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Third edition
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Gas analysis — Preparation of calibration gas mixtures using dynamic methods —

Part 7: **Thermal mass-flow controllers**

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

Partie 7: Régulateurs thermiques de débit massique

Reference number
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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).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 158, *Analysis of gases*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

This third edition cancels and replaces the second edition (ISO 6145-7:2009), which has been technically revised. The main changes compared to the previous edition are as follows:

— correction of some errors in the formulae in Annexes A and C;

— minor editorial corrections.

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

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

Part 7: Thermal mass-flow controllers

1 Scope

ISO 6145 is a series of documents dealing with various dynamic methods used for the preparation of calibration gas mixtures. This document specifies a method for continuous preparation of calibration gas mixtures, from nominally pure gases or gas mixtures by use of thermal mass-flow controllers. The method is applicable to preparation of mixtures of non-reacting species, i.e. those which do not react with any material of construction of the flow path in the thermal mass-flow controller or the ancillary equipment.

If this method is employed for preparation of calibration gas mixtures the optimum performance is as follows: the relative expanded measurement uncertainty U, obtained by multiplying the standard uncertainty by a coverage factor $k = 2$, is not greater than 2 %.

If pre-mixed gases are used instead of pure gases, mole fractions below 10^{-6} can be obtained. The measurement of mass flow is not absolute and the flow controller requires independent calibration.

The merits of the method are that a large quantity of the calibration gas mixture can be prepared on a continuous basis and that multi-component mixtures can be prepared as readily as binary mixtures if the appropriate number of thermal mass-flow controllers is utilized.

NOTE Gas blending systems, based upon thermal mass-flow controllers, and some including the facility of computerization and automatic control, are commercially available.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 6145-1, *Gas analysis — Preparation of calibration gas mixtures using dynamic volumetric methods — Part 1: Methods of calibration*

ISO 7504, *Gas analysis — Vocabulary*

ISO 12963, *Gas analysis — Comparison methods for the determination of the composition of gas mixtures based on one- and two-point calibration*

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 7504 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at: http://www.electropedia.org/

4 Symbols

C_p	Heat capacity (at constant pressure)
i,k	Indices for components in a gas or gas mixture
j	Index for a parent gas
m	Mass
p	Pressure
q	Number of components in the gas mixture
q_m	Mass flow rate
q_V	Volume flow rate
T	Temperature
V	Volume
Φ	Heat flux
ϕ	Volume fraction of a component in a parent gas
φ	Volume fraction of a component in a gas mixture
ρ	Density

5 Principle

The continuous preparation of calibration gas mixtures from nominally pure gases or other gas mixtures by the use of commercially available thermal mass-flow controllers is described. By adjustment of the set-points on the mass flow controllers to pre-determined values, it is possible to change the composition of the gas mixture rapidly and in a continuously variable manner. By selection of appropriate combinations of thermal mass-flow controllers and with use of pure gases, the volume fraction of the component of interest in the matrix gas can be varied by a factor of 1 000.

6 Set-up

6.1 General

To prepare a gas mixture, each gaseous component is passed through a calibrated thermal mass flow controller (TMC) at a known and controlled flow rate and at constant pressure. Accurate flow meters are used to measure the relevant flow rates in order to achieve an acceptable level of uncertainty regardless of the setting of the mass flow controller (see also ISO 6145-1).

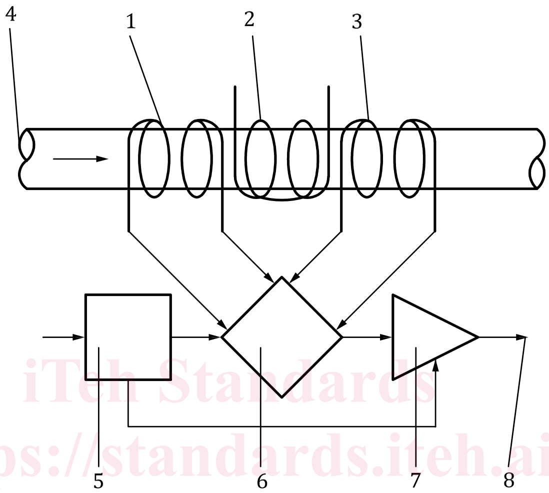
A TMC consists of a measuring unit for mass flow and a proportioning valve which is controlled by an electronic unit (see also Reference [1] and [2]).

