International Standard

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Gas analysis — Preparation of calibration gas mixtures — Dynamic volumetric methods — Part 1 : Methods of calibration

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Descriptors : gas analysis, calibration, gas mixtures, reference sample, preparation.

6145/1

Foreword

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International Standard ISO 6145/1 was prepared by Technical Committee ISO/TC 158, Analysis of gases.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to/any other international Standard implies itsb-e6f8-4cc6-b31blatest edition, unless otherwise stated. 38492c99347d/iso-6145-1-1986

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Gas analysis — Preparation of calibration gas mixtures — Dynamic volumetric methods — Part 1 : Methods of calibration

0 Introduction

Dynamic volumetric methods consist of the introduction of a given flow rate q_A of gas A into a constant flow rate q_B of a complementary gas B. Gas A can be either a pure component I or a mixture, defined by its concentration C_{IA} of the component I, obtained by another preparation method.

The flow rate of the resultant mixture M of gases A and B in which the concentration¹⁾ of component I is C_{IM} , is taken as q_{M} . The value of the final concentration C_{IM} is calculated from S. I the flow rates of gases A and B or from the ratio q_A/q_B or may be obtained from the measurement of the concentration of component I in the resulting mixture, as given by the equation 5-1:00 https://standards.ite a/cataloo/standards/si

$$C_{\rm IM} = C_{\rm IA} \left(\frac{q_{\rm A}}{q_{\rm A} + q_{\rm B}} \right)$$

It is assumed with this equation that C_{IB} , the concentration of component I in gas B, is zero.

The introduction of gas A can be continuous (e.g. permeation tube) or pseudo-continuous (e.g. piston volumetric pump). A mixing chamber should be inserted in the system before the analyser and is particularly essential in the case of pseudocontinuous introduction. The flow rate of component A is measured either directly in terms of volume or mass, or indirectly by measuring the variation of a physical property.

The dynamic volumetric preparation techniques produce a continuous flow rate of calibration gas mixtures into the analyser but do not generally allow the build-up of a reserve by storage under pressure.

The main techniques used for the preparation of the mixtures are :

- a) volumetric pumps;
- b) periodic injection;
- c) continuous injection;
- d) capillary;

- e) sonic orifices;
- f) mass flow controllers;
- g) diffusion;
- h) permeation.

DD

In all cases, and most particularly if very dilute mixtures are calculated from S in all cases, and most particularly if very dilute mixtures are concerned, the materials used for the apparatus should be chosen as a function of their resistance to corrosion and low absorption capacity (usually glass, PTFE or stainless steel). It by the equation $\frac{1}{12}$ -1:19 should, however, be pointed out that the phenomena are less har catalog/standards/sis/inportant_for dynamics volumetric methods than for static 38492c99347d/iso-614

> Numerous variants or combinations of the main techniques can be considered and mixtures of several constituents can also be prepared by successive dilutions.

> Some of these techniques allow calculation of the final concentration of the gas mixture from basic physical information (e.g. mass rates of diffusion, flow through capillaries). However, since all techniques are dynamic and rely on stable flow rates, this International Standard emphasizes calibration of the techniques by measurement of the individual flow rates or their ratios, or by determination of the final mixture concentration. The accuracy of the composition of the calibration gas mixture is determined by the method of calibration. The dynamic volumetric technique used has a level of precision associated with it. Information on the final mixture composition depends both on the calibration method and on the preparation technique.

1 Scope and field of application

The International Standard constitutes part 1 of ISO 6145, which deals with the various dynamic volumetric methods used in the preparation of calibration gas mixtures.

¹⁾ In this International Standard, the term "concentration" designates a concentration a mass ratio, a volume ratio, a molar ratio or a ratio of pressures.

It describes the calibration methods involved in the preparation of gas mixtures by dynamic volumetric techniques. It also gives a brief presentation of a non-exhaustive list of examples of dynamic volumetric techniques which are described in more detail in other parts of ISO 6145.

2 References

ISO 6142, Gas analysis - Preparation of calibration gas mixtures - Weiahina methods.

ISO 6143, Gas analysis - Determination of composition of calibration gas mixtures - Comparison methods.

ISO 6147, Gas analysis - Preparation of calibration gas mixtures - Saturation method.

ISO 6349, Gas analysis - Preparation of calibration gas mixtures - Permeation method.

ISO 7395, Gas analysis - Preparation of calibration gas mixtures — Mass dynamic method.

