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

Part 9:
Saturation method

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Partie 9: Méthode par saturation

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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 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.

International Standard ISO 6145-9 was prepared by Technical Committee ISO/TC 158, *Analysis of gases*.

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 Part 3 to ISO 6145, entitled *Periodic injections into a flowing stream*, has been withdrawn.

Annexes A and B form a normative part of this part of ISO 6145.

Introduction

Easily condensable gases and vapours are subject to surface adsorption, and it is difficult to prepare calibration gas mixtures containing these components by means of static methods. Moreover, these calibration gas mixtures cannot be kept under pressure near the saturation limit without condensation taking place. The saturation method can be employed in such cases.

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

In contrast to the methods presented in the other parts of ISO 6145, the method described in this part does not call for accurate measurement of flow rates since flow rates do not appear in the equations for calculation of the volume fraction.

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

Part 9:
Saturation method

1 Scope

This part of ISO 6145 specifies a method for the continuous production of calibration gas mixtures with a component which is easily condensable. In most cases the relative expanded uncertainty occurring on the volume fraction is less than 1 %.

2 Normative references

The following normative documents contain 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 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 6143, *Gas analysis — Comparison methods for determining and checking the composition of calibration gas mixtures*.

VDI 3490-3:1980, *Messen von Gasen — Prüfgase — Anforderungen und Maßnahmen für den Transfer* (Measurement of gases — Calibration gas mixtures — Requirements and precautions for the transfer).

3 Principle

The vapour pressure of a pure substance in equilibrium with its condensed phase depends only on temperature. At pressures close to atmospheric, and in the absence of significant gas phase interactions, such as occur with hydrocarbon mixtures, the volume fraction of the constituent can be calculated from knowledge of the temperature and pressure of the system.

If a complementary gas is brought into contact with the condensed phase of a constituent at a certain temperature, the saturation condition is reached more or less slowly. In order to accelerate the process, the complementary gas is passed through the condensed phase at a temperature T_1 and the gas mixture thus obtained is then cooled to a lower temperature T_2 which is below the dew-point. To ensure that saturation is attained, the difference in temperature, $(T_1 - T_2)$, should be at least 5 K.

The volume fraction φ_x of the constituent x is, to a good approximation, equal to the vapour pressure p_x of the constituent at temperature T_2 divided by the total pressure p of the gas mixture at the same temperature in the condenser, as follows:

$$\varphi_x = \frac{p_x}{p} \tag{1}$$

The value of the relevant partial pressure (vapour pressure) of the constituent at temperature T_2 can be found in tables or diagrams in references [1] to [4] of the Bibliography.

