Practice 7, PDA-Seminar

Brief explanation of the different stations

2024 PDA Europe
Freeze Drying in Practice

Agenda

Station 1

- Pressure Calibration > ATM
- Vacuum Calibration < ATM

Station 2

- Temperature Calibration

Station 3

- Shelf-Mapping

'You always measure wrong, you just have to know how much' David Packard (Packard-Bell)

Definitions

Calibration

*is comparing and documenting
the measurement of a device
to a traceable reference standard*

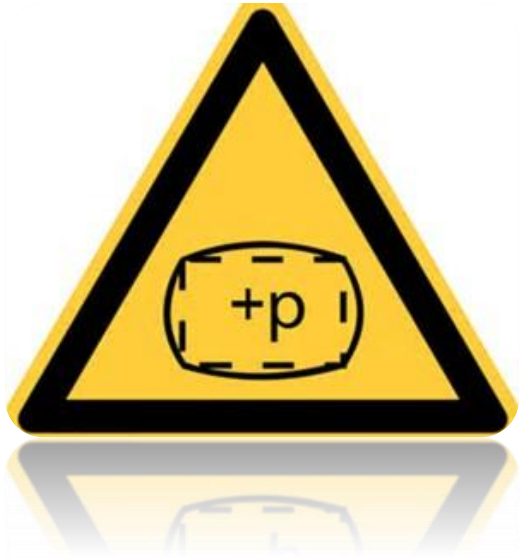
Adjustment

The act or process of adjusting to make a change to something in order to correct or improve it.

Why Calibration?

to minimize any measurement uncertainty by ensuring the accuracy of the equipment.

The calibration workstations



Pressure Calibration

Pressure Sensors in use

Device to be calibrated:

- **Sensor: piezo-resistive** pressure sensor

- **Type**: JUMO dTrans p31 pressure transducer

Nr.	Working Range (Measuring Range)	Sensor	Type	Deviation
1	50 ... 4000 mbar (0 ... 4000) mbar	4bar resistiv	Jumo p30 (491)	40mbar
2	20 ... 1600 mbar (0 ... 1600) mbar	1,6bar resistiv	Jumo p30 (489)	15mbar

- **Features**: Allowed media temperature up to 200° C, long term stability, overload resistance, high sensitivity, wide measuring range, fast response time, compact and lightweight
- **Application**: pressure vessel applications; SIP, Door control/closure, aeration,..
- **Miscellaneous**: robust, low price

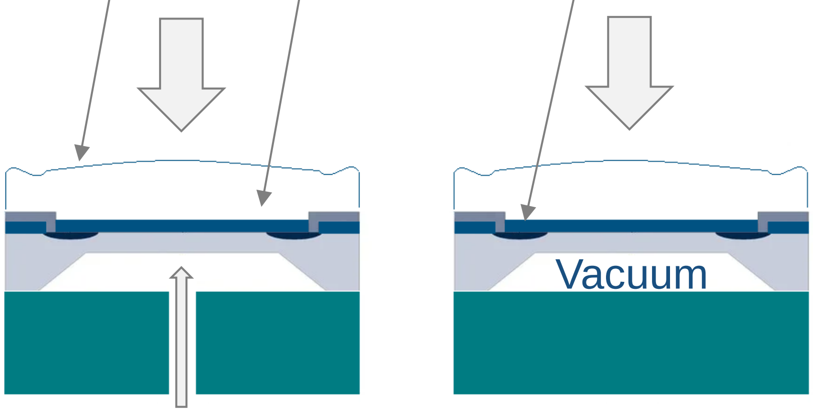
- **Measuring principle**: Determination of pressure indirectly via the deformation/deflection of an elastic element (membrane). Silicon crystal changes its electrical voltage (piezo-electrical) or by changing its resistance (piezo-resistive).

Principle of construction

Membrane of the sensor housing

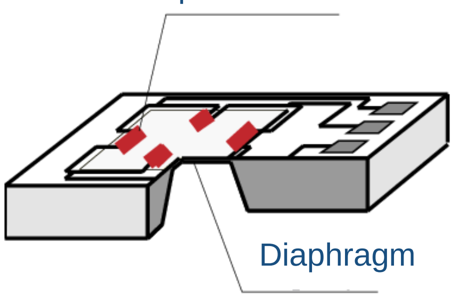
flexible membrane that deflects under the influence of an applied pressure

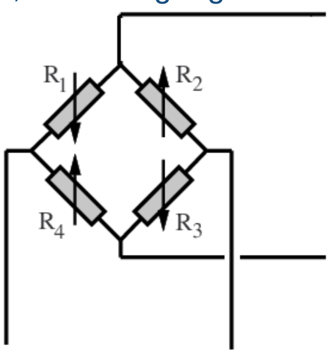
piezoresistive elements arranged in a specific pattern (Wheatstone bridge)



