RISE METROLOGY

Fredrik Arrhén

22 October 2019

Research Institutes of Sweden
Measurement Science & Technology



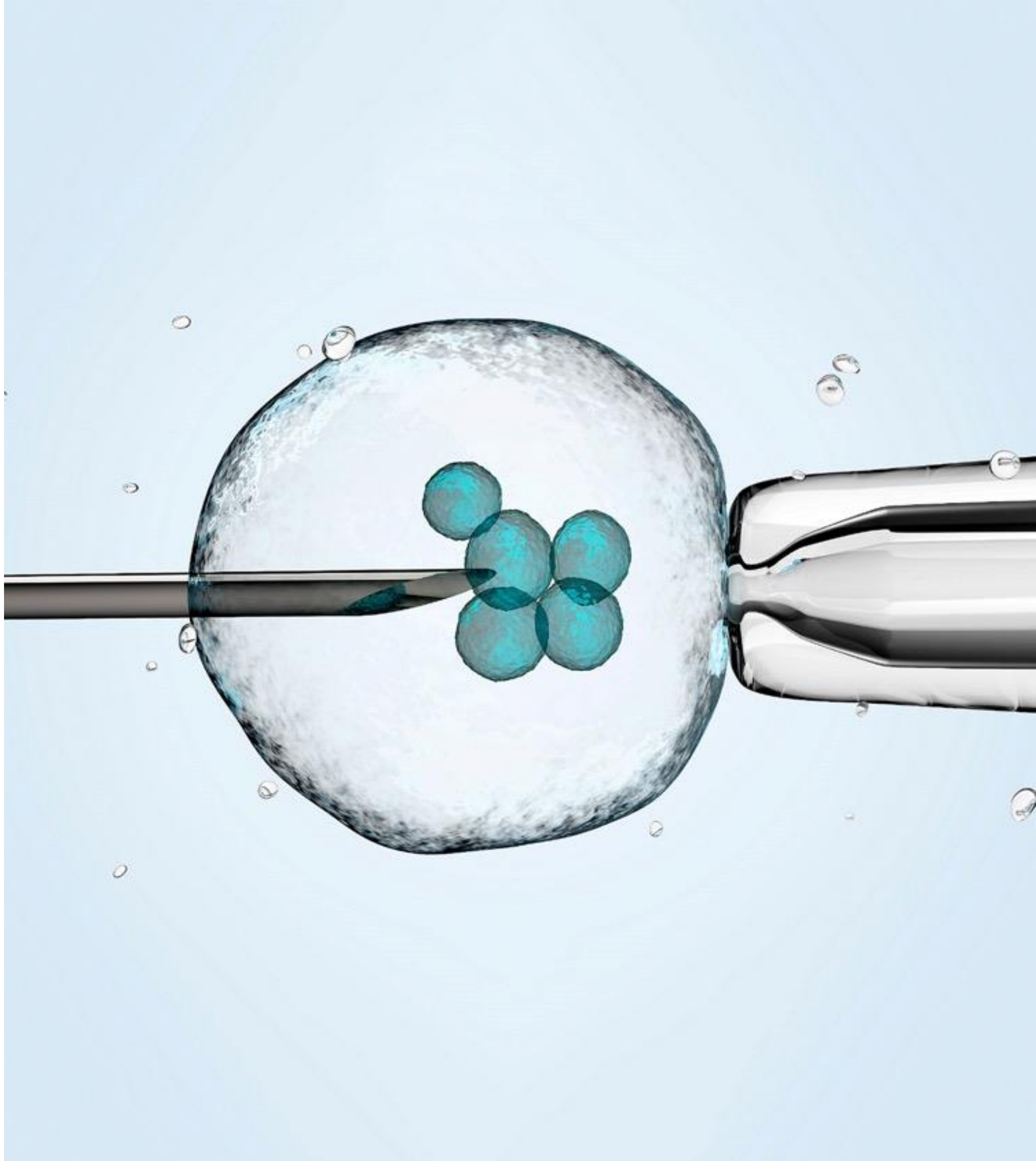
RISE´s Mission
from the Swedish Government

“The industrial research institutes shall be internationally competitive and facilitate sustainable growth in Sweden by strengthening competitiveness and renewal in the business community.”

Excerpt from the Research Bill 2016/17: 50 (Kunskap i samverkan).

One strong, unified institute for Sweden

- Sweden needs a strong, national innovation capacity to compete on the international stage and to meet major global challenges.
- The new RISE aims to build a stronger Swedish institute sector that will actively support Swedish industry, providing increased benefits for trade and industry, and society in general.



RISE in brief

- Present across the whole of Sweden. And beyond.
- 2,700 employees, 30 % with a PhD.
- Turnover approx. SEK 3 billion (2018).
- A large proportion of customers are SME clients, accounting for approx. 30 % industry turnover.
- Runs 100s of test and demonstration facilities, open for industry, SMEs, universities and institutes (RISE is owner and partner in 60 % of all Sweden's T&D facilities).

RISE testbeds and demonstration facilities

- **Unique infrastructures** for research, development and verification on lab and pilot scale
- **Physical or virtual environments** where businesses, academia, research institutes and the public sector can collaborate
- **Equipment** adapted for industrial applications with **qualified operators and technicians**
- **Expertise** in research, industrial applications and project management
- RISE owns, and has partnerships in, **more than 100 unique test beds and demonstration facilities.**

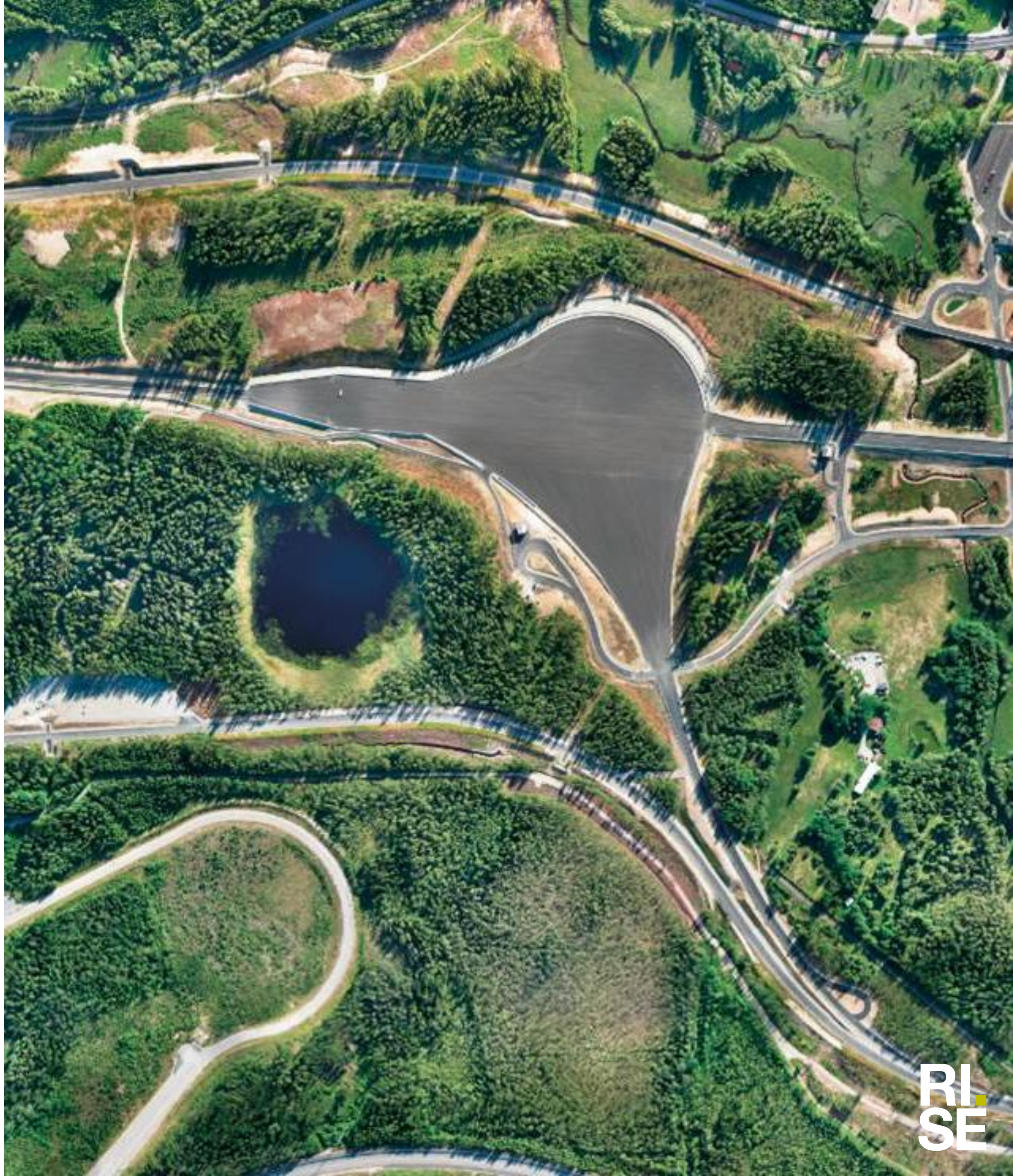
Awitar – a unique testing facility for autonomous vehicles

- The complexity of the vehicles on our roads – equipped with radar, cameras, and sensors – is growing rapidly. The disruptive testing of automotive electronics is therefore increasingly important and a prerequisite for transitioning to future autonomous vehicles, which must be able to communicate wirelessly with one another

- Awitar has been developed in close cooperation with the Swedish automotive industry. The new test facility for future vehicles is unique in its capacity and flexibility.

AstaZero – a test facility for future traffic environment in real size

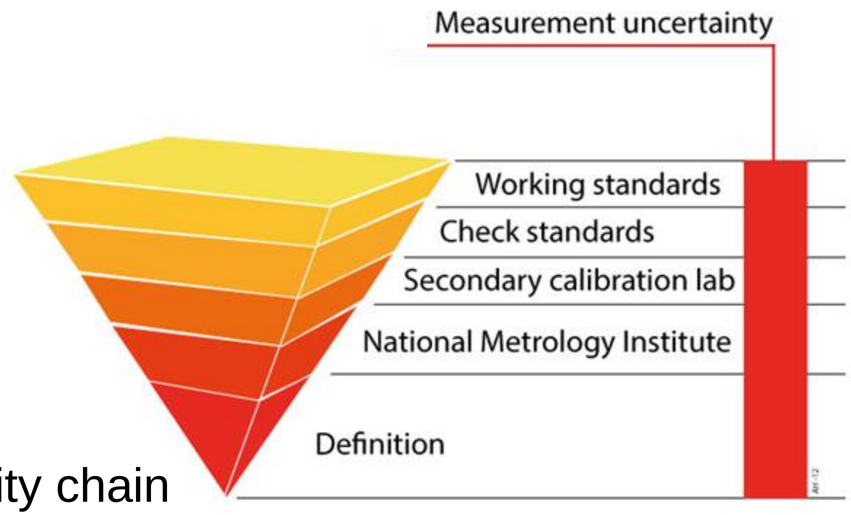
- A innovation and test facility for development and demo of advanced safety technology for vehicles and infrastructure
- Here, self driving vehicles are tested and companies, research institutes and universities can test and examine questions regarding infrastructure, city planning and safety.



RISE Metrology

RISE Metrology facts:

- >130 employees
- 15 technology groups
- ~20 laboratories
- Turnaround ~15 M€
- ~390 CMC entries in 13 areas



Traceability chain

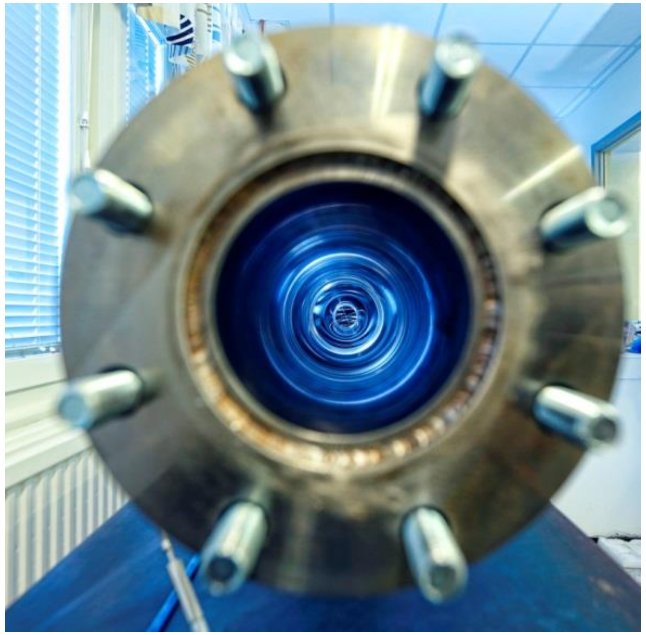
- **Mass**
- **Length**
- **Volume**
- **Pressure**
- **Force**
- **Acceleration**
- **Sound pressure**
- **Time / Frequency**
- **Electricity**
- **Photometry / Radiometry**
- **Temperature**
- **Chemical measurement technology**

EMPIR project with RISE participation

- The annual EMPIR calls for 2014-2018 have resulted in RISE participating in a total of 35 research projects
- Coordinator of two EMPIR projects in high voltage metrology
- Extensive activities in calls related to
  - Energy (ENG)
  - SI Broader Scope (SIB)
  - Industry (IND)
  - Pre-normative (NRM)
- Active participation in about 7 European Metrology Network (EMN) already existing established or under establishment

Project	Acronym	Coordinator	RISE contact
14IND06	Pres2vac	PTB	Zelan
14IND08	**Elpow**	**RISE**	**Bergman**
14IND10	MET5G	VSL	Hedekvist
14IND12	Innanopart	NPL	Ringstad
15HLT04	NeuroMet	LGC	Pendrill
15SIB04	QuADC	PTB	Bergsten
15SIB05	OFTEN	PTB	Hedekvist
15SIB07	PhotoLED	MIKES	Källberg
15NRM02	**UHV**	**RISE**	**Elg**
15NRM03	Hydrogen	LNE	Arrhenius
15RPT01	RFMicrowave	UME	Lundgren
15RPT04	TracePQM	CMI	Tarasso
16ENG01	MetroHyVe	NPL	Arrhenius
16ENG05	Biomethane	VSL	Arrhenius
16ENG08	MICEV	INRIM	Welinder
16ENV08	IMPRESS 2	NPL	Gustavsson
16NRM02	SURFACE	INRIM	Lindgren
16NRM04	MagNaStand	PTB	Johansson
16NRM05	Ion gauge	PTB	Zelan
16NRM08	BiRD	LNE-INM	Källberg
17IND03	LaVA	NPL	Bergstrand
17IND07	DynPT	MIKES	Arrhén
17IND10	LiBforSecUse	PTB	Tarasso
17IND13	Metrovamet	PTB	Büker
17IND14	WRITE	INRIM	Rieck
17NRM01	TrafoLoss	VSL	Bergman
17SIP05	CASoft	LNE	Pendrill
18HLT08	MeDDII	IPQ	Büker
18HLT09	NeuroMET2	LGC	Melin
18SIB01	GeoMetre	PTB	Bergstrand
18SIB03	BxDiff	LNE	Källberg
18SIB04	QuantumPascal	PTB	Zelan
18SIB06	TiFOON	NPL	Hedekvist
18SIB07	GIQS	PTB	Bergsten
18SIB08	ComTraForce	PTB	Wozniak

Dynamic metrology of mechanical quantities

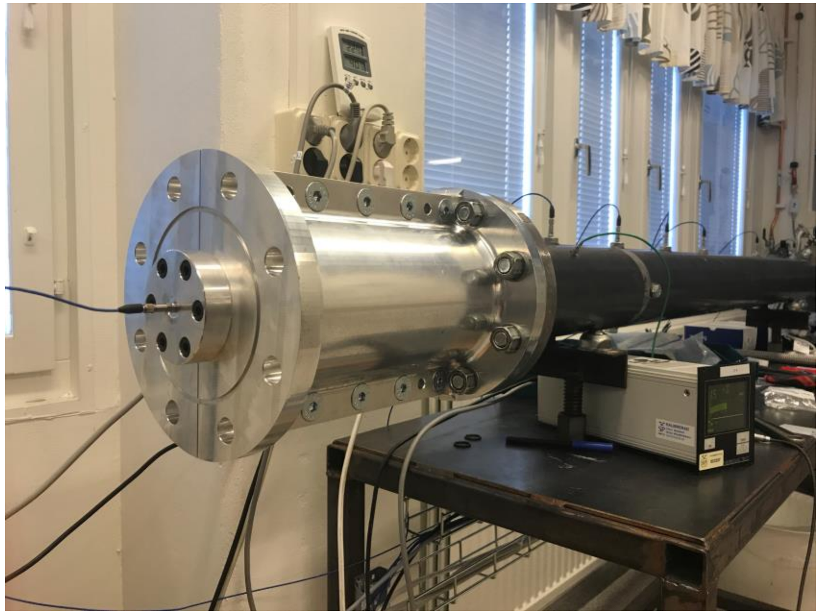


Challange:

Traceability is not available for reliable measurements of dynamic pressure, force and torque. For pressure, we are currently using a shock tube as signal source and hopefully also as primary standard. The challenge is understanding the shock spectra and optimize the dimensions.