6.2 Thermal mass-flow controller using a constant current supply

The flowing gas is passed through a heater connected to a constant current supply and the temperature is sensed upstream and downstream from the heater.

Figure 1 shows the working principle of a TMC and its key parts: heater, temperature sensors and associated circuitry. The two temperature sensors, one upstream and one downstream from the heater form two arms of a Wheatstone bridge circuit, which is balanced to give zero reading when there is no gas flow. When there is a gas flow through the system a temperature difference, ΔT, is established between the two sensors such that the heat flux, Φ, is given by Formula (1):

$$\Phi = C_p \Delta T q_m \quad (1)$$



Key

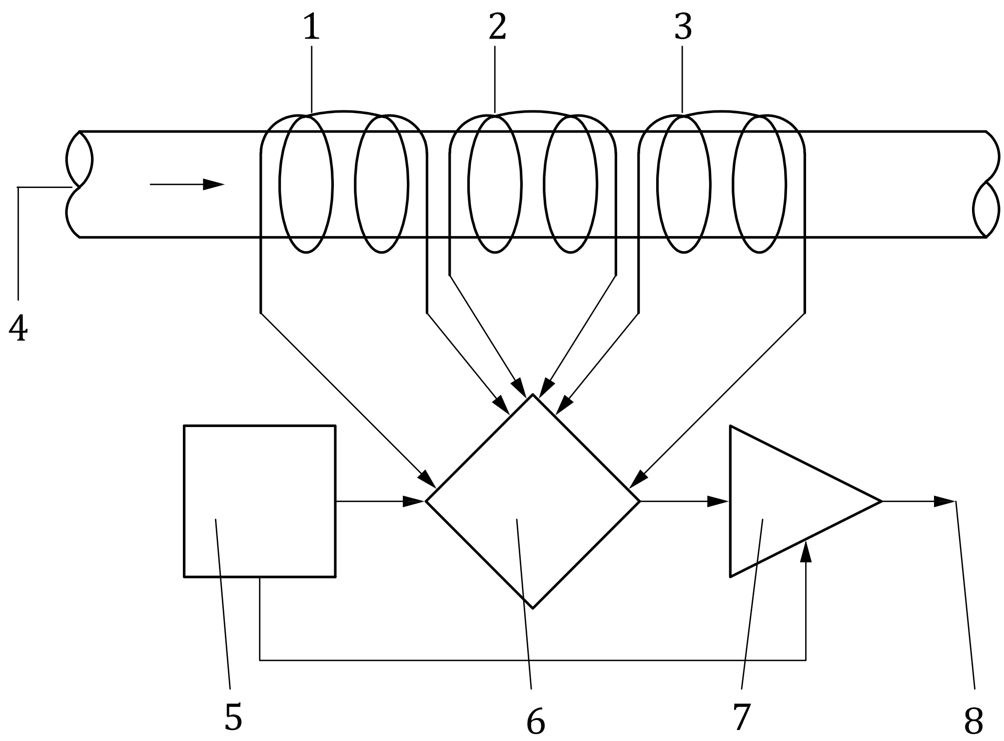
1 temperature sensor 1
2 heater
3 temperature sensor 2
4 gas supply
5 current supply
6 wheatstone bridge
7 differential amplifier
8 signal readout

Figure 1 — Principle of a thermal mass-flow controller with constant current supply

The difference in temperature between sensors results in a potential difference across the Wheatstone bridge circuit and thus a signal. The signal is compared with an adjustable reference voltage in a differential amplifier. The resulting output signal is in turn used for operating a control valve to regulate the flow of gas.

6.3 Thermal mass-flow controller under constant temperature control

In the system, shown in Figure 2, the parent gas passes through three heaters in sequence, each of which is connected into an arm of a self-regulating Wheatstone bridge. Instead of the difference in temperature being measured, the input to each heater is such that the temperature distribution along the flow path is uniformly maintained. The Wheatstone bridge current is proportional to the heat loss and therefore proportional also to the mass flow of the gas. The output signal is again used to operate a solenoid valve to control the mass flow rate.



Key

1 heater 1
2 heater 2
3 heater 3
4 gas supply
5 current supply
6 wheatstone bridge
7 differential amplifier
8 signal readout

Figure 2 — Thermal mass-flow controller under constant temperature control

In the preparation of multicomponent mixtures, it is generally necessary to use one mass-flow controller for each component. Dual-channel controllers are available and may be used in the preparation of binary mixtures or, for example, preparation of mixtures of a given gas in air.

