



SLOVENSKI STANDARD

SIST EN ISO 6142:2006

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Analiza plinov - Priprava kalibrirnih plinskih zmesi - Gravimetrijska metoda (ISO 6142:2001)

Gas analysis - Preparation of calibration gas mixtures - Gravimetric method (ISO 6142:2001)

Gasanalyse - Herstellung von Prüfgasen - Wägevverfahren (ISO 6142:2001)

Analyse des gaz - Préparation des mélanges de gaz pour étalonnage - Méthode gravimétrique (ISO 6142:2001)

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71.040.40 Kemijska analiza Chemical analysis

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN ISO 6142

August 2006

ICS 71.040.40

English Version

Gas analysis - Preparation of calibration gas mixtures - Gravimetric method (ISO 6142:2001)

Analyse des gaz - Préparation des mélanges de gaz pour
étalonnage - Méthode gravimétrique (ISO 6142:2001)

Gasanalyse - Herstellung von Prüfgasen - Wägeverfahren
(ISO 6142:2001)

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

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of 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.

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

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EN ISO 6142:2006 (E)**Foreword**

The text of ISO 6142:2001 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 6142:2006 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 6142:2001 has been approved by CEN as EN ISO 6142:2006 without any modifications.

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INTERNATIONAL STANDARD

ISO 6142

Second edition
2001-04-01

Gas analysis — Preparation of calibration gas mixtures — Gravimetric method

*Analyse des gaz — Préparation des mélanges de gaz pour étalonnage —
Méthode gravimétrique*

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ISO 6142:2001(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 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 6142 was prepared by Technical Committee ISO/TC 158, *Analysis of gases*, in collaboration with ISO/TC 193, *Natural gas*.

This second edition cancels and replaces the first edition (ISO 6142:1981), which has been revised to update the methods of preparation, estimation of the uncertainty and of validation of gravimetrically prepared calibration gases.

Annexes A to G of this International Standard are for information only.

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Gas analysis — Preparation of calibration gas mixtures — Gravimetric method

1 Scope

This International Standard specifies a gravimetric method for the preparation of calibration gas mixtures in cylinders of which the target accuracy of the composition has been pre-defined. It is applicable only to mixtures of gaseous or totally vaporized components which do not react with each other or with the cylinder walls. A procedure is given for a method of preparation based on requirements for the final gas mixture composition to be within pre-set levels of uncertainty. Multi-component gas mixtures (including natural gas) and multiple dilution mixtures are included in this International Standard and are considered to be special cases of the single component gravimetric preparation method.

This International Standard also describes the procedure for verifying the composition of gravimetrically prepared calibration gases. Provided rigorous and comprehensive quality assurance and quality control procedures are adopted during the preparation and validation of these gravimetric gas mixtures, calibration gases of the highest accuracy can be obtained for a wide range of gas mixtures, in comparison with other methods of preparing such gases.

2 Normative references

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The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 6141, *Gas analysis — Requirements for certificates for calibration gases and gas mixtures*.

ISO 6143:—1), *Gas analysis — Comparison methods for determining and checking the composition of calibration gas mixtures*.

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*.

IUPAC, *Commission on atomic weights and isotopic abundances: Atomic Weights of the Elements, biennial review*.

3 Principle

Calibration gas mixtures are prepared by transferring parent gases (pure gases or gravimetrically prepared mixtures of known composition) quantitatively from supply cylinders to the cylinder in which the calibration gas mixture will be contained. The amount of gaseous component added from the parent gas is determined by weighing after each successive addition.

1) To be published. (Revision of ISO 6143:1981)

ISO 6142:2001(E)

The amount of parent gas added to the cylinder in which the calibration gas mixture will be contained is determined by weighing either the supply cylinder or, alternatively, the cylinder in which the calibration gas mixture will be contained, before and after each addition. The difference in these two weighings corresponds to the mass of the gas added. The choice between these two weighing methods depends on which one represents the most suitable procedure for preparing the specified mixture. For example, the addition of small amounts of a specified component may best be performed by weighing a small, low-volume supply cylinder, before and after addition, on a highly sensitive, low-capacity balance.

A single-step preparation method may be used where the amount of each gaseous component required is large enough to accurately measure the mass of the cylinder, in which the calibration gas mixture will be contained, at each addition within the required composition uncertainty of the final calibration gas mixture. Alternatively, a multiple dilution method may be used to obtain a final mixture with acceptable uncertainty, particularly when low concentrations of the minor components are required. In this method, "pre-mixtures" are gravimetrically prepared and used as parent gases in one or more dilution steps.

The mass fraction of each component in the final calibration gas mixture is then given by the quotient of the mass of that component to the total mass of the mixture.

The gravimetric method scheme for preparing calibration gas mixtures, based on pre-set requirements for composition and the level of uncertainty, is given as a flow chart in Figure 1. The individual steps are explained in more detail in clause 4 (reference is given to the subclause for each step in Figure 1). An example of the gravimetric method scheme for preparing a calibration gas mixture following the Figure 1 flow chart is given in annex A.