Our tasks:

- Develop primary methods in pressure in collaboration with KTH (Stockholm) and ENSAM (Paris) within EMPIR 17IND07 DynPT
- In collaboration with the industry develop the traceability chain from the primary methods to industrial applications in research and production.
- Develop analysis methods including measurement uncertainties and suitable ways to distribute the data to end users.
- Develop new sensors in the area.
- Use the experience and knowledge acquired in pressure in force and torque, i.e. by participating in EMPIR a8SIB08 ComTraForce.



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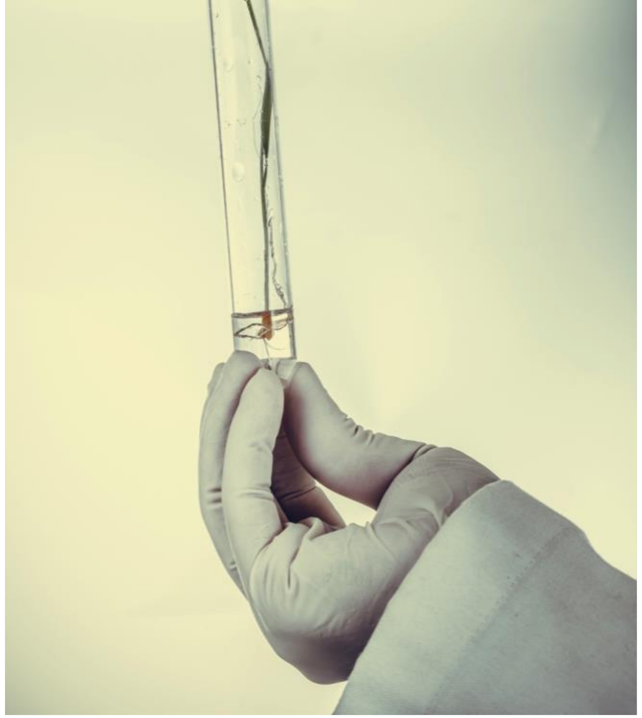
BASIC CONCEPTS IN VACUUM TECHNOLOGY

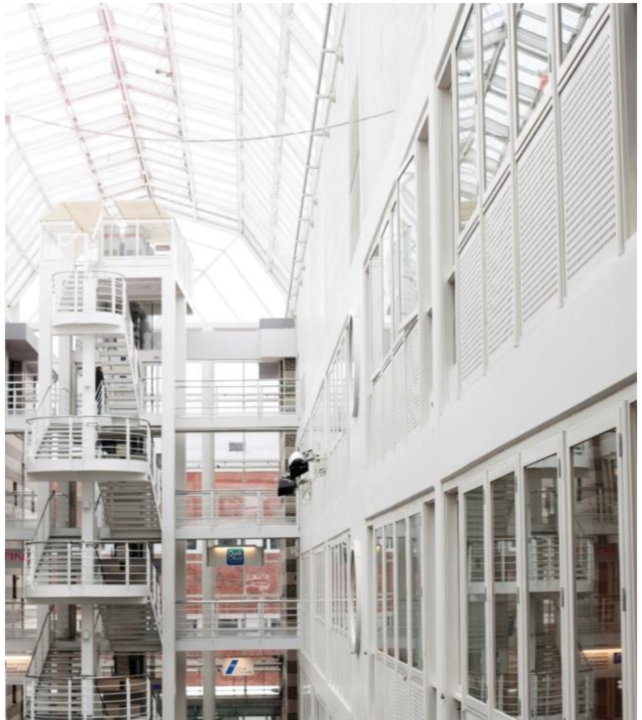
Fredrik Arrhén

22 Oktober 2019

RISE Research Institutes of Sweden

Säkerhet och transport
Mätteknik





Introduction

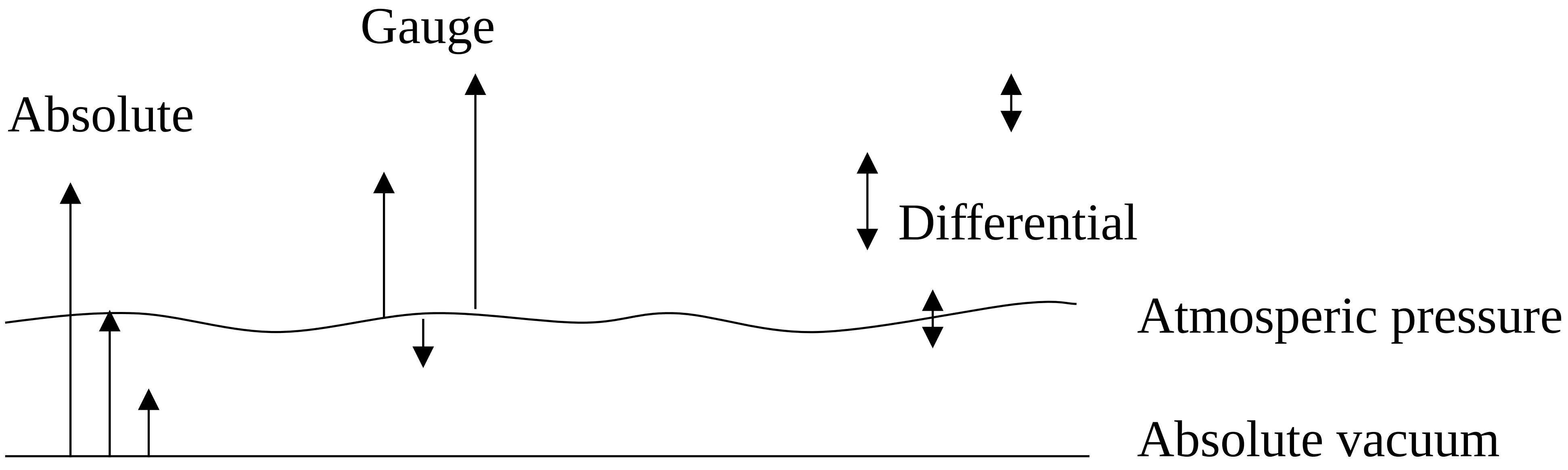
Absolute pressure: **Pressure with zero level at absolute zero.**

Gauge pressure: **Pressure with zero level at atmospheric pressure**

Differential pressure: **Difference between two arbitrary pressure levels**

Normal atmospheric pressure is 1013 hPa

At 20km above earth, the pressure is about 100 Pa



Pressure units

Pascal	**Pa**	**1 Pa**
Bar	**bar**	**100 000 Pa**
Hektopascal	**hPa**	**100 Pa**
millibar	mbar	100 Pa
Millimeter Hg (Torr)	mmHg	133,322 Pa
millimeter Vp	mmVp	≈ 9,8 Pa
normalatmosfär	atm	101 325 Pa
”kg per cm^2”	Kgf/cm^2	98 066,5 Pa

Use of vacuum

Using vacuum:

- Stops or minimize chemical reactions due to removal of active gases like oxygen
- Accelerate emission of captured gases and liquids.
- Generates forces due to pressure differences that can be used for transport of goods or fixing objects to a surface.
- ...

Inledning

Den gasblandning vi har i ett kärl kallas gaslast (Q = P*V).

Gaslasten beror på:

- urgasning ur vakuumsystemet
- urgasning ur material
- läckor

Vacuum ranges

Pressure range	Pressure hPa	Pressure Pa	Number density per cm^3	Mean free path in m
Atmospheric pressure	1,013.25	101,325	$2.7 \cdot 10^{19}$	$6.8 \cdot 10^{-8}$
Low vacuum (LV)	300…1	30,000…100	10^{19}…10^{16}	10^{-8}…10^{-4}
Medium vacuum (MV)	1…10^{-3}	100…10^{-1}	10^{16} …10^{13}	10^{-4}…10^{-1}
High vacuum (HV)	10^{-3}…10^{-7}	10^{-1}…10^{-5}	10^{13}…10^{9}	10^{-1}…10^{3}
Ultra-high vacuum (UHV)	10^{-7}…10^{-12}	10^{-5}…10^{-10}	10^{9}…10^{4}	10^{3}…10^{8}
Extremely high vacuum (XHV)	$<10^{-12}$	$<10^{-10}$	$<10^{4}$	$>10^{8}$

Ideal gas

An ideal gas have some properties not existing in real gases:

- **A very large amount of molecules moving randomly with different speed and in different directions.**
- **The total volume of the molecules is very small compared to the total volume of the gas.**
- **The molecules only interact with ach other at the collisions. The then acts as elastic balls.**

Ideal gas laws are quite easy to use and works as rough estimates for gas behavioour

Real gases

Real gases deviates from ideal gas behaviour in some ways:

- **The total volume of the molecules is not insignificant compared to the total volume**
- **The molecules interacts not only in direct collisions**

These deviations from ideal gas behaviour is corrected by the use of “virial coefficients” in the gas laws.

Useful relations

Boyle's law, keeping temperature and amount of gas constant , the following relation is valid:

$$P \cdot V = \text{constant}$$

Charles' law, for a given mass of an ideal gas at constant pressure:

$$P \sim T$$

- Combining these and some more relations leads to **the ideal gas law**

The ideal gas law

Amount of substance

Absolute pressure

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

Absolute temperature

Volume

Universal gas constant

Total mass

Alternative version:

$$p \cdot V = \frac{m}{M} \cdot R \cdot T$$

Molar mass

Partial pressure

The total pressure is the sum of the partial pressure of all gas components

$$p_{total} = \sum p = p_1 + p_2 + \ldots + p_n$$

Partial pressure in air

Gas	Partial pressure (Pa)	Partial pressure (mbar)	Volume (%)
Nitrogen, N_2	$79,1 * 10^3$	791	78
Oxygen, O_2	$21,2 * 10^3$	212	21
Argon, Ar	960	9,6	0,9
Carbon dioxide, CO_2	40	0,4	0,04
Neon, Ne	1,9	$1,9 * 10^{-2}$	
Helium, He	0,5	$5,0 * 10^{-3}$	
Krypton, Kr	0,101	$10,1 * 10^{-4}$	
Hydrogen, H_2	0,05	$5,0 * 10^{-4}$	
Xenon, Xe	0,009	$9,0 * 10^{-5}$	

Vacuum ranges

Pressure range	Pressure hPa	Pressure Pa	Number density per cm^3	Mean free path in m
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Pressure regions

Low vacuum:	The mean free path is much smaller that the dimensions of the chambers. Most collisions is between molecules, neglectable interaction with chamber walls. The gas acts as a normal media.
High vacuum:	The mean free path is much bigger than the dimensions of the chambers. Almost no interaction between gas molecules but mostly with chamber walls. Normal gas laws does not apply. The gas acts as particles.
Medium vacuum:	The mean free path is in the same region as the dimensions of the chambers. The gas acts as neither a pure media, nor as particles but something in between. Transition range. Special methods needed to calculate flow.

Clean systems

Importance of clean process and not touching inside of vacuum components:

Assuming:

- one fingerprint weights about 1 mg
- The components have a molar mass close to carbon dioxide, 1.6 kg/m3 at 100 kPa and 0°C

In a 10 l volume this will give a pressure of ~6 Pa

Material choises

	Metals	**Insulators, seals**
Good:	Stainless steel AL 6000 series	Glass, keramer
Decent:	Al, Cu, Ni	Silikone, viton
acceptable:	Fe, Sn, Pb	Plastic, polymers
Bad choise:	Zn, brass	Grease, fingerprints

MEASURING VACUUM

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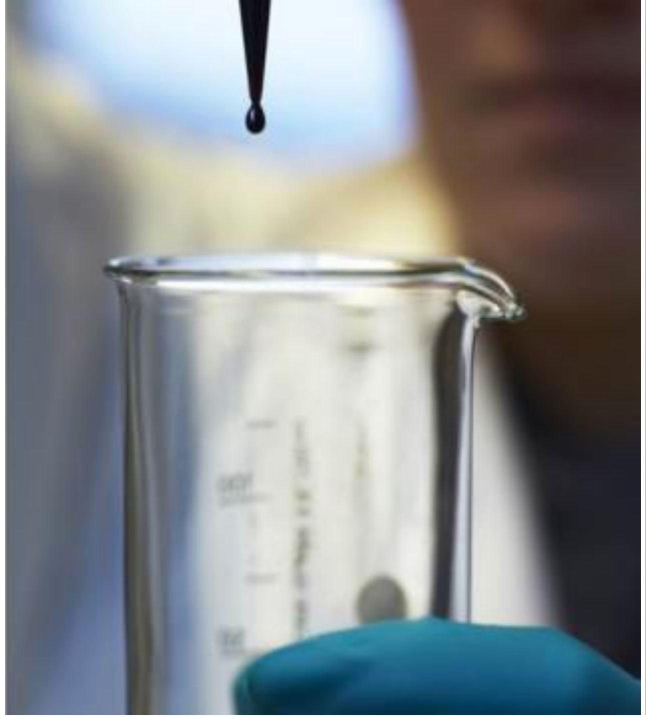
22 Oktober 2019



