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Analiza plinov - Priprava kalibracijskih plinskih zmesi - 1. del: Gravimetrijska metoda za zmesi razreda I (ISO 6142- 1:2015)

Gas analysis - Preparation of calibration gas mixtures - Part 1: Gravimetric method for Class I mixtures (ISO 6142- 1:2015)

Gasanalyse - Herstellung von Prüfgasen - Teil 1: Wägeverfahren für Prüfgase der Klasse I (ISO 6142- 1:2015)

Analyse des gaz - Préparation des mélanges de gaz pour étalonnage - Partie 1: Méthode gravimétrique des mélanges Classe I (ISO 6142- 1:2015)

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Gas analysis - Preparation of calibration gas mixtures - Part 1: Gravimetric method for Class I mixtures (ISO 6142-1:2015)

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Gasanalyse - Herstellung von Kalibriergasen - Teil 1: Wägeverfahren für Gemische der Klasse I (ISO 6142-1:2015)

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European foreword

This document (EN ISO 6142-1:2015) has been prepared by Technical Committee ISO/TC 158 "Analysis of gases".

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of an identical text or by endorsement, at the latest by March 2016, and conflicting national standards shall be withdrawn at the latest by March 2016.

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

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INTERNATIONAL STANDARD

ISO 6142-1

First edition
2015-08-01

Gas analysis — Preparation of calibration gas mixtures —

Part 1: Gravimetric method for Class I mixtures

Analyse des gaz — Préparation des mélanges de gaz pour étalonnage —
Partie 1: Méthode gravimétrique pour les mélanges de Classe I

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 158, *Analysis of gases*.

This first edition of ISO 6142-1 cancels and replaces ISO 6142:2001, which has been technically revised to update the methods of preparation, estimation of the uncertainty, and validation of the composition of gravimetrically prepared calibration gases. It also incorporates the Amendment ISO 6142:2001/Amd.1:2009.

ISO 6142 consists of the following parts, under the general title *Gas analysis — Preparation of calibration gas mixtures*:

— *Part 1: Gravimetric method for Class I mixtures*

A future part dealing with gravimetric method for Class II mixtures.

Introduction

The revision of ISO 6142 was initiated to provide better guidance to the users of this International Standard especially with respect to quality assurance measures and laboratory accreditation. In preparing the revision, it was decided to make accommodation for two types of calibration gas mixtures with different levels of quality assurance and with different levels of measurement uncertainty. The difference in the two classes can be summarized as follows:

Class I type calibration gas mixtures are prepared in accordance with this part of ISO 6142. The mixtures are individually verified. Provided rigorous and comprehensive quality assurance and quality control procedures are adopted during the preparation and verification of these mixtures, uncertainties may be achieved that are substantially smaller than by any other preparation method.

Class II type calibration gas mixtures are prepared in a similar manner to Class I calibration gas mixtures but these mixtures are not individually verified. Verification of Class II calibration gas mixtures can be based on random verification checks. These checks are monitored by means of statistical quality control to be described in a future part. For mixtures containing identical compounds and nominally identical amount–of–substance fractions, Class II type calibration gas mixtures will always have amount–of–substance fractions with larger uncertainties than their Class I counterparts.

Gas analysis — Preparation of calibration gas mixtures —

Part 1:
Gravimetric method for Class I mixtures

1 Scope

This part of ISO 6142 specifies a gravimetric method for the preparation of calibration gas mixtures in cylinders with traceable values for the amount-of-substance fraction (amount fraction) of one or more components. This part of ISO 6142 describes a method for calculating the uncertainty associated with the amount fraction of each component. This uncertainty calculation requires the evaluation of the contributions to the uncertainty due to factors including the weighing process, the purity of the components, the stability of the mixture, and the verification of the final mixture.

This part of ISO 6142 is only applicable to mixtures of gaseous or totally vaporized components, which may be introduced into the cylinder in the gaseous or liquid state. Both binary and multi-component gas mixtures (including natural-gas type mixtures) are covered by this part of ISO 6142. Methods for the batch production of more than one mixture in a single process are not included in this part of ISO 6142.

This part of ISO 6142 requires estimation of the stability of the mixture for its intended life time (maximum storage life), but it is not for use with components that react with each other unintentionally. This part of ISO 6142 also requires the impurities in each parent gas or liquid used in the preparation of the mixture to be assessed and quantified.