ISO 6145, Gas analysis - Preparation of calibration gas mixtures - Dynamic volumetric methods

- iTeh STANDA Ra) measurement of flow rate (mass or volume); - Part 2: Volumetric pumps.¹⁾
- Part 3: Periodic injections into a flowing gas stream. b) comparison method ; (standard
- Part 4: Continuous injection method.
- Part 5: Capillary calibration devices.¹⁾

c) tracer method; <u>ISO 6145-1:19</u>86

- https://standards.iteh.ai/catalog/standards/sisdiress.shemicakanalysis31b-Part 6: Sonic orifices.
- Part 7: Mass flow controllers, 1)
- Part 8: Diffusion.1)

38492c99347d/iso-6145-1-1986 Table 1 shows the applicability of each calibration method to the different preparation techniques.

Table 1 – Calibration methods applicable to the preparation techniques

		Calibration methods				
Preparation techniques	Comparison (ISO 6143)	Flow rate measurement	Tracer	Direct analysis		
Volumetric pumps	+	+	+	May be ap-		
Periodic injection	+		+	plicable; depends		
Continuous injection	+		+	ponents		
Capillary	+	+	+	-		
Sonic orifice	+	+	+			
Mass flow controllers	+	+	+			
Diffusion	+					
Permeation	+					

ISO 8959/2, Gas flow rate measurement - Volumetric method - Part 2: Bell prover. 1)

3 **Calibration methods**

3.1 General

The accuracy and precision of the concentration $C_{\rm IM}$ of a component I of a calibration mixture M depends at any time on the accuracy and precision of the calibration method, the frequency with which it is applied, and the stability of the regulation devices involved in the dynamic preparation technique.

To assess the instantaneous accuracy of the whole procedure, account has to be taken of the time required for the calibration method and the instantaneous possible variation of the principal parameters of the technique as well as any possible drift of these parameters during the calibration procedure.

According to the preparation technique for the gas mixtures used, calibration can be carried out by one of the following methods:

In general, the principles of the methods fall into two categories, as follows:

a) those in which the flow rates of component gases are measured either by volume or by mass and in which the concentration in the final mixture is calculated from the flow rate. Different techniques may be used for the individual components of a mixture and these may be calibrated by different methods. The principle of measurements of individual flow rates, however, remains;

b) those which operate directly on the final mixtures.

Since different principles are involved, they are given separately under each individual method.

Since the calibration methods have different principles and the equipment used for the realization of the gas flow rates is different, different units can be used to express the concentrations.

For calibrations using the comparison method, the concentration is expressed in mole fractions because most of the calibration gas mixtures used for the comparison will if possible be described in this way.

Using techniques based on volume flow rate leads in the first instance to volume ratios or percent volume. Recalculation of these data to mole fractions is possible but leads to an increase in the uncertainty because of the uncertainty of the density and molar-volume data. In this case the expression in volume fractions is preferred.

Calibration with the gravimetric method gives mass fractions -1:193.2.2 Description of flow measurement devices for the concentrations of components in gas mixtures These rds/sist/5038a0cb-c618-4cc6-b31bcan be recalculated to mole fractions by using the atomicso-6143.2.2.186 Soap-film flowmeter weights. Expression in mole fraction is therefore preferred.

3.2 Measurement of individual flow rates

3.2.1 General

Under some circumstances the total flow rate cannot be taken as the sum of two individual flow rates q_A and q_B which have been measured separately. These problems of non-additivity can be caused by deviations from the laws of ideal gas behaviour or by changes in conditions such as back-pressure or viscosity resulting from the blending of the two flow rates. Deviations from ideal behaviour can be predicted with reasonable accuracy and other non-additivity errors can be minimized by careful attention to apparatus design.

Flow rate measurement is normally carried out using one of the following :

- a) soap-film flowmeter;
- b) mercury-sealed piston flowmeter;
- c) wet gas meter;
- d) variable area flowmeter;
- e) thermal mass flow sensor.

The soap-film and mercury-sealed piston flowmeters share a common principle, i.e. that of timing the travel of a soap bubble or piston between carefully defined points. The wet gas meter is an integrating device which indicates the total volume of gas that has been passed through it (the dry gas meter, familiar from the domestic environment, has a similar integrating property but has not been included because it is less accurate). The variable area flowmeter ("rotameter") is a continuously indicating device. The thermal mass flow sensor measures mass flow rate as a function of heat flux.

Calibration of these flow rate measuring devices is carried out using one of the following methods:

f) bell prover;

3.2.2.1.1 Principle

- g) gravimetric method;
- h) weighing the volume of water.

The bell prover is a device for creating a constant and defined flow rate of gas, acting as a mechanically driven gas holder. The gravimetric method measures the mass of gas which has flowed at a constant rate for a defined time through the device to be calibrated. The volume of water equivalent to the measuring volume of a soap-film or mercury-sealed piston flowmeter can be accurately weighed under carefully controlled conditions.

