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Gas analysis - Preparation of calibration gas mixtures using dynamic methods - Part 1: General aspects (ISO/DIS 6145-1:2018)

Gasanalyse - Herstellung von Kalibriergasgemischen mit Hilfe von dynamische Verfahren - Teil 1: Kalibrierverfahren (ISO/DIS 6145-1:2018)

Analyse des gaz - Préparation des mélanges de gaz pour étalonnage à l'aide de méthodes volumétriques dynamiques - Partie 1 : Méthodes d'étalonnage (ISO/DIS 6145-1:2018)

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Part 1:

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

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Foreword

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This document was prepared by Technical Committee ISO/TC 158 Gas Analysis.

This third edition cancels and replaces the second edition (ISO 6145-1:2003), which has been technically revised.

The main changes compared to the previous edition are as follows:

- The techniques for the preparation of gas mixtures are described in an abbreviated manner since there is no need to repeat the text and formulae from each of the different parts of the ISO 6145 series. However, a summary table presenting the advantages and limitations of each method has been introduced.
- Recommendations regarding the handling of the dynamic mixing systems and quality considerations have been added.
- The methods and instruments to calibrate a dynamic system have changed, and are better described.
- The calculations to obtain composition and uncertainties are more detailed, and take into account the different ways of mixing gases (volume flow rates or mass flow rates)
- Clauses on certificates and verification have been added.

A list of all parts in the ISO 6145 series can be found on the ISO website.

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Introduction

This document is one of a series of standards which describes the various dynamic methods for the preparation of calibration gas mixtures.

Several techniques are available and the choice between them is decided on the basis of the desired gas composition range, the consistency of equipment with the application and the required level of uncertainty. This document aids with making an informed choice by listing all the advantages and limitations of the methods.

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

- a) piston pumps
- b) continuous injection
- c) capillary
- d) critical orifices
- e) thermal mass-flow controllers
- f) diffusion

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- g) saturation
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- h) permeation
- i) electrochemical generation <u>SIST EN ISO 6145-1:2019</u>

In dynamic methods, a gas A is introduced at a known constant volume or mass flow rate into a known constant flow rate of a complementary gas B. Gases A and B can be either pure gases or gas mixtures. The preparation process can be continuous (e.g., mass flow controllers, permeation tube) or pseudocontinuous (e.g., piston pump).

The dynamic 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.

1 Gas analysis — Preparation of calibration gas mixtures using

2 dynamic methods — Part 1: Methods of calibration

3 1 Scope

- 4 This document gives a brief overview of each of the dynamic techniques which are described in detail in
- 5 the following parts of ISO 6145. This document provides basic information to support an informed choice
- 6 for one or another method for the preparation of calibration gas mixtures. It also describes how these
- 7 methods can be linked to national measurement standards to establish metrological traceability for the
- 8 composition of the prepared gas mixtures.
- 9 Since all techniques are dynamic and rely on flow rates, this first part of ISO 6145 describes the
- calibration process by measurement of each individual flow rate generated by the device.
- 11 Methods are also provided for assessing the composition of the generated gas mixtures by comparison
- with an already validated calibration gas mixture.
- 13 This document provides general requirements for the use and operation of dynamic methods for gas
- 14 mixture preparation. It also includes the necessary expressions for calculating the calibration gas
- 15 composition and its associated uncertainty.
- Gas mixtures obtained by these dynamic methods can be used to calibrate or control gas analysers.
- 17 The storage of dynamically prepared gas mixtures into bags or cylinders is beyond the scope of this
- 18 document.

