
**Natural gas — Measurement of
properties — Calorific value and Wobbe
index**

*Gaz naturel — Mesurage des propriétés — Pouvoir calorifique et indice
de Wobbe*



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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.

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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 15971 was prepared by Technical Committee ISO/TC 193, *Natural gas*.

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Introduction

The amount of energy delivered by a flowing natural gas is often determined as the product of the volume delivered and the calorific value per unit volume of the gas. It is, therefore, important to have available standardized methods of determining the calorific value. In many cases, it is possible to calculate the calorific value of natural gas, with sufficient accuracy, given the composition (see ISO 6976). However, it is also possible, and sometimes a preferred alternative, to measure calorific value using any one of several techniques that do not require a compositional analysis. The methods currently in use, and the many factors that it is necessary to address in the selection, evaluation, performance assessment, installation and operation of a suitable instrument, are detailed herein. The measurement of the Wobbe index, a property closely related to calorific value, is discussed briefly in an informative annex, but is not considered in detail in the normative parts of this International Standard.

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Natural gas — Measurement of properties — Calorific value and Wobbe index

1 Scope

This International Standard concerns the measurement of calorific value of natural gas and natural gas substitutes by non-separative methods, i.e. methods that do not involve the determination of the gas composition nor calculation from it. It describes the principles of operation of a variety of instruments in use for this purpose, and provides guidelines for the selection, evaluation, performance assessment, installation and operation of these.

Calorific values can be expressed on a mass basis, a molar basis or, more commonly, a volume basis. The working range for superior calorific value of natural gas, on the volume basis, is usually between 30 MJ/m³ and 45 MJ/m³ at standard reference conditions (see ISO 13443). The corresponding range for the Wobbe index is usually between 40 MJ/m³ and 60 MJ/m³.

This International Standard neither endorses nor disputes the claims of any commercial manufacturer for the performance of an instrument. Its central thesis is that fitness-for-purpose in any particular application (defined in terms of a set of specific operational requirements) can be assessed only by means of a well-designed programme of experimental tests. Guidelines are provided for the proper content of these tests.

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 6976:1995, *Natural gas — Calculation of calorific values, density, relative density and Wobbe index from composition*

ISO 14532: 2001, *Natural gas — Vocabulary*

3 Terms and definitions

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

3.1 Calorific value and Wobbe index

3.1.1

superior calorific value

amount of heat that would be released by the complete combustion in air of a specified quantity of gas (on a molar, mass or volume basis), in such a way that the pressure, p , at which the reaction takes place remains constant and all the products of combustion are returned to the same specified temperature, T , as that of the reactants, all of these products being in the gaseous state, except for water formed by combustion, which is condensed to the liquid state at T

See ISO 6976.

3.1.2

inferior calorific value

amount of heat that would be released by the complete combustion in air of a specified quantity of gas (on a molar, mass or volume basis), in such a way that the pressure, p , at which the reaction takes place remains constant, and all the products of combustion are returned to the same specified temperature, T , as that of the reactants, all of these products being in the gaseous state

See ISO 6976.

3.1.3

Wobbe index

superior calorific value on a volumetric basis at specified reference conditions, divided by the square root of the relative density at the same specified metering reference conditions

See ISO 6976.

3.1.4

standard reference conditions

temperature, $T = 288,15$ K, and (absolute) pressure, $p = 101,325$ kPa, for the real dry gas

See ISO 13443.

NOTE Standard reference (or base) conditions of temperature, pressure and humidity (state of saturation) are defined for use only in natural gas and similar applications. For the calorific value on a volumetric basis, these conditions apply to both the metering and combustion of the gas. In the expression of physical quantities throughout this International Standard, these standard reference conditions as defined in ISO 13443 are taken to apply.

3.2 Water content of gas

3.2.1

saturated gas

natural gas which, at the specified conditions of temperature and pressure, is at its water dew-point

3.2.2

dry gas

natural gas which does not contain water vapour at a mole fraction greater than 0,000 05

See ISO 6976.

3.2.3

partially saturated or wet gas

natural gas which contains an amount of water vapour between that of the saturated gas and that of the dry gas, at the specified conditions of temperature and pressure

3.3 Performance classification

NOTE The following classification scheme is adopted in order to categorize the uncertainties associated with measurement of calorific value. The attached notes are explanatory, not parts of the definitions. The values given refer to an expanded uncertainty with a coverage factor of 2.

3.3.1

class 0

performance with which uncertainty limits of no greater than $\pm 0,1$ % in calorific value may be associated

NOTE Performance of this quality can currently be achieved only by instruments in which all operations are carried out in strict accordance with the best metrological practices and in which all relevant physical measurements are directly traceable to primary metrological standards. Typically, such an instrument is custom-built and installed in a purpose-built, environmentally controlled specialist laboratory; a specially trained and identified operator is likely required. Instruments of this type are sometimes known as "reference calorimeters" and all, to date, make measurements discontinuously on discrete samples of gas.

3.3.2**class 1**

performance with which uncertainty limits of no greater than $\pm 0,1 \text{ MJ/m}^3$ on a volume-basis calorific value (approximately 0,25 %) may be associated

NOTE This is the lowest level of measurement uncertainty currently available for any form of commercial instrument used in routine field (i.e. non-laboratory) operation. Even for the few types of instrument that are intrinsically capable of this performance, it is unlikely to be achieved unless installation is in accordance with both the manufacturer's instructions and the principles described in this International Standard, and operation is in accordance with the calibration, verification, maintenance and quality control procedures described in this International Standard.

3.3.3**class 2**

performance with which uncertainty limits of no greater than $\pm 0,2 \text{ MJ/m}^3$ on a volume-basis calorific value (approximately 0,5 %) may be associated

3.3.4**class 3**

performance with which uncertainty limits of no greater than $\pm 0,5 \text{ MJ/m}^3$ on a volume-basis calorific value (approximately 1,0 %) may be associated

3.4 Terms from metrology

NOTE The following definitions, including the Notes attached to them (except the Note to 3.4.6), are all taken from ISO 14111, where additional explanatory details are given.

3.4.1**accuracy**

closeness of agreement between a measurement result and the true value of the measurand

NOTE The term "accuracy", when applied to a set of measurement results, describes a combination of random components and a common systematic error or bias component.

3.4.2**trueness**

closeness of agreement between the average value obtained from a large series of measurement results and the true value of the measurand

NOTE The measure of trueness is usually expressed in terms of bias.

3.4.3**bias**

difference between the expectation of the measurement results and an accepted reference value

3.4.4**precision**

closeness of agreement between independent measurement results obtained under prescribed conditions

NOTE Precision depends only on the distribution of random errors and does not relate to the true value.

3.4.5**repeatability**

precision under conditions where independent measurement results are obtained with the same method on identical measuring objects in the same laboratory by the same operator within short intervals of time

NOTE Repeatability is expressed quantitatively based on the standard deviation of the results.

3.4.6

uncertainty

estimate attached to a measurement result which characterizes the range of values within which the true value is asserted to lie

NOTE An alternative, but equivalent, definition taken from Reference [1] is as follows: parameter, associated with the result of a measurement, that characterizes the dispersion of the values that can reasonably be attributed to the measurand.

3.4.7

calibration

set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards

3.4.8

verification

confirmation by examination and provision of objective evidence that specified requirements have been fulfilled

4 Principles of measurement

4.1 Introduction

Instruments capable of class 0 performance (hereafter, for brevity, called class 0 calorimeters) have been established in a few specialist laboratories; but since they are, inevitably, labour-intensive, spot-test instruments, not commercially available and not suitable for field operation, details of their installation, operation and maintenance are beyond the scope of the main part of this International Standard.

Nevertheless, measurements made using calorimeters of this type can have an important part to play in the "everyday" determination of calorific value, mainly as one possible accredited means for the provision of certified calibration gases (certified gaseous reference materials) having traceability to international metrological standards (see 7.2). They may also be used for research purposes and the resolution of disputes.

The principles upon which typical class 0 calorimeters operate, together with details of many of the other relevant factors, are given in Annex C. All class 0 calorimeters so far devised have, as their primary determination, the mass-basis calorific value. To be useful for most routine applications, it is necessary to convert this by some secondary means to the volume-basis value. In order to achieve a volume-basis calorific value with an uncertainty of $\pm 0.1\%$, it is usual to dedicate a density meter of sufficient accuracy for use with instruments of this type.

Instruments capable of class 1, class 2 or class 3 performance usually measure calorific value on the volume basis. They are normally designed for continuous, unattended operation in the field, producing an essentially continuous record of calorific value. Except for process gas chromatographs (which are not the subject of this International Standard), they are the only types of instrument that can sensibly be used for routine measurements of calorific value on natural gas passing through transmission and distribution systems.

The principle of operation may be either direct, indirect or inferential, within the meaning of these terms in accordance with ISO 14532. This International Standard is concerned mostly with the performance of these kinds of instruments. Some instruments have the additional facility of measuring relative density; in these cases, this capability is equivalent to making available the determination both of the calorific value on the mass basis and of the Wobbe index.

Depending upon the particular application, instruments can be required to record either the superior or the inferior calorific value. Although each particular type of instrument responds, in principle, to one or the other of these, most types can be set up so as to record, with little loss of accuracy for typical natural gases, the alternate value. To achieve this, the main requirement is that the instrument be set up using calibration gases that are correspondingly certified (see also 5.1.10.2).

4.2 Direct combustion calorimetry

Only those instruments that are true combustion calorimeters, in the sense that the energy released as heat by the combustion of gas is determined by means of thermometric measurements, fall into the “direct-measurement” category. All current commercial implementations determine the volume-basis calorific value.

In this type of instrument, the gas sample is metered volumetrically on a continuous basis, often through the use of a water-sealed “wet meter” (Reference [2], Chapter 4, and ISO 6145-1), before passing to a burner. The main measurement is of the quasi-stationary (equilibrium) rise in temperature of a continuously flowing (metered) heat-exchange medium with which the hot products of combustion do not mix.

The heat-exchange medium is usually air; water-flow calorimeters do exist in a wide variety of forms but all of these are now obsolete. The temperature rise is usually measured using resistance thermometry. Calibration is usually achieved by the use of gaseous reference materials (working standards) certified for calorific value.

Calorific values are usually measured by this method at ambient temperature and pressure. It is necessary, however, to refer the values recorded to specified reference conditions of temperature and pressure of both metering and of combustion. For this reason, prior information concerning the stability of the output with respect to variations of ambient temperature can be important (see 5.1.6).

It is also important to define the reference condition of water content for the gas, in particular if the instrument controls the water content of the gas (either by saturation or by drying) prior to or during the measurement process. At standard reference conditions, the difference between the superior calorific value of a dry gas and a saturated gas is approximately 1,7 %.

Instruments of this type are usually set up so as to record the superior calorific value. One of the main advantages of true combustion calorimeters is that there is no restriction on the composition of the sample gas for which they are expected to give the correct result.

Calorimeters based on this generic methodology (Reference [2], Chapter 10; Reference [6], Chapter 7; and References [3] to [5]) are often capable of class 1 performance, but typically have quite a sluggish response to changes in calorific value because of thermal inertia.

Typical examples of this kind of calorimeter are described in Annex D.

4.3 Indirect methods

4.3.1 General

Instruments that fall into the “indirect” category are those that measure some physicochemical property of the gas and use a known relationship, established by both practical observation and theoretical analysis, between calorific value and the property measured, in order to infer the calorific value, either superior or inferior, of the gas.

4.3.2 Stoichiometric combustion

Instruments of this type depend upon the principle that, for a gas mixture containing only alkane hydrocarbons and inert constituents, the volume-basis calorific value (either superior or inferior) is a linear function of the air-to-gas ratio required to achieve stoichiometric combustion.

There are at least two ways to implement this principle in a practical device. In one implementation, the stoichiometric point is determined by searching for the air-to-gas ratio at which the amount of oxygen in the products of complete combustion is zero. In an alternative implementation, the stoichiometric point is determined by searching for the air-to-gas ratio at which maximum flame temperature is achieved.

One disadvantage of instruments that operate on this principle is the requirement to confirm that the sample gas contains only alkane hydrocarbons and inert constituents. Any other constituent (for example, alkenes, hydrogen, carbon monoxide and, most severely, oxygen) can cause the instrument to give a false reading; in some cases, however, any errors can be accounted for by a correction procedure.

Instruments based on this principle^[7] are readily capable of at least class 2 performance and typically exhibit rapid response to changes in calorific value.

