



SLOVENSKI STANDARD

SIST ISO 6145-1:2004

01-oktober-2004

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Gas analysis -- Preparation of calibration gas mixtures using dynamic volumetric methods -- Part 1: Methods of calibration

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Analyse des gaz -- Préparation des mélanges de gaz pour étalonnage à l'aide de méthodes volumétriques dynamiques -- Partie 1: Méthodes d'étalonnage

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ICS:

71.040.40 Kemijska analiza Chemical analysis

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INTERNATIONAL STANDARD

ISO 6145-1

Second edition
2003-11-15

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

Part 1: Methods of calibration

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*Analyse des gaz — Préparation des mélanges de gaz pour étalonnage
à l'aide de méthodes volumétriques dynamiques —*

Partie 1: Méthodes d'étalonnage

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Reference number
ISO 6145-1:2003(E)

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Published in Switzerland

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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.

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

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

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

ISO 6145-1 was prepared by Technical Committee ISO/TC 158, *Analysis of gases*.

This second edition cancels and replaces the first edition (ISO 6145-1:1986), in which the estimated uncertainties in the calibration methods and techniques have now been combined in a square-root sum-of-squares manner to form the relative combined standard uncertainty. In comparison with the previous edition the periodic injection has been deleted (limited application).

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

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

Diffusion will be the subject of a future Part 8 to ISO 6145. Part 3 to ISO 6145, entitled *Periodic injections into a flowing gas*, has been withdrawn.

Introduction

This part of ISO 6145 is one of a series of standards which describes the various dynamic volumetric methods used for the preparation of calibration gas mixtures.

In dynamic volumetric methods a gas, A, is introduced at volume or mass flow rate q_A into a constant flow rate q_B of a complementary gas B. Gas A can be either a pure calibration component, i , or a mixture of i in A.

The volume fraction, $\varphi_{i,M}$ of i in the final calibration gas mixture is given in the following equation:

$$\varphi_{i,M} = \varphi_{i,A} \left(\frac{q_A}{q_A + q_B} \right)$$

where $\varphi_{i,A}$ is the volume or mass fraction of component, i , in the pre-mixed gas A, and is already known from its method of preparation. It is assumed that in this equation, $\varphi_{i,B}$, the concentration of component, i , in gas B, is zero.

The introduction of gas A can be continuous (e.g. permeation tube) or pseudo-continuous (e.g. volumetric pump). A mixing chamber should be inserted in the system before the analyser and is particularly essential in the case of pseudo-continuous introduction. The flow rate of component A is measured either directly in terms of volume or mass, or indirectly by measuring the variation of a physical property.

The dynamic volumetric preparation techniques produce a continuous flow rate of calibration gas mixtures into the analyser but do not generally allow the build-up of a reserve by storage under pressure.

The main techniques used for the preparation of the mixtures are:

- a) volumetric pumps;
- b) continuous injection;
- c) capillary;
- d) critical orifices;
- e) thermal mass-flow controllers;
- f) diffusion;
- g) saturation;
- h) permeation;
- i) electrochemical generation.

In all cases, and most particularly if very dilute mixtures are concerned, the materials used for the apparatus are chosen as a function of their resistance to corrosion and low absorption capacity (usually glass, PTFE or stainless steel). It should, however, be pointed out that the phenomena are less important for dynamic volumetric methods than for static methods.

Numerous variants or combinations of the main techniques can be considered and mixtures of several constituents can also be prepared by successive operations.

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Some of these techniques allow calculation of the final concentration of the gas mixture from basic physical information (e.g. mass rates of diffusion, flow through capillaries). However, since all techniques are dynamic and rely on stable flow rates, this part of ISO 6145 emphasizes calibration of the techniques by measurement of the individual flow rates or their ratios, or by determination of the composition of the final mixture.

The uncertainty of the composition of the calibration gas mixture is best determined by comparison with a gas mixture traceable to international standards. Certain of the techniques which may be used to prepare a range of calibration gas mixtures may require several such traceable gas mixtures to verify their performance over that range. The dynamic volumetric technique used has a level of uncertainty associated with it. Information on the final mixture composition depends both on the calibration method and on the preparation technique.

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

Part 1: Methods of calibration

1 Scope

This part of ISO 6145 specifies the calibration methods involved in the preparation of gas mixtures by dynamic volumetric techniques. It also gives a brief presentation of a non-exhaustive list of examples of dynamic volumetric techniques which are described in more detail in other parts of ISO 6145.

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 6142, *Gas analysis — Preparation of calibration gas mixtures — Gravimetric method*

<https://standards.iteh.ai/catalog/standards/sist/c70a4742-109d-45ab-b553-19a0634d7c03/iso-6145-1-2004>

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

ISO 7504, *Gas analysis — Vocabulary*

3 Terms and definitions

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

3.1

uncertainty of measurement

parameter, associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand

NOTE 1 Values of the individual statistical uncertainties found in some methods and techniques in this part of ISO 6145 are combined with the values of systematic uncertainties that also occur in a square-root sum-of-squares manner to provide a relative combined uncertainty, or in some cases as a relative expanded uncertainty by application of the coverage factor “2”.

