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**Guideline**      **Calibration of Measuring**  
**DKD-R 6-2**      **Devices for Vacuum**  
Part 4              Ionization Gauges

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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.

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**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 Different types of measuring systems with hot cathode
  - 1.1.1 Triode measuring systems
  - 1.1.2 "High-pressure" ionization measuring systems with hot cathode
  - 1.1.3 Bayard-Alpert measuring system (systems with thin collector)
  - 1.1.4 Bayard-Alpert measuring systems with modulator
  - 1.1.5 Bayard-Alpert measuring systems with hidden collector or with extractor
- 1.2 Different measuring systems with cold cathode
  - 1.2.1 Penning measuring system
  - 1.2.2 Inverted magnetron
  - 1.2.3 Other types with field emitter cathode

## **2 Pressure range**

Typically  $10^{-12}$  mbar to 1 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 suitable pressure measuring instruments such as, for example, ionization gauges, spinning rotor gauges and diaphragm 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 type.

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 gauge 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 (admission and discharge) 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.

### 3.3 Other recommendations

When turbomolecular pumps are used, it may be suitable to increase the compression by connecting two turbomolecular pumps in series, since the residual pressure in the ultra-high vacuum region is essentially due to hydrogen (H<sub>2</sub>) and for physical reasons the compression of a turbomolecular pump for light gases is relatively low.

## 4 Calibration item

Ionization 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.

## **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. sensitivity, emission current, measuring channel, mode of indication, configuration of the electrical output quantity: linear/logarithmic, etc.).

## **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  $\pm 1^\circ\text{C}$ .

## **8 Calibration procedure**

The vacuum gauges to be calibrated and the associated 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 recommended/specified by the manufacturer or agreed with the customer is to be taken into account. In the case of immersion systems, the mounting geometry (diameter, length, form, etc., of the connecting piece) is also to be stated.

## **9 Performance of calibration**

### **9.1 Prerequisites**

Prior to calibration,

1. the calibration item and the standards must be temperature-stabilized.  
If manufacturer's specifications are not available, the following are recommended as stabilization times:  
hot-cathode vacuum gauge: about 2 h  
cold-cathode vacuum gauge: about 0,5 h
2. if need be, the ionization gauge used as standard must be conditioned to enhance the stability (e.g. outgassing at a defined pressure in an argon atmosphere)
3. the calibration item and the standards must have been adjusted in accordance with section 6. The zero adjustments of calibration item and standard are to be made in accordance with 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
- 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	indication on calibration item	measuring deviation	expanded uncertainty
	mbar	mbar	%	%
1	1,09 E-08	1,15 E-08	5,5	9,2
2	2,03 E-08	2,15 E-08	5,9	8,1
3	5,11 E-08	5,38 E-08	5,3	8,1
4	1,03 E-07	1,10 E-07	6,8	6,1
5	2,00 E-7	1,91 E-07	-4,5	5,9

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 Ionization gauge

Ionization gauges are the most important measuring instruments to measure gas pressures in the high and ultra-high vacuum. They measure the pressure indirectly via the pressure-proportional particle number density. The gas in the sensors whose pressure will be measured is partially ionized. Ionization takes place through electrons which are accelerated in the electric field and thereby reach enough energy to form positive ions when colliding with gas molecules. The ions donate their charge back to the measuring electrode (ion catcher, collector) of the sensor. The ionic current produced (more exactly: the electron flow in the inlet of the measuring electrode necessary for neutralizing these ions) is a measure of the pressure, for the ion yield in the molecular region is proportional to the particle number density and thus to the pressure. After neutralization of the ions on the collector, the particles can again return into the gas atmosphere (e.g. noble gas atoms) or adhere to electrodes or walls. In this case, the pumping effect of the system is spoken of. The formation of the ions takes place either by collision with electrons emitted by a cathode (hot cathode or field-emitter cathode or similar) or in a discharge at high electric field strength, a so-called cold-cathode discharge. A distinction is made between two kinds of cold-cathode discharges: the Penning discharge (electric and magnetic field parallel to each other) and the magnetron discharge (electric and magnetic field vertical to each other).

Under otherwise identical conditions, the ion yield and thus the ionic current depend on the gas type, since some gases get more easily ionized than others.

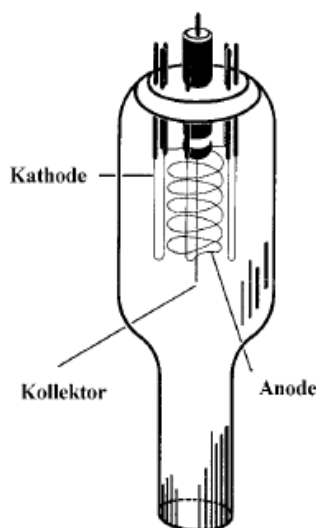


Figure 1:  
Hot-cathode ionization gauge  
Example: Bayard-Alpert measuring system

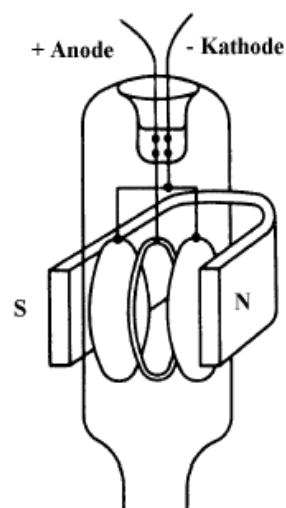


Figure 2:  
Cold-cathode ionization gauge  
Penning discharge: electric and magnetic field  
parallel to each other