Some practical details of these devices are given in Annex E.

4.3.3 Catalytic combustion

Instruments of this type are based on the principle that a determination of the amount of heat released during the complete oxidation (combustion) of a gas at a catalytic surface is a proper representative measure of its calorific value.

In one implementation, a semi-continuous (i.e. on-off-on) metered flow of fuel gas undergoes oxidation at the surface of a catalyst-coated conductor; the heat released by this combustion process raises the temperature of the conductor and so influences its electrical resistance. The electrical resistance can readily be used to follow the rise in temperature over the "on" period of the gas flow, and the integrated temperature rise for this period may then be used as an indicator of inferior calorific value.

In another implementation, the oxidation process takes place within a bed of powdered catalytic material. The flowrate of fuel (at a constant flowrate of air) that is necessary to maintain the reaction chamber at a constant temperature is then measured and used as an indicator of inferior calorific value.

Catalytic combustion instruments of the types described in this subclause^{[8], [9]} are at an advanced stage of development, but are not yet commercially available; their performance capabilities, therefore, cannot yet be assessed.

4.4 Inferential methods

The dividing line between "indirect" and "inferential" methods is rather indistinct. It can logically be argued that all determinations of calorific value are, in some sense of the word, inferential. Here, inferential methods are taken to be those that depend upon an empirical (or possibly semi-empirical) correlation between calorific value and some other measured property or properties.

Examples of relevant properties that may be used in this way as predictors of calorific value include compression factor (which is related to calorific value by means of the SGERG-88 equation^[10]) and speed of sound. Neither of these properties alone is sufficient to determine calorific value unambiguously (further information concerning the inert constituents is needed), and no commercial device has yet been produced to exploit such correlations. Nevertheless, the great precision with which speed of sound can readily be measured suggests a possible future role for a method based on this principle.

For the present, however, instruments that can best be classed as inferential are much less sophisticated, both in principle and in construction. In typical instruments of this kind, a supposedly constant proportion of the heat released by the combustion of a regulated flow of fuel gas is sensed (but not measured) by some particular device and related empirically to the calorific value.

In one long-established implementation of this principle (Reference [2], Chapter 10 and Reference [6], Chapter 6), the sensing device is the burner chimney itself, formed from two concentric metal tubes joined rigidly at the bottom; the two tubes expand differentially by an amount that depends on the heat transferred from the flue gases, and this may be used to give an indication of calorific value. In another implementation^[4], the sensor is a single metal thermal-expansion tube located in the effluent gas stream; and in a slightly more modern implementation, it is a similarly located thermopile, the output of which is taken as an indication of calorific value. In none of these instruments is the water of combustion condensed; consequently all, in principle, respond to the inferior calorific value.

As a consequence of their acknowledged simplicity, instruments of this general type cannot usually be expected to achieve better than class 3 performance except in the most favourable of circumstances.

Very many other principles of operation have been described over the years, but it is not the intention here to describe ideas which no longer find, have never found, or are unlikely to find reasonably widespread application. There are countless “dead-end” patents.

5 Performance assessment and acceptance tests

The flowchart given as Figure 1 provides an overview of the procedures that it is typically necessary to carry out in order to satisfy performance assessment and acceptance testing requirements. Specific details are provided in 5.1 and 5.2.

5.1 Performance assessment for instrument selection

5.1.1 General

For any application, it is necessary that an instrument for the measurement of calorific value meet some criteria of acceptability. Two common forms that these criteria may take for a commercially available instrument are

- a) a set of requirements that shall be met in order for the instrument to receive type-approval; in some cases, this can be issued by a statutory body responsible for the supervision of custody transfer or customer charging, and
- b) a technical specification for purchase contract purposes.

Annex B gives an example of a typical type-approval specification (Clause B.1) and of a typical technical specification forming part of the purchase documentation (Clause B.2).

5.1 relates to performance assessment tests that it is typically necessary to carry out on (usually) a single instrument as an exemplar of its type. In 5.1.2 to 5.1.13 are considered the factors that are those most often specified in a formal set of requirements, such as those referred to in this subclause. A purposeful test programme is likely to include the investigation of most, if not all, of these aspects of the instrumental performance.

The test programme shall include a specification for the calibration and other test gases that are required to carry out many aspects of the detailed testing and calibration, although some test gases might not require certification. Furthermore, any type-approval documentation issued as a result of the test programme shall include a specification for the gases for use on-site in the re-calibration and verification procedures, so as to ensure attainment and maintenance of the specified accuracy.

Instruments of all types considered in this International Standard generally perform optimally when left in continuous on-line operation; requirements calling for only intermittent or off-line operation demand special care and extra pre-testing (see 5.1.3).

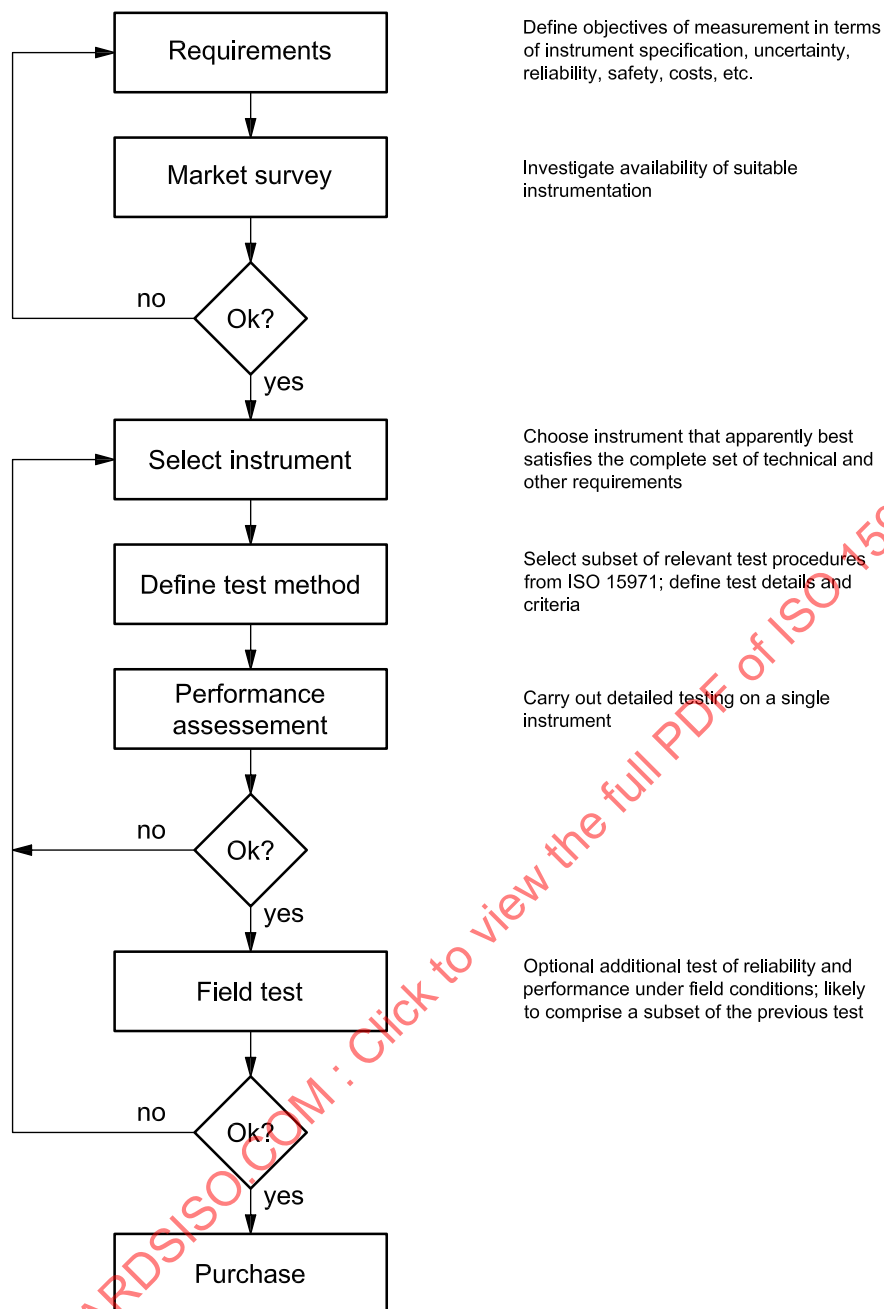


Figure 1 — Instrument evaluation, performance assessment and acceptance testing

Depending on the particular circumstances, performance assessment testing may be carried out by a regulatory authority, by an independent, accredited testing laboratory and/or by the purchaser. If carried out by a regulatory authority, the tests may lead to type-approval documentation. There is a clear trade-off between the length of time spent on a series of performance tests and the amount of detail that they yield about an instrument. Where a regulatory authority requires a thorough characterization of all aspects of performance, the test programme can easily extend over a period of one year or more.

A pro-forma checklist of the type given as Table 1 can be a useful means of keeping track of the progress of, and results from, a lengthy evaluation programme.

5.1.2 Continuity of operation

It is likely that an explicit requirement be for the instrument to be in-service and to operate correctly (and, for most applications, continuously) for a specified period of time. If the particular application requires intermittent operation only, then the trueness tests (see 5.1.3) should address this complicating factor as a priority before proceeding with other tests.

In the case of continuous operation, the instrument should be tested simply by letting it run without interruption or undue interference (such as unscheduled adjustments to settings) for a continuous period that exceeds the specified minimum required operating period by a specified percentage. Depending on the application, this may be anywhere from a few days to several months. It can, however, be possible to carry out other tests during this period, for example trueness and repeatability tests, without prejudice to the continuity (reliability) test.

Repeating the continuity (reliability) test, after routine maintenance in accordance with the manufacturer's instructions, at least once, is likely to be a worthwhile option. If the instrument cannot operate without breakdown or obvious malfunction for the specified period, then it fails this test.

The results from a completed test may be analysed in order to assess the period for which the specified instrument performance has been achieved. If this is less than the minimum for which correct operation is required, then the instrument has again failed. In this case, it can still be possible to use the instrument in applications where a shorter period of continuous satisfactory operation is acceptable.

If there is no explicit requirement for a minimum operating period, then it is possible to allow the frequency of maintenance operations to be determined by operational experience, i.e. the maintenance operations are performance-driven rather than requirement-driven.

Table 1 — Example of checklist for type-approval and acceptance testing

Property or test	Type approval testing			Factory acceptance test			On-site acceptance test		
	required	result	pass/fail	specified	result	pass/fail	specified	result	pass/fail
1 Continuity of operation continuous operation within maximum specified error of 0,25 MJ/m ³ test period	6 months	7 months	pass	1 month	1 month	pass	3 months	3 months	pass
2 Trueness of calorific value number of test gases range, MJ/m ³ linearity, MJ/m ³	7 31<CV<46 < 0,10			5 35<CV<42 < 0,10			application gases not tested		
3 Repeatability multiple readings type b) number of test readings spread, MJ/m ³	40 < 0,10			20 < 0,10			20 < 0,10		
4 Response to calorific value step-change step, MJ/m ³ 95 % response time	38 to 42 < 4 min			40 to 41 < 3 min			40 to 41 < 3 min		
		2 min 35 s	pass		1 min 53 s	pass		1 min 42 s	pass

Table 1 — Example of checklist for type-approval and acceptance testing (continued)

Property or test	Type approval testing			Factory acceptance test			On-site acceptance test		
	required	result	pass/fail	specified	result	pass/fail	specified	result	pass/fail
5 Temperature dependence maximum calorific-value error for temperature change									
5.1 Steady state range, °C error, MJ/m ³	15 to 30 < 0,10	0,13	fail	15 to 25 < 0,10	0,08	pass	natural variations (3 months) < 0,05 0,04 pass		
5.2 Dynamic range, °C rate, K/h error, MJ/m ³	15 to 30 5 < 0,20	0,14	pass	natural variations not tested			natural variations (3 months) < 0,05 0,04 pass		
6 Atmospheric pressure dependence range, hPa error MJ/m ³	970 to 1040 < 0,10	0,11	fail	natural variations < 0,05 0,05 pass			natural variations (3 months) < 0,05 0,03 pass		
7 Other environmental factors 7.1 Relative humidity interval range, % error, MJ/m ³ 7.2 Electrical supply variations voltage, AC error, MJ/m ³ frequency, Hz error, MJ/m ³ brown-out error MJ/m ³ 7.3 Electromagnetic interference and compatibility (EMC) conformance with ISO/IEC requirements standard tests	30 to 80 < 0,10 200 to 240 < 0,03 40 to 65 < 0,03 5 cycles at 100 VAC < 0,25	0,05 0,00 0,00 0,12	pass pass pass pass	natural variations < 0,10 0,05 pass 200 to 240 < 0,03 0,00 pass local site variations not tested local site variations not tested not applicable not tested			natural variations < 0,10 0,03 pass local site variations not tested local site variations not tested not applicable not tested		
8 Installation factors 8.1 Gas supply pressure range, kPa (mbar) error, MJ/m ³ 8.2 Air supply pressure range, kPa (mbar) error, MJ/m ³	0,5 to 4,0 (5 to 40) < 0,02 0,1 to 1,0 (1 to 10) < 0,02	0,01	pass fail	1,0 to 4,0 (10 to 40) < 0,02 < 0,02	0,01 0,00	pass pass	1,0 to 4,0 (10 to 40) < 0,02 0,3 to 0,5 (3 to 5) < 0,02	0,00 0,00	pass pass

