
**Gas analysis — Preparation of calibration
gas mixtures using dynamic volumetric
methods —**

**Part 7:
Thermal mass-flow controllers**

*Analyse des gaz — Préparation des mélanges de gaz pour étalonnage
à l'aide de méthodes volumétriques dynamiques —
Partie 7: Régulateurs thermiques de débit massique*

ISO 6145-7:2009

[https://standards.iteh.ai/catalog/standards/sist/e8b9e1a0-dacd-4593-bf90-
ce2bb4e81acb/iso-6145-7-2009](https://standards.iteh.ai/catalog/standards/sist/e8b9e1a0-dacd-4593-bf90-ce2bb4e81acb/iso-6145-7-2009)



PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 6145-7:2009

<https://standards.iteh.ai/catalog/standards/sist/e8b9e1a0-dacd-4593-bf90-ce2bb4e81acb/iso-6145-7-2009>



COPYRIGHT PROTECTED DOCUMENT

© ISO 2009

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword.....	iv
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Principle	2
5 Set-up	2
5.1 General	2
5.2 Thermal mass-flow controller using a constant current supply	2
5.3 Thermal mass-flow controller under constant temperature control	3
6 Preparation of gas mixtures	4
6.1 Description of the experimental procedure	4
6.2 Area of validity	6
6.3 Operating conditions	6
7 Calculations	7
7.1 Volume fraction	7
7.2 Sources of uncertainty	7
7.3 Uncertainty of measurement	8
Annex A (informative) Pre-mixed gases for preparation of mixtures of high dilution	9
Annex B (informative) Practical hints	10
Annex C (informative) Calculation of uncertainties	12
Bibliography	15

Foreword

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

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 6145-7 was prepared by Technical Committee ISO/TC 158, *Analysis of gases*.

This second edition cancels and replaces the first edition (ISO 6145-7:2001). In preparation of the first edition, it was assumed that each thermal mass-flow controller (TMC) would be configured for use at its optimum performance, and the uncertainty in the method was estimated on that basis. In this edition, therefore, extra precautionary text has been added to make it clear that the method shall not be employed, for example, to make a 10:1 binary mixture by using two thermal mass-flow controllers of identical range with one operated at its maximum, say, of 1 000 ml/min and the other at 100 ml/min. In the first edition, this necessary provision was only stated briefly in an informative annex; it has now been expanded and stated more explicitly in a normative part. Another major update is separation of the original Clause 3 into two clauses, one of which (Clause 4) defines the principle while the other (Clause 5) presents additional explanation to the user. The latter of these clauses now includes the necessary requirements. By introducing two new and relevant bibliographic references, the understanding of Annex B has been improved. Finally, some typing errors have been corrected.

ISO 6145 consists of the following parts, under the general title *Gas analysis — Preparation of calibration gas mixtures using dynamic volumetric methods*:

- *Part 1: Methods of calibration*
- *Part 2: Volumetric pumps*
- *Part 4: Continuous syringe injection method*
- *Part 5: Capillary calibration devices*
- *Part 6: Critical orifices*
- *Part 7: Thermal mass-flow controllers*
- *Part 8: Diffusion method*
- *Part 9: Saturation method*
- *Part 10: Permeation method*
- *Part 11: Electrochemical generation*

ISO 6145-3, entitled *Periodic injections into a flowing gas stream*, has been withdrawn.

Gas analysis — Preparation of calibration gas mixtures using dynamic volumetric methods —

Part 7: Thermal mass-flow controllers

1 Scope

This part of ISO 6145 is one of a series of International Standards dealing with dynamic volumetric methods used for the preparation of calibration gas mixtures. This part specifies a method for continuous production of calibration gas mixtures, containing two or more components, from pure gases or other gas mixtures by use of commercially available thermal mass-flow controllers.

If this method is employed for the preparation of calibration gas mixtures, the optimum performance is as follows: the relative expanded uncertainty of measurement, U , obtained by multiplying the combined standard uncertainty by a coverage factor $k = 2$, is not greater than 2 %.