2 Normative references

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

ISO 6141, *Gas analysis — Contents of certificates for calibration gas mixtures*

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

ISO 7504, *Gas analysis — Vocabulary*

ISO 14912, *Gas analysis — Conversion of gas mixture composition data*

ISO 16664, *Gas analysis — Handling of calibration gases and gas mixtures — Guidelines*

ISO 19229, *Gas analysis — Purity analysis and the treatment of purity data*

ISO/TS 29041, *Gas mixtures — Gravimetric preparation — Mastering correlations in composition*

ISO/IEC Guide 98-3, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

IUPAC, Commission on atomic weights and isotopic abundances: Atomic weights of the elements

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 7504 and ISO/IEC Guide 98-3 apply.

4 Symbols

A_z	atomic weight of element z
b_i	estimated amount fraction drift rate of component i
k	coverage factor
L_{ij}	limit of detection of impurity i in parent gas or liquid j
M_i	molar mass of component i
M_j	molar mass of parent gas or liquid j
M_k	molar mass of component k
M_Ω	molar mass of the final mixture
m_j	mass added of parent gas or liquid j
m_Ω	mass of the final mixture
q	number of components in the mixture
r	number of parent gases or liquids
p_F	filling pressure
$p_{F,\Omega}$	filling pressure of the final mixture
$p_i(T_L)$	saturated vapour pressure of component i at temperature T_L
R	ideal gas constant
T_F	filling temperature
T_L	lowest temperature to which the gas mixture will be exposed
t_d	decay time
t_s	shelf life of the mixture
$u(\ldots)$	standard uncertainty (of the quantity in parentheses)
$U(\ldots)$	expanded uncertainty (of the quantity in parentheses)
V_{cyl}	volume of the cylinder
ν_{zi}	stoichiometric coefficient for element z
w_i	mass fractions w_i of the components i in the final mixture
$w_{i,j}$	mass fraction of component i in parent gas or liquid j
x_c	amount-of-substance fraction of the “pure” component in the material being analysed
x_i	amount-of-substance fraction of component i
$x_{i,j}$	amount-of-substance fraction of component i in parent gas or liquid j
$x_{k,j}$	amount-of-substance fraction of component k in parent gas or liquid j
y_k^0	amount-of-substance fraction of component k at time $t = 0$

y_k^t	amount-of-substance fraction of component k at time t
y_i	amount-of-substance fraction of component i in the prepared mixture
y_k	amount-of-substance fraction of component k in the prepared mixture
$y_{k,\mathrm{ver}}$	analysed amount-of-substance fraction
Z_Ω	compressibility of the final mixture
Ω	final mixture

5 Principle

Calibration gas mixtures are prepared by transferring pure gases, pure liquids, or gravimetrically prepared mixtures of known composition quantitatively into a cylinder in which the calibration gas will be contained. The traceability to the SI of amount fractions of these mixtures arises from the correct execution of three steps:

a) the determination of the masses added;

b) the conversion of the added masses to amounts of substance, by knowledge of their chemical purity and appropriate relative atomic and/or molecular masses;

c) the verification of the final mixture against independent reference gas mixtures.

For Class II type calibration gas mixtures, the individual verification of the final mixture against independent reference gas mixtures is not required. The verification of Class II type calibration gas mixtures is described in a future part.

The mass of each component is determined by weighing either the supply cylinder, or 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 component added. The choice between these two weighing procedures depends on the uncertainty required for the amount fraction of the final mixture. Annex A provides more guidance on precautions to be taken when weighing, handling, and filling cylinders.

NOTE In the case of an addition of a small mass of a specified component, a highly sensitive balance is needed. If such a balance has insufficient capacity to weigh the final mixture, a small added mass can best be determined by weighing a low-volume supply cylinder before and after addition of the component to the main cylinder.

A single-step preparation method may be used when the mass of each component added is large enough to be measured accurately. Alternatively, a multiple-step dilution method may be used to obtain a final mixture with acceptable uncertainty, particularly when low amount fractions are required. In this method, “pre-mixtures” are prepared gravimetrically and used as parent gases in one or more of the steps.

An example of the steps used to prepare a calibration gas mixture is given in Annex B.

The determination of the purity of each material (liquid or gas) used in the preparation of the mixture is described in Clause 7. Clause 8 describes the determination of masses and the calculation of preparation uncertainty. The homogeneity and stability of the gas mixture are dealt with in Clause 9. The verification of the amount fraction of the components in the final mixture against independent standards is described in Clause 10. The calculation of the uncertainty of the calibration gas mixture is given in Clause 11.

The gravimetric method scheme for preparing calibration gas mixtures, based on requirements for composition and the level of uncertainty, is given as a flowchart in Figure 1. The individual steps are explained in more detail in the following clauses (reference is given to the subclause for each step in Figure 1).