INTERNATIONAL STANDARD

ISO 19685

First edition
2017-09

Vacuum technology — Vacuum gauges — Specifications, calibration and measurement uncertainties for Pirani gauges

Technique du vide — Manomètres à vide — Spécifications, étalonnage et incertitudes de mesure pour les jauges Pirani

Reference number
ISO 19685:2017(E)

Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 112, *Vacuum technology.*

Introduction

ISO 3567 and ISO 27893 are basic standards with no specific guidelines of a special type of vacuum gauge and are generally applicable. Detailed guidance for a specific gauge is intended to be given in separate technical specifications for the calibration of special types of gauges.

This document complements ISO 3567 and ISO 27893 when characterizing or calibrating Pirani gauges or using them as reference gauges.

Pirani gauges are widely used to measure pressures in the medium vacuum up to atmospheric pressure. The relevant parameters, calibration guidelines and uncertainties for the dissemination of the pressure scale and measurement of low and medium vacuum pressures by a Pirani gauge are described in this document.

Vacuum technology — Vacuum gauges — Specifications, calibration and measurement uncertainties for Pirani gauges

1 Scope

This document identifies parameters of Pirani gauges, their calibration procedure, and describes measurement uncertainties to be considered when operating these gauges.

This document applies to Pirani vacuum gauges operating over a pressure range of 0,01 Pa to 150 kPa.

This document complements ISO 3567 and ISO 27893 when calibrating Pirani gauges and using them as reference standards.

In addition, this document defines procedures to characterize Pirani gauges for response time and hysteresis.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3567:2011, *Vacuum gauges — Calibration by direct comparison with a reference gauge*

ISO 27893, *Vacuum technology — Vacuum gauges — Evaluation of the uncertainties of results of calibrations by direct comparison with a reference gauge*

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at http://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1 Definitions of components

3.1.1
thermal conductivity gauge
vacuum gauge in which the pressure is determined in relation to the transfer of thermal energy between the surfaces of two fixed elements maintained at different temperatures

Note 1 to entry: This gauge is based on the thermal conductivity of a gas being pressure dependent.

[SOURCE: ISO 3529-3:2014, 2.4.2.2, modified — Example has been deleted.]

3.1.2
Pirani gauge
thermal conductivity gauge in which the heated element is part of a Wheatstone bridge that supplies the energy to the element and by which the electrical resistance or the dissipated power of the element is being measured

Note 1 to entry: Some types of Pirani gauges have an extended upper limit of measurement range by using the heat convection inside the tube. This type is often called convection-enhanced Pirani gauge.

Note 2 to entry: See Annex A.

[SOURCE: ISO 3529-3:2014, 2.4.2.2.2, modified — Note 1 to entry has been replaced.]

3.2 Definitions of physical parameters

3.2.1
internal volume
volume of the gauge that is exposed to the vacuum system where it is attached to, counted from the connecting flange plane

3.2.2
thermal accommodation coefficient
ratio of the absolute temperature of the molecule after a hit with the heated element in a Pirani gauge and the absolute temperature of the heated element

3.2.3
long-term instability
quantity characterizing the typical change of relative measurement error $e = \frac{\Delta p}{p} = \frac{p_{\mathrm{UUC}} - p}{p}$ over time where the period needs to be specified

Note 1 to entry: Measurement errors to determine long-term instability of this gauge type are taken for nitrogen at a pressure of $p = (10 \pm 1)$ Pa (nitrogen) after a specified period. This quantity δ_t shall be a relative quantity and may be determined in two ways:

a) as the relative standard deviation of measurement error e_i obtained from at least three calibrations each being separated by the specified period

$$\delta_t = \sqrt{\frac{1}{n-1} \sum_{i=1}^{n} \left(e_i - \bar{e} \right)^2}$$

with n being the number of calibrations i, and

$$\bar{e} = \frac{1}{n} \sum_{i=1}^{n} e_i$$

b) as the mean of absolute (non-negative) changes of measurement error Δp_i between recalibrations separated by the specified period

$$\delta_t = \frac{\sum_{i=1}^{n-1} \left| e_{i+1} - e_i \right|}{n-1}$$

and n as described above

Note 2 to entry: The formula in a) is recommended when the measurement error does not show a significant drift but random variations, and the formula in b) when the measurement error shows a systematic and monotonic drift.