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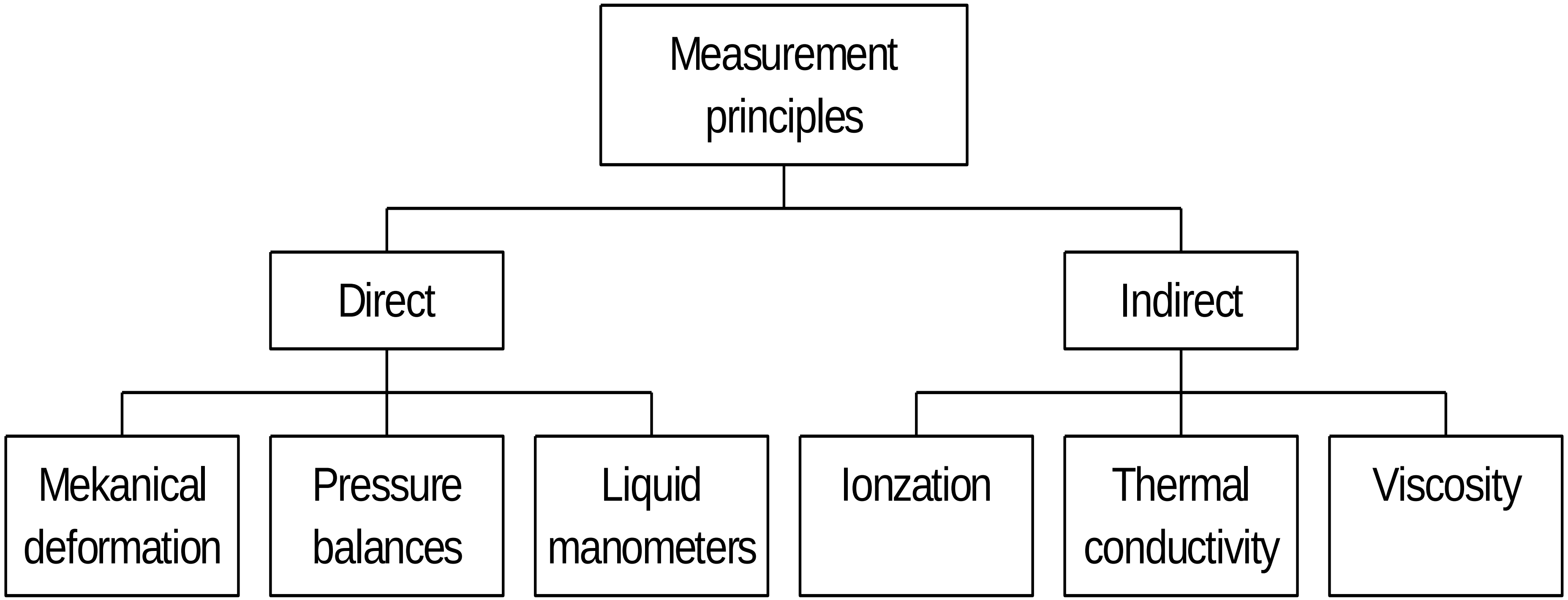
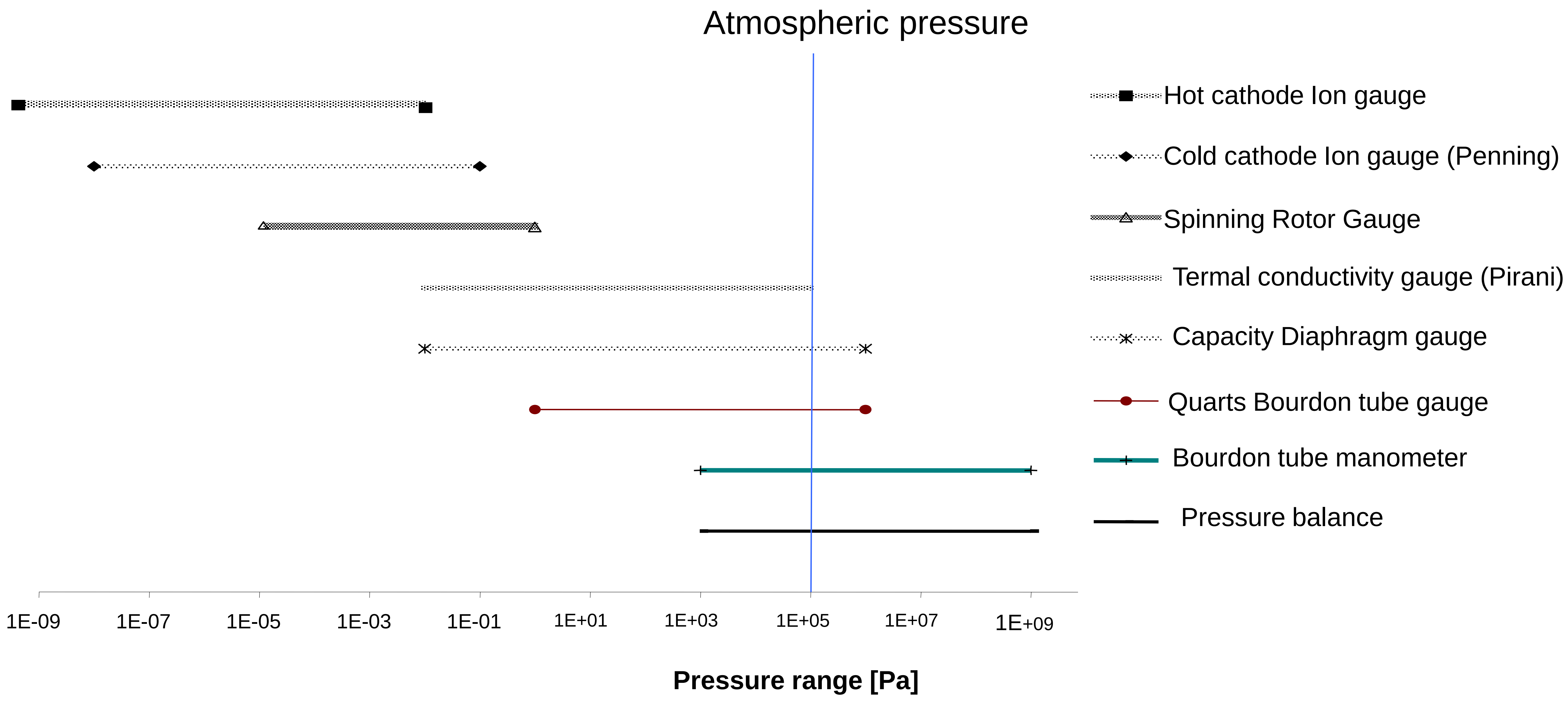
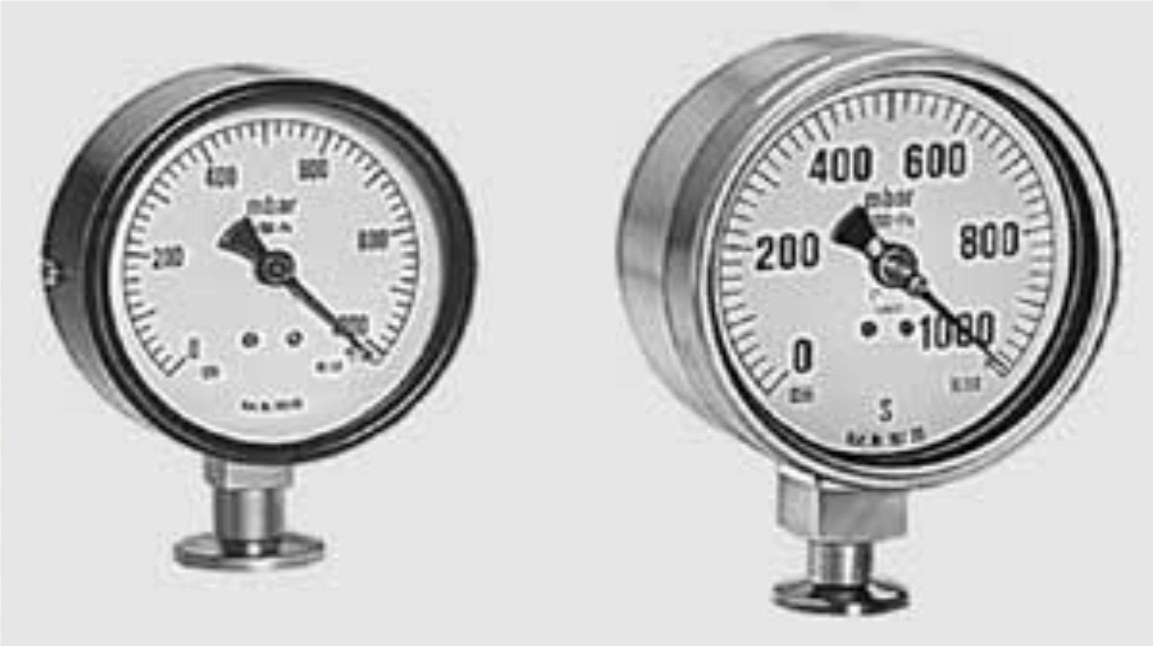
Different measuring principles



Measuring range for different vacuum gauges

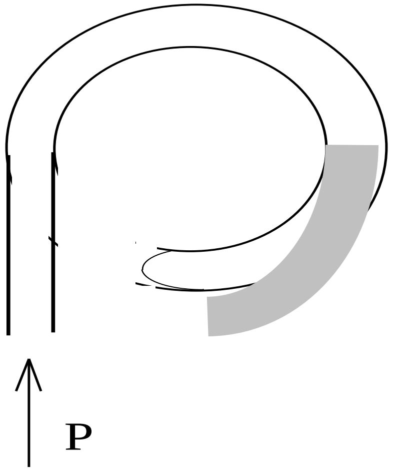


Bourdon tube gauge



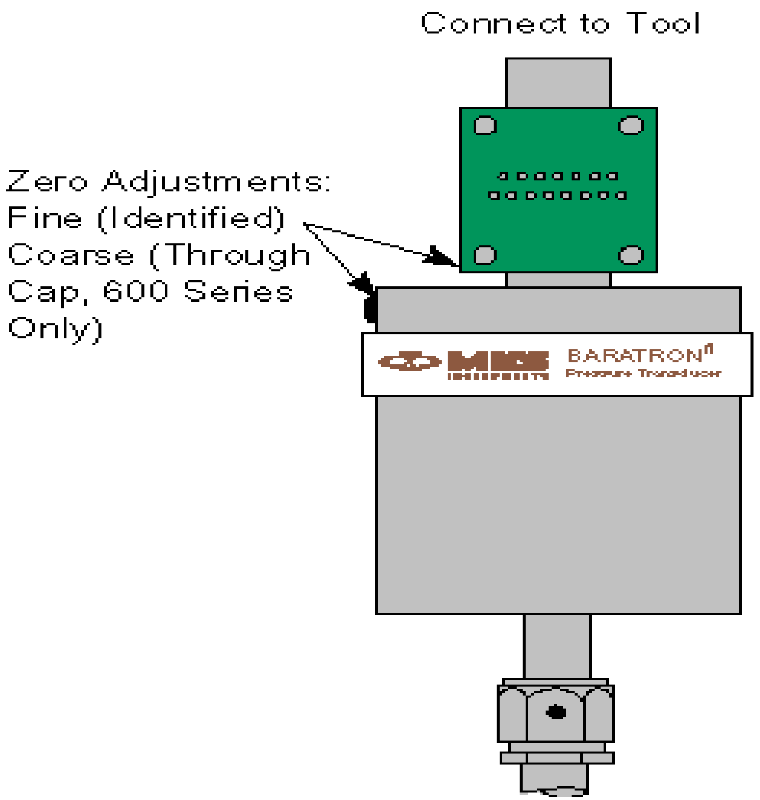
Bourdon tube gauge

- Still in production
- Very robust
- True pressure sensing
- Limited usefulness for HV

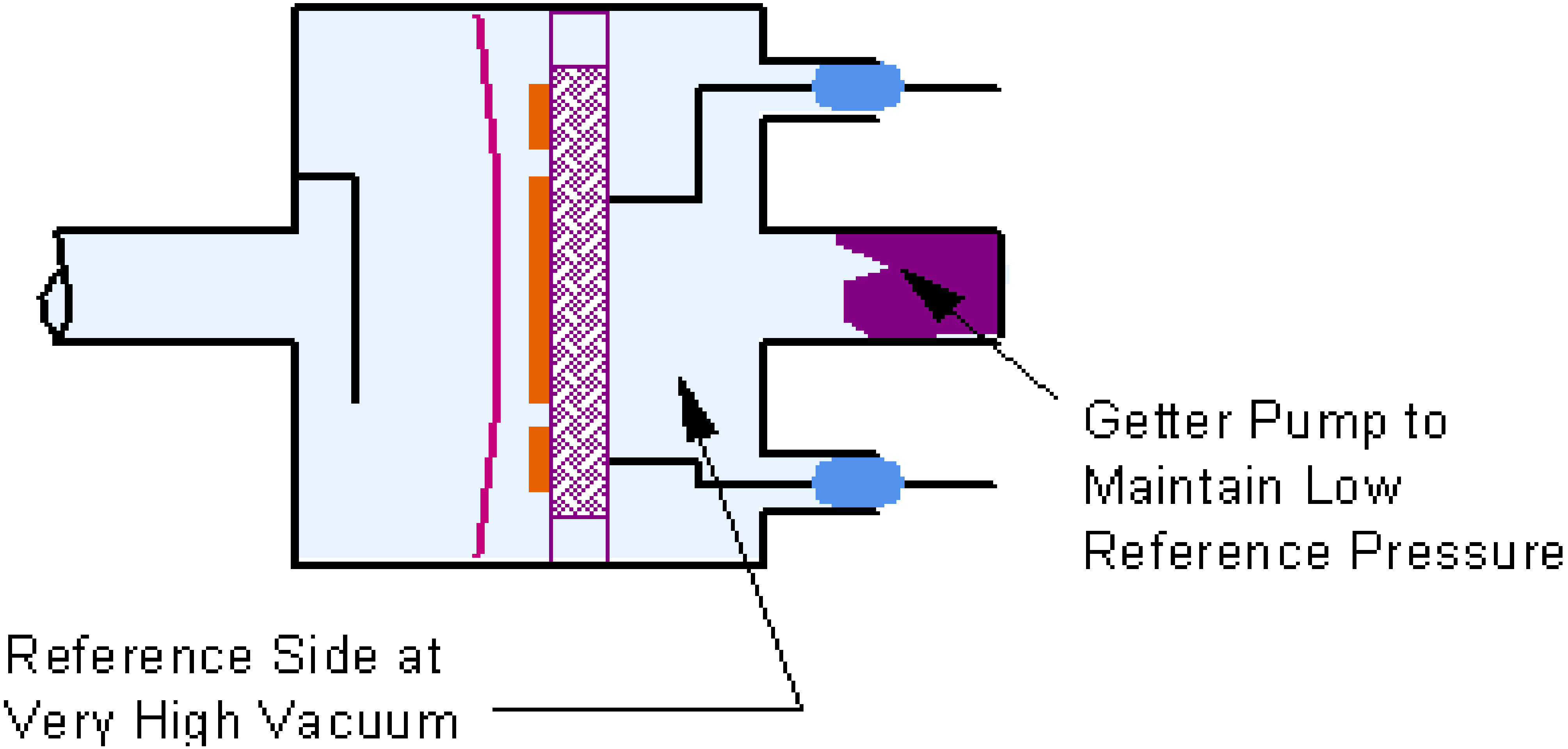


Capacitive gauges (CDG)

- 10^{-3} Pa to > 100 kPa
- True pressure sensing
- High sensitivity, useful over several decades
- Temperature sensitive, heated versions for highest precision
- May be manufactured for corrosive gases with special coatings