The bell prover and the gravimetric method can be used directly, where appropriate, to calibrate the various preparation techniques, but the information is more commonly transferred via one of the flow rate measurement devices.

A special soap-film flowmeter is used for this measurement. The size of the tube is chosen to be much larger than normal so that the volume and time measurements can both the made with greater accuracy. The measuring tube (see figure 1) is installed vertically in an insulated box, inside which air is continuously circulated at a controlled temperature in the range 23,3 to 23,5 °C with a variation of \pm 0,02 °C. The temperature is controlled by thermometers which are calibrated to 0,1 °C and which can be read to 0,01 °C.

The gas to be measured is saturated with water vapour at the temperature of the measuring tube by passing it through three successive wash bottles, each of which contains a sintered bubbler. It is then introduced into the measuring tube by the side-arm at the top. The solution used to produce the soap-film is introduced dropwise through the centre tube at the top. This solution consists of demineralized water containing 0,005 mol/l of sulfuric acid, 4 ml/l of a non-ionic surface-active agent¹⁾ and 1 ml/l of a colouring agent (e.g. mixed indicator) for better light absorption. When a drop of solution arrives at the narrow section at the top of the tube, a film forms across the tube and is carried down it by the gas flow. Photoelectric cells and lights are located at each side of the tube, positioned in such a way

¹⁾ A suitable product, available commercially, is Nekanil C. This information is given for the convenience of the user of this International Standard and does not constitute an endorsement of this product by ISO.

that they cannot move. As the soap-film passes the photoelectric cells, it first starts and then stops an accurate electronic timer. At the outlet of the tube the film bursts in a syphon, and the gas passes out of the system. The pressure in the measuring tube is measured by a U-tube manometer filled with water and connected to the outlet. Before a measurement is made, several bubbles should be formed and passed along the tube to ensure that the walls are thoroughly wetted.

3.2.2.1.2 Influence of temperature variation

The values measured are all recorded in the temperature band between 23.3 and 23.5 °C. The measuring tube is made of borosilicate glass having a coefficient of linear expansion of $3.3 \times 10^{-6} \text{ K}^{-1}$. The result is that, taking into account the control of temperature to \pm 0,02 °C, there is a maximum variation in the volume of the measuring tube of approximately 2×10^{-7} and a maximum variation in the volume of gas of 7×10^{-5} .

3.2.2.1.3 Influence of the pressure drop

Variations in solubility over the temperature range likely to be used for this measurement will not significantly affect the likely error.

3.2.2.1.8 Reduction in vapour pressure of water

The reduction in the partial pressure of water caused by the surface active agent or the indicator can only be about 1 % relative. This should remain the same between calibration and measurement and so the errors are represented by the variation in this reduction, according to the degree of saturation (see 3.2.2.1.6). If the variation is 10 % relative, this is 10 % of 3×10^{-4} , i.e. 3×10^{-5} .

3.2.2.1.9 Error summation

The sum of the possible errors described in 3.2.2.1.2 to 3.2.2.1.8 is as follows:

22212 Influence of the pressure drop	temperature	7×10^{-5}
	pressure	1×10^{-4}
The pressure in the soap-film flowmeter will not vary from at-	diffusion	0
this can be measured to 0,1 mbar, giving an error of 10^{-4} .	film thickness	2×10^{-5}
	humidity	3×10^{-4}
3.2.2.1.4 Diffusion across the film ireh STAND		4×10^{-5}
Diffusion of the gas across the soap-film will not occur since and vapour pressure		3×10^{-5}
the composition is identical on each side.	Total	5.6×10^{-4}

3.2.2.1.5 Influence of film thickness

<u>ISO 6145-1:1986</u>

ss https://standards.iteh.ai/catalog/standards/sst/Mercury-sealed/piston-flowmeter

The thickness of the soap-film layer and of the volume of 4 and 9347d/iso-6145-1-1986 contained in the measuring tube can be determined accurately with a burette. Two drops per measurement are enough to produce a bubble which traverses the measuring volume without bursting. The thickness of the film varies the volume. The volume of a drop of liquid is 0.03 ml compared with a tube volume of 1 356 ml, i.e. an error of 2 \times 10⁻⁵ and with a reproducibility of \pm 20 % on two drops.