Table 1 — Example of checklist for type-approval and acceptance testing (*continued*)

Property or test	Type approval testing			Factory acceptance test			On-site acceptance test		
	required	result	pass/fail	specified	result	pass/fail	specified	result	pass/fail
8.3 Sample clearing time clearing time		not tested			not tested		< 3 mins	2 min 15 s	pass
8.4 Air movement (draughts) error, MJ/m ³					no require- ment			not applicable	
9 Effect of non-alkane gases test gas A: 25 mol % H ₂ nominal actual composition: 0,703 CH ₄ , 0,267 H ₂ , 0,022 C ₂ H ₆ , 0,008 C ₃ H ₈ CV = 31,98 MJ/m ³ (ISO) error, MJ/m ³ test gas B: ___mol % C ₂ H ₄ nominal	< 0,04	0,02	pass		not tested no require- ment		< 0,04	0,02	pass
10 Integrity of computed average values error, MJ/m ³ - per hour - per day	 < 0,001 < 0,001	 0,000 0,000	 pass pass	 < 0,001 < 0,001	 0,000 0,000	 pass pass	 < 0,001 < 0,001	 0,000 0,000	 pass pass
11 Security of instrument and its record 11.1 Physical security keyswitch/password 11.2 Electronic security power cut automatic restart retention of record			pass pass pass pass			pass pass pass pass			pass pass pass pass
12 Fail-safe operation power failure gas supply failure air supply failure flame failure			pass pass pass pass			pass pass pass pass			pass pass pass pass
NOTE Numerical values and other entries are entirely notional and, in particular, do not correspond to those that can be devised for any real set of tests for any specific type of calorific-value measuring device.									

5.1.3 Trueness of calorific value

The requirement is likely to be for the instrument to measure calorific values to within a specified value of trueness, or bias, over a specified range of calorific values (systematic error analysis).

Usually, these tests refer to an instrument left in continuous operation. However, if the particular application requires intermittent operation only, then it is important that the tests include a preliminary investigation in which an assessment is made of the length of time (from “cold”) during which it is necessary for the instrument

to be in operation before it is demonstrably able to produce “settled” results; for direct-measurement instruments, this period of “warm-up drift” may be as long as several days. The remainder of this subclause assumes that the warm-up period has been exceeded.

In order to carry out the trueness assessment, it is necessary to have available a number (typically 5 to 7) of test gases of certified (traceable) calorific value and uncertainty, one of which conforms to the requirements of the (single) calibration gas (often high-purity methane) for use in the routine operation of the instrument. These gases are normally synthetic mixtures of the major components of natural gas that the instrument is expected to analyse and, insofar as possible, similar in composition to that natural gas. The range of calorific values covered should be somewhat wider than the operational range given in the requirements; it is normal for one of the test gases to have a calorific value below the minimum value of the operational range and for one to have a calorific value above the maximum value of the operational range.

Tests carried out with a set of gases such as these (using the routine calibration gas as a datum) enable the assessment not only of the trueness of the recorded calorific value at each point, but also of the linearity of the instrumental response function. Tests for each gas should typically be carried out a number of times so as to obtain a representative average value (this relates to the repeatability of readings; see 5.1.4).

Further tests of trueness should normally be carried out using at least three test gases of substantially different compositions but having approximately the same calorific value. This serves as a check that the instrumental trueness is not a function of the composition of the gas. Where large changes in gas composition are expected in operational use, it can be useful to use more than three test gases to check for compositional effects.

All trueness tests should be carried out at a very early stage of a typical test programme; it is advisable to repeat the tests at intervals during, and at the conclusion of, the continuity (reliability) test (see 5.1.2).

The gases used for the trueness tests should have uncertainties on the certified calorific values that are less than half the accuracy requirement for the operation of the instrument.

The results of the tests should be analysed to determine the trueness of the measured calorific value over the specified range of calorific values.

5.1.4 Repeatability

There are at least three distinct concepts of repeatability (random error analysis) that can be identified in the context of calorific-value measurement, each of which satisfies the proper metrological criteria for classification as repeatability. Each may be quantified by appropriate tests.

- a) Independent readings are taken at fixed intervals of time with the instrument running continuously on the same test gas and with no calibration during the entire test period.
- b) Independent readings are taken at fixed intervals of time with the instrument running continuously, but switched to run on another gas (e.g. line gas) between each pair of readings, and with no calibration during the entire test period. In this case, it is necessary to give the instrument time to “settle” after each switching to the test gas before the reading is taken.
- c) Independent readings are taken at fixed intervals of time with the instrument running continuously, but with a single point calibration carried out between each pair of readings. It is necessary to give the instrument time to “settle” before each reading is taken. (For the purpose of this test, it can be necessary to reduce the interval between calibrations below that used under operational conditions.)

The results of the tests may be analysed by calculating the standard deviation on the respective data sets and comparing the results with the specified requirements.

For each type of test, the most relevant test gas to use is the one closest in calorific value to that of the gas expected to be received in normal operation. However, it is not necessary that the test gas for this application be carefully certified. All that is required is a gas of reliably constant and roughly known calorific value.

The relevant “fixed intervals of time” and the number of readings in a repeatability test depend very much on the operational characteristics of the instrument. Many more measurements can conveniently be taken with modern rapid-response devices than can be taken in the same period with traditional direct-measurement calorimeters, which typically exhibit rather sluggish response (see 5.1.5). Typically, for a rapid-response device, the time interval is measured in minutes, with each reading the average over 1 min or 2 min of measurement whereas, for a traditional calorimeter, the time interval can be 1 h, with each reading the average of perhaps half-an-hour's measurements.

There is likely to be an operational requirement for the instrument to measure calorific values within a specified repeatability, for type a), type b) or type c) measurements or, possibly, for more than one type.

It is normal for at least one additional set of repeatability tests to be carried out on a second gas, preferably at the same time as the trueness tests, so as to investigate whether the repeatability is a function of calorific value. Further repeatability tests should be carried out at regular intervals (e.g. two months to three months) in order to determine whether the repeatability varies with time.

5.1.5 Response time

The requirement is likely to be for the instrument to respond, within a specified time, either completely or to within a specified percentage of completion, to a step-change in calorific value.

Tests for this characteristic may be carried out by switching between two test gases of different calorific value and monitoring the output while the instrument continues to run normally. For this purpose, it is not necessary that the test gases be of accurately known calorific value. Several tests should be carried out with changes of calorific value in both the upward and downward direction.

There are a number of factors to bear in mind when performing tests of this type, namely the following.

- a) In many cases, it might not be usual for step-changes of calorific value to occur in operational practice, so this test, then, does not properly mimic operational reality; however, if step-changes (or near step-changes) are expected in field operation, then the test gases used should, by preference, be such as to mimic these changes.
- b) The response time may depend strongly on the magnitude of the change in calorific value, so that tests on more than one pair of test gases can be useful.
- c) The tests should be designed to record any overshoot (under-damping) of the correct calorific value.
- d) Some instruments update their display at fixed intervals of time rather than continuously and, therefore, the response proceeds in discrete steps rather than smoothly. In this case, the user should recognize and take this effect into account and, if the reporting interval is user-selectable, it should be set to the minimum available for the duration of the tests.
- e) Steps should be taken to ensure that the response time measured is the fundamental characteristic of the instrument itself and is not a measure of any delay (clearing time) caused by the design of the gas-supply system for the instrument, i.e. an installation effect (see 5.1.9).

The results of these kinds of test may be expressed in various ways. If the characteristic response to a step-change in calorific value roughly follows a logarithmic equation, the calorific value, $H(t)$, reported by the instrument at time, t , after a step-change in calorific value of $[H(\infty) - H(0)]$ at time $t = 0$, can be expressed as given in Equation (1):

$$H(t) - H(0) = [H(\infty) - H(0)] [1 - e^{-t/\tau}] \quad (1)$$

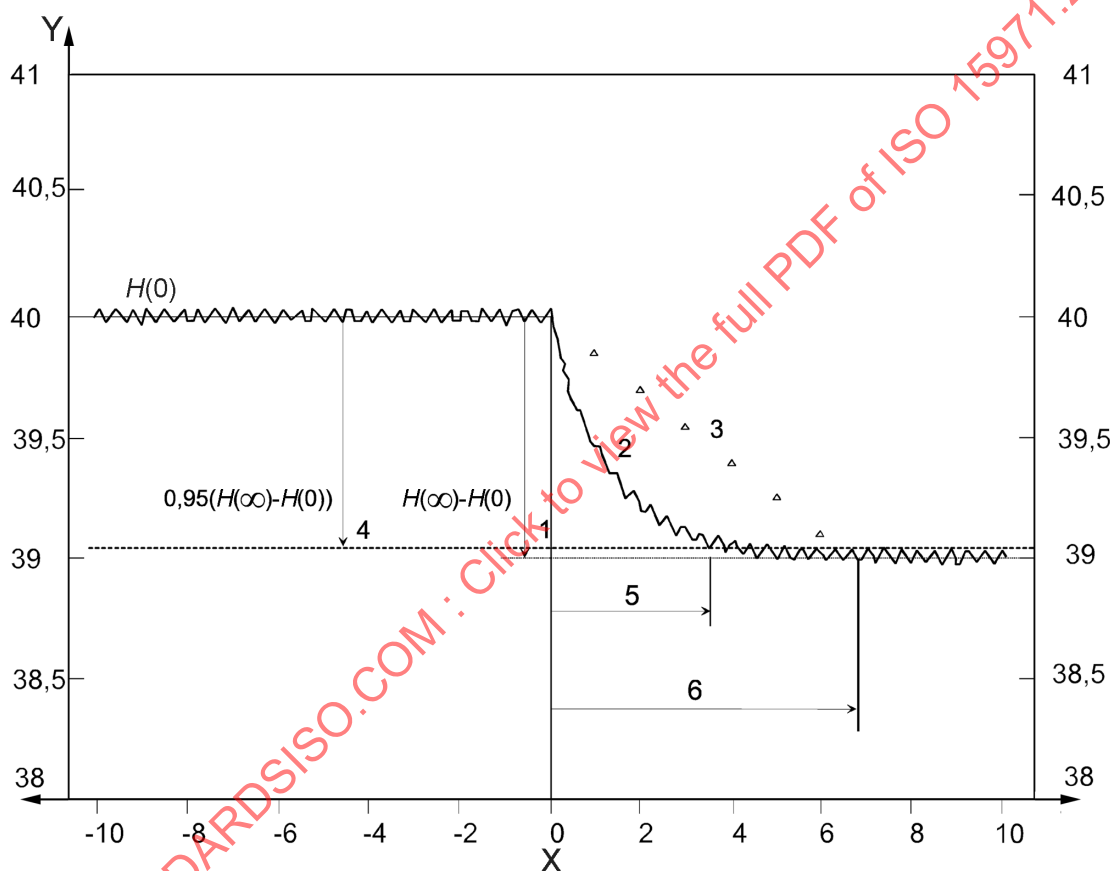
where the observed value of τ (the relaxation time) is equal to the time taken to reach 63 % completion.

Not all instruments exhibit exponential behaviour. In either case, the time taken to achieve 95 % completion can be considered as a more convenient measure of the speed of response. Figure 2 illustrates the typical response characteristic for the two types of calorific-value record, namely

- a) for a continuous-reading instrument (device A),
- b) for a discrete-reading instrument, i.e. the output value is updated at finite but regular intervals (device B).