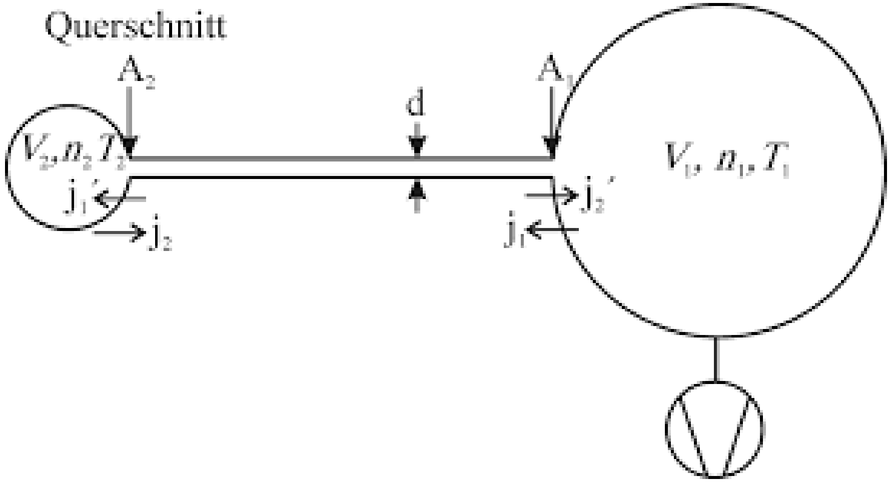
CDG



Getter Pump to
Maintain Low
Reference Pressure
Reference Side at
Very High Vacuum

CDG

Heated version require correction for ”thermal transpiration” in the range below ~100 Pa

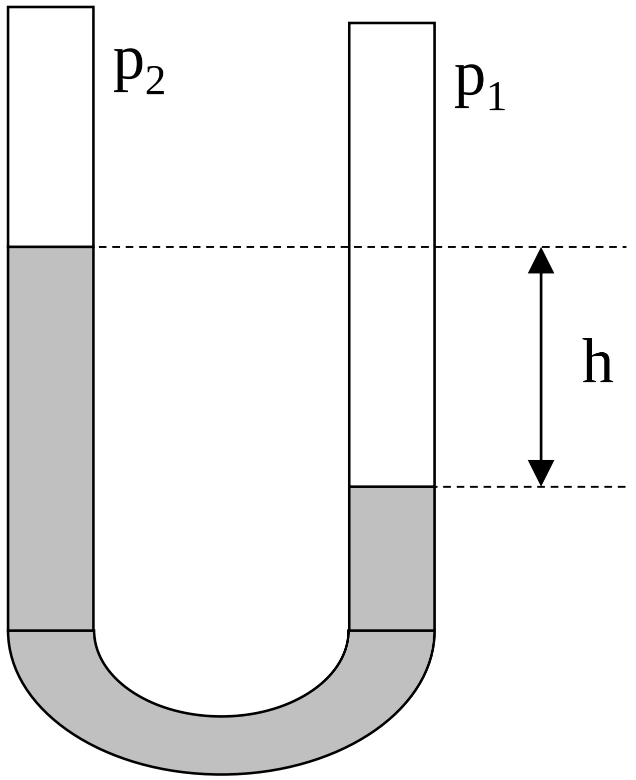


Pressure balances

- Primary standard for pressures above 1 kPa
- Very accurate, down to 1 ppm of pressure
- Laboratory equipment, slow, expensive.
- Influenced by air density, gravity and several other effects.

Liquid manometer

- $\mathbf{p_1 = \rho g h + p_2}$
- ρ = liquid density
- g = local gravity ≈ 9,81 m/s²
- h = Height difference between the two liquid levels
- p_1 resp p_2: applied pressure on each leg in the manometer



Liquid manometer

Simple to use

Hard to get precise readings, influenced by density and temperature inhomogenities

Slow

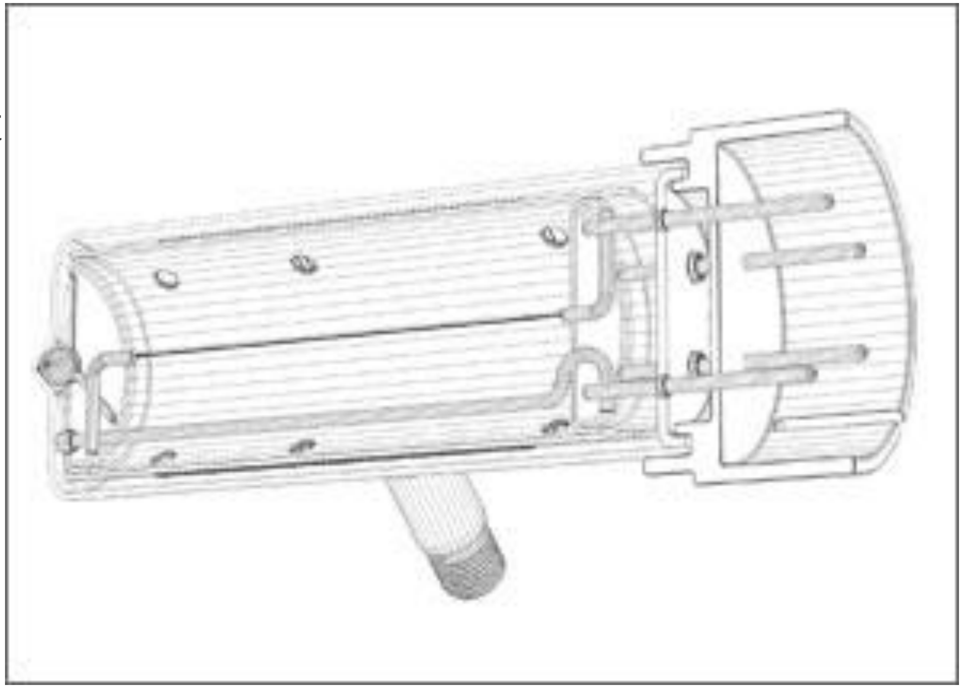
Ranges: ~1 Pa – 150 kPa (mercury)

0,01 Pa – 100 Pa (vacuum oil)

Best uncertainties ~10 ppm

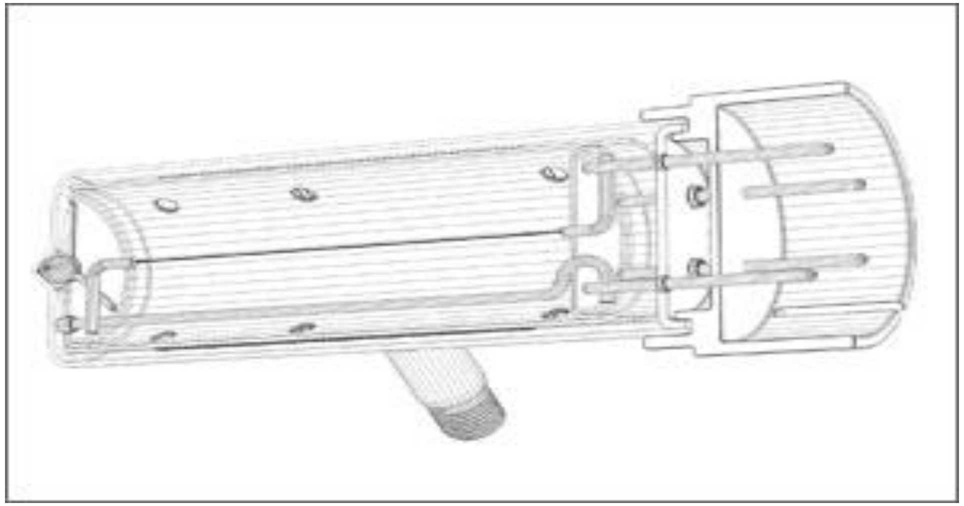
Pirani gauge

- 10^{-3} – 10 hPa
- Cheap and simple to use
- Robust, can stand suden pressure changes without damages
- Measures thermal conductance
- Fast

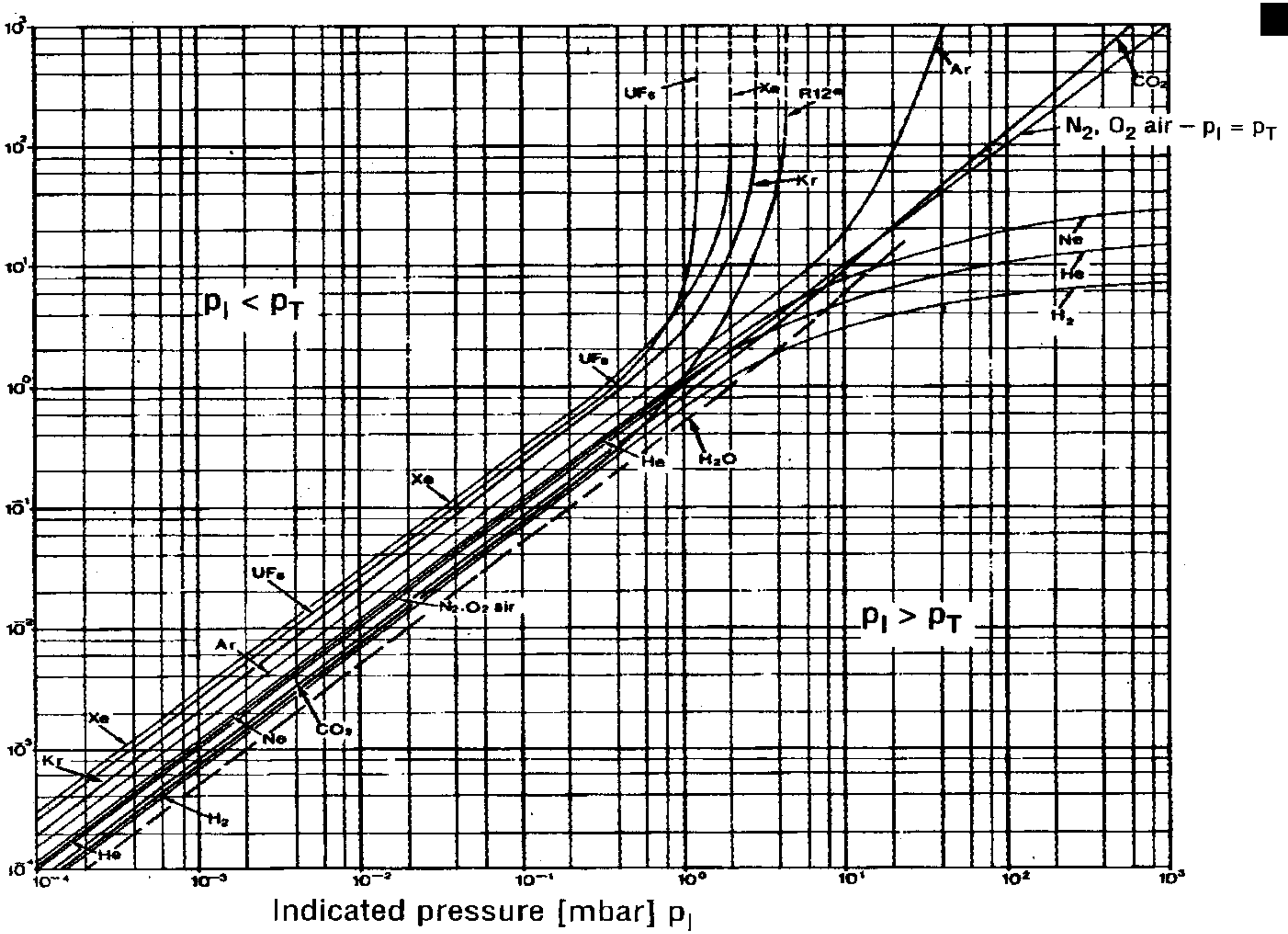


Pirani gauge

- Affected by high temperatures and moisture
- Low precision
- Gas dependant
- Sensitive to contamination
- Position sensitive, have to be calibrated/characterized in same position as used



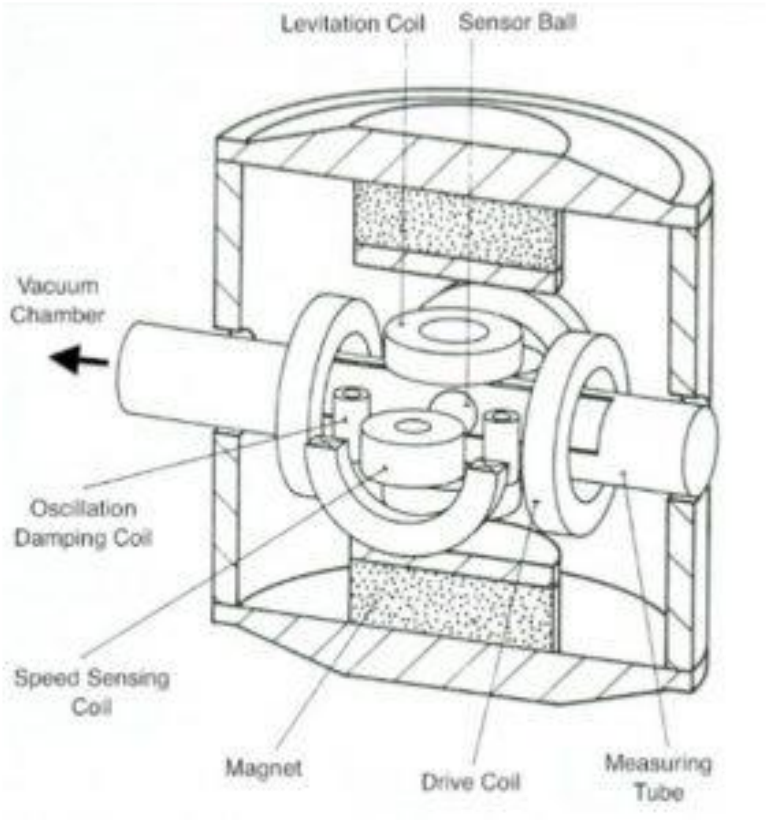
Pirani gauge



Calibration curves of THERMOVAC gauges for various gases, based on nitrogen equivalent reading

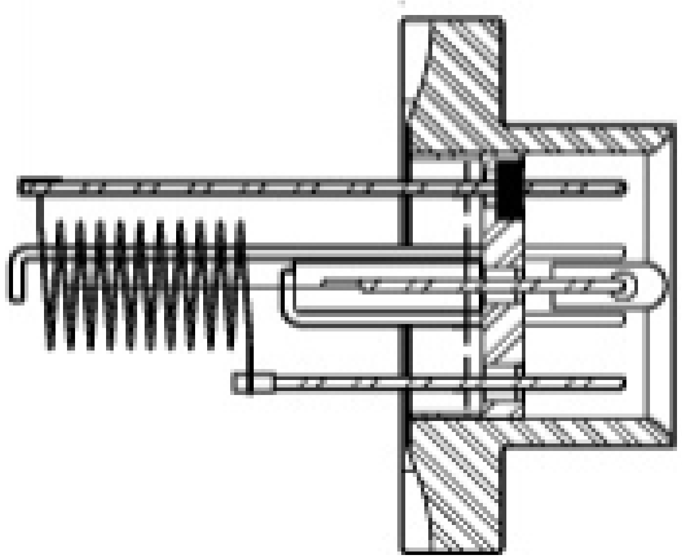
Spinning rotor gauge, SRG (gas viscosity meter)

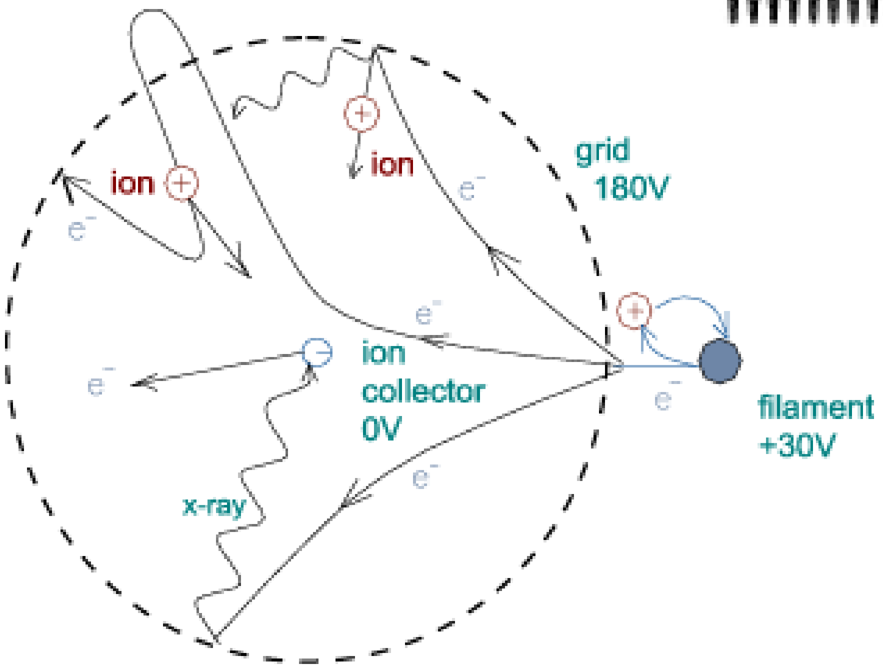
- 10^{-7} – 10 Pa
- Very accurate
- Gas dependant
- Slow (1 s – 30 s)
- Vibration sensitive
- Viscosity meter



Hot-cathode Ion Gauge

- 10^{-10} - 10^{-2} hPa
- Gas depending
- Can not withstand high pressures
- Measures the ionization effect of the gas molecules
- Several designs exist to minimize effects of background radiation and other parasitric effects





Hot-cathode ion gauge

Too high pressure (~10^{-2} Pa) will destroy the filament

Requires degassing with regular interval

Affects local vacuum since it both pumps and release gas.