Relative Sensor   Absolute Sensor

piezoresistive elements, or strain gauges

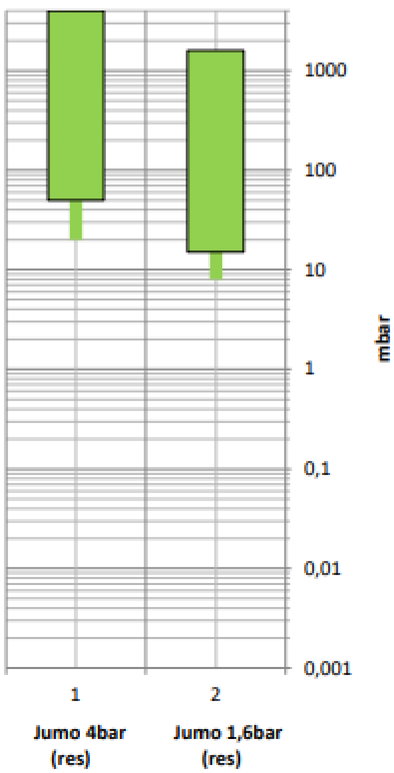


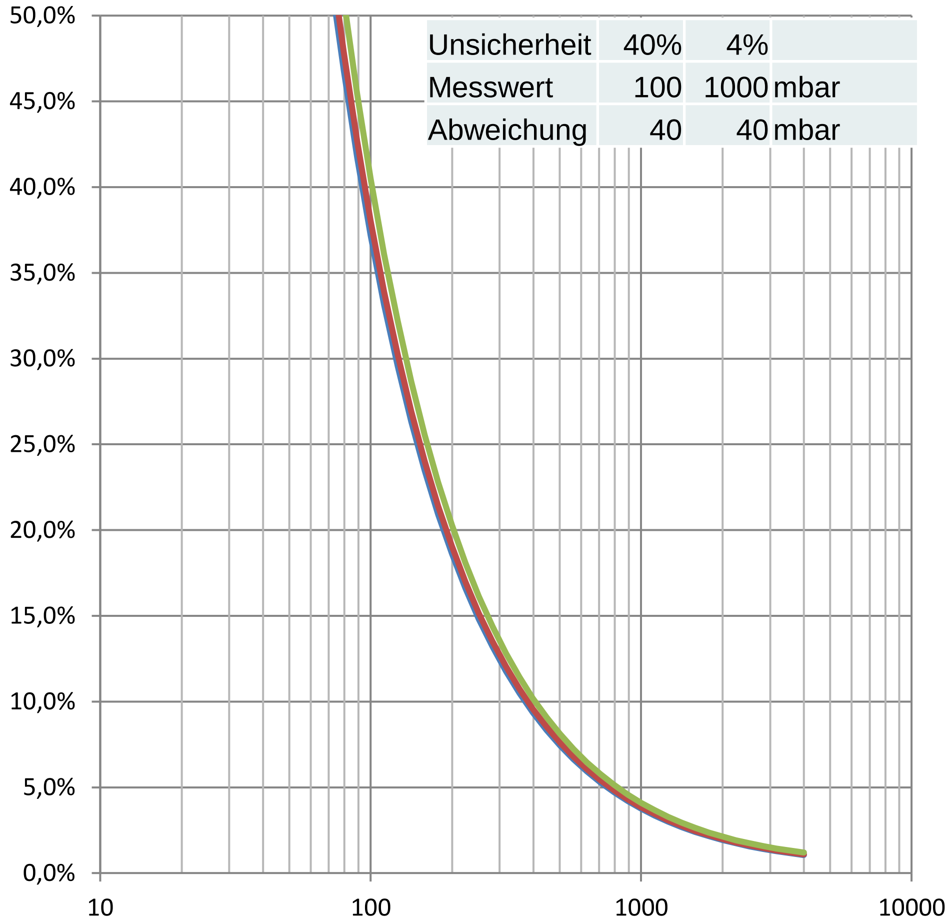


Wheatstone bridge

Under pressure/vacuum the diaphragm deforms, causing mechanical stress on stain gauges.
A Wheatstone bridge is used for the resistor arrangement.

Pressure Sensors uncertainty & useful range





Unsicherheit	40%	4%	
Messwert	100	1000	mbar
Abweichung	40	40	mbar

Pressure Sensor Calibrator (reference)

- Mecotec reference display as calibrated standard (0,001 ... 4.000 mbar)

- Hand "pressure" pump for generating vacuum and overpressure

- Calibration vessel (recipient) for holding up to three sensors

Vacuum Calibration

Vacuum Sensors -Pirani- in use

Device to be calibrated:

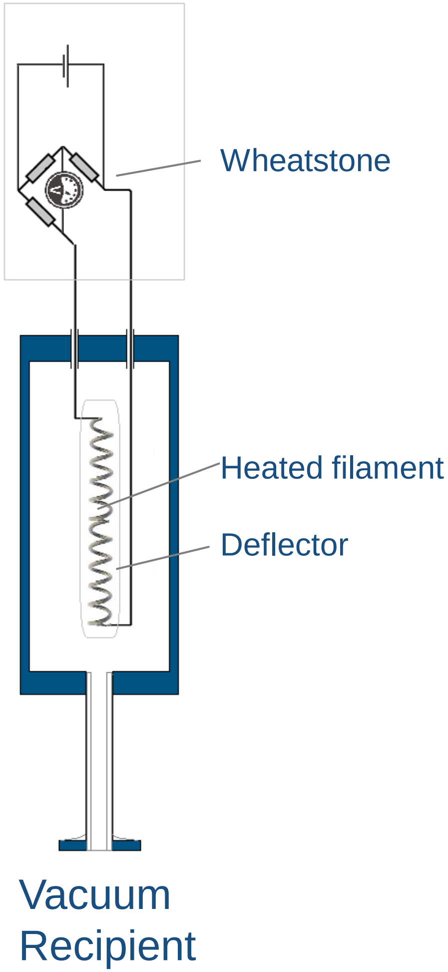
- **Sensor: Pirani** probes (gas dependent)
  - **Type: Thyracont VCP63MV** Pirani sensor with Platinum/Rhodium-Filament