If pre-mixed gases are used instead of pure gases, mole fractions below 10^{-6} can be obtained. The measurement of mass flow is not absolute and the flow controller requires independent calibration.

The merits of the method are that a large quantity of the gas mixture can be prepared on a continuous basis and that multicomponent mixtures can be prepared as readily as binary mixtures if the appropriate number of thermal mass-flow controllers is utilized.

NOTE Gas-blending systems based upon thermal mass-flow controllers, some including the facility of computerization and automatic control, are commercially available.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 6143, *Gas analysis — Comparison methods for determining and checking the composition of calibration gas mixtures*

ISO 6145-1:2003, *Gas analysis — Preparation of calibration gas mixtures using dynamic volumetric methods — Part 1: Methods of calibration*

ISO 7504, *Gas analysis — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 7504 apply.

4 Principle

Continuous production of calibration gas mixtures, containing two or more components, from pure gases or other gas mixtures by the use of commercially available thermal mass-flow controllers is described. By adjusting the set-points on the flow controllers to pre-determined values, it is possible to change the composition of the gas mixture rapidly and in a continuously variable manner. By selecting appropriate combinations of thermal mass-flow controllers and with the use of pure gases, the volume fraction of the component of interest in the complementary gas can be varied by a factor of 1 000.

5 Set-up

5.1 General

To prepare the gas mixture, each gaseous component is passed at a known, controlled flow rate, and at constant pressure, from a calibrated thermal mass-flow controller. Use accurate flowmeters in measuring the relevant flows in order to reach an acceptable measure of uncertainty regardless of the setting of the mass-flow controller (see also ISO 6145-1:2003, Table 1).

A thermal mass-flow controller consists of a measuring unit for mass flow and a proportioning valve which is controlled by an electronic unit (see also References [1] and [2]).

5.2 Thermal mass-flow controller using a constant current supply

The flowing gas is passed through a heater connected to a constant current supply and the temperature is sensed upstream and downstream from the heater.

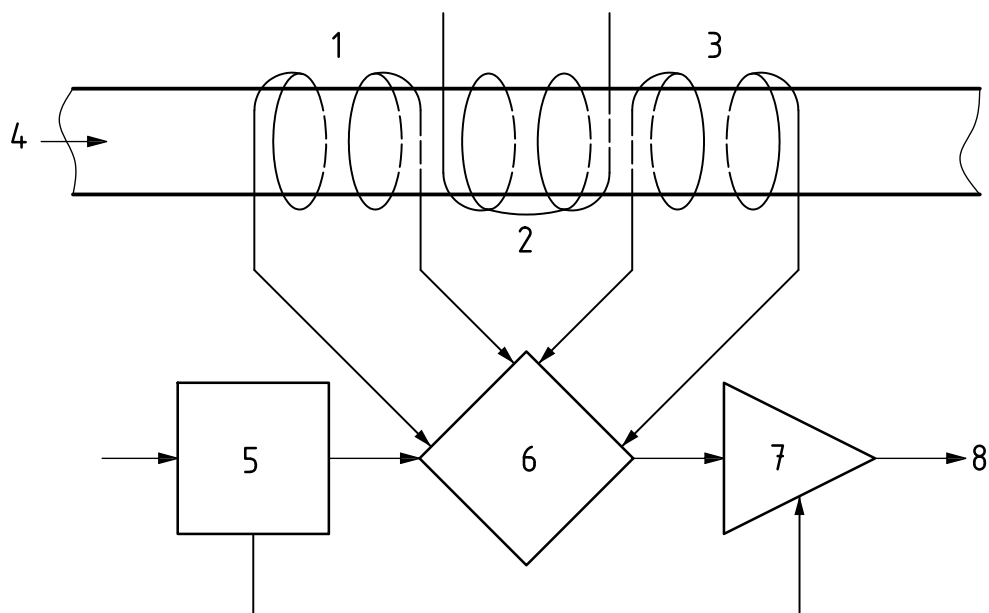
Figure 1 shows the principle of a thermal mass-flow controller: heater, temperature sensors and associated circuitry. The two temperature sensors, one upstream and one downstream from the heater form two arms of a Wheatstone bridge circuit, which is balanced to give a zero reading when there is no gas flow. When there is a gas flow through the system, a temperature difference, ΔT , is established between the two sensors such that the heat flux, Φ , is given by:

$$\Phi = c_p \Delta T q_m \quad (1)$$

where

c_p is the heat capacity per unit mass, or molar heat capacity, of the gas at constant pressure;

q_m is the mass flow rate.