In both cases, the use of the 95 % completion as the preferred response time characteristic is emphasized.

Modern indirect instruments generally exhibit response times in the range from several seconds up to a few minutes, whereas traditional direct-measurement recording calorimeters have response times generally in the region of tens of minutes.



Key

- X time, expressed in minutes
- Y calorific value, expressed in megajoules per cubic metre
- 1 true calorific value
- 2 device A (continuous reading)
- 3 device B (discrete reading)
- 4 95 % completion
- 5 response time for device A
- 6 response time for device B

Figure 2 — Typical response of instrumental record to a step-change in calorific value

5.1.6 Temperature dependence

5.1.6.1 General

Normally, there are likely to be two requirements, one concerning the response of an instrument when the change in ambient temperature is significant but takes place very slowly (steady-state performance), the other concerning the response when the change in ambient temperature takes place relatively rapidly (dynamic performance). In the first case, the requirement is likely to be either

- a) to demonstrate that the instrument measures calorific values to within specified uncertainties when operated within a specified range of steady ambient temperatures, or
- b) to define the operational range of steady ambient temperatures within which the instrument measures calorific values to within specified uncertainties.

In the second case, the requirement is likely to be to define a maximum value for the rate of change of ambient temperature which allows the instrument to operate without significant degradation of the result.

5.1.6.2 Steady-state performance

The steady-state temperature dependence of the instrument can conveniently be assessed by running it in an environmentally controlled test room capable of achieving the relevant range of temperatures, at a series of fixed (steady) temperatures within and at the extremities of this range.

By preference, it should be possible to achieve the required temperature control without the imposition of excessive air currents in the room, as these can give rise to adverse effects on instrumental performance (see 5.1.9) that mask the actual effects of temperature variation. Relatively simple tests, carried out with the instrument operating on a gas of constant but not necessarily certified calorific value, readily demonstrate any direct, steady-state functional dependence of recorded calorific value on temperature.

5.1.6.3 Dynamic performance

Some instruments have been observed to exhibit either or both of two rather more subtle effects, the possibility of which should also be considered in the evaluation programme. Both are more likely to occur for direct-measurement calorimeters, as for these it can be difficult, or deemed inappropriate, to remove them electronically.

Firstly, an instrument can exhibit a transient dependence upon temperature, i.e. a dynamic change in recorded calorific value that occurs while the instrument is not in thermal equilibrium following a change in temperature, but which dies away as a new thermal equilibrium condition is approached.

Secondly, there can be a dependence of the recorded calorific value on the rate of change of temperature as well as its absolute value.

The two effects are usually interlinked and their experimental investigation is not easy; one reason for this is the rarity of test rooms in which the rate of change of temperature is controllable. For the investigation of these two effects in particular, both upward and downward changes of temperature should be carried out.

5.1.6.4 Summary

Any or all of the various forms of temperature dependence can be significant if an instrument is required to operate in a building that has a heating system but no air conditioning, or poor-quality stepwise (on-off-on) air-conditioning control; in the worst cases, the ambient temperature range in the instrument room or enclosure can be as wide as 280 K to 330 K, while rates of temperature change of up to ± 5 K/h can occur.

Tests should be carried out to assess the systematic effects of temperature on the accuracy of the measured calorific value.

In addition to causing bias, variations of ambient temperature can also cause corresponding variations of repeatability (see 5.1.4). For this reason, the repeatability should be determined at several different temperatures within the operating range and both these factors should be taken into consideration when estimating the overall uncertainty for any set temperature.

The results should be analysed so as to establish

- a) the temperature range, and
- b) the rates of temperature change over which the instrument satisfies the specific requirements of the application.

5.1.7 Atmospheric pressure dependence

The requirement is likely to be for the instrument to measure calorific values to within specified uncertainties over the range of atmospheric pressure (and its maximum rate of change) likely observed at the site(s) where the instrument is to be installed. Local atmospheric-pressure records covering the last few years should be sufficient to establish the operational envelope.

Specific tests of the instrument's functionality with respect to atmospheric pressure cannot be carried out. Instead, the instrument should be left to operate on a gas of constant but uncertified calorific value (or possibly a set of gases, but not line gas) for a substantial period of time during which the atmospheric pressure varies by a substantial amount; for example, deep depressions are particularly valuable. During this period, it is necessary to log a sufficient number of readings of recorded calorific value and atmospheric pressure (perhaps hourly) in order to allow any correlation between the two to be both detectable and quantifiable. A graphical time-series plot is generally adequate.

The instrument fails its evaluation test if the effect of variations in atmospheric pressure is to degrade the trueness of the recorded calorific value beyond a specified limit.

5.1.8 Other environmental factors

5.1.8.1 General

Various other environmental factors have the potential to degrade the trueness of measurements of calorific value. There is likely to be a requirement to guarantee either that, in normal operation, the instrument is not exposed to any such factor, or that the particular response of the instrument is within specified limits. The factors in 5.1.8.2 to 5.1.8.5 can be identified.

5.1.8.2 Relative humidity

The requirement in this case depends on the range of relative humidity expected in the environment within which the instrument is required to operate. Even in a good-quality environmental test facility, it is difficult to vary the relative humidity systematically without inducing a secondary effect on the ambient temperature. Consequently, any direct effect of variation in relative humidity is inherently difficult to investigate, but the principles by which to approach this are similar to those for investigating the effect of variation of atmospheric pressure (see 5.1.7).

5.1.8.3 Electrical supply

The requirement in this case is likely to be that the instrument continues to function, without discernible degradation of the calorific-value measurement, over the complete range of supply-voltage and frequency variations expected at the installation site. In the case of voltage, at least, a test of the instrument's performance in this respect is easy to set up. In addition, the instrument should be robust against electrical spiking and against short (i.e., a few cycles) losses of power (brown-out). In the case where continuous operation is required at a site prone to electrical failure, consideration may be given to the installation of an uninterruptible power supply or an automatic back-up power facility.

5.1.8.4 Electromagnetic interference and compatibility

These factors should be investigated in accordance with methods given in relevant standards (see IEC 61000-6-1, IEC 61000-6-2, IEC 61000-6-3 and IEC 61000-6-4) and appropriate certification shall be obtained. Tests should be designed in order to establish the upper limit of electromagnetic disturbance compatible with acceptable performance of the instrument; this implies that, during the relevant tests, the instrument shall be fully operational and giving a live output.

5.1.8.5 Air cleanliness

It is generally important that air for combustion (and for other instrumental purposes) should be of an appropriate quality. Specific tests to investigate the effects of poor-quality air are difficult to set up but, depending on the particular circumstances, can be deemed necessary if there is no guarantee of good-quality air; requirements on air quality are discussed more fully in 6.2.1.

5.1.9 Installation factors

5.1.9.1 General

Various installation factors have the potential to degrade the trueness of measurements of calorific value. There is likely to be a requirement to guarantee either that, in normal operation, the instrument is not exposed to any such factor, or that the particular response of the instrument is within specified limits. The factors in 5.1.9.2 to 5.1.9.4 may be identified.

5.1.9.2 Gas and air supply pressures

In some cases, it is necessary to supply the gas to the instrument at distribution line-gas pressure (i.e. a few kPa above ambient pressure) whereas, in other cases, very much higher, regulated pressures (specified by the manufacturer) are necessary. There can be a similar requirement on the supply pressure for any air required for combustion and/or instrument operation. Either way, an assessment should be carried out to determine the response of the instrument to variations of the relevant supply pressure(s) from the nominal value(s). The tests can usefully cover the ranges from 50 % to 200 % of the nominal values, with each being varied independently of the other.

5.1.9.3 Sample clearing time

Any instrument can make measurements only on the gas that is presented at its sample inlet; it is, therefore, important that the clearing time of the sample line between the original sampling point and the instrument's sample inlet be short compared with the time scale during which changes in calorific value are expected. It may, however, be sufficient to carry out calculations instead of tests on this item [see 6.1 a) to 6.1 c) for a fuller discussion].

5.1.9.4 Air movement (draughts)

Some instruments, particularly those where temperature is measured as a fundamental part of their operation (e.g. direct-measurement calorimeters), can be sensitive to excessive air movement in their immediate vicinity. In an air-conditioned environment, air movement is, to a greater or lesser extent, inevitable. Tests may be carried out on the effects of air movement on the performance of an instrument by the local introduction of forced draughts using a suitable fan; because of the thermal inertia, it should not be expected that any instrument response to a forced draught take place immediately. For some of the smaller instruments, it can be practicable to avoid any problem of this sort by use of a ventilated, protective cabinet or other enclosure.

5.1.10 Effects of non-alkane gases

5.1.10.1 Stoichiometric combustion

For some instruments, the principle of operation (i.e. the underlying theory or correlation) relies on the knowledge that particular components (e.g. oxygen) or groups of components (e.g. alkenes) are absent from the sample gas. Although these components are not normally found in natural gas, they can be found in substitute natural gases and other mixtures that are interchangeable with natural gas (e.g. propane-air). Consequently, if the instrument can be exposed to more than trace amounts of such non-alkane components (interferents), it is necessary to assess to what extent its function is impaired by their presence. There is likely to be a requirement that the instrument remain within its trueness specification even with (named) interferents present at up to specified mole fractions. Alternatively, a guarantee of the absence of interferent components can be sufficient.

Instruments of this type do not exhibit the proper response to a change in calorific value if that change is accompanied by changes in the mole fraction of any combustible non-alkane component or oxygen.

The effects of non-alkane combustibles can be calculated, to a good approximation, from theoretical considerations (see Annex F). In some instances, this can be an acceptable alternative to elaborate tests involving the preparation and use of certified gases containing one or more interferent.

5.1.10.2 Inferential instruments

For instruments whose primary determination is of the inferior calorific value, there exists the possibility of a different non-alkane effect. This occurs only if the measured inferior calorific value is used to infer (either by multiplying by a constant factor or, equivalently, falsely calibrating on a superior calorific value) the superior calorific value (see 4.1).

In this case, the problematic components are hydrogen, non-alkane hydrocarbons and, most seriously, carbon monoxide. The presence of these can cause incorrect results. This occurs because the relationship (percentage difference) between the inferior and superior calorific values for these components differs from that of the alkane hydrocarbons. For example, for carbon monoxide, the inferior and superior calorific values are equal, whereas for the alkanes the former is about 10 % lower than the latter.

For a mixture containing a mole fraction, x , of any non-alkane component (with the balance being methane), it can be shown that the error, ΔH , in determining superior calorific value by this method is given by Equation (2):

$$\Delta H = x(1,11 \cdot H_I - H_S) \quad (2)$$

The factor 1,11 arises as the quotient H_S/H_I of the superior and inferior calorific values for methane at standard reference conditions. A mixture of 0,2 mole fraction of carbon monoxide in methane, for example, results in an error of 0,26 MJ/m³. Values for H_S and H_I for individual components are given in ISO 6976.

Most inferential instruments exhibit this effect and it can be relevant to assess its impact on any application calling for the measurement of superior calorific value of gases likely to contain significant amounts of combustible, non-alkane components.

5.1.11 Integrity of computed average values

For some applications, there is likely to be a requirement for the instrument to report the calorific values as averages based on prescribed time intervals (e.g. hourly, daily, weekly, monthly).

It is clearly a fundamental requirement that the desired averages are correctly calculated from the relevant set of "instantaneous" calorific values measured by the instrument. Consequently, the assessment programme should include a validation of the way in which specific interval averages are calculated by the instrument's software from the (semi-)continuous record, and/or a thorough check (by manual calculation) that the average values reported are correct.

5.1.12 Security of the instrument and its record

5.1.12.1 General

For some applications there is likely to be a requirement that the instrument and its record are secure against unauthorized interference. It is necessary, then, that the assessment programme address the factors in 5.1.12.2 and 5.1.12.3.

5.1.12.2 Physical security

The requirement is likely to be that access to any adjustment device can be denied, by physical isolation, sealing or otherwise. For example, the air-to-gas ratio adjustment on a commonly used form of recording calorimeter is a critical setting, interference with which can be prevented either by sealing the adjustment device or by installing a lockable cover. Adjustment devices that it can be necessary to re-set during calibration fall within the general requirement, but it is necessary to provide access to these for personnel authorized to carry out any routine, non-automated calibration procedure, and access to all parts of the instrument can be required during maintenance operations. In some cases, operating the instrument in a locked room can represent sufficient physical security.

