INTERNATIONAL STANDARD

ISO 6145-10

First edition
2002-02-01

Gas analysis — Preparation of calibration gas mixtures using dynamic volumetric methods —

Part 10: Permeation method

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

Partie 10: Méthode par perméation

Reference number
ISO 6145-10:2002(E)

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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.

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 part of ISO 6145 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 6145-10 was prepared by Technical Committee ISO/TC 158, *Analysis of gases*.

It cancels and replaces ISO 6349:1979 which has been technically revised.

ISO 6145 consists of the following parts, under the general title *Gas analysis — Preparation of calibration gas mixtures using dynamic volumetric methods*:

— *Part 1: Methods of calibration*

— *Part 2: Volumetric pumps*

— *Part 4: Continuous injection method*

— *Part 5: Capillary calibration devices*

— *Part 6: Critical orifices*

— *Part 7: Thermal mass-flow controllers*

— *Part 9: Saturation method*

— *Part 10: Permeation method*

Diffusion will be the subject of a future part 8 to ISO 6145. Part 3 to ISO 6145, entitled *Periodic injections into a flowing gas stream*, has been withdrawn by Technical Committee ISO/TC 158, *Analysis of gases*.

Annex A of this part of ISO 6145 is for information only.

Introduction

This part of ISO 6145 is one of a series of standards dealing with various dynamic volumetric methods used for the preparation of calibration gas mixtures.

Gas analysis — Preparation of calibration gas mixtures using dynamic volumetric methods —

Part 10: Permeation method

1 Scope

This part of ISO 6145 specifies a dynamic method using permeation membranes for the preparation of calibration gas mixtures containing component mole fractions ranging from 10^{-9} and 10^{-6}. A relative expanded uncertainty of 2,5 % of the component mole fraction can be achieved using this method. In the mole fraction range considered, it is difficult to maintain some gas mixtures, for example in cylinders, in a stable state. It is therefore desirable to prepare the calibration gas immediately before use, and to transfer it by the shortest possible path to the place where it is to be used. This technique has been successfully applied in generating low content calibration gas mixtures of, for example, sulfur dioxide (SO_2), nitrogen dioxide (NO_2) and benzene (C_6H_6) in air.

If the carrier gas flow is measured as a gas mass-flow, the preparation of calibration gas mixtures using permeation tubes is a dynamic-gravimetric method which gives contents in mole fractions.

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this part of ISO 6145. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 6145 are encouraged to investigate the possibility of applying the most recent edition of the normative document 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 6145-1, *Gas analysis — Preparation of calibration gas mixtures using dynamic volumetric methods — Part 1: Methods of calibration*

3 Principle

The calibration component [for example SO_2, NO_2, ammonia (NH_3), benzene, toluene, xylene] is permeated through an appropriate membrane into the flow of a carrier gas, i.e. the complementary gas of the mixture obtained. The calibration component, of known purity, is contained in a tube, which is itself contained in a temperature-controlled vessel. This vessel is purged at a known and controlled flow rate by the carrier gas. The composition of the mixture is determined from the permeation rate of the calibration component as well as the flow rate of the high quality carrier gas, free from any trace of the calibration component and from any chemical interaction with the material of the permeation tube.

The permeation rate of the calibration component through the membrane depends upon the component itself, the chemical nature and structure of the membrane, its area and thickness, the temperature, and the partial pressure gradient of the calibration component across the membrane. These factors can be kept constant by proper operation of the system.

The permeation rate can be measured directly by mounting the tube on a microbalance and weighing the tube either continuously or periodically.

4 Reagents and materials

4.1 Permeating substances for calibration, of the highest possible purity so as to avoid any effect of impurities on the permeation rate; if this is not possible, the nature and quantities of the impurities shall be known and allowance made for their effect.

4.2 Carrier gas, of known purity, established by an appropriate analytical technique, for example, gas chromatography (GC) and/or Fourier transform infrared (FTIR) spectrometry.

5 Apparatus

5.1 Permeation apparatus, typically consisting of one of two modes (5.1.1 and 5.1.2) of application of the permeation method.

The materials of the permeation apparatus shall be chosen so as to avoid any effect on the content of the calibration component by sorption (chemical or physical). The smaller the desired final content, the greater the effect of adsorption phenomena. If possible, use glass as the housing of the temperature-controlled permeation tube. Choose flexible and chemically inert tube materials and metals, especially having regard to the transfer of the gas between the permeation apparatus and the analyser. Pay special attention to all junctions so as to keep them free from leaks.

