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**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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Printed 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 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.

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## 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.

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

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.

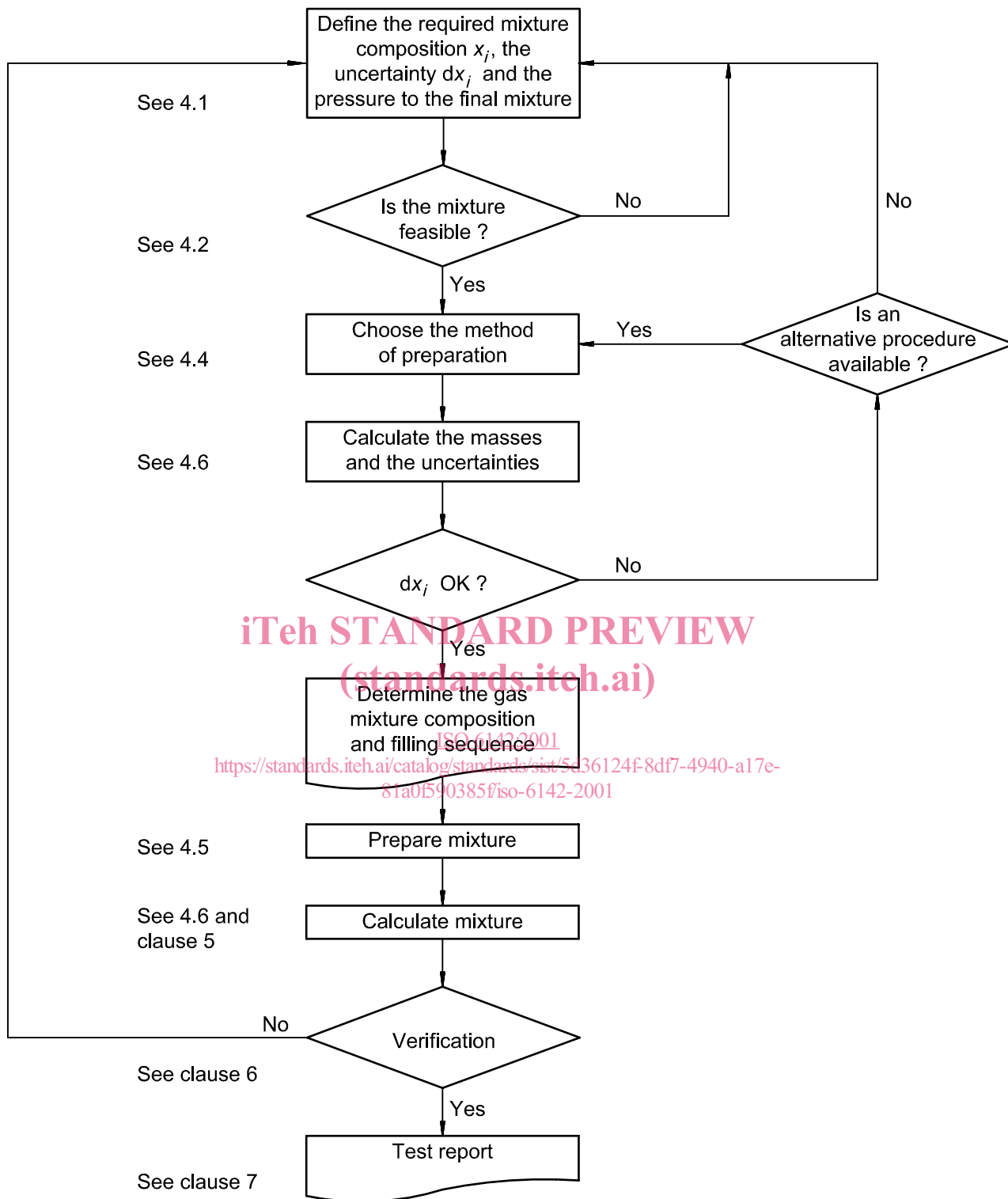


Figure 1 — Gravimetric method scheme for preparing calibration gas mixtures

#### 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  $\text{NO}_2$  to  $\text{N}_2\text{O}_4$ , 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 NO<sub>2</sub>). 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;

$x_{\text{pure}}$  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).

$$m_i = \frac{x_i M_i}{\sum_{j=1}^N x_j M_j} \cdot m_f \quad (2)$$

where

- $x_i$  is the mole fraction of component  $i$ ;
- $x_j$  is the mole fraction of component  $j$ ;
- $M_i$  is the molar mass of component  $i$ ;
- $M_j$  is the molar mass of component  $j$ ;
- $N$  is the number of components in the final mixture;
- $m_f$  is the mass of final mixture.

After the target masses have been calculated, a preparation procedure is selected and the uncertainties associated with the preparation process are calculated. If the calculated uncertainty for that procedure proves to be unacceptable, another procedure shall be adopted. It may be necessary to perform an iterative process to select a procedure with acceptable uncertainty.