5.1.12.3 Electronic security

Any electronic device linked to the instrument during field operation should be capable of requesting only the transmission of data and the reading of these from the instrument (interrogation); it should not be capable of any other two-way transmission, as this opens up the possibility of alteration of software parameters, corruption of stored data, etc.

5.1.13 Fail-safe operation

There is likely to be a requirement, at least when the intention is for use in continuous and unattended operation, that the instrument shut down to a safe condition (which shall include isolation of the gas supply to the instrument and reporting of the shutdown to an external alarm), in a number of potentially hazardous circumstances within the instrument. These normally include

- a) extinguishment of the main burner flame,
- b) extinguishment of any pilot burner flame,
- c) loss of electrical supply,
- d) recorded calorific value outside of pre-set shutdown limits.

Note that "shutdown" limits may be different from "alarm" limits, and that the general shutdown requirement may apply only to the instrument and not necessarily to the entire installation facility.

It is normally quite straightforward to test the satisfactory operation of the fail-safe mechanisms by the simple simulation of these conditions. It can also be useful to test the response of the instrument to these conditions in the situation that it is inadvertently left in a "fail-safe override" mode.

In the more modern instruments, it is usual also to have several "invisible" operating parameters (e.g. temperatures, flowrates) under the automatic surveillance of an on-board microprocessor. In this case, the instrument can shut itself down for a wider variety of reasons (usually some operating parameter outside of pre-set limits) than those that the microprocessor is normally able to report to an operator. It is impracticable to test all of these functions.

For several types of instrument, there is an option to allow one or more attempts at an automatic restart in the event, for example, that the gas or electricity supply is restored after a failure. Clearly, it is possible to simulate this situation and thereby test this aspect of the instrument's performance.

5.2 Factory and site acceptance tests

5.2.1 General

In 5.2 are considered the tests that should be carried out on each individual instrument, or at least a representative sample of each type of instrument, in order to confirm that each conforms in construction, performance, etc., with the instrument specified and type-tested according to the provisions detailed in 5.1.2 to 5.1.13.

For each of the three distinct aspects of acceptance testing, there shall be a formal procedure. The flowchart given as Figure 3 provides a summary of the various procedures which shall be carried out.

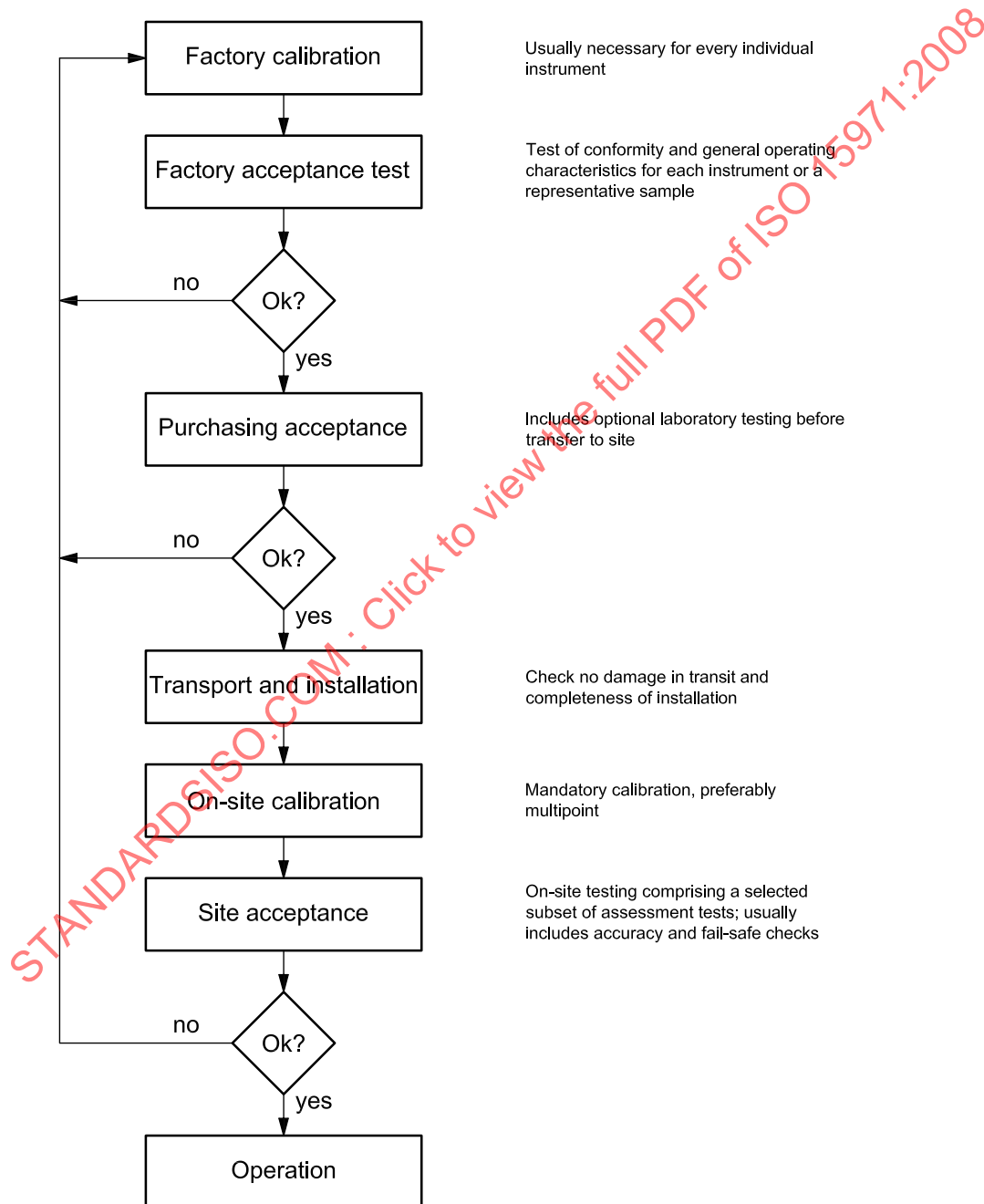


Figure 3 — Factory and site acceptance testing

5.2.2 Factory acceptance tests

Factory acceptance testing is normally carried out in such a way as to demonstrate the conformity of instrument manufacture, set-up and general operational characteristics to that of the instrument(s) used for the performance assessment tests. Witnessing of an appropriate and agreed set of factory acceptance tests, either by the purchaser or by an authorized body, is likely an important feature of the procedure.

The results of factory acceptance testing of this kind can form part of the documentation that the purchaser requires the supplier to provide.

5.2.3 Purchasing acceptance

Factory acceptance tests witnessed by a representative of the purchaser can be used as one part of a purchasing-acceptance schedule. However, there can be additional technical requirements and/or tests, that it is necessary to check or to carry out in the purchaser's laboratory.

Other than this, purchasing acceptance is likely to be largely an exercise in the provision, handling and checking of the proper documentation.

5.2.4 Site acceptance tests

The purpose of site acceptance testing is twofold: first, to check that an installation is complete; second, to demonstrate that each individual instrument is capable of operating within an agreed envelope of satisfactory performance, for the whole range of expected operational conditions. Site acceptance tests shall normally be carried out by the purchaser and, in general, their content depends primarily on the results gained in the performance assessment tests.

These tests should, therefore, follow a test programme designed, in particular, to address and reflect any concerns about specific aspects of the detailed performance assessment tests where satisfactory performance is close to the edge of the instrument's capability, or where there is reason to believe that performance can sensibly vary from instrument to instrument. In this sense, the tests should comprise a purposeful sub-set of the tests described in 5.1. Tests specific to the particular location can also be necessary; this can be the case, for example, when any of the relevant environmental factors is expected to show extreme variations.

As an absolute minimum, there shall be a single-point calibration, an accuracy test using a test gas of traceable calorific value and a check on the fail-safe mechanisms. A multipoint calibration, if possible, is strongly recommended.

In addition, it can be useful to run another (proven) instrument in parallel with the new instrument for a substantial period and/or to take regular samples of the gas being analysed by the instrument for independent laboratory determination of calorific value.

6 Sampling and installation guidelines

6.1 Sampling

All of the instruments to which this International Standard applies are mainly intended for use as on-line devices, within the sense of that term in accordance with ISO 14532. Because, however, different types of instrument can have widely differing requirements for the gas-supply pressure and flowrate, it is not possible to give specific requirements for the sampling system that apply to all instruments. Nevertheless, the general provisions of those parts of ISO 10715 that relate to direct (continuous) sampling may be taken to apply, and the following general guidelines can be given.

- a) The gas-supply line should follow the shortest practicable route from the original sampling point, and the internal diameter of the line should be as small as possible (typically 3 mm or 6 mm) without offering too

great a viscous restriction to flow. These considerations relate to the minimization of the volume and hence clearing (residence) time for the line.

- b) It might not be easy to make tests of the clearing time for the sample line, and calculations based on the volume of the sample line and the rate of gas usage by the instrument can be a more practicable approach to resolving this question.
- c) If the rate of gas usage is low compared with the hold-up in the sample line, then it can be necessary to give consideration to the introduction of a bleed-off in the sample line; typically, this takes the form of a suitably sized purge system installed close to the gas sample inlet of the instrument, with the purge gas either being dissipated through a flame trap to the external atmosphere, or burnt externally.
- d) Any substantial pressure reductions required between the sampling point and the instrument's gas-supply inlet should take place in stages as close to the original sampling point as practicable. The pressure-reduction valve and local pipework shall be heated by a (calculated) amount sufficient to prevent cooling (and hence condensation of components from the sample gas) due to gas expansion or exposure.
- e) For the remainder of the pipework, after the final pressure reduction and up to the sample inlet of the instrument, it can be necessary to apply further heating and/or insulation to counteract the effects of exposure to cold conditions or wind.
- f) The sample line should be of seamless stainless steel with as few welded joints as possible. The system shall be leak-tested regularly, preferably at the time of instrument maintenance.
- g) The sample line shall include a suitable filter to guard against the ingress of dust and other foreign materials.
- h) All valves, regulators and other fittings in the sample line shall be internally free of grease and other contaminants.

Occasionally, the particular application can require an instrument being used for off-line spot sampling. In this case, the user should be aware that this requires that the instrument be run for a period greater than its possibly lengthy "warm-up" time (see 5.1.3) before reliable results can be obtained and that, partially for this reason, the application is likely to demand the availability of rather large samples. The guidelines a) to h) given above remain applicable.

6.2 Installation guidelines

6.2.1 Combustion calorimeters

These instruments (usually known as recording calorimeters) rely on heat exchange and thermometric measurements and, for reasons related to this, can exhibit a dependence of the recorded calorific value on ambient temperature. For any particular type of recording calorimeter, the exact magnitude of any dependence on temperature can vary slightly from instrument to instrument as a result of minor constructional differences.

The generic characteristics of the type should, nevertheless, have been established by tests carried out in accordance with 5.1.6, and from this information it should be possible to determine the temperature stability necessary to achieve measurements that remain within the specified trueness.

For many existing installations, the specified requirement (for class 1 performance) is that the temperature remain within a band of ± 2 K around a stipulated set temperature. It is usually necessary that the set temperature itself be within a specified range, for example 288 K to 293 K. There can also be a requirement for the maximum allowable rate of change of the temperature. All of these factors have implications for the operational characteristics of the air-conditioning system(s) that can be needed to control the environment of the calorimeter room.

The calorimeter should always be sited where exposure to direct sunlight is impossible, and away from walls that can be exposed to wind. An "internal" room is preferable. No sources or sinks of heat (including other instruments) should be placed in close proximity to the calorimeter; steam and/or hot water pipes should not run close to the calorimeter. Fluorescent electric lighting, preferably left on permanently, is recommended.

Recording calorimeters normally take the air required for combustion from within the calorimeter room; consequently, the calorimeter room air should not contain combustible (fuel) components, as these can erroneously enhance the recorded calorific value. There is often a stipulated figure (usually quoted in $\mu\text{mol/mol}$) for the maximum allowable concentration of combustible gases in the room air.

Table 2 gives the approximate mole fractions of various combustible components that, if present in the room air, cause an enhancement of recorded calorific value of 20 % of the accuracy specification for class 1, class 2 and class 3 performance. The table is based on a typical value for the combustion air-to-gas ratio of 14:1.

Table 2 — Mole fractions of components in room air leading to enhancements of 20 % of the accuracy specification for typical combustion calorimeters^a

Component	Mole fraction $\mu\text{mol/mol}$		
	Class 1	Class 2	Class 3
Hydrogen	120	240	590
Methane	38	76	190
Ethane	22	43	110
Propane	15	30	76
Butane	12	23	59
Pentane	10	19	48
^a For an air-to-gas ratio of 14:1.			

