



SLOVENSKI STANDARD SIST EN ISO 6144:2006

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Gas analysis - Preparation of calibration gas mixtures - Static volumetric method (ISO 6144:2003)

Gasanalyse - Herstellung von Prüfgasen - Volumetrisch - statisches Verfahren (ISO 6144:2003)

Analyse des gaz - Préparation des mélanges de gaz pour étalonnage - Méthode volumétrique statique (ISO 6144:2003)

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71.040.40 Kemijska analiza Chemical analysis

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN ISO 6144

August 2006

ICS 71.040.40

English Version

Gas analysis - Preparation of calibration gas mixtures - Static volumetric method (ISO 6144:2003)

Analyse des gaz - Préparation des mélanges de gaz pour étalonnage - Méthode volumétrique statique (ISO 6144:2003)

Gasanalyse - Herstellung von Prüfgasen - Volumetrisch - statisches Verfahren (ISO 6144:2003)

This European Standard was approved by CEN on 21 July 2006.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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EN ISO 6144:2006 (E)**Foreword**

The text of ISO 6144:2003 has been prepared by Technical Committee ISO/TC 158 "Analysis of gases" of the International Organization for Standardization (ISO) and has been taken over as EN ISO 6144:2006 by Technical Committee CEN/SS N21 "Gaseous fuels and combustible gas", the secretariat of which is held by CMC.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by February 2007, and conflicting national standards shall be withdrawn at the latest by February 2007.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

Endorsement notice

The text of ISO 6144:2003 has been approved by CEN as EN ISO 6144:2006 without any modifications.

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INTERNATIONAL STANDARD

ISO 6144

Second edition
2003-02-01

Gas analysis — Preparation of calibration gas mixtures — Static volumetric method

*Analyse des gaz — Préparation des mélanges de gaz pour
étalonnage — Méthode volumétrique statique*

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ISO 6144:2003(E)**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 6144 was prepared by Technical Committee ISO/TC 158, *Analysis of gases*.

This second edition cancels and replaces the first edition (ISO 6144:1981), which has been technically revised.

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Gas analysis — Preparation of calibration gas mixtures — Static volumetric method

1 Scope

This International Standard specifies a method for the preparation of calibration gas mixtures by a static volumetric method and provides a procedure for calculating the volumetric composition of the mixture. It can be used either with binary gas mixtures (containing one calibration component in a complementary gas, which is usually nitrogen or air^{1, 2)}) or with mixtures containing more than one component in the complementary gas. This International Standard also specifies how the expanded uncertainty in the volume fraction of each calibration component in the mixture is determined by a rigorous evaluation of all the measurement uncertainties involved, including those associated with the apparatus used for the preparation of the gas mixture and those associated with the experimental procedure itself.

NOTE 1 This International Standard is generally applicable to the preparation of calibration gas mixtures containing calibration components in the concentration range 10×10^{-9} (10 ppb — parts per billion) to 50×10^{-6} (50 ppm — parts per million) by volume. However, gas mixtures may be prepared at larger or smaller volume fractions, provided that the components used in the static dilution process are selected appropriately.

NOTE 2 A relative expanded uncertainty of not greater than $\pm 1\%$ at a level of confidence of 95 % may be achievable at these concentrations, provided that:

- the purities of the parent gases have been determined by analysis and any significant impurities and the uncertainties in their measured concentrations have been taken into account;
- no significant adsorption effects or chemical reactions occur between the gaseous constituents and the internal surfaces of the apparatus, and there are no reactions between any of the gaseous components, i.e. between the calibration component and complementary gas or between the calibration components themselves;
- all the relevant apparatus used in the preparation of a calibration gas mixture have been calibrated with assigned measurement uncertainties which are appropriate to calculating the final expanded uncertainty for the calibration gas mixture prepared.

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 7504, *Gas analysis — Vocabulary*

3 Terms and definitions

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

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4 Principle

A calibration gas mixture consists of one or more calibration components in a complementary (diluent) gas, mixed in a suitable gas-mixing chamber. These calibration components are generally pure gases taken from cylinders, or from pure, volatile liquids that are allowed to evaporate into the gas-mixing chamber. The gas mixture is prepared using syringes to inject:

- either known volumes of gaseous calibration components (each at a pressure of about 1×10^5 Pa);
- or known masses or volumes of liquid calibration components;

These are injected into a volume of complementary gas contained in the mixing chamber (which is also at a pressure of about 1×10^5 Pa). Further complementary gas is then added to increase the overall pressure of the gas mixture to an accurately measured value above ambient atmospheric pressure. This final (above-atmospheric) pressure is required so that the calibration gas mixture will subsequently flow out of the mixing chamber and can be used to calibrate a gas analyser, which is usually operated at ambient pressure.