**Key**

- 1 temperature sensor 1
- 2 temperature sensor 2
- 3 heater
- 4 gas supply
- 5 current supply
- 6 Wheatstone bridge
- 7 differential amplifier
- 8 signal readout

iTeh STANDARD PREVIEW
(standards.iteh.ai)

[ISO 6145-7:2009](https://standards.iteh.ai/catalog/standards/sist/e8b9e1a0-dacd-4593-bf90-ce2bb4e81acb/iso-6145-7-2009)

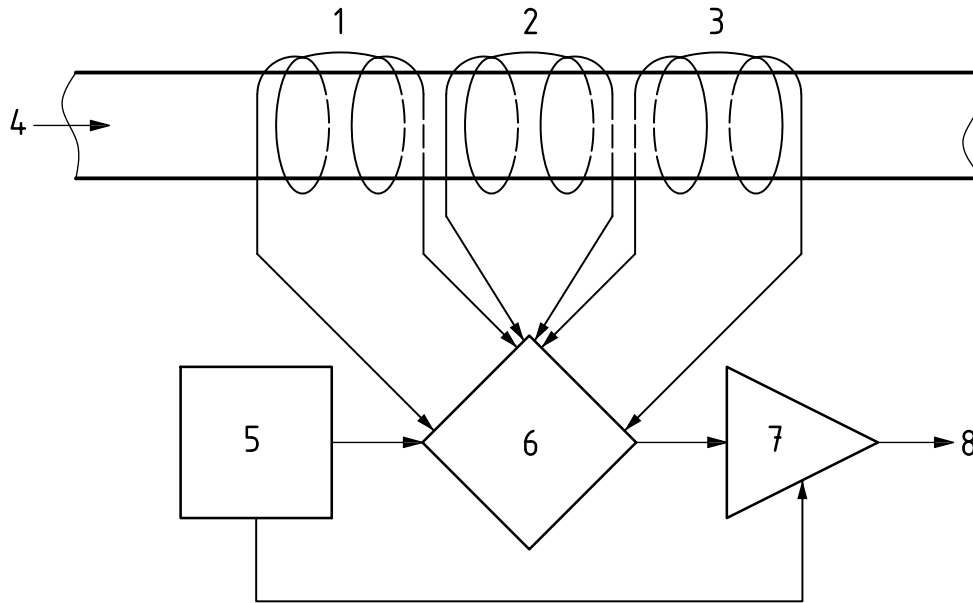
<https://standards.iteh.ai/catalog/standards/sist/e8b9e1a0-dacd-4593-bf90-ce2bb4e81acb/iso-6145-7-2009>

Figure 1 — Principle of a thermal mass-flow controller with constant current supply

The difference in temperature between sensors results in a potential difference across the Wheatstone bridge circuit and thus a signal. The signal is compared with an adjustable reference voltage in a differential amplifier. The resulting output signal is in turn used for operating a control valve to regulate the flow of gas.

5.3 Thermal mass-flow controller under constant temperature control

In this system (see Figure 2), the gas passes through three heaters in sequence, each of which is connected to an arm of a self-regulating Wheatstone bridge. Instead of the difference in temperature being measured, the input to each heater is such that the temperature distribution along the flow path is maintained uniform. The Wheatstone bridge current is proportional to the heat loss and therefore proportional also to the mass flow of the gas. The output signal is again used to operate a solenoid valve to control the mass flow rate.