19 **2 Normative references**

- The following documents are referred to in the text in such a way that some or all of their content
- 21 constitutes requirements of this document. For dated references, only the edition cited applies. For
- 22 undated references, the latest edition of the referenced document (including any amendments) applies.
- 23 ISO 6143, Gas analysis Comparison methods for determining and checking the composition of calibration
- 24 gas mixtures
- 25 ISO 7504, Gas analysis Vocabulary
- 26 ISO 12963, Gas analysis -- Comparison methods for the determination of the composition of gas mixtures
- 27 based on one- and two-point calibration
- 28 ISO 14912, Gas analysis Conversion of gas mixture composition data
- 29 ISO 19229, Gas analysis Purity analysis and the treatment of purity data

30 3 Terms and definitions

- For the purposes of this document, the terms and definitions given in ISO 7504 and the following apply.
- 32 **3.1**
- 33 mass flow rate
- $34 q_m$
- 35 mass of gas per unit of time

- 36 **3.2**
- 37 **volume flow rate**
- q_1
- 39 volume of gas per unit of time
- 40
- ISO and IEC maintain terminological databases for use in standardization at the following addresses:
- 42 IEC Electropedia: available at http://www.electropedia.org/
- 43 ISO Online browsing platform: available at https://www.iso.org/obp

44 **4 Symbols**

Symbol	Definition
i,k	Indices for components in a gas or gas mixture
j	Index for a parent gas
m	Mass of a component
М	Molar mass of a component
n	Number of parent gases
p	Pressure
q	Number of components in the gas mixture
q_m	Mass flow rate
q_n	Amount-of-substance flow rate
R	Ideal gas constant
r	Number of parent gases
T	Temperature
V	Volume
$q_{ u}$	Volume flow rate SISTEN ISO 6145-1:2019
v htt	Mass fraction of a component in a parent gas 345380-949a-4185-a3a1-
W	Mass fraction of a component in a gas mixture 1-2019
X	Amount-of-substance fraction of a component in a parent gas
У	Amount-of-substance fraction of a component in a gas mixture
Z	Compressibility factor
φ	Volume fraction of a component in a parent gas
ϕ	Volume fraction of a component in a gas mixture

45 **5 Principle**

46 **5.1 General**

- 47 All preparation techniques described in ISO 6145 (all parts) are based on the combination of gas flows.
- 48 These flow rates can be measured on a volume or mass basis. The composition is calculated from the flow
- rate data and the composition of the parent gases.
- 50 It is applicable only to
- 51 pure gases,
- 52 gas mixtures, or
- 53 totally vaporized components at ambient pressure.
- which do not react with each other or with any surfaces of the mixing device.

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- For the calculation of the composition, it is essential to appreciate the composition of the parent gases
- used for preparing the calibration gas mixture. Even if such gases are considered "pure", their purity shall
- be verified in accordance with ISO 19229. The corresponding compositional data of these parent gases
- shall be used in the calculation of the composition, as described in Clause 7 of this document.
- 59 Practically, all preparation systems are furthermore sensitive to changes or fluctuations in the conditions
- under which the calibration gas mixture is prepared. These conditions typically include the pressure and
- temperature of the gases, as well as the dynamic effects of combining flows and homogenization of the
- 62 calibration gas mixture, among others. In the subsequent parts of ISO 6145, attention is drawn to these
- effects, and care shall be taken to follow these instructions.
- 64 Several techniques are available and the choice between them should be decided based on the desired
- composition range, the availability of equipment and the required uncertainty.
- A set-up of a gas mixing system in principle is described in each part of ISO 6145.
- Depending on its principle, each dynamic method will generate gas mixtures of composition based on
- 68 either volume fraction, mass fraction, amount fraction or mass concentration. The calibration procedure
- 69 will also affect the expression of gas mixture composition (mass, volume or amount fraction). The final
- fraction and its associated uncertainty depend both on the calibration method and on the preparation
- 71 technique.

72

88

5.2 Suitability of the method to the application

- Before preparing a gas mixture, it is necessary to consider the suitability of the dynamic system to the
- application. Pressure and flow rates should be consistent with the analyser to which the dynamic system
- 75 will be linked.
- Be sure to respect the manufacturer recommendations. Check if the dynamic method is sensitive to
- external parameters, such as temperature or atmospheric pressure, and respect recommendations given
- 78 in each ISO 6145 part.
- 79 Depending on the principle used by the dynamic system, the achievable range of concentration in the
- 80 final mixture will differ. In order to compare the capabilities of each method, a dilution ratio is estimated
- as follows:
- use of pure components as parent components (for example: in cylinders, or permeation tubes or
 by syringe injection);
- by by mge mjection,
- only one step dilution is considered.
- 85 This dilution ratio could be extended for some dynamic systems by two step dilution.
- When choosing the technology of the dynamic method, the user shall take into consideration the
- advantages and limitations of each method.

