NIST HANDBOOK 150-2H

**National
Voluntary
Laboratory
Accreditation
Program**

Calibration Laboratories

Technical Guide for Thermodynamic Measurements

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Preface

The Calibration Laboratories Accreditation Program was developed by the National Voluntary Laboratory Accreditation Program (NVLAP) at the National Institute of Standards and Technology (NIST) as a result of interest from private industry and at the request of the National Conference of Standards Laboratories (now the NCSL International). The goal of the program is to provide a means by which calibration laboratories can be assessed for competency. This voluntary program is not designed to serve as a means of imposing specific calibration procedures or minimum uncertainties on applicant laboratories; instead, the program allows for all scientifically valid calibration schemes and requires that laboratories derive and document their measurement uncertainties.

To accomplish this goal, NVLAP employs technical experts on a contract basis, to serve as assessors in each of the following eight fields of physical metrology calibration:

- electromagnetic dc/low frequency,
- electromagnetic rf/microwave frequency,
- time and frequency,
- ionizing radiation,
- optical radiation,
- dimensional,
- mechanical, and
- thermodynamics.

NIST Handbooks 150-2A through 150-2H are technical guides for the accreditation of calibration laboratories, with each handbook corresponding to one of the eight fields of physical metrology calibration. They are intended for information and use by:

- NVLAP technical experts in assessing laboratories,
- staff of accredited laboratories,
- those laboratories seeking accreditation,
- other laboratory accreditation systems,
- users of laboratory services, and
- others needing information on the requirements and guidelines for accreditation under the NVLAP Calibration Laboratories Accreditation Program.

NOTE   The Calibration Laboratories Accreditation Program has been expanded to cover chemical calibration for the providers of proficiency testing and certifiers of spectrophotometric NTRMs. (See NIST Handbooks 150-19 and 150-21.) Other NVLAP handbooks in the chemical calibration area are expected in the future.

The assessor uses NIST Handbook 150, *NVLAP Procedures and General Requirements*, and the appropriate guides (NIST Handbooks 150-2A through 150-2H) to validate that a laboratory is capable of performing calibrations within the laboratory's stated uncertainties. These technical guides and other relevant technical information support assessors in their assessments of laboratories. Along with inspecting the facilities, documentation, equipment, and personnel, the assessor can witness a calibration, have an item recalibrated, and/or examine the results of measurement assurance programs and round-robins to collect objective evidence.

NIST Handbooks 150-2A through 150-2H supplement NIST Handbook 150, which contains Title 15 of the U.S. Code of Federal Regulations (CFR) Part 285 plus all general NVLAP procedures, criteria, and policies. The criteria in NIST Handbook 150 originally encompassed the requirements of ISO/IEC Guide 25:1990 and

the relevant requirements of ISO 9002 (ANSI/ASQC Q92-1987). These handbook criteria have been updated to incorporate the requirements of ISO/IEC 17025:1999. The entire series of Handbooks 150-2A through 150-2H comprises information specific to the Calibration Laboratories Program and neither adds to nor detracts from requirements contained in NIST Handbook 150.

Any questions or comments on this handbook should be submitted to the National Voluntary Laboratory Accreditation Program, National Institute of Standards and Technology, 100 Bureau Drive, Stop 2140, Gaithersburg, MD 20899-2140; phone (301) 975-4016; fax (301) 926-2884; e-mail NVLAP@nist.gov.

Acknowledgments

NIST Handbook 150-2 was first available as a draft covering all eight fields of physical metrology calibration in one volume. It has been separated into eight handbooks to allow easier updating and electronic downloading from the NVLAP web site. The preparation of these documents has been a joint effort, with input from representatives of other government agencies, laboratories, and the private sector. Acknowledgment of their efforts is in order; however, the listing of individual names is impractical. The submissions by individuals and companies offering suggestions for improvement to this document were also very welcome, as were the contributions of those who attended the public workshops.

We thank all the NIST measurement divisions for their work in writing or contributing to the individual handbooks. Listed below are those from the NIST measurement divisions who deserve special thanks for input to Handbook 150-2H, *Technical Guide for Thermodynamic Measurements*:

- Dr. Peter H. Huang and Dr. James R. Whetstone (Humidity),
- Dr. Dean Ripple, Mr. Gregory Strouse and Dr. B. W. Mangum (Thermometry),
- Dr. Charles R. Tilford, Mr. Patrick Abbott, Dr. Ren-Fang Chang, and Dr. Archie Miiller (Leaks and Vacuum), and
- Dr. Albert Lee, Dr. Douglas Olson, Dr. Charles D. Ehrlich, Mr. Stephen W. Doty, Mr. Robert G. Driver, Mr. Bernard E. Welch, and Mr. Donald B. Ward (Pressure).

Additional thanks go to those who actively participated in the Technical Guide Workshop held November 1993 and to those who served as points of contact within fields of calibration. They include: Ms. Georgia L. Harris, Mr. Norman B. Belecki, Dr. Theodore D. Doiron, Mr. Robert M. Judish, Mr. Thomas C. Larason, Ms. Sally S. Bruce, and Dr. Donald B. Sullivan. A special thanks is owed to Mr. James L. Cigler for work in developing the content and format of this guide, and to Ms. Vanda White for her editorial expertise in making this a readable document.

Above all, we wish to thank Mr. Jon M. Crickenberger, the editor of the first three drafts of this document, for literally hundreds of hours of his work in creating this guide. It was he who tasked the contributors to produce the technical content, assembled the results of their efforts into a consistent format, and provided the general commentary. Without Jon's dedicated effort to this monumental task, this guide would never have been published.

NVLAP has edited the individual handbooks and made changes resulting from comments by individuals to earlier draft versions. This editing has been to a different extent for each parameter. Every effort was made to include all pertinent information relevant to an ISO/IEC 17025-derived technical guide.

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Summary

This guide presents the general technical requirements (i.e., on-site assessment and proficiency testing) of the laboratory accreditation program for calibration laboratories along with specific technical criteria and guidance applicable to thermodynamic measurements. These technical guidelines are presented to indicate how the NVLAP criteria may be applied.

Any calibration laboratory (including commercial, manufacturer, university, or federal, state, or local government laboratory) engaged in calibration in thermodynamic measurements listed in this handbook may apply for NVLAP accreditation. Accreditation will be granted to a laboratory that complies with the criteria for accreditation as defined in NIST Handbook 150. Accreditation does not guarantee laboratory performance – it is a finding of laboratory competence.

Fields of calibration covered: Specific calibration parameters and related stimulus and measurement devices in areas of thermodynamic measurement.

Scope of accreditation:
- Calibration parameter(s), range, and uncertainty level (for Best Measurement Capability).
- Types of measuring and test equipment
- Quality assurance system for measuring and test equipment

Period of accreditation: One year, renewable annually.

On-site assessment: Visit by an assessor(s) to determine compliance with the NVLAP criteria before initial accreditation, in the first renewal year, and every two years thereafter. Preassessment and monitoring visits are conducted as required. All calibration parameters or general areas of calibration within the specific scope of accreditation requested will be assessed.

Assessors: Selected from technical experts with experience in the appropriate areas of calibration and quality systems assessment.

Proficiency testing (measurement assurance): Each laboratory is required to demonstrate its capability to successfully perform calibrations as part of on-site assessment or by documented successful completion of an approved Measurement Assurance Program (MAP) or round-robin intercomparison. Proficiency testing may be required for initial accreditation, or where other evidence of measurement assurance is not evident, and may be conducted annually thereafter. Advance notice and instructions are given before proficiency testing is scheduled.

Fees: Payments are required as listed on the NVLAP fee schedule, including the initial application fee, administrative/technical support fee, on-site assessment fee, and proficiency testing fee.

1 General information

1.1 Purpose

The purpose of this handbook is to amplify the general requirements for accreditation by NVLAP of calibration laboratories in the area of thermodynamic measurements covered by the Calibration Laboratories Program. It complements and supplements the NVLAP programmatic procedures and general requirements found in NIST Handbook 150, *NVLAP Procedures and General Requirements*. The interpretive comments and additional guidelines contained in this handbook make the general NVLAP criteria specifically applicable to the Calibration Laboratories Program.

This handbook does not contain the general requirements for accreditation, which are listed in NIST Handbook 150, but rather provides guidelines for good calibration laboratory practices, which may be useful in achieving accreditation.

1.2 Organization of handbook

The handbook is organized in two sections. The first section provides additional explanations to the general procedures and requirements contained in NIST Handbook 150. The second section provides details and guidance very specific for thermodynamic calibration laboratories.

1.3 Description of Calibration Laboratories Accreditation Program

On May 18, 1992, as a result of the petition and public notice process, the Director of the National Institute of Standards and Technology published in the *Federal Register* a notice of intent to develop the Calibration Laboratories Accreditation Program under the procedures of the National Voluntary Laboratory Accreditation Program. On June 2, 1994, the procedures and general requirements under which NVLAP operates, Title 15, Part 285 of the U.S. Code of Federal Regulations (CFR), were revised to:

a) expand the procedures beyond testing laboratories to include accreditation of calibration laboratories,

b) update the procedures to ensure compatibility with generally accepted conformity assurance and conformity assessment concepts,

c) incorporate international changes, especially with relevant International Organization for Standardization/International Electrotechnical Commission (ISO/IEC) documents (e.g., ISO/IEC Guides 25 (now ISO/IEC 17025:1999), 38, 43, and 58, and the ISO 9000 series), and

d) facilitate and promote acceptance of the calibration and test results between countries to avoid barriers to trade.

Calibration laboratory accreditation is offered in eight fields of physical metrology calibration covering a wide variety of parameters and includes accreditation in multifunction measuring and test equipment calibrations. Specific requirements and criteria have been established for determining laboratory qualifications for accreditation following prescribed NVLAP procedures. The criteria address quality systems, staff, facilities and equipment, test and calibration methods and procedures, manuals, records, and calibration/certification reports.

On September 18, 1992, a public workshop was held at NIST Gaithersburg and attended by a mix of private sector and government personnel. The workshop reviewed a draft handbook, which included general requirements, as well as very specific technical requirements for dc voltage calibrations at all levels. As a result of the workshop, the draft handbook was revised to take the form of a Calibration Laboratories Program Handbook, which included the general requirements for laboratories (using ISO/IEC Guide 25 as a basis), and eight companion Technical Guides covering the specific requirements for each field of calibration offered for accreditation.

On May 18, 1993, a public workshop on the revised draft program handbook was held at NIST Boulder and attended by more than 60 industry and government personnel. Comments from this workshop, as well as responses to a survey/checklist mailing, were used to prepare the final draft of the handbook, now entitled *NVLAP Procedures and General Requirements* (NIST Handbook 150), published in March 1994. NIST Handbook 150 has since been revised to incorporate ISO/IEC 17025:1999.

A public workshop for the Calibration Laboratories Technical Guides was held at NIST Gaithersburg, on November 22 through 24, 1993. More than 60 industry and government personnel attended and provided comments on the draft version of the Technical Guide for each of eight fields of calibration. As a result, the eight Technical Guides were incorporated into a draft Handbook 150-2, *Calibration Laboratories Technical Guide*, covering the fields being offered for accreditation. [In 2000, Handbook 150-2 (draft) was divided into eight handbooks, one for each calibration area.]

The need for technical experts to serve as assessors was advertised, and the first group of assessors was selected and trained during a four-day session held from November 16 through 19, 1993, in Gaithersburg, using materials developed by NVLAP.

The Calibration Laboratories Accreditation Program officially began accepting applications when notification was given in the *Federal Register* dated May 11, 1994. Applications are accepted and processed following procedures found in NIST Handbook 150.

1.4 References

1.4.1 The following documents are referenced in this handbook.

a) NIST Handbook 150, *NVLAP Procedures and General Requirements*; available from:

> National Voluntary Laboratory Accreditation Program
> National Institute of Standards and Technology
> 100 Bureau Drive, Stop 2140
> Gaithersburg, MD 20899-2140
>
> Phone: (301) 975-4016
> Fax: (301) 926-2884
> E-mail: nvlap@nist.gov
> NVLAP Web site: http://www.nist.gov/nvlap

b) ISO/IEC/BIPM (BIPM is the Bureau International des Poids et Mesures, the International Bureau of Weights and Measures) *Guide to the Expression of Uncertainty in Measurement* (GUM), 1993.

c) ISO/IEC 17025: 1999: *General requirements for the competence of testing and calibration laboratories.*

d)   ISO/IEC Guide 43: 1997, *Proficiency testing by interlaboratory comparisons, Part 1 and Part 2.*

e)   ISO/IEC/BIPM *International Vocabulary of Basic and General Terms in Metrology* (VIM), 1993.

ISO documents b) through e) are available from:

Global Engineering Documents (paper copies)
Order phone: (800) 854-7179

American National Standards Institute (ANSI) (electronic copies)
Electronic Standards Store
ANSI web site: http://www.ansi.org

f)   Taylor, Barry N., and Chris E. Kuyatt, *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*, NIST Technical Note 1297, U.S. Government Printing Office, Washington DC, 1994. Available on-line at http://physics.nist.gov/Document/tn1297.pdf.

g)   NCSL International Recommended Practice RP-15: *Guide for Interlaboratory Comparisons*, 1999.

h)   ANSI/NCSL Z540-1-1994, *Calibration Laboratories and Measuring and Test Equipment—General Requirements.*

i)   ANSI/NCSL Z540-2-1997, *U.S. Guide to the Expression of Uncertainty in Measurement.*

j)   NCSL International Recommended Practice RP-7: *Laboratory Design*, 1993.

NCSL International documents are available from:

NCSL International
2995 Wilderness Place, Suite 107
Boulder, CO 80301-5404
Phone: (303) 440-3339
Fax: (303) 440-3384
E-mail: orders@ncsli.org
Web site: http://www.ncsli.org

k)   Ehrlich, C. D., and S. D. Rasberry, "Metrological Timelines in Traceability," *J. Res. Natl. Inst. Stand. Technol.* **103**, 93 (1998).

l)   Croarkin, M. C., Measurement Assurance Programs, Part II: Development and Implementation, *NBS Special Publication 676-II*, U.S. Government Printing Office, Washington, DC, 1985.

1.4.2   Additional references specific to thermodynamic measurements are listed in Sections 2.2 through 2.8.

1.5 Definitions

Definitions found in NIST Handbook 150 apply, but may be interpreted differently or stated differently, when necessary to amplify or clarify the meaning of specific words or phrases as they apply to specific technical criteria.

1.5.1   Best uncertainty or best measurement capability: Smallest uncertainty of measurement a laboratory can achieve within its scope of accreditation, when performing more or less routine calibrations of nearly ideal measurement standards intended to define, realize, conserve or reproduce a unit of that quantity or one or more of its values, or when performing more or less routine calibrations of nearly ideal measuring instruments designed for the measurement of that quantity.

1.5.2   Proficiency testing: Determination of laboratory performance by means of comparing and evaluating calibrations or tests on the same or similar items or materials by two or more laboratories in accordance with predetermined conditions. For the NVLAP Calibration Laboratories Accreditation Program, this entails using a transport standard as a measurement artifact, sending it to applicant laboratories to be measured, and then comparing the applicant's results to those of a reference laboratory on the same artifact.

1.5.3   Traceability: Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties. [VIM:1993, 6.10]

A single measurement intercomparison is sufficient to establish uncertainty relationships only over a limited time interval (see reference 1.4.1 k)); internal measurement assurance (see reference 1.4.1 l)), using control (check) standards, is required to fully demonstrate that uncertainties remain within stated levels over time. For the purposes of demonstrating traceability for NVLAP accreditation, a laboratory must demonstrate not only that there is an unbroken chain of comparisons to national standards, but also that this chain is supported by appropriate uncertainties, measurement assurance processes, continuous standard maintenance, proper calibration procedures, and proper handling of standards. In this way, traceability is related to these other areas of calibration.

1.6 NVLAP documentation

1.6.1   Accreditation documents

Laboratories granted NVLAP accreditation are provided with two documents: Scope of Accreditation and Certificate of Accreditation.

The Scope of Accreditation lists the "Best Uncertainty" that an accredited laboratory can provide for a given range or nominal value within a given parameter of measurement. This "Best Uncertainty" is a statement of the smallest uncertainty that a laboratory has been assessed as capable of providing for that particular range or nominal value. The actual reported value of uncertainty for any particular measurement service that the accredited laboratory provides under its scope may vary depending on such contributors as the statistics of the test and uncertainties associated with the device under test.

1.6.2   Fields of calibration/parameters selection list

The Calibration Laboratories program encompasses eight fields of physical metrology calibration, with multiple parameters under each field. Each field is covered by a separate handbook (NIST Handbooks 150-2A through 150-2H). (Fields of accreditation under Chemical Calibration are covered by separate handbooks.) Depending on the extent of its calibration capabilities, a laboratory may seek accreditation to all or only selected fields and parameters within the scope of the program. The fields of calibration and their related parameters are given on the Fields of Calibration and Parameters Selection List, which is provided to a laboratory seeking accreditation as part of the NVLAP application package for the program. Additional fields of calibration and/or parameters may be added to the Calibration Laboratories program upon request

of customer laboratories and/or if decided by NVLAP to be in the best interest of the Calibration Laboratories Program.

The laboratory is requested to indicate on the Fields of Calibration/Parameters Selection List the parameter(s) for which accreditation is desired, along with appropriate ranges and uncertainties. There is also provision for an applicant laboratory to request accreditation for parameters not currently listed on the Selection List, or for accreditation of the quality system employed for assuring Measurement and Test Equipment (M & TE) used in support of product certification. Request for accreditation of quality assurance systems for M & TE will be treated as a separate field of calibration for the purpose of setting appropriate fees. Once a laboratory meets all the requirements for accreditation for the Fields of Calibration/Parameters Selection List, this information will become the basis for the Scope of Accreditation document.

1.6.3   Checklists

Checklists enable assessors to document the assessment of the laboratory against the NVLAP requirements found in NIST Handbook 150. The NVLAP Calibration Laboratories Accreditation Program incorporates the NVLAP General Operations Checklist. The questions are applicable to evaluating a laboratory's ability to operate a calibration program, and address factors such as the laboratory's organization, management, and quality system in addition to its calibration competency.

The NVLAP General Operations Checklist is numbered to correspond to the requirements in NIST Handbook 150. Comment sheets are used by the assessor to explain deficiencies noted on the checklist. Additionally, the assessor may use the sheets to make comments on aspects of the laboratory's performance other than deficiencies.

1.7 Assessing and evaluating a laboratory

1.7.1   On-site assessment

1.7.1.1   The NVLAP lead assessor will schedule with the laboratory the date for on-site evaluation, and will request the quality manual and documented quality and calibration procedures in advance of the visit to reduce time spent at the laboratory; such materials will be returned by the assessor. NVLAP and the assessor will protect the confidentiality of the materials and information provided. The laboratory should be prepared to conduct routine calibrations, have equipment in good working order, and be ready for examination according to the guidance contained in this handbook, the requirements identified in NIST Handbook 150, and the laboratory's quality manual. The assessor will need time and work space to complete assessment documentation while at the laboratory, and will discuss these needs at the opening meeting of the on-site assessment.

1.7.1.2   NVLAP technical assessors are provided with the NVLAP General Operations Checklist to help ensure the completeness, objectivity, and uniformity of the on-site assessment.

1.7.1.3   When accreditation has been requested for a considerable number of fields of calibration and parameters, the assessment may range from observing calibrations in progress, requiring repeat measurements on completed calibrations, to listening to laboratory staff describe the calibration process. The depth into which the assessor performs the assessment depends on the number of fields of calibration and associated parameters for which accreditation is requested and the time required to perform a given calibration.

1.7.1.4 The assessor, or the assessment team, does the following during a typical on-site assessment:

a)   Conducts an entry briefing with the laboratory manager to explain the purpose of the on-site visit and to discuss the schedule for the day(s). At the discretion of the laboratory manager, other staff may attend the briefing.

b)   Reviews quality system manual, equipment and maintenance records, record-keeping procedures, laboratory calibration reports, and personnel competency records. At least one laboratory staff member must be available to answer questions; however, the assessor may wish to review the documents alone. The assessor(s) does not usually ask to take any laboratory documents with him/her, and previously supplied documents will be returned.

c)   Physically examines equipment and facilities, observes the demonstration of selected procedures by appropriate personnel assigned to perform calibrations, and interviews the personnel. The demonstrations must include preparation for calibration of devices, and the setup and use of measuring and test equipment, standards and systems.

d)   Holds an exit briefing with the laboratory manager and staff to discuss the assessment findings. Deficiencies are discussed and resolutions may be mutually agreed upon. Items that must be addressed before accreditation can be granted are emphasized, and outstanding deficiencies require response to NVLAP within 30 days. Items that have been corrected during the on-site and any recommendations are specially noted.

e)   Completes an On-site Assessment Report, as part of the exit briefing, summarizing the findings. The assessor(s) attaches copies of the completed checklists to this report during the exit briefing. The report is signed by the lead assessor and the laboratory's Authorized Representative to acknowledge the discussion. This signature does not necessarily indicate agreement; challenge(s) may be made through NVLAP. A copy is given to the representative for retention. All observations made by the NVLAP assessor are held in the strictest confidence.

