INTERNATIONAL STANDARD

ISO 6142

Second edition
2001-04-01

AMENDMENT 1
2009-05-15

Gas analysis — Preparation of calibration gas mixtures — Gravimetric method

AMENDMENT 1: Liquid introduction

Analyse des gaz — Préparation des mélanges de gaz pour étalonnage — Méthode gravimétrique

AMENDEMENT 1: Introduction de liquides

Reference number
ISO 6142:2001/Amd.1:2009(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

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 2.

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

Amendment 1 to ISO 6142:2001 was prepared by Technical Committee ISO/TC 158, *Analysis of gases*.

Gas analysis — Preparation of calibration gas mixtures — Gravimetric method

AMENDMENT 1: Liquid introduction

Page iii, Contents

Add “**Annex H** (normative) **Liquid introduction**”.

Page iv, Foreword

Replace the next to last paragraph with the following and delete the last paragraph.

> This second edition of ISO 6142 cancels and replaces the first edition (ISO 6142:1981), which has been revised to update the methods of preparation, estimation of the uncertainty and of validation of gravimetrically prepared calibration gases. An annex on liquid introduction has been added.

Page 1, Normative references

Replace the reference to ISO 6143:— with the following:

> ISO 6143:2001, *Gas analysis — Comparison methods for determining and checking the composition of calibration gas mixtures*

Delete the reference to Footnote 1) and the footnote “To be published. (Revision of ISO 6143:1981)”.

Page 6, 4.5

Add the following paragraph at the end of the subclause.

> “In cases where liquid components are introduced into gravimetrically prepared calibration gas mixtures, follow the guidance for preparation methods in Annex H. Annex H is only applicable to mixtures whose final composition is totally vaporized and which contain components that do not react with each other or with the cylinder wall.”

Page 10, 6.2

Replace the reference to “ISO 6143:—” with “ISO 6143:2001”.

Page 10, 6.3

In list items a) and b), replace the references to “ISO 6143:—” with “ISO 6143:2001”.

Page 15, A.5.2.3

Renumber the reference to Footnote 2) and the footnote itself to Footnote 1).

Page 15, A.5.3

Renumber the reference to Footnote 3) and the footnote itself to Footnote 2).

Page 36, Bibliography

Replace Reference [16] by the following, deleting Footnote 4).

[16] ISO/TS 14167, *Gas analysis — General quality assurance aspects in the use of calibration gas mixtures — Guidelines*

Page 37, Bibliography

Add the following reference.

[20] ISO 16664, *Gas analysis — Handling of calibration gases and gas mixtures — Guidelines*

Page 36

Add the following annex before the Bibliography.

Annex H
(normative)

Liquid introduction

H.1 Principle

The introduction of a liquid component into a gas mixture requires dedicated introduction methods and equipment. As the gas mixtures are prepared gravimetrically and as the amount of liquid is normally small due to the vapour pressure, a highly sensitive, low capacity balance is required. The liquid is either introduced into an evacuated cylinder, where it vaporizes, or is introduced as a volume of vaporized liquid.

In some cases, a liquid component may be introduced directly into the cylinder as long as the cylinder undergoes a final mixing stage to ensure complete homogenization.

Several methods of liquid introduction are described that result in a good calibration gas preparation. For a better understanding, some are described in more detail. Other methods may exist with equal or even better performance.

H.2 Methods

H.2.1 General guidance

It is important that the liquid fully vaporize in the gas mixture and that it remain in the gaseous phase. Normally, the vapour pressure of a component at specified conditions is used to calculate the maximum amount of liquid to be introduced.

IMPORTANT — To prevent condensation, the fraction of the vapour pressure shall be kept low enough in relation to the dewpoint. This limits the maximum amount of substance fraction to be produced at a certain pressure.

NOTE A maximum of 70 % is usually found to be sufficient. This is a safety measure for transport conditions, which may differ from production conditions. See also 4.2.2. For guidance on transport, see ISO 16664 [20].

H.2.2 Syringe method

A gas-tight syringe is filled with the liquid to be introduced. A syringe with a graduated scale is useful for estimating the amount of liquid in the syringe. It is best to first weigh the syringe after filling it, then to weigh it again after injection. The difference between these two weighings corresponds to the amount of liquid introduced.

The liquid is introduced into the vacuum cylinder by injection through a septum that is closed off during pressurization with the matrix gas. An example of this setup is shown in Figure H.1.