Note 3 to entry: If the output signal of the gauge is not pressure (e.g. voltage or current), this signal shall be converted to pressure based on the manufacturer's specification, before the measurement error is calculated.

Note 4 to entry: Long term instability can be determined by recalibrations with a more accurate gauge or a primary standard. This often requires a transport which itself can lead to an instability of the calibrated value. For this reason, it is not reasonable to assume a linear relationship of instability with time (e.g. δ_t for a period of 2 years is not 2 times δ_t for a period of 1 year).

Note 5 to entry: If not specified otherwise, it is recommended to determine δ_t over a period of 1 year. This is usually a reasonable compromise between costs and influence of transport on the one hand and a possible drift and lowest possible measurement uncertainty on the other hand.

EXAMPLE If δ_t was determined to 6,5 % according to the formula in b), where the period of recalibration was 1 year, the report will be "δ_t = 6,5 % for a period of one year".

3.2.4
response time
τ
time that elapses when the Pirani gauge is exposed to a sudden pressure change of nitrogen between 1 kPa to 100 Pa (up or down to be specified), until the Pirani shows 90 % of the pressure change difference

Note 1 to entry: See Figure 1.

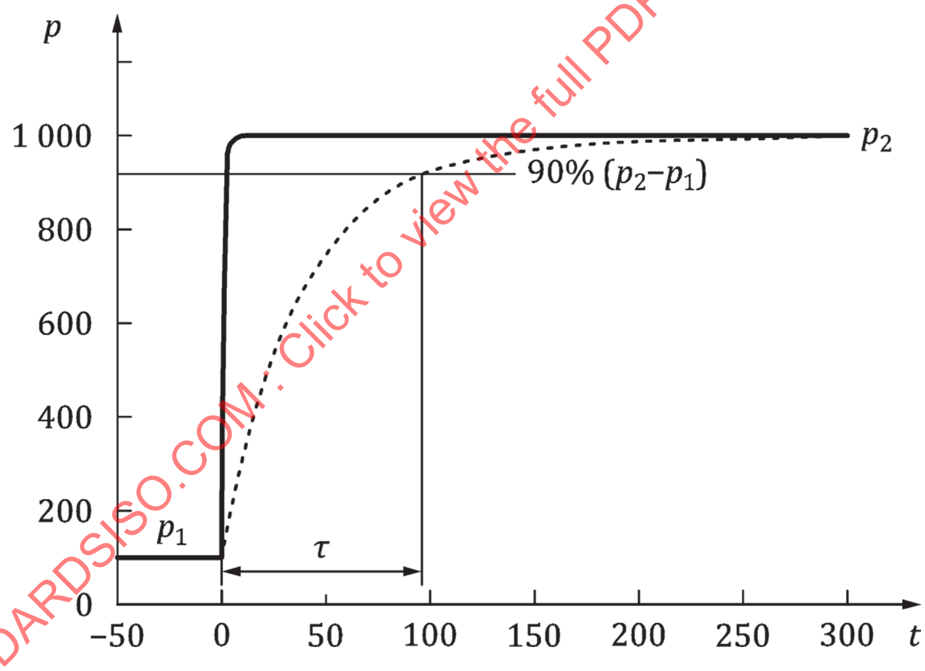

Figure 1 — Illustration to explain definition of response time τ

3.2.5
hysteresis
relative difference of indication of a Pirani gauge when exposed to the same pressure in a rising and decreasing sequence

