DEUTSCHER KALIBRIERDIENST



# Guideline DKD-R 6-2

# Calibration of Measuring Devices for Vacuum

Part 3

Electrical Diaphragm 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



## Address:

Deutscher Kalibrierdienst at the
Physikalisch-Technische Bundesanstalt

| | |
|---|---|
| Bundesallee 100 | D-38116 Braunschweig |
| P.O. Box 33 45 | D-38023 Braunschweig |
| Office telephone | +49 531 592 1901 |
| Fax | +49 531 592 1905 |
| E-Mail | dkd@ptb.de |
| Internet | www.dkd.info |

## 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 in 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.

## Contents

## 1 Scope of application

Directly measuring electrical diaphragm gauges, e.g. capacitive, piezoresistive and similar pressure transducers for absolute pressure measurement.

## 2 Pressure range

Typically $10^{-6}$ 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 the reference or working standard. These have been directly or indirectly traced back to a national standard. The standards used are vacuum gauges with long-term stability.

The working 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. Working standards are subject to approval by the PTB. The standards can be very different as regards their type.

### 3.2 Special features when piston gauges are used as standards

The vacuum chamber (glass bell) of the piston gauge must be sufficiently evacuated to a residual gas pressure $p_{res}$ (recommended: $p_{res}$ <= 0,05% · p). The residual gas pressure must be measured. The calibration pressure $p_{kal}$ is obtained as the sum of pressure difference $p_{kol}$ at the piston and the residual gas pressure:

$$p_{kal} = p_{kol} + p_{res}$$

The uncertainty of the residual gas pressure must be allowed for in the calculation of the uncertainty of the calibration pressure.

If possible, a separate pump should be used to evacuate the bell.

### 3.3 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 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 1 % by volume.

## 4 Calibration item

Electrical diaphragm gauges 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 agreed by the customer and the calibration laboratory.

The stability of the zero point in particular is an indication of the state of the diaphragm gauge. Rests of air humidity and other residues, e.g. process substances, must have been completely removed from the measuring cell of the calibration item. This is achieved by evacuation, possibly assisted by baking out.

If necessary, the following special functional tests can be carried out:

a) Repeated filling with dry gas to twice to three times the FS but not beyond the maximum permissible pressure, with subsequent evacuation below the limit of resolution. The scatter of the zero indication should lie within the manufacturer's specifications. If the scatter is larger or if manufacturer's specifications are not available, the calibratability should be discussed with the customer. If after every cycle the zero indication is shifted in the same direction, the calibration item is not calibratable (e.g. due to contamination, defects, e.g. reference vacuum loss).

b) Recording of the zero signal for 12 h or more, e.g. with a y/t recorder or data acquisition system. If the ambient temperature influences the zero point, this must also be recorded. On the basis of the manufacturer's specifications for the temperature coefficient, a statement on the temperature stability of the calibration item can be made.

## 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, configuration of the output signal, etc.). Adjustments (e.g. linearity, FS) 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 variation should not exceed ± 1°C.

## 8 Calibration procedure

The vacuum gauges to be calibrated and the associated reference and working standards are connected to a vacuum chamber 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 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, the calibration items and standards must be

1. temperature-stabilized.

   If manufacturer's specifications are not available, the following are recommended as stabilization times:
   non-thermostated devices: 1 hour
   thermostated devices: 4 hours

2. be adjusted in accordance with section 6.

   The zero point of calibration item and standard is to be adjusted in accordance with the manufacturer's specifications. If such specifications are not available, a residual pressure not exceeding 1/10 of the response threshold (in manufacturer's specifications frequently referred to as resolution) of the calibration item is to be adjusted.

Hint:

The time for thermostating can additionally be used for evacuating and outgassing the measuring cell. This also serves to reduce the time for producing the vacuum necessary for zero adjustment.

### 9.2 Adjustment of calibration pressures

Unless otherwise agreed with the customer, at least ten calibration points uniformly distributed over the calibration range shall be recorded. For calibrations over several decades, at least three calibration points per decade shall be recorded (e.g. 1, 2, 5).

Unless otherwise agreed, 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.

To check the zero point stability, the zero point of the calibration item can be checked after termination of the measurement series.

## 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
- calibration method (e.g. DIN 28418 or this Guideline)
- mounting position of calibration item
- adjustments on calibration item.

The calibration certificate should contain a table with all measurement values. It must include at least:

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

Furthermore, the calibration certificate can state:

- the deviations
- the relative deviations
- further measurement values, calculations, etc.

Example of calibration result representation

(pressure transducer with 10 mbar measurement range and 2-10 V signal output)

| calibration pressure mbar | calibration item | | deviation mbar | expanded uncertainty mbar |
|---|---|---|---|---|
| | output signal $U_a$ V | calculated pressure* mbar | | |
| 0,1000 | 2,084 | 0,105 | 0,005 | 0,0036 |
| 0,2008 | 2,163 | 0,204 | 0,003 | 0,0070 |
| 0,5001 | 2,396 | 0,495 | -0,005 | 0,0165 |
| 1,0220 | 2,810 | 1,013 | -0,009 | 0,0304 |
| etc. | | | | |

* calculated in accordance with manufacturer's specifications:

$$p = (U_a - 2\ \text{V}) \cdot (10\ \text{mbar}/8\ \text{V})$$

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

### 10.1 Corrections

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 note:

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

### 10.2 Thermal effusion

In most diaphragm gauges for low pressure ranges, the measuring cells (sensors) are heated to increase the stability. The result normally is a temperature gradient between the measuring cell and the vacuum chamber, and the pressure in the measuring cell is higher. In the range <1 mbar this leads to systematic deviations of some (typically 3 to 4) percent. The functional relation is either specified by the manufacturer or can be determined by a balancing calculation.
If the corrected values are stated in the calibration certificate, the correction procedure used must be expressly stated or described.