1.7.2   Proficiency testing

1.7.2.1   Background

Once the quality system review and on-site assessment steps have been satisfactorily completed, it is necessary to gather another set of data points to aid in deciding whether or not the applicant laboratory is competent to perform calibrations within the fields of interest to the uncertainties claimed. In the eight fields of calibration covered by Handbooks 150-2A through 150-2H, there are approximately 85 parameters of interest. Under most parameters there are several subsets, referred to as ranges. For example, in the pressure and leak field, parameters can range from 10^{-4} Pa to 102 MPa in value. In view of the many possible ranges, proficiency testing could be conducted in scores of areas. NVLAP reserves the right to test by sampling in any area; hence, applicant laboratories must be prepared, with reasonable notice, to demonstrate proficiency in any of a number of parameters.

1.7.2.2   Proficiency testing vs. measurement assurance

There is an important difference between proficiency testing and measurement assurance. The objective of proficiency testing is to determine through a measurement process that the laboratory's measurement results compare favorably with the measurement results of the audit laboratory (NIST or one designated by NVLAP), taking into account the relative uncertainties of the measurements assigned by both the applicant and audit laboratories (see 1.7.2.5 below). The objective of proficiency testing is not to determine and certify

the total uncertainty of the applicant laboratory, as is done in a Measurement Assurance Program (MAP) with NIST, but to verify (through the assessment process) that the uncertainty claimed by the applicant laboratory is reasonable, and then use the claimed uncertainty to test that the measurement result obtained through the proficiency test is acceptable.

It is neither the intention nor the mission of NVLAP to conduct MAPs or to otherwise provide traceability for laboratories. Laboratories obtain these services from the NIST measurement divisions. NVLAP assesses the implementation, application, and documentation of MAPs by laboratories. NVLAP accreditation encourages the use of MAPs by the calibration laboratory community, and MAP results produce objective evidence that NVLAP assessors look for as part of the assessment process.

1.7.2.3 Requirements

NVLAP's proficiency testing program uses a sampling approach. All applicant laboratories are required to complete an annual proficiency test in one parameter under each field of calibration for which it has applied to be accredited. For the purposes of the NVLAP Calibration Laboratories Accreditation Program, the results of the proficiency test are considered as objective evidence, along with the on-site visit, of a laboratory's ability to perform competent calibrations. Proficiency testing is conducted annually using different parameters in each field; however, those laboratories accredited in only one parameter within a field are retested in the same parameter.

1.7.2.4 Uncertainty determination

The applicant laboratory is required to perform a measurement or series of measurements on an artifact using the same calibration method, apparatus, and personnel that is typical of that used to calibrate its customers' equipment. The laboratory must be able to identify and quantify all sources of uncertainty that affect the measurement. The laboratory should attach an overall uncertainty to the measurement by combining all uncertainty contributions, in their type A and type B components, in the root-sum-squared method as described in the *Guide to the Expression of Uncertainty in Measurement* (see reference 1.4.1 b)). The confidence limit used should be $k = 2$, which is equivalent to a 95 % confidence probability. It is these 95 % confidence level uncertainties that are used in section 1.7.2.5 and throughout the handbook.

Although the laboratory's accreditation scope is based on its "Best Measurement Capability," the laboratory should keep customer expectations in mind when providing a measurement uncertainty to its customers. Uncertainties for calibrated measuring instruments occur from many sources – the device itself, the calibration facility, the conditions of the calibration, the procedures used by the calibration staff, and the calibration test procedure used. Metrologically competent instrument owners will wish to have their device calibrated so that the resulting data describes the measurement performance they can expect to see after the calibration. This means that the calibration conditions and procedures need to quantify the short-term stability (repeatability, see NIST TN 1297, reference 1.4.1 f)) and long-term stability (hysteresis effects, "turn-off-turn-on" reproducibility, possible day-to-day effects), if these are thought to be worth quantifying, for the instrument over the ranges of measurement conditions. If the instrument owner expects to use the device after it is removed and reinstalled, and if this feature is suspected of adding an additional source of uncertainty, then the "take-out-put-back" reproducibility should be appropriately quantified through the calibration process. This "take-out-put-back" reproducibility would be pertinent in the case of transfer standards or artifacts that go from laboratory to laboratory for proficiency testing or laboratory intercomparisons, for example.

This document is intended to guide the laboratory and the assessor as to whether the laboratory is capable of performing calibrations within the laboratory's stated uncertainties, not to instruct the calibration laboratory on how to apply its standards.

1.7.2.5 Pass/fail criteria

The performance of the proficiency test is judged by calculating the error of the measurement, normalized with respect to the uncertainty of the measurement, using the following equation:

$$E_{normal} = \left| (Value_{lab} - Value_{ref}) / (Uncertainty_{ref}^2 + Uncertainty_{lab}^2)^{1/2} \right|$$

where

E_{normal} = normalized error of the applicant laboratory,
$Value_{lab}$ = the value as measured by the applicant laboratory,
$Value_{ref}$ = the value as measured by the reference laboratory,
$Uncertainty_{ref}$ = the uncertainty of the reference laboratory, and
$Uncertainty_{lab}$ = the uncertainty of the applicant laboratory.

To pass the proficiency test, the applicant laboratory must have a value for E_{normal} less than 1 (i.e., $E_{normal} < 1$). The results may be plotted graphically, with lines representing the limits of uncertainty of the measurements. The anonymity of each applicant laboratory will always be preserved.

1.7.2.6 Scheduling and handling

Proficiency testing is scheduled by NVLAP-designated reference laboratories. These sites are NIST laboratories or NVLAP-accredited laboratories that have been found to have the ability to perform the required proficiency tests to an uncertainty level appropriate for the laboratories they evaluate. The proficiency test is scheduled independently and not to correspond with the on-site visit. Applicant laboratories are notified in advance as to the approximate arrival time of the measurement artifact. Instructions for performing the test, reporting the results, communicating with the reference laboratory, and shipping are included along with the artifact as part of the proficiency test package. Applicant laboratories are instructed to perform all required measurements within a reasonable time and are told where to ship the artifacts once the testing has been completed.

1.7.2.7 Notification of results

NVLAP notifies each laboratory of its own results in a proficiency test. If a laboratory has received its on-site assessment prior to the completion of the proficiency test, the status of that laboratory's accreditation is contingent upon successful completion of proficiency testing. The laboratory's accreditation status may be changed to reflect a partial accreditation, or may be completely suspended pending demonstration of the laboratory's ability to successfully complete the proficiency test at a later date.

1.7.3 Traceability

1.7.3.1 Establishing traceability

Laboratories must establish an unbroken chain of comparisons leading to the appropriate international or national standard, such that the uncertainties of the comparisons support the level of uncertainty that the laboratory gives to its customers. Generally speaking, the uncertainties of the comparisons increase as they

move from a higher (international or national level) to a lower level standard. This uncertainty chain is the evidence of traceability and must be documented accordingly. Traceability does not simply mean having standards calibrated at the national laboratory, but must consider how a measurement, with its corresponding uncertainty, is transferred from the national level to the calibration laboratory's customers.

1.7.3.2 Considerations in determining traceability

Without some type of measurement assurance process, one cannot be reasonably certain that the comparisons have been transferred properly to the laboratory's customers. The measurement process itself must be verified to be in control over time. Therefore, traceability is not a static concept that, once established, may be ignored; it is dynamic. Process control exercised in each calibration provides the assurance that a valid transfer of the international or national standard has taken place. This assurance may be accomplished through the use of tools such as check standards and control charts. Also, the laboratory's primary standards must be maintained in such a way as to verify their integrity. Examples of this may be having more than one primary standard to use for intercomparisons, monitoring the primary standard with a check or working standard (looking for changes), and verifying a primary standard on a well-characterized measurement/calibration system. Using scientifically sound measurement procedures to transfer the primary standard value to the working level and the customer's item is essential to establishing traceability. If the procedure itself yields the wrong result, there is no way the laboratory can perform a calibration traceable to the international or national standard. Handling the laboratory's standards affects the measurement process, and therefore the ability to transfer the standard's value to the customer. Examples of handling problems are dirty or improperly cleaned standards, maintaining standards in an improper environment, not maintaining custody and security, and improper handling of standards during the measurement process.

1.7.3.3 Relationship to existing standards

The above discussion illustrates how traceability is dependent on many aspects of the measurement process and therefore must be considered in all phases of calibration. It is not just coincidental that the factors addressed above are main topics of concern in ISO/IEC 17025:1999.

1.7.4 Uncertainty

NVLAP recognizes the methodology for determining uncertainty as described in the *Guide to the Expression of Uncertainty in Measurement*, published by ISO. To be NVLAP-accredited, a laboratory must document the derivation of the uncertainties that it reports to its customers. These uncertainties will appear on the scope issued to each accredited laboratory to an accuracy appropriate to the standards, procedures, and measuring devices used.

2 Criteria for accreditation

2.1 Introduction

2.1.1 Applicant laboratories are assessed using the requirements in NIST Handbook 150, *NVLAP Procedures and General Requirements*. This guide, NIST Handbook 150-2H, was developed from a NIST measurement laboratory perspective and provides examples and guidelines, not requirements, to assessors and interested calibration laboratories, on good laboratory practices and recommended standards. Therefore, the guide language reflects this philosophy through the use of "shoulds" instead of "shalls" (along with other less prescriptive language) when describing criteria. The requirements presented here are not absolute since specific requirements depend on the measurement uncertainty for which an applicant laboratory wishes to be accredited. This is a business decision for each laboratory and beyond the scope of NVLAP. Simply stated, to be accredited, an applicant laboratory must have a quality system and be able to prove (and document) that it is capable of doing what it says it does (i.e., correctly calibrate to a stated uncertainty) within the framework of NIST Handbook 150. Accreditation will be granted, and therefore may be referenced in calibration reports, etc., only for those specific parameters, ranges and uncertainties using calibration methods and procedures for which a laboratory has been evaluated. Calibrations performed by a laboratory using methods and procedures not considered appropriate for the level of measurements being made, and which have not been evaluated by the accreditation process, are outside the scope of accreditation and may not be referenced as "accredited" calibrations on calibration reports, etc.

2.1.2 The following sections 2.2 onward detail specific calibration guidelines for thermodynamic measurements. This guide is dynamic in that new parameters may be added and existing criteria updated and improved.

2.2 Hygrometer calibrations

2.2.1 Scope

2.2.1.1 This section sets out the specific technical criteria in accordance with which a laboratory should demonstrate that it operates, if it is to be recognized as competent to carry out hygrometer calibrations.

2.2.1.2 This section may also be used as a guide by hygrometer calibration laboratories in the development and implementation of their quality systems.

2.2.2 References

a) ASHRAE STANDARD: Spc.41.6-1994R, *Standard Method for Measurement of Moist Air Properties*.

b) ASTM STANDARD: D4230-83(1996), *Standard Test Method of Measuring Humidity with Cooled-Surface Condensation (Dew-Point) Hygrometer*.

2.2.3 Quality System

When the calibration standard used by the laboratory is either a two-pressure humidity generator or a two-temperature humidity generator, the three action items described below are required:

a)   **Temperature standard for the saturator and the test chamber**

When the temperature standard used by the laboratory for measurements of temperature in the saturator and the test chamber is a standard platinum resistance thermometer (SPRT), or a thermistor thermometer, the calibration of the thermometer should be checked according to good laboratory practice (e.g., every 6 months).

b)   **Pressure standard for the saturator and the test chamber**

When the pressure standard used by the laboratory for measurements of pressure in the saturator and the test chamber is a pressure gage, a pressure transducer, a mercurial barometer, or a manometer, the calibration of the pressure measuring device should be checked according to good laboratory practice (e.g., every 6 months).

c)   **Electrical resistance standard for the dew-point hygrometer**

When an electrical resistance measuring device is used by the laboratory for measurements of 4-wire resistance associated with a dew-point hygrometer, its calibration should be checked by standard resistors at an interval determined by the observed stability of the device, but not to exceed one year.

2.2.4   Personnel

One of the persons working in the laboratory should understand measurement uncertainty and have a background in mathematics and general physics, as well as having a working knowledge of the literature pertinent to measurement of moisture concentration in gases.

2.2.5   Accommodation and environmental conditions

2.2.5.1   When a bath fluid such as N-propyl alcohol is used for controlling temperature of the humidity generator system, the laboratory should have adequate ventilation.

2.2.5.2   For calibrations of dew-point hygrometers, the temperature of the laboratory should be controlled at a higher value than the dew-points to be measured to avoid moisture condensation in connection tubing. Otherwise, connection tubing should be heated to an appropriate temperature.

2.2.5.3   Vibrations in the laboratory should be reduced to levels where they do not affect the measurements.

2.2.6   Equipment

2.2.6.1   The laboratory should have the humidity generation system needed to produce a stable water vapor concentration. These may be either active or passive methods.

2.2.6.2   The laboratory should have equipment for making temperature measurements of sufficient accuracy to achieve the desired moisture uncertainty.

2.2.6.3   The laboratory should have equipment for making pressure measurements of sufficient accuracy to achieve the desired moisture uncertainty.

2.2.6.4   The laboratory should have equipment for making sufficiently accurate gas flow measurements.

2.2.6.5   The laboratory should have equipment for making sufficiently accurate voltage, current or resistance measurements.

2.2.6.6   The water used in the saturator of the humidity generator or similar devices should be distilled or high purity water with a resistivity greater than 0.5 MΩ cm.

2.2.7   Measurement traceability

2.2.7.1   The temperature standard used by the laboratory should be traceable to temperature reference standards developed and maintained by a national measurement institute (NMI) such as NIST.

2.2.7.2   The pressure standard used by the laboratory should be traceable to pressure reference standards (as opposed to traceable to force and dimension) developed and maintained by a national measurement institute (NMI) such as NIST.

2.2.7.3   Dew-point hygrometer or psychrometer as the reference standard

When the reference standard used by the laboratory is a dew-point hygrometer or a psychrometer, one of the two action items described below is recommended.

a)   The hygrometer or the psychrometer should have been calibrated by an NMI.

b)   The hygrometer or the psychrometer may have been evaluated by the supplier, if the supplier documented in detail the preparation and calibration, showing direct traceability to an NMI, or to a laboratory accredited by an International Laboratory Accreditation Cooperation (ILAC) signatory.

2.2.8   Test and calibration methods and method validation

2.2.8.1   All computer programs used in data logging and analysis should be documented.

2.2.8.2   When calibrations are performed for chilled mirror type of dew-point hygrometers, the four action items described below are recommended:

a)   The mirror surface should be cleaned using pure ethyl or methyl alcohol before calibration.

b)   The mirror chamber should be maintained at a temperature higher than that of a dew-point to be measured.

c)   The saturator of the humidity generator system should be maintained at a stable temperature and pressure for a sufficient period of time before measurement.

d)   The dew-point measurement should not be obtained until adequate water vapor pressure equilibrium is reached.

2.2.8.3   When calibrations are performed for dew-point hygrometers by comparison against a reference standard hygrometer, all the hygrometers should be connected in parallel with respect to the sample gas inlet.

2.2.8.4   When calibrations are performed for relative humidity sensors, the three action items described below are required:

a) The sensors should be installed in the test chamber of the humidity generator.

b) The test chamber of the humidity generation system should be maintained at a stable temperature and pressure for a sufficient period of time before measurement such that the temperature and pressure are satisfactorily stated for the uncertainties quoted.

c) The relative humidity measurement should not be obtained until adequate thermal and water vapor pressure equilibrium conditions are reached.

2.2.8.5 When calibrations are performed for psychrometers, the four action items described below are required.

a) The fluid stored in the reservoir of the sensor head should be distilled or high purity water.

b) The sensor head should be installed in the test chamber of the humidity generator.

c) The test chamber of the humidity generator system should be maintained at a stable temperature and pressure for a sufficient period of time before measurement.

d) The volume flow rate of the sample gas passing through the sensor head should be approximately 140 liters per minute.

2.2.9   Assuring the quality of test and calibration results

For the purposes of proficiency testing of laboratories seeking or maintaining accreditation, the laboratory should calibrate a hygrometer over the accreditation range of dew/frost-points and/or relative humidities, and at calibration intervals of 10 °C and/or 10 % relative humidity at a given dry-bulb temperature. Proficiency testing may consist of round-robin testing among other accredited laboratories, or may consist of submission of a calibrated hygrometer and its calibration to NIST.

2.3 Thermometer calibrations

2.3.1   Scope

2.3.1.1   This section contains the specific technical criteria in accordance with which a laboratory should demonstrate that it operates, if it is to be recognized as competent to carry out thermometer calibrations.

2.3.1.2   This section may also be used as a guide by thermometer calibration laboratories in the development and implementation of their quality systems.

2.3.1.3   For Accuracy Class I (see section 2.3.3), additional requirements beyond the scope of this handbook are necessary. Further information on these requirements may be obtained from:

Dr. Dean Ripple
NIST, 100 Bureau Dr., Stop 8363
Gaithersburg, MD 20899-8363
301-975-4801

2.3.2   References

a) ASTM Annual Book of Standards, Volume 14.03, *Standards Relating to Temperature Measurement,* ASTM, Philadelphia, PA.

b) Burns, G.W., Scroger, M.G., NIST SP 250-35, *The Calibration of Thermocouples and Thermocouple Materials,* April 1989.

c) Wise, J.A., NIST SP 250-23, *Liquid-In-Glass Thermometer Calibration Service,* Sept. 1988.

d) Wise, J.A., Soulen, R.J., NBS Monograph 174, *Thermometer Calibration, A Model for State Calibration Laboratories* (Appendix A: NBS Monograph 150, *Liquid-In-Glass Thermometry*), Jan. 1986.

e) Mangum, B. W., Furukawa, G. T., NIST TN1265, *Guidelines for Realizing the International Temperature Scale of 1990 (ITS-90),* August 1990.

f) BIPM – *Supplementary Information for the International Temperature Scale of 1990,* Bureau International des Poids et Mesures, Pavillon de Breteuil, F-92310 Sevres, France, 1990.

g) BIPM – *Techniques for Approximating the ITS-90,* Bureau International Des Poids et Mesures, Pavillon de Breteuil, F-92310 Sevres, France, 1990.

h) Strouse, G. F. and Tew, W. L., NISTIR 5319, *Assessment of Uncertainties of Calibration of Resistance Thermometers at the National Institute of Standards and Technology,* 1994.

i) Meyer, C. W., Strouse, G. F., Tew, W. L., NISTIR 6138, *A Revised Assessment of Calibration Uncertainties for Capsule Type Standard Platinum and Rhodium-Iron Resistance Thermometers,* 1998.

j) Mangum, B. W., NIST Technical Note 1411, *Reproducibility of the Temperature of the Ice Point in Routine Measurements,* 1995.

k) Wise, J.A., NISTIR 5341, *Assessment of Uncertainties of Liquid-in-Glass Thermometer Calibrations at the National Institute of Standards and Technology,* Jan. 1994.

l) Ripple, D., Burnes, G.W., Scroger, M.G., NISTIR 5340, *Assessment of Uncertainties of Thermocouple Calibrations at NIST,* 1994.

2.3.3   Definitions

Accuracy class: Level of performance associated with the level of total expanded uncertainty ($k=2$), should conform to the following table:

Accuracy Class	Total Expanded Uncertainty ($k=2$)	
I	$\leq \pm$ 0.005 °C	
II	$> \pm$ 0.005 °C	to $\leq \pm$ 0.05 °C
III	$> \pm$ 0.05 °C	to $\leq \pm$ 0.20 °C
IV	$> \pm$ 0.20 °C	to $\leq \pm$ 1.0 °C
V	$> \pm$ 1.0 °C	to $\leq \pm$ 5.0 °C

NOTE   The uncertainty of thermometers calibrated by the laboratory will vary depending upon the temperature range of application, even for the same thermometer. Thus, a laboratory may perform calibrations of Accuracy Class II in some cases and Accuracy Class III or IV in other cases because of the temperature ranges involved. Also, the accuracy class assigned is dependent on the types of thermometers calibrated.