Different housing:

- Naked gaugemeasure ambient vacuum more accurately but is affected by environment (other gauges, local ”chamber”)
- Encapsuled gauge is more robust when transferred to other systems and not affected by environment in the same way

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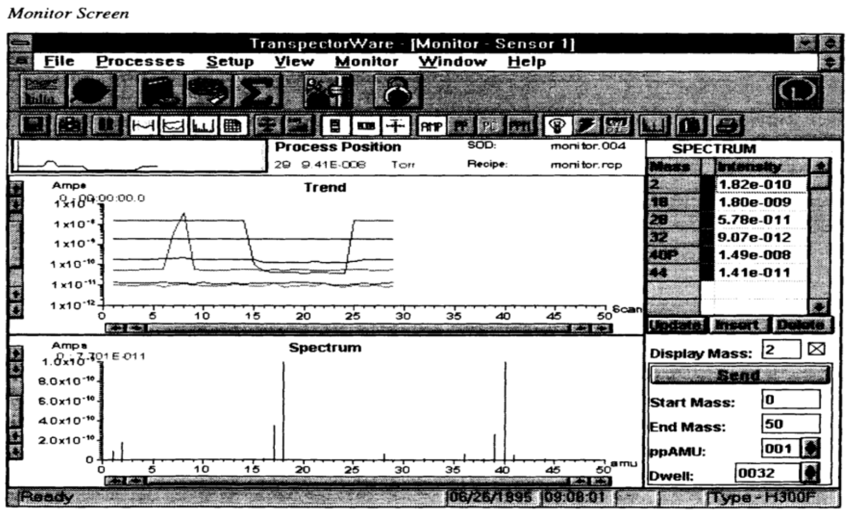
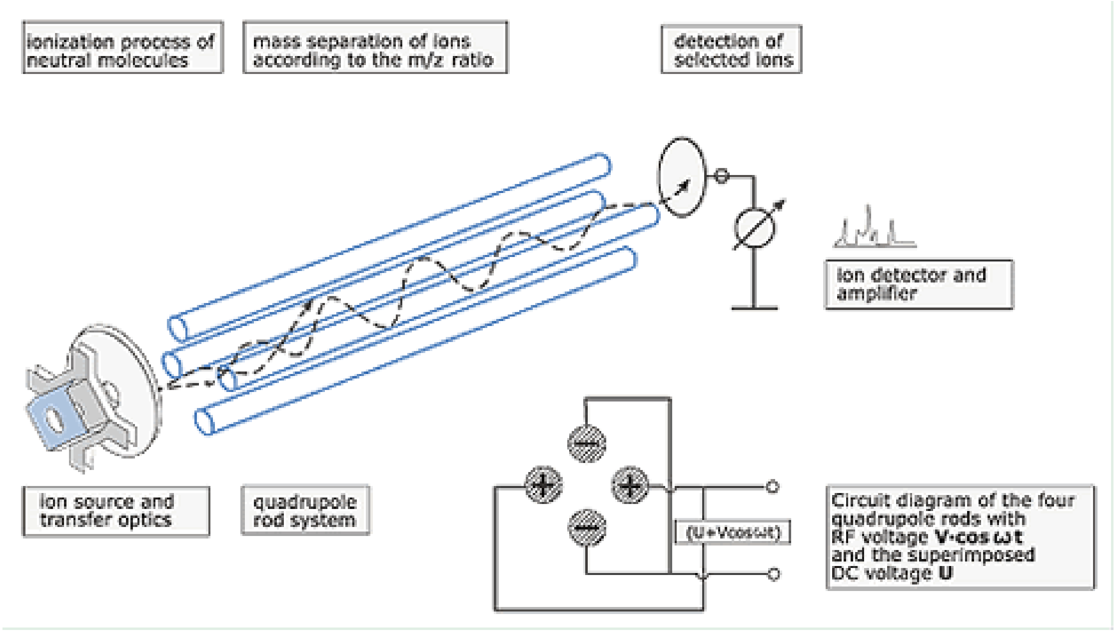
Cold-cathode ion gauge (Penning gauge)

- $5*10^{-7}$ - 10^{-2} hPa
- Gas depending
- Rought but robust
- Sensitive for contaminations
- Measures ionization of the gas
- Often used to check zero level on other gauges like pirani gauges or CDGs

Residual gas analyzer (RGA)

- Useful in UHV- and HV range
- Measures both total and partial pressure
- Hard to trust, drifts over time, (~60%3 months
- For accurate measurements, reference gases are to be used in conjunction with measurements.





Further comment

Today, there are several hybrids on the market, containing two or more sensors giving a bigger working range, for example CDG/Pirani or Pirani/Ion gauge

This gives a big dynamic range in one measuring head.

It might give problems in the overlap range, for example with gas mixture corrections

Conclusion indirect measuring vacuumeters

Type	Range (Pa)	Range (hPa)	Comments
Penning gauge	10^{-5} - 1	10^{-7} – 10^{-2}	Rough but reliable
Hot Ion gauge	10^{-1} – 10^{-8}	10^{-3} – 10^{-10}	Liniar, not so robust
RGA	10^{-2} – 10^{-10}	10^{-4} –10^{-12}	Expensive, slow
Pirani	10^{-2} - 10^{5}	10^{-4} - 10^{3}	Cheap, easy to use but low precision
SRG	10^{-5} - 2	10^{-7} – $2*10^{-2}$	Accurate but slow and sensitive to vibrations, expensive

VACUUM PUMPS

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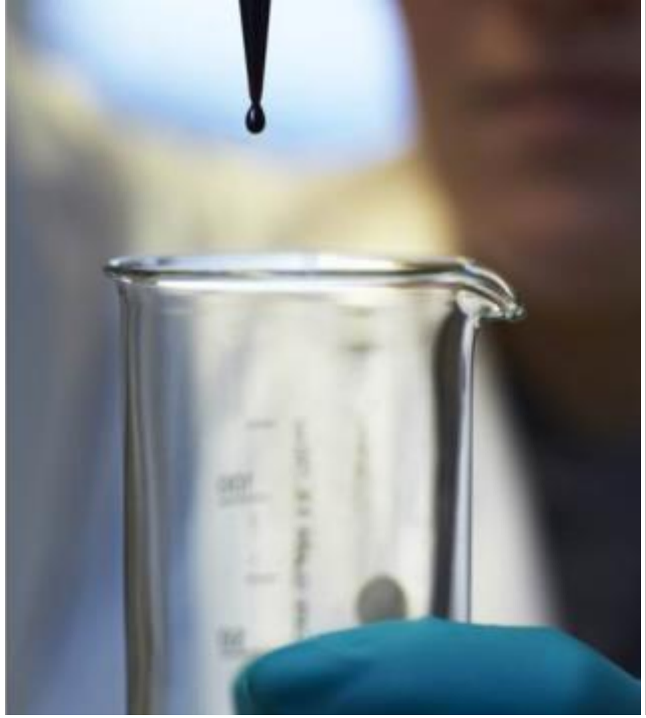
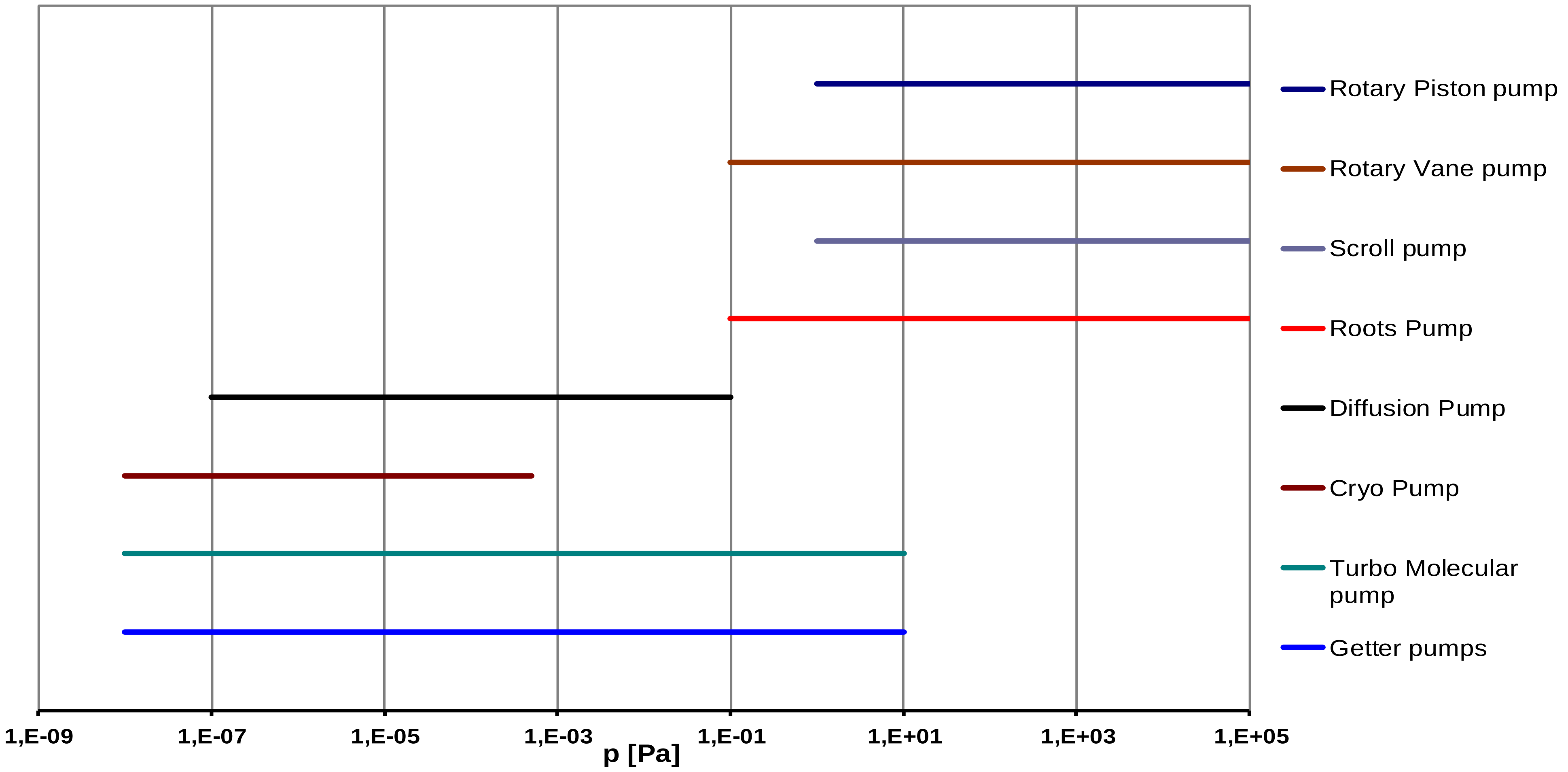
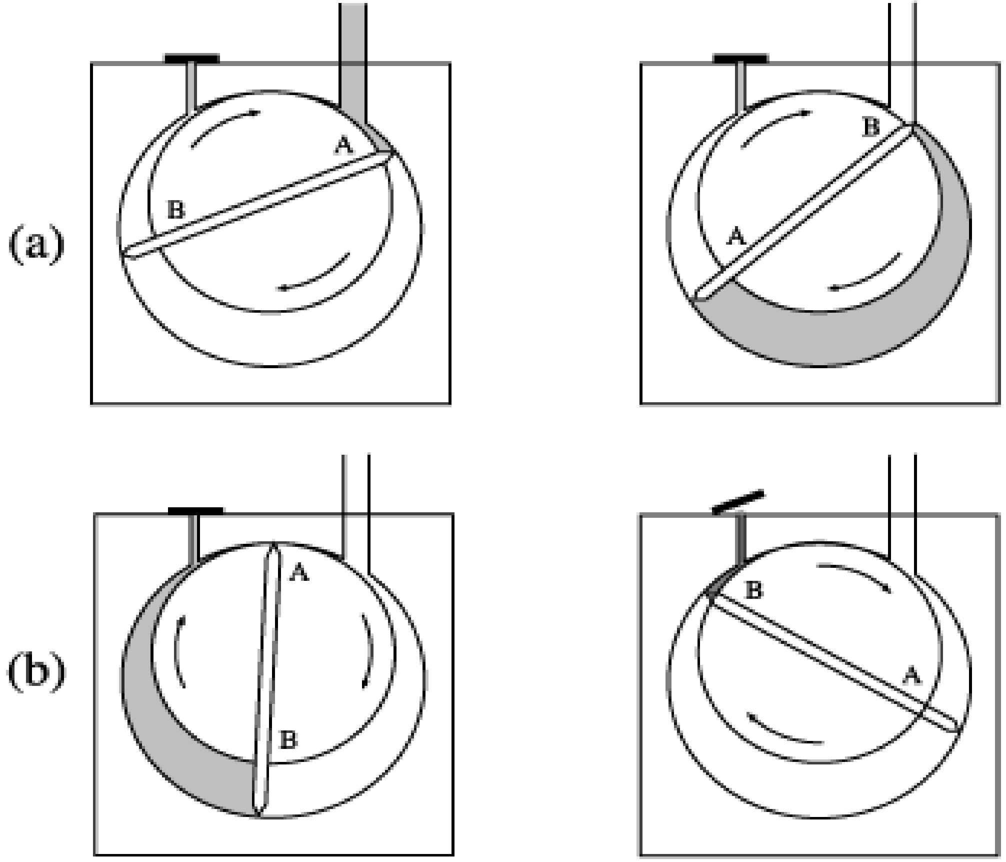


