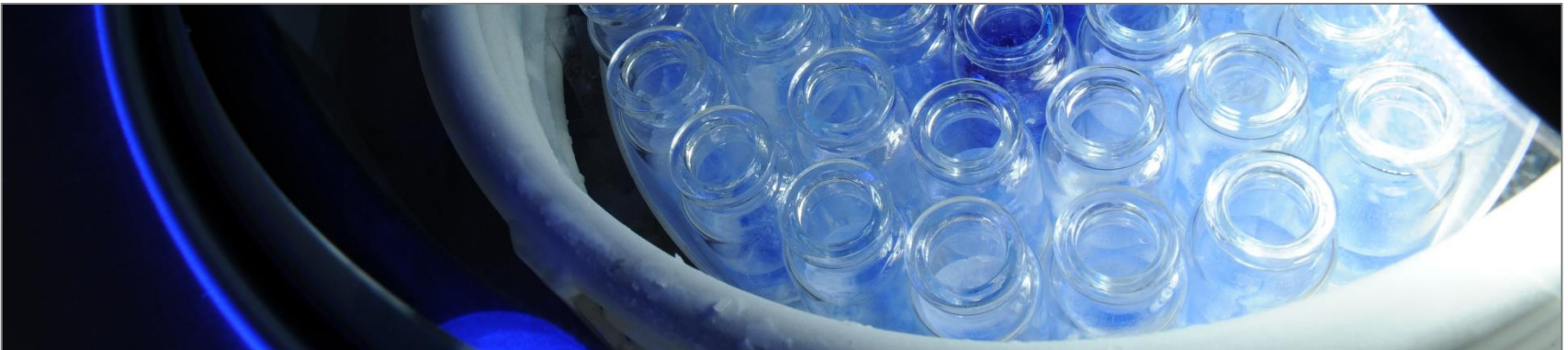

Practice 7, PDA-Seminar

Brief explanation of the different stations

2023 PDA Europe
Freeze Drying in Practice



Agenda

Station 1

- Pressure Calibration > ATM
- Vacuum Calibration



Station 2

- Temperature Calibration



Station 3

- Shelf-Mapping

Definitions

Calibration

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