2.3.4 Assuring the quality of test and calibration results

2.3.4.1   Fixed-point cell as the reference standard

When the reference standard used by the laboratory is a fixed-point cell, the four action items described below are required, at the minimum, as indicated by the application table that follows their description.

a)   Records of complete phase equilibrium plateaus obtained for each cell (except for the triple point of water) upon receipt and once per year thereafter should be maintained.

b)   A check thermometer should be used for each fixed point, and control charts maintained.

c)   The triple point of water or ice point should be measured after every check thermometer measurement at another fixed point temperature.

d)   Any thermometer used in a fixed-point cell should adequately track the hydrostatic head correction over the bottommost 3 cm.

Item	Accuracy class to which item applies				
	I	II	III	IV	V
a)	x	x	x	x	
b)	x	x	x		
c)	x	x			
d)	x	x			

2.3.4.2 SPRT or RIRT as the reference standard

When the reference standard used by the laboratory is a standard platinum resistance thermometer (SPRT) or a rhodium-iron resistance thermometer (RIRT), the two action items described below are required as indicated by the application table that follows them.

a)   There should be documentation (i.e., control charts) to show that the resistance of the instrument at the triple point of water or ice point has not changed since its last calibration by more than the equivalent shown in the table below. If, since the last calibration, the resistance of the thermometer has changed at the triple point of water or ice point by the equivalent shown in the table below, a new calibration should be performed.

b)   If a digital temperature indicator is used to determine temperature, the calibration of the measured thermometer and the indicator calibration should be checked periodically at the triple point of water or ice point. If, since the last calibration, the readout of the indicator from measuring the thermometer at the triple point of water or ice point has changed by the equivalent shown in the table below, a new calibration should be performed.

Item	Accuracy class				
	I	II	III	IV	V
a)	0.002 °C	0.003 °C	0.005 °C	0.05 °C	0.1 °C*
b)	0.002 °C	0.003 °C	0.005 °C	0.05 °C	0.1 °C*

* at the ice point

2.3.4.3 IPRT as the reference standard

When the reference standard used by the laboratory is an industrial platinum resistance thermometer (IPRT), the two action items described below are recommended as indicated by the application table that follows them.

a)   There should be documentation (i.e., control charts) to show that the resistance of the instrument at the triple point of water or ice point has not changed since its last calibration by more than the equivalent shown in the table below. If, since the last calibration, the resistance of the thermometer has changed at the triple point of water or ice point by the equivalent shown in the table below, a new calibration should be performed.

b)   If a DVM, DMM, or digital temperature indicator is used to determine temperature, the calibration of the measured thermometer and the indicator calibration should be checked periodically at the triple point of water or ice point. If, since the last calibration, the readout of the indicator from measuring the thermometer at the triple point of water or ice point has changed by the equivalent shown in the table below, a new calibration should be performed.

Item	Accuracy class				
	I	II	III	IV	V
a)	0.002 ˚C	0.003 ˚C	0.005 ˚C*	0.05 ˚C*	0.1 ˚C*
b)	0.002 ˚C	0.003 ˚C	0.005 ˚C*	0.05 ˚C*	0.1 ˚C*

* at the ice point

2.3.4.4 Thermistor thermometer as the reference standard

When the reference standard used by the laboratory is a thermistor thermometer, the two action items described below are recommended as indicated by the application table that follows them.

a)   There should be documentation (i.e., control charts) to show that the resistance of the instrument at the triple point of water or ice point has not changed since its last calibration by more than the equivalent shown in the table below. If, since the last calibration, the resistance of the thermometer has changed at the triple point of water or ice point by the equivalent shown in the table below, a new calibration should be performed.

b)   If a DVM, DMM, or digital temperature indicator is used to determine temperature, the calibration of the measured thermometer and the indicator calibration should be checked periodically at the triple point of water or ice point. If, since the last calibration, the readout of the indicator from measuring the thermometer at the triple point of water or ice point has changed by the equivalent shown in the table below, a new calibration should be performed.

Item	Accuracy class				
	I	II	III	IV	V
a)	0.002 °C	0.003 °C	0.005 °C*	0.02 °C*	0.1 °C*
b)	0.002 °C	0.003 °C	0.005 °C*	0.02 °C*	0.1 °C*

* at the ice point

2.3.4.5 Thermocouple as the reference standard

a)   When the reference standard used by the laboratory is a thermocouple, special techniques must be used to ensure the reliability of the thermocouple as a reference standard. Unlike other thermometer types, recalibration of a reference standard thermocouple in a thermal environment different than the environment of use may give highly misleading results. This occurs because thermoelectric variations from contamination, material loss, or mechanical cold-working will result in thermoelectric inhomogeneity of the thermocouple, and the response of the thermocouple will depend on the details of the thermal profile along the length of the thermocouple.

b)   Examples of acceptable tests of the reliability of thermocouple reference standards include:

1) Periodic measurement of inhomogeneity of the thermocouple, at a temperature sufficiently low or a duration sufficiently short that the inhomogeneity test does not itself change the thermocouple properties.

2) In situ calibration of the reference standard thermocouple. For example, when a new TC is begun in service as a standard, it may first be used to check the calibration of the previous standard.

3) Calibration of test thermometers or check thermometers using standards that are both new and used.

c)   Control charts of tests similar to the ones above should be maintained. The results of these tests will dictate the lifetime of the reference standards. Once the acceptable limit of drift has been exceeded, recalibration is not recommended. A new reference thermocouple should be used.

d)   Measurement of thermocouples at fixed-points near ambient (triple point of water (TPW), Ga melting point) are poor indicators of thermocouple drift at significantly higher temperatures.

2.3.4.6 Liquid-in-glass thermometer as the reference standard

Only total-immersion, mercury-in-glass thermometers should be used as reference standard thermometers. The stability of these thermometers is limited by the stability of the bulb volume, which may slowly change over long periods of time at temperatures near ambient, or may change more rapidly upon exposure to higher

temperatures. Bulb stability should be monitored by checking the ice point on a regular basis and maintaining appropriate control charts that indicate both calendar date of the ice point reading and the time of exposure above 100 °C. The interval between ice point checks should be determined by the rate of change in ice point readings observed on the control charts. The initial interval for ice point checks of thermometers of unknown stability is indicated in the chart below.

<u>Accuracy class</u>

	I	II	III	IV	V
Interval			(after each thermal cycle)	weekly	monthly

2.3.5 Personnel

2.3.5.1   For accuracy class I, one of the persons working in the laboratory should have attended the NIST Precision Thermometry Workshop and the NIST Fixed-point Cell Mini-Workshop, or have an equivalent background.

2.3.5.2   For accuracy classes II through IV, one of the persons working the laboratory should have specialized training or experience in the field of thermometry.

2.3.6 Accommodation and environmental conditions

2.3.6.1   For all of the accuracy classes, the environmental conditions of the laboratory should be controlled.

2.3.6.2   The effects of variation in the temperature and humidity of the laboratory should be accounted for in the uncertainty budget. This may be accomplished by measuring the repeatability of thermometer calibrations under varying environmental conditions, or by referring to manufacturers' specifications for the laboratory equipment used in calibrations.

2.3.6.3   The temperature and humidity of the laboratory should be controlled such that the environment meets all manufacturers' specifications for the laboratory equipment used in the calibrations.

2.3.7 Equipment

2.3.7.1   Reference standards

The following table indicates which reference standard is acceptable for each class.

Acceptable reference standard	Accuracy class				
	I	II	III	IV	V
Fixed-point cell	x	x	x		
SPRT and/or RIRT	x	x	x	x	x
IPRT	x	x	x	x	x
Thermistor thermometer	x	x	x	x	x
Gold/platinum thermocouple		x	x	x	x
Type S, R or B thermocouple				x	x
Total-immersion liquid-in-glass			x	x	x

2.3.7.2 Fixed-point cell as the reference standard

a)　The purity of the fixed-point material should be ≥99.9999 % and the other starting materials of construction of the cells should be of ultra-high purity also. If the cells are unsealed, they should be filled at all times with an inert gas, such as argon.

b)　The cells should be of the defining fixed points of the ITS-90, or well-characterized, stable and reproducible secondary fixed points.

2.3.7.3 SPRT or RIRT as the reference standard

a)　A system having adequate resolution and uncertainty for the desired accuracy class should be used to measure a reference SPRT or RIRT. Recommendations for specific situations are given below.

b)　A resistance bridge having at least the resolution shown below is recommended. A ratio bridge and standard resistors may also be used:

Accuracy class	Claimed total expanded uncertainty (k=2)	Minimum bridge resolution
I	±0.002 °C	0.000 01 Ω
II	≤±0.01 °C	0.000 1 Ω
III	±0.05 °C	0.000 5 Ω
IV	near ±0.20 °C	0.001 Ω
V	near ±1.0 °C	0.02 Ω

c)　A DVM or DMM with the resolution shown below, and a constant-current source with provision for reversing the current, may be used. The current should be known to the same accuracy as the DVM or DMM. Alternatively, a DMM with offset compensation may be used. In either case, the current used should be equal to the current used in the calibration of the reference standard.

Accuracy class	DVM or DMM resolution (digits)
I	8 1/2
II	8 1/2
III	6 1/2
IV	6 1/2

d)   A digital temperature indicator having adequate resolution and uncertainty for the desired accuracy class may be used to measure a reference SPRT or RIRT.

2.3.7.4 IPRT as the reference standard

a)   A system having adequate resolution and uncertainty for the desired accuracy class should be used to measure a reference IPRT, SPRT or RIRT. Recommendations for specific situations are given below.

b)   A resistance bridge having at least the resolution shown below, as a function of claimed total uncertainty, is recommended. A ratio bridge and standard resistors may also be used:

Accuracy class	Claimed total expanded uncertainty (k=2)	Minimum bridge resolution
I	±0.002 °C	0.000 01 Ω
II	±0.01 °C	0.000 1 Ω
III	±0.05 °C	0.000 5 Ω
IV	near ±0.20 °C	0.002 Ω
V	near ±1.0 °C	0.01 Ω

c)   A DVM or DMM with the resolution shown below, and a constant-current source with provision for reversing the current, may be used. The current should be known to the same accuracy as the DVM or DMM. Alternatively, a DMM with offset compensation may be used. In either case, the current used should be equal to the current used in the calibration of the reference standard.

Accuracy class	DVM or DMM resolution (digits)
I	8 1/2
II	8 1/2
III	6 1/2
IV	6 1/2

d)   A digital temperature indicator having adequate resolution and uncertainty for the desired accuracy class may be used to measure a reference IPRT.

2.3.7.5   Thermistor as the reference standard

a)   A system having adequate resolution and uncertainty for the desired accuracy class should be used to measure a reference thermistor.

b)   A resistance bridge or resistance ratio bridge having adequate resolution and uncertainty for the desired accuracy class may be used to measure a reference thermistor.

c)   A DVM or DMM with the resolution shown below, and a constant-current source with provision for reversing the current, may be used. The current should be known to the same accuracy as the DVM or DMM. Alternatively, a DMM with offset compensation may be used. In either case, the current used should be equal to the current used in the calibration of the reference standard.

Accuracy class	DVM or DMM resolution (digits)
I	8 1/2
II	8 1/2
III	6 1/2
IV	6 1/2

d)   A digital temperature indicator having adequate resolution and uncertainty for the desired accuracy class may be used to measure a reference thermistor.

2.3.7.6   Liquid-in-glass as the reference standard

When a liquid-in-glass thermometer is used as a reference standard, and the thermometer is read to better than one-half of a scale division, the optical magnification of scale graduations and procedures to avoid parallax errors should be suitable for the desired resolution and accuracy.

2.3.7.7   Thermocouple as the reference standard

a)   If the reference standard is a noble metal thermocouple used with a scanner, a scanner with low-thermal switches should be used.

b)   Any method for reference junction compensation must be demonstrated to give results equivalent to a true 0 °C reference junction temperature.

c)   The effects of thermal emf of any wiring or instrumentation between the thermocouple readout and the actual reference thermocouple must be accounted for, and uncertainties for these effects included in the uncertainty budget.

d)   The offset voltage of the thermocouple readout (i.e., the reading of the instrument when the inputs are appropriately shorted) should be monitored, and corrected for, if necessary.

Item	Accuracy class to which item applies			
	II	III	IV	V
a)	x	x	x	
b)	x	x	x	
c)	x	x	x	
d)	x	x		

e)   A DVM or DMM with the resolution shown below, or an equivalent digital readout, may be used.

Accuracy class	DVM or DMM resolution (digits)
II	8 1/2
III	7 1/2
IV	6 1/2
V	5 1/2

2.3.8   Measurement traceability

2.3.8.1   Fixed-point cell as the reference standard

When the reference standard is a fixed-point cell, the five action items described below are recommended as indicated by the application table that follows them.

a)   The cell should be certified by an NMI that has participated in the appropriate CCT key comparison (certification in this context means that the cell is tested and found to be acceptable as an ITS-90 defining standard to within some stated uncertainty) or

b)   The cell may have been certified by the supplier, if the supplier either documented in detail the preparation and certification, showing direct traceability to an NMI or through a laboratory accredited by an ILAC signatory, or is accredited as a temperature calibration laboratory by an accrediting body recognized by ILAC.

c)   The maximum expanded uncertainty ($k=2$) of the temperature of the cell should be as indicated below.

d)   Fixed-point cells should be recertified or compared with a cell of certification less than one year old, at the indicated intervals.

e)   An alternative to having fixed-point cells certified is to perform a Measurement Assurance Program (MAP) with NIST using SPRTs as the transfer standard to provide a total calibration system check to verify the stated calibration uncertainties. The interval between MAPs is indicated below. Because the accuracy of a fixed-point realization depends on many factors in addition to the accuracy of the cell itself, a MAP is a superior test of proficiency at realizing the ITS-90. For information on a MAP, please contact G. Strouse at 301-975-4803 or at gstrouse@nist.gov.

Item	Accuracy class			
	I	II	III	IV
a)	x	x[*]		
b)		x[**]		
c)	±0.001 °C	±0.005 °C	±0.01 °C	±0.02 °C
d)	5 years	7 years		
e)	3 years	5 years		

[*] for total uncertainties ≤±0.01 °C
[**] for total uncertainties in range ±0.01 °C to ±0.05 °C

2.3.8.2   SPRT or RIRT as the reference standard

When the reference standard is an SPRT or RIRT, the three action items described below are recommended as indicated by the application table that follows them.

a)   The minimum calibration interval for the SPRT or RIRT is determined by the results of the statistical process control specified in section 2.3.4.2 b). Calibration of the SPRT or RIRT shall be performed

by an NMI or a laboratory accredited by an ILAC signatory.

b)   If a bridge is used, it should be validated annually, and all reference resistors used with the bridge should be calibrated traceable to national standards.

c)   If a DVM or DMM is used, it should be calibrated annually.

Item	Accuracy class				
	I	II	III	IV	V
a)	x	x	x	x	x
b)	x	x	x	x	
c)	x	x	x	x	x

2.3.8.3   IPRT as the reference standard

When the reference standard is an IPRT, the four action items described below are recommended as indicated by the application table that follows them.

a)   The minimum calibration interval of the IPRT is determined by the results of the statistical process control specified in section 2.3.4.3 b). Calibration of the IPRT shall be performed by an NMI or a laboratory accredited by an ILAC signatory.

b)   As an alternative to 2.3.8.3 a), the IPRT may be calibrated annually by an NMI or a laboratory accredited by an ILAC signatory.

c)   If a bridge is used, it should be validated annually, and all reference resistors used with the bridge should be calibrated traceable to national resistance standards.

d)   If a DVM or DMM is used, it should be calibrated annually.

Item	Accuracy class				
	I	II	III	IV	V
a)	x	x	x	x	x
b)				x	x
c)	x	x	x		
d)	x	x	x	x	x

2.3.8.4   Thermistor as the reference standard

When the reference standard is an thermistor, the four action items described below are recommended as indicated by the application table that follows them.

a)   The minimum calibration interval of the thermistor is determined by the results of the statistical process control specified in section 2.3.4.4 b). Calibration of the thermistor shall be performed by an NMI or a laboratory accredited by an ILAC signatory.

b)   As an alternative to 2.3.8.4 a), the thermistor may be calibrated annually by an NMI or a laboratory accredited by an ILAC signatory.

c)   If a bridge is used, it should be validated annually, and all reference resistors used with the bridge should be calibrated traceable to national standards.

d)   If a DVM or DMM is used, it should be calibrated annually.

Item	Accuracy class				
	I	II	III	IV	V
a)	x	x	x	x	x
b)				x	x
c)	x	x			
d)	x	x	x	x	x

2.3.8.5   Thermocouple as the reference standard

a)   Documentation should show that the thermocouple calibration is traceable to national standards, indicate the annealing procedure used prior to and during the thermocouple's use, and document the total duration of use at elevated temperatures.

b)   If reference standard thermocouples are cut from a larger lot of thermocouple material and the calibration of the thermocouple taken as equal to that of a sample of the lot, the homogeneity of each lot of thermocouple material shall be tested and documented. The readout used to measure the thermocouple emf should be calibrated at least annually.

c)   The thermocouple material should be protected from chemical contamination at high temperature from such sources as metal or oxide vapors. A small amount of contamination is allowable near the measuring junction, if that junction is in a region of the calibration apparatus with small thermal gradients.

2.3.8.6   Liquid-in-glass thermometer as the reference standard

If a liquid-in-glass thermometer is the reference standard, it should have been calibrated traceable to national temperature standards. Periodic inspection of the thermometer for separated columns and monitoring and/or correction of the ice-point depression (see section 2.3.4.6) is required to ensure continued traceability.

2.3.9   Test and calibration methods and method validation

2.3.9.1   All computer programs used in data logging and analysis should be documented in detail. Documentation should include:

a)   The scope, purpose, and application of each program; and

b)   For specialized software, evidence of the correctness of calculations and algorithms by comparison of results computed by independent means.

2.3.9.2   Methods should be in place to prevent unintended and unvalidated modification of programs.

2.3.9.3 When calibrations are performed by comparison against a reference thermometer, the accuracy of the calibration often depends on the degree of thermal equilibrium between the test and reference thermometers, rather than the accuracy of the calibration of the reference thermometer. To address the issues of proper thermal equilibrium, the following actions should be taken.

a) The uncertainty budget should include components for the temperature stability and uniformity of the comparison medium.

b) The stability and uniformity of the comparison medium should be determined by direct measurement in the actual apparatus used for calibrations.

c) When the comparison apparatus is a stirred liquid bath, with a medium consisting of a fluidized bed, oil, or a hygroscopic liquid (such as chilled alcohol), check standards should be used to verify the continued uniformity and stability of the bath. The implementation of check standards should be designed so that the measurements of these check standards are sensitive to variations in the stability and uniformity of the comparison medium. For example, in a liquid bath, check standard thermometers should be placed farther away from the reference thermometer than the test thermometers. Similarly, the sequence of reading the check thermometers should be chosen to ensure that these measurements are at least as sensitive to fluctuations in the temperature of the medium as the measurements of the test thermometers.

d) As an alternative to 2.3.9.3 c), 2.3.9.3 b) may be performed at the indicated intervals.

Item	Accuracy class				
	I	II	III	IV	V
a)	x	x	x	x	x
b)	x	x	x		
c)	x	x	x	x	x
d)	2 mon	4 mon	6 mon	1 year	1 year

e) When the reference standard is a total-immersion mercury-in-glass thermometer, corrections obtained from measurements at the ice point should be made for all temperature measurements.

f) The ice-point bath should be made according to accepted procedures from ice made from distilled water. This applies to accuracy classes III, IV and V.

2.3.10   Handling of test and calibration items

In addition to the general requirements set forth in the Program Handbook, it should be noted that SPRT's are susceptible to and need protection from shock and vibration in shipping, handling, and storage.

2.4 Calibration of leak artifacts between (10^{-14} and 10^{-6}) mol/s by comparison with a standard permeation leak

2.4.1 Introduction

2.4.1.1   Leaks or leak artifacts are devices that provide controlled delivery of gases at relatively low flow rates (generally between 10^{-14} mol/s to 10^{-6} mol/s (2×10^{-9} std. cm³/s to 2×10^{-2} std. cm³/s at 0 °C)). For the purposes of this document, leaks are defined as flows less than 10^{-6} mol/s (2×10^{-2} std. cm³/s at 0 °C). Leak artifacts can be used for calibration of leak detection instrumentation such as mass spectrometers or halogen detectors. There are many types of leaks and applications for which they are used. In this document a distinction will be made between physical leaks and permeation leaks.

a)   Physical leaks are generally composed of a gas supply, a physical restriction, and a gas outlet. The physical restriction may come in numerous forms such as a capillary, crimped capillary, sintered material, orifice, or other restrictive geometry.

b)   Permeation leaks use a glass, quartz, or polymer barrier to restrict the flow of the gas of interest via diffusion. Helium glass permeation leaks will be solely addressed in this document.