Robert Boyle f. 1627

Vacuum Pumps



Rotary vane pump



- Down to ~0,1 Pa
- Oil lubricated
- Oil sealed
- Effective
- Very common
- Robust
- Can result in oil contamination

Animation

Roots pump

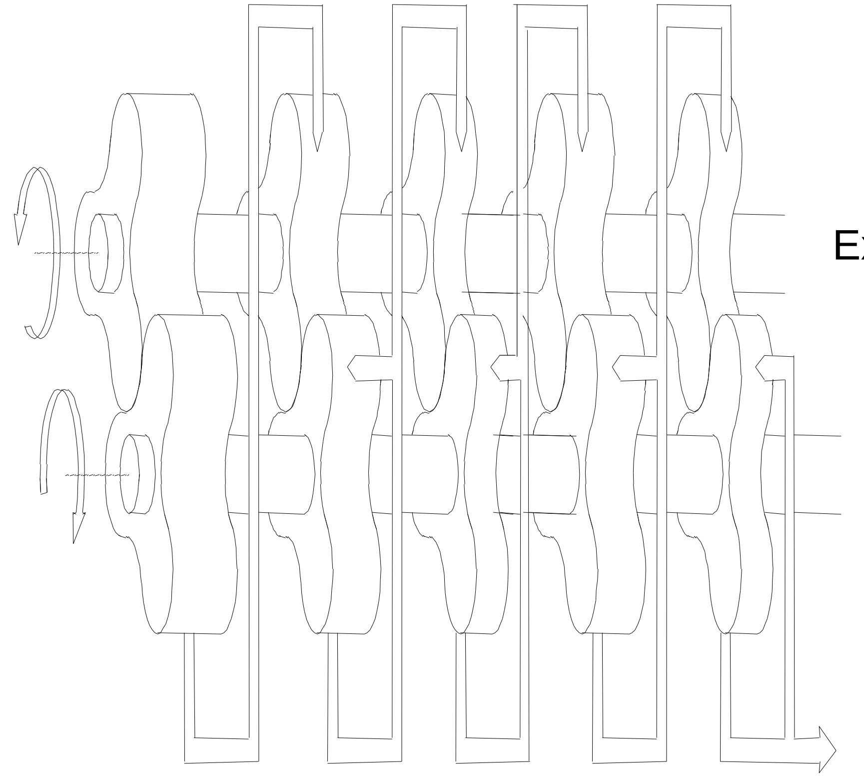


Bild från Alcatel

- Down to ~1 Pa
- Dry pump
- Old principle but recently used
- Requires many steps for high vacuum

[ROOTS pump animering]

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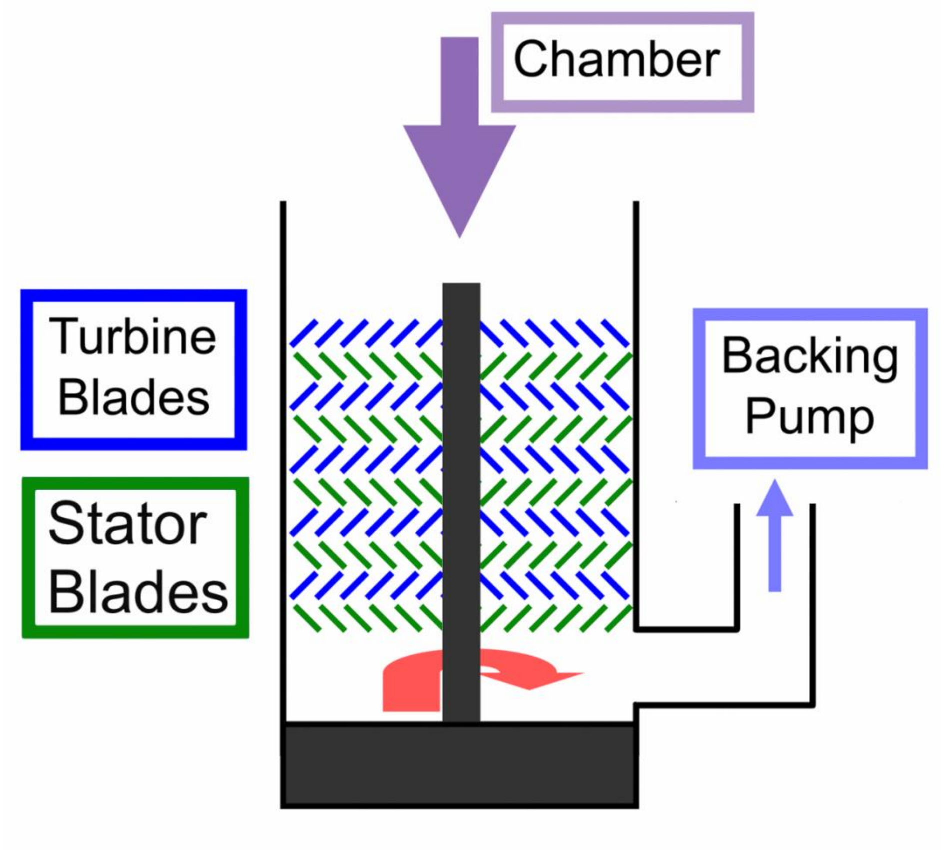
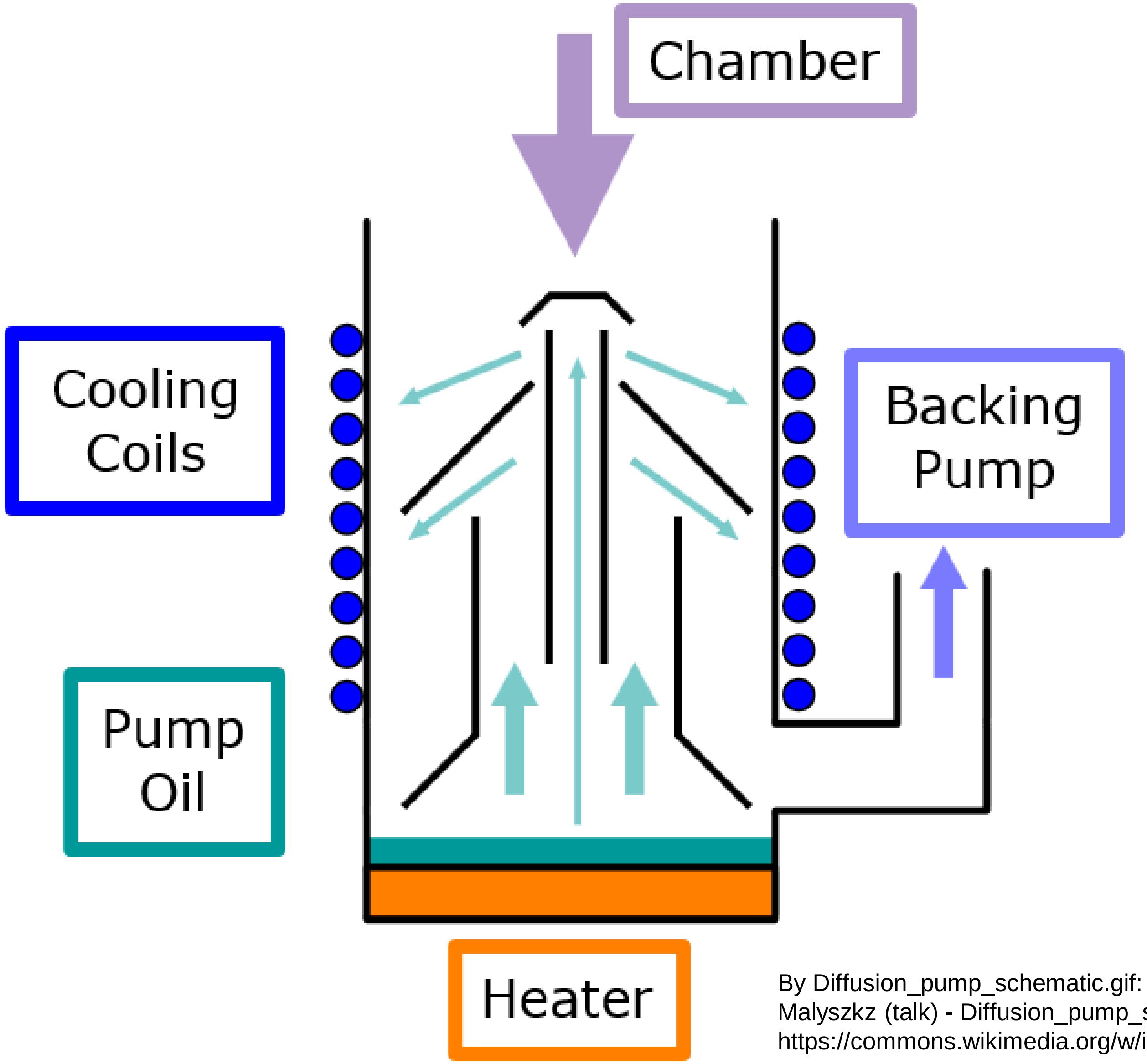
Turbomolekylar pump (turbopump)



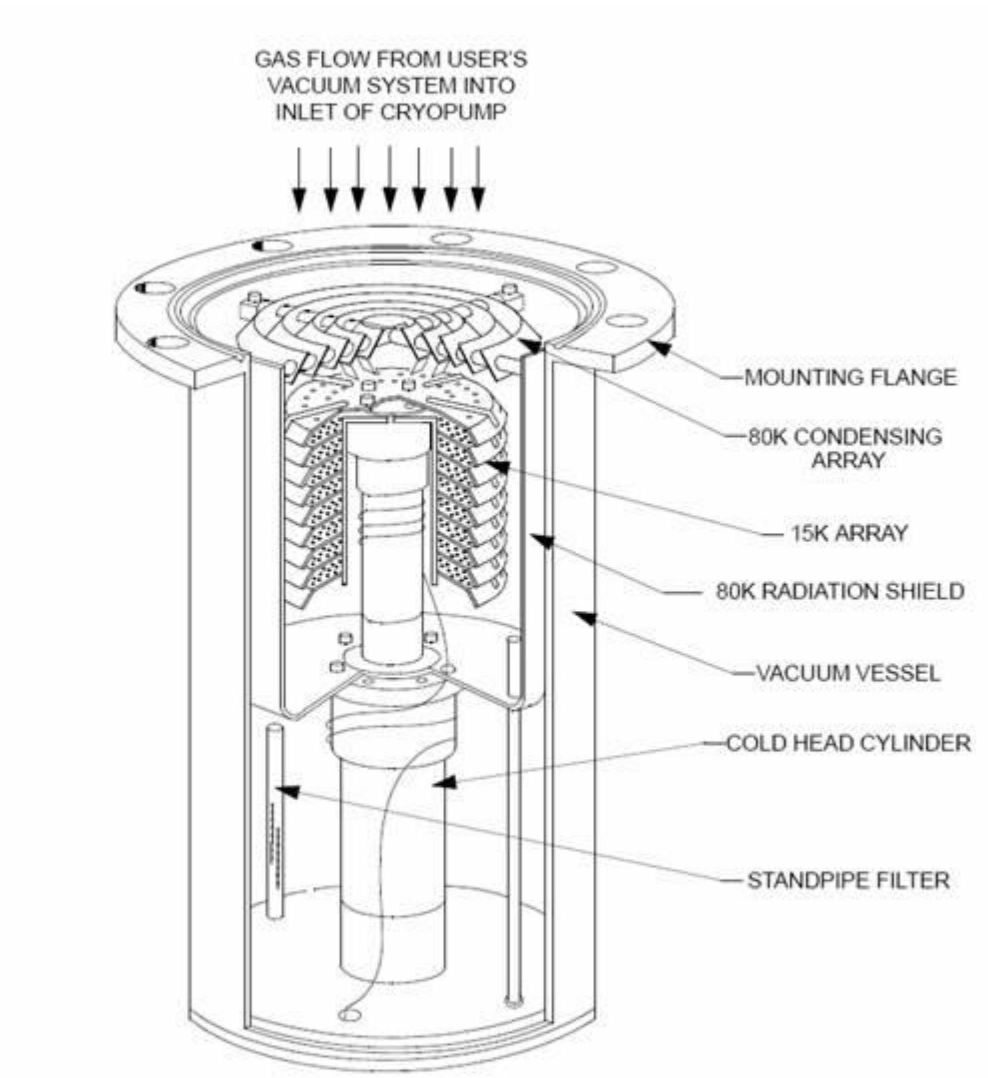
- Down to 10^{-9} Pa
- Very efficient
- Different pumping speed for different gases
- Oil lubricated or with magnetic suspension
- Requires roughening pump

Oil diffusion pump



- Down to ~10^{-5} Pa
- High risk for oil contamination
- Requires roughning pump
- Vibration free
- High capacity

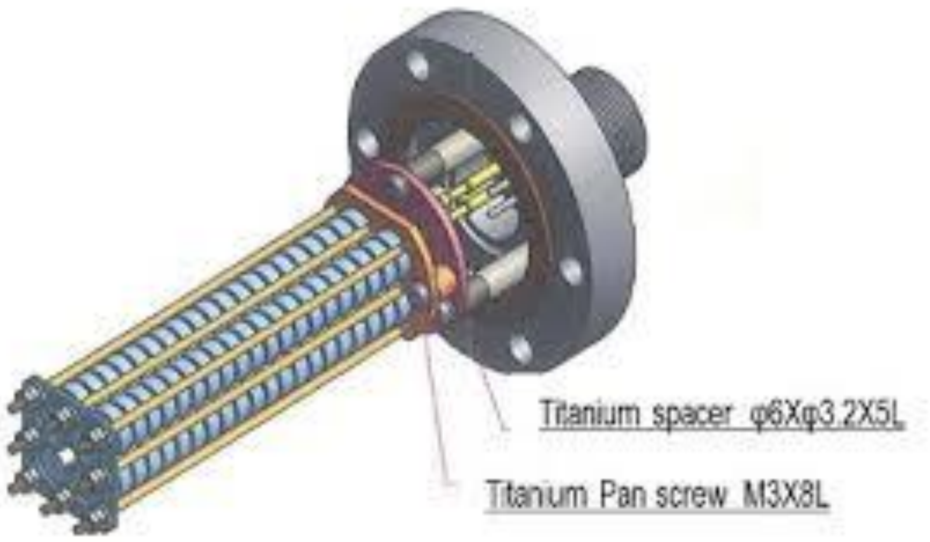
Cryo pump



- Gas depending pumping speed
- High pumping speed
- Not for continuous use
- Have to be regenerated

Getter pump

- Extremely large area
- Binding the gas to the surface
- Constant pumping speed in HV and UHV range
- Very good for Hydrogen and its isotopes
- Vibration free
- Needs regeneration
- Can be used in combination with turbo pumps