4 Symbols and abbreviated terms

Symbol	Designation	Unit
e	Relative measurement error	1 or in %
p	Pressure, traceable to SI	Pa
Δp	Measurement error (measured pressure p_{UUC} minus reference pressure p)	Pa
p_{UUC}	Measured pressure by unit under calibration (UUC)	Pa
δ_t	Long-term instability as defined in 3.2.3	1 or in %
τ	Response time	ms
UUC	Unit under calibration	

5 Principle of Pirani gauge

The Pirani gauge is one type of a thermal conductivity vacuum gauge. In the Pirani gauge, gas molecules conduct heat away from a heated element to a surrounding wall that is usually at room temperature (see Figure A.1). In a certain pressure range, the heat taken away by the molecules is proportional to molecular density and hence pressure. The corresponding temperature change of the heated element, which is often a filament, causes a change in resistance. The resistance is part of a Wheatstone type bridge circuit that supplies the energy to the element and by which the electrical resistance or the dissipated power of the element is being measured. The bridge circuit may keep constant either the temperature of the heated element, or the heating power, or voltage or current. The linear measurement range is highest, when the temperature is kept constant. More detailed information is given in Reference [1].

The heat conductivity is strongly dependent on the gas species. It depends on the degrees of freedom of the molecule, its mass and the thermal accommodation coefficient of the molecule with the surface of the material of the heated element[2]. Therefore, also the signal measured by the Pirani gauge is strongly gas dependent. The thermal accommodation coefficient is significantly affected by impurities on the surface, which makes the Pirani gauges sensitive to contamination by vacuum processes. Coverage of the heated element caused by such processes also changes the lower measurement limit and, due to the change of filament diameter, the sensitivity.

At higher pressures, in the viscous flow regime, the heat conductivity through the gas becomes pressure independent. The lighter the gas molecule, the lower the pressure this occurs. This limits the useful measuring range of the Pirani gauge. Convection is still causing some heat dissipation from the heated element, but the sensitivity of the Pirani gauge is very small in this regime.

The lower measurement limit and resolution of the Pirani gauge is determined by the heat conductivity to the mechanical support structure of the heated element and radiation. For this reason, ambient temperature changes do affect the sensitivity of the gauge significantly below 10^{-1} Pa.

The wall temperature of the gauge head may also be influenced by the heated elements itself. At higher pressures > 1 kPa, more heating power for the heated element is needed to keep the temperature constant than at lower pressures and, as a consequence, the surrounding wall is heated up. This again reduces the heating power and may cause hysteresis.

NOTE In recent years, another type of thermal conductivity gauge was commercialized. The heated element (filament) is not operated at a constant temperature, but is cyclically heated up to a defined temperature threshold by an increasing voltage ramp[3]. The time to heat the element up to the threshold is pressure dependent. In terms of measurement uncertainties, this type of thermal conductivity gauge is similar as the Pirani gauge and this standard can be applied to these type of gauges as well.

6 Specifications for Pirani gauge

6.1 General

The following features and specifications are required, in order to enable users of the gauges to estimate the measurement uncertainty and/or to disseminate the pressure scale.

6.2 Measuring principle

It shall be specified how the heat dissipation in the gauge head is measured (continuously, pulsed, convection-enhanced).

6.3 Type of output

The type of output (analogue, digital) and the unit (or units) of indicated signal (e.g. voltage, current, pressure) shall be specified. If the indicated signal output is not given in pressure units, the conversion of the signal unit to pressure given in the manufacturer's manual shall be used.

6.4 Range of output and output display resolution

The range of output and the corresponding resolution for digital output shall be specified.

6.5 Measurement range and full scale

The measurement pressure range and full scale for nitrogen shall be specified. The measurement range generally depends on the measurement uncertainty. The measurement range for a given measurement uncertainty for nitrogen of a newly produced gauge shall be specified by the manufacturer. The measurement range is the range between minimum and maximum pressure where the reading of the gauge is within the defined measurement uncertainty limits. If the pressure range is not given in pascal, the value in pascal shall be given in addition.