These considerations result in a preparation procedure whereby a filling sequence consisting of several stages is selected in which gases are transferred into a cylinder in which the calibration gas mixture will be contained and subsequently weighed. Each stage has its own associated uncertainty and when combined, remain within the required level of uncertainty. This procedure shall be used in the subsequent preparation.

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#### 4.5 Preparation of the mixture

Precautions to be taken when weighing, handling and filling cylinders are given for information in annex C.

To achieve the intended composition of the mixture, a tool is required. Normally the parameters used in targeting this composition are pressure and/or mass. When pressure is used for targeting this composition, temperature effects, resulting from the pressurization and the compressibility of the introduced components, is of importance. In particular, non-ideal behaviour of certain components makes it difficult to establish a simple relationship between added pressure and added mass. However, the compression factor, which quantifies these deviations from ideal behaviour, is a function of pressure, temperature and composition and can be calculated and used to predict the required pressure.

A more direct way of targeting the desired masses is by use of a balance on which the cylinder is placed to observe the difference in mass which occurs during transfer.

## 4.6 Calculation of the mixture composition

The mole fractions of the components in the final mixture are calculated using equation (3):

$$x_i = \frac{\sum_{A=1}^P \left( \frac{x_{i,A} \cdot m_A}{\sum_{i=1}^n x_{i,A} \cdot M_i} \right)}{\sum_{A=1}^P \left( \frac{m_A}{\sum_{i=1}^n x_{i,A} \cdot M_i} \right)} \quad (3)$$

where

- $x_i$  is the mole fraction of the component  $i$  in the final mixture,  $i = 1, \dots, n$ ;
- $P$  is the total number of the parent gases;
- $n$  is the total number of the components in the final mixture;
- $m_A$  is the mass of the parent gas  $A$  determined by weighing,  $A = 1, \dots, P$ ;
- $M_i$  is the molar mass of the component  $i$ ,  $i = 1, \dots, n$ ;
- $x_{i,A}$  is the mole fraction of the component  $i$ ,  $i = 1, \dots, n$ , in parent gas  $A$ ,  $A = 1, \dots, P$ .

A method for deriving this formula is given for information in annex D.

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## 5 Calculation of uncertainty

**5.1.1** The uncertainty in the values of the mole or mass fractions of the components in a gravimetrically prepared calibration gas mixture indicates the dispersion of values which can reasonably be attributed to these fractions.

The procedure for evaluating the uncertainty may be summarized in 5.1.2 to 5.1.7.

**5.1.2** Identify the steps taken in the preparation procedure. Following equation (3) in 4.6, three categories can be identified that will influence the uncertainty:

- the uncertainty in the weighing of the parent gases;
- the uncertainty in the purity of the parent gases;
- the uncertainty in molar masses.

**NOTE** The parent gases may themselves be gravimetrically prepared mixtures.

**5.1.3** For each step in the gravimetric preparation procedure, a list shall be made of all sources of uncertainty, i.e., a list of all factors that may influence the resulting composition. A list of possible error sources is given for information in annex E. Some of these uncertainty contributions, for example the standard deviation in the repeated weighings, can be determined by repeated measurements (type A evaluation). For a well-characterized measurement under statistical control, a combined or pooled estimate of variance  $s_p^2$  (or a pooled experimental standard deviation  $s_p$ ) that characterizes the measurement may be available. In such cases, when the value of the measurand  $q$  is determined from  $n$  independent observations, the experimental variance of the arithmetic mean  $\bar{q}$

of the mean observations is more closely estimated by  $s_p^2/n$  than by  $s_q^2/n$  and the standard uncertainty more closely estimated by  $u = s_p/\sqrt{n}$ . For uncertainty contributions that cannot be estimated by repeated measurements (type B evaluation), a realistic evaluation should be made to estimate this contribution. This applies, for example, to adsorption/desorption effects and thermal effects on the cylinder that influence the balance. Variations in some parameters can be decreased by monitoring and/or controlling these and then calculating the appropriate correction factors. For example, the uncertainty of the buoyancy effect may be decreased by accurately monitoring the ambient pressure, humidity and temperature conditions and using these to calculate the density of air at the time of weighing. Each significant uncertainty contribution shall be evaluated as a standard uncertainty, i.e. as a single standard deviation.

NOTE More details about type A and type B evaluations of standard uncertainty are given in the Guide to expression of uncertainty in measurement [17].

**5.1.4** For each contribution to the total uncertainty, decide which ones merit evaluation (significant contributions) and which ones can be neglected (insignificant contributions). As the total certainty is the sum of squared contributions, a contribution equalling less than 1/10 of the largest contribution can safely be neglected.

NOTE This method cannot always be applicable to the purity analysis of the parent gases, as some insignificant impurities can be critical to the mixture under preparation (for example, some impurities can react with the minor component). In such cases, an evaluation of the influence of the parent gas purity on the contribution to total uncertainty is necessary.

