H-25-23

INTERNATIONAL STANDARD ISO 6147

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Gas analysis — Preparation of calibration gas mixtures — Saturation method

Analyse des gaz — Préparation des mélanges de gaz pour étalonnage — Méthode par saturation

First edition — 1979-02-15

ISO 6147-1979 (E)

UDC 543.27 : 53.089.68

Ref. No. ISO 6147-1979 (E)

Descriptors : gases, gas mixtures, gas analysis, calibrating, reference samples.

Price based on 2 pages

FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 6147 was developed by Technical Committee ISO/TC 158, *Analysis of gases*, and was circulated to the member bodies in November 1977.

It has been approved by the member bodies of the following countries :

Australia
Belgium
Czechoslovakia
Egypt, Arab Rep. of
France
Germany, F. R.
India
Ireland
Mexico
Netherlands
Poland
Romania
South Africa, Rep. of
Spain
Turkey
United Kingdom
U.S.A.
U.S.S.R.
Yugoslavia

No member body expressed disapproval of the document.

Printed in Switzerland

Gas analysis — Preparation of calibration gas mixtures — Saturation method

1 SCOPE AND FIELD OF APPLICATION

This International Standard describes the principles of a saturation method for the continuous preparation of calibration gas mixtures, one of the components of which is readily condensable. An accuracy of 3 % relative value may be obtained on the concentration, expressed as a pressure ratio[1].

Readily condensable gases often exhibit very marked sorption phenomena; it is, therefore, difficult to prepare mixtures comprising such gases by static methods. Moreover, the use of high pressures precludes the achievement of concentrations close to the saturation limit during the use of the mixture at atmospheric pressure. In these cases, recourse should be made to the method described in this International Standard.

2 PRINCIPLE

The vapour pressure of a pure gas in equilibrium with its condensed phase depends only on the temperature. If the temperature and the total pressure of the gas mixture are known, its concentration may be deduced therefrom.

In practice, when a make-up gas is brought into contact with the condensed phase of a component at a given temperature, saturation is attained only very slowly. To accelerate this process, the make-up gas is bubbled through the condensed phase at a temperature T_1 and the resulting mixture is cooled to temperature T_2 in a condenser. To be certain of obtaining saturation, the temperature difference $T_1 - T_2$ should be sufficiently large.

The cooling system shall ensure the steady removal of heat (any on/off control system shall be rejected since it introduces temperature gradients). The coefficient of exchange between the condenser and the gases must be large : a good means for obtaining this effect is to use a packing which is a good heat conductor (balls, sintered materials).

The temperature regulation may be obtained by an assembly either filled with a cooling liquid or comprising Peltier-effect elements energized by an adequate direct current source. This assembly may alternatively, be replaced by a vessel filled with a cryogenic liquid.

The pressure p of the gas mixture in the condenser shall be maintained constant at temperature T_2 (see clause 4) and shall be recorded. The condensate formed in the condenser shall be evacuated continuously by a pump.

The component concentration C is given by the equation

$$C = \frac{p_c}{p}$$

where

p_c is the saturated vapour pressure of the component at temperature T_2;

p is the total pressure of the gas mixture in the condenser.

The saturated vapour pressure value, p_c, shall be determined, for each component, from suitable reference sources of such data (published tables or diagrams). When the method described in this International Standard is to be used to prepare a calibration gas mixture required in another International Standard, the latter shall indicate the values of p_c required for the component concerned.

---

1) The transformation of the value of concentration from one set of units to another becomes more difficult as one gaseous component nears its saturation point.

3 PRACTICAL EXAMPLE

The make-up gas (1) is taken from a cylinder and brought through the pressure regulator (2) to a constant absolute pressure, for example 1 200 mbar*. It then goes through a flowmeter (3) and a fine regulation valve (4) before penetrating upwards into the saturator (5) (containing the component which has been condensed) through a sintered plug (7) or a hollow cylinder of sintered material. To avoid pressure variations downstream of the saturator, it is recommended that the condensed component level be kept constant (additional filling system).

It should be ascertained that no liquid droplets (mist) are transported from the cooled region onwards. This control should be made by means of a suitable filter also cooled to T_2.

The condenser gas piping includes in its lower part a separator in which condensate is evacuated (18) continuously by means of a pump (17).

In the example shown in the figure, the condensate is discharged through the descending branch of the Y-shaped tube. The pump discharge rate shall be chosen to be much lower than the total gas flow rate because the volume ratio between the gas phase and the condensed phase is approximately 1 000 : 1 and only a part of the component absorbed by the make-up gas is evacuated as condensate.

It can be verified that sufficient saturation is obtained by making the condensate flow visible.

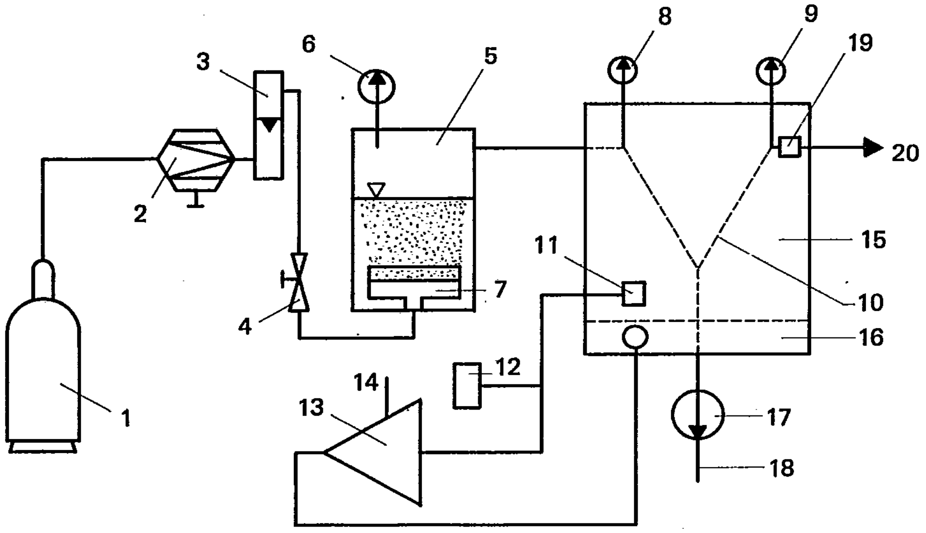
4 CALCULATION OF THE ERROR

The relative accuracy depends on the one hand on the total pressure of the gas in the condenser and on the other hand on the saturated vapour pressure. Whereas the total pressure is known with satisfactory accuracy, the accuracy of the vapour pressure value depends on :

- the accuracy of condensate temperature measurement;
- the temperature control ($\Delta T_2 < 0{,}2$ °C can be obtained);
- the accuracy of the tables or diagrams used;
- the purity of the components and of the make-up gas (water vapour content, solvent).

An estimate of the accuracy can only be made for each individual case by taking account of the actual situation; for example, the solubility of the carrier gas in the condensed phase at T_2 may interfere in certain cases.

A verification of the correct functioning of the system may be necessary, for example by determination of the mixture dew point.



1	Make-up gas	11	Temperature probe
2	Absolute pressure regulator	12	T_2 control
3	Flowmeter	13	Regulator with switch
4	Fine regulation valve	14	Power supply
5	Saturator	15	Controlled-temperature bath
6	Thermometer (T_1)	16	Peltier elements
7	Sintered material	17	Pump
8	Pressure gauge	18	Condensate evacuation
9	Thermometer (T_2)	19	Filter
10	Condenser	20	Calibration gas

Schematic diagram of a practical example

---

* 1 mbar = 100 Pa