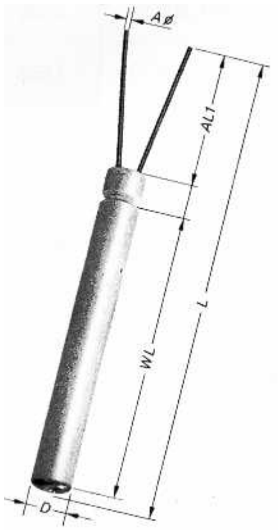
Part of the voltage drops along the wires with their resistance R_L(which usually is not precisely known).

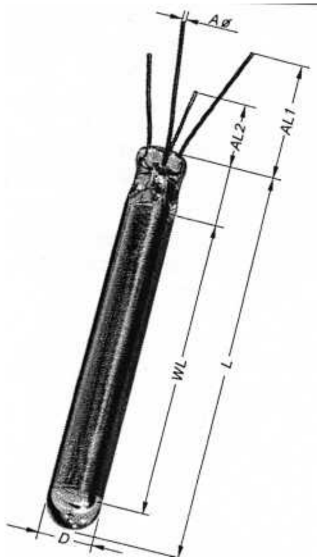
Therefore, U_M has to be measured <u>separately</u> (blue wires):

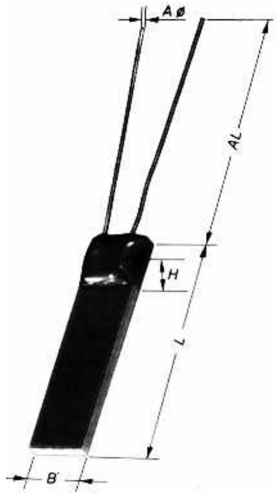
Four-wire-technique

T-measurement - Resistance T-detectors

Examples of resistance sensors (Heraeus)







Ex.1:
Pt in ceramics
-200°C to +850°C
WL 20mm
D 1.5mm
t1/2 (water) 0.2s
(air 1 m/s) 5s

Ex.2:
Pt in glass, 2 filaments
0°C to +600°C
WL 30mm
D 5mm
t1/2 (water) 0.8s
(air 1 m/s) 13s

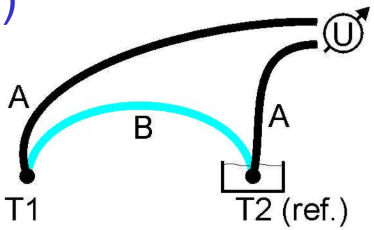
Ex.2:
Pt film on ceramics
-70°C to +500°C
L min. 3.9mm
B min. 1.9mm
t1/2 (water) 0.1s
(air 1 m/s) 5s

Complete resistance thermometers (resistors in housing)

two galvanically separated filaments:
one for T-display
one for process control

T-measurement - Thermocouple (Seebeck-effect)

Advantages: small, fast, simple, electrical signal
Disadvantages: reference T needed



standard:
$T_{ref} = 0°C$

Type	Material	Symbol	Temp. range (°C)
K	nickel-chromium / nickel „chromel-alumel“	NiCr-Ni	-200…900 (1300)
J	iron / copper-nickel „iron-konstantan“	Fe-CuNi	-200…700 (1200)
N	nickel-chromium-silicon / nickel-silicon, „nicrosil-nisil“	NiCrSi-NiSi	-200…1200
E	nickel-chromium / copper-nickel „chromel-konstantan“	NiCr-CuNi	-200…900 (1000)
T	copper / copper-nickel „copper-konstantan“	Cu-CuNi	-200…400
S	platinum-10rhodium./.platinum	Pt10%Rh-Pt	0…1300 (1700)
R	platinum-13rhodium./.platinum	Pt13%Rh-Pt	0…1300 (1600)
B	platinum-30rhodium./.platinum-6rhodium	Pt30%Rh-Pt6%Rh	0…1800

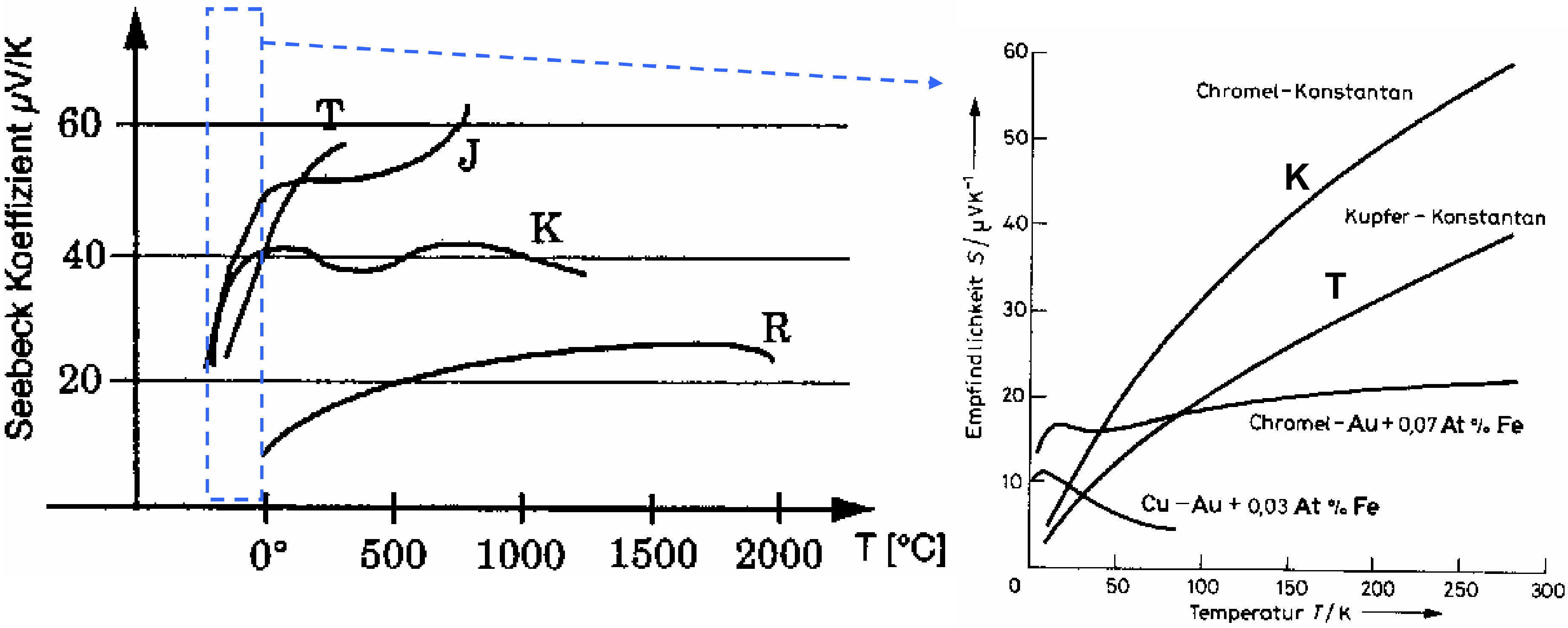
values in ( ): not for permanent use, not in reactive atmosphere

More information => separate sheet

T-measurement - Thermocouple

Thermovoltage generally not linear in T

Low T: generally more problematic.



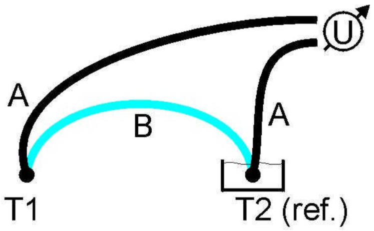
Seebeck coefficient:

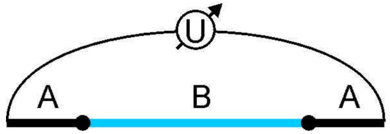
$\Delta U_{th}/\Delta T$ μV/K

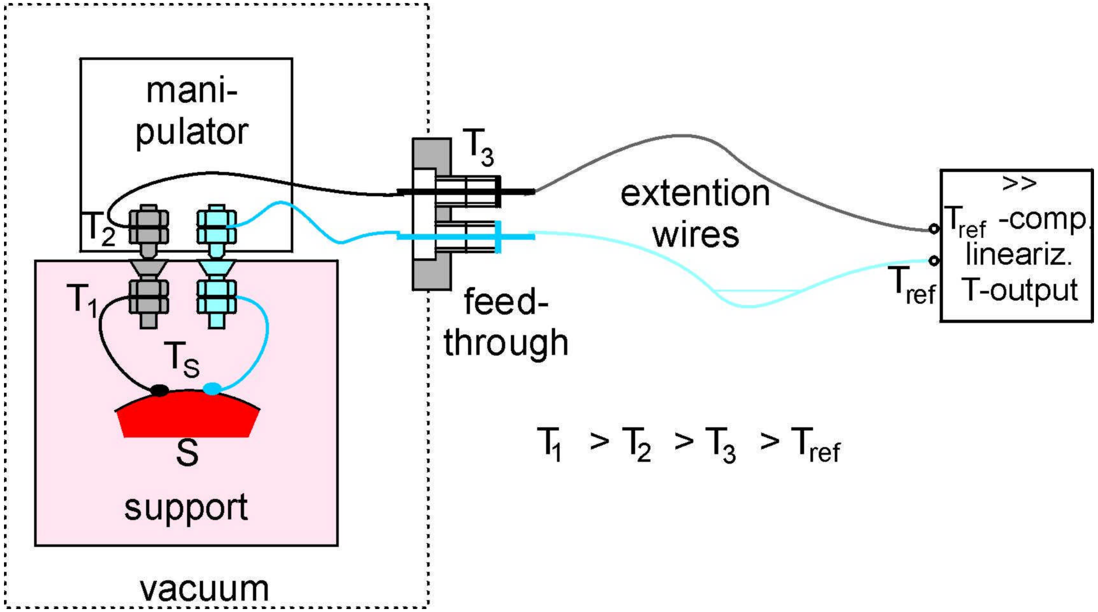
change of thermovoltage per degree

T-measurement - Thermocouple

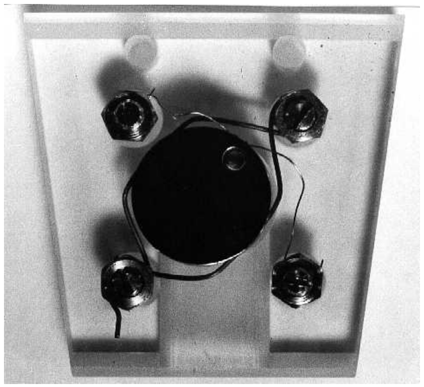
Wiring and unintended thermovoltages

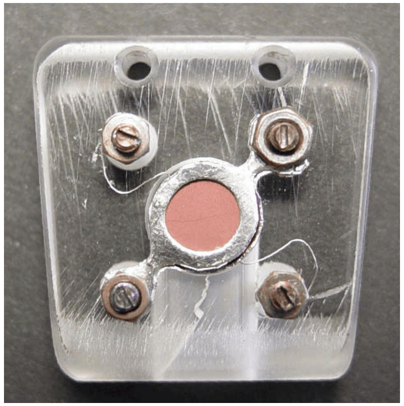






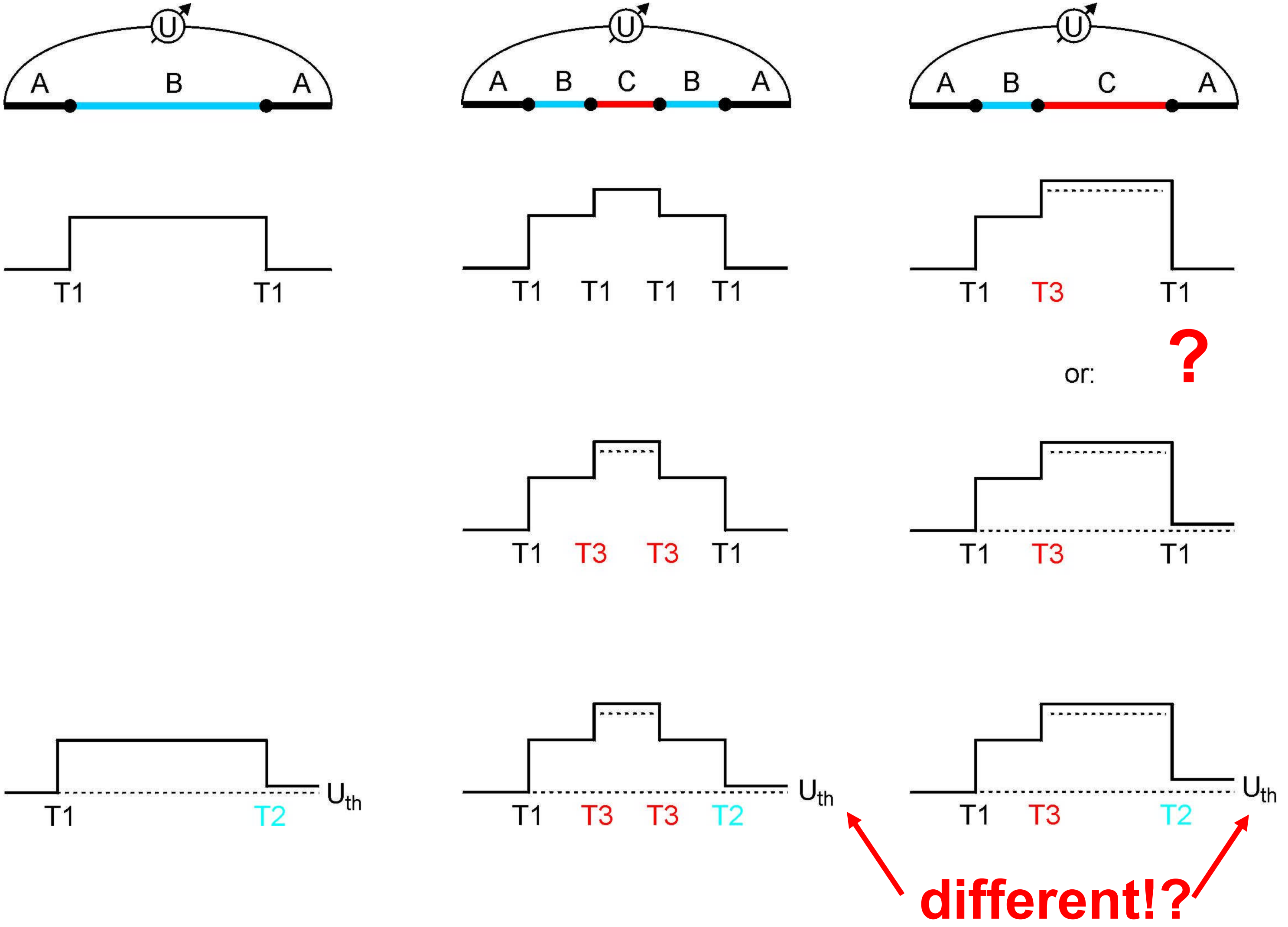
$T_1 > T_2 > T_3 > T_{ref}$





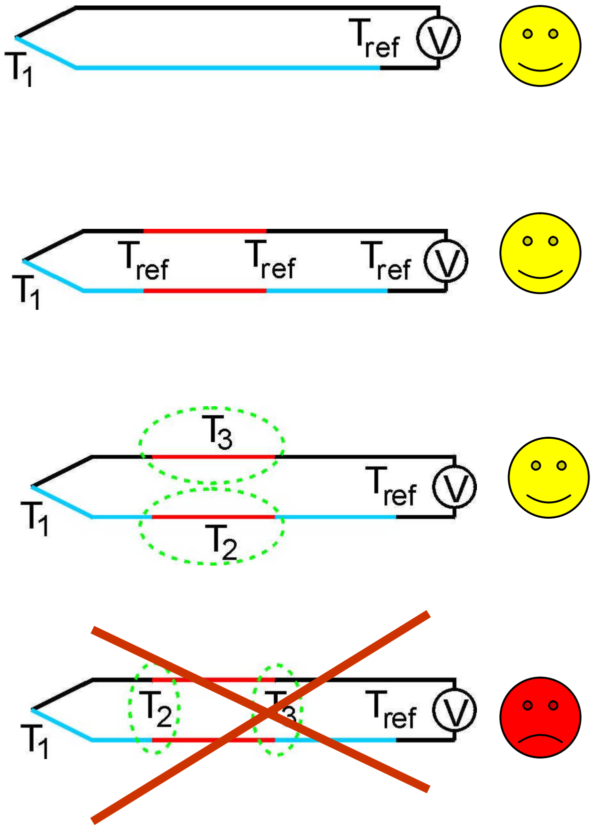
T-measurement - Thermocouple

Wiring and unintended thermovoltages



Thermovoltages additive (like electrochem. potentials) ?