**5.1.5** The combined uncertainty due to the contributions from the molar masses of the components, the weighing results and the purity analyses is obtained by uncertainty propagation of equation (3) in 4.6. In this equation, the targeted component quantities  $x_i$  are expressed as functions of a number of input quantities  $y_1, y_2, \dots, y_q$ , i.e.

$$x_i = f_i(y_1, y_2, \dots, y_q) \tag{4}$$

Here the input quantities  $y_i$  comprise the molar masses  $M_i$ , the parent gas masses  $m_A$ , and the mole fractions  $x_{j,A}$ . According to the rules of uncertainty propagation the standard uncertainties  $u(x_i)$  are given as follows:

$$u^2(x_i) = \sum_{r=1}^q \left( \frac{\partial f_i}{\partial y_r} \right)^2 \cdot u^2(y_r) + 2 \sum_{r=1}^{q-1} \sum_{s=r+1}^q \left( \frac{\partial f_i}{\partial y_r} \right) \left( \frac{\partial f_i}{\partial y_s} \right) \cdot u(y_r, y_s) \tag{5}$$

In this equation  $u(y_r)$  are the standard uncertainties of the input quantities, and  $u(y_r, y_s)$  are the covariances between different input quantities, should these be correlated.

Correlations can be made, for example, between different parent gas masses  $m_A, m_B$ , if these are determined as differences between the results of successive weighings. Correlations can also be made between the mole fractions  $x_{j,A}, x_{k,A}$  of different components in the same parent gas, due to the fact that these mole fractions add up to unity. To avoid making such correlations, the primary input quantities can be considered instead. For example, in the case of parent gas masses, these are the results of successive weighings, starting with the empty cylinder in which the calibration gas mixture is to be contained (see informative annex C). In the case of the mole fractions of parent gas components, the problem of correlation can be resolved by expressing the mole fraction of the main component as the difference of the sum of the mole fractions of all the other components from unity (see 4.3), which are generally uncorrelated. If, in this manner, the targeted component quantities  $x_i$  have been expressed as functions of uncorrelated input quantities  $z_1, z_2, \dots, z_p$ , i.e.

$$x_i = g_i(z_1, z_2, \dots, z_p) \tag{6}$$

the combined uncertainties  $u(x_i)$  are simply given by:

$$u^2(x_i) = \sum_{t=1}^p \left( \frac{\partial g_i}{\partial z_t} \right)^2 \cdot u^2(z_t) \tag{7}$$

or by:

$$u^2(x_i) = \sum_{t=1}^p [c_i \cdot u(z_t)]^2 \equiv \sum_{t=1}^p u_t^2(x_i) \quad (8)$$

where  $c_i$ , the sensitivity coefficient is given by:

$$c_i \equiv \frac{\partial g_i}{\partial z_t}, \quad u_t(x_i) \equiv |c_i| \cdot u(z_t)$$

**5.1.6** The total uncertainty is given by the result of this calculation, combined with the contributions from all other sources of significant error. Simple methods for evaluating uncertainty contributions based on estimates of maximum errors are described in annex F.

**NOTE** The total uncertainty is only applicable to stand-alone applications of single analyte contents. In any joint application of several analyte contents or the complete composition, covariances have to be taken into account, whose estimation is beyond the scope of this International Standard.

**5.1.7** In order to obtain the expanded uncertainty, the combined standard uncertainty is multiplied by the coverage factor,  $k$ .

**NOTE 1** A coverage factor,  $k$ , is typically in the range from 2 to 3.

**NOTE 2** Within ISO TC 158, a coverage factor of  $k = 2$  has been agreed, unless specific reasons necessitate another choice.

**NOTE 3** For a normal distribution, a coverage factor of  $k = 2$  corresponds to a coverage probability of approximately 95 %.

More information on the estimation of corrections and correction uncertainty is given for information in annex F. A computer program implementing the recommended methods for calculating gas mixture composition as well as uncertainty is given for information in annex G.

## 6 Verification of calibration gas mixture composition

### 6.1 Objectives

The objective of verifying the composition of a calibration gas mixture is to check that the composition, calculated from the gravimetric process, is consistent with measurements made on the mixture by independent means, for example by an analytical comparison method. This verification acts to highlight significant errors in the preparation process of the individual gas mixture. Moreover, further checks may be required over a period of time to demonstrate the stability of specific mixtures.

Verification of the composition of a gas mixture may be achieved by:

- a) establishing consistency between prepared mixtures and appropriate traceable standards (see note);
- b) establishing consistency between several nominally similar prepared mixtures;
- c) monitoring continuing production of validated mixtures using a suitable statistical process control method.

**NOTE** A traceable standard refers to a mixture of appropriate metrological quality that is traceable, through an unbroken chain of comparisons with stated uncertainties, to a national or an International Standard.

When seeking to verify a prepared mixture and confirm its composition, the mixture components may all be in a range where suitable traceable standards are readily available, so that consistency may easily be demonstrated. However, often the reason for relying on gravimetric preparation is that one or more components are outside the range where traceable standards already exist, so verification shall be performed by other methods, such as