Figure H.1 — Example of liquid introduction via a syringe

When using this method, it is important to eliminate the loss of component in the syringe and especially in the needle. It is therefore recommended to replace the needle after filling and before weighing. Especially with very volatile components, the remaining liquid droplets may vaporize during weighing.

H.2.3 Glass tube method

A glass tube with one open end is weighed, then filled with liquid. After filling, the tube is sealed by melting the open end of the glass tube. The sealed tube is weighed again. The difference between the two weighings corresponds to the amount of liquid introduced. The glass tube is then packed into the filling line, or even in the gas cylinder, and broken by the high-pressure matrix gas.

It is important to prevent the broken glass from entering the cylinder valve during the filling process and later use.

The sealing of the glass tube will cause some vaporization of the liquid introduced into the tube.

This effect should be evaluated for the different liquids as the effect increases with more volatile components. To prevent this, cool the tube before sealing.

H.2.4 Vapour in a receptacle

This method uses a certain quantity of vapour in a closed receptacle. The receptacle may have various shapes, but spheres are mostly used.

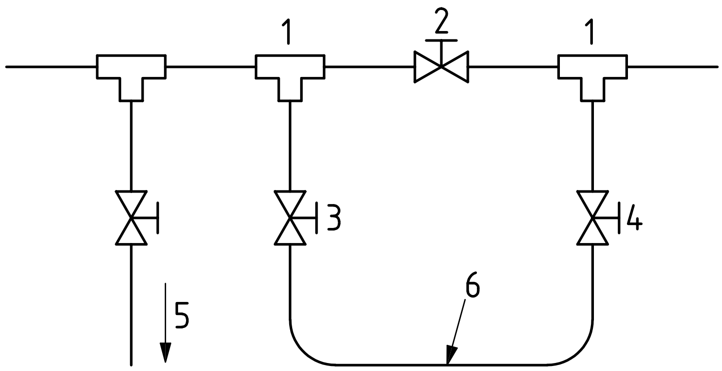
The evacuated receptacle is connected to a flask of liquid, and the vapour allowed to flow into the receptacle until the vapour pressures in the flask and in the receptacle are equal.

It is important that the temperature of the receptacle and the transfer lines be higher than the temperature of the flask of liquid to avoid condensation.

After the system is equilibrated, the receptacle is disconnected and accurately weighed. After weighing, it is connected to the final evacuated cylinder. After equilibration, the receptacle is weighed again to determine the amount of vapour transferred into the cylinder.

H.2.5 U-tube method

A U-tube is typically constructed from stainless steel (see Figure H.2). The length of the U-tube is designed so that the volume is nominally the same as the volume of liquid required. Different volume tubes can easily be made by altering the U-tube length.



Key

1 T-type connector

2, 3, 4 shut off valves

5 vacuum

6 U-tube line

Figure H.2 — U-tube construction

The U-tube is dismantled so that the U part retains the two shut off valves (3 and 4). The U-tube with open valves is then weighed on a high accuracy balance. Both valves are opened and liquid is then introduced into the U-tube and the valves are then closed. The U-tube plus the liquid is again weighed. The difference between the weighing readings corresponds to the mass of liquid. The U-tube is then reconnected to the filling assembly. One end of the filling assembly is connected to the cylinder to be filled and the other to a gaseous component which is to be introduced. The system excluding the U-tube is then evacuated (valve 2 open, valves 3 and 4 closed).

The gas to be introduced is then allowed to fill the filling assembly. Valve 2 is then closed and valves 3 and 4 are then opened. The filling gas pushes the liquid into the recipient cylinder. Before the final mass of gas has been achieved in the recipient cylinder, valves 3 and 4 should be closed and valve 2 opened. This is to remove any liquid trapped behind valve 2. Once the required mass of gas has been transferred, the recipient cylinder is isolated.

The U-tube method has the advantage that liquids can be introduced into the recipient cylinder at any stage of the cylinder filling process. Unfortunately, the U-tube method can introduce some air contaminants into the recipient cylinder if the correct tube length is not used. Air above the liquid in the U-tube should be kept to a minimum.