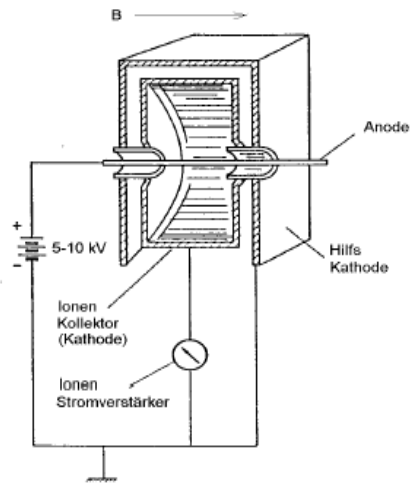


Figure 2b:  
Cold-cathode ionization gauge  
(Inverted) magnetron discharge: electric and magnetic field  
vertical to each other

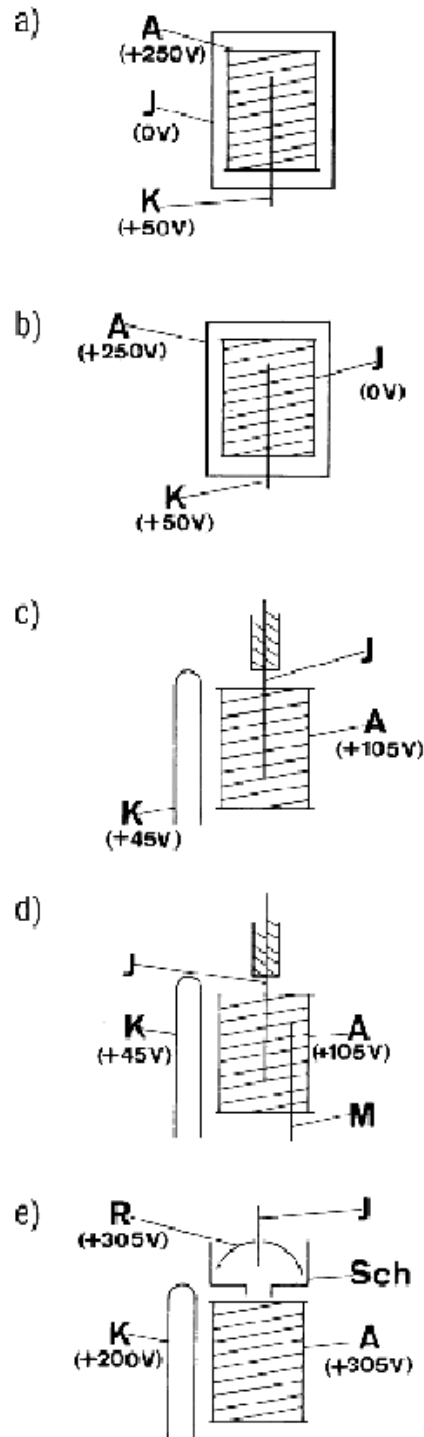


Figure 3: Electrode schematic of different hot-cathode ionization gauges

- (a) Conventional triode gauge system
- (b) Triode gauge system for pressures up to 1 mbar
- (c) Bayard-Alpert ionization gauge system
- (d) Bayard-Alpert ionization gauge system with modulator
- (e) Extraction gauge system

- J ion catcher
- Sch shield
- M modulator
- A anode
- K cathode
- R reflector

## Annex B

### B.1 Calibration system

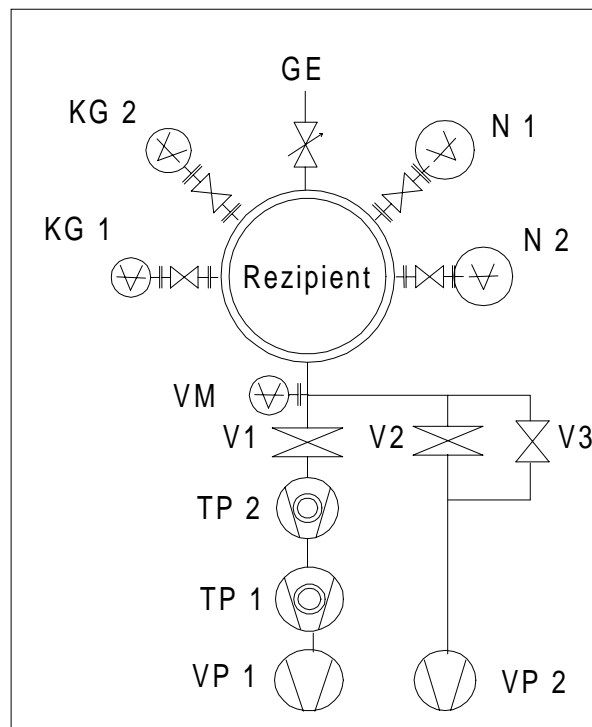


Figure 4: Example of calibration system recipient

Symbols:

GE	gas inlet
N 1,2...	standards
KG 1,2...	calibration items
VM	vacuum gauge for residual pressure indication, e.g. ionization 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
TP2	turbomolecular pump 2
TP1	turbomolecular pump 1 (to increase the compression for hydrogen)
VP1	backing pump for TP1
VP2	pump for pre-evacuation

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