At each stage in the preparation procedure, the mixture is homogenized, usually by means of a suitable stirring device, and then left to equilibrate to ambient atmospheric temperature.

The volume fraction of each calibration component in the calibration gas mixture is determined by calculation of the ratio of the volume of the calibration component to the total volume of the mixture.

5 Apparatus

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5.1 Gas-mixing chamber, consisting of (standards.itoh.ai) to 5.1.1 to 5.1.8.

NOTE An example of a suitable gas-mixing chamber is described in Annex A.

5.1.1 Vessel, comprising the gas-mixing chamber itself, of sufficient internal volume to deliver the amount of calibration gas mixture required for any subsequent instrumental calibrations, manufactured of a suitable material that is inert to all the component gases, and designed both to be evacuable and to withstand the required above-atmospheric operating pressures. It shall also have vacuum/high-pressure flanges to allow access to the components that are mounted within the mixing chamber.

NOTE 1 Vessels with internal volumes of $0,1 \text{ m}^3$ to $0,5 \text{ m}^3$, capable of operating up to pressures of about 2×10^5 Pa (2 bar) and of maintaining a vacuum of better than $0,1 \times 10^2$ Pa (0,1 mbar), have been found to be suitable (see Annex A).

NOTE 2 Mixing chambers manufactured from borosilicate glass or stainless steel have been found to be suitable for the commonly used gaseous species (e.g. gas mixtures which contain SO_2 , NO, NO_2 , CO and C_6H_6 as the calibration components). However, care shall be taken in selecting the materials of the mixing chamber, and of the other components which come into contact with the gas mixtures, so that they do not affect the mixture's stability adversely — particularly when more reactive gas mixtures are to be prepared.

5.1.2 Vacuum pump, capable of evacuating the mixing chamber and its associated components to a low pressure, and including a suitable vacuum shut-off valve. This low pressure shall be defined either so that any gaseous contamination resulting from the residual low pressure has no effect on the accuracy of the concentration of the gas mixture prepared, or so that a quantitative correction for the effect of this residual low pressure may be made to the concentration of the mixture.

NOTE The residual gas pressure is generally due mainly to nitrogen from residual air. However, care must be taken to ensure that other gases that may react with the constituents of the gas mixture are not present at significant concentrations in this residual gas (e.g. traces of water vapour when acid gases are being used as calibration components, or traces of oxygen in the case when nitric oxide calibration mixtures are being prepared).

5.1.3 Gas line, used for the injection of the complementary gas, and including appropriate metering and shut-off valves.

5.1.4 Pressure, vacuum and temperature gauges, used to monitor these parameters inside the mixing vessel.

5.1.5 Septum, enabling a gas or liquid of known volume or mass to be injected into the mixing chamber from a metering syringe (5.2).

5.1.6 Motor-driven gas-mixing device, e.g. a fan, enabling the gaseous components in the gas-mixing chamber to be homogenized, and designed to provide satisfactory mixing of all the gaseous components to a given degree of homogeneity within a specified time. Experimental tests shall be carried out to demonstrate that the mixing device is able to achieve the required homogeneity within the specified time.

5.1.7 Pressure relief valve, used to ensure that the maximum internal safe working pressure specified for the mixing vessel and its associated components is not exceeded.

5.1.8 Outlet-gas sampling line, enabling the gas mixture prepared to be used for calibration purposes, and having a device for equalizing the internal pressure of the gas mixture in the mixing chamber with atmospheric pressure so as to enable the gas mixture to be determined at ambient pressure for calibration purposes.

5.2 Calibrated metering syringe, which can be used to inject, by means of a piston, a known volume of gas or liquid through a needle. The syringe shall have gastight seals to ensure that no significant leakage of the gas or liquid takes place.

NOTE 1 Glass syringes having polytetrafluoroethene (PTFE) bushings as seals, and with internal volumes of 10 ml, 5 ml, 1 ml, 0,5 ml and 0,1 ml, have been found to be suitable when used with mixing chambers of volumes which are in practical use, and when used to prepare gas mixtures for the calibration of ambient-air analysers.

NOTE 2 It is recommended that the internal volume of the syringe be measured experimentally with a maximum relative uncertainty of $\pm 1\%$ (at a level of confidence of 95 %). In addition, the syringe should have a maximum leak rate of 10×10^2 Pa (10 mbar) per hour after evacuation to 5 Pa (5×10^{-2} mbar), in order that it has satisfactory leaktightness.

5.3 Apparatus for filling the metering syringe, consisting of the components specified in 5.3.1 to 5.3.9.

NOTE An example of a suitable set-up for filling the syringe is described in Annex A.

