DEUTSCHER KALIBRIERDIENST

Guideline DKD-R 6-2

Calibration of Measuring Devices for Vacuum

Part 5

Pirani Gauges

Published by the Accreditation Body of the Deutscher Kalibrierdienst (DKD) at the Physikalisch-Technische Bundesanstalt in co-operation with its Technical Committee "Pressure and Vacuum".

Deutscher Kalibrierdienst (DKD)

The DKD comprises calibration laboratories in industrial enterprises, research institutes, technical authorities, inspection and testing institutes. They are accredited and supervised by the DKD. They calibrate measuring instruments and material measures for measurands and measurement ranges specified within the scope of accreditation. The DKD calibration certificates issued by these laboratories prove traceability to national standards as required in the ISO 9000 family and ISO/IEC 17025.

Calibrations carried out by DKD laboratories ensure that the user may rely on measurement results. They increase the customers' confidence and competitiveness on the national and international markets and serve as a metrological basis for the inspection of measuring and test equipment within the framework of quality assurance measures.

Calibrations offered by the DKD cover electrical measurands, length, angles and other geometrical quantities, roughness, coordinate and form measuring techniques, time and frequency, force, torque, acceleration, pressure, flowrate, temperature, humidity, medical measurands, acoustic measurands, optical measurands, ionizing radiation and other measurands.

Publications: see Internet

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Foreword

DKD Guidelines are application documents for the general criteria and procedures which are laid down in DIN EN ISO/IEC 17025 and DKD publications. The DKD Guidelines describe technical and organizational processes serving the calibration laboratories as a model for laying down internal procedures and regulations. DKD Guidelines can become an integral part of quality manuals of calibration laboratories. The application of the Guidelines supports equal treatment of the devices to be calibrated at the different calibration laboratories and improves the continuity and verifiability of the work of the calibration laboratories.

The DKD Guidelines will not impede the further development of calibration procedures and sequences. Deviations from guidelines and new methods are permitted in agreement with the Accreditation Body if they are justified by technical aspects.

The present Guideline was prepared by the Technical Committee "Pressure and Vacuum" in cooperation with the PTB and adopted by the Advisory Board of the DKD. With its publication it is binding for all DKD calibration laboratories unless separate procedural instructions approved by the Accreditation Body are available.

This document is a translation of the German Guideline R 6-2. In case of any disputes the respective German version is binding.

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1 Scope of application

1.1 Pirani gauges
1.2 Heat-convection gauges operating on the convection principle
1.3 Thermoelectric vacuum gauges
1.4 Other heat-convection gauges

2 Pressure range

Typically 10^{-4} mbar to 1000 mbar.

3 Standards and measuring equipment

3.1 Reference and working standards

The calibration is carried out by direct comparison of the measurement values for the calibration item with those of a reference or working standard. These have been directly or indirectly traced back to a national standard.
The standards used are suitable pressure measuring instruments such as, for example, diaphragm gauges or spinning rotor gauges. They are calibrated at regular intervals and are traced back directly or indirectly to a national standard.
The standards documented in the quality manual of the DKD laboratory are calibrated at an accredited laboratory and a calibration certificate is issued for them in which the expanded uncertainty under reference conditions is stated. The standards are subject to approval by the PTB. They can be very different as regards their design.
For calibration outside these reference conditions, corrections to the pressure calculation are to be carried out. The measurement uncertainties to be attributed to the influence quantities effective in the measurement are to be taken into account in the uncertainty budget as further uncertainty components.

3.2 Apparatus

(according to ISO/CD 3567 as of 09/99)

- The volume of the vacuum chamber should at least be 20 times the total volume of the connected vacuum gauge, including the associated connecting lines.
- The vacuum chamber should be such that the ratio between wall surface and volume is as small as practically possible (ideal case: sphere); this ratio should not exceed the value given by a right circular cylinder whose length is twice the diameter.
- The connection between vacuum chamber and the rest of the vacuum system must be such that the entering gas flow strikes neither the vacuum gauges to be calibrated nor the standards nor the orifices opening on the vacuum gauges.
- The standards and the vacuum gauges to be calibrated must be arranged on the test chamber so that pressure and temperature differences do not lead to considerable errors (equivalent measuring connections). The conductance of the tube connections between measuring chamber and vacuum gauge should at least be some litre per second to keep the influence of adsorption and desorption effects small. The gas flow (inlet and evacuation) must not reach the active zone of the vacuum gauge directly.