Adjustment

The act or process of adjusting



The calibration workstations





Pressure Calibration



Pressure Sensors in use

Device to be calibrated:

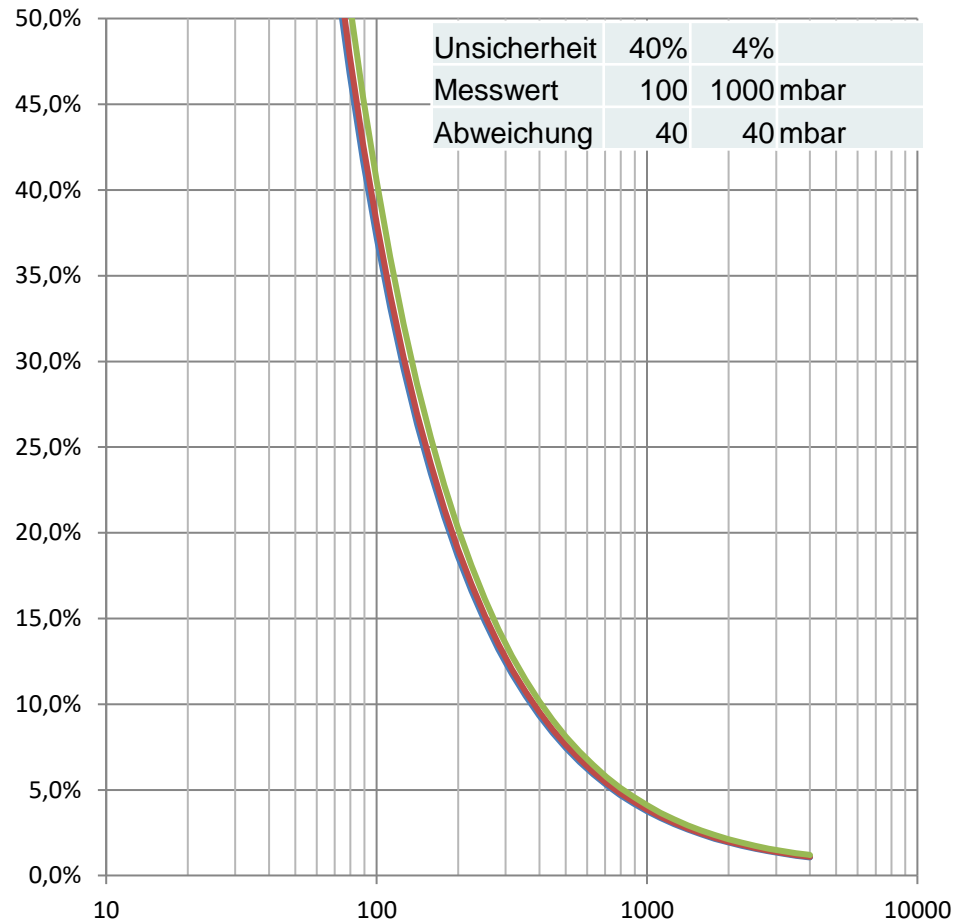
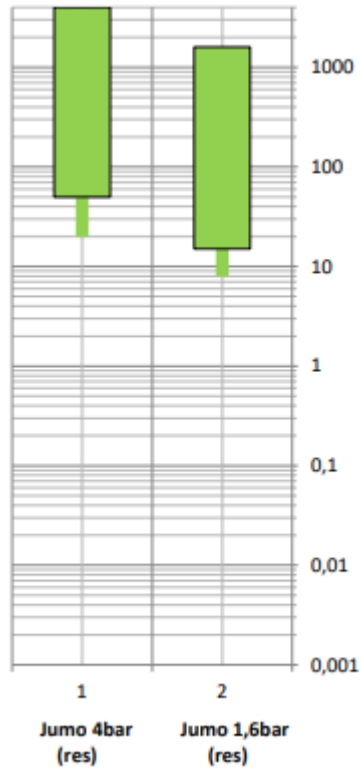
- **Sensor:** piezo-resistive pressure sensor
- **Type:** JUMO dTrans p31 pressure transducer