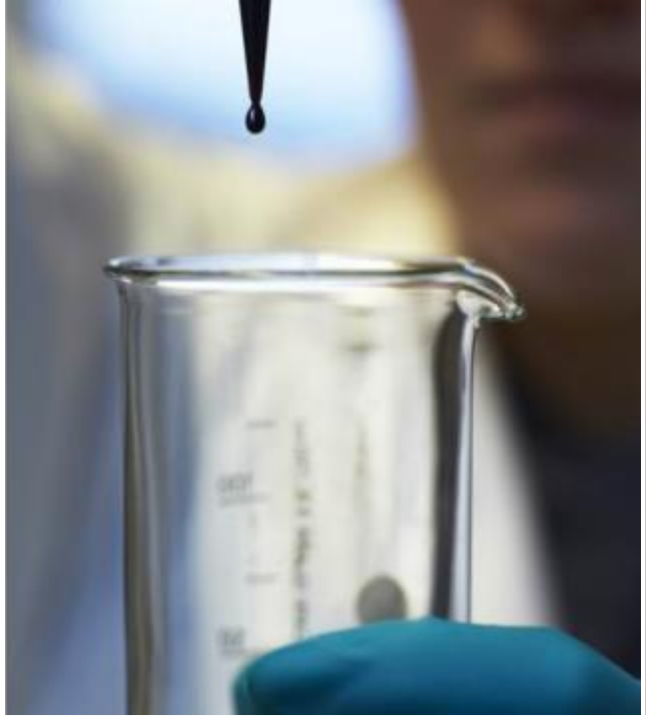
VACUUM FLANGES

Fredrik Arrhén

22 Oktober 2019

RISE Research Institutes of Sweden

Säkerhet och transport
Mätteknik



Flanges - requirements

Flanges are non-permanent joints in the vacuum system

They have to:

- Easy (or possible) to mount and dismount
- Have minimla leakage in the working range
- Be able to carry some mechanical load
- Be standarized
- Have maximal conductance

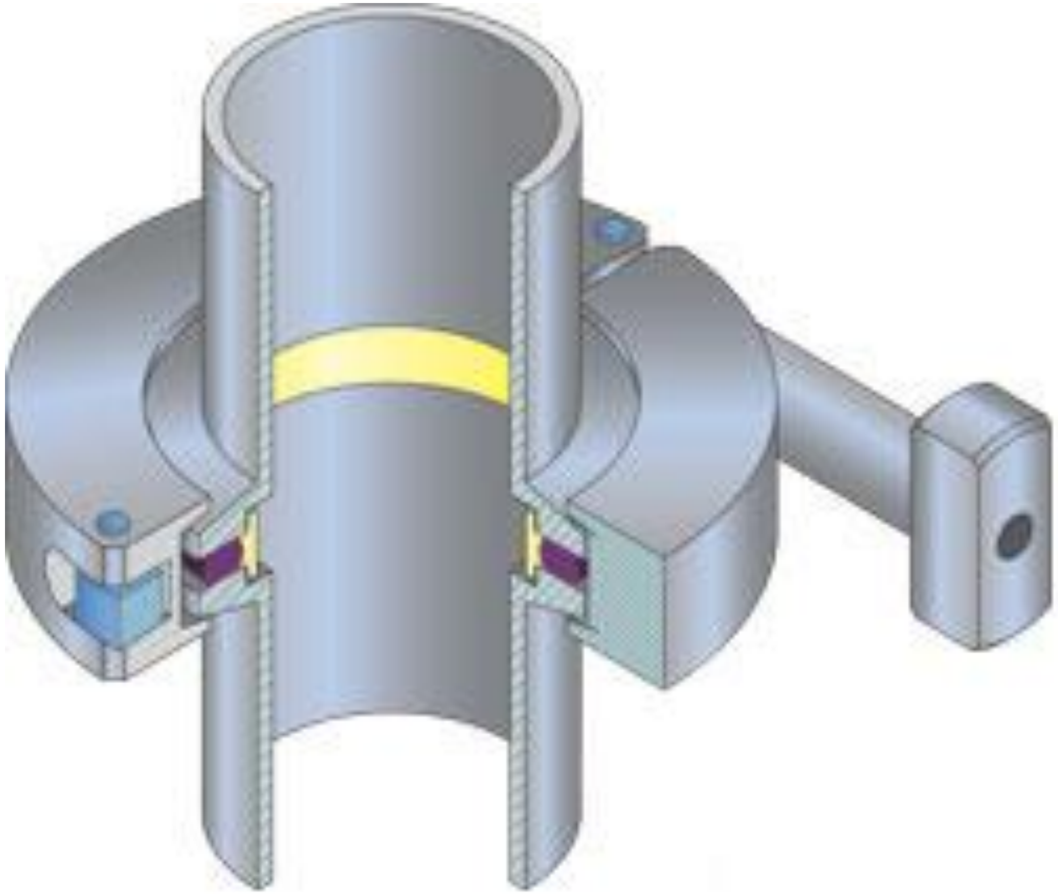
Diferent types of flanges

Three variants are standarized today:

- KF (Klein Flansche)
- ISO (Large ISO)
- CF (ConFlat)

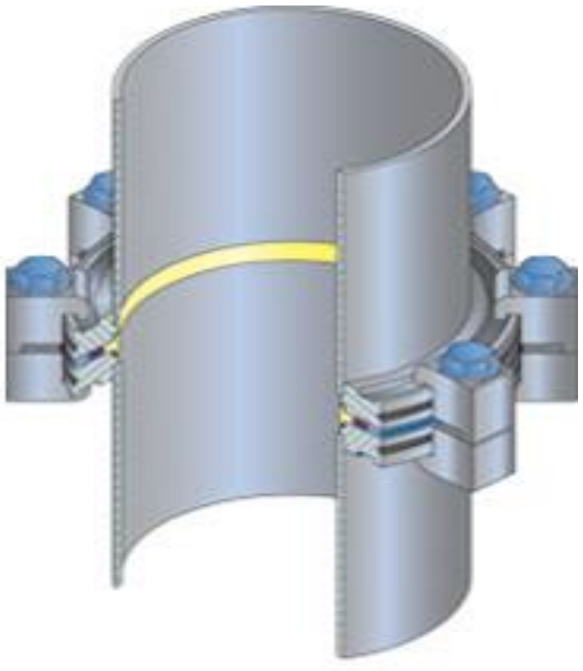
KF-flange (”Klein Flanche”

- KF is elastomer sealed flange using clamping ring
- Sizes from 10 to 50 mm inner diameter
- Can take some mechanical load, mainly to support the own weight
- The elasomer seal is mounted on a centering ring for support
- Useful in the range down to HV range and in temperatures between 0°C and 100-180°C depending on elastomer material.
- Quick and easy to use

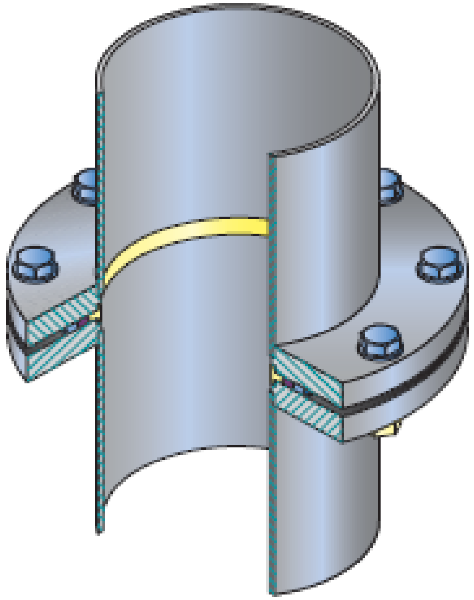


ISO-flange

- Also an elastomer-sealed flange
- The seal is mounted between two centering rings
- In diameters between 63 and 630 mm inner diamaeter.
- Can take higher mechanical load that KF.
- The flanges are either clamped together (ISO-K) or bolted together (ISO-F).
- The two versions can be mixed using ”half-clamps”.
- Useful down to HV and tempereratures between 0°C and 100-180°C depending on elastomer material.
- Very common on turbo pumps



ISO-K



ISO-F

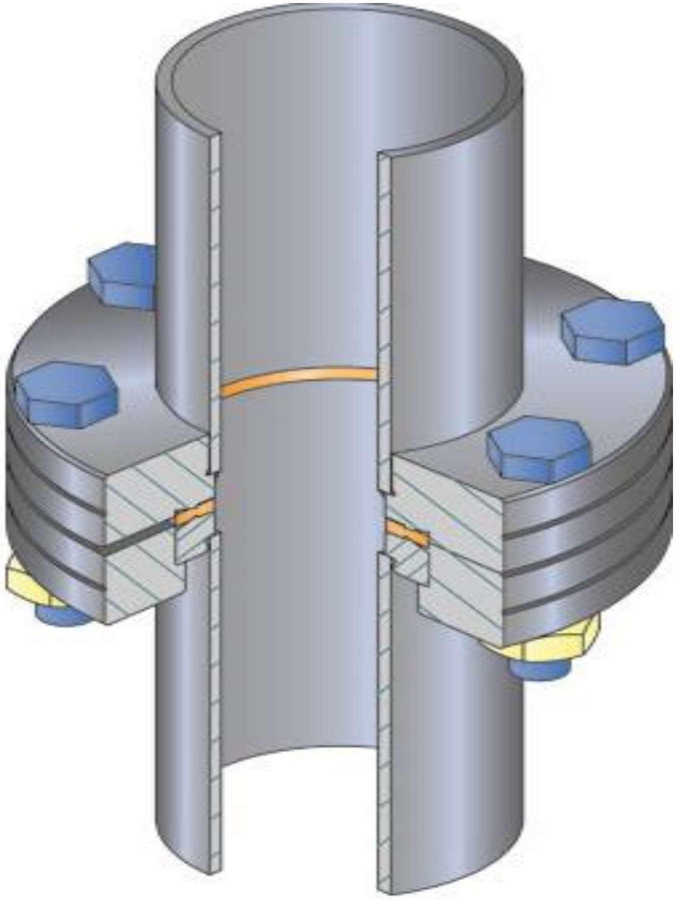
O-rings and elastomer materials

- Both ISO-K, ISO-F and KF are limited in performance by the elastomer material
  - Nitrile rubber (buna-N): cheap, maximum temperature ~100°C, high permeability for water, not suitable for oxygen processes, short lifetime.
  - Fluorocarbon (viton); very common, maximum temperature ~180°C to 200°C, relatively low permeability except for helium, long lifetime.
  - Perfluorocarbon (Kalrez, Chemraz), best high temperature performance, up to 250°C, otherwise like fluorocarbon

- Use of vacuum grease is under debate!

CF-flange (ConFlat)

- In diameters between 16 and 250 mm inner diameter.
- Have a metal seal between two knife egges, one in each flange.
- The seal is normally copper which can be silver coated for high temperature use
- Bolted together directly on the flange
- Both rotating and fixed flange ring exist
- The seal is not reusable
- Useful in pressures down to ~10^{-11} Pa and temperatures between -196°Cand 450°C.
- Special care to be taken for the bolts when used in extreme temperatures



Adapters

- Of course you always have the wrong flange on your system
- There are a vast range of adapters between different sizes and different flanges.
- On top here is a adapter between CF16 till ’Cajun VCR’, a common connector on CDGs and in gas injection lines
- … and underneath an adapter between KF16 and NPT thread.

Standardized dimensionens

- Most components have standardized dimensions
- For example a 90° angle can easily be replaced by a T-connector later if needed.

When the standard components doesn’t fit

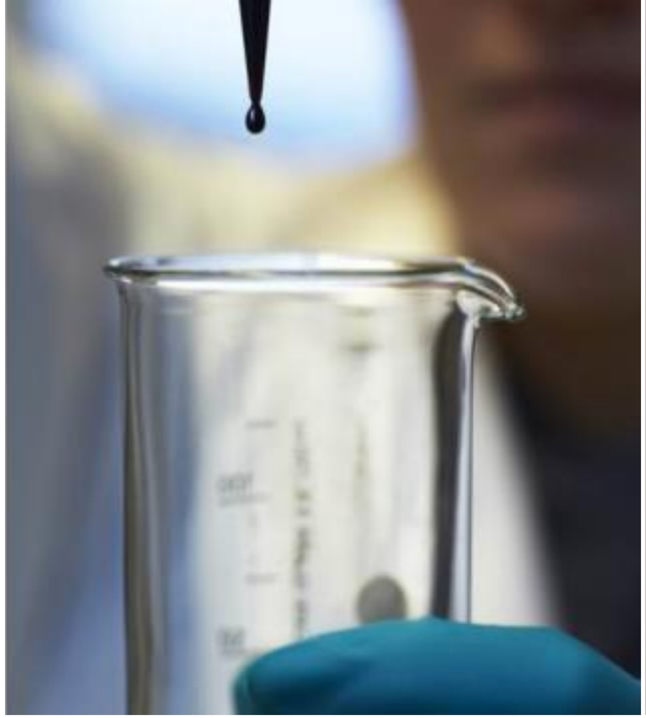
- Of course there are flexiple components when standard dimensions doesn’t fit
- Or when vibration damping is needed

And then there are special components.

TRACEABILITY AND MEASUREMENT UNCERTAINTY

Fredrik Arrhén

22 Oktober 2019



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Mätteknik

Measuremetn quality and how to measure more precise

Content:

- Terms
- Measurement uncertainty in short
- Tracability
- Measurement and calibration stategies

Terminology in metrology

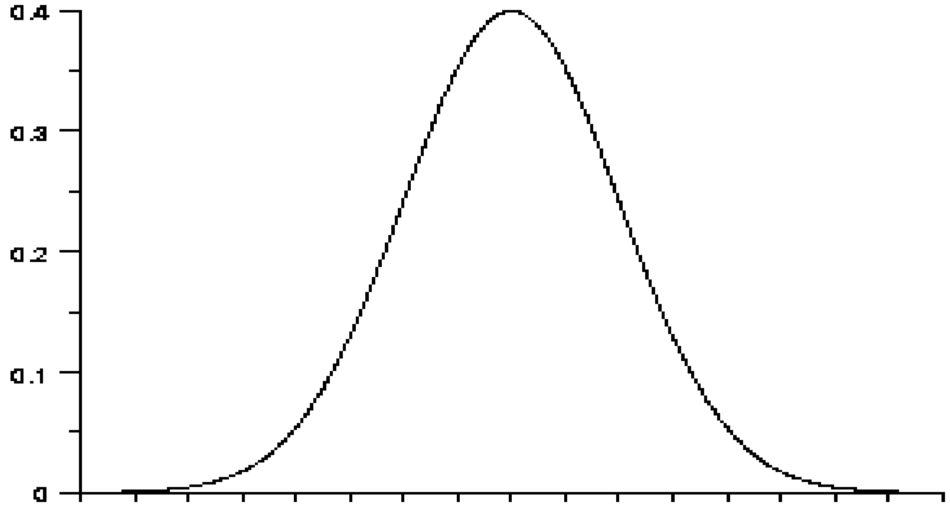
- **Calibration:** Establish the relation between the readout and the true value.
- **Adjustment:** To make an instrument provide prescribed indications corresponding to given values of a quantity to be measured.
- **Measurement error:** Indicated value minus true value
- **Correction term:** Value to be added to indicated value to compensate for systematic effects.
- **Mesurement uncertainty:** An interval within where the true value is, “non-negative parameter characterizing the dispersion of the quantity values being attributed to a measurand, based on the information used”

Measurement uncertainty in reality

- The GUM-mettod (Guide to the expression of Uncertainty in Measurements):

- All effects are treated using statistical methods

- All contributions are treated as standard deviations



Measurement uncertainty in reality

Some expressions:

- Expanded uncertainty
  The interval within which the true value is expecxted to be with a certain confidence level, normally 95%.