4 Preparation of the mixture

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4.1 Mixture composition and uncertainty

The composition of the final gas mixture is, by the principle of the gravimetric method, defined by the mass of each component. Gas composition is preferentially expressed as a mole fraction (mol/mol). If other quantities of composition are required (for example mass concentration or volume fraction) then the applicable conditions (pressure and temperature) shall be given and the additional uncertainty contributions shall be determined and considered in the calculation of the uncertainty in the composition of the calibration gas. The uncertainty of the final mixture composition is expressed as an expanded uncertainty, i.e. the combined standard uncertainty multiplied by a coverage factor.

The molar masses of the components, and their uncertainties, needed for the conversion of mass fraction to mole fraction, shall be derived using the most recent publication of the commission on atomic weights and isotopic abundances of the International Union of Pure and Applied Chemistry (IUPAC).

4.2 Feasibility of obtaining the gas mixture

4.2.1 General

Gas mixtures potentially capable of reacting dangerously shall be excluded for safety reasons. These phenomena shall be taken into account when considering the feasibility of preparing the required gas mixture, described in 4.2.2 to 4.2.4.

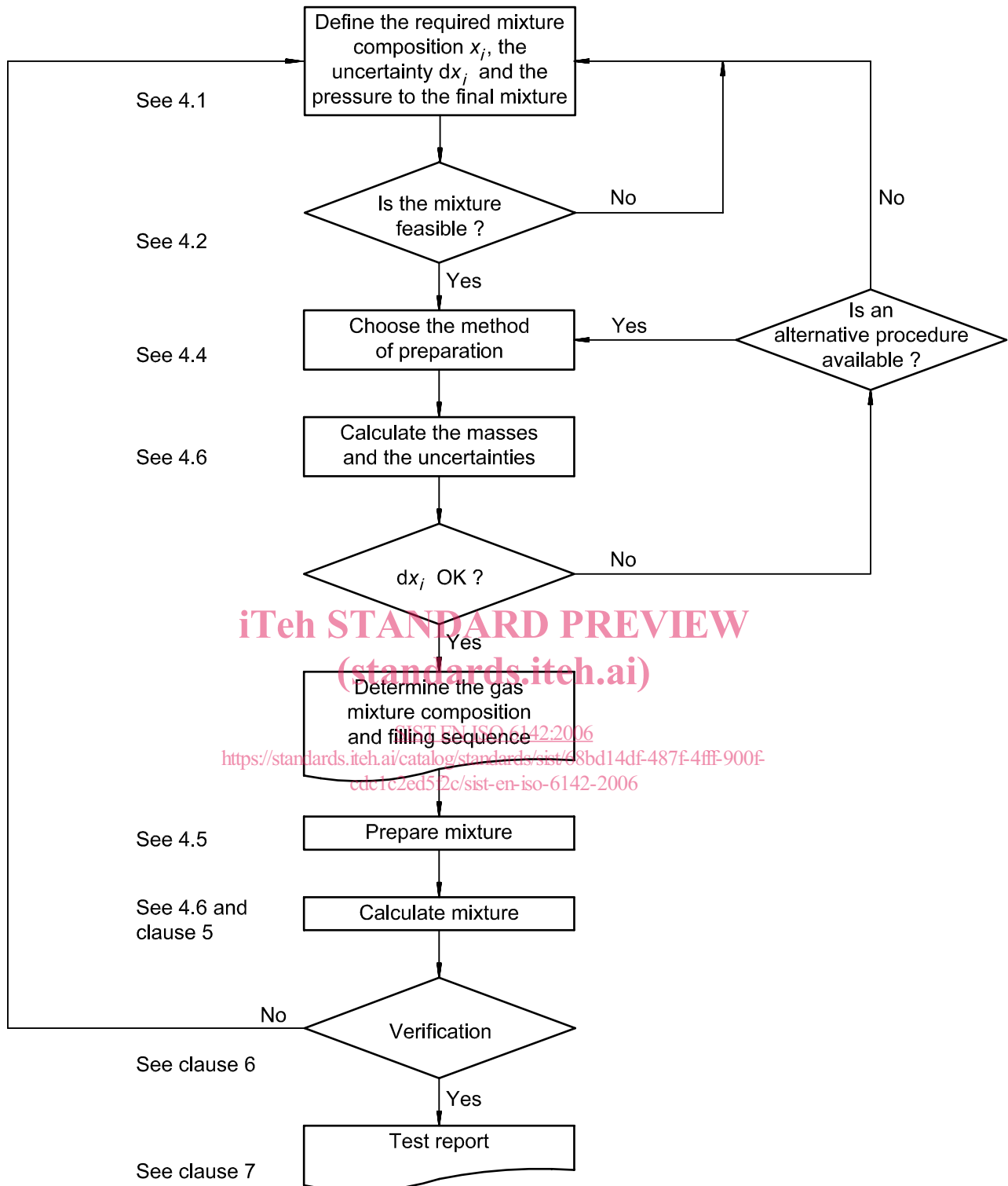


Figure 1 — Gravimetric method scheme for preparing calibration gas mixtures

ISO 6142:2001(E)**4.2.2 Condensation of the vapour to either a liquid or a solid phase**

When preparing, storing or handling gas mixtures which contain condensable components (see annex B), the following measures shall be taken to prevent condensation because loss by condensation will change the gas phase composition.