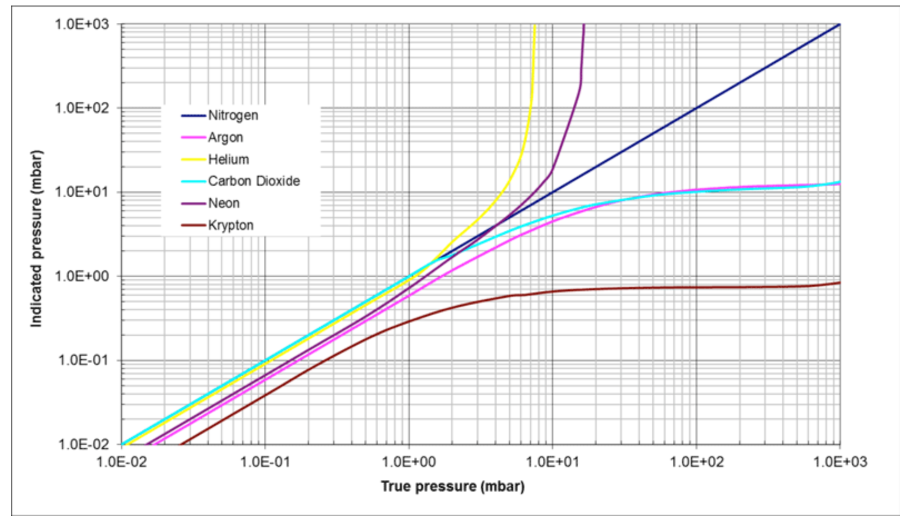
Nr.	Working Range (Measuring Range)	Sensor	Type	Deviation
4A	0,005 ... 1000 mbar (0,0005 ... 1000) mbar	Pirani **Gefriertrocknung**	Thyracont VCP63	10% vom Messwert bei <10mbar
4B	0,005 ... 1000 mbar (0,0005 ... 1000) mbar	Pirani **Belüften**	Thyracont VCP63	(30% vom Messwert bei >10mbar)

- **Features**: comparably cheap sensor, stable measuring values (low drift affinity), **Applications**: Comparative pressure measurement, all vacuum application
- **Miscellameous**: needs block valve for CIP, can be sterilized SIP (not powered) +150° C no add. sensor heater required. Critical in ATEX applications (filament >+60° C)

- **Measuring principle**: heated filament changes resistance due to reduced thermal conductivity

Principle of construction

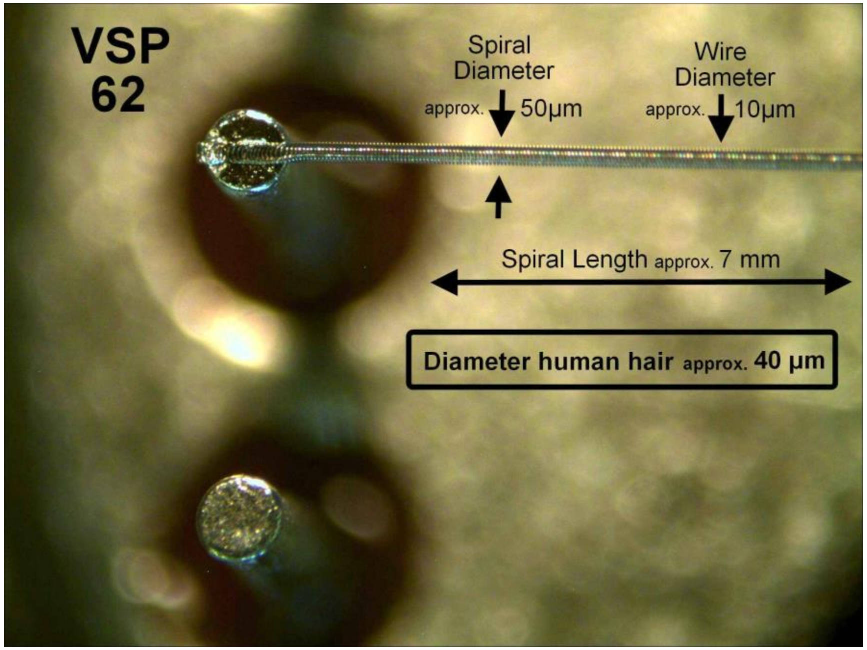




Gas dependency, thermal conductivity

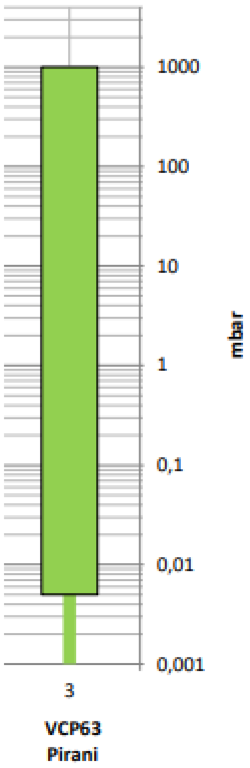
Nitrogen gas has a much higher thermal conductivity compared to the thermal conductivity of Argon gas!