# Annex A

## A.1 Capacitive diaphragm pressure transducer

**(capacitance manometer, capacitive vacuum gauge)**

A measuring cell accommodates an elastic diaphragm subdividing it into two separate, leak-proof chambers. The diaphragm bends according to the amount of pressure applied. At a small distance from it, according to the sensor design, one or two electrodes are placed on an isolated beam, either in the form of a disc or in the form of a ring and a disc. Together with the electrode(s), the diaphragm which either is itself electrically conducting or has a conducting surface forms a parallel-plate capacitor whose capacitance is a function of the distance of the diaphragm from the electrode(s) or directly depends on the pressure. The capacitance is converted into d.c. voltage which is linear to the pressure.
In absolute pressure transducers, the space behind the diaphragm is permanently evacuated so that the pressure applied before the diaphragm always occurs as absolute pressure. For the absolute pressure measurement using differential pressure transducers, the space behind the diaphragm must be evacuated to such an extent that the residual pressure is smaller than the resolution.

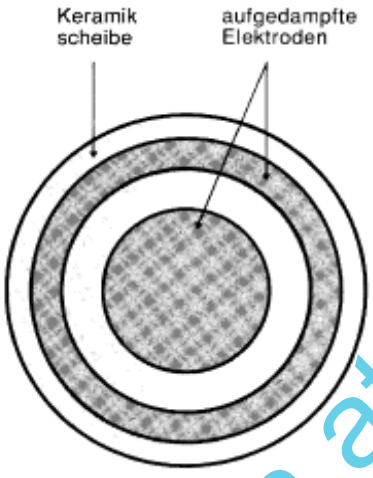



Figure 1: Measuring cell

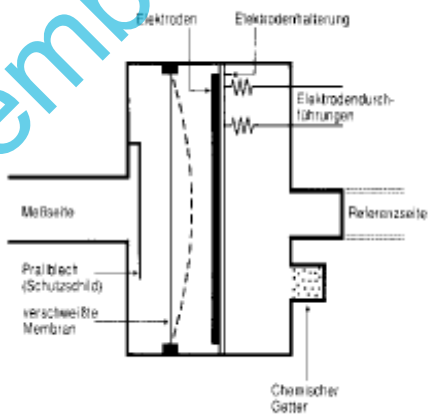



Figure 2: Electrode arrangement

## A.2 Thermal effusion

**(Thermal transpiration)**

This effect results with diaphragm gauges with heated measuring cell in the region of molecular flow. Due to the higher temperature of the measuring cell and the associated higher particle velocity, a higher pressure results in the measuring cell. The pressure below which the effect occurs depends on the particle size for the type of gas and typically is 1 mbar. For very small pressures, the ratio of the pressure $p_M$ in the measuring cell to the pressure $p_R$ in the recipient is

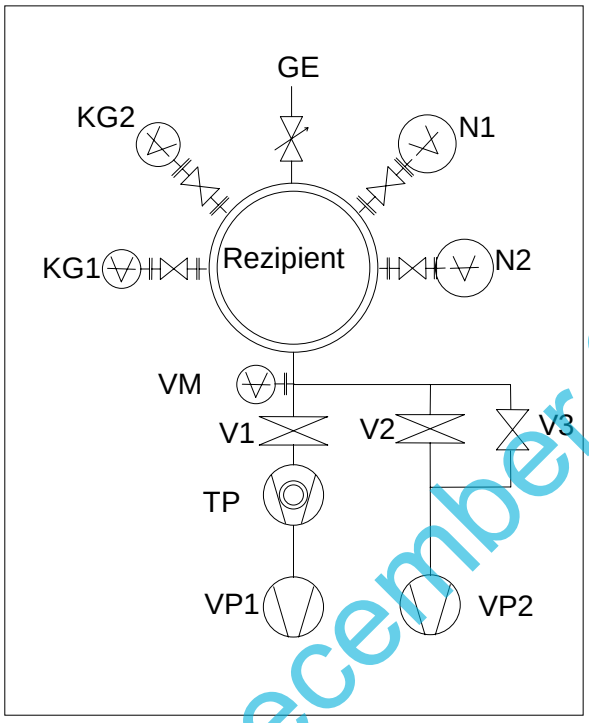
$$p_M / p_R = \sqrt{\frac{T_M}{T_R}}$$

The deviation in the transitional region between viscous and purely molecular flow is described by a nonlinear transition function.

## Annex B

### B.1 Calibration system

Figure 3: Example of 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. ion or Pirani gauge, possibly also for pump control |
| V1 | flow reduction valve, conductance adjustable for dynamic pressure regulation between 0,001 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 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.