5.3 Piston pumps

- 89 Part 2 of ISO 6145 specifies a volumetric method for the dynamic preparation of calibration gas mixture
- 90 using piston pumps. Two or more piston pumps, combined in a gas-mixing pump, are driven with a
- 91 defined ratio of strokes. The stroke volume of each piston pump is individually determined by the
- geometry (cross section) of the cylinder and the height of stroke of the piston. The composition is rapidly
- 93 changed by mechanical changing of the ratio of strokes. Suitable peripherals for gas feeding and
- homogenisation of the final mixture are recommended.

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- 95 The calibration of the stroke volume is made by dimensional measurements in the SI base unit of length.
- 96 Uncertainty evaluation of the gas mixture composition and an assessment of potential sources with
- 97 quantification of significant sources of uncertainty are out lined in detail in part 2 of ISO 6145.
- 98 By combination of piston pumps with appropriate stroke volumes and stroke ratios, the composition of
- 99 the calibration gas mixtures can be changed by dilution ratios from 1:1 up to 1:104. Higher dilution ratios
- can be prepared by a two stage dilution.
- The merits of the method are that the composition and the associated uncertainty of the calibration gas
- mixture are calculated from the geometric stroke volume and the ratio of strokes of the piston pumps.
- The content of each component is directly expressed in volume fractions and in amount fractions.
- Final mixture flow rates from 5 l/h to 500 l/h can be prepared depending on the equipment used.

105 **5.4 Continuous (syringe) injection**

- Part 4 of ISO 6145 specifies a method for preparation of calibration gas mixtures using continuous
- injection.
- The calibration component, either in the gaseous or liquid phase, is displaced from a reservoir through a
- capillary into a complementary inert gas stream. The system may contain a syringe, whose plunger is
- 110 continuously driven by a suitable variable-speed motor. Alternatively, the component may be forced
- through the capillary by controlled pressurization of the reservoir.
- This method applies also to multi-component mixtures of known composition. In the case of liquids, the
- calibration component is vaporized while mixing with the complementary gas.
- The flow rate of the calibration component is determined by the geometry (cross-section) of the syringe
- and the linear velocity of the plunger or by the continuous weighing of the reservoir.
- Using this method, gas mixtures can be prepared with dilution ratios from 1×10^4 up to 1×10^7 .

117 **5.5 Capillary**

- Part 5 of ISO 6145 specifies a method for the continuous preparation of calibration gas mixtures from
- pure gases or gas mixtures using capillary tubes in single or multiple combinations.
- A constant flow of gas from a capillary tube under conditions of constant pressure drop is added to a
- controlled flow of complementary gas. The complementary gas may be derived from another capillary
- 122 tube.
- This application is used in industrial gas mixing panels for the production of specific gas atmospheres.
- Gas dividers can be used to divide gas mixtures prepared from pure gases or gas mixtures into controlled
- proportions by volume.
- Using this method, gas mixtures of dilution ratios from 1:1 up to 1:10⁴ can be prepared.

127 **5.6 Critical flow orifices**

- Part 6 of ISO 6145 specifies a method for the continuous preparation of calibration gas mixtures by use
- of critical flow orifice systems.
- When passed through a critical orifice at increasing upstream pressure P_{in} , the volume flow rate of gas
- passing through the orifice will increase. When the ratio of the gas pressure downstream P_{out} and the gas
- pressure upstream P_{in} of the orifice has reached the critical value, the volume flow rate of the gas becomes
- independent with respect to P_{out} and is proportional to P_{in} .