The flow range of the carrier gas is kept constant by a control system and is monitored by a flowmeter. The value of the flow rate can, for example, be controlled by means of a mass flow controller and determined using a mass flowmeter.

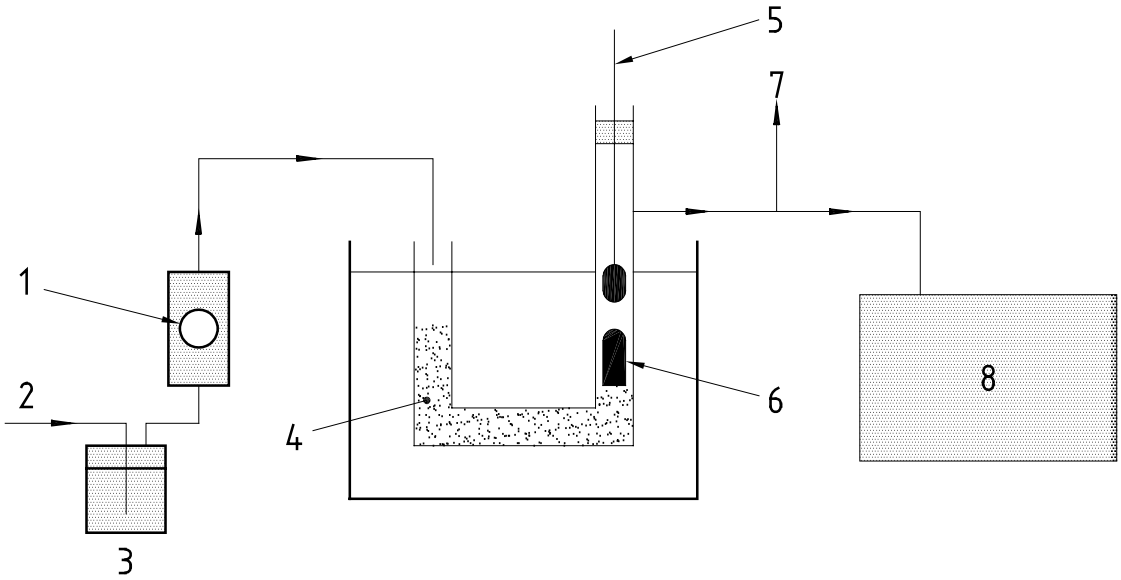
The existence of an outlet for surplus gas enables the analyser under calibration to take the gas flow rate necessary for its proper operation, the remainder of the flow of gas being vented to atmosphere.

5.1.1 Periodic-weighing-mode permeation apparatus, consisting of a permeation tube kept in a temperature-controlled enclosure, swept by carrier gas. The permeation tube is periodically removed from the enclosure to be weighed.

Typical examples are given in Figures 1 and 2.

5.1.2 Continuous-weighing-mode permeation apparatus, consisting of a permeation tube kept in a temperature-controlled enclosure, swept by carrier gas. The permeation tube is suspended from a weighing device and weighed continuously.

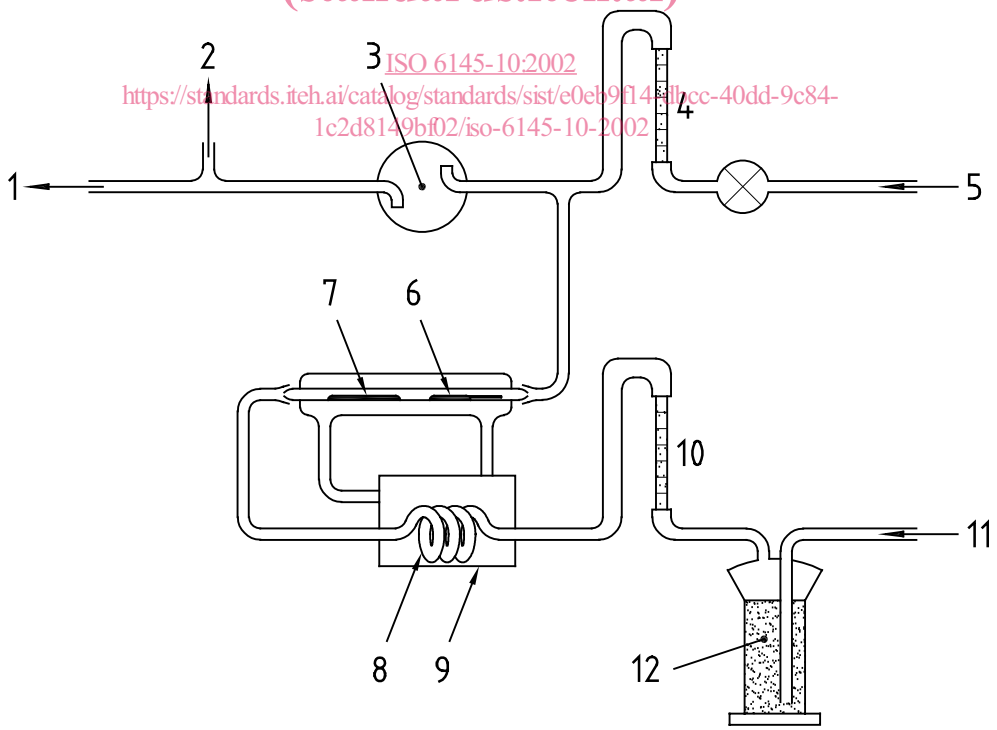
A typical example is given in Figure 3.