In addition, the room air should not become vitiated by inefficient removal of combustion products. It is usually considered necessary to provide a forced supply of fresh air into the calorimeter room. The ventilation required to maintain the carbon dioxide level below a specified level (e.g. 0,15 %) can be calculated^[11]; alternatively, a minimum number of fresh air changes per hour may be stipulated, with periodic testing of the carbon dioxide level to ensure compliance.

The atmosphere should also be sufficiently clean and free of dust that it is not necessary to change air filters in the air supply ducting between maintenance operations; if the room air is not filtered, a build-up of dust within the instrument can result in a steady drift of the record.

Finally, the whole installation should be so sited that it is beyond the range of vibrations from heavy traffic or machinery, as this can affect the accuracy of any water-sealed metering device that forms part of the instrument.

6.2.2 Stoichiometric combustion instruments

Measurements made with instruments of this type are, in general, not prone to dependence upon ambient temperature, although a weak dependence can, in some cases, be observed. Thus, there is not normally a requirement to maintain the instrument in a thermo-stable environment.

However, some instruments of this type require the supply of both gas and air at stable, elevated pressures (typically a few hundred kilopascals), and the means of providing this regulation can give rise to a degree of temperature dependence. Tests carried out in accordance with 5.1.6 should have shown the extent of any temperature dependence and the consequent requirement for any temperature control.

Instruments of this type are demanding in their requirement for purity of air supply; indeed, they are in some respects less tolerant of impurities than combustion calorimeters. The following five potential problems can be identified.

- a) If the oxygen content of the air for combustion is vitiated by inefficient removal of combustion products, or any other external reason, then erroneous results are certain.
- b) If room air is used for combustion, then any large humidity variations inevitably cause the oxygen content to vary slightly; correspondingly variable results can then be observed. (Some instruments, however, dry the combustion air internally and thereby avoid this problem.)
- c) If the air for combustion is supplied from a cylinder of synthetic air, then there is a good chance that the oxygen content is sufficiently unreliable as to make the measurements equally unreliable. However, if the instrument is calibrated while operating on the same cylinder of synthetic air, compensation for this effect is made automatically (see also 7.1.4).
- d) If room air is used for combustion, then any combustible (fuel) components present can erroneously enhance the recorded calorific value, as is the case for combustion calorimeters (see 6.2.1). Table 3 gives the approximate mole fractions of various combustible components that, if present in the room air, cause an enhancement of recorded calorific value of 20 % of the accuracy specification for class 1, class 2 and class 3 performance.
- e) If the air for combustion is supplied through a compressed air system, then hydrocarbon vapours from oil in the compressor can be present. Efficient filtering, for example by the use of active carbon filters, can help to remove these, but there remains a possibility of erroneous enhancement of the calorific value from this source.

Table 3 — Mole fractions of components in room air leading to enhancements of 20 % of the accuracy specification for typical stoichiometric devices ^a

Component	Mole fraction μmol/mol		
	Class 1	Class 2	Class 3
Hydrogen	170	330	830
Methane	53	110	270
Ethane	30	61	150
Propane	21	43	110
Butane	16	33	82
Pentane	13	27	67
^a For an air-to-gas ratio of 10:1.			

6.2.3 Catalytic combustion instruments

As instruments of this type are not yet commercially available, no specific information can be given here.

6.2.4 Inferential instruments

Instruments of this type usually exhibit a strong sensitivity to both ambient temperature and pressure. In general, therefore, it is preferable to install any of the current range of inferential instruments in a temperature-controlled environment. In this case, as the instruments generally are rather smaller than combustion calorimeters, it can be possible to achieve this by use of a large thermostatic enclosure rather than a thermo-stable room.

However, if such a solution is considered too cumbersome just to achieve class 3 performance, then it is essential to record the ambient temperature and pressure on a continuous basis, in order that corrections to the record, based on some kind of site-specific calibration, can be made.

So far as other installation factors are concerned, the considerations relevant to this type of instrument are rather similar to those for combustion calorimeters. In particular, the performance of these instruments is affected by the presence of hydrocarbons in the air used for combustion but, since the air for combustion is always in excess, performance is unaffected by minor changes in oxygen content.

Nevertheless, neither the air quality nor the air-to-gas ratio should be allowed to vary by amounts that lead to significant variations in the thermal conductivity and/or heat capacity of the effluent gases, as these properties directly influence the efficiency of heat transfer to the sensor mechanism and thereby influence the measurement result.

7 Calibration

7.1 Calibration procedures

7.1.1 General

The calibration of an instrument for the routine measurement of calorific value is a corrective metrological procedure (see Note). It consists of using the calorific values recorded by the instrument for one or more certified reference gas mixtures (calibration gases; see 7.2) in order either

- a) to make adjustments to the instrument that reduce the difference between the measured and certified values to zero, or
- b) to derive a correction that, when applied to the measured value, produces the corrected value.

The actual process of adjustment or correction may be either manual or automatic, depending on the type of instrument.

The following principles apply to all types of calorific-value measurement apparatus and equipment currently in use. For most types of instruments, at separate times and different intervals, it is necessary to perform both multipoint and single-point calibrations.

NOTE In strict metrological terminology (see 3.4.7), the term calibration is not taken to include or imply the corresponding corrective procedure. For the purposes of this International Standard, however, and in particular to distinguish “calibration” from “verification”, calibration is taken to include a corrective procedure, either by applying an adjustment or by correction.

7.1.2 Multipoint calibration

Multipoint calibrations should cover the entire calorific-value working range of the instrument. They are usually carried out at long intervals of time (see 7.1.4).

Multipoint calibrations permit correction to the instrument sensitivity as well as to a specific single set point (datum). The single set point should be that of the calibration gas nearest in calorific value to the middle of the working range (see last paragraph of this subclause).

The process of multipoint calibration requires the sequential use of several calibration gas mixtures that span the range of calorific values for which the instrument is suitable. The number of gases required depends on the application of the instrument, but is unlikely to be more than five; normally three should suffice. The decision on this matter depends not only on the particular application but also on the linearity of the instrument, which should have been established during trueness testing (see 5.1.3). It is advisable for one calibration gas to be up to 1 MJ/m³ lower in calorific value than the minimum value expected in service, and one up to 1 MJ/m³ higher than the maximum value.

The calorific values measured for all the calibration gases are compared to the certified values. If this comparison satisfies user-defined and predetermined acceptance criteria, then the instrument is certified as remaining within calibration. Otherwise, adjustments may be made or corrections revised so as to bring the instrument back to this state.

For some types of instrument, it is impossible to perform multipoint calibration because the adjustment is not accessible; in this case, multipoint verification (see 8.1.3) should be carried out instead.

7.1.3 Single-point calibration

Single-point calibrations should generally be carried out using a calibration gas close to the middle of the range of calorific values expected to be measured by the instrument. They are usually carried out at short intervals of time (see 7.1.4).

Single-point calibrations do not allow correction to sensitivity, only to the offset of the single set point. The known offset is often used to derive a simple correction factor, sometimes known as the record correction, to be applied equally to all measured calorific values until the next calibration.

Because of the low uncertainty of its calorific value (see 7.2), the use of high-purity methane as a single-point calibration gas is generally acceptable for natural gas operations in cases where instrumental response is known from performance-assessment tests to be highly linear (see 5.1.3). However, the single-point calibration gas should not be the same as the gas used for routine verification (see Clause 8).

7.1.4 Calibration frequency

The required frequency of routine calibrations, both single-point and multipoint, shall be established during the performance evaluation tests. The calibration frequencies adopted may be different from the recommendations made by the instrument manufacturer.

Single-point calibrations are usually carried out at short intervals of time (e.g. hourly, daily or weekly). The interval adopted shall be set such that the measurement error of the instrument before a new calibration is not expected to exceed a user-defined percentage (e.g. 50 %) of the uncertainty specified for the class of performance required.

Occasionally, it can be useful to carry out an unscheduled calibration, either single-point or multipoint. The requirement for this is most often and most reliably suggested by unusual or erratic results from the routine verification tests (see Clause 8).

In the particular case of a stoichiometric instrument using synthetic air for combustion, it is advisable to carry out at least a single-point calibration after each change of cylinder. This is in addition to being sure that "air" of sufficient quality for the purpose (possibly prepared gravimetrically) is part of the overall specification.

Multipoint calibrations should generally be carried out

- a) following installation and commissioning, and after any maintenance or adjustment that can affect the sensitivity of the instrument,
- b) at regular but long intervals of time (e.g. annually), and/or
- c) following a verification test that gives results outside of any pre-set divergence limits (see 8.1.2).

7.2 Calibration gases

Calibration gases are certified reference gas mixtures, where the certification is for calorific value, including a statement of the associated uncertainty. Typically, they are synthetic mixtures (e.g. methane-ethane-nitrogen). Sometimes a dry natural gas (provided it is always stored above its hydrocarbon dew point) or methane of specified and checked purity is suitable.

It is necessary to certify the gases by a process that provides an uncertainty on the certified calorific value that is significantly less than the uncertainty requirement for the calorific values to be measured by the instrument. To achieve this, the uncertainty of the certified calorific value shall be not more than one-half of the uncertainty specified for the application. In practical terms, the best that can presently be achieved for mixtures in the certification process is about $\pm 0,05 \text{ MJ/m}^3$ (95 % confidence limit).

Currently available certification procedures that provide the required levels of trueness are as follows:

- a) use of a class 0 calorimeter, maintained and operated by a national standards institute or an accredited laboratory, which provides traceability to recognized national metrological standards;
- b) use of a gas of known composition, prepared using a documented gravimetric procedure such as that described in ISO 6142, carried out at a national standards institute or an accredited laboratory, which provides traceability (of composition) to recognized national standards in the manner described in ISO 14111; in this case, the certified calorific value shall then be calculated from the composition by the internationally recognized method in accordance with ISO 6976;
- c) use of methane of known purity (e.g. 0,999 9+ mole fraction for class 1 applications) where applicable; in this case, the impurities present in the gas should be checked and the calorific value of methane given in ISO 6976 shall be used.

Any or all of these methods may be specified in documentation giving type approval to an instrument.

8 Verification

8.1 Verification procedures

8.1.1 General

Verification is a non-corrective metrological procedure, the purpose of which is to confirm that the results produced by the instrument in question are, for the same test gas or gases, unchanged within specified limits (see 8.1.2) since the previous verification.

Verification should be carried out at specified and pre-established intervals (typically daily or weekly, depending upon the type of instrument), and should also be carried out whenever an anomaly is detected during a quality-control procedure (see Clause 10).

For all types of instrument, single-point verification is applicable; however, for instruments where multipoint calibration is impossible, multipoint verification should also be carried out, but with a lesser frequency.

It is necessary to consider a number of operational details, applicable to both single-point and multipoint verification.

- a) The instrument should remain in its normal operational state during verification (i.e. it should not be switched, either manually or automatically, to a calibration cycle).
- b) The results of any verification processes should not be included in any calculation of averages from the routine on-line measurements.
- c) A stable measurement should be achieved before the verification result is accepted.
- d) It is necessary to make suitable arrangements for an orderly transfer from one cylinder of reference gas to the next whenever the first is close to depletion.

8.1.2 Single-point verification

The single-point verification procedure consists simply of measuring the calorific value of a reference gas close to the maximum or minimum calorific value normally measured by the instrument. It is not necessary for this reference gas to be of certified calorific value; all that is required is a guarantee that, from verification to verification, the calorific value remains unchanged.

In general, the verification gas should be sensibly different from the single-point calibration gas (so as to exercise the instrument), but in applications where the normal working range of the instrument is very narrow this can be difficult to arrange.

As in the case of calibration gases, natural gas (provided that it is stored and transported at temperatures above its hydrocarbon dew-point) or a synthetic mixture compressed into a high-pressure cylinder is suitable as a single-point verification gas. In this case, homogeneity in the cylinder is a far more important criterion than detailed knowledge of the composition or calorific value.

Before putting an instrument into service, the user should decide not only on the frequency of verification tests but also on the criteria that determine whether the instrument passes or fails each test.

One possible strategy is to set a simple "divergence limit". Then, if the absolute difference in results between two successive verifications is less than the specified limit, the instrument is allowed to continue in service; if not, then an adjustment or single-point calibration should be carried out, after which the instrument is returned to service.