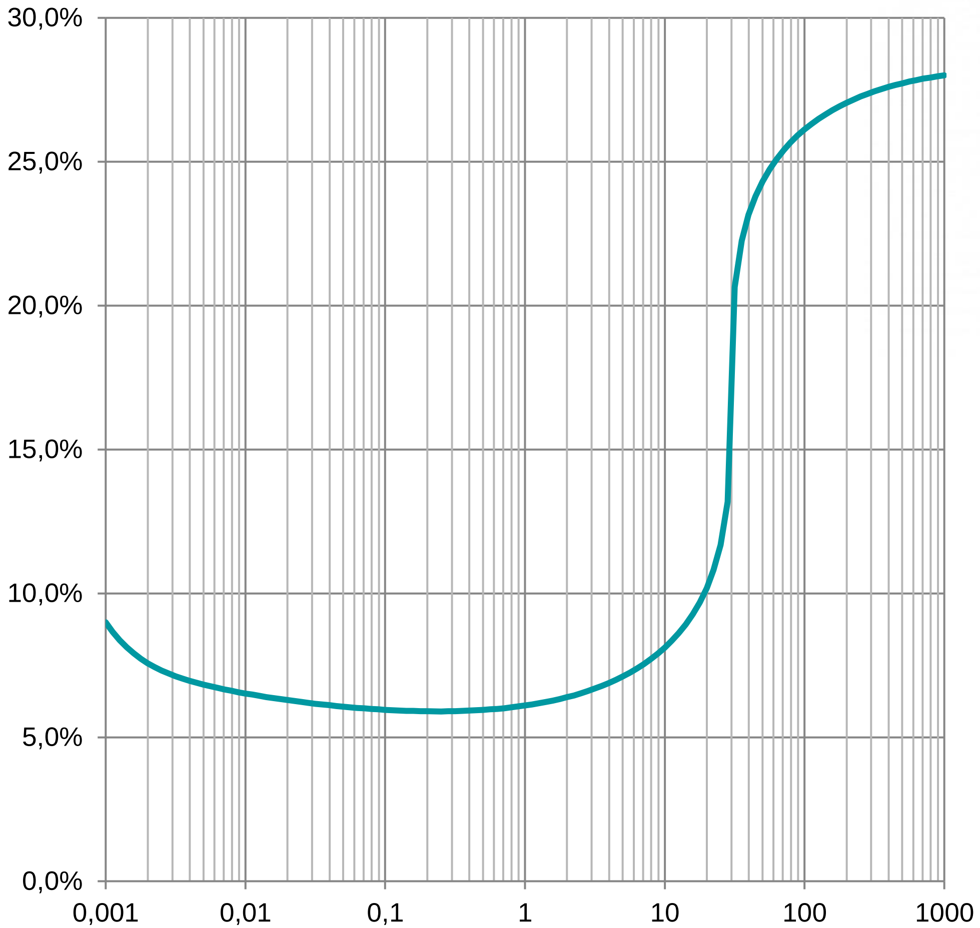
Pirani filament under microscope



- Avoiding exposure to filament
- *Humidity, Product, Corrosion,*

Vacuum Sensors uncertainty & useful range





Vacuum Sensors -capacitive- in use

Device to be calibrated:

- **Sensor: Capacitive probes** (absolute, gas independent probes):
  - **Type:** **Pfeiffer CMR363 / 364** (temp.-compensated)
    Pfeiffer CMR373 / 374 (temp.-controlled/regulated)
    Azbil V8C (5 .. 0,0005), SPG7A-P13 (10-0,001) P12 (1 .. 0,0001)

Nr.	Working Range (Measuring Range)	Sensor	Type	Deviation
5	0,5 … 100 mbar ..(0,1 … 100) mbar	100mbar kapazitiv	CMR362* Pfeiffer CMR372* CLR392*	0,2 mbar
6	0,05 … 10 mbar ..(0,01 … 10) mbar	10mbar kapazitiv	CMR363 Pfeiffer CMR373 CLR393	0,02 mbar
7	0,005 … 1 mbar (0,001 … 1) mbar	1mbar kapazitiv	CMR364 Pfeiffer CMR374 CLR394	0,002 mbar

CMR37X CMR36X

Azbil V8C

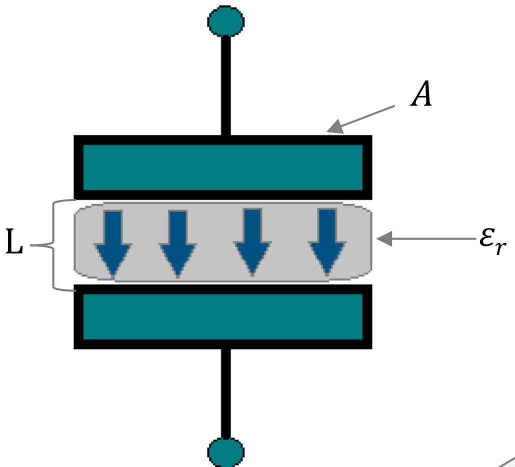
- **Features**: temperatur-controlled, temperatur-compensated, unregulated,
- **Applications**: all vacuum applications, corrosive gas resistant
- **Miscellameous**: cannot be sterilized (SIP). → MKS Barathron 627, 628, 631, …