2.4.1.2   Flow rates are generally quoted at standard conditions for leak measurement of P = 101 325 Pa and T = 0 °C. When referenced to the specific temperature, std. cm³/s can be converted to mol/s by multiplying by 4.45×10^{-5}.

2.4.2 Scope

2.4.2.1   This section provides general guidelines for calibration of leaks by comparison with a standard permeation leak. The section is tutorial in nature describing the best practices to minimize measurement errors. The section is not all-inclusive and cannot be used to determine the ability of a laboratory to calibrate leaks to within a specified tolerance.

2.4.2.2   This section does not address calibration of physical leaks.

2.4.2.3   This section will describe the various types of leaks and their properties, the process for direct comparisons, the required equipment, the procedure for calibration of leaks, assessment of uncertainties in the measurement process, and the recommended use and handling of leaks.

2.4.3 References

a)   Tison, S. A., "A Critical Evaluation of Thermal Mass Flow Meters," *J. Vac. Sci. Technol.*, **A 14**, 2582, (1996).

b)   Abbott, P. J., and Tison, S. A., "Commercial helium permeation leak standards: Their Properties and Reliability," *J. Vac. Sci. Technol.*, **A 14**, (1996).

c)   Tison, S. A., and Mohan, P., "Using Characterized Variable Reservoir Helium Permeation Leaks to Generate Low Flows," *J. Vac. Sci. Technol.*, **A 12**, 564, (1994).

d)   Tison, S. A., "Experimental Data and Theoretical Modeling of Gas Flow Through Metal Capillary Leaks," *Vacuum,* **44**, 1171, (1993).

e)   Ehrlich, C. D., and Tison, S. A., "NIST Leak Calibration Service," *NIST Special Publication 250-38*, (1992).

f)   Ehrlich, C. D., Tison, S. A., Hsiao, H. Y., and Ward, D. B., *J. Vac Sci. Technol.* **A 8**, 4086, (1990).

g)   Hyland, R. W., Ehrlich, C. D., and Tilford, C. R., "Transfer Leak Studies and Comparisons of Primary Leak Standards at the National Bureau of Standards and Sandia National Laboratories," *J. Vac. Sci. Technol.*, **A 4**, 334, (1986).

h)   Ehrlich, C. D., "A Note on Flow Rate and Leak Rate Units," *J. Vac. Sci. Technol.*, **A 4**, 2384, (1986).

i)   McMaster, R. C., et al., *Nondestructive Testing Handbook,* Leak Testing, American Society for Nondestructive Testing, Vol. 1, American Society for Metals, (1982).

2.4.4 Accommodation and environmental conditions

2.4.4.1   Leaks should be kept clean at all times.

2.4.4.2   The leak rate of the permeation leak is dependent upon the temperature of the leak, the composition and construction of the leak element, as well as the reservoir helium concentration behind the leak element. As helium flows out of the leak element, the reservoir concentration decreases, and thus the leak rate decays. It is important to determine the decay rate of the leak since it can vary over time depending on how the leak is constructed, how it is used, and how it is stored (see 2.4.4.3).

2.4.4.3   If an isolation valve exists on the outlet side, it should not be closed for an extended period of time. Closing the valve for a few minutes during the measurement process is acceptable, but if helium is allowed to build up, the leak rate of the artifact will be affected. After closure of this valve for extended periods of time, re-equilibration of the leak can take up to two weeks.

2.4.4.4   Helium glass permeation leaks are fragile and may be damaged by rough handling.

2.4.5 Equipment

2.4.5.1   Glass permeation leaks are the most widely used because of their selectivity for specific gas permeation, most often helium. A glass permeation leak is usually composed of a tubular glass element which is sintered to metal and then welded into a reservoir which completely encapsulates the element. The reservoir is filled with helium which diffuses across the glass element according to the concentration gradient of helium within the glass. The concentration gradient is dependent on the concentration of helium at the surface of the glass element. The concentration of the adsorbed helium is dependent upon the solubility of the helium in the glass.

2.4.5.2   The permeation rate of the element is a function of the geometry, type of glass, reservoir helium concentration and temperature. The permeation rate is generally a linear function of the reservoir concentration. The temperature dependence has been experimentally determined to be an exponential function of the form

$$Q_m = A\,T\,e^{-\frac{E}{RT}}, \tag{1}$$

where in compatible units, Q_m is the molar leakage rate (mol/s), A is a constant incorporating the helium concentration, diffusivity, solubility, and geometrical properties of the leak element, T is the absolute temperature, E is the diffusivity activation energy, and R is the ideal gas constant. The constants A and E are experimentally determined. The constant A is a function of the helium concentration and will change proportionally as the helium in the reservoir is depleted. The activation energy E may vary from (2.0 to 4.0) KJ depending upon the type of glass, but is usually very stable with time.

2.4.5.3 Although the temperature dependence is known to be exponential, it is common practice to approximate Eq. (1) with the linear form

$$Q_m = Q_{cal} [1 - \alpha(T - T_{cal})], \qquad (2)$$

where in compatible units, Q_{cal} is the molar leak rate at the reference temperature (T_{cal}), α is the linear temperature coefficient, and T is the temperature for which the leak rate is to be obtained. The linear temperature coefficient typically varies from 2 % to 5 % and is only a function of glass type. Numerous errors can be made in using a linear temperature coefficient. It is recommended that linear temperature coefficients not be used if the ambient temperatures vary by more than 5 °C from the calibration temperature.

2.4.5.4 Most commercially available glass permeation leaks have a closed reservoir with a fixed helium reservoir concentration. The constant A in Eq. (1) in this case only changes due to depletion of the helium in the reservoir. The physical time constant required for equilibration due to a step change in temperature of a leak is generally on the order of a few minutes or less. The main obstacle to accurate temperature compensation is the accurate determination of the temperature of the glass leak element. In many instances, changes in the leak reservoir may require up to two hours for the leak element to equilibrate.

2.4.5.5 Changes in the leak reservoir helium concentration may require up to 2 weeks before the leak rate equilibrates to within 1 % of its steady state value for typical commercial glass permeation leaks. It should be noted that the leak rate is proportional to the helium concentration gradient within the element and not the reservoir pressure. As such, changes in the helium concentration on either side of the element will cause changes in the leak rate. It is therefore recommended that any shut-off valve on the vacuum side of the leak be left open so that helium is not allowed to accumulate during storage.

2.4.6 Test and calibration methods and method validation

2.4.6.1 The direct comparison leak calibration utilizes a mass-spectrometer-type leak detector or a mass spectrometer with a vacuum system. The leak rate of an unknown leak is determined by comparison with a calibrated leak of known flow using the mass spectrometer to make the comparison measurements. It is recommended that both the calibrated leak and the unknown leak be mounted on the system concurrently with isolation valves so that the flows can be independently directed to the mass spectrometer.

2.4.6.2 Required equipment

a) **Helium leak detector** - The system should have a tuned magnetic sector helium mass spectrometer as the helium detector. Most commercial helium leak detectors employ this type of detector and this document is written for this particular type of instrument. The system should have a means to mount the calibrated and unknown leaks together and have the ability to isolate them from the detector independently. The pumping system should be capable of maintaining a vacuum below 1×10^{-4} Pa at all times during the measurement process.

b) **Calibrated leaks** - Calibrated helium permeation leaks are required to determine the sensitivity of the detector. The number of calibrated leaks required depends upon the range of the leak rate measurements to be performed and is discussed subsequently. Most commercial leaks have a fixed gas reservoir and as such the leak rate will decay as a function of time from calibration date and the initial fill pressure. The decay rates may be estimated based upon the initial quantity of gas in the reservoir and the initial calibrated leak rate. Typical leaks may have decay rates from 20 % to 1 % per year. Leaks may also change due to damage to the element or the reservoir, which may increase the decay rate or completely deplete the reservoir. For this reason, a calibrated leak will be defined as a leak which has been previously calibrated within a twelve month period from the time of test. Use of statistical process control may be justification for using a longer period.

2.4.6.3　Apparatus preparation

To use the instrument for calibration of leaks, the sensitivity should be determined as a function of leak rate. The sensitivity of the instrument should be determined using the procedures in the following paragraphs. The instrument's sensitivity should be determined at two points per decade and should span the leak rates so that extrapolation is not required. Two examples follow:

a) It is desired to calibrate a leak with a nominal rate of 1.3×10^{-12} mol/s. Two calibrated leaks are available with rates of 4×10^{-12} mol/s and 4×10^{-13} mol/s. The sensitivity of the instrument was determined using the procedures in the next section to be 4.45×10^{-11} (amps)/(mol/s) and 4.67×10^{-11} (amps)/(mol/s) respectively for the two calibrated leaks. The uncertainty in the sensitivity over this range of measurement is then computed to be the percent change in the sensitivity between the two measured values: in this case 5 %.

b) To calibrate leaks from 4×10^{-14} mol/s to 4×10^{-12} mol/s, a minimum of three calibrated leaks are required with the following nominal leak rate values: 1×10^{-14} mol/s $\pm$ 10 %, 1×10^{-13} mol/s $\pm$ 10 %, and 1×10^{-12} mol/s $\pm$ 10 %. The sensitivity of the instrument should be determined at two points on the scale for which the unknown leak is to be calibrated.

2.4.6.4　Leak rate measurement

a) The calibrated and unknown leaks should be installed on the comparison system and be pumped by the system until a stable base pressure is achieved. Operation of mass spectrometers with changing background pressures has been demonstrated to affect the sensitivity of numerous instruments and is not recommended. Provision should be made for measuring the temperature of the calibrated and unknown leaks by direct attachment of temperature probes to the leak reservoirs. The leaks should be allowed to thermally equilibrate so that the temperature changes no more than 1 °C/h.

b) The mass spectrometer should be tuned to helium according to the manufacturer's recommendations and any required zeroing of electronic components should be performed. The helium signal of the mass spectrometer should be recorded as a function of time for a minimum period of one minute and the average ion current for the helium signal, I_{O1}, calculated. The calibrated leak should be valved into the mass spectrometer and adequate time be allowed for the signal to equilibrate. This equilibration time will be a function of the system volume and pumping speed. After the signal has equilibrated, the signal should be recorded as a function of time for a minimum period of one minute; the temperature of the calibrated leak. T_{S1}, should be recorded concurrently. The average of the ion current, I_{C1}, should be calculated,. The calibrated leak should then be valved out and the unknown leak valved into the mass spectrometer and time allowed for the signal to equilibrate. Upon equilibration, the signal should be recorded as a function of time for a minimum period of 1 minute

and the average ion current value for the unknown leak, I_{U1}, determined. The temperature of the unknown leak, T_{U1}, should be recorded concurrently. The unknown leak should be valved out and the calibrated leak valved into the mass spectrometer. Upon equilibration, the signal should be recorded as a function of time for a minimum period of 1 minute and the ion current average value for the calibrated leak, I_{C2}, determined. The calibrated leak should be valved out and the ion signal allowed to equilibrate. Upon equilibration, the signal should be recorded as a function of time for a minimum period of 1 minute and the average ion current value, I_{O2}, calculated. The unknown leak rate can then be calculated with the following equation where $Q_C(T_{C1})$ and $Q_C(T_{C2})$ are the leak rates of the calibrated leak at the respective temperatures.

$$Q_U(T_{U1}) = \frac{Q_C(T_{C1}) + Q_C(T_{C2})}{2} \left[\frac{I_{U1} - (I_{O1} + I_{O2})/2}{\dfrac{I_{C1} + I_{C2} - I_{O1} - I_{O2}}{2}} \right] \tag{3}$$

This equation assumes that the mass spectrometer sensitivity is constant over the range of the measurements. Differences between $Q_C(T_{C1})$ and $Q_C(T_{C2})$ greater than a few percent indicate excessive changes in the leak rate due to temperature fluctuations during the measurement. Differences between I_{C1} and I_{C2} of more than a few percent indicate large instabilities in the sensitivity of the mass spectrometer and lead to unreliable measurements. The helium sensitivity of the instrument can be defined in the following manner:

$$S(Q_C) = \frac{\dfrac{Q_C(T_{C1}) + Q_C(T_{C2})}{2}}{\dfrac{I_{C1} + I_{C2} - I_{O1} - I_{O2}}{2}} \tag{4}$$

where $S(Q_C)$ is the sensitivity of the mass spectrometer at the leak rate of the calibrated leak. Eq. (3) can be rewritten by combining Eqs. (3) and (4) as

$$Q_U(T_{U1}) = S(Q_C)[I_{U1} - (I_{O1} + I_{O2})/2] \tag{5}$$

c) Apart from aberrant behavior of the mass spectrometer, the systematic uncertainties in the leak rate measurement of the unknown leak at temperature T_{U1} may be calculated with the following expression,

$$\frac{\delta Q_U(T_{U1})}{Q_U(T_{U1})} = \left[\left[\frac{\delta Q_C(T_C)}{Q_C(T_C)} \right]^2 + \left[\frac{\delta S(Q_U)}{S(Q_U)} \right]^2 + (\alpha_C \delta T_C)^2 + (\alpha_U \delta T_U)^2 \right]^{1/2} \tag{6}$$

Terms   1   2   3   4

where $\delta Q_C(T_C)$ is the uncertainty in the leak rate of the calibrated leak at temperature T_C, $\delta S(Q_U)$ is the uncertainty in the sensitivity of the mass spectrometer at leak rate Q_U, α_C is the linear temperature coefficient of the calibrated leak, δT_C is the uncertainty in the temperature of the calibrated leak (degree Celsius), α_U is the linear temperature coefficient of the unknown leak and δT_U is the

uncertainty in the temperature of the unknown leak. All uncertainties are expressed at the two standard deviation level of confidence unless otherwise stated. The uncertainty in the leak rate of the calibrated leak at the appropriate temperature can be obtained from its calibration report. The uncertainty in the sensitivity of the mass spectrometer can be estimated from previous characterization of the instrument. The errors due to uncertainties in the sensitivity of the mass spectrometer can be reduced by closely matching the standard and unknown leak rates. The uncertainty in the temperature of the calibrated leak includes the uncertainty of the temperature measuring device and the effects of temperature gradients that may exist between the measurement spot and the actual leak element. The temperature coefficient of the unknown leak if not previously measured should be estimated for use in Eq. (6). An average number of 4 % / °C would be appropriate for most commercial leak elements. The uncertainty in the temperature of the unknown leak includes the uncertainty of the temperature measuring device and the effects of temperature gradients that may exist between the measurement spot and the actual leak measurement. If an exponential model for temperature dependence is used, terms 3 or 4 from Eq. (6) may be replaced by $(E \, \delta T)/RT^2$.

d) To determine the random errors associated with the measurements, it is recommended that the process be repeated a minimum of five times. The random uncertainty in the unknown leak rate will then be given by the two sigma value of the standard deviation of the mean unknown leak rate. The total uncertainty of the measurement process can be obtained by a root-sum-of-squares combination of all uncertainty components. The two sigma systematic uncertainty in the measurements can be obtained by multiplying the result of Eq. (6) by two to produce results for 95 % confidence levels.

2.4.6.5   Special considerations

Many modern commercial leak detectors utilize built-in calibrated leaks and autocalibration procedures which are used to determine the sensitivity of the instruments. They then use the measured sensitivity to calculate the unknown leak rate. These systems may be used if the following precautions are followed:

a) The built-in leak is itself calibrated.

b) The leak detector accounts for the temperature dependence of the calibrated leak.

c) The built-in calibrated leak is of the appropriate range for the measurement.

d) The uncertainty of the sensitivity of the instrument has been determined according to the preceding paragraphs.

e) The built-in, autocalibration procedure is verified by measuring a calibrated leak of approximately the same leak rate as the built-in leak and comparing the measured value with the calibrated value for consistency.

2.5 Pressure calibrations

2.5.1 Areas of commonality among parameters

2.5.1.1 Scope

a) This section contains specific technical criteria a laboratory engaged in pressure calibrations should follow if it is to be recognized as competent to carry out calibrations of, and/or use, pressure artifacts. The artifact calibrations currently included in the accreditation program are:

 1) Deadweight pressure gages

   a. Pneumatic deadweight piston gages (gage mode)

   b. Pneumatic deadweight piston gages (absolute mode)

   c. Hydraulic deadweight piston gages

   d. Pneumatic deadweight ball gages

 2) Pressure transducers

b) It is assumed that the pressure artifacts used by the calibration laboratory for calibrating other pressure artifacts have been previously calibrated through a traceable path to the pressure laboratory of a recognized standards authority.

c) This section does not address technical requirements for laboratories that perform effective area calibrations of deadweight pressure gages using dimensional metrology, due to the possible large range of uncertainties associated with the variability of the models used to calculate effective area from dimensional measurements (see 2.5.1.8, Estimation of uncertainty of measurement).

2.5.1.2 References

a) ASME/ANSI Performance Test Code 19.2-1987, reaffirmed 1998, *Pressure Measurement, Instruments and Apparatus, Supplement on Instruments and Apparatus, Part 2.*

b) UL 404, *Standard for Safety; Gauges Indicating Pressure for Compressed Gas Service*, Underwriters Laboratories Incorporated, July 30, 1979.

2.5.1.3 Assuring the quality of test and calibration results

a) All sources of variability for the calibration should be monitored by subsystem calibration (e.g., thermometer, humidity measuring devices) and the use of check standards to ensure that the calibrations are carried out under controlled conditions. The laboratory should maintain some form of statistical process control (SPC) commensurate with the accuracy levels needed for the calibration. The SPC control parameters should be based on measurements of check standards (or closure parameters) and the repeatability of multiple measurements. The frequency and number of process control checks should be appropriate for the level of uncertainty and reliability claimed for the calibration.

b) The laboratory should have a manual outlining the procedures to be followed for each type of calibration. For calibrations involving assessment of accuracy classification, the procedure should name the accuracy classifications that are covered by the procedure.

2.5.1.4   Accommodation and environmental conditions

a) The temperature in the pressure calibration laboratory should be consistent with the requirements of the equipment, as specified by the manufacturer, and consistent with good pressure measurement practice. The temperature should be such that the operating pressure fluids maintain their hydrostatic properties. The NIST Pressure and Vacuum Group normally references to 23 °C. Thermal expansion coefficients of piston/cylinder materials range from approximately 4.0×10^{-6} / °C for tungsten carbide to 12.0×10^{-5} / °C for steel. Attention should be paid to achieving a reasonable time-rate-of-change of temperature during the calibration period.

b) The relative humidity in the pressure calibration laboratory should be from 40 % to 60 %, with a temporal rate of change less than ± 2 % / h. It should be carefully monitored, and should not exceed that specified by the manufacturer of the equipment. Attention should be paid to achieving a reasonable time-rate-of-change of relative humidity during the calibration period.

c) Excessive vibration should be avoided in the pressure calibration laboratory. If an obvious source of vibration exists, it should not adversely affect the laboratory's claimed accuracy or uncertainty level.

2.5.1.5   Equipment

a) The laboratory should have the equipment needed to make auxiliary measurements on artifacts (e.g., thermometer, leveling capabilities, humidity indicator, electronic equipment, etc.).

b) The laboratory should have temperature measuring capabilities suitable to the calibration procedure. The measured temperature should be used, when uncertainties warrant, to make corrections to the effective area of deadweight pressure gages, or to compensate for the temperature dependence of pressure transducers.

c) A laboratory that certifies artifacts to accuracy classes should demonstrate a measurement uncertainty satisfactorily within the accuracy class designation.

2.5.1.6   Handling of test and calibration items

a) Pressure artifacts should be cleaned where required and carefully handled. Pressure artifacts should be stored in a manner that prevents damage. Manufacturer's recommendations should be followed.

b) Pressure artifacts may be fragile, requiring special packing and shipping methods to be used. The manufacturer's manual should also be consulted.

2.5.1.7   Personnel

Persons performing cross-floating pressure calibrations should be properly trained.

2.5.1.8 Estimation of uncertainty of measurement

The uncertainty in the pressure and/or effective area should be derived (and reported) from a model of the measurement system which includes (as applicable) the uncertainties due to:

a)   The pressure standard;

b)   Reproducibility of measurement system; and

c)   Other factors as appropriate.