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
- **Application:** SIP, Door 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.

Pressure Sensors uncertainty & useful range



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:

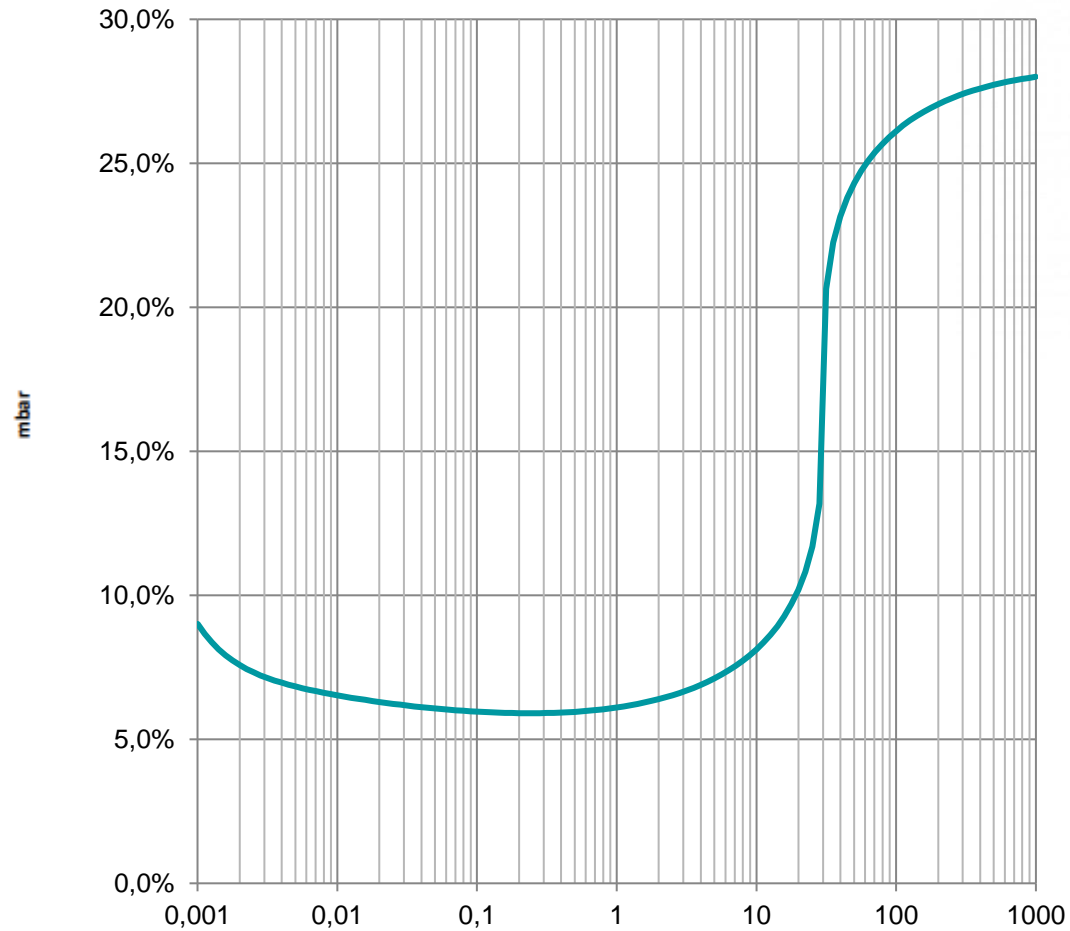
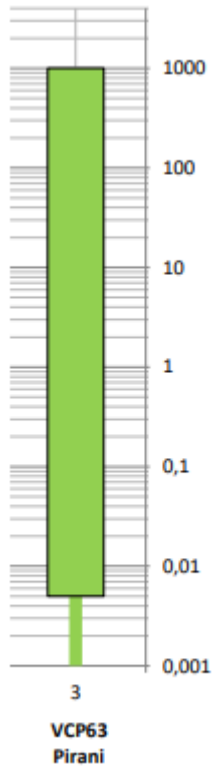
- **Sensor: Pirani** probes (gas dependent)
 - **Type: Thyracont VCP63MV** Pirani sensor with Platinum/Rhodium-Filament