T-measurement - Thermocouple

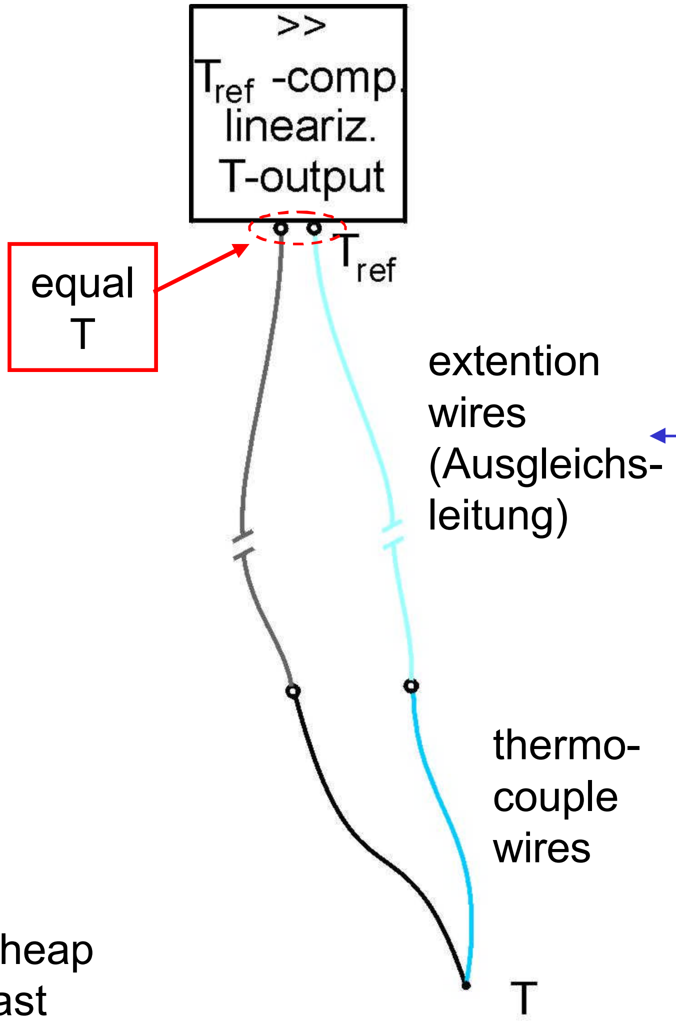


Thermovoltages not simply additive!

When T-gradients exist:

all materials in each branch must be made from the same (thermocouple) material (or extention wire material)

Self-made thermocouples



extention wires:
- cheap material
- same thermovoltage (up to 200°C)

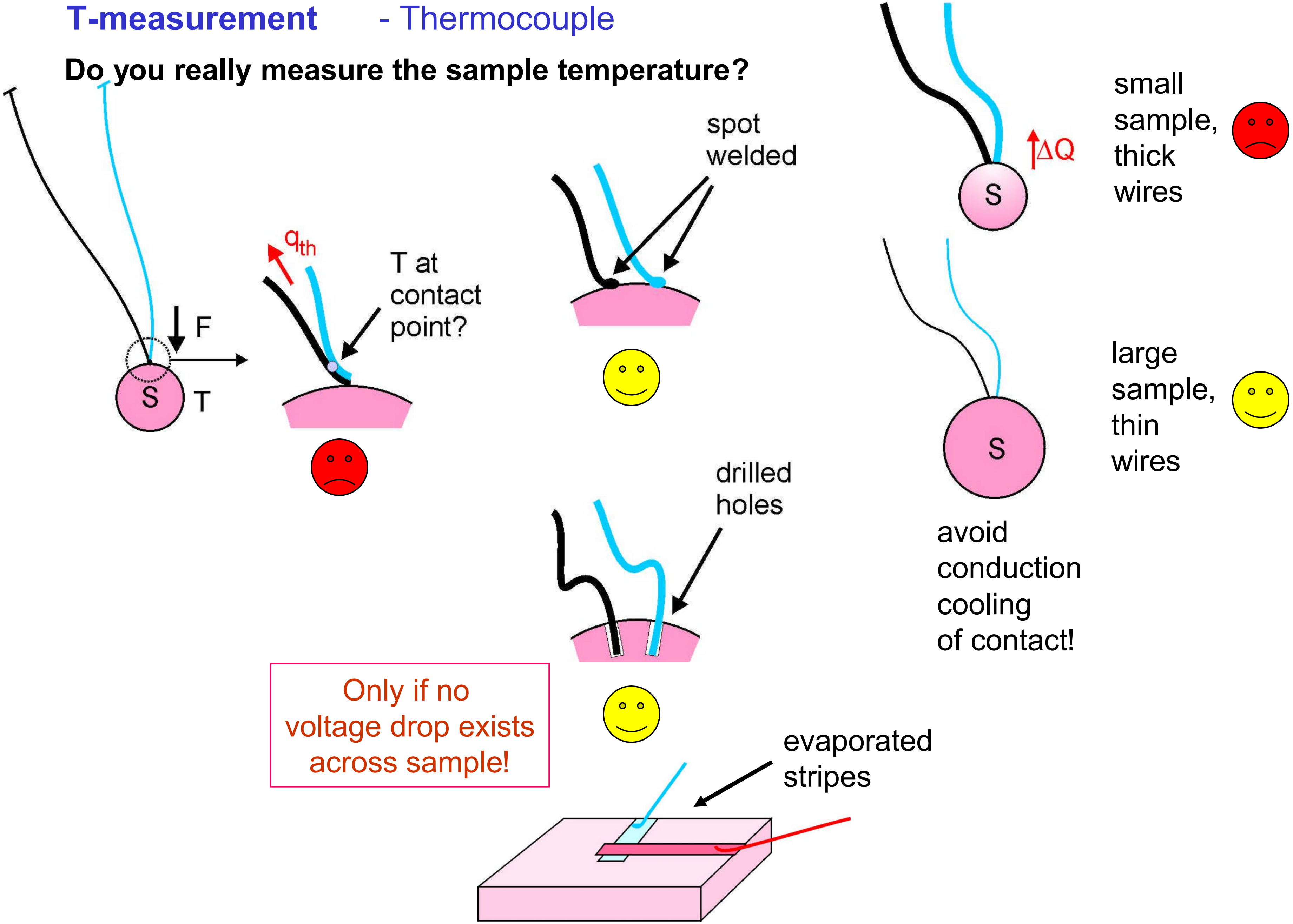
simple wires, "self-made"

🙂 cheap
fast

☹ no galvanic separation

T-measurement - Thermocouple

Do you really measure the sample temperature?



T at contact point?

spot welded

drilled holes

Only if no voltage drop exists across sample!

evaporated stripes

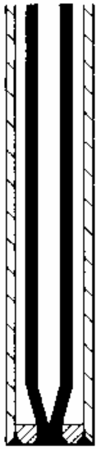
small sample, thick wires

large sample, thin wires

avoid conduction cooling of contact!

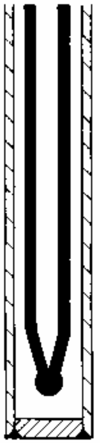
T-measurement - Thermocouple

Escapsulated thermocouples (Mantelthermoelemente)



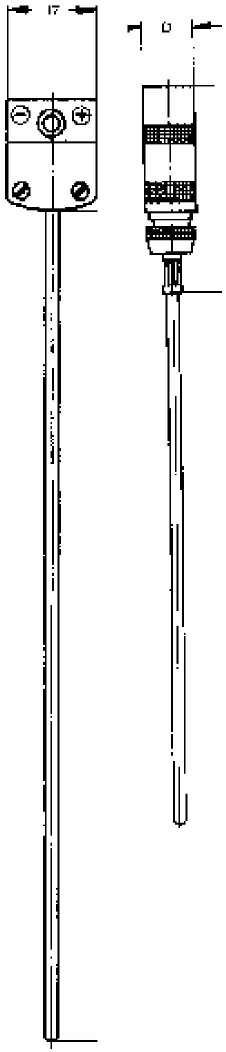
☹ grounded*

☺ fast

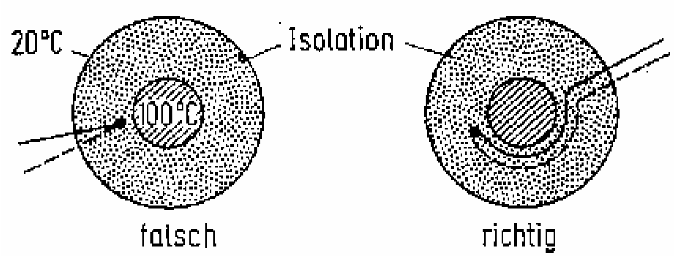


☺ isolated

😐 slower

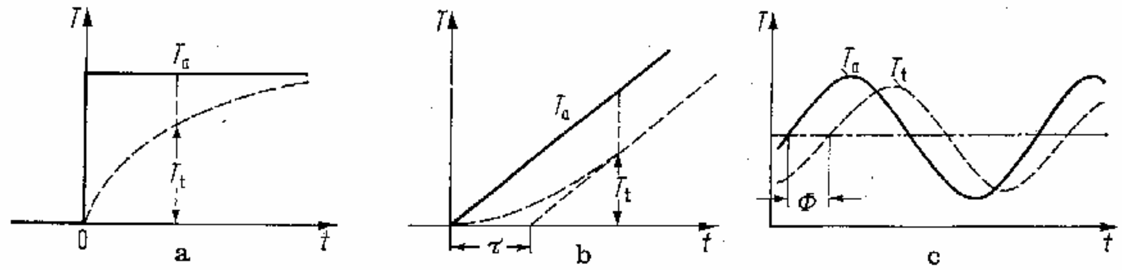


	stainless steel	Iconel
d (mm)	0.5 – 3	0.25 – 6
l (m)	200 – 1000	
isol.-R	>1000 MΩ	
bending-r	2 x d	



measurement in solids

Time response



T-jump

continuous T-change

periodic T-change

* not shielded, bias, noise, inductive voltages, ground loops

T-measurement - Thermocouple

Accuracy

Voltage measurement (floating ground? noise? ground loops?)
see manual, ask electronics workshop

Reproducibility of thermomaterials and thermovoltages
Ex.: K-type, allowed +/-2.5° or 0.0075 |t| (+/-7.5° at 1000°C)

Linearization of thermovoltage
see manual, ask electronics workshop

Thermal contact
your responsibility!