2.5.1.9 Measurement traceability

An effective area measurement is traceable at the national level when it can be related to appropriate primary pressure standards in the pressure laboratory of the national standards laboratory through an unbroken chain of comparisons. While it is possible to have physical area measurements traceable at the national level to the dimensional laboratory of the national standards laboratory, taking this route of traceability to the national standards laboratory does not guarantee that the effective area determined from these measurements will yield the same effective area as would be obtained from traceability to the pressure laboratory, since different models or methods of handling the same dimensional data might be used. If traceability to the dimensional laboratory is used, then a complete traceability statement will include not only the traceability of the dimensional measurements, but a complete statement of the dimensional data and the model that was used to determine the effective area, including all uncertainties.

2.5.2 Unique areas of concern

The following sections list those areas considered unique to calibrations performed in pressure/effective area measurements. Where these situations exist, the assessor should give special attention to these areas when considering accreditation of the calibration laboratory.

2.5.3 Pneumatic deadweight piston gage calibration (gage mode)

2.5.3.1 Scope

This section contains specific technical criteria a laboratory engaged in pneumatic deadweight piston gage calibrations should follow if it is to be recognized as competent to:

a)   Use pneumatic deadweight piston gages in the gage mode of operation to perform calibrations, or

b)   Certify pneumatic deadweight piston gages in the gage mode to accuracy class.

2.5.3.2 References

a)   Heydemann, P. L. M., and Welch, B. E., *Piston Gages*, International Union of Pure and Applied Chemistry, Experimental Thermodynamics, Vol. II, Chapter 4, Part 3, pages 147-202, B. LeNeindre and B. Vodar, editors, Butterworths, London, 1975.

b)   *NCSL International Recommended Intrinsic/Derived Standards Practice (RISP) 4 on Deadweight Pressure Gauges*, NCSL International, Boulder, CO, 1998.

c)   Dadson, R. S., Lewis, S. L., and Peggs, G. N., *The Pressure Balance: Theory and Practice*, Her Majesty's Stationary Office, London, 1982.

2.5.3.3   Assuring the quality of test and calibration results

A pneumatic deadweight piston gage or a high-quality pressure transducer may be used as a process control check standard. The laboratory should have a manual outlining the procedures to be followed for the use and/or calibration of pneumatic deadweight piston gages in the gage mode of operation.

2.5.3.4   Accommodation and environmental conditions

In addition to the general factors discussed in section 2.5.3.8, the following factors should be considered and may be significant when performing calibrations of pneumatic deadweight piston gages in the gage mode of operation: leveling, reference level, operating position, air currents, electrostatic charging, magnetization, system cleanliness, system leakage, spin time, and mass loading.

2.5.3.5   Equipment

a)   The laboratory should have the equipment necessary to perform calibrations of pressure measuring equipment using a pneumatic deadweight piston gage. Such equipment should include a clean pressurized gas source and adequate plumbing and valves. For higher-quality measurements, thermometers, a barometer, a hygrometer, rotational-speed indicator, and other related measuring equipment may be used. If dissimilar fluids are used, some means of fluid separation is required.

b)   When applicable, the laboratory should have the equipment necessary to perform calibrations of other deadweight pressure gages using the cross float method or other documented procedures. Such equipment should include a clean pressurized gas source, adequate plumbing and valves, and piston/cylinder position indicators. In addition, a means of determining balance conditions should be provided, such as through the use of fall rate measurements, differential pressure cells, or equivalent. For higher-quality measurements, thermometers, a barometer, a hygrometer, rotational-speed indicator, and other related measuring equipment may be used. If dissimilar fluids are used, some means of fluid separation is required.

2.5.3.6   Test and calibration methods and method validation

A manual outlining the procedures to be followed for all of the various uses of pneumatic deadweight piston gages by the calibration laboratory should be maintained. Such uses might include the calibration of pressure measuring equipment or other deadweight pressure gages. For calibrations involving assessment of accuracy classification, the procedure should name the accuracy classifications that are covered by the procedure.

2.5.3.7   Handling of test and calibration items

a)   Pneumatic deadweight piston gages should be cleaned where required and carefully handled. After assembly of the piston/cylinder and associated parts, adequate time should be provided to ensure that they have come to thermal equilibrium with their environment. Specific guidelines for this process should be stated in the measurement procedure.

b)   When loading masses directly on pistons in pneumatic deadweight piston gages, care should be taken to avoid lateral forces that may bend or break the piston. Similar care should be taken when hand-rotating the masses.

c) Pistons/cylinders should be stored in a manner that prevents damage. Manufacturer's recommendations should be followed.

2.5.3.8 Reporting the results

a) The uncertainty in the pressure generated and measured by a pneumatic deadweight piston gage should be derived (and reported) from the uncertainty in the previously determined effective area of the gage plus a model of the measurement system which includes (as applicable) the uncertainties due to:

1) Mass loaded on the gage;

2) Local acceleration of gravity;

3) Local air density;

4) Density of the masses;

5) Thermal expansion coefficients of piston/cylinder materials;

6) Measured temperature;

7) Density of pressurizing fluid;

8) Height differential between reference levels of instruments; and

9) Other factors as appropriate.

b) The uncertainty in the effective area of a deadweight pressure gage under test calibrated by a pneumatic deadweight piston gage using the cross float method should be derived (and reported) from the uncertainty in the previously determined effective area of the pneumatic deadweight piston gage plus a model of the measurement system which includes (as applicable) the uncertainties due to:

1) Mass loaded on the gages;

2) Local acceleration of gravity;

3) Local air density;

4) Density of the masses;

5) Thermal expansion coefficients of piston/cylinder materials;

6) Measured temperatures;

7) Density of pressurizing fluid;

8) Height differential between reference levels of instruments; and

9) Other factors as appropriate.

c)   Due to the "balancing" nature of the cross float method, some of these uncertainties become relatively inconsequential, such as the local acceleration of gravity.

2.5.4 Pneumatic deadweight piston gage calibration (absolute mode)

2.5.4.1   Scope

This section contains specific technical criteria a laboratory engaged in pneumatic deadweight piston gage calibrations should follow if it is to be recognized as competent to: (1) use pneumatic deadweight piston gages in the absolute mode of operation to perform calibrations, or (2) certify pneumatic deadweight piston gages in the absolute mode to accuracy class.

2.5.4.2   References

a)   Heydemann, P. L. M., and Welch, B. E., *Piston Gages*, International Union of Pure and Applied Chemistry, Experimental Thermodynamics, Vol. II, Chapter 4, Part 3, pages 147-202, B. LeNeindre and B. Vodar, editors, Butterworths, London, 1975.

b)   *NCSL International Recommended Intrinsic/Derived Standards Practice (RISP) 4 on Deadweight Pressure Gauges*, NCSL International, Boulder, CO, 1998.

c)   Dadson, R. S., Lewis, S. L., and Peggs, G. N., *The Pressure Balance: Theory and Practice*, Her Majesty's Stationary Office, London, 1982.

2.5.4.3   Assuring the quality of test and calibration results

A pneumatic deadweight piston gage or a high-quality pressure transducer may be used as a process control check standard. The laboratory should have a manual outlining the procedures to be followed for the use and/or calibration of pneumatic deadweight piston gages in the absolute mode of operation.

2.5.4.4   Accommodation and environmental conditions

In addition to the general factors discussed in section 2.5.4.8, the following factors should be considered and may be significant when performing calibrations of pneumatic deadweight piston gages in the absolute mode of operation: leveling, reference level, operating position, bell jar reference pressure (vacuum), electrostatic charging, magnetization, system cleanliness, system leakage, spin time, and mass loading.

2.5.4.5   Equipment

a)   The laboratory should have the equipment necessary to perform calibrations of pressure measuring equipment using a pneumatic deadweight piston gage in the absolute mode of operation. Such equipment should include a clean pressurized gas source, a vacuum pump, and adequate plumbing and valves. For higher-quality measurements, thermometers, a rotational-speed indicator, and other related measuring equipment may be used. If dissimilar fluids are used, some means of fluid separation is required.

b)   When applicable, the laboratory should have the equipment necessary to perform calibrations of other deadweight pressure gages using the cross float method or other documented procedures. Such equipment should include a clean pressurized gas source, a vacuum pump, adequate plumbing and valves, and piston/cylinder position indicators. In addition, a means of determining balance conditions

should be provided, such as through the use of fall rate measurements, differential pressure cells, or equivalent. For higher-quality measurements, thermometers, a rotational-speed indicator, and other related measuring equipment may be used. If dissimilar fluids are used, some means of fluid separation is required.

2.5.4.6   Test and calibration methods and method validation

A manual outlining the procedures to be followed for all of the various uses of pneumatic deadweight piston gages in the absolute mode of operation by the calibration laboratory should be maintained. Such uses might include the calibration of pressure measuring equipment or other deadweight pressure gages. For calibrations involving assessment of accuracy classification, the procedure should name the accuracy classifications that are covered by the procedure.

2.5.4.7   Handling of test and calibration items

a)   Pneumatic deadweight piston gages should be cleaned where required and carefully handled. After assembly and pump-down of the piston/cylinder and associated parts, adequate time should be provided to ensure that they have come to thermal equilibrium within their vacuum environment. Specific guidelines for this process should be stated in the measurement procedure.

b)   When loading masses directly on pistons in pneumatic deadweight piston gages, care should be taken to avoid lateral forces that may bend or break the piston. Similar care should be taken when rotating the masses.

c)   Pistons/cylinders should be stored in a manner that prevents damage. Manufacturer's recommendations should be followed.

2.5.4.8   Reporting the results

a)   The uncertainty in the pressure generated and measured by a pneumatic deadweight piston gage in the absolute mode of operation should be derived (and reported) from the uncertainty in the previously determined effective area of the gage plus a model of the measurement system which includes (as applicable) the uncertainties due to:

1)   Mass loaded on the gage;

2)   Local acceleration of gravity;

3)   Bell jar pressure;

4)   Density of the masses;

5)   Thermal expansion coefficients of piston/cylinder materials;

6)   Measured temperature;

7)   Density of pressurizing fluid;

8)   Height differential between reference levels of instruments; and

9)   Other factors as appropriate.

b)   The uncertainty in the effective area of a deadweight pressure gage under test calibrated by a pneumatic deadweight piston gage in the absolute mode of operation using the cross float method should be derived (and reported) from the uncertainty in the previously determined effective area of the pneumatic deadweight piston gage plus a model of the measurement system which includes (as applicable) the uncertainties due to:

1)   Mass loaded on the gages;

2)   Local acceleration of gravity;

3)   Bell jar pressure;

4)   Density of the masses;

5)   Thermal expansion coefficients of piston/cylinder materials;

6)   Measured temperatures;

7)   Density of pressurizing fluid;

8)   Height differential between reference levels of instruments; and

9)   Other factors as appropriate.

c)   Due to the "balancing" nature of the cross float method, some of these uncertainties become relatively inconsequential, such as the local acceleration of gravity.

2.5.5   Hydraulic deadweight piston gage calibration

2.5.5.1   Scope

This section contains specific technical criteria a laboratory engaged in hydraulic deadweight piston gage calibrations should follow if it is to be recognized as competent to: (1) use hydraulic deadweight piston gages to perform calibrations, or (2) certify hydraulic deadweight piston gages to accuracy class.

2.5.5.2   References

a)   Heydemann, P. L. M., and Welch, B. E., *Piston Gages*, International Union of Pure and Applied Chemistry, Experimental Thermodynamics, Vol. II, Chapter 4, Part 3, pages 147-202, B. LeNeindre and B. Vodar, editors, Butterworths, London, 1975.

b)   *NCSL International Recommended Intrinsic/Derived Standards Practice (RISP) 4 on Deadweight Pressure Gauges*, NCSL International, Boulder, CO, 1998.

c)   Dadson, R. S., Lewis, S. L., and Peggs, G. N., *The Pressure Balance: Theory and Practice*, Her Majesty's Stationary Office, London, 1982.

2.5.5.3   Assuring the quality of test and calibration results

A hydraulic deadweight piston gage or a high-quality pressure transducer may be used as a process control check standard. The laboratory should have a manual outlining the procedures to be followed for the use and/or calibration of hydraulic deadweight piston gages.

2.5.5.4   Accommodation and environmental conditions

In addition to the general factors discussed in section 2.5.5.8, the following factors should be considered and may be significant when performing calibrations of hydraulic deadweight piston gages: leveling, reference level, operating position, air currents, electrostatic charging, magnetization, system cleanliness, system leakage, spin time, and mass loading.

2.5.5.5   Equipment

a)   The laboratory should have the equipment necessary to perform calibrations of pressure measuring equipment using a hydraulic deadweight piston gage. Such equipment should include a hydraulic pressure generating source and adequate plumbing and valves. For higher-quality measurements, thermometers, a barometer, a hygrometer, rotational-speed indicator, and other related measuring equipment may be used. If dissimilar fluids are used, some means of fluid separation is required.

b)   When applicable, the laboratory should have the equipment necessary to perform calibrations of other deadweight pressure gages using the cross float method or other documented procedures. Such equipment should include a hydraulic pressure generating source, adequate plumbing and valves, and piston/cylinder position indicators. In addition, a means of determining balance conditions should be provided, such as through the use of fall rate measurements, differential pressure cells, or equivalent. For higher-quality measurements, thermometers, a barometer, a hygrometer, rotational-speed indicator, and other related measuring equipment may be used. If dissimilar fluids are used, some means of fluid separation is required.

2.5.5.6   Test and calibration methods and method validation

A manual outlining the procedures to be followed for all of the various uses of hydraulic deadweight piston gages by the calibration laboratory should be maintained. Such uses might include the calibration of pressure measuring equipment or other deadweight pressure gages. For calibrations involving assessment of accuracy classification, the procedure should name the accuracy classifications that are covered by the procedure.

2.5.5.7   Handling of test and calibration items

a)   Hydraulic deadweight piston gages should be cleaned where required and carefully handled. After assembly of the piston/cylinder and associated parts, adequate time should be provided to ensure that they have come to thermal equilibrium with their environment. Specific guidelines for this process should be stated in the measurement procedure.

b)   When loading masses directly on pistons in hydraulic deadweight piston gages, care should be taken to avoid lateral forces that may bend or break the piston. Similar care should be taken when hand-rotating the masses.

c)   Pistons/cylinders should be stored in a manner that prevents damage. Manufacturer's recommendations should be followed.

2.5.5.8   Reporting the results

a)   The uncertainty in the pressure generated and measured by a hydraulic deadweight piston gage should be derived (and reported) from the uncertainty in the previously determined effective area of the gage plus a model of the measurement system which includes (as applicable) the uncertainties due to:

1)   Mass loaded on the gage;

2)   Local acceleration of gravity;

3)   Local air density;

4)   Density of the masses;

5)   Thermal expansion coefficients of piston/cylinder materials;

6)   Measured temperature;

7)   Density of pressurizing fluid;

8)   Height differential between reference levels of instruments; and

9)   Other factors as appropriate.

b)   The uncertainty in the effective area of a deadweight pressure gage under test calibrated by a hydraulic deadweight piston gage using the cross float method should be derived (and reported) from the uncertainty in the previously determined effective area of the hydraulic deadweight piston gage plus a model of the measurement system which includes (as applicable) the uncertainties due to:

1)   Mass loaded on the gages;

2)   Local acceleration of gravity;

3)   Local air density;

4)   Density of the masses;

5)   Thermal expansion coefficients of piston/cylinder materials;

6)   Measured temperatures;

7)   Density of pressurizing fluid;

8)   Height differential between reference levels of instruments; and

9)   Other factors as appropriate.

c)   Due to the "balancing" nature of the cross float method, some of these uncertainties become relatively inconsequential, such as the local acceleration of gravity.

2.5.6 Pneumatic deadweight ball gage calibration

2.5.6.1   Scope

This section contains specific technical criteria that a laboratory engaged in pneumatic deadweight ball gage calibrations should follow if it is to be recognized as competent to:

a)   Use pneumatic deadweight ball gages to perform calibrations, or

b)   Certify pneumatic deadweight ball gages to accuracy class.

2.5.6.2   References

a)   Heydemann, P. L. M., and Welch, B. E., *Piston Gages*, International Union of Pure and Applied Chemistry, Experimental Thermodynamics, Vol. II, Chapter 4, Part 3, pages 147-202, B. LeNeindre and B. Vodar, editors, Butterworths, London, 1975.

b)   *NCSL International Recommended Intrinsic/Derived Standards Practice (RISP) 4 on Deadweight Pressure Gauges*, NCSL International, Boulder, CO, 1998.

2.5.6.3   Assuring the quality of test and calibration results

A pneumatic deadweight ball gage, a pneumatic deadweight piston gage or a high-quality pressure transducer may be used as a process control check standard. The laboratory should have a manual outlining the procedures to be followed for the use and/or calibration of pneumatic deadweight ball gages.

2.5.6.4   Accommodation and environmental conditions

In addition to the general factors discussed in section 2.5.6.8, the following factors should be considered and may be significant when performing calibrations of pneumatic deadweight ball gages: leveling, reference level, air currents, electrostatic charging, magnetization, system cleanliness, system leakage, rotary motion, and mass loading.

2.5.6.5   Equipment

a)   The laboratory should have the equipment necessary to perform calibrations of pressure measuring equipment using a pneumatic deadweight ball gage. Such equipment should include a clean pressurized gas source adequate for the operation of the gage, and adequate plumbing and valves. For higher-quality measurements, thermometers, a barometer, a hygrometer, and other related measuring equipment may be used. If dissimilar fluids are used, some means of fluid separation is required.

b)   When applicable, the laboratory should have the equipment necessary to perform calibrations of other deadweight pressure gages using the cross float method or other documented procedures. Such equipment should include a clean pressurized gas source and adequate plumbing and valves. A piston/cylinder position indicator should be used, when appropriate. In addition, a means of determining balance conditions should be provided, such as through the use differential pressure cells, or equivalent. For higher-quality measurements, thermometers, a barometer, a hygrometer, and other related measuring equipment may be used. If dissimilar fluids are used, some means of fluid separation is required.

2.5.6.6   Test and calibration method and method validation

A manual outlining the procedures to be followed for all of the various uses of pneumatic deadweight ball gages by the calibration laboratory should be maintained. Such uses might include the calibration of pressure measuring equipment or other deadweight pressure gages. For calibrations involving assessment of accuracy classification, the procedure should name the accuracy classifications that are covered by the procedure.

2.5.6.7   Handling of test and calibration items

a)   Pneumatic deadweight ball gages should be cleaned where required and carefully handled. After assembly of the ball/nozzle and associated parts, adequate time should be provided to ensure that they have come to thermal equilibrium with their environment. Specific guidelines for this process should be stated in the measurement procedure.

b)   When loading masses on pneumatic deadweight ball gages, care should be taken to avoid lateral forces that may damage the ball or nozzle. In addition, the weights should never be spun unless the ball is floating.

c)   The gage should be stored in a manner that prevents damage. Manufacturer's recommendations should be followed.

2.5.6.8   Reporting the results

a)   The uncertainty in the pressure generated and measured by a pneumatic deadweight ball gage should be derived (and reported) from the uncertainty in the previously determined effective area of the gage plus a model of the measurement system which includes (as applicable) the uncertainties due to:

    1)   Mass loaded on the gage;

    2)   Local acceleration of gravity;

    3)   Local air density;

    4)   Density of the masses;

    5)   Thermal expansion coefficients of ball/nozzle materials;

    6)   Measured temperature;

    7)   Density of pressurizing fluid;

    8)   Height differential between reference levels of instruments;

    9)   Flow rate through the nozzle; and

    10)   Other factors as appropriate.

b)   The uncertainty in the effective area of a deadweight pressure gage under test calibrated by a pneumatic deadweight ball gage using the cross float method should be derived and reported from the

uncertainty in the previously determined effective area of the pneumatic deadweight ball gage plus a model of the measurement system which includes (as applicable) the uncertainties due to:

1) Mass loaded on the gages;

2) Local acceleration of gravity;

3) Local air density;

4) Density of the masses;

5) Thermal expansion coefficients of ball/nozzle materials;

6) Measured temperatures;

7) Density of pressurizing fluid;

8) Height differential between reference levels of instruments; and

9) Other factors as appropriate.

c)   Due to the "balancing" nature of the cross float method, some of these uncertainties become relatively inconsequential, such as the local acceleration of gravity.

2.5.7   Pressure transducer calibrations

2.5.7.1   Scope

This section contains specific technical criteria a laboratory engaged in pressure transducer calibrations should follow if it is to be recognized as competent to: (1) use pressure transducers as standards to calibrate other pressure measuring equipment, or (2) certify transducers to accuracy class.