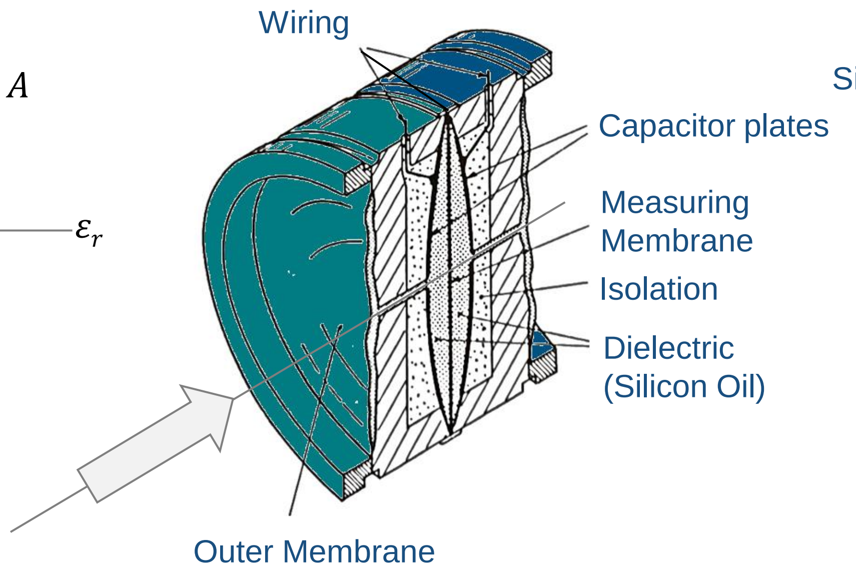
- **Measuring principle**: deflection of membrane causing a change in capacity

Principle of construction

- Functional design of an capacitor (two plates and a dielectric isolator)

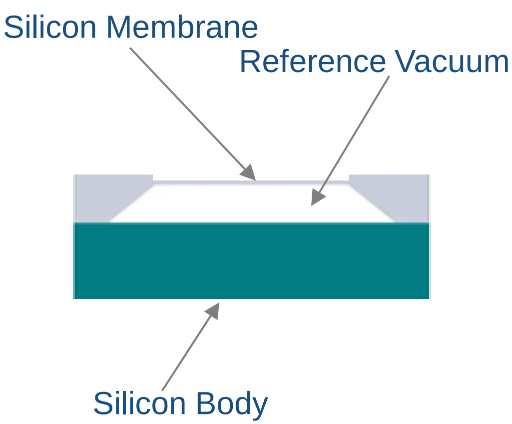
$$C = \varepsilon_0 \cdot \varepsilon_r \cdot \frac{A}{L}$$