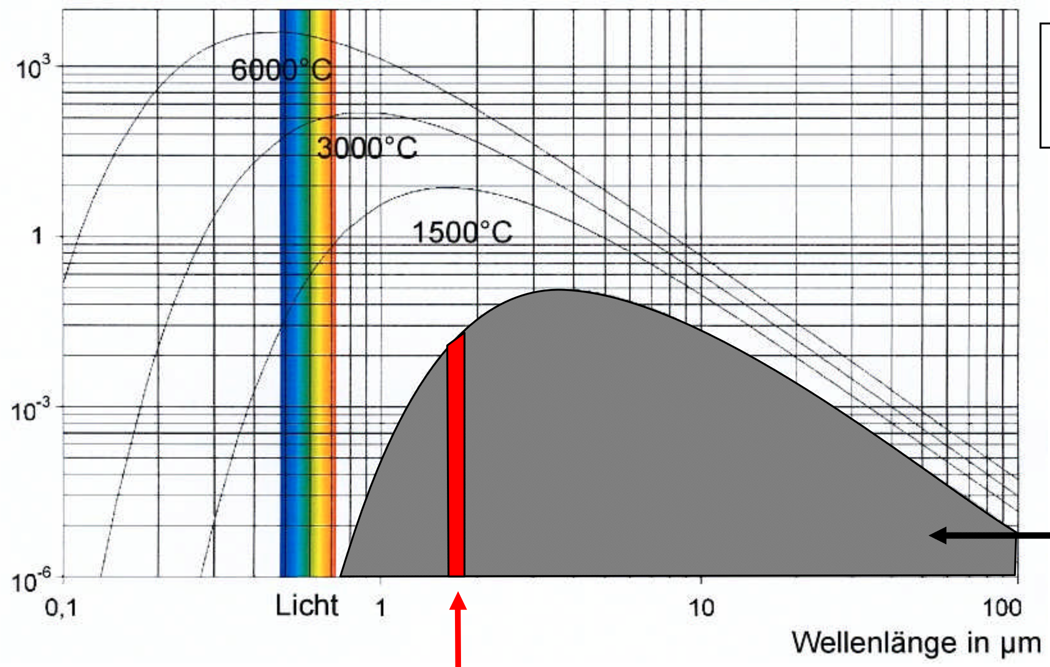
Thermal loss by heat conductivity
your responsibility!

Reproducibility

Usually high

T-measurement - Pyrometer, Thermal radiation measurement

non-contact measurement



Spectral distribution of light emission, black-body radiation

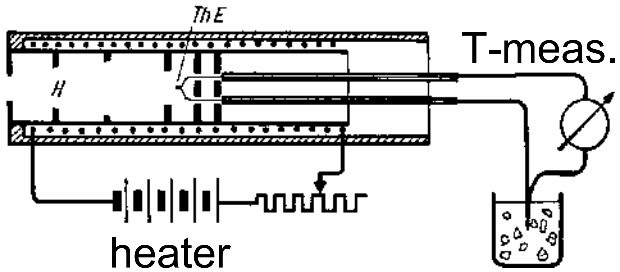
$I_{tot} \sim T^4$ (Stefan Boltzmann), total-radiation pyrometer, thermal sensors (bolometer, thermopile)

I_{part}, weaker (but still strong) T-dependence band- or partial radiation pyrometer, photon sensors: photo diodes

T-measurement - Pyrometer

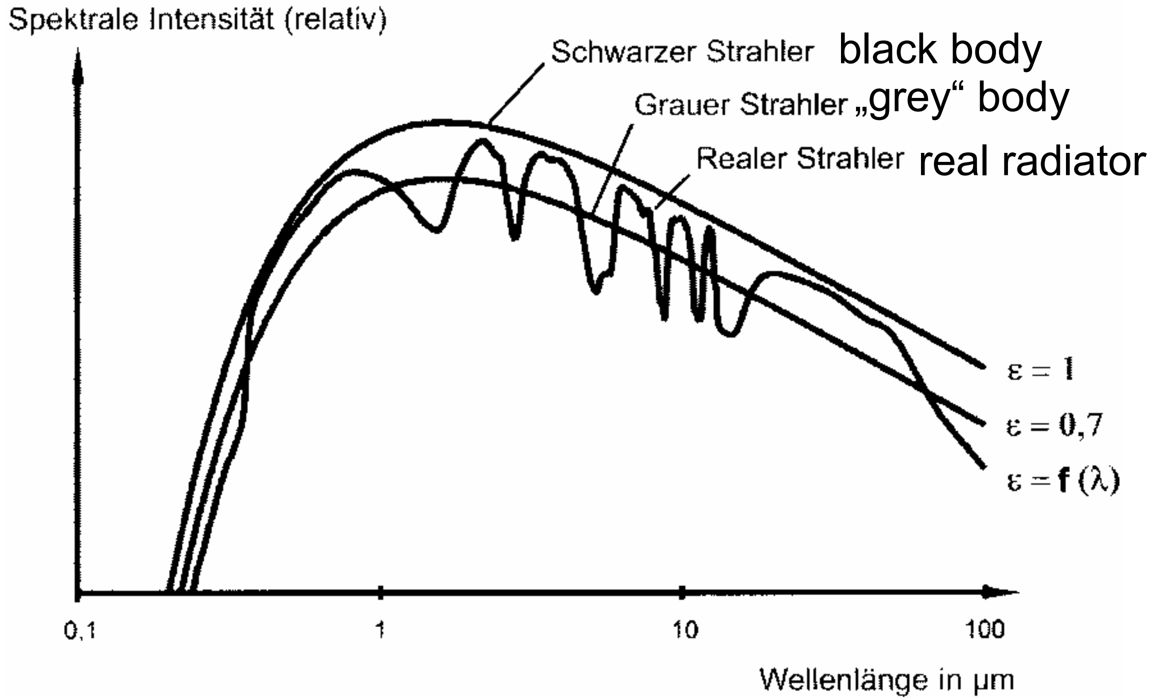
Cavity radiator (black-body)

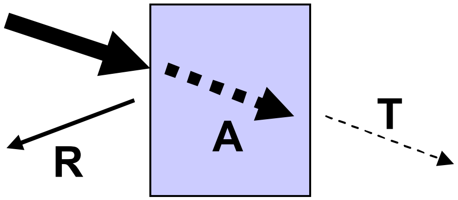
black-body radiation



Kirchhoff:
emissivity = absorptivity
$\varepsilon(\lambda) = \alpha(\lambda)$
black body:
ε = const = 1
„grey body“
ε = const < 1
„real“ or „colored“ radiator
$\varepsilon = f(\lambda)$

attention with transparent materials!