- During the preparation of the gas mixture, the filling pressure shall be set safely below the dew-point vapour pressure of the final mixture at the filling temperature. To prevent condensation at intermediate stages, this condition shall be fulfilled for every intermediate mixture as well. If condensation of an intermediate mixture cannot be safely excluded, measures shall be taken to vaporize any possible condensate and to homogenize the gas phase at an appropriate later stage.
- During the storage of the gas mixture, the storage temperature shall be set so as to maintain the filling pressure safely below the dew-point vapour pressure of the mixture at that temperature.
- During the handling of the gas mixture, the same condition on the handling temperature applies. Furthermore, to prevent condensation during mixture transfer, the transfer lines shall be heated if required.

In informative annex B, some guidance is given for estimating the maximum filling pressure for introducing components of a gas mixture at which no condensation of the condensable components is expected to occur. An example of this estimation is given in B.2 for a natural gas mixture.

4.2.3 Reactions between mixture components

Before preparing a gas mixture, it is necessary to consider possible chemical reactions between the components of the mixture. The method cannot be used to prepare mixtures

- containing potentially interactive substances (e.g. hydrochloric acid and ammonia),
- producing other possible dangerous reactions including explosions (e.g. mixtures containing flammable gases and oxygen),
- producing strong exothermic polymerizations (e.g. hydrogen cyanide), and
- which can decompose (e.g. acetylene).

Exceptionally this method can be used for substances undergoing dimerization, such as NO_2 to N_2O_4 , which is a reversible reaction.

A comprehensive compilation of reactive combinations is not available. Therefore, chemical expertise is necessary to assess the stability of a gas mixture.

For dangerous reactions and dangerous combinations, to be excluded for safety reasons, some information can be found in regulations on dangerous goods and in gas supplier handbooks.

4.2.4 Reactions with container materials

Before preparing a gas mixture, it is necessary to consider possible chemical reactions of mixture components with materials of a high-pressure cylinder, its valve and the transfer system. Special consideration shall be given to the attack by corrosive gases with metals and possible reactions with elastomers and greases used, for example, in the valve seat and seals. Such reactions should be prevented by using only materials that are inert to all components of the mixture. If this is not possible, measures shall be taken to minimize corrosive attack on the materials with which the gases make contact so as to prevent any significant effect on mixture composition and any danger in storage and use.

Information on the compatibility of gases with container materials is given in gas sampling guidelines, corrosion tables and gas supplier handbooks.

4.3 Purity analysis of primary gas standards

The accuracy achievable by the gravimetric method will depend significantly on the purity of the parent gases used for the preparation of the calibration gas mixture. Impurities in the parent gases are often one of the most critical contributors to the uncertainty of the final mixture composition. The uncertainty contributions depend on the amount of impurities present in the pure, parent gases and upon the accuracy with which these impurities have been measured. In many cases the purity of the major component (matrix gas) is of most importance. This is especially true when the mole fraction of the minor component is low and is likely to be an impurity in the major component. It is also important to evaluate critical impurities that may react with the minor component (e.g. oxygen present in pure nitrogen will react with NO to form NO₂). The result of purity analysis of parent gases shall be incorporated into a purity table containing the mole (or mass) fractions of all components with accompanying uncertainties derived from analysis.

Generally, impurities in a nominally “pure” parent gas are established by analysis and the mole fraction of the major component is conventionally determined by difference such that

$$x_{\text{pure}} = 1 - \sum_{i=1}^N x_i \quad (1)$$

where

x_i is the mole fraction of impurity i , determined by analysis;

N is the number of impurities likely to be present in the final mixture;

x_{pure} is the mole fraction “purity” of the “pure” parent gas.

When an impurity, likely to be present in the “pure” parent gas, is not detectable by the analytical method used, the mole fraction of the expected impurity shall be set equal to half of the value of the detection limit of the analytical method. The uncertainty of the determination of this mole fraction is based upon a rectangular distribution between zero and the value of the detection limit of the analytical method. In this way, the gravimetric method assumes that there is an equal likelihood that the impurity may be present in the “pure” parent gas at a level up to its value of the detection limit. Hence, the content of an undetected impurity forms a rectangular distribution from which its standard uncertainty is defined as half the value of the detection limit divided by $\sqrt{3}$.

4.4 Choice of preparation procedure

When choosing a suitable preparation procedure, a number of considerations shall be made to ensure the most appropriate method is used. The following is a list of parameters which shall be considered:

- pressure at which the gases are available and possibility of condensation (see annex B);
- maximum filling pressure of the cylinder to be used;
- established composition of each parent gas mixture used;
- filling method, i.e. direct method, multiple dilution, transfer method (use of small cylinder separately weighed on a low-capacity, high-resolution balance);
- characteristics of the type of balance to be used with its determined performance specifications;
- requirements for the preparation tolerance.

First calculate the value of the masses desired, or target masses m_i , of each component i , using equation (2).