- The residual gas pressure, i.e. the pressure prevailing in the vacuum chamber without gas being admitted must not exceed 10% of the lowest calibration pressure. If a smaller uncertainty is to be reached, the residual gas pressure must be lower.
- The vacuum gauges must not exert an influence on one another; if need be, suitable precautions have to be taken.
- The purity of the gas should be equivalent to a maximum impurity level of 0,1% by volume.

4 Calibration item

Pirani gauge with indication and/or analog output and/or digital interface.

5 Calibratability

Handling of the calibration commission presupposes that the calibration item is calibratable (suitable for calibration), i.e. the state of the calibration item at the time of calibration should comply with the generally accepted rules of technology as well as with the particular specifications of the manufacturer's documentation. The calibratability is to be ascertained by external inspections and functional tests.

External inspections cover, for example:

- visual inspection for damage (pointer, inscriptions, readability of indications, set-up of measuring system, sealing surface), contamination and cleanliness. Recommendation: have decontamination certified by customer.
- check whether the documents necessary for calibration (technical data, operating instructions) are available.

Functional tests cover, for example:

- tightness of calibration item
- electrical function
- perfect function of operating elements (e.g. adjustability of zero point)
- adjusting elements in defined position
- faultless execution of self-check and/or self-adjusting functions; if appropriate, internal reference values are to be read out via the EDP interface.

Note: If repair measures must be taken to provide calibratability, this work should be arranged by the customer and the calibration laboratory.

The stability of the indication and the reproducibility in particular are important criteria for the state of the vacuum gauge. Residues of air humidity and other residues, e.g. of process substances must be removed from the measuring cell of the calibration item. This is achieved by evacuation, possibly assisted by baking out.

6 Adjustments of calibration item

Prior to calibration, the adjustments of the vacuum gauge must be made in accordance with the manufacturer's specifications or in agreement with the customer (e.g. measuring channel, thermostating, gas type, configuration of the output signal, etc.). Adjustments (e.g. for zero point, full-scale) should be made in agreement with the customer.

7 Ambient conditions

The calibration should be carried out at an ambient temperature of 20°C to 26°C, preferably at 23°C. The temperature variations should not exceed ± 1°C.

8 Calibration method

The vacuum gauges to be calibrated and the appropriate reference and working standards are connected to the vacuum chamber (see 3.2) in which the pressures are adjusted. The vacuum chamber must be so designed that the pressures at the measuring points agree to such an extent that comparisons can be carried out with the accuracy necessary.

An example of a calibration system is given in Annex B.

The vacuum gauge is to be calibrated as a whole (measuring chain), if possible.

The mounting position of the pressure transducer recommended/specified by the manufacturer or agreed with the customer is to be taken into account.

9 Performance of calibration

9.1 Prerequisites

Prior to calibration,

1. the calibration item and the standards must be temperature stabilized. Unless otherwise specified by the manufacturer, a stabilization time of 0,5 h is recommended.

2. calibration item and standards must have been adjusted in accordance with section 6. The zero adjustments and the adjustments of the full-scale deflection of the calibration item (if setting devices are available) are made according to the manufacturer's specifications.

9.2 Adjustment of calibration pressures

Unless otherwise agreed with the customer, at least three calibration pressures per decade (e.g. 1, 2, 5) but at least 10 calibration pressures on the whole shall be recorded.

Recording of the calibration values takes place from small to large pressures, in the ascending direction. In each measuring point one has to wait until the output quantities of calibration item and standard have reached a steady state.

10 Evaluation, calibration result

The main components of the pressure measuring facility are each provided with a calibration mark; as to measuring chains, each device will be provided with a calibration mark.

In addition to the requirements of DKD-5, the following statements are to be made in the calibration certificate:

- measuring gas
- mounting position of calibration item
- auxiliary measuring equipment used
- adjustments on calibration item.

In accordance with Guideline DKD-5, the measurement values can be represented in different ways. If they are represented in the form of a table, this must at least contain:

- the calibration pressure
- the signal (e.g. pressure indication, d.c. voltage output) of the calibration item.