$\alpha + r\ (+ t + \text{lum}) = 1$

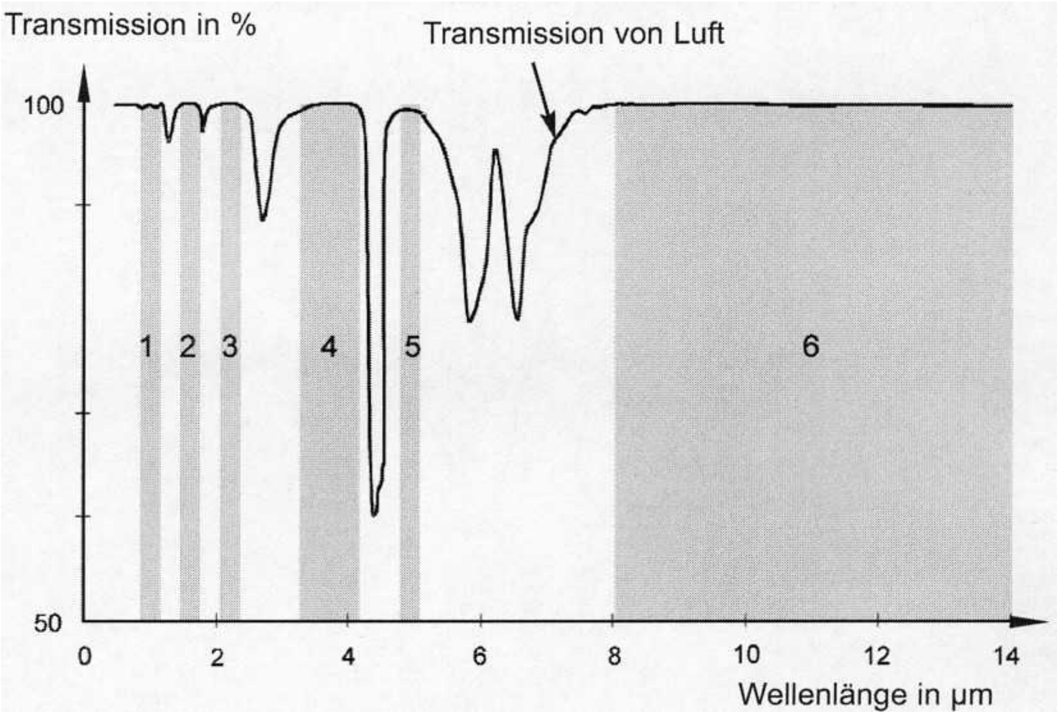
If r is small: $\alpha\ (=\varepsilon)$ is high
high: low

$P = e\, P_s$

emission power = emissivity x black-body emission power

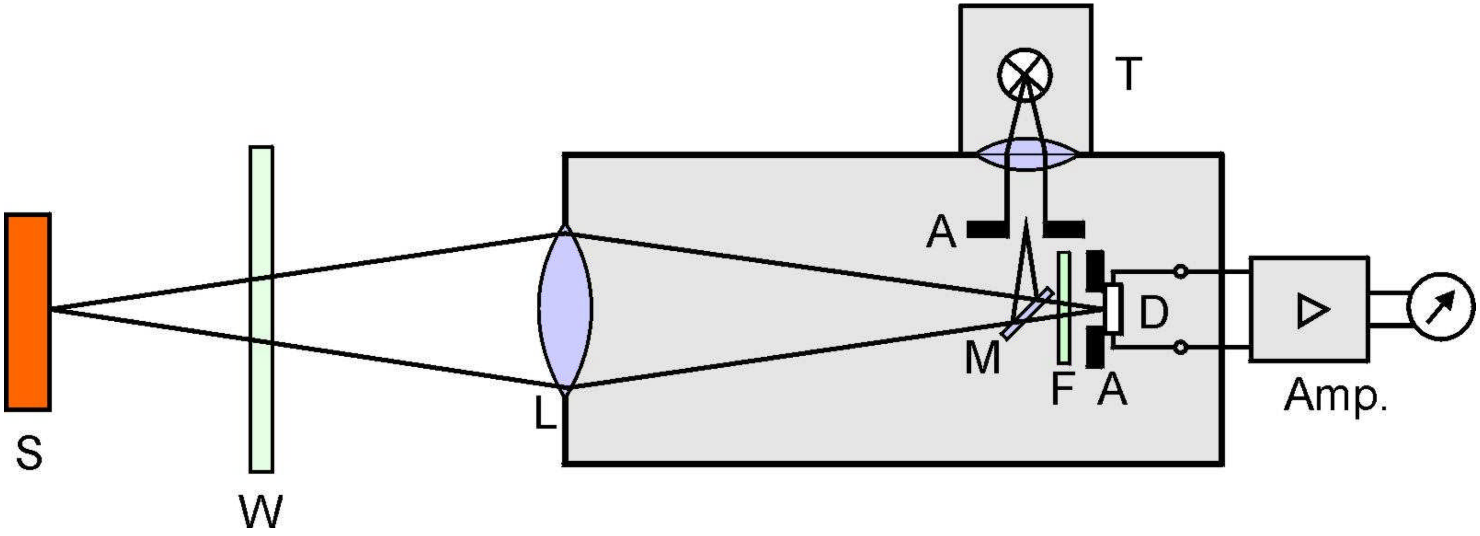
T-measurement - Pyrometer

„Atmospheric windows“
In order to avoid absorption effects of the air in the light path, partial radiation pyrometers use regions without absorption.



Window	Used detector type
1	Si diode
2	Ge-diode
3	PbS-diode
4	PbSe-diode, bolometer (resistance change) thermopile (ser. of thermocouples)
5, 6	bolometer (resistance change) thermopile (ser. of thermocouples)

T-measurement - Pyrometer



must be transparent for the used wavelength!

S	sample
W	window
L	lens
M	semitransparent or removable mirror
F	filter, attenuator
A	apertures
D	radiation detector
T	lamp for sighting

T-measurement - comparison

	Resistance thermometer	Thermocouple	Pyrometer
Advantages	exact almost linear wide T-range	very small very fast very wide T-range easy to make self	non-contact very high T quite fast
Disadvantages	not very small not very fast $\tau \sim 1$ s	not linear esp. at low T	only for T>~400°C line of sight necessary emissivity-problem => low precision
Price	medium	lowest	high

pressure

Pressure measurement

This lecture does not deal with:

- how to make vacuum (pumps)
- how to make a vacuum device (materials)
- gas flow and flow ranges

(see class 2002/2003, ask for manuscript)

Pressure, definition:

p = F/A = force / area (N m^{-2})

1 N m^{-2} = 1 Pa;
10^5 Pa = 1 bar
100 Pa = 1 mbar

1 Torr = 1 mm Hg = 1.333 mbar

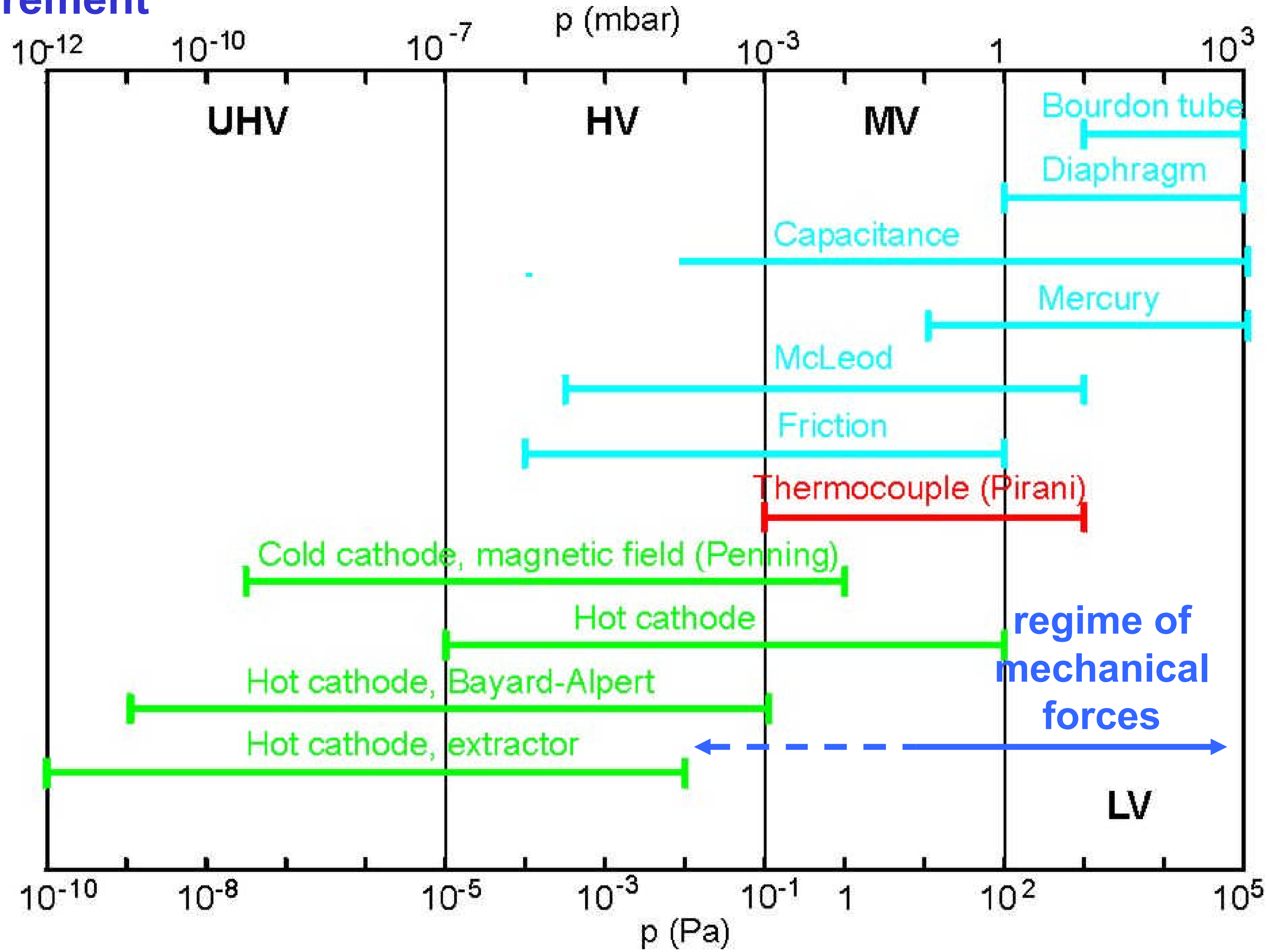
Pressure measurement

LV low vacuum
(Grobvakuum)