2.5.7.2   References

a)   ANSI B40.1 - 1991, *Gauges, Pressure, Indicating Dial Type - Elastic Element.*

b)   ISA-S37.3, 1982, reaffirmed 1995, *Specifications and Tests for Strain Gage Pressure Transducers.*

c)   ASME/ANSI Performance Test Code 19.2-1987, reaffirmed 1998, *Pressure Measurement, Instruments and Apparatus, Supplement on Instruments and Apparatus, Part 2.*

2.5.7.3   Assuring the quality of test and calibration results

A high-quality pressure transducer may be used as a process control check standard. The laboratory should have a manual outlining the procedures to be followed for the use and/or calibration of all pressure transducers used in the laboratory, including all different modes of operation and operating fluids that are used for each transducer type.

2.5.7.4 Accommodation and environmental conditions

In addition to the general factors discussed in section 2.5.7.8, the following should be considered and may be significant when performing calibrations of pressure transducers: proper electronic and mechanical setup and other influences which may affect the performance of the transducer such as wiring, hysteresis, orientation, system cleanliness, system leakage, etc.

2.5.7.5 Equipment

The laboratory should have the equipment necessary to perform calibrations of pressure measuring equipment using a pressure transducer. Such equipment should include an appropriate fluid source, pressure generator, adequate plumbing and valves, and readout. For higher-quality measurements, thermometers, a barometer, regulated electronic equipment and other related measuring equipment may be used. If dissimilar fluids are used, some means of fluid separation is required.

2.5.7.6 Test and calibration methods and method validation

A manual outlining the procedures to be followed for all of the various types and uses of pressure transducers in the calibration laboratory should be maintained. Such uses might include the calibration of other pressure transducers or pressure measuring equipment. For calibrations involving assessment of accuracy classification, the procedure should name the accuracy classifications that are covered by the procedure.

2.5.7.7 Handling of test and calibration items

Pressure transducers should be kept clean and carefully handled. After initial setup, adequate time should be provided to ensure that the system has come to thermal equilibrium with the environment. Specific guidelines for this process should be stated in the measurement procedure. Manufacturer's recommendations should be followed.

2.5.7.8 Reporting the results

a) The uncertainty in the calibration of the pressure measuring equipment under test should be derived (and reported) from the uncertainty in the previously determined calibration of the standard pressure transducer which includes (as applicable) the uncertainties due to:

1) Zero drift or shift;

2) Pressure hysteresis;

3) Temperature hysteresis;

4) Short and long term stability;

5) Full scale drift;

6) Attitude dependence;

7) Warm-up time;

8) Supply-voltage dependence;

9) Resolution and sensitivity;

10) Height differential between reference levels of instruments; and

11) Other factors as appropriate.

b) The final uncertainty of the calibration of the pressure measuring equipment under test will include the type A uncertainties from the random effects of the calibration and also the type B uncertainty from the standard pressure transducer.

c) The pressure measuring equipment under test should be operated according to, and perform as specified in, the manufacturer's operation manual.

2.6 Calibration of ionization gages from (10^{-7} to 10^{-1}) Pa using a calibrated ionization gage reference standard

2.6.1 Scope

This section contains specific criteria for the equipment and procedures used for the calibration of hot-cathode or cold-cathode ionization vacuum gages using a calibrated hot-cathode ionization gage as a reference standard over the nominal range of (10^{-7} to 10^{-1}) Pa.

2.6.2 Accommodation and environmental conditions

A "normal" laboratory environment is adequate. Temperature should not vary by more than ±1 °C and a clean workspace should be provided for equipment preparation and assembly.

2.6.3 Handling of test and calibration items

2.6.3.1 It should be kept in mind at all times that the calibration system and vacuum sensing elements (tubes) are very sensitive to contamination and that surfaces exposed to the vacuum should not be touched without wearing clean plastic gloves.

2.6.3.2 Exposure of calibration items and parts to oil vapor (e.g., exhaust from mechanical vacuum pumps) and industrial atmospheres should be avoided. If vacuum surfaces are contaminated they should be cleaned by washing with a water and alkaline detergent and thoroughly rinsed with deionized water. Drying can be accelerated by rinsing with reagent-grade ethyl alcohol.

2.6.3.3 Mild contamination (e.g., fingerprints) should be removed by swabbing and/or rinsing with reagent-grade acetone and/or alcohol.

2.6.4 Equipment

2.6.4.1 Materials of construction

The calibration chamber, high vacuum pumping system, and gas inlet and control systems should be of metal construction with no elastomer tubing. For calibration pressures above 10^{-5} Pa, valves with elastomer bonnet and poppet seals can be used. For lower pressures, the components exposed to sub-atmospheric pressures,

including the relevant gas inlet and control components, should be bakeable and of all-metal (stainless steel, copper, and/or aluminum, no brass) construction. In general good ultra high vacuum (UHV) practices should be followed.

2.6.4.2   Design

a)   The calibration chamber should be large enough to accommodate mounting ports for the test gage(s), reference standard(s), and at least one check standard. The chamber can be spherical or cylindrical in design, or assembled from standard UHV components. However, the components should have a minimum diameter of 35 mm, and the length to diameter ratio should not exceed 5. The calibration gas inlet should be downstream from the gages and directed so that molecules cannot go directly from the gas inlet to any of the gages. Provision should be made for temperature equilibration of calibration gas with chamber, i.e., lots of molecular collisions with the walls. A baffle at the inlet works well. "O" ring compression type seals are allowed for the mounting of test gages for pressures down to 10^{-4} Pa. However, metal-gasketed Conflat[1]-type seals are required for the reference and check standards, and are required for the test gages as well, for pressures below 10^{-4} Pa (as a general rule, all-metal flanges are preferred for all pressures). For pressures below 10^{-4} Pa, the chamber should be equipped for baking at 100 °C. For pressures below 10^{-5} Pa the chamber and vacuum appendices, including all gages, should be equipped to be baked at 200 °C.

b)   The gas inlet system should provide a supply of clean gas at the rates required to cover the entire range of calibration pressures. At any given pressure, the flow of calibration gas should be stable to within 1 % over a period of 15 min, or the time required to obtain a complete set of calibration data, including data for the reference and check standard, and test gage(s), whichever is longer. Again, the connection to the calibration chamber should be all-metal downstream from the flow-controlling valve. Gas reservoirs, ballasts, or storage volumes that are part of the flow/pressure control system should not contain elastomer tubing or vessels, although "O" ring seals can be used in valves and fittings upstream of the flow-control valve.

c)   The calibration chamber pump should meet the following criteria: The pump should be a clean UHV-type pump such as a

- Turbomolecular pump,
- Well baffled and trapped diffusion pump,
- Cryocondensation or cryoabsorption pump, or
- Ion pump.

These pump types are listed in the descending order of general preference for calibration purposes; however, specific conditions or cost factors may dictate the choice. The pump should be capable of evacuating the chamber, with all gages mounted, to a pressure a factor of 10 lower than the lowest calibration pressure (with baking if necessary). The combined pressure instabilities in the calibration chamber due to the pump and the gas supply should not exceed 2 % over 15 min or the time to take all necessary data at a particular pressure. The pressure stability can be improved by restricting the conductance between the pump and calibration chamber proper. If so, the pumping speed in the calibration chamber should still be adequate to maintain the base pressure a factor of 10 below the lowest calibration pressure, and it will be desirable to include provision for bypassing the restriction so that full pumping speed is available for initial pumpdown.

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[1]Brand names cited here are used for identification purposes and do not constitute an endorsement by NIST.

2.6.4.3   Reference standard

a)   The reference standard(s) should be a calibrated Bayard/Alpert[2]-type, hot-cathode ionization gage, and its associated controller or equivalent. The gage should be mounted to the calibration chamber with a Conflat-type seal, welded to a glass-to-metal transition if appropriate. In general, glass-tubulated gages give the most linear performance for pressures above 10^{-3} Pa and tungsten filaments give the most stable performance except for reactive gases such as water.

b)   The reference gage controller should control the emission current to within $\pm$ 1 % and maintain filament and grid bias voltages constant to within $\pm$ 1 V. Corrections should be made for any emission current changes that exceed 1 %. If glass-tubulated gage tubes are used, it is recommended that noise-free dc filament-heating current be used; filament currents with unfiltered spikes can cause pressure-dependent changes in the gage sensitivity. The ion current measuring circuit should be of the feedback type and maintain the collector within 100 mV of the system reference potential (generally ground). Corrections should be made for the input impedance of the voltmeter used to verify this potential (generally, an input impedance of 10^8 Ω or more is adequate). The ion current measuring circuitry should be separately calibrated against a current standard over the range corresponding to the pressure calibration range. This calibration should be periodically checked to verify that significant errors are not introduced by changes in the circuitry since the last vacuum calibration of the reference standard.

c)   The reference gage(s) should be calibrated at three or more approximately evenly spaced points per decade, with at least one repeat on separate days. The imprecision of the reference gage readings should be evaluated from the calibration data.

2.6.5 Test and calibration methods and method validation

2.6.5.1   Gage handling and mounting

a)   As noted before, the interior surfaces of the vacuum components and all sealing areas should be protected from mechanical damage and contamination. As a general rule, it is preferable that the reference and check standard gages remain mounted on the calibration system, unless this would expose them to damage or contamination. It is also advisable to retain an additional check standard that is not normally operated on the calibration system, but is used only to resolve apparent discrepancies between the reference and check standards.

b)   The test gages should be mounted on the calibration system and evacuated. Leak tightness should be verified by helium leak testing with a sensitivity of 10^{-13} mol/s (10^{-9} std cc/s) and/or the achievement of a pressure two orders of magnitude below the lowest calibration pressure. Particular attention should be paid to the leak integrity of the gage mounting seals; not only are these one of the more likely sources of leaks, if they do leak they can cause a significant local pressure increase in the gage. This local pressure increase can be a significant problem with "O" ring compression seals.

c)   Wherever possible, ion gages should be mounted so that there is no direct line-of-sight communication between any two gages. This can be achieved by the design of the chamber, or by mounting the gages with an intervening 4cm (1 ½ in) elbow or tee so that any ion exiting from a gage will strike a grounded metal surface before it reaches another gage. In cases where it is not possible to prevent line-of-sight communication, the effects of this should be evaluated by operating both gages at a stable

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[2]Brand names cited here are used for identification purposes and do not constitute an endorsement by NIST.

pressure at least two orders of magnitude higher than the base pressure, establishing equilibrium readings, and then turning off first one gage and then the other while monitoring the operating gage to check for changes in its reading. Typically, some fraction or all of an hour may be required to complete this check as there will be an initial temperature-induced transient when a gage is turned off or on. The percentage change in the reading of the operating gage should be included as an additional error source and the magnitude should be added to the rest of the calibration uncertainty.

d)   This same type of test should be conducted for cold-cathode gages, even if there is no line-of-sight communication, to check for gage pumping effects. In this case it is only necessary to cycle the cold-cathode gage on and off while monitoring the reference standard.

2.6.5.2   Gage outgassing

The gages can be effectively outgassed by operating them at normal emission currents while the calibration system and gages are baked. If the system is not baked, or the baking system does not effectively heat the gages, glass-tubulated gages can be baked and outgassed by wrapping the tube in fiberglass insulation and operating them for a period of at least 4 h. If resistive or electron bombardment heating of the grid is employed, the duration of this heating should not exceed the manufacturer's recommendation. Following outgassing of the grid, at least 8 h should be allowed before calibration data are taken.

2.6.5.3   Gage controller operation

a)   Before taking calibration data, the controllers for all hot-cathode gages should be checked, if possible, as follows: With the gage in normal operation, the grid and filament dc biases should be measured with a voltmeter accurate to within 0.1 V and recorded. The filament bias should be measured at each end of the filament and the average value noted for inclusion in the report. If the controller has a mechanical meter readout for the collector current or equivalent pressure, the mechanical offset should be determined by disconnecting the collector current input (do not short the input, leave it open-circuited) and switching the controller to the highest pressure or current range. If the meter is equipped with an adjustment mechanism, it should be adjusted to read zero to within the readability of the gage. Otherwise, the offset should be noted. Any electrical offset should then be determined by switching to the lowest or most sensitive pressure or current range (leave the collector current input open-circuited). If there is provision for an electrical zero, the readout should be adjusted to zero to within the reading tolerance. If not so equipped (as is generally the case), the zero offset of the test-gage controller should be noted for inclusion in the report. The offset of the reference- and check-standard controllers should be noted and compared with previous values. If the offset is 1 % or more of the reading at the lowest calibration pressure, the subsequent calibration readings should be corrected for the offset. The electron emission current can be measured by placing a 1000 Ω resistor, calibrated with an uncertainty no greater than 1 Ω, in series with the grid connection, and measuring the dc voltage across the resistor with a voltmeter with an uncertainty that does not exceed 0.1 V.

CAUTION: Note that the voltmeter will be exposed to the grid bias voltage, typically 150 V to 200 V, as a common mode voltage.

b)   With the exception of the zero offset readings, these same measurements should be repeated with the gage and controllers operating at the highest calibration pressure. The results should again be recorded for use in the reports.

2.6.5.4   Data taking procedures

a)   Following baking (if required) and outgassing of the gages, the system base pressure should be monitored to verify that it does not change by more than 10 % of the lowest calibration pressure over the course of an hour.

b)   As a general rule, the most consistent results are obtained by starting at the lowest pressures and proceeding to higher pressures. It is useful to check for by obtaining additional calibration data with a sequence of decreasing pressure points, however, reliable results should not be expected for pressures within two orders of magnitude of the base pressure.

c)   Once a calibration pressure is established, the reference standard should be monitored to verify pressure stability. At a minimum, the pressure should change in a monotonic manner, with the change not exceeding 1 % over the period required to obtain the necessary calibration data for all gages. At each calibration point, all gages should be read at least twice in sequence, starting and ending with the reference standard. If the reference standard readings do not repeat to within the greater of 1 % or the readability of the standard, the test should be repeated. If the reference standard readings persistently do not repeat to within the required tolerance, but do indicate a constant monotonic drift in the pressure, calibration data can be obtained from the average of two readings taken in a sequence so that for all gages the first and second reading are equally spaced about the same point in time, e.g., if three gages, A B and C, are to be read, they should be read in an evenly spaced sequence: A B C C B A. If the readings for any gage do not repeat to within the readability of the gage, or are not consistent with the pressure drift indicated by the reference standard, additional data should be taken to determine if any of the readings are anomalous and to determine representative random errors.

d)   Data should be obtained for at least three pressures per decade, located to within ± 20 % of either an evenly spaced logarithmic or evenly spaced arithmetic sequence.

2.6.6 Reporting the results

The calibration report should include complete identification of the reference standard and test gage, model and serial numbers, and any identification numbers associated with the gage tubes and controllers.

2.7   Calibration of vacuum gages from 10^{-5} Pa to 1 Pa using the spinning rotor gage (SRG) as a reference standard for direct comparison

2.7.1 Introduction

This section establishes the criteria used to assess the competence of calibration laboratories in the area of vacuum calibrations using a spinning rotor gage for comparison calibrations in the pressure range 10^{-5} Pa to 1 Pa.

2.7.2 Background

A given laboratory involved in the calibration of vacuum gages using a spinning rotor gage can rely on a number of different designs of the calibration system, depending on the calibration load, accuracy and pressure range over which calibrations are to be performed. The demands on vacuum system performance are nowhere so demanding as in the calibration of vacuum gages. To obtain acceptable levels of accuracy,

care should be taken in the design and maintenance of the calibration system and associated reference and check standards.

2.7.3 Scope

This section contains specific technical criteria for a laboratory engaged in calibrations of vacuum gages within the pressure range from 10^{-5} Pa to 1 Pa (133.322 Pa = 1 Torr) using the Spinning Rotor Gage (SRG) as a reference standard for direct comparison. The gages to be calibrated currently included in the accreditation program are:

a) Spinning rotor gages;

b) Cold-cathode ionization gages; and

c) Hot-cathode ionization gages.

2.7.4 References

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b) Setina, J., and Looney, J. P., "Behavior of Commercial Spinning Rotor Gages in the Transition Regime," *Vacuum*, **44**, (1993), 577.

c) Dittmann, S., Lindenau, B. E., and Tilford, C. R., "The molecular drag gage as a calibration standard," *J. Vac Sci. Technol.*, **A 7** (1989), 3356.

d) McCulloh, K. E., Wood, S. D., and Tilford, C. R., "The zero stability of spinning rotor vacuum gages," *J. Vac Sci. Technol.*, **A 3** (1985), 1736.

e) *Vacuum calibrations using the spinning rotor gage*, Available from NIST Pressure and Vacuum Group, Building 220, Room A55, Gaithersburg, MD 20899.

f) Tilford, C. R., "Pressure and Vacuum Measurements," Chapter 2 in *Physical Methods of Chemistry, 2nd Ed. Volume 6: Determination of Thermodynamic Properties*, Rossiter, B. W., and Beatzold, R. C., ed., John Wiley & Sons, New York, (1992).

g) Sasaki, Y. T., "A survey of vacuum material cleaning procedures," *J. Vac Sci. Technol.*, **A 9** (1991), 2025.

h) Berman, A., *Total Pressure Measurements in Vacuum Technology*, Academic Press, NY (1985).

i) O'Hanlon, J. F., *A user's guide to vacuum technology*, Wiley Interscience, NY (1989).

2.7.5 Accommodation and environmental conditions

2.7.5.1 The temperature in the calibration area should be nominally equivalent to the temperature of the environment in which the test artifact will be used, with a maximum allowable rate of change and maximum temperature gradients in the calibration area depending on the uncertainty level needed for calibration. The

uncertainties due to temperature measurement, temperature rate of change and temperature variations across the calibration system should be calculated and added to the total uncertainty of the calibration.

2.7.5.2   The relative humidity in the calibration room should not exceed 50 %.

2.7.5.3   Excessive vibration should be avoided in the calibration area. If an obvious source of vibration does exist, proper precautions should be taken to prevent adverse effects on the laboratory's measurements.

2.7.5.4   Excessive electromagnetic fields should be avoided in the calibration area. Obvious sources of excessive electromagnetic interferences should be eliminated or proper precautions (i.e., shielding) taken to prevent adverse effects on the laboratory measurements.

2.7.6 Equipment

2.7.6.1   The calibration system should consist of an all-metal (stainless steel, aluminum) high-vacuum chamber with test ports to which the SRG and the test gages are attached, a vacuum pumping sub-system and a sub-system for introducing test gas to the calibration chamber. The chamber and associated sub-systems should be of all-metal (stainless steel, aluminum and/or copper, no brass) construction, with no elastomer tubing. Valves with elastomer bonnet and poppet seals can be used. In general, good UHV practices should be followed.

2.7.6.2   The calibration chamber should be large enough to accommodate mounting ports for the test gage(s), reference standard(s), and at least one check standard (see Sec. 2.7.6.6). The chamber can be spherical or cylindrical in design, or assembled from standard UHV components. The components should have a minimum diameter of 35 mm, and the length to diameter ratio should not exceed 5. The calibration inlet should be downstream from the gages and directed so that molecules cannot go directly from the gas inlet to any of the test ports. "O" ring compression type seals are allowed (but not preferred) for the mounting of the test gages for calibration pressures down to 10^{-4} Pa. The reference and check standards should be attached using metal-gasketed, bakeable seals. As a general rule, metal-gasketed seals are preferred for all pressures. For calibration pressures at or below 10^{-4} Pa, the chamber should be equipped for baking to 100 °C.

2.7.6.3   The vacuum pumping sub-system should consist of a clean, UHV pump which has a time-independent pumping speed, e.g., turbomolecular or diffusion pump, which is backed by a mechanical displacement forepump. In descending order of preference the pumping systems which are acceptable are: turbo-molecular pump, well trapped and baffled diffusion pump, cryocondensation pump, and ion pump. The laboratory should demonstrate that the pumping system (via the use of a hot cathode ionization gage) is of suitable capacity to reduce the vacuum chamber to below 10^{-6} Pa to allow for proper determination of the SRG residual drag. The pumping system should be of suitable capacity to continuously exhaust the test gas without extreme changes in the effective pumping speed. Diffusion pumps should be properly cold trapped and baffled to minimize pump fluid backstreaming.

2.7.6.4   The calibration chamber should be of such a design that ensures the pressure at each of the test ports is equal, and that the direction of the gas molecules be randomly distributed. For calibration pressures above 0.1 Pa, the calibration system could be either a static pressure or dynamic expander (continuous-flow) type calibration system. For calibration pressures below 0.1 Pa, the test calibration system should be of the continuous-flow design. If static pressure systems are to be used, the laboratory should determine the outgassing rate of the vacuum chamber. The uncertainty due to changes in the pressure from outgassing should be calculated and added to the total uncertainty of the calibration. The combined pressure instabilities in the calibration chamber due to pump speed variations and fluctuations in the gas supply flow should not

exceed 2 % over 15 min, or the time necessary to obtain all of the calibration data, whichever is longer. The pressure stability can be improved by restricting the conductance between the pump and the calibration chamber by the use of an orifice. If so, the pump speed to the chamber should still be adequate to maintain a base pressure of 10^{-6} Pa. It is also desirable that the design include provision for bypassing the pump speed restriction so that full pump speed is available for initial exhaust and pump down.