4 Apparatus

4.1 Equipment for producing calibration gas mixtures by the saturation method, an example of which is shown in Figure 1.

A continuous flow of complementary gas from the supply (1) is first conducted through a filter (2) containing quartz fibre material to remove suspended particles.
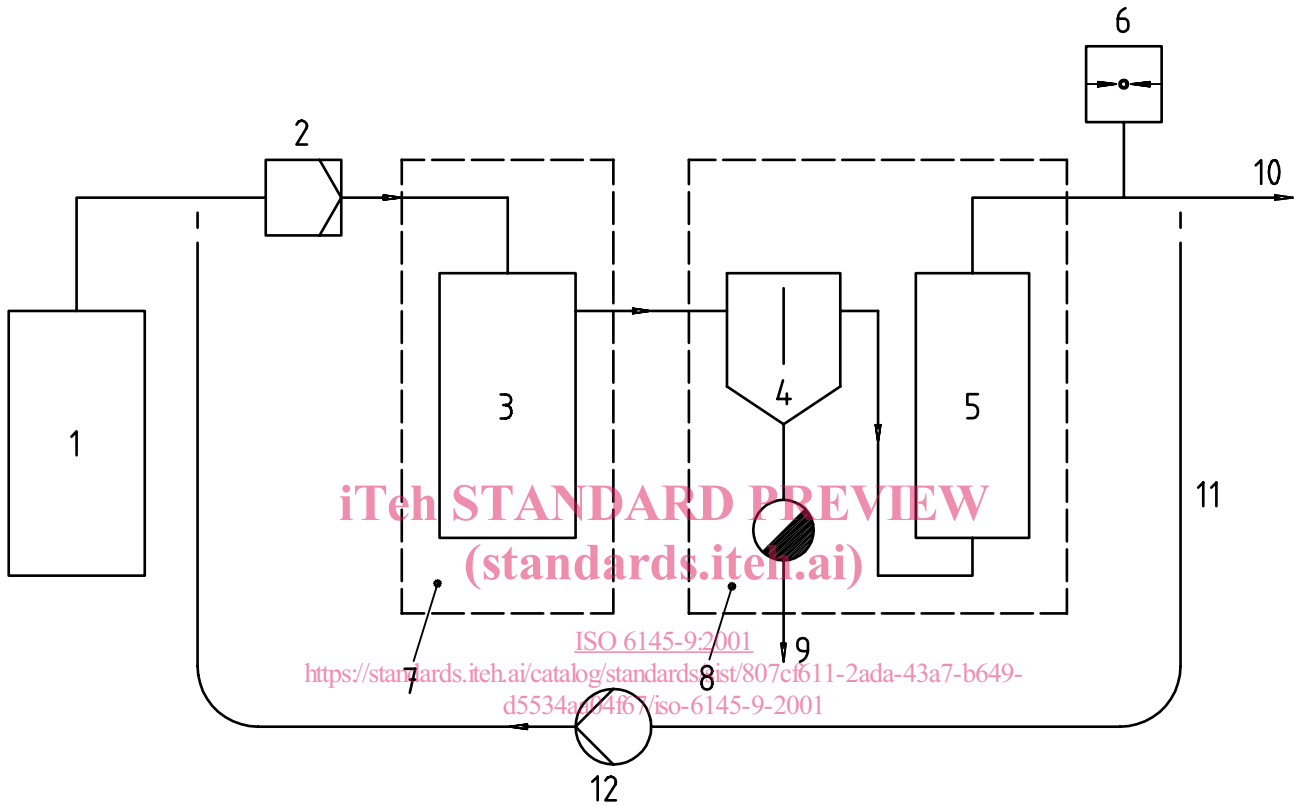
Key

1	Supply of complementary gas
2	Filter for suspended particles
3	Saturator
4	Condenser, made of a material that exhibits adequate thermal conductivity (e.g. copper or stainless steel)
5	Pressure-equalizing vessel with baffles
6	Pressure gauge
7	Constant-temperature control (T_1)
8	Constant-temperature control (T_2)
9	Condensate outlet
10	Calibration gas mixture outlet
11	Circulation system
12	Circulation pump

NOTE   Items 11 and 12 are only required where a recycling system of calibration gas is employed.

Figure 1 — Schema of equipment for producing calibration gas mixtures by the saturation method

The following procedure shall be followed for the assembly and use of the equipment so as to minimize errors in the volume fraction of the constituents.

a) Gas preparation:

Clean and dry the complementary gas before it is introduced into the saturator.

b) Compatibility of the apparatus:

Use only gas-bearing lines and components for the apparatus made from a material that exhibits almost no interaction with the constituents of the calibration gas mixtures.

c) Selection of the apparatus:

Use only connecting lines of which the cross-sections are sufficiently large to ensure that the pressure drop produced by the flow resistance remains negligibly small.

d) Pressure control:

Measure the total pressure at the outlet of the pressure-equalizing vessel.

e) Temperature control:

Comply with the regulations for transfer of calibration gas mixtures specified in VDI 3490-3. Ensure that the temperature of the gas line is sufficiently higher than T_2 to prevent condensation; when appropriate, a heated connecting line shall be provided.

f) Instrumentation:

Use only precision instruments for measurement: thermometers with an error of $|\Delta T| < 0{,}05$ K, pressure-measuring devices with an error of $|\Delta p| < 1$ hPa [1 mbar] [1].

g) Purity:

Use only components of purity $> 99{,}99$ % because certain impurities, when present, affect the vapour pressure.

5 Procedure

5.1 Installation

Arrange the cooling surfaces so as to obtain identical temperatures of the gas and the condenser at the condensate outlet. Place the pressure-equalizing vessel with baffles after the condenser in order to remove aerosols from the gas stream. Maintain the pressure-equalizing vessel at the same temperature as the condenser. Ensure that the temperature of the cooling medium in the vessel, holding the condenser and the pressure-equalizing vessel, remains constant by means of suitable cooling and heating elements via a control circuit.

In addition to the temperature T_2, maintain the pressure, p, of the gas mixture constant in the condenser and display it. Collect the condensate produced in the condenser in a condensate receiver or remove it continuously by pumping.