Key

- 1 heater 1
- 2 heater 2
- 3 heater 3
- 4 gas supply
- 5 current supply
- 6 Wheatstone bridge
- 7 differential amplifier
- 8 signal readout

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 6145-7:2009
<https://standards.iteh.ai/catalog/standards/sist/e8b9e1a0-dacd-4593-bf90-ce2bb4e81acb/iso-6145-7-2009>

Figure 2 — Thermal mass-flow controller under constant temperature control

In the preparation of multicomponent mixtures, it is in general necessary to use one mass-flow controller for each component. Dual-channel controllers are available and may be used in the preparation of binary mixtures or, for example, in the preparation of mixtures of a given gas in air.

6 Preparation of gas mixtures

6.1 Description of the experimental procedure

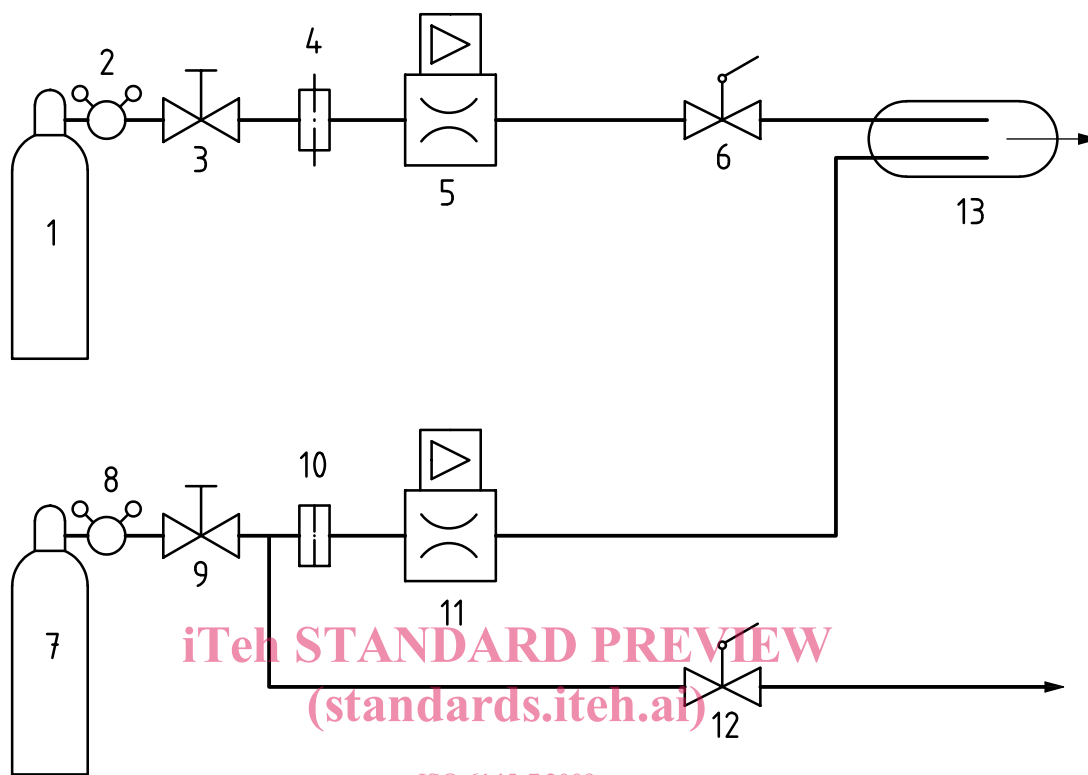
A schematic diagram of the arrangement used for the preparation of binary mixtures is shown in Figure 3.

The pressure and temperature at the time of the calibration shall be recorded.

Depending on the gases to be mixed and the fact that they are not ideal, the volume fraction can be somewhat influenced by the ambient pressure and temperature. The pressure and temperature at the time of calibration of the analyser should be as near as possible to those prevalent at the time the thermal mass-flow controllers were checked by the comparison method given in ISO 6143 (see 7.3).