6.6 Expected measurement uncertainty

The expected measurement uncertainty in the measurement range as given in 6.5 for the type of device as can be achieved by any individual device of this type shall be specified. It should be given as relative quantity of reading in %. It shall be specified how the gauge has to be adjusted to meet the measurement uncertainties. If an adjustment at a certain pressure has to be made, the gas species has to be specified.

NOTE The expected measurement uncertainty of type of device includes the measurement error. In the case of the Pirani gauge, this is mainly caused by the nonlinearity of the gauge.

The measurement uncertainty of an individual gauge is given by a calibration certificate (see 8.6). The measurement uncertainty of an individual gauge may be significantly smaller than the expected measurement uncertainty for the same type of device, since it does not need to include the measurement error.

6.7 Material exposed to gas

The material of the gauge head exposed to operating gas shall be specified.

6.8 Connecting flange

The type and size of the flange of the gauge head shall be specified.

6.9 Interface and pin-connections

The protocol of communication with a computer shall be specified; The connector type and the function of the pin connections (pin-out) shall be specified.

6.10 Warm-up period

The warm-up period for optimum performance in the measurement range (the indication is within the measurement uncertainty limits within the measurement range) shall be specified.

6.11 Compatibility between gauge tube and controller

At least one type of controller compatible with the gauge head shall be specified. If no controller is recommended, the technical data of the controller for proper operation of the gauge need to be given.

6.12 Normal operating (environment) conditions

Temperature of the gauge head and/or of the operating gas and humidity range for reliable measurement of pressure shall be specified at which the gauge can be operated.

6.13 Electrical power supply

Necessary voltage (AC or DC), frequency and power and, if applicable, their stabilities (e.g. maximum ripple noise) shall be specified.

6.14 Cable length

The cable length between the gauge head and controller shall be specified.

NOTE A longer cable can be sensitive to electro-magnetic interference.

6.15 Maximum bake-out temperature

The maximum temperatures shall be specified respectively for the gauge head and cables. If the control unit or indicating unit is directly attached to the gauge head (“integrated” or “active” gauge), but can be removed, this shall be stated and the maximum bake-out temperature for both the gauge head only and gauge head with control unit attached shall be given.

6.16 Dimensions of gauge head and controller

The dimensions of the gauge head, controller and, if applicable, the dimensions of cut-out for panel mounted controllers should be specified in outline drawings in SI units. It may be expressed as width, depth and height ($w \cdot d \cdot h$). Other units can be used in addition.

6.17 Mounting orientation

Mounting orientation of the gauge (vertical, horizontal, arbitrary) should be specified.

NOTE The sensitivity (calibration results) may depend on the orientation.

7 Additional (optional) specifications for Pirani gauge

7.1 Long-term instability

Long-term instability shall be expressed in relative units for a specified period (e.g. 1 month, 1 year). For such measurements, pure gases at stable or repeatable pressures shall be used and the gauge be operated under its normal conditions. Typical values may be given.

7.2 Relative sensitivity factor

The relative sensitivity factors may be given in a table or graph (if dependent on pressure) for various gas species.

7.3 Hysteresis

The hysteresis as defined in 8.7 may be specified.

7.4 Response time

The response time as defined in 8.8 may be specified.

7.5 Inspection record and calibration certificate

A document of an inspection record that shows proper function of the instrument (readings and true pressures at the limits of the measurement range for nitrogen) may be supplied with the gauge. If a calibration certificate is available, it shall contain information on how it is traceable to a national standard concerning vacuum.

7.6 Configuration

The configuration (active, passive) may be specified.

7.7 Weight

Weight of the gauge (e.g. sensor, controller, cable) should be specified.

7.8 Storage and transport condition

Conditions of storage and transportation should be specified to avoid damage and harm to the gauge, e.g. gas environment, cleanliness, temperature, relative humidity, vibration, shock, etc.

7.9 Photographs

For a clear outlook and details, a photograph or drawing of the upright gauge tube and the front and back panels (connector side) of the gauge controller is recommended.