5.3.1 Evacuatable gas reservoir, capable of containing gas at above-atmospheric pressure so as to enable the metering syringe to be filled to that pressure, its internal surfaces being made of a material that does not react with any of the calibration components.

NOTE A gas reservoir with an internal volume of about 100 ml, capable of operating up to a pressure of $1,4 \times 10^5$ Pa (1,4 bar) and of maintaining a vacuum of better than $0,1 \times 10^2$ Pa (0,1 mbar), has been found suitable.

5.3.2 High-pressure gas cylinder, containing the selected pure gas component (or a pre-mixture containing a higher concentration of gas mixture).

5.3.3 Pressure regulator, used to enable the pressure of gas in the reservoir to be adjusted to a pre-defined pressure above that of the ambient atmosphere.

5.3.4 Septum, constructed of appropriate material, enabling the needle of the metering syringe to be introduced into the gas reservoir.

5.3.5 Vacuum pump, enabling the gas reservoir and its associated components to be evacuated to below the required vacuum.

NOTE It is important to ensure that any gaseous component which may be hazardous, and which is exhausted by the vacuum pump to the atmosphere, is vented in a safe way.

5.3.6 Pressure gauge, used to monitor the pressure of the gas in the reservoir during the various stages of the preparation procedure.

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5.3.7 Gas shut-off valves, used to isolate the gas cylinder from the reservoir, and the gas reservoir from the vacuum pump.

5.3.8 Pressure-relief valve, capable of relieving a gas pressure selected to protect the apparatus (typically $1,4 \times 10^5$ Pa). It should be vented to a safe location.

5.3.9 Suitable vessel, for use when the metering syringe is to be filled with a volatile liquid rather than a gas, and enabling the syringe to be filled in a manner that prevents the ingress into the syringe of any components other than the volatile liquid.

6 Procedure for preparing the calibration gas mixture

6.1 Determination of the volume of the gas-mixing chamber

There are a number of ways by which the internal volume of the gas-mixing chamber may be determined in practice. The major component of this volume will be the internal volume of the empty vessel itself and this is normally measured by filling it with water, or with another liquid of known density, and then determining the increase in mass due to this liquid. However, other methods may be used where these have the required accuracy. Following this, the internal volumes of the components within the gas-mixing chamber shall be determined by, for example, geometric measurement or liquid displacement. Corrections shall then be made for the volumes of these additional components when determining the net volume of the chamber. Corrections for some of the components (e.g. the gas-mixing device) will lead to a smaller volume whereas others (e.g. the pressure gauge and the outlet pipes leading to the shut-off valves) will lead to an increase in volume.

These measurements of the volumes of the components that make up the total gas-mixing chamber (see 5.1) may have been carried out at different temperatures. In such cases, corrections shall be made, where significant, to convert the measured volumes to a common ambient temperature. A further correction may need to be made if the complete mixing chamber with all its components is used at a different ambient temperature, provided such a correction is significant.

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6.2 Conditioning the gas-mixing chamber before use

A new gas-mixing chamber will normally contain ambient air, and this may contain trace pollutants at levels that would affect the accuracy of the calibration gas mixture. In addition, the inside surfaces of the chamber may be contaminated with a surface layer which may react with some species which are put in the mixing chamber. It is therefore necessary to condition the mixing chamber before use so as to avoid any potential contamination of the calibration gas from these causes. Do this by evacuating the chamber to a pressure of less than 5×10^2 Pa (5 mbar). Then fill the chamber to above-atmospheric pressure with the complementary gas which is to be used in the preparation of the calibration gas mixture. Subsequently, connect the outlet from the mixing chamber to a set of instruments designed to measure air pollutants (e.g. analysers which monitor SO_2 , NO_x , CO and hydrocarbons). These shall have sufficient detection sensitivity to determine whether significant concentrations of the relevant gaseous impurities exist in the complementary gas in the mixing chamber at this time. Allow the complementary gas in the mixing chamber to flow into these analysers, and observe the concentrations of any impurities detected.

Repeat this procedure, involving the evacuation of the mixing chamber followed by re-filling with complementary gas, several times or until all relevant gaseous impurities are below the concentrations required to prevent significant contamination of the calibration gas mixtures to be prepared in the chamber. In circumstances where this cannot be achieved, either clean the mixing chamber by other means so as to remove the contaminants, or make a suitable correction when calculating the concentration of the calibration component in the calibration gas mixture so as to allow for such impurities.

In circumstances where such an analysis of the impurities present in the complementary gas is the only one that is carried out, the detection limits of the analysers used shall represent the upper limits for the concentrations of the impurities which could be present in the calibration gas mixture. In these cases, the concentrations represented by these detection limits shall be taken into account in the determination of the expanded uncertainty of the prepared calibration gas mixture. In cases, however, where additional, more