Concentrations of calibration gas mixtures are normally expressed as volume fractions but manufacturers' accuracy specifications for thermal mass-flow controllers are expressed in terms of percentage of the full scale of the instrument. The relative expanded uncertainty of 2 %, which is quoted in the scope of this part of ISO 6145, is 2 % of the volume fraction of the calibration component of the mixture. This value assumes optimum use of each thermal mass-flow controller in the system, which means that each is operated at, or

very near to, its maximum flow rate. Thus, if a thermal mass-flow controller is operated at 10 % of full scale, the expanded uncertainty expressed as a percentage of maximum flow (as distinct from relative expanded uncertainty) can be $\pm 1\%$, but, if it is expressed instead in terms of a percentage of the actual flow rate, the expanded uncertainty becomes 10 %.



ISO 6145-7:2009

<https://standards.iteh.ai/catalog/standards/sist/e8b9e1a0-dacd-4593-bf90-ce2bb4e81acb/iso-6145-7-2009>

Key

Complementary gas:

- 1 cylinder of pressurized gas
- 2 pressure regulator
- 3 shut-off valve
- 4 filter against contamination
- 5 thermal mass-flow controller
- 6 shut-off valve

Calibration component:

- 7 cylinder of pressurized gas
- 8 pressure regulator
- 9 shut-off valve
- 10 filter against contamination
- 11 thermal mass-flow controller
- 12 shut-off valve
- 13 mixing vessel

Figure 3 — Mixing apparatus for production of binary gas mixtures by means of thermal mass-flow controllers

A binary mixture containing the calibration component at a volume fraction of 1:10 could be prepared using two thermal mass-flow controllers, each of full scale 1 000 ml/min, by operating one at 100 ml/min and the other at 1 000 ml/min. However, the expanded uncertainty in the flow rate of the former would be $\pm 10,00\%$ of the flow rate and the relative expanded uncertainty in the volume fraction would be $\pm 10,05\%$. To prepare the mixture to a relative expanded uncertainty of 2 %, the mixture shall be prepared using one thermal mass-flow controller of full-scale range 100 ml/min and the second one of full scale range 1 000 ml/min, both being operated at very close to full scale.

The same requirement shall be observed relative to preparation of multicomponent mixtures.

NOTE 1 A method for which there is no requirement for calibration against external standards of gas flow rate or volume fraction is described briefly in Annex B, and the reference to the publication which provides the complete description is given in the Bibliography.

Gas cylinders (1) and (7) containing respectively the complementary gas and the calibration component are connected to the thermal mass-flow controllers (5) and (11) through pressure regulators (2) and (8) and shut-off valves (3) and (9). The two in-line filters (4) and (10) provide protection against contamination. The gases from the flow controllers enter the mixing vessel (13).

The recommended working range for the pressure regulators is 60 kPa [0,6 bar¹] to 600 kPa (6,0 bar). The pressure regulator for the gaseous component shall also be suitable for the particular component involved (e.g. the diaphragm shall be of stainless steel or other corrosion-resistant material). Similarly, the thermal mass-flow controllers shall be suitable for use with the gaseous components and for the requirements of the gas mixture.

Set the input pressures appropriate to the controllers using the pressure regulators and open the shut-off valves (3), (6) and (9). Purge the inlet path of the gaseous component through the shut-off valve (12), which shall be of a type which can be operated rapidly.

Adjust the set-points of the controllers so as to obtain the respective flow rates in the correct ratio for the desired composition of the binary gas mixture; meanwhile, continue the purging process of the input tube for the component gas by multiple opening and closing of valve (12) until a total volume of gas at least 10 times the volume of the flow path has been vented.