- Coverage factor (k)

  The coverage factor used to reach the confidence level (normally 95%) wanted.

- Confidence level

  The level with which the uncertainty is to be expressed at, normally 95%.

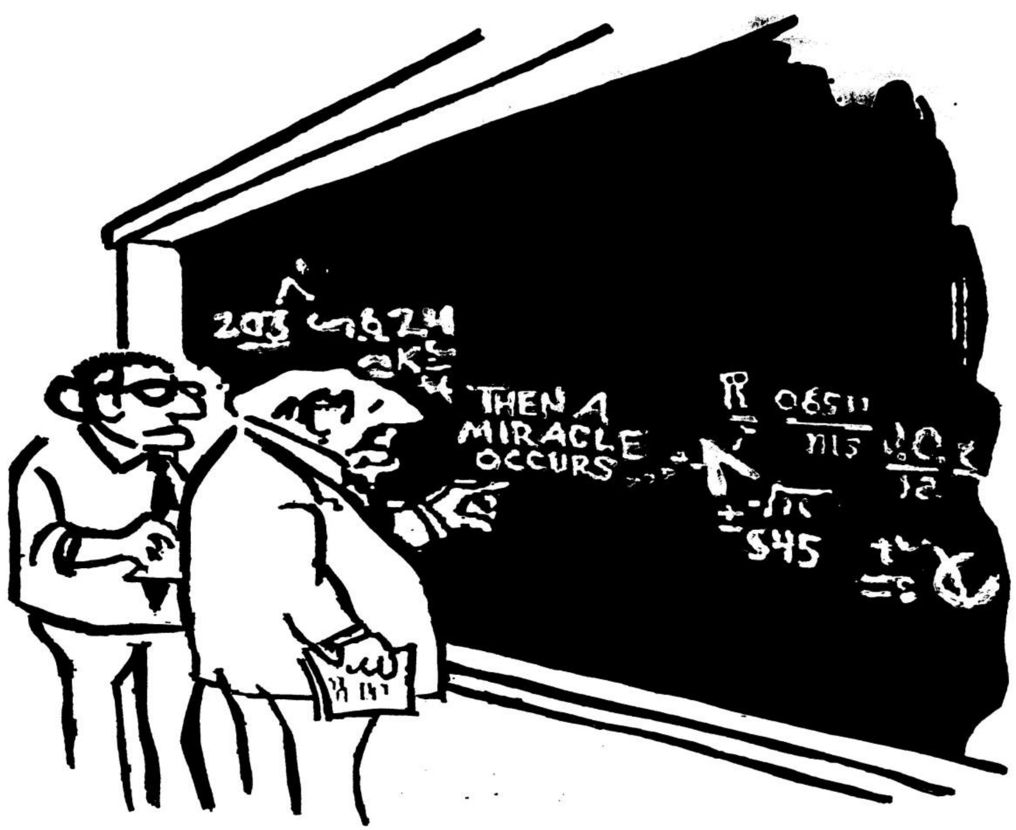
Exemple on an uncertainty calculation osäkerhetsdiagram

Simplified uncertaity calculation according to EA-4/02

Components

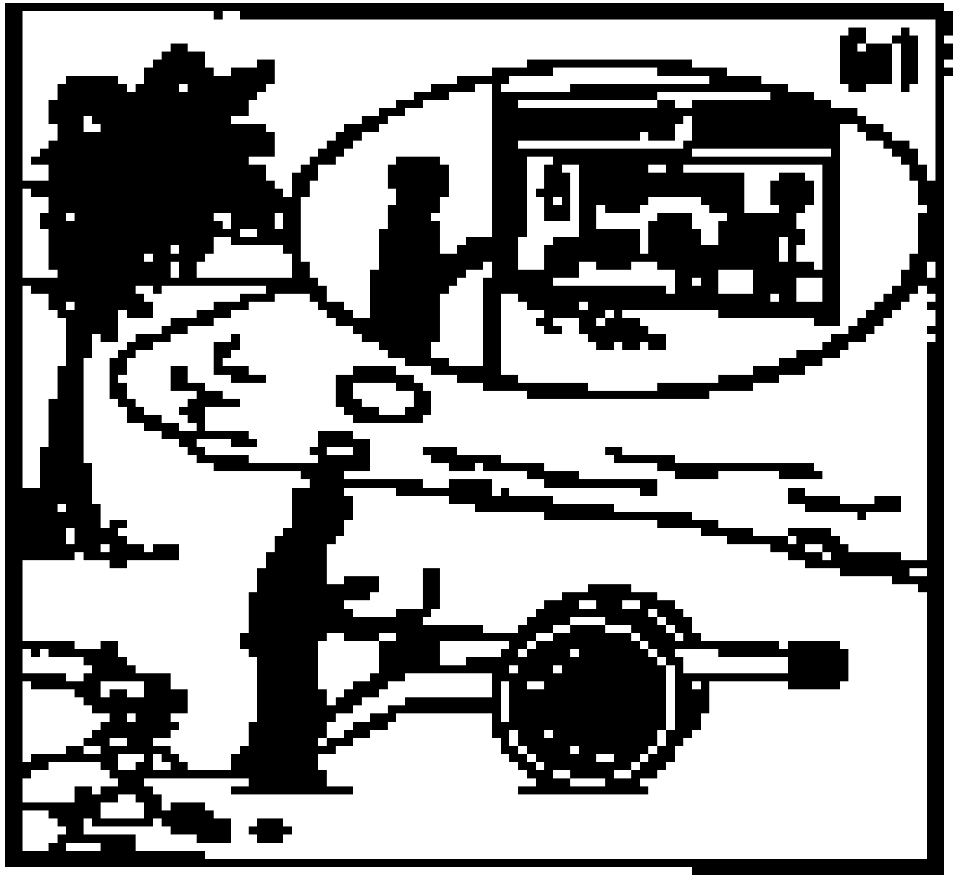
Quantity	Uncertainty	Unit	Distribution	Sensitivity factor	unit	Contribution	Comment
Calibration	0,09	mbar	normal, k=2	1	mbar/m	0,05	
Drift	0	mbar	rektangel	1	mbar/m	0,00	From spec
Resolution	0,005	mbar	rektangel	1	mbar/m	0,00	
Specifikation	1	mbar	rektangel	1	mbar/m	0,58	Spec/year
Error from modelling	0,035	mbar	rektangel	1	mbar/m	0,02	
					total k=1	0,58	
				k-factor:	2		
		Decimals	3		**total (95%)**	**1,159**	

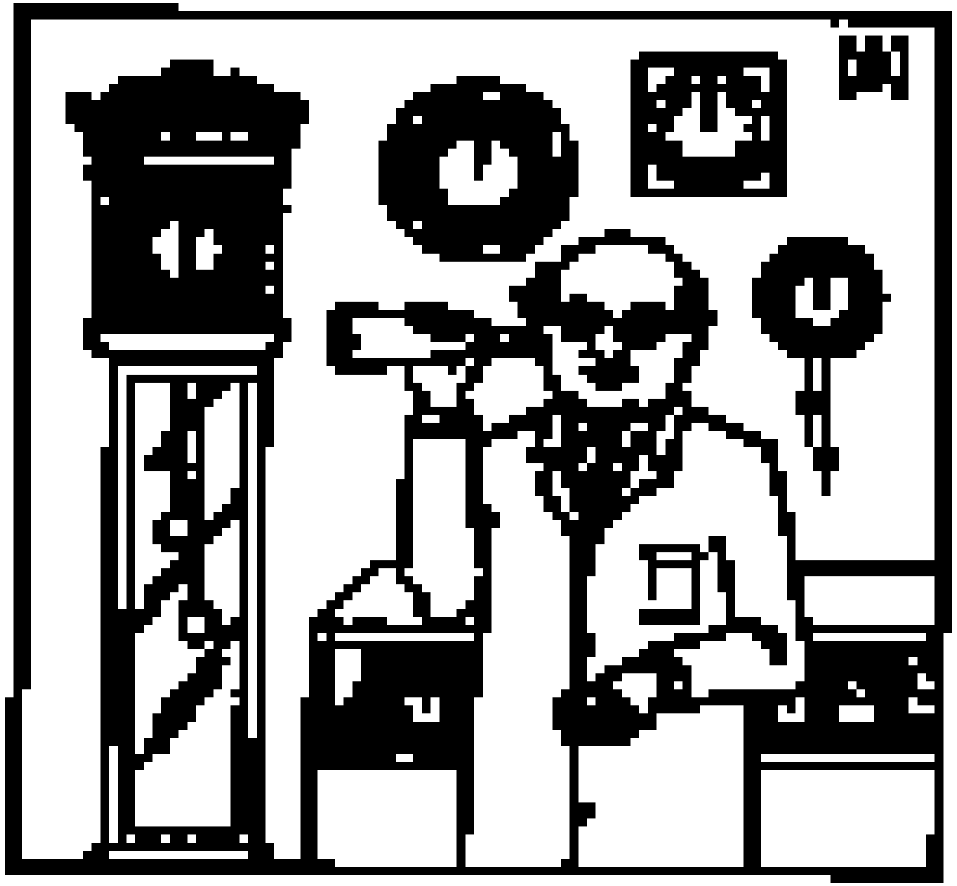
In reality



"I think you should be more explicit here in step two."

The Zanzibar effect





Traceability

What's the use of tracability?

- A requirement for making measurements comparable whenever or whereever they are made.

- Necessary for reproducibility of measurements.

Traceability

What is traceabiity?

“Property of a measurement result whereby the result can be related to a reference through a documented unbroken chain of calibrations, each contributing to the measurement uncertainty”

Traceability

- SI, seven bvase units:
- **Metre**
- **Kilo**
- **Sekund**
- Ampere
- Kelvin
- Candela
- Mol

Static expansion system

Simple principle:

V_1=1dm³

V_2=9dm³

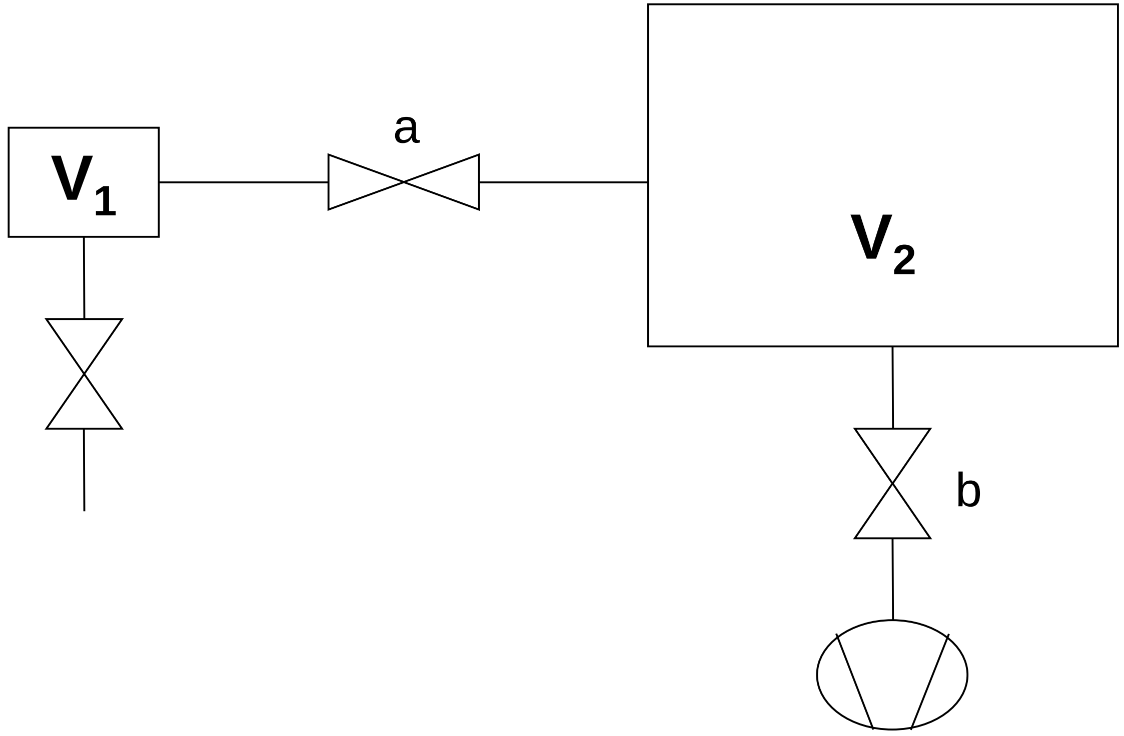
P_1=1,0 kPa

P_2=0,00001 Pa~0kPa

Close valve b open valve a.

P=0,1 kPa

Close valve a, open valve b and repeat until wanted pressure is reached.



Static expansion system

Static expansion system

- Useful from atmosperic pressure down to ~10^{-6}Pa
- Requires very well known volume ratios.
- Influenced by changes in valve volumes and temperatures
- Simple and reliable principe
- Uncertainties between 0,1 – 10% depending on pressure range.

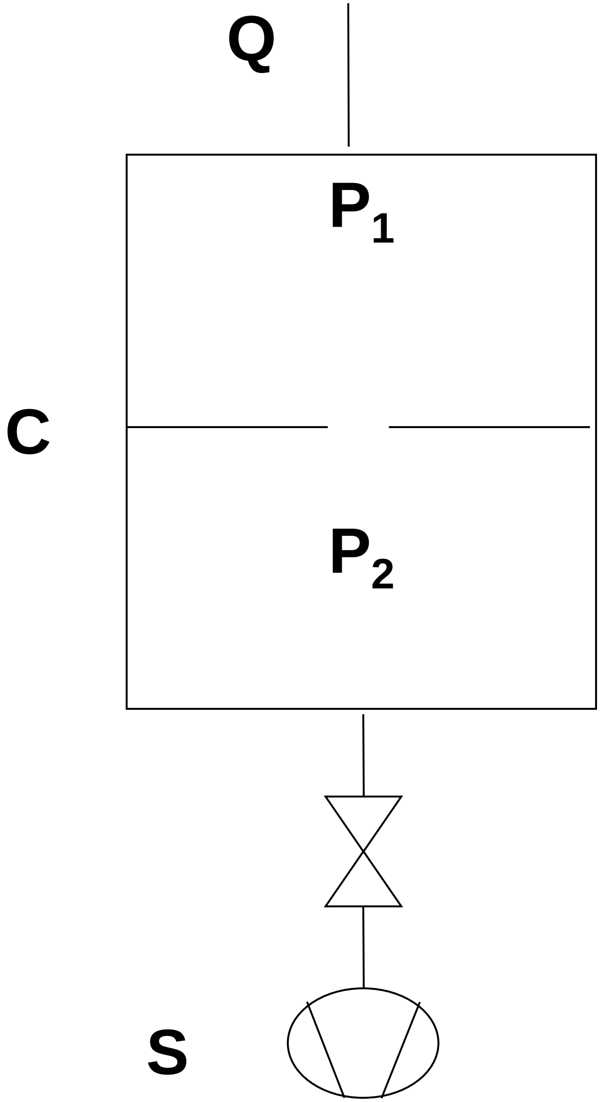
Dynamic expansion system or Orifice flow system

- Useful below ~10^{-3}Pa

- More complex principle:
- Kowing Q, P_1, C and S, P_1 and P_2 can be calculated.

- Q and C Hard to determine with enough. precision

- Uncertainties between 1-10% of pressure.



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