2.7.6.5   The gas inlet sub-system should be of such a design as to provide a stable flow of clean gas to the calibration chamber. At any given pressure, the flow of calibration gas should be stable to within 1 % over a period of 15 min, or the time required to obtain a complete set of calibration data, including data from the Reference and Check Standards, whichever is longer. The connection to the calibration chamber should be all-metal downstream from the flow control valve. The use of piezoelectric valves for flow control should be avoided.

2.7.6.6   The laboratory should maintain at least one other internal reference or check standard SRG at all times for quality control.

2.7.6.7   The laboratory should have the temperature measuring capability suitable to the calibration procedure. A temperature measurement accuracy of 0.1 K should prove sufficient.

2.7.6.8   The laboratory should have a 50x laboratory microscope available to periodically inspect the surface of secondary standard SRG to monitor for changes in surface condition.

2.7.6.9   The laboratory should have the necessary equipment (oscilloscope or 100 kHz spectrum analyzer) to monitor the SRG pick-up signal for excessive noise and unwanted interferences.

2.7.6.10   The laboratory should have the capability of baking the calibration system to at least 100 °C.

2.7.6.11   The laboratory should have the necessary test equipment to monitor and ensure the vacuum system's integrity and cleanliness, and ensure that the ultimate pressure of the system is low enough to accurately determine the SRG residual drag or offset. This equipment must include at least one hot cathode ionization gage whose sensitivity has been determined by calibration against the reference standard SRG, and a helium leak detector or residual gas analyzer. If a helium leak detector is to be used, it should have a minimum sensitivity of 10^{-13} mol/s (10^{-9} std cc/s).

2.7.6.12   The laboratory should have the necessary equipment to clean and handle the vacuum components. The laboratory should have a documented and detailed procedure for cleaning vacuum components.

2.7.7 Test and calibration methods and method validation

2.7.7.1   Generic concerns

a)   The major factor in the calibration of vacuum gages is the design of the calibration system. The calibration system should be designed, assembled, and tested in accordance with accepted practice, and constructed using suitable materials.

b)   A major factor in the calibration of vacuum instrumentation is the vacuum system cleanliness and integrity. The vacuum tightness should be tested prior to the start of every calibration.

c)   The vacuum system should be demonstrated to be leak free and have a suitably low outgassing rate which does not affect gage calibration.

d)   The calibration system should avoid the use of "O" ring or elastomeric seals for attaching test or artifact standard gage, except that "O" ring compression type seals are allowed (but not preferred) for the mounting of the test gages for calibration pressures down to 10^{-4} Pa. The calibration system should avoid the use of butterfly-type isolation valves for the test gages.

2.7.7.2   Spinning rotor gages

a)   The calibration of the SRG should be recognized as a property of the rotor itself and not of the suspension head or gage thimble.

b)   A major factor which affects the accuracy of gage calibrations using a Spinning Rotor Gage is the stability of the residual drag. The residual drag for the SRG should be monitored continuously prior to, during, and after the calibration tests. The residual drag should be determined with the vacuum chamber configured as it will be used at the time of test (i.e., pump restriction, if any, in place, valve to the flow lines open, but flow control valve closed). The period of monitoring should be a minimum of 24 h prior to the start of test. The average value of the residual drag and its dependence upon the rotational frequency of the rotor should be determined and properly accounted for in subsequent data analysis. The residual drag should be monitored, and uncertainties in the calibration due to the instability of the residual drag should be computed, and added to the total uncertainty of the calibration.

c)   Another factor which affects the accuracy of the SRG is the condition of the rotor's surface. The surface condition should be periodically monitored by visual inspection. The SRG should be recalibrated upon loss of the magnetic suspension while the rotor is spinning.

d)   The laboratory should conduct periodic comparison tests with a least one check or control standard to maintain quality control. Recalibration of the standard SRG should be performed at NIST or other accredited laboratory when shifts greater than 2 % in the control gage to standard SRG reading ratio at 10^{-2} Pa are observed, if these cannot explicitly be attributed to a change in the control gage's calibration.

2.7.7.3   Hot-cathode ionization gages

a)   Ionization gages should be calibrated as a complete system consisting of the gage tube or gage head, cables, and the ionization gage controller. The identification number of the gage tube should be engraved on the gage flange or painted onto the gage tube.

b)   A factor in the calibration of hot-cathode ionization gages is careful placement on the calibration system. No two ionization gages should be mounted in a manner whereby two gages could be "looking" directly at one another as gage interactions can effect calibration accuracy. The calibration laboratory should test the system design for gage interaction effects.

c)   The ionization gage controller should control the emission current to within ± 10 %, and maintain the filament and grid bias voltages constant to within ± 1 V. Corrections should be made for any changes in the emission current changes which exceed 1 %. Emission currents should not exceed 1 mA, except for electron bombardment degassing, if used. If glass-tubulated gages are used, it is recommended that a dc heating current be used; filament currents with unfiltered negative-going spikes can cause pressure-dependent changes in the gage sensitivity. The ion current measuring circuit should be of the feedback type, and maintain the collector within 100 mV of system reference potential (usually

ground). The ion current measuring circuitry should be separately calibrated against a current standard over the range of calibration to check for changes in the controller electronics with time.

d)   Hot-cathode ionization gages should be calibrated at three or more, approximately evenly spaced points per decade, with at least one repeat calibration on separate days. The short -term or long-term uncertainty, including uncertainty due to finite resolution from the controller display, should be evaluated from the calibration data. Calibration data should be obtained over the entire range for which the calibration certificate is to be issued.

e)   Before calibration data is taken, the controllers for all hot-cathode gages should be checked for normal operation of the grid and filament bias voltages. The bias voltages should be measured and recorded with a voltmeter accurate to 0.1 V.

f)   Before calibration, the electrical zero of the controller should be checked by switching to the lowest pressure or most sensitive setting and disconnecting the collector current input, i.e., open circuited (do not short the input).

g)   For ion gage controllers with mechanical output meters, the mechanical offset should be determined by disconnecting the collector current input (i.e., open circuit operation) and switching the controller to the highest pressure or current range (i.e., lowest sensitivity scale) and recording the meter reading. If the meter is equipped with an adjustment mechanism, it should be adjusted to read zero.

h)   Before the introduction of the calibration gas, the pressure indication at base vacuum (with the system configured as it will be used for calibration) should be recorded.

i)   The electron emission should be measured and recorded by placing a 1000 Ω resistor, calibrated with a 95 % level uncertainty of no greater than 1 Ω, in series with the grid connection and measuring the dc voltage across the resistor with a floating potential or battery operated voltmeter with an uncertainty that does not exceed 0.1 V. Care should be taken at this point as the voltmeter will be exposed to the grid bias voltage, typically 150 V to 200 V, common mode voltage.

2.7.7.4   Cold-cathode ionization gages

a)   Cold-cathode ionization gages should be calibrated as a complete system consisting of the gage head, cable and gage controller.

b)   A factor in the calibration of cold-cathode ionization gages is the effect of the (potentially) large pumping speed of cold-cathode gages. Gage calibrations should be conducted in a manner whereby gage pumping effects will not adversely affect the gage calibration.

c)   A factor in the calibration of cold-cathode ionization gages is careful placement on the calibration system. No two ionization gages should be mounted in a manner whereby two gages could be "looking" directly at one another as gage interactions can effect calibration accuracy. The calibration laboratory should test the system design for gage interaction effects.

d)   Cold-cathode ionization gages should be calibrated at five or more approximately evenly spaced points per decade, with at least two repeat calibrations on separate days. The imprecision, including imprecision due to finite resolution from the controller display, should be evaluated from the calibration data.

2.7.8 Handling of test and calibration items

2.7.8.1 Reference and check standard gages, as well as the test gages should be clean and free from particulate, oil or other contamination.

2.7.8.2 Care should be taken when handling the SRG rotor as finger acids and oils can cause changes in the surface characteristics. The rotor should not be touched without clean plastic gloves and/or plastic tweezers.

2.7.9 Records

2.7.9.1 The uncertainty should be derived and reported from a suitable model of the measurement system which includes uncertainties from:

a) Uncertainty in the reference standard SRG calibration;

b) Uncertainty in the reference standard SRG residual drag determination and its frequency dependence;

c) Uncertainty in the measured temperature and the effect of thermal gradients across the calibration system and thermal drifts during the course of the calibration data;

d) Random error in the Reference Standard SRG readings due to environmental effects;

e) Random reading errors in the test gage; and

f) Other factors as they may apply.

2.7.9.2 Supporting documentation, calculations and appropriate test data, which substantiate the uncertainty model, should be maintained by the calibration laboratory and made available for inspection.

2.7.9.3 Records of data (temperature, residual drag measurements, etc.), which are used to determine the calibration uncertainty, should be maintained and made available for inspection.

2.7.10 Reporting the results

The calibration report should include complete identification of the reference standard and test gage, model and serial numbers, and any identification numbers associated with the gage tube(s).

2.8 Calibration of vacuum and low-pressure gages between (10^{-2} and 1000) Pa using capacitance diaphragm gages as reference standards

2.8.1 Background

2.8.1.1 The capacitance diaphragm gage, CDG, is a pressure measuring system consisting of a pressure sensor, electrical circuitry to convert the physical reaction of the sensor into an electrical signal, and signal conditioning circuitry to process this signal into an output.

2.8.1.2 The sensor is essentially a two sided cavity, separated by a non-porous diaphragm. The sensor may have vacuum permanently sealed on one side, and a port on the other to allow for absolute pressure

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measurements, or it may have ports on both sides of the cavity to allow for differential measurements. In the latter case, the reference side should be continually evacuated for an absolute pressure measurement. The diaphragm forms the movable side of a variable capacitor. To construct the sensor, electrodes are added to one or both sides of the diaphragm to form the stationary plate(s) of the capacitor. See 2.8.11.1 d) and e) for examples of additional construction details.

2.8.1.3　　Some capacitance diaphragm transducers are manufactured that can be operated at temperatures in excess of 300 °C or below liquid nitrogen temperatures. The user should be aware that special considerations may be necessary for calibration and use at elevated or reduced temperatures. These include thermal transpiration (discussed later in this document), and temperature corrections. The manufacturer should be consulted for guidance.

2.8.1.4　　In general, the design and construction of CDGs results in high sensitivity and compatibility with clean, leak-tight systems.

a)　　In addition to a wide selection of ranges, various materials and types of construction are available, depending on the needs of the user. See 2.8.11.1 d) through g) for more details.

b)　　It is recognized that cost, complexity, and performance tradeoffs result in a wide variety of inherent uncertainties in commercial CDGs. Sometimes unintentional quality variations exist even within a group of the same type. It will not therefore be possible at any point in this document to give uncertainty statements that apply to all CDGs. References 2.8.11.1 i), j), and k) are examples of existing documentation of performance and uncertainties.

2.8.2 Scope

2.8.2.1　　This section is intended to provide guidance and criteria for the use of capacitance diaphragm gages (CDGs) as reference standards for calibrating other CDGs (see 2.8.11.1 b)), thermal conductivity gages (see 2.8.11.1 a)), and similar devices for absolute pressures in the range 10^{-2} to 1000 Pa (10^{-4} to 10 Torr).

2.8.2.2　　An overview of several commonly-used calibration procedures will be given.

2.8.3 Definitions

The *Dictionary of Terms*, Ref. 2.8.11.1 c), contains many definitions appropriate for this document. Below are several additional definitions for the purposes of this document, which may expand or modify other corresponding definitions.

a)　　**Absolute pressure:** The force per unit area molecules exert on a surface.

b)　　**Base pressure:** The lowest absolute pressure achievable in a given system.

c)　　**Calibration factor:** A factor by which an observed parameter must be multiplied to convert the parameter into a "true" (as defined by some standard) value. This factor may not be constant over the range of interest.

d)　　**Reference pressure:** The pressure on the reference side of a differential device. It may or may not be the system base pressure.

e)　　**Sensor:** A device that converts pressure to some measurable unit.

f) **Transducer:** A device converting the parameter or property being sensed or measured into a convenient form for analysis. In this document, it comprises a sensor plus electronics that translates pressure into an analogous electrical signal.

g) **Transfer standard:** Any measurement device can be a transfer standard, used to transfer "true" values of a property or parameter from itself to another device. However, the word "standard" implies that long- and short-term stabilities and sources of random and systematic uncertainties have been investigated, are understood, and are satisfactory for the desired transfer.

2.8.4 Assuring the quality of test and calibration results

2.8.4.1 A check standard is a device that may or may not be fully calibrated but which is known to be stable. It is not necessarily the same as the reference standard. It is used to make occasional comparisons at several pressure points simply to confirm that the device being checked (in particular, a working standard) does or does not need recalibration.

2.8.4.2 The check standard may be used as often as desired to compare several points with either a working or primary standard, with the first comparison having been done as soon as possible after a newly calibrated standard became available. After each comparison, the data is examined to see if any changes have occurred which are beyond some limit acceptable within the particular laboratory. If no such change is observed, then either the standard and check standard have changed in the same way, or neither the standard or check standard has changed (within the acceptable limit). Failure to detect drifts because of the former situation would become less likely if more than one check standard is used either simultaneously or during alternate checks. If a larger than acceptable change has occurred, and no independent means is available to determine which of the two devices has shifted, then the standard should be recalibrated, even though it may have been the check standard which changed.

2.8.4.3 It is recommended that the check standard be left evacuated and with power on when not in use.

2.8.4.4 Checking intervals should be a documented part of the laboratory procedures.

2.8.4.5 If the record of comparisons between the reference and check standards indicates that a systematic difference has developed between the standards, then this difference should be taken into account in the uncertainty of the reference standard.

2.8.4.6 Control charts can be used to identify trends or changes in the behavior of either the test gage or reference standard.

2.8.5 Personnel

Personnel performing calibrations should be familiar with the principle of operation and general characteristics of both CDGs and whatever device is being calibrated, plus all ancillary equipment.

2.8.6 Accommodation and environmental conditions

2.8.6.1 Capacitance diaphragm gages (CDGs)

CDGs have a small internal volume, high sensitivity, rapid response, can generally be used with a wide variety of gases, and are generally compatible with data acquisition systems. The long- and short-term

instabilities vary even among CDGs of the same type, and are affected by such things as temperature variations, position, and vibration. These environmental factors are considered below.

2.8.6.2   Temperature considerations for CDGs

a)   All CDGs respond in some degree to external temperature changes, as do the instruments that are being calibrated. Although many CDGs and other vacuum sensors have built-in heaters and sensors for temperature control, they all should be protected during both calibration and subsequent use from local air currents, such as those from air conditioning vents and cooling fans. If extreme temperature variations are encountered, it may be necessary to actively control the temperature of the immediate surroundings, even for CDGs with built-in temperature control. The degree to which the lack of temperature stability is a problem should be inferred from the behavior in a particular environment. For instance, the drift of the indicated pressure with zero applied pressure can have a significant temperature dependence.

b)   If condensable gases are present in the system, care should be taken to avoid temperatures where liquid can form.

2.8.6.3   Cleanliness of CDGs

Strict attention should be paid to maintaining cleanliness in order to insure leak-tight joints, low outgassing rates, and to prevent contamination of the instruments and/or the calibration manifold and standard. Regarding the possibility of contaminants, it is useful or perhaps even crucial to know the history of any device to be calibrated before placing it on the calibration manifold, to prevent the transfer of such contaminants.

2.8.6.4   Stability of CDG equipment

a)   It is suggested that CDGs being used as reference or check standards be kept on and evacuated when not in use. This keeps them in a relatively stable temperature environment, as well as unstressed, clean, and outgassed before the next use. All of these precautions will help prevent shifts in calibration and minimize the next start-up time.

b)   The output of the reference standard with zero applied pressure should be monitored prior to any calibration in order to develop a historical basis for predicting the possible shifts with time in the reference standard. This monitoring should continue for at least a 24-hour period after the reference standard has been operational for at least 48 hours.

2.8.6.5   Certification of equipment

a)   If applicable, all ancillary equipment (voltmeters, etc.), should be within their certification period and have known uncertainties which can be factored into the final uncertainty of the instrument being calibrated. Proper operation of computers and data gathering devices should be verified. Be aware that the addition of data-logging equipment can introduce errors such as noise, non-linearity, and voltage offsets. If possible, one should perform all calibration procedures with such equipment in place.

b)   CDG components and subsystems may be interchangeable according to the manufacturer's specifications. However, elements comprising the measurement system, particularly reference and check standards, should be calibrated as a unit.

2.8.6.6   Base pressure considerations

a)   The system base pressure can be ignored if it is insignificant relative to the desired uncertainty at the lowest calibration point. Otherwise, the base pressure would have to be known with an acceptable degree of uncertainty by some independent means. The base pressure can be no greater than some fraction (acceptable to the calibrator) of the lowest desired calibration pressure. The base pressure can be determined for example with an ionization gage, thermocouple gage, or another CDG. The operation of any base pressure indicator should be thoroughly understood.

b)   Note that, if the base pressure is at least two orders of magnitude below that of the lowest desired calibration pressure, its contribution to that pressure is no more than 1 %. Thus, if the base pressure is known to within ± 50 %, the contribution to the uncertainty of the lowest point is only 0.5 %. If it is totally ignored, its contribution to the uncertainty at the lowest pressure becomes 1.5 %. The base pressure should therefore be consistent with the user's requirements. In general, the lower the base pressure of the calibration system, the easier it will be to perform the calibrations.

2.8.6.7   Calibration interference considerations

There is always the possibility that a CDG and the instrument(s) being calibrated might interact with each other or with some other component (e.g., a source of electromagnetic noise) of the calibration system. As an example, there have been instances where the output voltage of one CDG signal conditioner has been affected by the proximity of another signal conditioner. The signal from one reference oscillator can beat against another (either through a ground loop or electromagnetic radiation), and cause oscillations in the output voltage. If this voltage is being used for control purposes, the consequences can be significant. This usually shows up as an unusually noisy reading of one or more of the instruments, and its source may be located by turning them on and off one at a time. This problem may be relieved by slightly changing the reference oscillator frequency.

2.8.6.8   Vibration

a)   The CDG sensors and many other vacuum sensors are sensitive to vibration. If there is an acceleration of the sensing element in the proper direction, a force will be exerted which is indistinguishable from pressure. Shock or vibration may cause an output proportional to the level of those frequency components lying within the bandwidth of the signal processing system. For most CDG systems, this is usually below 100 Hz.

b)   For the CDG sensor, the effects can be minimized by proper orientation of the diaphragm relative to the principle axis of vibration. For both the CDG and other sensors, additional minimization can be achieved by concentrating on isolating sources of vibration (e.g., pumps) of the system to which the gages are attached, by mounting the transducers on vibration isolators (rubber or cork sheets), and by adding mass to the system. If vibration cannot be adequately controlled by the above methods, additional filtering of the output signal can be added, at the expense of frequency response and possibly signal amplitude. This latter effect should be checked, and if found, appropriate actions taken.

2.8.7 Equipment

2.8.7.1   Initial adjustments

a)   There is a pervasive notion that any instrument that comes into a calibration laboratory should be adjusted to "within specifications" before it is used. However, calibration history can be made

irrelevant by such readjustments, which can include changing the span (sensitivity) setting. (This does not refer to the routine adjustments such as nulls and full scale settings of the readout.) Therefore, a calibration should be performed before any internal electronic adjustments are made.

b)   Obviously, if an instrument is so far out of adjustment as to make it unusable, realignment procedures should be undertaken. However, in general, this is not a good idea. The purpose of the calibration is to provide corrections to the readings. In general, the magnitudes of these corrections are of no consequence as long as they are accurately determined and used as appropriate. Therefore readjusting the electronics to make these small corrections is not necessary.

2.8.7.2   The calibration system

a)   In general, the CDG that is used as the reference standard should not be used for in situ calibrations on process equipment. In many cases the system on which the gages to be calibrated are being used involves sources of contamination or vibration which could affect the behavior of the standard. It is recommended that a separate calibration manifold be constructed, and that gages to be calibrated be moved from the system where they are being used to the calibration manifold.

b)   In all cases, there are precautions and factors that should be observed.

   1)   It is important that the interconnecting plumbing between the pumps, standard, and instrument being calibrated has a large enough conductance to prevent pressure gradients that will affect the calibration. These gradients will arise from normal outgassing and small system leaks as well as from transient pressures if there are changes for example in pump speeds or gas law (pressure-volume-temperature) effects.