When the system has been thoroughly purged, feed the gases via the thermal mass-flow controllers to the mixing vessel (13), constructed from inert materials. Provided that the resistance to flow downstream of the mixing vessel (13) is low in relation to the flow being delivered at the source, the mixture flows at ambient atmospheric pressure to the instrument. (standards.iteh.ai)

NOTE 2 Although for most applications the gas mixture will be transmitted at the prevailing ambient atmospheric pressure, this method may also conceivably be applied to convey mixtures at elevated exit pressures. However, in this case, it would be necessary to give due consideration to changes in c_p and density of the gaseous components with pressure in order to assess the validity of this procedure. <https://standards.iteh.ai/catalog/standards/sist/4e81acb/iso-6145-7-2009>

6.2 Area of validity

The method is applicable to the preparation of mixtures of non-reacting species, i.e. those which do not react with any material of construction of the flow path in the thermal mass-flow controller or the ancillary equipment. Particular care shall be exercised if the method is considered as a means of preparing gaseous mixtures which contain components that form potentially explosive mixtures in air. Steps shall be taken to ensure that the apparatus is safe, for example by means of in-line flame arrestors in addition to the items listed in 6.1.

This method is not absolute and each thermal mass-flow controller shall be calibrated for the particular gas for which it is to be used.

6.3 Operating conditions

The conditions for efficient operation of the sensor system are that there shall be no heat loss or heat gain, other than that which results from the flow of gas, between the region of the heater and that of the downstream sensor, and that there shall be uniform temperature distribution across the gas stream. The assumption that c_p is constant is valid only over a restricted range of temperature. The general precautions common to all dynamic techniques of preparation shall be observed. It is essential that attention be paid to the materials used in the construction of the flow system. Only materials of low porosity, that are non-adsorbing are suitable. The pipework shall be clean and all unions secure.

1) 1 bar = 100 kPa = 0,1 MPa = 1 N/m².

Unless independence of the thermal mass-flow controller to its orientation has been established, it shall be maintained in the orientation in which it was calibrated. Controllers shall be calibrated for the components in question and it may be necessary to consult the manufacturer of the controller if the type of gas is to be changed; it may be necessary for the sensor to be changed.

7 Calculations

7.1 Volume fraction

The volume fraction is determined with reference to any of the methods of calibration described in Clause 4 of ISO 6145-1:2003. Due consideration shall be given to the uncertainty associated with the method selected.

Calibration of the thermal mass-flow controller will define the mass flow rate, q_m , or the volumetric flow rate, q_V , dependent on the method used.

$$q_m = \frac{\Phi}{c_p \Delta T} \quad (2)$$

$$q_V = \frac{\Phi}{c_p \rho \Delta T} \quad (3)$$

where ρ is the density of the component.

The mole fraction is:

$$x_A = \frac{q_{m,A}}{M_A} \bigg/ \left(\frac{q_{m,A}}{M_A} + \frac{q_{m,B}}{M_B} \right) \quad (4)$$

<https://standards.iteh.ai/catalog/standards/sist/e8b9e1a0-dacd-4593-bf90-ce2bb4e81acb/iso-6145-7-2009>

where

M_A and M_B are the molar masses of components A and B, respectively;

$q_{m,A}$ and $q_{m,B}$ are the values of mass flow rate, q_m , for components A and B, respectively.

The corresponding volume fraction is:

$$\varphi_A = q_{V,A} / (q_{V,A} + q_{V,B}) \quad (5)$$

7.2 Sources of uncertainty

Commercially available thermal mass-flow controllers usually indicate the gas flow rate in volume units as an analogue or digital display. Typical claims for accuracy are $\pm 1\%$ of full scale, provided that the ambient temperature is maintained within $\pm 5^\circ\text{C}$ of the temperature at which the instrument was calibrated. The corresponding claims for set-point repeatability are $\pm 0,2\%$ of full scale.

It is assumed that pressures and temperatures respectively are measured with the same instruments during calibration and use, so that the standard uncertainties in these measurements are constant throughout.

From Equations (2) and (3):

$$\frac{u(q_m)}{q_m} = \left\{ \left[\frac{u(\Phi)}{\Phi} \right]^2 + \left[\frac{u(c_p)}{c_p} \right]^2 + \left[\frac{u(\Delta T)}{\Delta T} \right]^2 \right\}^{1/2} \quad (6)$$