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
- **Miscellaneous:** needs block valve for CIP, can be sterilized SIP (not powered) +150° C
no add. sensor heater required. Critical in ATEX applications
- **Measuring principle:** heated filament changes resistance due to reduced thermal conductivity

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)

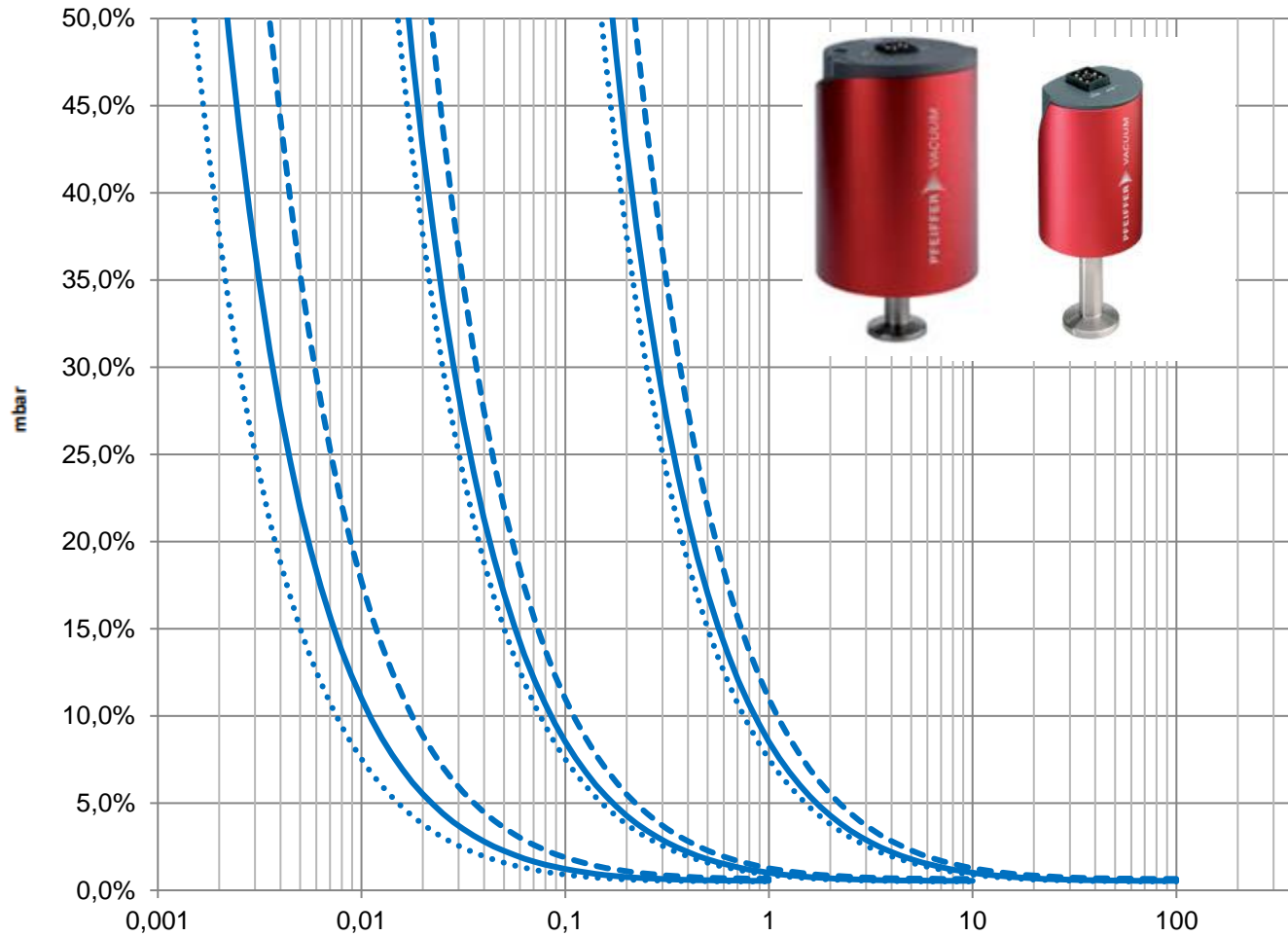
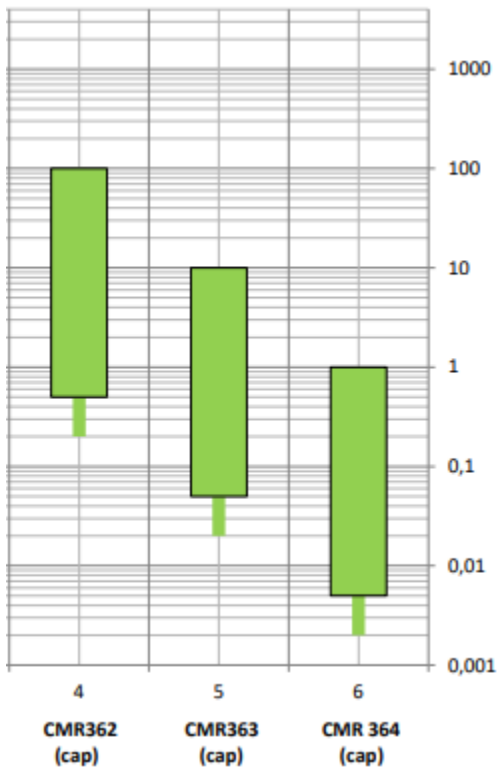
Nr.	Working Range (Measuring Range)	Sensor	Type	Deviation
5	0,5 ... 100 mbar ..(0,1 ... 100) mbar	100mbar kapazitiv	CMR362* Pfeiffer CMR372* CLR392*	0,2 mbar
6	0,05 ... 10 mbar ..(0,01 ... 10) mbar	10mbar kapazitiv	CMR363 Pfeiffer CMR373 CLR393	0,02 mbar
7	0,005 ... 1 mbar (0,001 ... 1) mbar	1mbar kapazitiv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