   2)   Some types of gradient effects are easily detected simply by closing a valve or isolating the pumps and observing changes in the outputs of the different gages on the system. This is also a good test of the overall leak integrity of the system.

   3)   If absolute calibrations at pressures below about 0.1 Pa (10^{-3} Torr) are desired, high vacuum techniques should be used in constructing and maintaining the system.

   4)   Whatever type of calibration system is used, it should be kept as clean and dry as possible. This means, for example, back-filling with dry gas to a pressure slightly above 1 atmosphere before opening, and not leaving it exposed to atmospheric conditions any longer than necessary.

   5)   Records should be kept of times required to evacuate to base pressure, and of any revisions or unusual events/observations.

   6)   Vacuum leaks and pressure leaks are not necessarily symmetric. It is not sufficient to leak test a system for vacuum use by applying a pressure greater than 1 atmosphere and looking for some manifestation of a pressure leak.

   7)   Precautions should be taken to protect all gages against overpressure.

c)   **Connecting to the system**

Connect the transducers to be calibrated to the calibration system following the manufacturer's instructions and any requirements of the calibrating organization. The reference standards should be connected using VCR-type connectors (most CDGs are supplied with this type connector).

d)   **Leak testing**

After connecting the standard and devices to be calibrated to the calibration system, the system should be evacuated and checked for leaks by an appropriate method (e.g., helium leak detector). In addition to preventing the system from reaching its required base pressure, leaks may cause pressure differentials that can lead to calibration errors. Compliance with acceptable leak rates should be verified before calibration.

e)   **Port choice**

Absolute transducers have only one port for pressure application. Differential CDGs and other transducers have a pressure port and a second port for the reference pressure. Since many differential transducers are linearized only for positive differential pressures, the pressure should be applied from the correct direction. It will not, in general, damage a differential transducer to pressurize it in the reverse direction, but the behavior will not be optimal, and an existing calibration for pressurization in one direction may not be valid for the other.

f)   **Position**

1)   The gravitational field of the earth acts on the diaphragm of a CDG sensor, and on the sensing element of any transducer. The possible effects of orientation should therefore be considered. In particular, mounting a CDG transducer with the plane of the diaphragm parallel with the surface of the earth will produce a one "g" deflecting force, whereas mounting it with the plane perpendicular produces no deflecting force. The result is that the output between the two positions will be different.

2)   In general, there is no problem if the transducer is rotated about an axis perpendicular to the plane of the diaphragm, as this does not alter the plane of the diaphragm relative to gravitational forces. Therefore, the unit should be mounted in the same orientation during calibration and use.

3)   It is suggested that the transducer be mounted in such a way to prevent particles from falling into the diaphragm volume. For example, the pressure port of an absolute transducer should face either horizontal or downwards unless the manufacturer recommends otherwise.

4)   Sensitivity is not affected by the diaphragm orientation.

g)   **Support**

Some transducers are not meant to be attached to the system or operated without some form of support. In particular, these should not be suspended by their own tubing. Consult the manufacturers' instructions if unsupported operation is necessary.

h)   **Warm-up**

1)   Warm-up should occur under vacuum in order to minimize elastic recovery effects, outgassing time, and temperature changes due to pump-down prior to calibration.

2)   For vacuum sensors with no active temperature regulation which operate at or near room temperature, the time allowed for warm-up may be minutes to hours. This will depend in large part on the temperature sensitivity of the gage (consult the manufacturer) and the stability of the ambient temperature.

3)   For temperature regulated CDGs, at least 8 hours should be allowed for warm up. The reference standard should be warmed up for a minimum of 48 hours. Warm-up includes any and all ancillary equipment in the calibration area. Monitor the zero drift of the CDG as a function of time until its stability is satisfactory.

i)   **Initial adjustments**

The calibration laboratory should decide as a matter of policy whether or not test instruments are to be adjusted to minimize deviations from the calibration pressures. However, no adjustments should ever be made to the reference or check standards other than to reset the zero of the instrument or full scale voltage output voltages prior to use. If scale or linearity adjustments are ever made the instrument should be considered to no longer be in calibration and cannot be used as a standard until it is recalibrated by a competent laboratory.

j)   **Zeroing**

1)   Zeroing is to be done after the previously specified periods of warm up. For CDGs this consists of either zeroing both the signal conditioner and the sensor output or zeroing the sensor output alone. The manufacturer's instructions should be followed. The following configurations are typical:

- CDGs with single zero adjustment.

- CDGs with multiple zero adjustments.

- CDGs with multiple zero adjustments, and signal conditioner zero adjustment.

2)   With the latter type CDGs there are additional adjustments, which vary from manufacturer to manufacturer, related to the sensitivity. Instruction manuals should be consulted.

3)   The zeroing of the gage to be calibrated should be done according to manufacturers' instructions. In the case of thermal conductivity gages, this may involve setting a single potentiometer.

k)   **Control unit zeroing**

1)   If applicable, the electronics that controls the sensors and ancillary equipment should be set to read zero and full-scale when the corresponding voltages are applied by the front panel selector switch. Generally, these adjustments and corresponding instructions are available for commercial CDGs. If the laboratory performing the calibration is using its own electronics for power and/or readout and control, provisions should be made for these adjustments.

2) If any of the gages has multiple ranges, zeroing on one range may not set the zero for another.

3) Upon completing the zeroing procedures, record zeros on all ranges. If there are significant changes in zero as one goes from one range to another, these offsets may be used to correct the data on different ranges. Some laboratories use only the zero offset determined for the most sensitive range, and make no further zero adjustments as the calibration proceeds to different ranges. Shifts are then accounted for during data reduction, and the results appear in the calibration report.

4) If only one range is to be used, then the zero should be adjusted for that range.

l) **Zeroing absolute gages** - Two ways to establish the zero of absolute gages are suggested.

1) The preferred method is to establish a base pressure which, when its uncertainty is considered, is low enough to be an insignificant percentage of the lowest calibration pressure. For example, if the base pressure is known only to within an order of magnitude at 10^{-3} Pa, then one can only be sure it is less than 10^{-2} Pa. If the first calibration point is at 1 Pa, then this base pressure and its uncertainty can at most contribute a 1 % uncertainty to the first point.

As a second example, suppose the base pressure is known to be 10^{-2} Pa to within 1 %. Then the uncertainty contributed by the base pressure at a calibration point of 10^{-2} Pa is only 1 %.

2) An alternate method is to establish a low pressure, known by other means, within the calibration system, and set both the transfer standard CDG and the gage to be calibrated to indicate that pressure, accounting for thermal transpiration effects and height difference (pressure head) effects if applicable.

m) **Zeroing differential gages**

Using clean metal tubing and as short a path as possible, connect the pressure and reference ports together and evacuate to the reference pressure. An arrangement incorporating a valve and by-pass valve is suggested. Evacuate the system by opening both valves. Then close valve, but not the bypass valve, and wait for a sufficiently stable output and set the zero.

n) **Pre-cycling**

1) After warm-up, it is suggested that the CDG be cycled from zero to full scale and back. Also, depending upon manufacturer's recommendations, the CDG may benefit from cycling to atmospheric pressure.

2) Pre-cycling is particularly important for differential CDGs, since there is no way of knowing where they may be on their hysteresis loop before use. It also permits a check of the overall system behavior before the actual calibration.

3) Cycling introduces stresses in the sensor leading to a zero offset that can take hours to dissipate. Such zero effects can be of the order of 0.01 % of full-scale. They may gradually diminish, or may remain at the 0.01 % of full-scale level. In order to have repeatable low pressure (relative to the full-scale of the particular CDG) data, be sure that enough time is allowed for the system to return to a stable zero before calibration. Cycling stress is one reason why, either during a calibration or actual use, it is recommended that data be taken on the low pressure range first.

o) **CDG over-ranging or over-pressuring**

1) Absolute CDGs are made to withstand an applied absolute pressure of at least 10^5 Pa (1 atmosphere) without harm. Consult the manufacturer's literature. In order to achieve the best accuracy it is suggested that absolute CDGs with full scale ranges less than a 133 kPa be equipped with a permanent isolation valve, which is closed when the calibration system is cycled beyond the range of the CDG.

2) Differential CDGs operating with a vacuum reference should receive the same considerations. Care should be taken to disconnect the measurement port first in order to avoid reverse overpressure.

3) Consult manufacturer's literature for overpressure limits for other types of gages.

p) **The calibration gas**

The calibration gas should be compatible with the vacuum system, the standard, and the gages being calibrated. If it is necessary to correct for height differences between the standard and the gage under calibration, the mean molecular weight of the gas should be known. For thermal transpiration calculations, the mean molecular diameter should be known.

q) **Dielectric considerations for gages with capacitance sensing**

1) In single or double-sided CDGs, or any gages depending on a capacitance measurement, where the gas is in contact with the electrodes, the gas becomes part of the dielectric medium. The capacitance and therefore the gage performance are dependent on the dielectric constant of the calibration gas.

2) The gas used for calibration should have, within the accuracy limits required by the calibration, the same dielectric constant as that to be used in the calibration. For pressures near 1 atmosphere, the commonly used calibration gases have nearly the same value for the dielectric constant. For examples: air-1.00059, nitrogen-1.00058, hydrogen-1.000264, carbon dioxide-1.000985 (see 2.8.11.1 n)). The dielectric constant ϵ minus 1 is proportional to absolute pressure, i.e.,

$$\epsilon - 1 = K \cdot P \qquad (1)$$

so that effects arising from dielectric constant variations become less important with decreasing absolute pressure ranges. However, if a differential CDG is calibrated at a high line pressure, then subsequently pumped for use at vacuum, consideration should be given to the effect of the resulting change in dielectric constant on the calibration. Even in the case of a single-sided, dual electrode CDG where the dielectric change affects both capacitor plates, the changes will not be exactly the same. This can cause second-order calibration shifts.

3) There can be extreme circumstances. For highly ionizing gases such as tritium, free electrons can cause a substantial change in the effective impedance of the capacitors at most pressures above a few tenths of a pascal. Do not calibrate with nitrogen, and then use that same calibration with a highly ionizing gas. See 2.8.11.1 o).

r) **Thermal transpiration**

Thermal transpiration is frequently a cause of calibration errors. The phenomenon occurs where the mean free path becomes of the order of the internal diameter of the interconnecting tubing between two volumes at different temperatures. In that case, the higher-temperature volume will be at higher pressure than the lower-temperature one. Such a condition can exist in the application of any heated transducer or gage or in their calibration. Thermal transpiration affects both absolute and differential transducers when operated at pressures below approximately 100 Pa (see 2.8.11.1 p) to s).

s) **Height difference (pressure head) considerations**

If two pressure devices are connected together with a height difference between them, the lower device will read a pressure greater than that of the uppermost device. If the height difference (in meters) is h, then the pressure difference in pascals is given by

$$dP = \rho\, gh \tag{2}$$

where ρ is the density in kg/m^3 of the gas, and g is the local value of gravity in m/sec^2.

Since the (ideal gas) density can be calculated from

$$\rho = mP/RT \tag{3}$$

where m is the molecular weight in kg/mol (e.g., for nitrogen, m = .028014 kg/mol), R is the gas constant (8.31441 J/mol-K), and T is the absolute temperature in K, it follows that the fractional pressure variation with height is given by

$$dP/P = (mg/RT) \cdot h \tag{4}$$

For room-temperature nitrogen, the correction amounts to about 116×10^{-6} difference in the pressure for every meter difference in height, regardless of the pressure. Failure to account for pressure differences arising from height differences can be significant.

Corrections should also be made for any significant pressure differential between the standard and the test gage.

2.8.8 Test and calibration methods and method validation

2.8.8.1 Considerations for calibrations using CDGs

The calibration techniques by which a calibrated CDG may be used to calibrate other vacuum devices are varied, and depend in part on the capabilities and needs of the person doing the calibrating. The needs in turn depend on the end use. Several acceptable techniques will be described. Whichever method is used, it is essential that a proper uncertainty analysis be performed.

2.8.8.2 The calibration

a) Assuming that all of the set-up, warmup, and zeroing procedures discussed above have been followed, for both the transfer standard and the device to be calibrated, the calibration consists of applying

pressure, waiting an appropriate time for pressure equilibrium to be achieved, and recording the indications of the standard and device under calibration as close to simultaneously as possible. The equilibration time after applying a new pressure will be a function of the calibration method and system volumes. At each point, the operator should watch the comparative indications of standard and device being calibrated. Once they start tracking each other, it can be assumed that the system has equilibrated.

b)   Many gages have provision for checking the zero and full-scale indications of the electronics and auxiliary data acquisition equipment. For best accuracy, these can and should be checked occasionally during calibration. This procedure is not the same as zeroing the transducer indication, and therefore does not involve resetting the system to zero pressure. Consult the manufacturer's instruction manual for further information.

c)   Reference 2.8.11.1 i) gives additional information on calibration techniques, including those discussed below.

2.8.8.3   Data order

a)   The calibration should proceed from the lowest to the highest pressures, and then return, with the final points being again at zero. Because of mechanical stresses and the possible attendant zero shifts, the data taken during the return to lower pressures may show increasingly greater offsets from the data taken with increasing pressure, even if long pauses are made between data points. Should this occur, a decision will have to be made whether or not to include data obtained below a pressure where the offsets exceed acceptable uncertainty limits. If this is done, then the instrument should be used in a manner consistent with this calibration, that is, do not use the calibrated gage for determining descending pressures below the limit specified in the calibration report. In any gage that is routinely used to cover the full range of increasing and decreasing pressures, provision for any differences in ascending and descending pressures observed during calibration should be a part of the uncertainty statement.

b)   It may also be necessary to decide whether observed differences between the ascending and descending data arise from the effect of hysteresis or from a zero offset, but this decision can usually be made based on the pattern of the offsets (hysteresis will show as larger offsets near mid-range, diminishing as the high and low ends are approached) as well as the actual offsets between the initial and final indicated zeros.

c)   When zero drifts are significant it may be beneficial to return to zero between each data point. Establish a pressure, return to zero and take the difference between the two readings.

2.8.8.4   General comments on data acquisition

a)   Data should be taken at pressure intervals determined by the individual needs. If no satisfactory procedure has already been established, it is recommended that the calibration consist of no less than 10 equally-spaced points per decade on each scale of interest.

b)   At least two readings should be taken at each setting to determine whether there is a significant random variation about the mean pressure. If the two readings have a significant difference, at least two additional readings should be taken to allow a determination of the mean value and its random error (see 2.8.11.1 t)).

c)   Many devices have a range switch that provides different sensitivities for different decades of pressure. At or near the pressure where a range change is to be made, take data on both ranges. There may be a significant (tenths of a percent) discontinuity in the pressure indication when the range is changed. Such discontinuities should be dealt with in the data reduction.

2.8.8.5   General comments on calibrated differential gages

a)   The calibration of differential transducers is often attempted with atmospheric reference pressure. There may be substantial sensitivity errors associated with change in the reference line pressure, and in particular with using the gages calibrated with atmospheric reference pressure under vacuum conditions. Dielectric effects and distortion effects may be significant.

b)   Thermal transpiration effects do not appear until absolute pressures far below atmospheric are used. Therefore, a heated gage calibrated with atmospheric reference pressure, connected to a room temperature system and then used with a vacuum reference pressure, may produce measurements with errors of the order of several percent if thermal transpiration is not accounted for(see 2.8.11.1 p) to s)).

c)   The changes in the transducer output behavior should be understood and documented before using a gage calibrated with an atmospheric reference pressure as a vacuum reference standard.

2.8.8.6   Calibrations using a calibrated CDG as a transfer standard

a)   It is assumed that the transfer standard has an acceptable calibration and is in good working condition. All corrections given in its calibration report should be applied or otherwise be accounted for. Thermal transpiration effects should be accounted for in heated CDGs.

b)   After the calibration system has been evacuated to its base pressure, isolate the system pump and establish each calibration pressure by one of two methods:

   1)   Static Method: For pressures above approximately 10 Pa, admit a sufficient quantity of gas to raise the pressure to the desired value.

   2)   Dynamic Method: For lower pressures, adjust the pressure to the desired value by simultaneously pumping on the calibration system through a throttling valve while adjusting the flow of gas into the system.

2.8.9 Records

2.8.9.1   To provide objective evidence that calibration schedules are complied with and that the accuracy of each calibrated gage is maintained, records should be kept for each instrument that is calibrated.

2.8.9.2   Calibration data and ancillary records should be retained for a sufficient period of time to satisfy all regulatory and contractual requirements and to allow decisions to be made to adjust the assigned calibration interval.

2.8.9.3   The records should include the gage identification, calibration history including any unusual observations or circumstances, necessary information required to locate each gage in the calibration chain (if applicable), and a clear indication of when the next calibration should be performed on any gage. Similar information should be kept concerning the standards against which the calibrations are to be performed.

2.8.9.4   The user should identify and document the possible sources of uncertainty arising from things such as height corrections, temperature corrections or effects, pressure drops along the lines, thermal transpiration corrections, transducer orientations, operator skill (do two different operators get the same results?), etc. A possible way to evaluate the calibration technique is to repeatedly perform calibrations of the standard itself, or of an appropriate pressure measuring device which has been independently and reliably calibrated, and to document the random and systematic differences between these measurements and those given on the accompanying calibration certificate.

2.8.9.5   Calibration intervals

Until a history is obtained which indicates otherwise, it is wise to keep the initial calibration intervals on a device relatively short (probably no more than a year apart). Calibration history and interim treatment should be accounted for rather than adhering to an inflexible schedule. Comparisons at several points against a check standard can be used between regularly scheduled calibrations to determine if gross changes have occurred, and to determine if a scheduled complete calibration is even necessary. Regular comparisons with check standards, perhaps before each use, can serve to build a large part of the history referred to in the first sentence. Ref. 2.8.11.1 ab) may provide additional guidance.

2.8.9.6   Measurement traceability

If required, documentation should be available to trace the parameter values assigned to the standard to values in terms of nationally recognized standards. See Ref. 2.8.11.1 ac). If a reference standard is calibrated by a laboratory other than the National Institute of Standards and Technology (NIST), traceability to NIST should be established.

2.8.9.7   Assessment of uncertainties associated with the standard CDG

The historical records of the standard will allow assessment of its long-term stability, which may not be initially available. Analysis of the records may indicate that a revision in the overall uncertainty statements is necessary. Determination of the magnitudes of drifts and random behavior in the lowest useable range of the standard CDG should be done periodically, becoming part of its total uncertainty statement.

2.8.10   Reporting the results

2.8.10.1   The calibration report, at a minimum, should properly identify the gage and the standard used, date of calibration, temperature of the calibration manifold, calibration gas, calibration method, operator, and any special circumstances. It should also include some method for recovering the "true" pressure from the indicated gage values, and contain a statement of the overall estimated uncertainty. In the case of differential calibrations, the nominal base reference pressure should be reported.

2.8.10.2   A table containing the indicated values of the gage and the standard, along with the associated differences (or corrections), is a common method of presenting the data. Plots of these differences as a function of the gage reading are highly recommended, as they provide an easy method for comparison with previous calibrations as well as a quick method for predicting corrections for pressures between the actual calibration points. The "true" pressure (as determined by the reference standard) is sometimes presented as a function of the gage indication. The function may result from a least-squares curve fit. For low-range, heated CDGs or other heated gages, it is suggested that if curve fitting is used, a separate curve be fitted for the region affected by thermal transpiration.

2.8.10.3   On gages having range switches, large range switch-related zero shifts, if they occur, are easily handled when corrections are presented in tabular form. If equations of "true" (as read by the standard) pressure as a function of the gage reading are fitted to the data, it may be prudent to independently fit the data of each decade. If there are no range change offsets or if they are deemed negligible, then, depending on the pressure range covered and the accuracies required, a single fitted equation may suffice.

2.8.10.4   Thermal transpiration

Because of thermal transpiration, the temperature and species of the gas being measured and the temperature of the transducer should appear on every calibration report. This will permit a user or calibrator who is working with a different set of conditions to make appropriate corrections to observations.

2.8.10.5   Uncertainties

a)   The calibration report should include a statement of the estimated uncertainties, associated with the indicated pressures of the gage over the range of pressures used in the calibration. The uncertainties should include those arising from the calibration standard, the method used, and those associated with the gage itself, such as long- and short-term instabilities.

b)   Past performance of the gage should be taken into account during the discussion of uncertainties. For example, there may be predictable drifts with time that can be accounted for, or there may have been such a drastic shift from a prior calibration that its continued use under any circumstance should be questioned.

2.8.11   References

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