7.10 Maximum power consumption

Maximum power consumption of the gauge head with one of the recommended controllers near atmospheric pressure should be specified. If the gauge head is stand alone ("active gauge"), the maximum power consumption for this should be given.

7.11 Set point of pressure

It should be specified, whether external control set points are available.

8 Calibration and other measurement procedures

8.1 General

If the calibration is performed by direct comparison with a reference gauge, ISO 3567 applies. In the following subclauses, details for Pirani gauges are given with reference to ISO 3567, if applicable.

If in rare cases the calibration is performed by a primary standard, the user shall apply the following procedures correspondingly.

8.2 Conditions for calibrations

It is recommended that the complete measurement chain—gauge head, cable and indicating unit—is calibrated. If this is not the case, the calibrating laboratory shall ensure that all necessary equipment used for recording the output (e.g. a digital voltmeter) is calibrated according to ISO/IEC 17025.

For example, if an active gauge requires a voltmeter to read the output, and the client does not provide the voltmeter, the calibration laboratory shall provide a calibrated voltmeter. In this case, the client needs to be aware that the calibration values are only valid if the client uses calibrated equipment at their site.

The calibration item shall be 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. This is to be ascertained by external inspections and functional tests.

The inspection shall include:

— visual inspection for contamination (surface coverage) and cleanliness inside of the head;

— visual inspection for damage (pointer, inscriptions, readability of indications, sealing surface);

— check whether documents necessary for calibration (technical data, operating instructions) are available.

It is recommended to require a declaration by the user that the gauge is not contaminated by toxic, corrosive, explosive, radioactive, microbiological or other harmful substances.

If the calibrating laboratory decides that a cleaning procedure needs to be performed before a calibration, the client needs to be informed or the device is returned. In many cases, however, the best option may be to replace the gauge head with a new unit, which is then calibrated.

Functional tests shall include:

— leak tightness of calibration item;

— operational function (e.g. off/on, adjustability of zero point);

— adjusting elements of controller in the defined positions.

The stability of the indication and the reproducibility in particular are important criteria for the status of the vacuum gauge. Residues of air humidity and other residues, e.g. of process substances shall be removed from the gauge head of the calibration item. This is achieved by evacuation, possibly assisted by a bake out.

8.3 Assembly and adjustments of calibration item

The gauge should be mounted in the orientation as recommended by the manufacturer or as agreed with the customer. It is recommended to mark the upwards direction during calibration on the gauge by a sticker.

For convection-enhanced Pirani gauges, often a horizontal orientation of the heated wire is required to work properly at higher pressures.

Before any adjustment and calibration, the gauge shall be in a stable temperature condition. After evacuation and switching on, a warm-up time according to the manufacturer's specifications should be considered. Unless otherwise specified by the manufacturer, a stabilization time of 30 min is recommended.

It is customary to make adjustments at full scale and zero of the gauge before a calibration.

Some clients may need the calibration data before any adjustment, e.g. because they are unable to make the same adjustments on site. According to ISO/IEC 17025, this is to be reviewed with the client before the adjustment is made.

The calibration gas species shall be recorded. All Pirani settings should be confirmed with the customer including the measuring channel, heating, output signal configuration.

8.4 Calibration

It is recommended to use as reference gauge one or several capacitance diaphragm gauges or vacuum gauges with similar or better accuracy covering the whole measurement range for calibration (see ISO 3567:2011, 6.6 and B.10).

Due to the poor linearity of the Pirani gauge, calibration by a primary standard is normally not worth the effort.

The reading of the Pirani gauges is strongly dependent on gas species. Therefore, it is recommended that the client requires a calibration with that gas species which is prevailing in the vacuum system when the gauge is in use, provided that this gas species can be handled by the calibration laboratory. If nothing else is specified, the calibration test gas is nitrogen (see ISO 3567:2011, 6.4 and B.2).

8.5 Measurement uncertainties during calibration

ISO 27893 applies. The individual uncertainty components depend on the specific design of the Pirani gauge.