MEMS Sensors

***M**icro**ME**chanical**S**ystems*



$Capacity = C$

$Dielectric\ constant = \varepsilon_0$

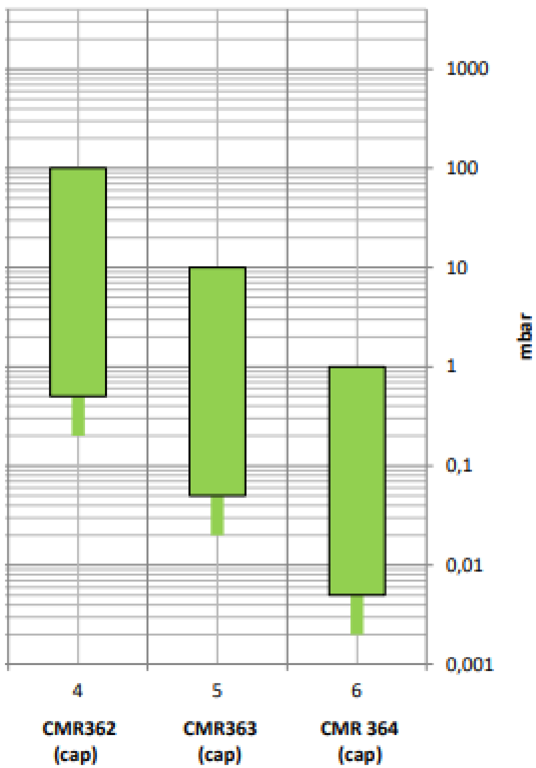
$Dielectric = \varepsilon_r$

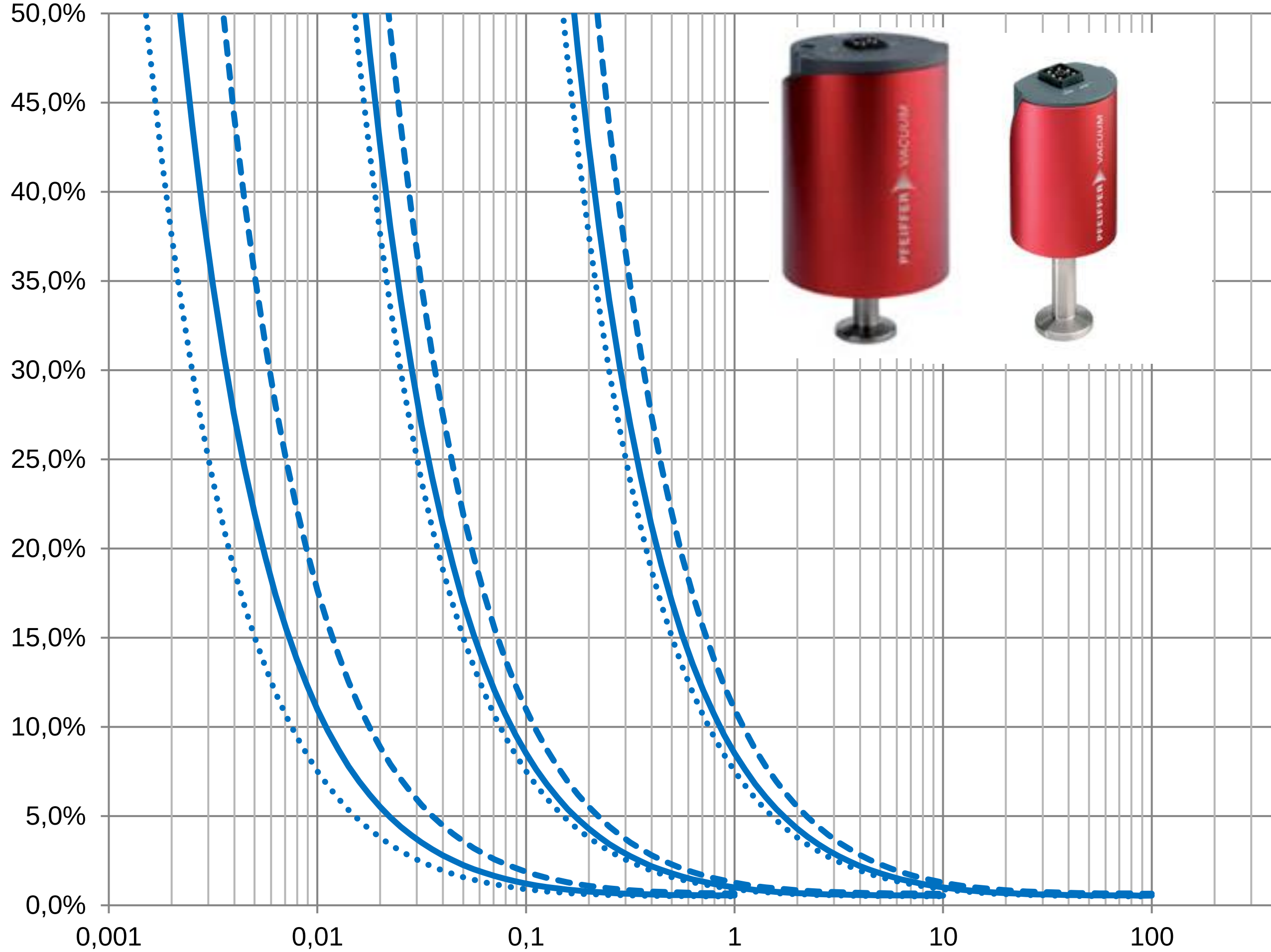
$Surface = A$

$Distance\ between\ plates = L$

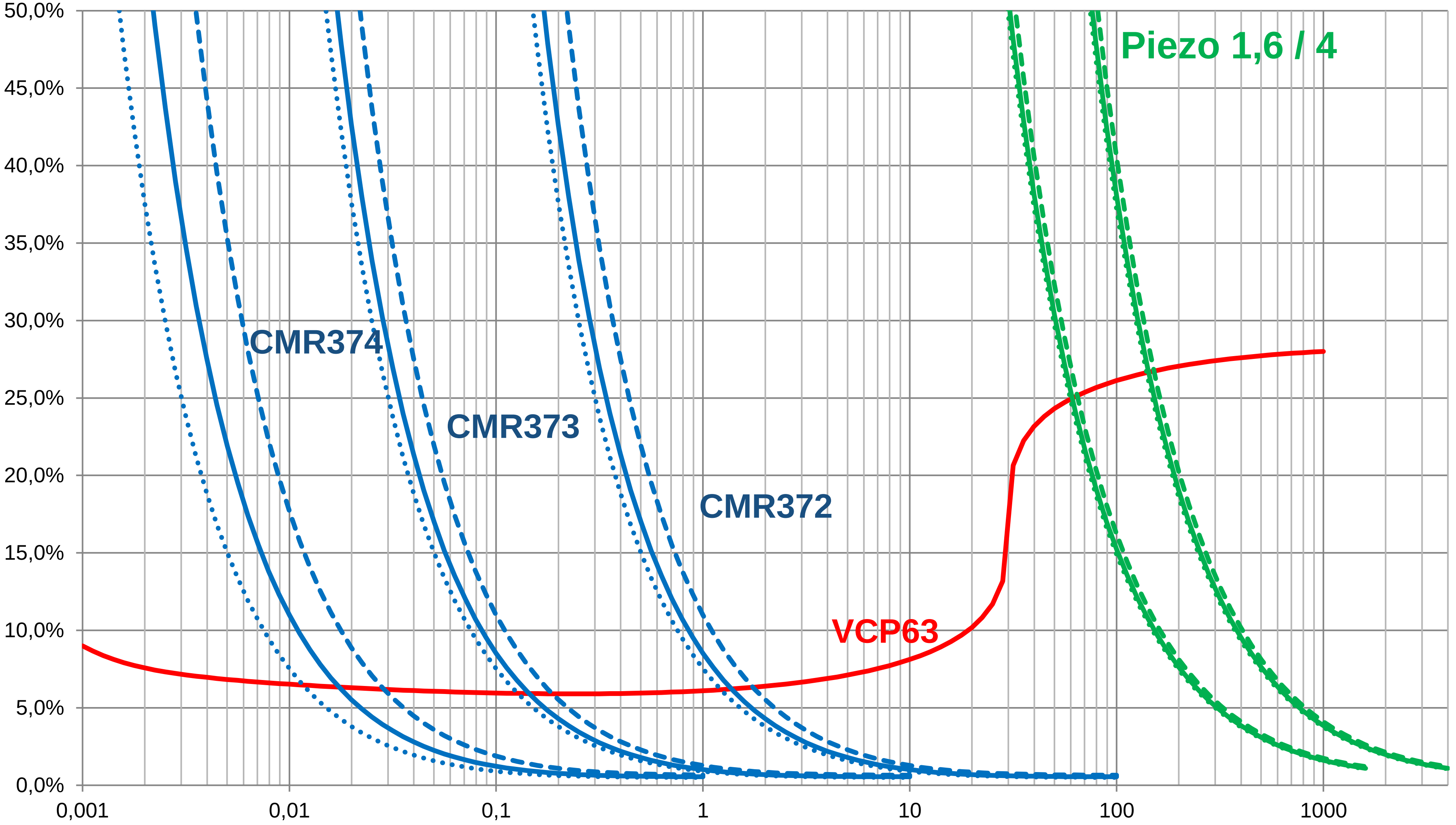
Vacuum Sensors uncertainty & useful range

- 2,5 decades max!