NOTE 2 In keeping with Reference [1] of the Bibliography, the uncertainty of the composition of a mixture is expressed as a relative expanded uncertainty.

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4 Calibration methods

4.1 General

4.1.1 The uncertainty in the composition i , M of a component i of a calibration mixture M depends at any time on

- a) the uncertainty of the calibration method,
- b) the frequency with which it is applied,
- c) the stability of the control devices involved in the dynamic preparation technique.

To assess the uncertainty of the whole procedure, possible instantaneous variations and drift of the principle parameters of the technique during the calibration procedure shall be considered.

According to the preparation technique for the gas mixtures used, calibration can be carried out by one of the following methods:

- measurement of flow rate (mass or volume);
- comparison method;
- tracer method;
- direct chemical analysis.

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Table 1 shows the applicability of each calibration method to the different preparation techniques.

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Table 1 — Calibration methods applicable to the preparation techniques

Preparation techniques	Calibration methods			
	Comparison with ISO 6143 ^a	Flow rate measurement ^a	Tracer ^a	Direct analysis
Volumetric pumps	+	—	+	May be applicable; depends on nature of components
Continuous injection	+	—	+	
Capillary	+	+	+	
Critical orifice	+	+	+	
Thermal mass flow controllers	+	+	+	
Diffusion	+	—	—	
Saturation	+	—	—	
Permeation	+	—	—	

^a The pluses refer to the measurement of a volume flow. In principle, flow rate measurement can also be performed for continuous injection methods, diffusion methods and permeation methods. Here, mass flows are measured rather than volume flows. For diffusion and permeation tubes the mass flow may be measured continuously using a suspension balance.

4.1.2 In general, the principles of the methods fall into two categories, as follows.

- Principles in which the flow rates of component gases are measured either by volume or by mass and in which the concentration in the final mixture is calculated from the flow rate. Different techniques may be used for the individual components of a mixture and these may be calibrated by different methods. The principle of measurements of individual flow rates, however, remains.
- Principles which operate directly on the final mixtures.

Since different principles are involved, they are given separately under each individual method.

Since the calibration methods rely upon different principles and the equipment used for the realization of the gas flow rates is different, different units can be used to express the contents.

For calibrations using the comparison method, the content is expressed as a mole fraction or mole concentration because most of the calibration gas mixtures used for the comparison, if possible, are described in this way.

Using techniques based on volume flow rate leads in the first instance to volume fractions. Recalculation of these data to mole fractions is possible but leads to an increase in the uncertainty because of the uncertainty of the density and molar-volume data. In this case, the expression in volume fractions is preferred.

Calibration by the gravimetric method gives mass fractions for the contents of components in gas mixtures. These can be recalculated to mole fractions by dividing by the respective atomic or molar masses. Expression in mole fraction is therefore preferred.

Under some circumstances, the total flow rate cannot be taken as the sum of two individual flow rates q_A and q_B which have been measured separately. These problems can be caused by deviations from the ideal gas laws or by changes in conditions such as backpressure or viscosity resulting from the blending of the two flow rates. Deviations from ideal behaviour can be predicted with reasonable accuracy and other uncertainties can be minimized by careful attention to apparatus design.

4.1.3 Flow rate measurement is normally carried out using one of the following:

- a) primary devices, based on absolute principles, for example:
- gravimetric method;
- b) methods which may be considered as potentially primary when the volume of the device is determined by weighing the relevant volume of water, or another suitable liquid of higher density:
- mercury-sealed piston,
 - bell-prover;
- c) many other devices available for the measurement of volume flow, some of which are listed below (calibration of these devices is carried out by using one of the above primary or potentially primary methods):
- soap-film meter,
 - wet-gas meter,
 - thermal mass flow sensor,
 - variable area flow meter.

The soap-film and mercury-sealed piston flow meters share a common principle, i.e. that of timing the travel of a soap bubble or piston between carefully defined points either electronically or by observation, for example by means of a cathetometer. The volume between these points can be determined by filling with water, which is subsequently weighed (see Annex A).

The wet-gas meter is an integrating device which indicates the total volume of gas that has been passed through it (the dry-gas meter, familiar from the domestic environment, has a similar integrating property but has not been included because it is less accurate). The variable area flow meter is a continuously indicating device. The thermal mass flow sensor measures mass flow rate as a function of heat flux.

NOTE These devices are fully described in Annex B.

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4.1.4 Calibration of these flow-rate measuring devices is carried out using one of the primary or potentially primary methods:

- a) gravimetric method;
- b) mercury-sealed piston;
- c) bell prover.

The gravimetric method measures the mass of gas, which has flowed at a constant rate for a defined time through the device to be calibrated. The mercury-sealed piston drives a defined volume of gas over a measured time period into the device to be calibrated. The bell prover is a device for creating a constant and defined flow rate of gas, acting as a mechanically driven gasholder.