H.2.6 Minicylinder method

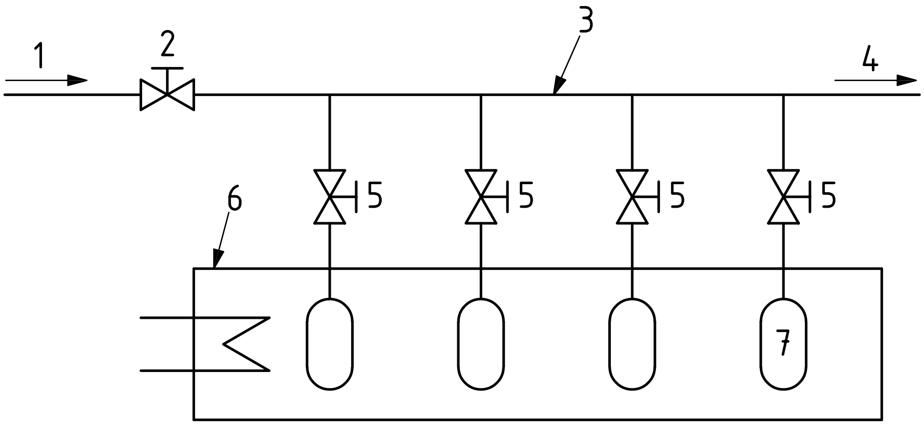
H.2.6.1 General

This method uses a set of minicylinders filled with liquid to be transferred into the calibration gas cylinder.

These minicylinders can be configured in parallel or in series.

H.2.6.2 Parallel configurations

An example for a typical configuration is shown in Figure H.3.



Key

1 inlet
2 inlet valve
3 transfer line
4 outlet
5 shut off valves
6 heating system
7 minicylinder

Figure H.3 — Minicylinders in parallel

First, the calculated mass of each component is introduced into each of the evacuated minicylinders (7).

These components are introduced one after the other into the calibration gas cylinder, starting with the component of lowest vapour pressure.

After evacuation of the complete filling system, inlet valve 2 is closed and the valve of the evacuated calibration gas cylinder opened.

The shut off valve (5) of the first minicylinder then is opened to balance the pressure in the filling system (transfer line, minicylinder and calibration gas cylinder).

This step is repeated for each minicylinder.

The remaining components in the transfer system are purged with the matrix gas (pure gas or pre-mixture) or an additional component (pure gas or pre-mixture) through the outlet (4) into the calibration gas cylinder.

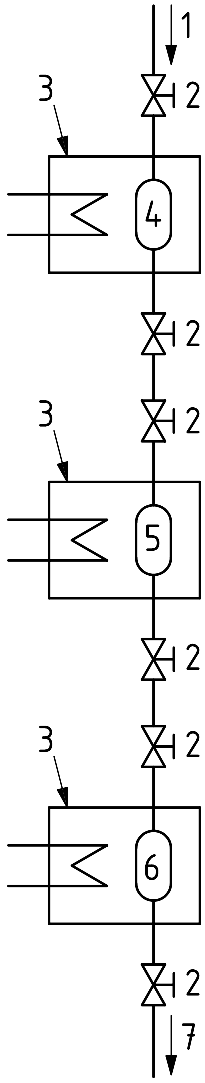
The exact mass of each component is determined by the weighing difference between the minicylinders before and after the filling process (in the calibration gas cylinder).

For components with vapour pressures lower than the vapour pressure of *n*-butane, the minicylinders should be heated during the filling process.

H.2.6.3 Serial configuration

A similar approach is used with three minicylinders combined in line (see Figure H.4).

Again, first the calculated mass of each component is introduced into the evacuated minicylinders.



Key

1 inlet transfer line
2 two-way valves
3 heating system
4, 5, 6 minicylinders
7 outlet transfer line

Figure H.4 — Minicylinders in series

These components are introduced one after the other through the outlet (7) into the calibration gas cylinder, starting with the component in minicylinder 6.

Starting with the outlet valve of minicylinder 6, one valve after the other, excluding the inlet valve of minicylinder 4, is opened to balance the pressure in the system.

Thereafter, the inlet valve of minicylinder 4 is opened to purge the remaining components with matrix gas (pure gas or pre-mixture) or an additional component (pure gas or pre-mixture) through the outlet (7) into the calibration gas cylinder.

The exact mass of each component is determined by the weighing difference between the evacuated and the filled minicylinders.

NOTE The mass of the remaining air in the connections between the minicylinders and in the transfer line from minicylinder 6 to the calibration gas cylinder can be calculated from these volumes.

The uncertainty contributions should be well estimated.

In cases where 3-way valves are installed for the evacuation of the connections between the minicylinders and in front of minicylinder 6, no remaining air need be considered.

H.3 Validation

In addition to the uncertainty sources listed in this International Standard, liquid introduction methods may have supplementary sources of uncertainty. These shall be included in the total uncertainty budget; however, it is best to prevent them by following the advice given in this annex.

In the case of liquid introduction, validation is essential as these liquids tend to stick to the wall of the cylinder.

ICS 71.040.40

Price based on 8 pages