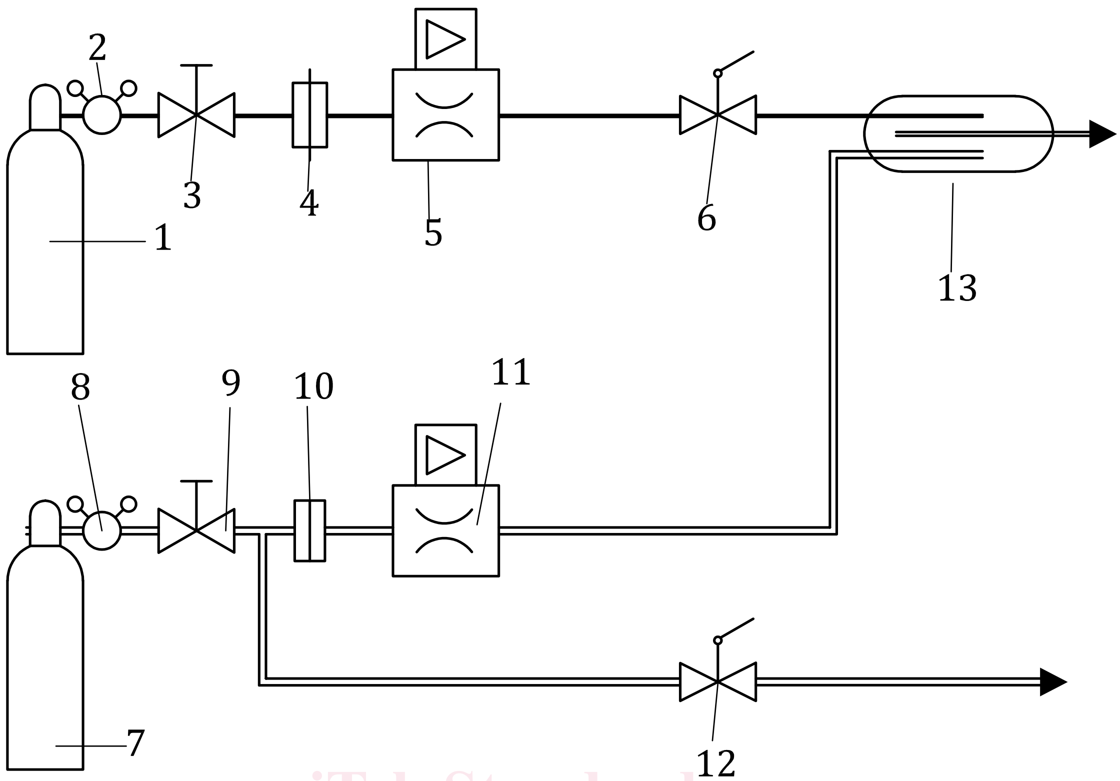
7 Preparation of gas mixtures

7.1 Description of the experimental procedure

A schematic diagram of the arrangement for preparation of binary mixtures is shown in Figure 3.

The pressure and temperature at the time of the calibration shall be recorded. Depending on the gases to be mixed and their departure from ideality, the volume fraction can be somewhat influenced by the ambient pressure and temperature. The pressure and temperature at the time of calibration of the analyser should be as near as possible to those prevalent at the time the TMCs were checked by the comparison method ISO 6143 or ISO 12963 (see 7.3).

Compositions of calibration gas mixtures are normally expressed by volume fractions but manufacturers' accuracy specifications for thermal mass-flow controllers are usually expressed in terms of percentage of the full scale of the instrument. The relative expanded uncertainty of 2 %, which is quoted in the Scope of this document, is 2 % of the volume fraction of the calibration component of the mixture. This value assumes optimum use of each TMC in the system, which means that each is operated at, or very near to, its maximum flow rate. Thus, if a TMC is operated at 10 % of full scale, the expanded uncertainty expressed as percentage of maximum flow (as distinct from relative expanded uncertainty) can be ±1 %, but if expressed instead as a percentage of the actual flow rate the relative expanded uncertainty becomes 10 %.