Key

1	Flowmeter	5	Thermometer
2	Carrier gas	6	Permeation tube
3	Drier	7	Outlet for surplus gas
4	Filter	8	Analyser

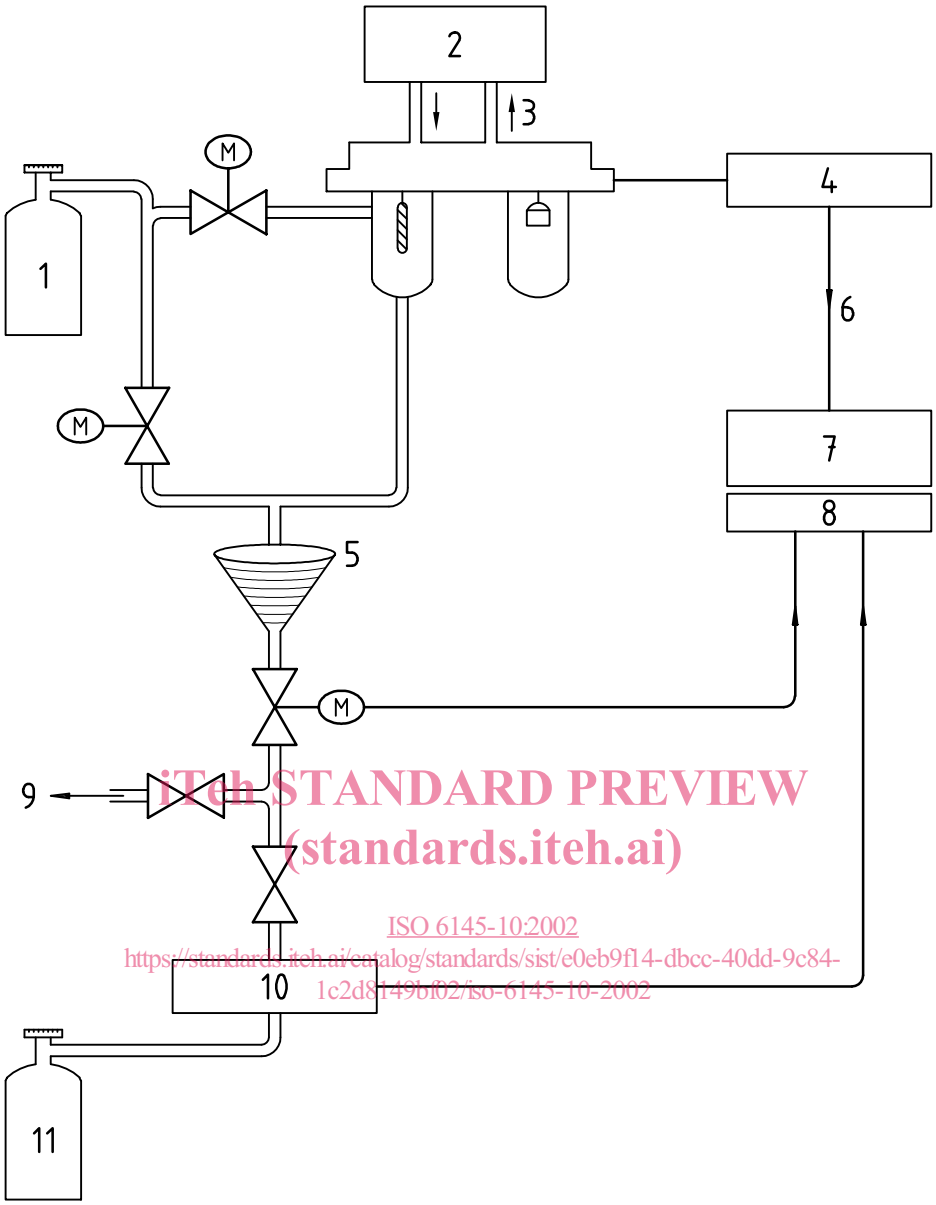
Figure 1 — Example 1 of a periodic-weighing-mode permeation apparatus