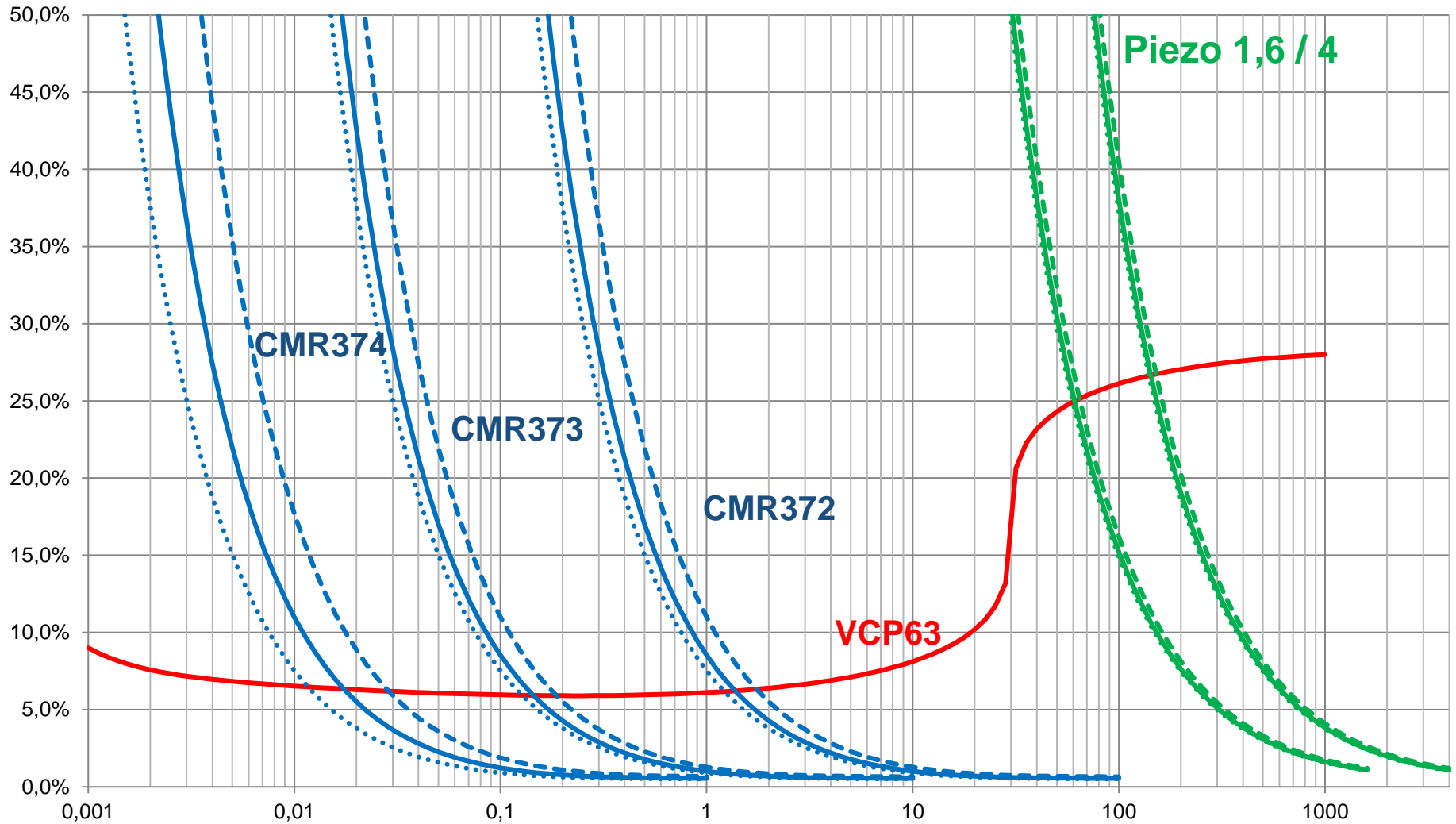
- **Features:** temperatur-controlled, temperatur-compensated, unregulated,
- **Applications:** all vacuum applications, corrosive gas resistant
- **Miscellaneous:** cannot be sterilized (SIP). → MKS Baratron 627, 628, 631, ...
- **Measuring principle:** deflection of membrane causes a change in capacity