Key

Matrix gas:

1 cylinder of pressurized gas
2 pressure regulator
3 shut-off valve
4 filter against contamination
5 thermal mass-flow controller
6 shut-off valve

Calibration component:

7 cylinder of pressurized gas
8 pressure regulator
9 shut-off valve
10 filter against contamination
11 thermal mass-flow controller
12 shut-off valve
13 mixing vessel

Figure 3 — Mixing apparatus for preparation of binary gas mixtures by means of thermal mass-flow controllers

A binary mixture containing the calibration component at volume fraction 1:11 could be prepared by use of two TMCs each of full scale 1 000 mL/min by operating one at 100 mL/min and the other at 1 000 mL/min. However, the expanded uncertainty associated with the flow rate of the former would be ±10 % of flow rate and the relative expanded uncertainty in the volume fraction would be ±9 %. Use one TMC with a full scale range of 100 ml/min and a second one with a full scale range of 1 000 ml/min, both being operated very close to full scale, so that the mixture has a volume fraction with a relative expanded uncertainty of 2 %.

The same requirement shall be observed relative to preparation of multi component mixtures.

A method for which there is no requirement for calibration against external standards of gas flow rate or volume fraction is described briefly in Annex B, and the reference to the publication which provides the complete description is given in the bibliography.

As shown in Figure 3, gas cylinders (1) and (7)containing the matrix gas and the component of interest respectively are connected to the thermal mass-flow controllers (5) and (11) through pressure regulators (2) and (8) and shut-off valves (3) and (9). The two in-line filters (4) and (10) provide protection against contamination. The gases from the flow controllers enter the mixing vessel (13).

The recommended working range for the pressure regulators is 60 kPa (0,6 bar) to 600 kPa (6,0 bar). The pressure regulator for the "gaseous component" shall also be suitable for the particular component involved (e.g. the diaphragm shall be of stainless steel or other corrosion resistant material). Similarly, the thermal mass-flow controllers shall be suitable for use with the gaseous components and for the requirements of the gas mixture.

Set the input pressures appropriate to the controllers using the pressure regulators and open the shut-off valves (3), (6) and (9). Purge the inlet path of the gaseous component through the shut-off valve (12), which shall be of a type which can be operated rapidly.

Adjust the set points of the controllers so as to obtain the respective flow rates in the correct ratio for the desired composition of the binary gas mixture; meanwhile, continue the purging process of the input tube for the component gas by multiple opening and closing of valve (12), until a total volume of gas at least 10 times the volume of the flow path has been vented.

When the system has been thoroughly purged, feed the gases via the thermal mass-flow controllers to the mixing vessel (13), constructed from inert materials. Provided that the resistance to flow downstream of the mixing vessel (13) is low in relation to the flow being delivered at the source, the mixture flows at ambient atmospheric pressure to the instrument.

Although for most applications the gas mixture will be transmitted at the prevailing ambient atmospheric pressure, this method may also conceivably be applied to convey mixtures at elevated exit pressures. However, in this case it would be necessary to give due consideration to changes in C_p and density of the gaseous components with pressure in order to assess the validity of this procedure.

7.2 Range of validity

As stated in the scope, this method is applicable to preparation of mixtures of non-reacting species, i.e. those which do not react with any material of construction of the flow path in the thermal mass-flow controller or the ancillary equipment. Particular care shall be exercised if the method is considered as a means of preparation of gaseous mixtures which contain components which form potentially explosive mixtures in air. Steps shall be taken to ensure that the apparatus is safe for example by means of in-line flame arrestors in addition to the items listed in 6.1.

This method is not absolute and each thermal mass flow controller shall be calibrated for the particular gas or gas mixture for which it is to be used.

7.3 Operating conditions

The conditions for efficient operation of the sensor system are that

— there shall be no heat loss or heat gain, other than that which results from the flow of gas, between the region of the heater and that of the downstream sensor, and that

— there shall be uniform temperature distribution across the gas stream.

The assumption that C_p is constant is valid only over a restricted range of temperature. The general precautions common to all dynamic techniques of preparation shall be observed. It is essential that attention is paid to the materials used in the construction of the flow system. Only materials of low porosity that do not cause adsorption of any of the components in the gases or gas mixture are suitable. The tubing shall be clean and all unions secure.

Unless independence of the thermal mass-flow controller to its orientation has been established, it shall be maintained in the orientation in which it was calibrated. Controllers shall be calibrated for the components in question and it may be necessary to consult the manufacturer of the controller if the type of gas is to be changed; it may be necessary for the sensor to be changed.