Key

1	Outlet for surplus gas	5	Diluent gas	9	Water bath
2	Sampling system	6	Thermometer	10	Flowmeter 1
3	Mixing bulb	7	Permeation tube	11	Carrier gas
4	Flowmeter 2	8	Copper tubing	12	Drier

Figure 2 — Example 2 of a periodic-weighing-mode permeation apparatus



Permeation tube

Mass flow controller

Tare mass

Key

1	High purity air/N_2	5	Gas blender	9	Flow rate calibration facility
2	Temperature controller	6	RS232 link	10	Gas analyser
3	Water	7	PC (acquisition, analysis and diagnostics)	11	Stable mixture requiring certification
4	Microbalance controller	8	16-bit ADC		

Figure 3 — Continuous-weighing-mode permeation apparatus

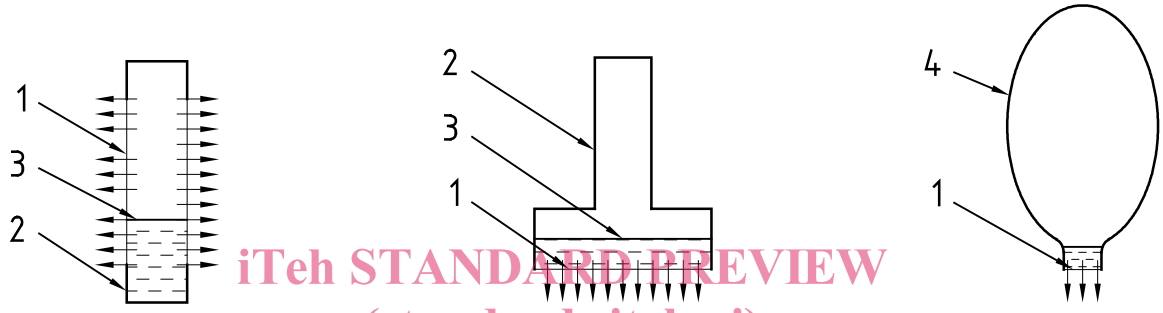
5.2 Permeation membrane, made from polymers and having sufficient chemical and mechanical resistance, e.g. suitable polytetrafluoroethylene (PTFE), polyethylene, polypropylene or a copolymer of tetrafluoroethylene and hexafluoropropylene (FEP).

Take into account variations of the material characteristics which occur with a change of temperature.

5.3 Permeation tubes, or containers, made of stainless steel or glass, fitted with a permeation membrane (5.2) and capable of holding the calibration component in the liquid phase and gaseous phase; the membrane through which the permeation takes place may be in contact with the liquid phase only, or with the gaseous phase only, or with both.

See examples given in Figure 4.

Before use, keep the permeation tube in an airtight container under an anhydrous atmosphere in a cold place (e.g. in a refrigerator at approximately 5 °C) so as to maintain the diffusion rate as low as possible, hence to minimize loss of the calibration component and avoid any condensation on the tube.



a) Cylindrical tube fitted with a membrane in contact with both phases

b) Tube fitted with a membrane in contact with only the liquid phase

c) Container fitted with a membrane in contact with only the gaseous phase

Key

1 Membrane
2 Stainless steel
3 Liquid level
4 Glass

Figure 4 — Examples of permeation tubes and container

6 Procedure

6.1 Preliminary checks and operating conditions

6.1.1 Permeation tube

Before use, assess the purity of the product of the permeation tube by collecting a sample of the permeated gas for analysis by an appropriate analytical technique [e.g. GC or FTIR] so as to quantify any likely major contaminants. This information may be provided by the suppliers of the tube and, if so, a certificate of analysis by an accredited body shall be provided.

Periodically check the permeation rate of the tube at a known, fixed temperature by measuring the mass loss. This gives a good indication as to the purity of the permeated gas. If the permeation rate changes by more than 10 % at the known, fixed temperature, discard the permeation tube.

When first using the permeation tube, allow the system to reach a state of equilibrium before carrying out the first weighing so as to ensure that the permeation rate is well stabilized at the constant value. The time needed to reach