MV mean vacuum
(Feinvakuum)

HV high vacuum
(Hochvakuum)

UHV ultrahigh vac.
(Ultrahochvak.)



Pressure ranges for different vacuum gauges.

Blue: Direct measurement of force.

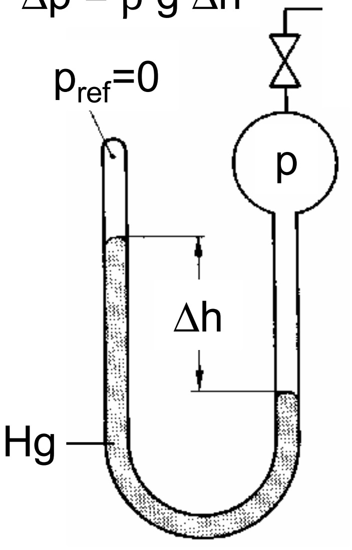
Red: Indirect, p-dependence of thermal conductivity.

Green: Indirect, p-dependence of ion current in electrical discharge.

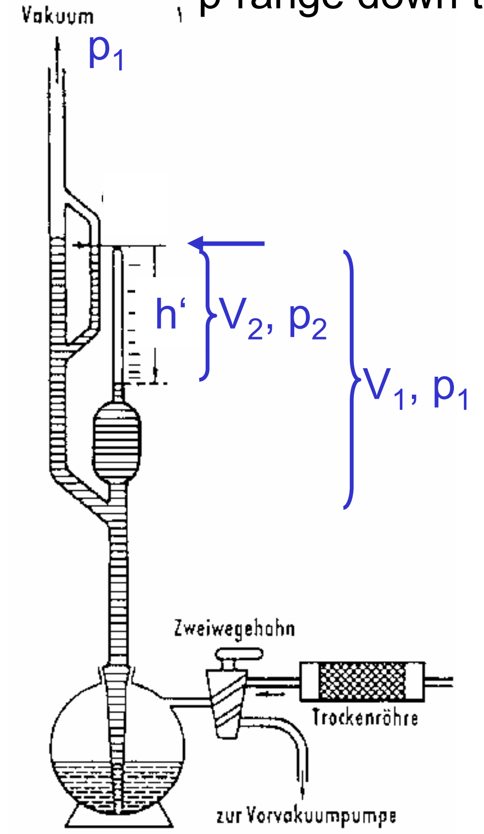
p-measurement - Direct measurement of mechanical force

Hg U-tube
- absolute p
- no gas dependence
- limited precision for $p<10$ mbar

$$\Delta p = \rho\, g\, \Delta h$$



Hg-compression gauge (McLeod)
- absolute
- no gas dependence (gas condensation!?)
- p-range down to ~10^{-5} mbar



How to measure:
1. All Hg in reservoir p_1 in volume V_1
2. Pump up Hg until arrow V_1 gets compressed to V_2, p_1 rises to p_2

Acoording to

$p\,V = \nu\, R\, T = \text{const.}$:

$p_1\, V_1 = p_2\, V_2$

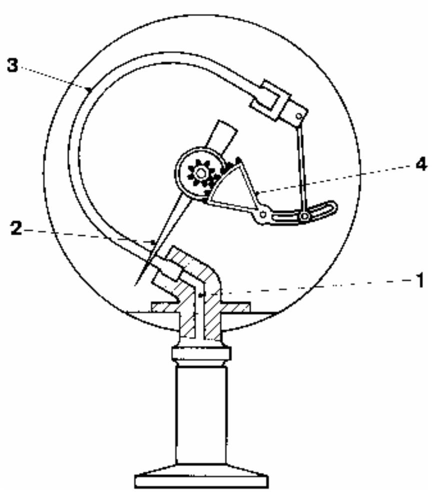
$p_2 = p_1\, V_1/V_2 >> p_1$

Primary standard for p-calibration!

p-measurement - Direct measurement of mechanical force

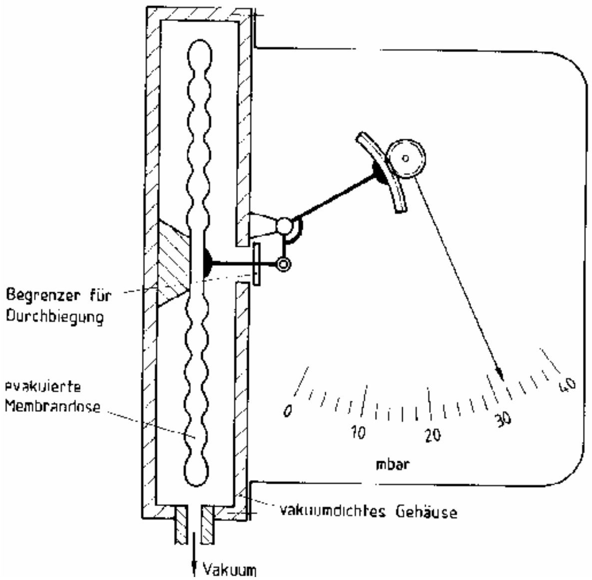
mechanical pressure indication

Bourdon tube



p > 10 mbar

Diaphragm



Mechanical barometer

p > 1 mbar

So far:
not well suited for process control.

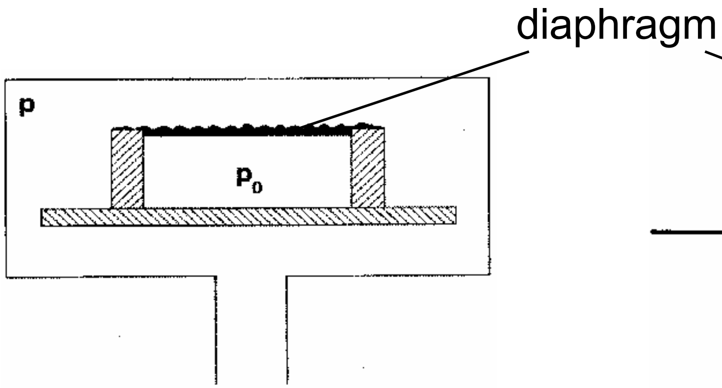
Wanted:
Electrical output signals

p-measurement - Direct measurement of mechanical force

Piezoresistive diaphragm:

Diaphragm: Semicond.