Vacuum Sensors uncertainty & useful range

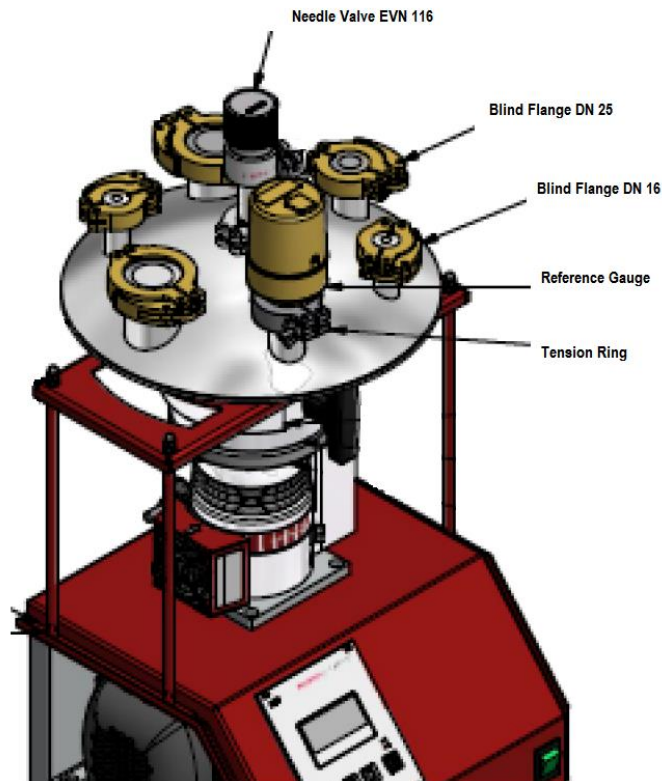
- 2,5 decades max!



Vacuum Sensors uncertainty overview



Vacuum Sensor Calibrator (reference)



- HiCube80
- turbo-molecular pump $1e-7$ mbar
- Rotary vane pump >1 mbar
- 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. coefficient)
- Resistance of 100Ω at 0° C

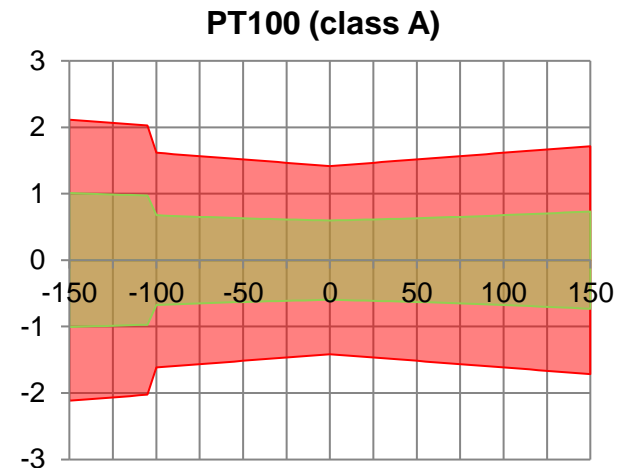
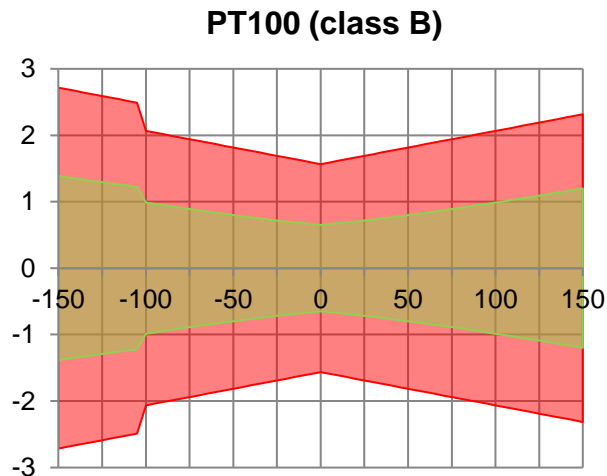
Measuring point	Sensor	Manuf.	Working Range (Measuring Range)	Deviation
Stellflächen-/ Eiskondensator- Vorlauftemperatur	PT100 (B) 3-Leiter	Jumo	-80 ... 50 °C	± 1,0 K
			(-150 ... 150 °C =	± 1,5 K
(Kundenanforderung Präzisionsmessfühler)	PT100 (A) 3-Leiter	Jumo	-80 ... 50 °C (-150 ... 150 °C)	± 0,8 K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



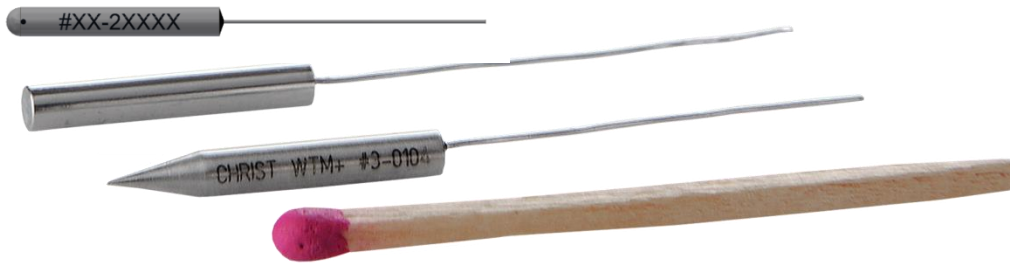
- DIN EN 60751:2009
- Klasse A: $dT = \pm (0,15 \text{ } ^\circ\text{C} + 0,002 \cdot T)$
- Klasse B: $dT = \pm (0,30 \text{ } ^\circ\text{C} + 0,005 \cdot T)$