The value set for the divergence limit depends strongly on the particular application. In general, however, it is recommended that the limit be set equal to 50 % or 100 % of the uncertainty associated with the required class of performance, i.e. 0,05 MJ/m³ or 0,10 MJ/m³ for class 1 and so on.

A strategy that simply compares present performance with the immediate past performance does not pick up problems such as long-term drift. To do this, a cumulative-sum control chart (see ISO 5725-6) that records the results of all verification tests should be maintained. A pre-set limit to the cumulative long-term drift can then be used to trigger an unscheduled maintenance operation, including recalibration.

8.1.3 Multipoint verification

For some types of instrument, the process described in 7.1.2 as multipoint calibration is impossible to perform, in the sense that there is no access to those instrument settings that allow the application of a correction to the sensitivity. In this case, multipoint verification shall take the place of multipoint calibration.

The process of multipoint verification is identical to that of multipoint calibration, except that no correction is made. The criteria that the results of multipoint verification need to satisfy in order that the instrument shall be allowed to continue in service are

- a) that test results are unchanged, within pre-set limits, since the previous verification,
- b) that the test results are correct within the uncertainty values of the certified test gases.

These criteria are, of course, closely interlinked. As for single-point verification, the pre-set limits can sensibly be set equal to the uncertainty associated with the required class of performance.

If an instrument fails a multipoint verification test, this probably indicates the need for corrective maintenance. If maintenance demonstrates the need for corrective adjustments, then it will probably be necessary to consult with the instrument supplier.

8.2 Verification gases

The main point requiring emphasis here is that it is not necessarily required that single-point verification gases be certified for calorific value, whereas multipoint verification gases do require this certification.

The general requirements for single-point verification gases are discussed in 8.1.2 and it is not necessary that they be repeated.

The requirements for multipoint verification gases are identical to those of multipoint calibration gases (see 7.2).

9 Maintenance

9.1 Preventive maintenance

It is normal practice to carry out, as a minimum, all maintenance procedures as recommended by the manufacturer, and at the prescribed frequency.

Additional preventive maintenance can be required when the performance assessment tests (see 5.1) have suggested that, for a particular application, the required performance is likely to be achieved only by more stringent or more frequent attention.

In some cases, there is likely to be a requirement that preventive maintenance can be carried out without damage to, or interference with, any officially set protective marks or seals. It can be necessary, however, to break such devices and officially re-set them after any maintenance operation that demands at least partial recalibration of the instrument.

The schedule of maintenance operations should include requirements for the instrument cabinet or test room as well as for the instrument. This can involve items such as air filters, etc.

9.2 Corrective maintenance

Corrective maintenance is any set of operations carried out in order to repair an instrument malfunction. The malfunction in question can be either catastrophic (i.e. the instrument shuts down and/or the record is lost) or it can be that routine verification procedures show that the recent record is unsatisfactory (inaccurate).

It is not possible to give general guidelines for the content of corrective maintenance procedures, but procedures relevant to various types of failure can likely be found in the particular manufacturer's instrument manuals. Calibration of an instrument is mandatory after the performance of corrective maintenance.

10 Quality control

10.1 General

Taken together, calibration, verification, maintenance and quality control, plus "live" operation, constitute the set of distinct procedures that shall be required for normal post-acceptance routine application of an instrument. A flow chart showing a typical sequence in which these procedures shall be carried out is given as Figure 4.

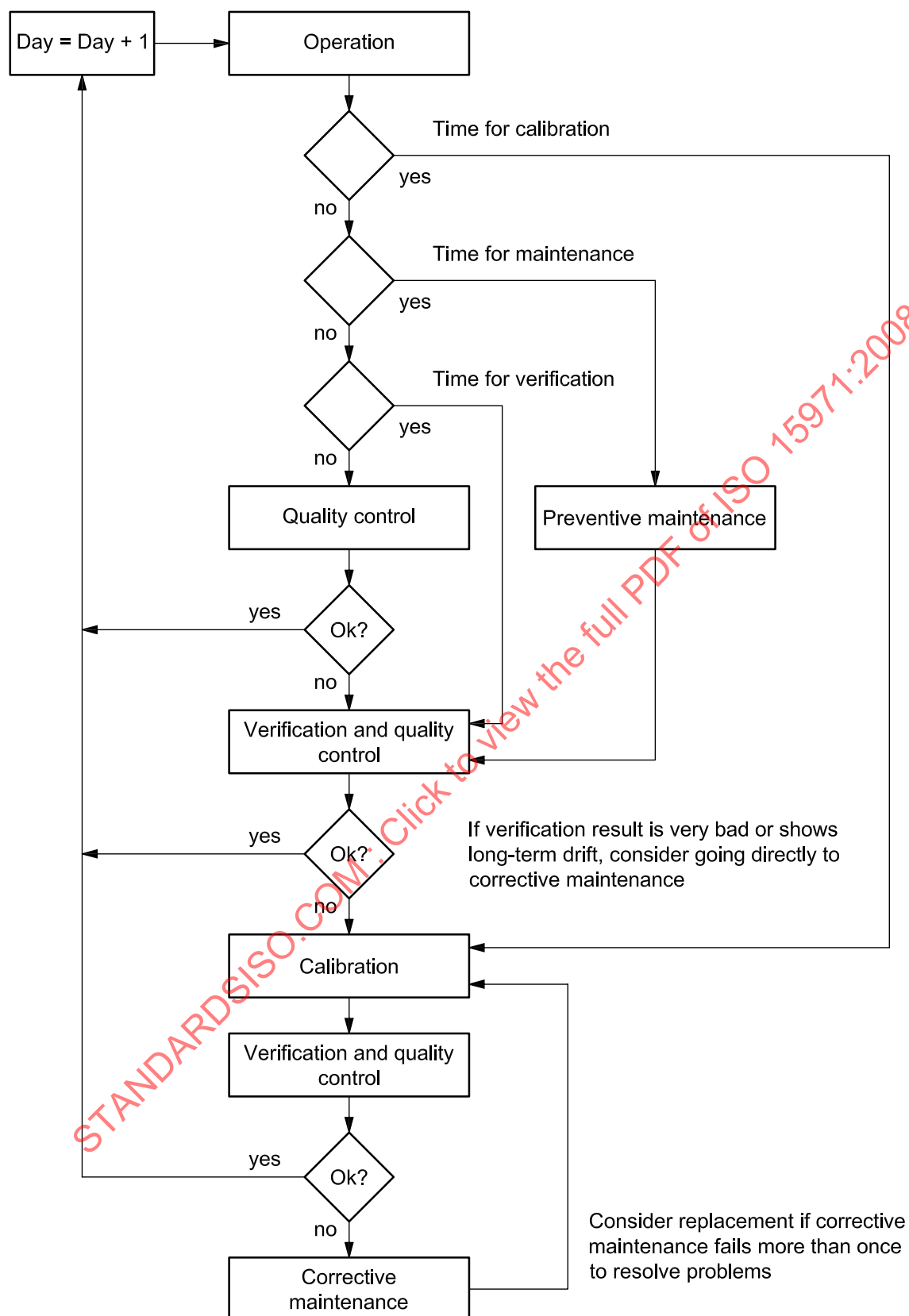


Figure 4 — Operational procedures

Quality control, in the context of routine calorific value measurement, is that set of operations or observations that together ensure the proper functioning of the measurement system. The latter includes the instrument itself and all of the ancillary equipment (e.g. the sampling line).

Apart from regular verification, which constitutes the major quality control mechanism, the procedure is generally passive (i.e. non-interventionist) and visual. It is necessary that it be carried out according to a regular schedule (e.g. daily or weekly) during a period of normal measurement with which it shall not interfere. It may also be carried out if any apparent measurement anomaly is detected during normal operation.

The procedure should involve working through a checklist that includes all of the environmental parameters and instrumental factors that are relevant to the proper functioning of the system and that are accessible to measurement (e.g. temperatures, flowrates) or observation (e.g. flame structure). The checklist of parameters is instrument-specific.

The observed values of the various parameters from the checklist should be compared with those established during installation or during the previous calibration. In the event that any parameter falls outside of pre-established acceptable limits, the possibility of active intervention, such as verification, calibration or maintenance, should be considered.

A log of all data recorded during quality-control procedures should be kept for an extended period (e.g. 10 years) for possible retrospective qualitative or quantitative analysis.

10.2 Environmental parameters and ancillary equipment

10.2.1 General

The relevant items are mainly those listed in 10.2.2 to 10.2.6 although there can be others in particular applications:

10.2.2 Ambient conditions

These include ambient temperature, atmospheric pressure and relative humidity. They are unlikely to influence the performance of the instrument under normal circumstances although (see 5.1.6) it is likely that there are temperature limits within which the instrument is required to operate. If these temperature limits are exceeded then, whether or not instrument performance is apparently affected, steps should be taken to get the environment back within specification. In the event of a correlation between any of these three parameters and the recorded calorific value (i.e. of a magnitude greater than that observed in the performance assessment tests), it is probably indicative of instrumental malfunction, which should be investigated. It can be convenient to maintain continuous records of these parameters, e.g. using thermo-baro-hygrograph-type instruments.

10.2.3 Combustion-air quality

There should be a check on the status of any air-filtration equipment external to the instrument; this shall be maintained in accordance with the manufacturer's instructions. It can also be useful to make spot-checks of the air quality using commercial aspirated-tube chemical detection equipment.

10.2.4 Calibration and verification gases

The pressure in each cylinder of gas used by the instrument should be checked

- a) in order to detect possible leakage,
- b) to enable the procurement of replacements in good time, bearing in mind that certified gases are unlikely to be available "off-the-shelf".

10.2.5 Status of ancillary devices

The activity status of any device connected to the instrument, such as a pen recorder, printer or data-acquisition unit should be checked. Observations that should be made include a check on the proper operation of any pen or printer and of the amount of any chart roll remaining unused.

10.2.6 Abnormal electrical transmissions

Any devices electrically connected to the instrument should be checked in order to avoid any influence of abnormal transmissions on the instrument.

10.3 Instrumental factors

Rather few instrumental factors are generally amenable to much more than a superficial check. Where possible, the readings of any air and gas flowrate indicators should be recorded but, beyond that, the available checks that do not interfere with instrument function are purely observational. These should include the following:

- a) integrity of all visible electrical connections;
- b) where accessible, the condition of any sensor elements (e.g. exposed thermocouples);
- c) shape, size, structure, colour, etc., of any exposed flame, including that on any external pilot burner.

An observation of any these factors that differs sensibly from previous observations should be investigated, with the likelihood that the instrument requires maintenance and/or recalibration.

Annex A (normative)

Symbols and units

Symbol	Meaning	Unit
H_s	Volume-basis superior calorific value	MJ/m ³
H_i	Volume-basis inferior calorific value	MJ/m ³
p	Absolute pressure	kPa
t	Time	s
T	Temperature	K
τ	Relaxation time	s
x	Mole fraction	—

Annex B (informative)

Examples of type-approval and technical specifications

B.1 Example of type-approval specification

a) Instrument accuracy

When calibrated with a certified gaseous reference mixture of uncertainty no greater than $\pm 0,05 \text{ MJ/m}^3$, the instrument shall measure calorific values with a total uncertainty of less than $\pm 0,10 \text{ MJ/m}^3$.

b) Reporting of results

The instrument shall provide printed reports of calorific value at intervals of time not exceeding 1 h, and printed reports of the daily average calorific value immediately after midnight.

c) Type-approval programme

Type-approval testing of the instrument shall include tests carried out in accordance with the performance assessment clauses of ISO 15971, and shall establish the operational envelope within which the required accuracy is achieved. The boundaries of this envelope shall define the operating conditions for the acceptable use of the instrument.

B.2 Example of technical specification

The purchase documentation, of which this example technical specification can form one part, is likely to include a number of other important requirements, not least of which are the purchase price, delivery time and technical back-up available.