Si diaphragm, with deformation dependent R-bridge



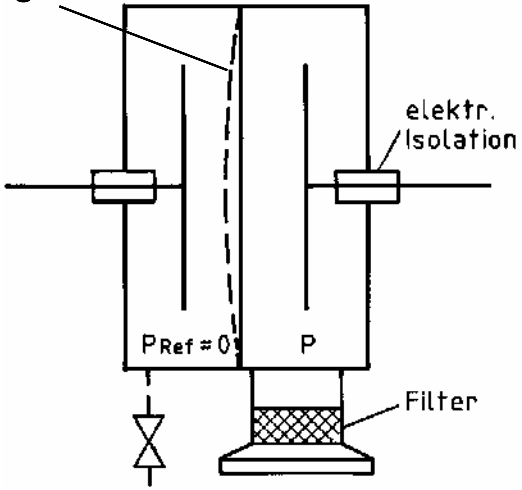
Advantage:
- simple, robust
- insensitive to high p
- gas-independent
- also for higher p available

Problems:
- limited precision

Range: 0.1 – 55 000 mbar
Precision:
+/- 10 mbar

Capacitance diaphragm:

Diaphragm: ceramics or stainless steel



Dp deforms diaphragm and changes the capacitance, electricval measurement

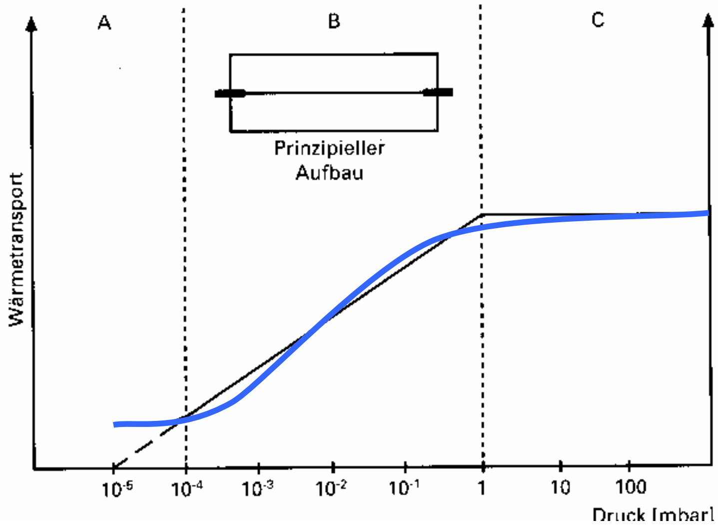
Advantage:
- simple, robust
- insensitive to high p
- high precision

Problems: no

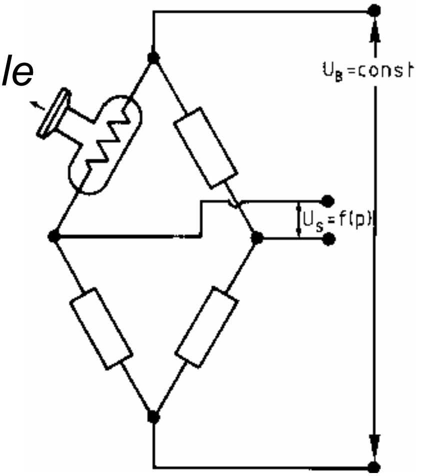
Range: 10^{-4} – 1000 mbar
Precision:
0.15% typically

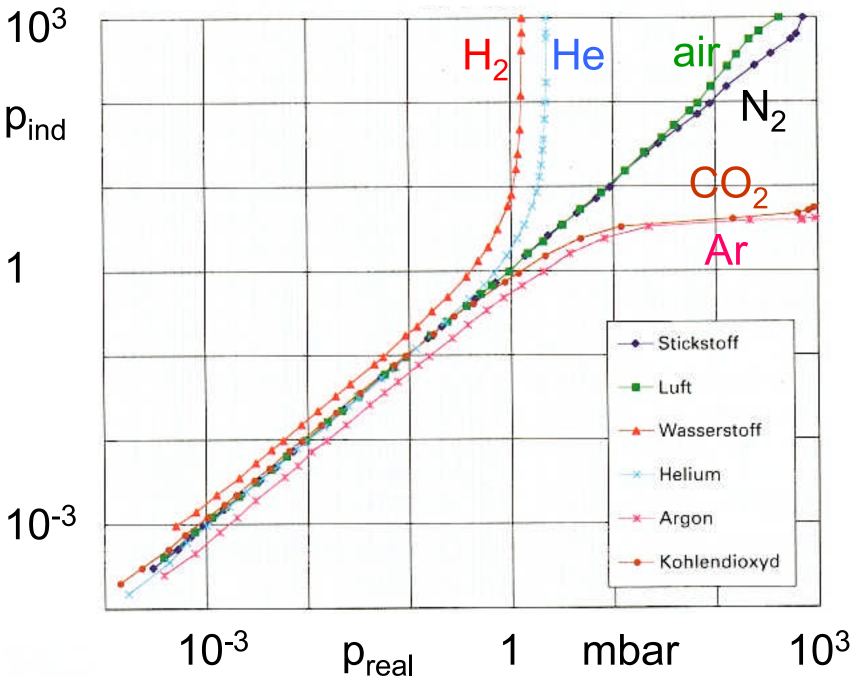
p-measurement - Indirect measurement, heat conductivity

Pirani



Principle





A wire is heated and at the same time its R is measured. The T and hence the R depends on cooling by heat conduction through the gas.

gas dependent pressure indication

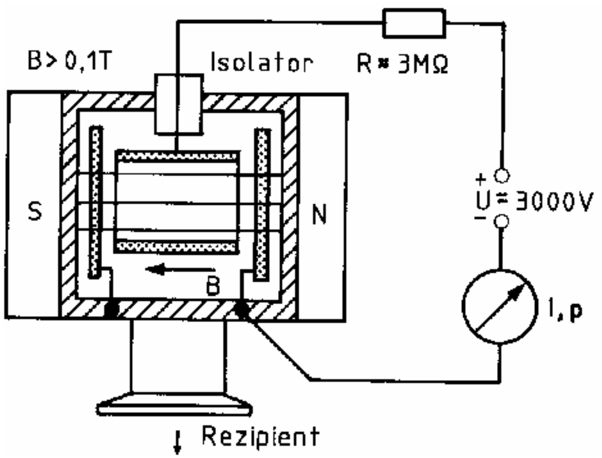
Advantage:
- simple, robust
- insensitive to high p

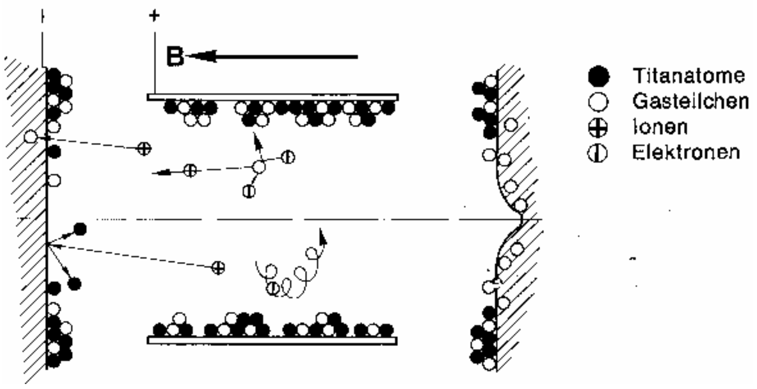
Problems:
- gas dependent
- highly non-linear p-characteristics
- low precision for $p < 10^{-3}$ mbar and $p > 10^{-1}$ mbar

Range: 10^{-3} – 10 (100)
Precision:
5% at 10^{-3} mbar
(+ gas dependence!)

p-measurement - Indirect measurement, gas ionization

Cold cathode, inverse magnetron, **Penning**





working principle:
like in ion getter pump (diode type):
each Penning pumps!

Incidentally produced ions move to anode, electrons to cathode. Electrons are forced on long spiral paths by B-field in order to increase the probability to produce further ions by impact. The ion/ electron current is proportional to the gas density and thus p.

Advantage:
- simple, robust
- insensitive to high p

Problems:
- gas dependent
- ignition
- maintain discharge at low p
- leak currents (contamination)

Range: $(10^{-10})\ 10^{-8} – 10^{-2}$
Precision:
low +/- 30% at
(+ gas dependence!)

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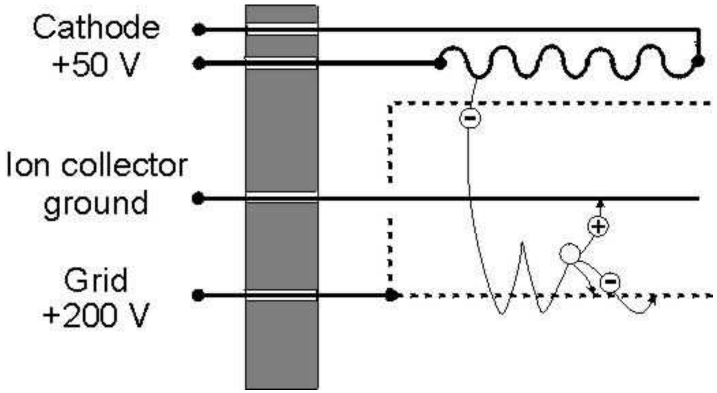
Hot cathode, **Bayard-Alpert**-type



The hot cathode emits electrons which pass the grid several times before they hit it. They ionize particles.
Ions are collected by a thin wire (collector).
X-ray limit:
e^- generate X-rays when hitting the grid which generate a photoionization current from the collector.
Its size determines the low-p limit.

Advantage:
- linear for $p < 10^{-4}$ mbar
- wide range $10^{-3} – 10^{-11}$ mbar

Problems:
- gas dependent
- cathode burns at $p > 10^{-2}$ mbar (safety circuit necessary)

Range: $3\text{x}10^{-11} – 10^{-3}$
Precision:
rel. high, +/-10% at 10^{-7} mbar
(+ gas dependence!)