Furthermore, the calibration certificate may state:

- the measuring deviations
- the relative measuring deviations
- further measurement values and calculations.

Example of calibration result representation:

Consecutive number	Calibration pressure mbar	Indication on calibration item mbar	Measuring deviation %	Expanded uncertainty %
1	1,004	1,1 E+0	9,6	5,2
2	2,048	2,1 E+0	2,5	5,0
3	4,992	4,9 E+0	-1,8	5,0
4	9,903	1,0 E+1	1,0	5,0
5	20,16	2,4 E+1	19,0	7,5

The expanded uncertainty is to be stated in accordance with DKD-5.

Corrections applied to the measurement value are to be precisely described.

If the expanded uncertainty and the deviation are stated in the table, the calibration certificate must contain the following remark:

"The expanded uncertainty relates to the indication of the calibration item given in the table after this has been corrected by the deviation from the calibration pressure."

Annex A

A.1 Pirani gauge

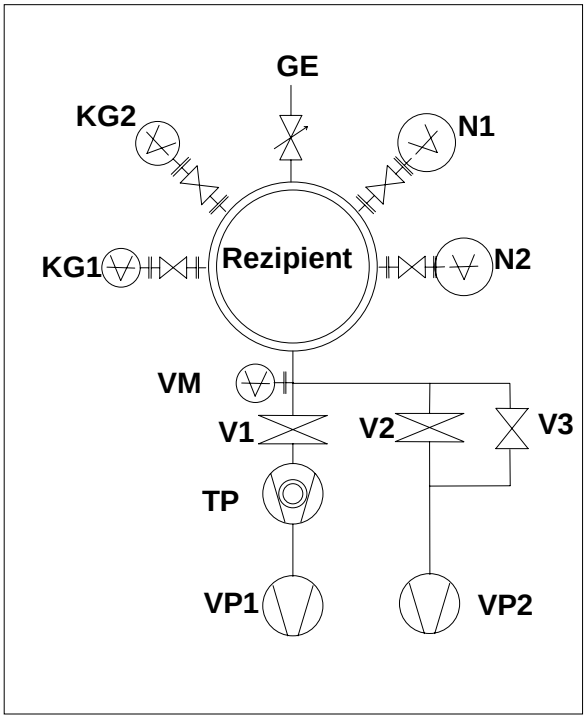
In a Pirani gauge the heat flow transferred due to the heat conduction of gas from an electrically heated thin wire to the wall of the measuring tube serves as the measure of the gas pressure. The heating wire typically consists of tungsten, platinum or nickel. The Pirani design which is widely used measures the temperature-dependent electrical resistance of a heating wire (spiral or straight). The heating wire is integrated as the variable resistor into a measuring bridge which is balanced at 10^{-3} mbar to 10^{-5} mbar. The output voltage of the bridge then is a measure of the pressure in the measuring tube. Controlled systems in which the temperature of the heating wire is kept constant are very frequent. The supply of the necessary electrical energy is pressure-dependent.

In thermoelectric vacuum gauges, a thermocouple is used to measure the temperature of the heating wire.

All Pirani gauges measure in dependence on the gas type and are in most cases balanced for nitrogen or air. The typical measurement range lies between 10^{-3} and 100 mbar. Some designs have measurement ranges from some 10^{-4} mbar to 1000 mbar.

Annex B

B.1 Calibration system



Symbols:

GE	gas inlet
N 1,2...	reference standards
KG 1,2...	calibration items
VM	vacuum gauge for residual pressure indication, e.g. ionization or Pirani pressure gauge, possibly also for pump control
V1	flow reduction valve, conductance adjustable for dynamic pressure regulation between 0,001 mbar and 10 mbar. If complete shut-off is not possible, an additional stop valve must be provided.
V2	stop valve
V3	valve with small conductance, parallel to V2, for slow discharge
TP	turbomolecular pump
VP1	backing pump for TP1
VP2	pump for pre-evacuation

Standard and calibration item can be flanged via stop valves. Unnecessary flooding of the recipient for exchange is thus avoided and the transport of the vacuum gauges in vacuo is possible.

If low residual pressures are necessary, it may be necessary to bake out the calibration system.