Note to vendor: The following are minimum requirements.

a) Performance:

- range of superior calorific value (dry at standard reference conditions): (36 to 45) MJ/m^3
- uncertainty over range: $\pm 0,10 \text{ MJ/m}^3$
- repeatability: $\pm 0,05 \text{ MJ/m}^3$
- time for 95 % response to 1 MJ/m^3 change: < 4 min

b) Environmental conditions for specified performance:

- temperature range: (10 to 50) $^{\circ}\text{C}$
- temperature stability: no requirement
- relative humidity: (30 to 85) %
- electromagnetic compatibility: to level 3

- c) Calibration:
- calibration gas: $\pm 0,05 \text{ MJ/m}^3$
 - calibration interval: no requirement
- d) Electrical supply: (240 $\pm$ 20) VAC single phase at (50 $\pm$ 5) Hz (or vendor to supply transformer)
- e) Automatic re-ignition: Re-ignition procedure tried three times; then to safe state if re-ignition not successful
- f) Safety features: Fail-safe circuits to shut down to safe state in event of hazardous state occurring
- g) Calorific-value record: Continuous digital display with hard-copy printout; print interval selectable

Annex C (informative)

Class 0 mass-basis calorimetry

C.1 Principle

The distinguishing feature of calorimeters capable of class 0 performance is that all operations are carried out in strict accordance with the best metrological practices and all relevant physical measurements are traceable, through an unbroken chain of comparisons, to international metrological standards.

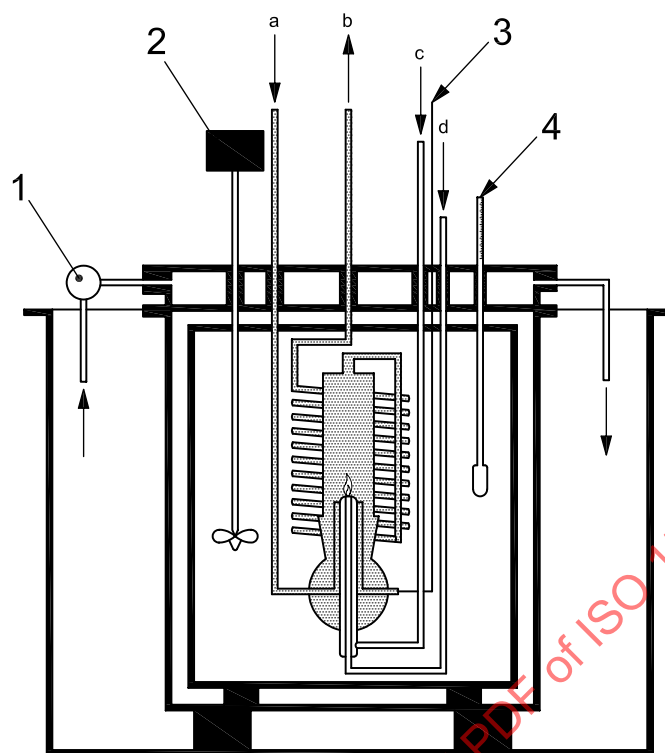
All current class 0 calorimeters fall into the “direct” measurement category and measure calorific value on a mass basis. They depend on the determination, using thermometric measurements, of the energy released as heat from the combustion of a discrete mass of gas.

In the usual configuration, the main measurement is of the dynamic increase in temperature of a stirred heat-exchange fluid within a sealed container, which is compared with the temperature rise produced during calibration (see Clause C.3). It is necessary to take great care to account for all minor sources and sinks of heat in this type of calorimeter [12], [13], [14].

Mass-basis calorific values are usually measured at, and referenced to, the international thermochemical standard reference temperature, $T_{\text{ref}} = 298,15 \text{ K}$ (25°C), by this technique. The values obtained are then restated, if so required, on a volumetric basis using a measured value for the density of the gas at T_{ref} . It is also necessary to make corrections for any change in the reference conditions, but these factors are beyond the scope of this International Standard.

C.2 General method

The main features of a typical class 0 mass-basis calorimeter are shown in Figure C.1. The apparatus is comprised of an external water bath, normally thermostatically controlled to within 10 mK at about 2 K above T_{ref} , within which the calorimeter assembly is mounted. This assembly consists of two concentric copper/brass vessels, the inner one mounted within the outer by means of thermally insulating supports. The vessels are separated by an air gap, and the facing surfaces gold-plated and highly polished in order to reduce radiative heat transfer.

**Key**

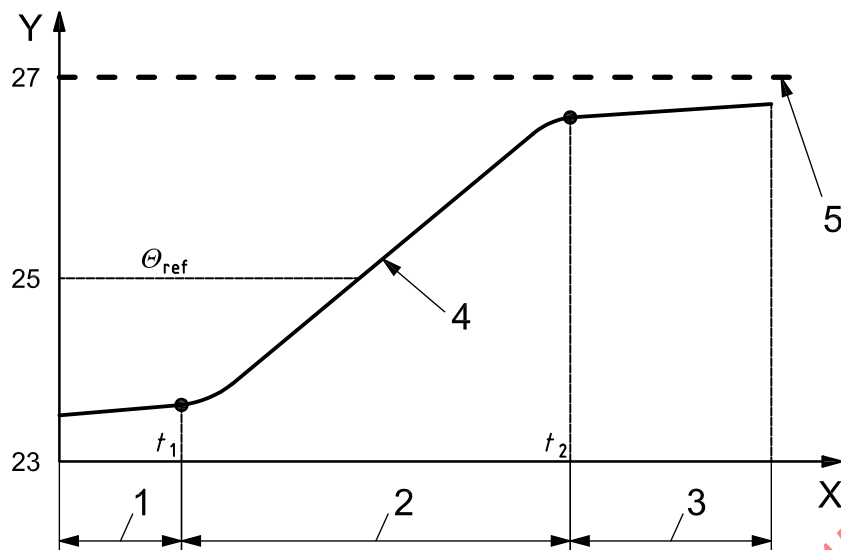
- | | | | |
|---|----------------------------|---|-------------------------|
| 1 | water pump | a | Secondary oxygen. |
| 2 | stirrer motor | b | Combustion products. |
| 3 | spark ignition electrode | c | Primary oxygen + argon. |
| 4 | quartz crystal thermometer | d | Fuel gas. |

Figure C.1 — Schematic diagram of class 0 calorimeter

The inner vessel contains the calorimeter burner and coiled heat-exchange unit (typically made of Pyrex glass with a silica burner jet), a temperature-measuring device (typically a platinum-resistance or quartz thermometer), a constant-speed stirrer, a sleeve for a cold finger and, where used, an electrical heater for calibration. This vessel is filled with heat-exchange fluid (usually water) and sealed to prevent changes in the mass of fluid that affect the energy equivalent (i.e. calibration constant) of the calorimeter. During a combustion run, fuel gas, a primary argon-oxygen mixture and secondary oxygen enter through inlet lines shown in the figure. A double-jet system permits pre-mixing of the fuel gas with the primary argon-oxygen just before the combustion zone. The argon is present only to stabilize the flame and to give good combustion characteristics.

An experimental run, either calibration or a measurement, consists of three parts. First, the inner vessel is force-cooled to about 2 K below T_{ref} and then allowed to rise slowly in temperature (with no flow of fuel gas) as a result of heat transfer from the environment; the temperature is measured as a function of time until the heat-exchange fluid reaches (say) 1,5 K below T_{ref} (fore-period).

The main period is then initiated, either by igniting (at the spark electrode) the fuel gas, which now begins to flow, or (for an electrical calibration) by switching on electrical power. The temperature increase is now recorded continuously (i.e. every few seconds) until a temperature of (say) 1,5 K above T_{ref} is reached, at which time the fuel gas flow is stopped (or electrical power switched off), but the temperature-time measurements are continued until the heat-exchange fluid is close to 2 K above T_{ref} (after-period). The temperature-time plot for the whole run has the form shown in Figure C.2.



Key

X	time, t , expressed in minutes	4	calorimeter temperature
Y	temperature, θ , expressed in degrees Celsius	5	tank temperature
1	fore-period	t_1	ignition
2	main period (approximately 20 min)	t_2	extinction
3	after-period		

Figure C.2 — Temperature-time plot for class 0 calorimeter

The temperature discrimination during all phases of the run is normally 1 mK. Readings of temperature, together with many of the switching and other operations needed in the course of a run, are under computer control.

The data collected can be used to determine either an energy equivalent for the calorimeter system or, using a known value of the energy equivalent, the heat input from the known mass of fuel gas burned, and hence its mass-basis calorific value.

This is a very broad outline of a typical system. An experimental run has many additional intricacies and it is necessary that the data analysis take account of these, particularly those that concern the many small but non-negligible sources and sinks of heat, e.g. the spark-ignition system, the energy of stirring, the water vapour lost in effluent gases, etc. [13].

Class 0 calorimeters of this sort are labour-intensive spot-test instruments that are designed to carry out measurements to the highest achievable accuracy on discrete samples of gas. The requirements for metrology, environmental control and data acquisition systems and the test duration of more than 1 day for each determination make these instruments unsuitable for installation in field-testing situations where the instruments described in 4.2, 4.3 and 4.4 are normally used.

C.3 Calibration

C.3.1 Electrical heating method

In this method^[14], calibration is achieved by the input of an accurately determined amount of energy (released as heat), using an electrical resistance device as the source. This is operated, as described in Clause C.2, over a known period of time, during which the thermal response of the calorimeter's heat-exchange fluid is followed, in order to determine the energy equivalent of the calorimeter. This method has the capability of providing traceability, through the use of certified devices (e.g. platinum resistance thermometers, resistors, voltmeters, time signals, etc.), to national metrological standards.

C.3.2 Combustion method

In this method^[13], calibration is achieved by burning an accurately determined mass of a gas of accurately known standard enthalpy of combustion over a period of time, during which the thermal response of the calorimeter's heat-exchange fluid is followed, in order to determine the energy equivalent of the calorimeter.

In practice, ultra-high-purity methane, the conventional true value (including its associated uncertainty) for which is given in ISO 6976, is the only gas that satisfies the requirements of good metrological practice. In this context, methane can properly serve as a gaseous reference material. The primary traceability is performed to this value for the standard enthalpy of combustion (and only back through this to the more conventional metrological standards), but ancillary equipment (thermometry, mass-pieces, etc.) is capable of direct traceability to national standards.

In this method, heat is dissipated in the calorimeter during calibration at exactly the same point (i.e. at the burner) as during a run on a sample gas. Consequently, the internal heat-flow patterns should be more alike than is the case with electrical calibration, and there can be less scope for the introduction of systematic error from this particular source. This apparent advantage, however, is offset by the presence in the overall uncertainty budget of a significant contribution, not present in the electrical calibration method, from the uncertainty of the standard enthalpy of combustion of the calibration gas.

Annex D (informative)

Direct combustion calorimetry

The main principles of direct, continuously recording combustion calorimetry are presented briefly in 4.2, and so are not repeated here, except to reiterate that a steady flow of gas, metered volumetrically, is burned under controlled conditions in an excess of air so as to produce a quasi-stationary (equilibrium) temperature rise in a continuously counter-flowing heat-transfer medium with which it does not mix. The equilibrium temperature rise in the heat-transfer medium is taken as a measure of the volume-basis superior calorific value of the gas.

A schematic diagram of a typical commercial implementation of these principles is shown as Figure D.1, and a simplified "thermal circuit" for this is shown as Figure D.2. Other, more or less distinct, implementations are known, but are no longer in widespread use.

In this device, the fuel gas, the air for combustion and the heat-exchange air are all metered (at atmospheric pressure and in the saturated state) in three separate water-sealed "wet-meters". All three meters are mounted on the same axle, in a single large tank of water (the level of which is continuously maintained by means of a pump-and-weir system) and are driven, through a system of gears, by a single electric motor. As a consequence, the ratio between the amounts of fuel and air delivered by each meter remains constant, irrespective of changes in motor speed, atmospheric pressure or ambient temperature. At constant calorific value, therefore, the temperature difference between the nickel resistance thermometers at the inlet and outlet of the heat-exchanger unit remains constant. A typical temperature difference for natural gas is about 20 K.

The two resistance thermometers are mounted in the opposite arms of a Wheatstone bridge arrangement, and the out-of-balance signal is conditioned and used as input to a recording device, such as a pen recorder, or to a data-acquisition unit.

In principle, the temperature difference observed between the inlet and outlet thermometers can be used as an unambiguous determinant of the superior calorific value of the saturated gas only if

- a) all of the energy liberated as heat of combustion is transferred to the heat-exchange air, and
- b) all of the water produced by combustion is condensed to the liquid state.

Neither of these requirements can be fully achieved in practice, and so it is not possible to secure an exact heat balance that allows the calculation of calorific value from first principles. Nor, for a variety of reasons, is the instrument fully insensitive to variations in ambient temperature.

Nevertheless, instruments of this type are readily capable of class 1 performance given proper calibration and operation within an air-conditioned and temperature-controlled room.