5.2 Operation of direct system

Load the gas with the constituent to be dosed in the saturator (item 3 in Figure 1) at temperature T_1. Ensure that the condensation temperature of the constituent in the flow of complementary gas is higher than the temperature T_2 of the following condenser (item 4 in Figure 1). Cool the gas mixture in the condenser until part of the constituent condenses. The condensate is discharged via the condensate outlet (item 9 in Figure 1). After the condenser, the calibration gas mixture passes through an pressure-equalizing vessel (item 5 in Figure 1) in which any aerosol that may still be present is separated. Both the condenser (item 4 in Figure 1) and pressure-equalizing vessel (item 5 in Figure 1) are located in a thermostatically controlled container (item 8 in Figure 1) at temperature T_2. A pressure gauge (item 6 in Figure 1) at the outlet (item 10 in Figure 1) for the calibration gas mixture provides a reading of the pressure.

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1) 1 bar = $10^5 \text{N}/\text{m}^2 = 0{,}1 \text{MPa}$

5.3 Operation of a closed circulation system

A closed circulation system can also be used. This system operates continuously and when in use will eliminate any lengthy delays in the procedures required to attain equilibrium conditions. The calibration gas is circulated around an additional circuit (item 11 in Figure 1) by means of a pump (item 12 in Figure 1). After several passages of gas around the circuit to establish equilibrium, sample gas can be extracted at the sample point (item 10 in Figure 1). Gas taken out of the system is to be carefully made up by fresh complementary gas from the supply (item 1 in Figure 1), thus avoiding any pressure changes.

NOTE It is possible to check whether saturation is achieved in the condenser by observing the condensate flow. Since the volume fraction of the gaseous to condensed phases is about 1 000 : 1 and only a fraction of the components picked up by the complementary gas is separated out in the condenser as condensate, the volume flow rate through the outlet (item 9 in Figure 1) is very low. A physical dew point measurement on the outlet gas can be carried out to confirm equilibration has been achieved.

6 Uncertainty of measurement

The relative uncertainty depends on the total pressure of the gas in the condenser and on the saturated vapour pressure. Whereas the total pressure is known with satisfactory uncertainty, the uncertainty of the vapour pressure value depends on

— the uncertainty of condensate temperature measurement;

— the temperature control (i.e. $\Delta T < 0{,}05$ K can be measured);

— the uncertainty of the vapour pressure data used;

— the purity of the components.

The relative expanded uncertainties in the volume fraction of constituent x shall be estimated with the aid of the determinable individual uncertainties using equation (2).

The relative standard uncertainties of measurement are combined in a square root sum-of-squares manner to form the overall relative expanded uncertainty as follows:

$$\frac{U\left(\varphi_x\right)}{\varphi_x} = 2\sqrt{\left\{\left[\frac{u\left(p_x\right)}{p_x}\right]_{T_2}\right\}^2 + \left[\frac{u\left(p\right)}{p}\right]^2 + \left\{\left[\frac{T_2}{p_x}\left(\frac{\mathrm{d}p_x}{\mathrm{d}T}\right)_{T_2} - 1\right] \times \left[\frac{u\left(T\right)}{T}\right]_{T_2}\right\}^2} \tag{2}$$

where

$\dfrac{U\left(\varphi_x\right)}{\varphi_x}$ is the relative expanded uncertainty in the volume fraction of the constituent;

$\left[\dfrac{u\left(p_x\right)}{p_x}\right]_{T_2}$ is the relative standard uncertainty in the vapour pressure curve at working point T_2;

$\dfrac{u\left(p\right)}{p}$ is the relative standard uncertainty in the measurement of the total pressure;

$\left(\dfrac{\mathrm{d}p_x}{\mathrm{d}T}\right)_{T_2}$ is the increase in the vapour pressure curve at working point T_2;

$\left[\dfrac{u\left(T\right)}{T}\right]_{T_2}$ is the relative standard uncertainty in the temperature measurement of T_2.

NOTE 1 To provide a confidence level (relative expanded uncertainties) of approximately 95 %, the relative standard uncertainties given by equation (2) have been multiplied by a coverage factor of 2.

There exists the possibility of over-saturation as a stable phase through solvation capability in the matrix gas.

The component concentration may be checked by comparison with a reference gas mixture. This may be difficult at low concentrations. In this case, it may be possible to use an instrument that has been assessed for traceability.

NOTE 2   Examples of calculations are given in annex A.

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