More information
=> separate sheet

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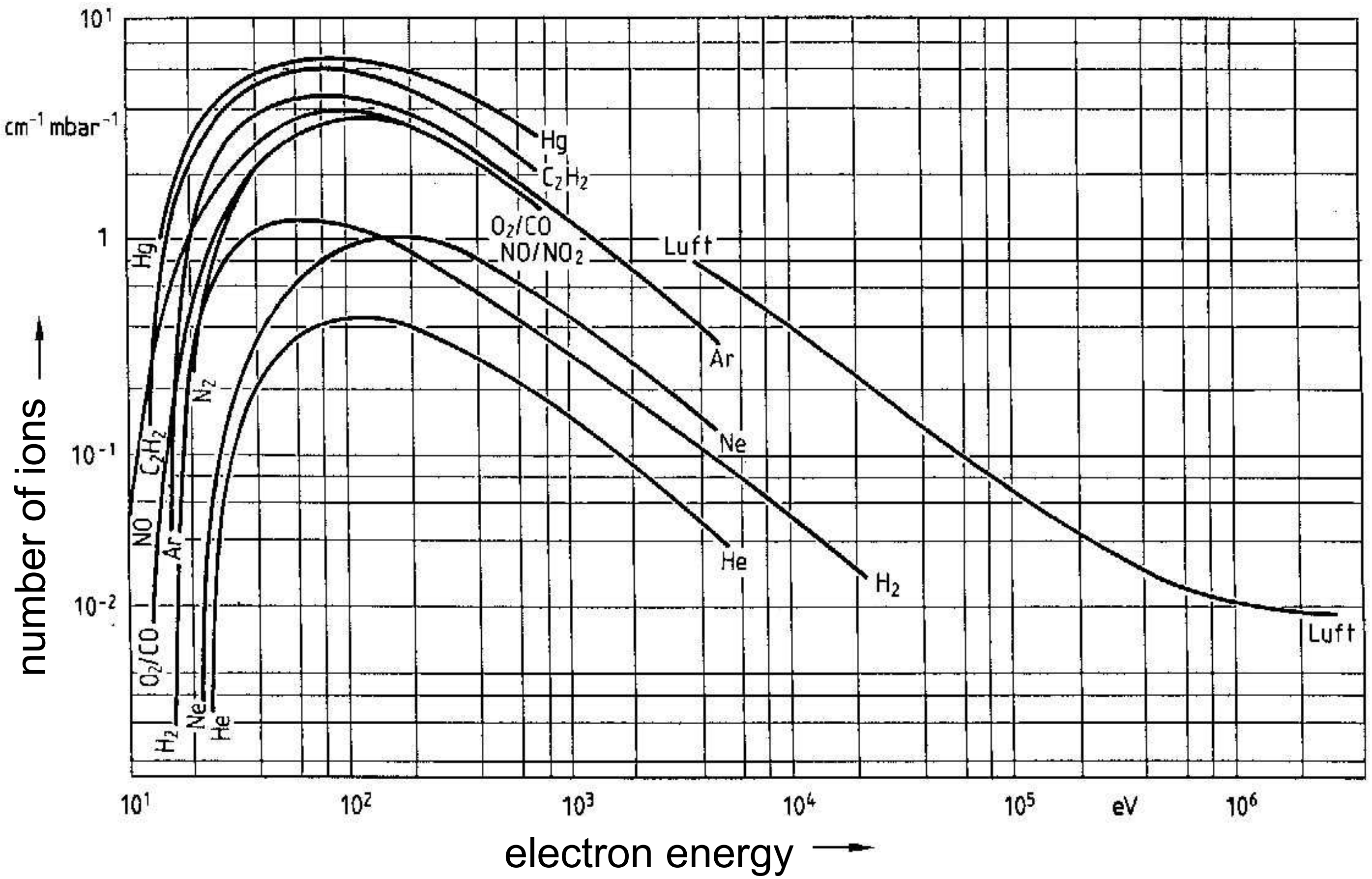


Molecule	ion gauge sens. S
He	**0.19**
H_2	0.44
N_2	**1**
CO	1.02
H_2O	1.25
Ar	1.37
CH_4	1.49
C_2H_6	2.53
C_6H_6	**5.18**

F. Nakao, Vacuum 25 (1975) 431)

The energy needed for ionization depends on the kind of the gas (ionization potential) and determines the onset of the curves. The maximum ionizability occurs for all gases at around 100 eV

Consequence for p-measurement:

- need to know gas composition
- divide p-indication by sens. factor $p = p_{indic}/S$

p-measurement - gauge combinations

For wide p-ranges and process control:
- combination of two principles as one unit („Full Range“ or „Wide Range“ gauges)
- automatic switching between gauges
- digital indication

Examples:

Piezo + Pirani
(diaphragm) (heat cond.)
1200 mbar – 10^{-3} mbar

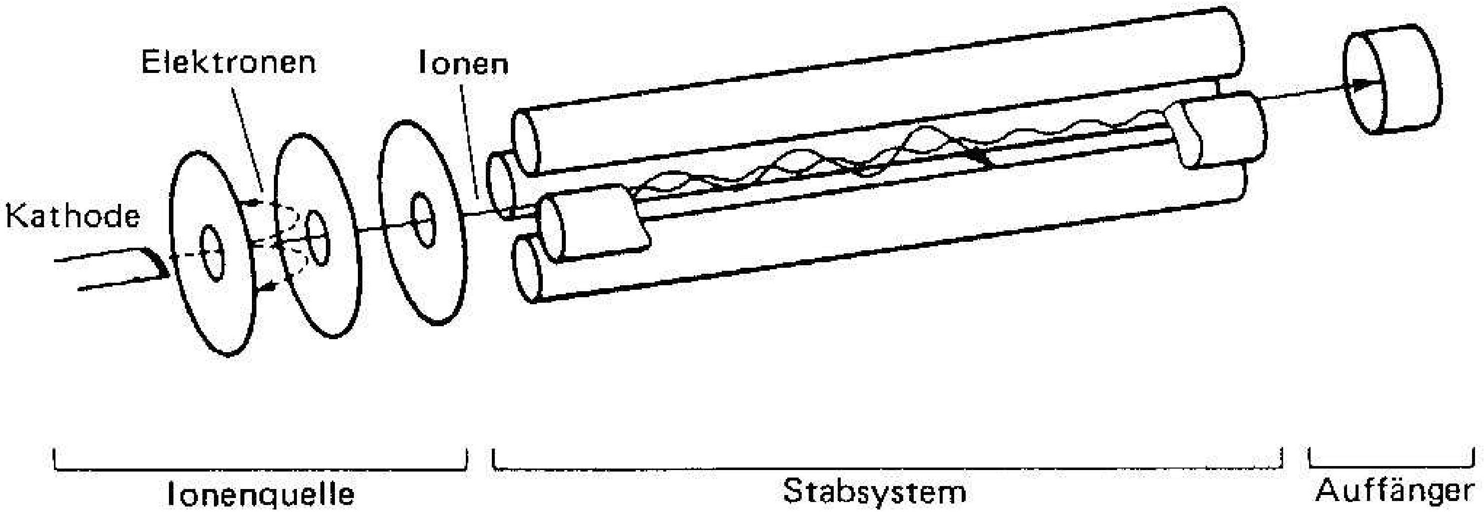
Pirani + Penning
(heat cond.) (cold cathode)
100 mbar – 10^{-8} mbar

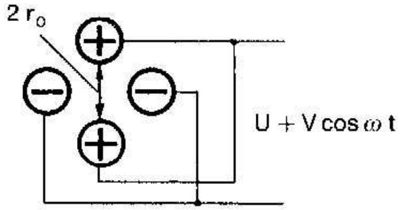
Pirani + Bayard-Alpert
(heat cond.) (hot cathode)
100 mbar – 10^{-11} mbar

Be careful:
digital indication
makes you believe
in high precision!

The actual pressure
may be up to +/-30%
wrong due to
the measuring principle
and in addition
depends mostly
on the kind of gas!

p-measurement - quadrupole mass spectrometer (QMS)





Quadrupole rod system,
applied DC voltage U
and AC voltage V cos Wt

QMS consisting of ionizer, mass separator (rod system) and collector (Faraday cup or multiplier).
Particles are excited to vibrations in the separator.
For given values of U and V, the amplitude for a particle with certain m/e remains limited and the particle is transmitted.
All others are in resonance, hit a rod and are neutralized.

T and p:

Don‘t simply believe what your instruments indicate!

T: Sensor - type?
T-range?
linearity?
mounting?

p: Sensor - type?
cleanliness?
mounting -
degassing of surrounding?