The following list gives an overview of contributions related to the Pirani gauge to the uncertainty during calibration:

— resolution of signal output;

— short term instability (repeatability);

— zero drift;

— full-scale drift;

— change of ambient temperature.

8.6 Calibration certificate

ISO/IEC 17025 and ISO 3567 apply (see ISO 3567:2011, Clause 8).

In addition to the points given in ISO 3567 to be mentioned in the certificate, the adjustment procedure before the calibration needs to be described, in particular which gas species was used for the adjustment near 100 kPa.

If the output signal of the Pirani gauge is not pressure, the mathematical model given by the manufacturer by which the output signal (e.g. voltage, current) is converted to pressure should be repeated in the certificate.

In the special case of Pirani gauges, the measurement uncertainties in the calibration certificate can be rather small and can appear to a non-experienced user that it is a very accurate instrument. At later use, however, significant additional uncertainties much greater than those given in the certificate may apply. For this reason, it is strongly recommended to add the following sentence in the certificate:

"In the special case of Pirani gauges, the measurement uncertainties in the calibration certificate can be rather small and it may seem to a non-experienced user to be a very accurate instrument. On further use, however, significant, additional uncertainties may arise. Particularly large sources of uncertainty include: use of a different gas (even when corrected for known gas species), exposure to corrosive or non-inert gas species, use of interpolated values (not given in this certificate, i.e. non-linearity), use at different ambient temperature, long-term instability, contamination, and thermal relaxation at higher pressures."

NOTE Often, the Pirani inhibits a significant measuring error which depends on pressure. The uncertainty given in the certificate applies after prior correction for this error.

8.7 Measurement procedure for hysteresis

If hysteresis is to be determined, a reference gauge or standard with insignificant hysteresis effect needs to be used. Hysteresis measurement shall be done in the following manner:

a) pump to high vacuum of at least 1 mPa, take readings in increasing sequence at 1 Pa, 10 Pa, 100 Pa and 1 000 Pa for nitrogen;

b) expose gauge to 80 kPa, wait 5 min;

c) take readings in downwards direction at 1 000 Pa, 100 Pa, 10 Pa, 1 Pa;

d) pump to high vacuum of at least 1 mPa.

Both directions should be passed through as fast as possible, but a stable reading within the resolution limit shall be achieved for each target pressure. The mean of three cycles should be determined.

8.8 Measurement procedure of response time

If response time needs to be characterized, it shall be measured with nitrogen as test gas in the following manner.

Downward step change: After installation on a suitable vacuum system and warm-up time, expose the gauge to a pressure of 1 kPa. Reduce the pressure to 100 Pa by expansion into a suitable vacuum chamber within 10 ms or less by opening a fast valve.

Upwards step change: After installation on a suitable vacuum system and warm-up time, expose the gauge to a pressure of 100 Pa. Increase the pressure to 1 kPa by expansion from a suitable reservoir within 10 ms or less by opening a fast valve.

All the mentioned pressures are nominal and should be met within ±20 % of their nominal values.

NOTE 1 The pressure steps can be realized by means of accordingly sized and prefilled volumes with a fast opening valve in between. For a downwards step, an expansion from a small volume into a 9-times-larger, evacuated volume is possible. The same pressure step can also be realized by expansion into a 99-times-larger volume, but prefilled with 91 Pa. For an upwards step, the small volume is filled with 100 Pa and a 9-times-larger reservoir volume is filled with 1 100 Pa.

Suitable valves with sufficiently high conductance and fast opening times are needed for the expansion.

NOTE 2 If the electronics of the controller recording the gauge output are slow compared to the pressure step of 10 ms, the electronics will dominate the response time, and not the physical measurement principle.

9 Measurement uncertainties at use

Some measurement uncertainties may not be important during a calibration, but play a role at later use, e.g. when used as reference gauge.

The following list gives an overview of contributions to the uncertainty in addition to those listed in 8.5:

— long-term instability;

— nonlinearity;