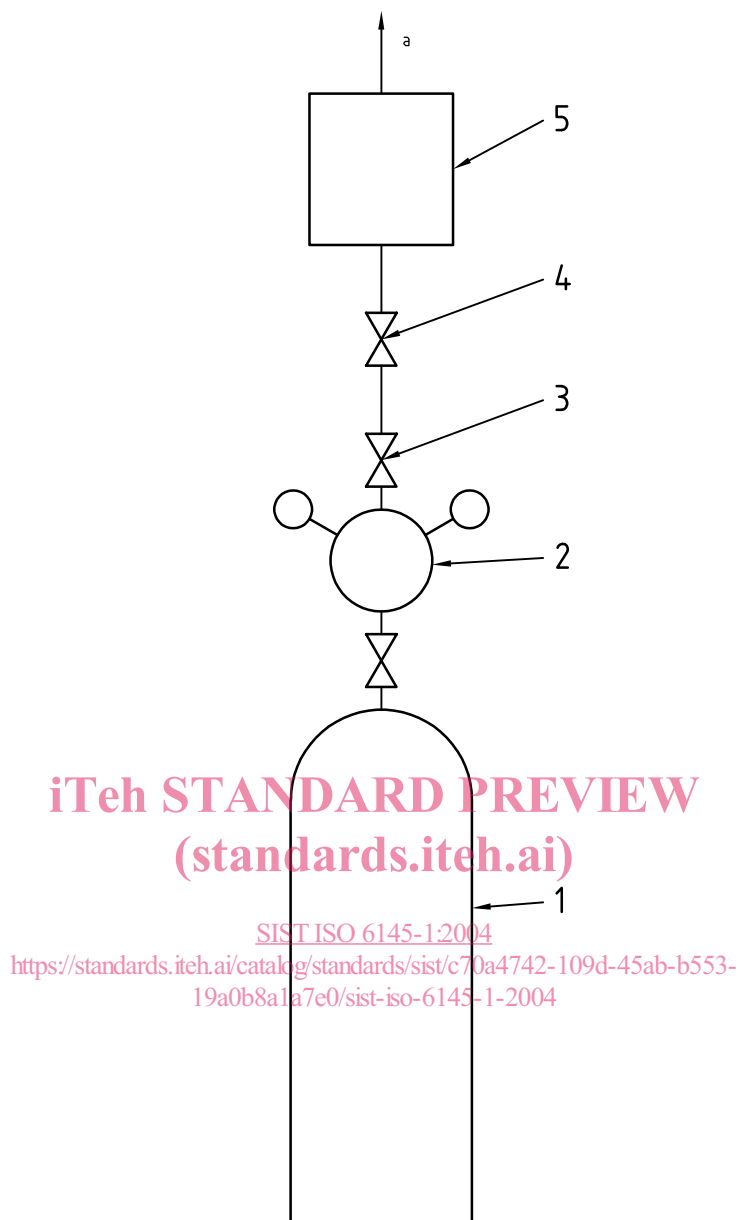
The bell prover and the gravimetric method may be used directly, where appropriate, to calibrate the various preparation techniques, but the information is more commonly transferred via one of the flow-rate measuring devices.

4.2 Description of primary or potentially primary measuring devices**4.2.1 Gravimetric method****4.2.1.1 Principle**

Gas from a cylinder flows at a constant rate through the device to be calibrated. This is continued for a sufficiently long period for the loss of mass from the cylinder to be accurately measured. The procedure provides data in terms of mass flow, which can then be converted to molar flow rate or, with assessed uncertainty, to a volume flow rate.

The gas cylinder and flow-rate measuring device are set up as shown in Figure 1. The cylinder (1) is fitted with a pressure regulator (2) on the outlet of which a precision needle valve (3) and shut-off valve (4) lead to the device to be calibrated (5). The dead volume between the needle valve outlet and the shut-off valve is minimized by using the smallest size of tubing and fittings commensurate with the desired gas flow rate. The temperature and pressure of the gas are measured at the inlet to the device to be calibrated.

The cylinder valve is opened, the pressure regulator is set to a value of, e.g. 100 kPa (1 bar) gauge, and the needle valve is adjusted to the desired flow rate. When conditions are seen to be steady, the shut-off valve is closed and the pipe-work is disconnected at the outlet of this valve. The cylinder, regulator, needle valve and shut-off valve are weighed as a single unit. The pipe-work is reconnected and the shut-off valve is opened to re-start the flow at the same rate. After the gas has flowed for a period long enough for the mass used to be measured accurately, the shut-off valve is closed and the cylinder, regulator, needle valve and shut-off valve weighed as before. During this period, the gas flow is accurately measured by first calculating the volume of gas from the change in mass, then the flow rate from the volume and the time.

**Key**

- 1 cylinder
- 2 pressure regulator
- 3 needle valve
- 4 shut-off valve
- 5 device to be calibrated

^a To vent.

Figure 1 — Gravimetric method