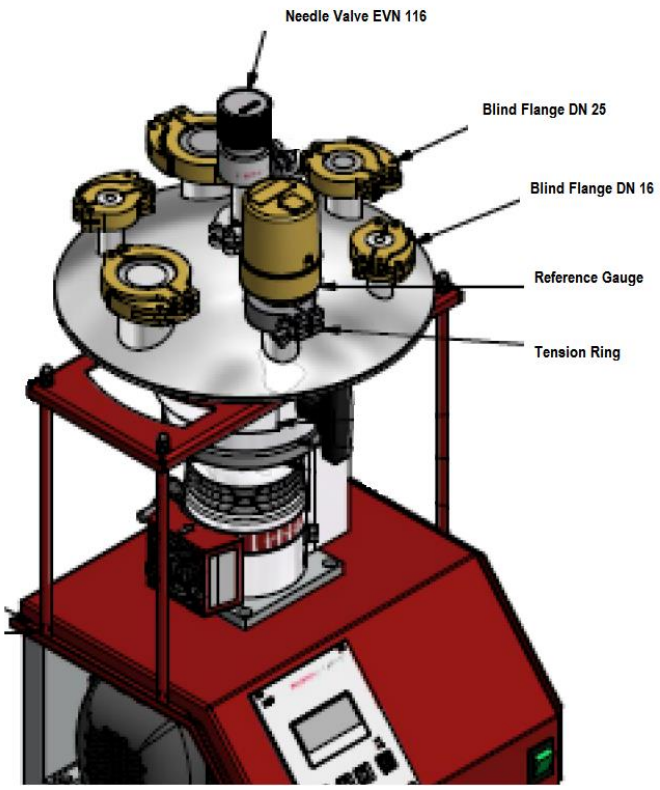




Vacuum Sensors uncertainty overview



Vacuum Sensor Calibrator (reference)



- HiCube80
- turbo-molecular pump 1e-7 mbar
- Rotary vane pump >1mbar
- Reference gauge CMR372/374/375
- Calibration vessel (recipient) in symmetric shape
- Micro aeration valve

Temperature Calibration

Temperature sensors -wired- in use

Device to be calibrated:

- **Sensor:** resistance thermometer PT100
  - **Type**: JUMO Platin Sensor - PTC (positive temp. coeffizient)
  - Resistance of 100Ω at 0° C

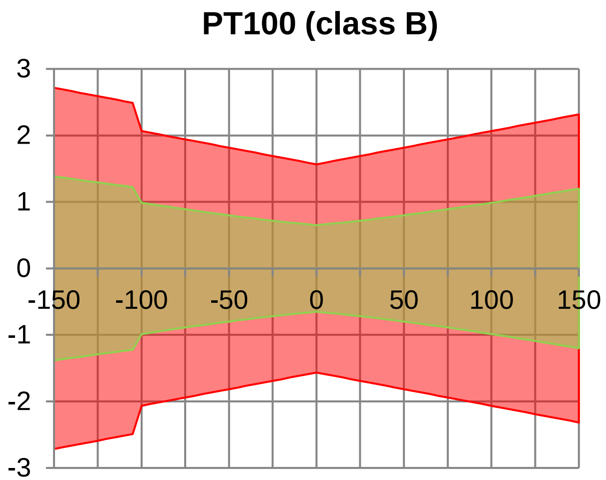
Measuring point	Sensor	Manuf.	Working Range (Measuring Range)	Deviation
Stellflächen-/ Eiskondensator-Vorlauftemperatur	PT100 (B) 3-Leiter	Jumo	-80 … 50 °C (-150 … 150 °C =)	± 1,0 K ± 1,5 K
(Kundenanforderung Präzisionsmessfühler)	PT100 (A) 3-Leiter	Jumo	-80 … 50 °C (-150 … 150 °C)	± 0,8 K ± 1,2 K
Filter-/ Sterilisationstemperatur	PT100 (B) 3-Leiter	Jumo	-110 … 140 °C (-150 … 150 °C)	± 1,0 K ± 1,5 K
Produkttemperatur/ LyoTemperatur	PT100 (B) 2-Leiter	Jumo	-60 … 50 °C (-150 … 150 °C)	± 1,7 K ± 3,0 K

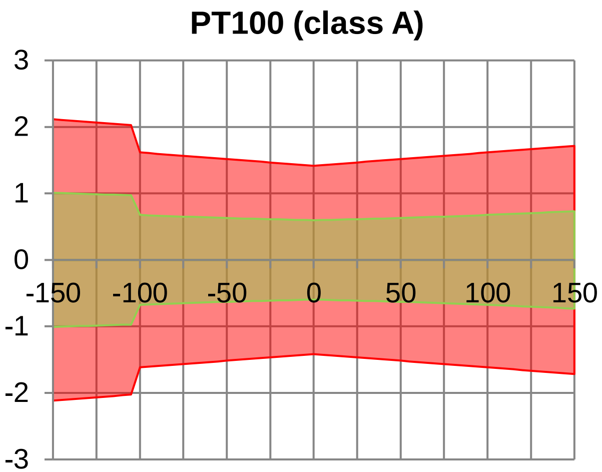
  - **Features**: Available in different designs
  - **Application**: Temperature measurement in all applications
  - **Miscellaneous**: + almost no drift, + low deviation,

  - **Measuring principle**: Resistance thermometers measure the temperature based on the temperature dependency of an electrical conductor .