Temperature sensors -wireless- in use

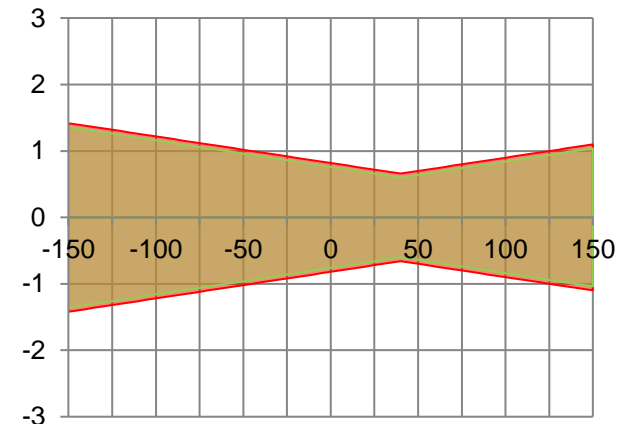
Device to be calibrated:

- **Sensor:** WTMplus Wireless Temperature Measurement Sensor
- **Type:** WTM, WTMplus, WTMplus 2.0

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



WTM+



- **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

- Dry-calibrator i.e. Ametek Jofra, Isotech, etc.
- Temperature range $-70 \dots +140^{\circ} \text{C}$
- Fluke thermometer with WTMpuck

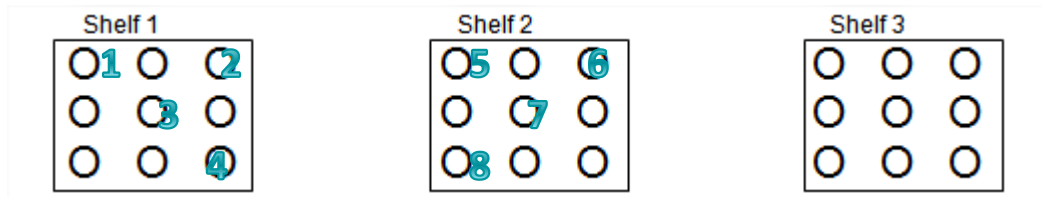


Shelf temperature distribution (Shelfmapping)

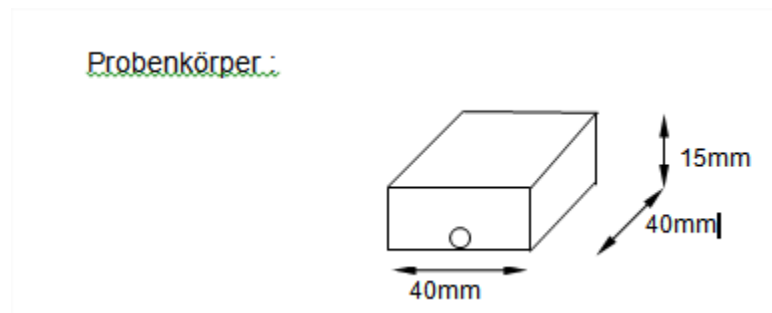


Stellflächentemperaturverteilung - Aufbau

- 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

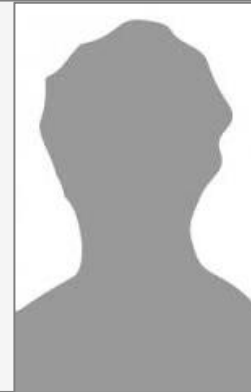


- An exact, direct temperature measurement on the surface of the shelves is technically not possible. For this purpose and to mitigate surrounding effects thermal-aluminium-blocks are required
- To avoid heat convection, vacuum must be applied : ca 1 mbar
- As per default distribution is measured at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



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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.