Measuring and working ranges

- Deviation depending on sensor + measuring loop
- ! temperature-dependent conductor resistance (wire, connector, length, diameter, material, temperature. etc.)





- DIN EN 60751:2009
- Klasse A: dT = ± (0,15 ° C + 0,002 · T)
- Klasse B: dT = ± (0,30 ° C + 0,005 · T)

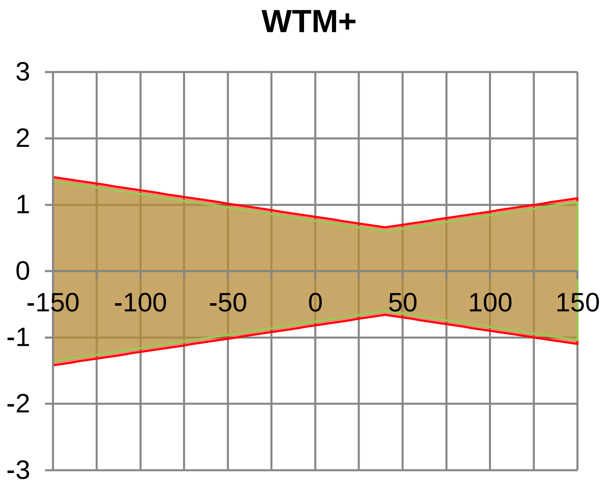
Temperature sensors -wireless- in use

Device to be calibrated:

- **Sensor:** WTMplus **W**ireless **T**emperature **M**easurement Sensor

  - **Type**: WTM, WTMplus, WTMplus 2.0

Measuring point	Sensor	Manuf.	Working Range (Measuring Range)	Deviation
Produkttemperatur	WTMplus	RSSI	-60 ... 50 °C (-150 ... 150 °C)	± 1,0 K ± 1,5 K



- **Features**: Available as in different designs
- **Application**: Temperature measurement in all applications
- **Miscellaneous**: + low deviation, + NO temperature dependent cable resistance + low max. error due to digital communication

- **Measuring principle**: temperature dependent Quarz is detuned by temperature change. The temperature dependency affects the oscillation frequency.

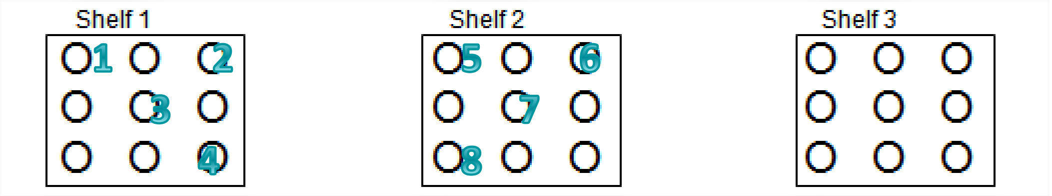
Temperature Calibrator

- Dryblock-calibrator i.e. Ametek Jofra, Isotech, etc.
- Temperature range -70 .. +140° C
- Fluke thermometer with WTMpuck

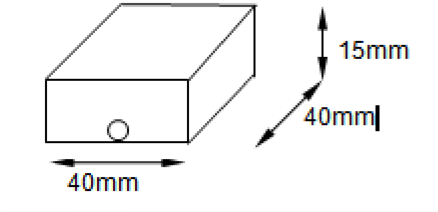
Shelf temperature distribution (Shelfmapping)

Shelfmapping - Arangement

- Reference ISO 13408-3 – Part 3 8.4.11f
- Distribution of temperature probes on all shelves.
- Inter- and intra shelf variations are recorded with 20 channels (measuring points per recorder) placed on 1 + n shelves
- Position of each sensor is documented

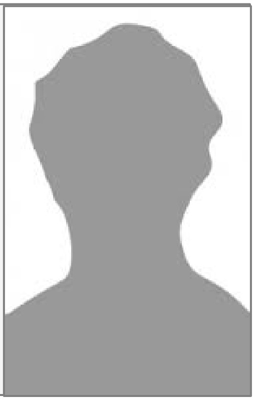


- An exact, direct temperature measurement on the surface of the shelves is technically not possible. For this purpose and to mitigate surrounding effects thermal-aluminium-blocks are used
- To avoid heat convection, vacuum must be applied between 10 .. 5 mbar
- As per default the distribution is measured at +20, -40, 0, +40° C
- Stabilisation time ~ 15 .. 30 min
- Acceptance criteria +/- 1K from average value. +/- 2K deviation to shelf inlet value.



Kontakt

Markus Wehner
Martin Christ Gefriertrocknungsanlagen GmbH
An der Unteren Söse 50, 37520 Osterode am Harz
Tel: 05522-5007-8521
Fax: 05522-5007-9921
Mail: m.wehner@martinchrist.de



Foto, Daten & Diagramm Quellennachweis

- Jumo „Elektrische Temperaturmessung“ ISBN 13-978-3-935742-06-1
- Pfeiffer Vakuum Asslar “The Vacuum Know-How Book Vol II.“
- Christ Produktmanagement, Produktdatenblatt Messtechnik 2.0.
- Industriesensorik, Vogel ISBN 978-3-8343-9076-5