3.2.2.1.6 Differences in humidity

The saturator is of high efficiency, such that a variation of 1 % relative humidity between one gas and another corresponds to a variation in the volume of the gas of

$$\frac{21,5}{760} \times \frac{1}{100}$$

i.e. 3×10^{-4}

3.2.2.1.7 Solubility of gases in water

Typical values for solubility in 1 ml of water at ambient temperature are 0,02 ml of hydrogen, 0,03 ml of oxygen and 0,87 ml of carbon dioxide.

Even if the water is pure the error in the volume resulting from the carbon dioxide dissolved in 0,6 ml of the liquid just introduced into the measuring tube amounts only to 0,05 ml per 1 356 ml, i.e. an error of 4 \times 10⁻⁵.

3.2.2.2.1 Principle

A precision bore glass measuring tube (see figure 2) is set vertically in an insulated box fitted with temperature regulation. The temperature is maintained constant to within \pm 0.02 °C.

The measuring tube is divided into a number of sections by photoelectric cells serving as sensors, and the actual volume between two adjacent photoelectric cells is determined by filling with water and weighing (see 3.2.3.3).

A constant flow moves a frictionless piston with a constant speed upwards. The displaced volume can be estimated from the dimensions of the tube or measured from the water calibration.

The piston, made of plastics (e.g. PVC) or glass contains a horizontal, circular groove, filled with mercury. The purity of the mercury is such as to ensure that the piston does not stick in operation.

The piston is allowed to attain a constant speed before time counting is allowed to start at sensor 1.

Depending on the flow rate and the tube size, counting is stopped when the piston passes sensor 2 or sensor 3. Sensors can be of the reflection type because of the high reflectance of the mercury ring. Because of a high back-pressure caused by the weight of the piston, the measured pressure difference is of the order of 1 mbar up to 10 mbar. The measuring sequence starts with closing side 1 of the 3-way valve (see figure 2). As soon as the piston passes sensor 1, time counting starts; it stops after the piston passes the next sensor. The 3-way valve resets its position and the piston falls down on the spring. The flowmeter is then ready to start again.

3.2.2.2.2 Influence of temperature variation

The measuring tube is made of borosilicate glass having a coefficient of linear expansion of $3,3 \times 10^{-6} \text{ K}^{-1}$. The result is that, taking into account the control of temperature to \pm 0,02 °C, there is a maximum variation in the volume of the tube of approximately 2×10^{-7} and a maximum variation in the volume of gas of 7×10^{-5} .

3.2.2.2.3 Influence of the pressure drop

The pressure difference over the mercury-sealed piston depends on the mass and dimensions of the piston.

Pressure differences from 1 mbar to 10 mbar are found in practice. For the volume calculation a correction for this pressure difference should be made:

$$V = V_{o} \left(\frac{p + 1\ 013}{1\ 013} \right)$$
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The accuracy of the determination of this pressure is about S 0,01 mbar, which gives an error on the volume of 10^{-5} .

3.2.2.2.4 Diffusion accross the piston

The construction of the mercury-sealed piston does not provide for the possibility of keeping the same composition of the gas on both sides. Though diffusion along the mercury seal is still possible, the effect is considered negligible in general practice.

3.2.2.2.5 Error summation

The sum of the possible errors described in 3.2.2.2.2 to 3.2.2.2.4 is as follows:

Total	8 × 10 ⁻⁵
diffusion across piston	0
pressure	1×10^{-5}
temperature	7×10^{-5}

3.2.2.3 Wet gas meter

3.2.2.3.1 Principle

A wet gas meter consists of a drum which can rotate about a horizontal axis within an outer case. The drum is divided into four compartments of equal capacity which are arranged radially about the axis of rotation. Each compartment has an inlet opening at one end and an outlet at the other. The drum rotates in water or oil which is contained in the outer case and the level of which is adjusted to act as a seal so that the compartments fill with, trap and deliver gas in succession. The positions of the inlet and outlet openings are such that at no time are both unsealed by being above the fluid level. The vanes which separate the compartments dip below the fluid at all times, but are not continued to the spindle, so that fluid is able to pass to and from each compartment as the drum rotates.

Figure 3 shows the operation of a single compartment of the meter. In (a) the compartment is filled with fluid with both connectors sealed. Rotation of the drum brings it to position (b), where gas can flow into the compartment and by doing so continues to rotate the drum. At position (c) both connectors are sealed once more with a defined volume of gas in the compartment, which is then discharged during further rotation (d).

A pointer attached to the drum spindle rotates against a circular scale on the front of the instrument and a simple mechanical integrator records the number of revolutions. Meters are generally available in the ranges 0,25 to 25 I/revolution, suitable for maximum flow rates of 140 to 3 000 I/h. Meters with external water jackets are available for precise temperature control.

If the fluid in the meter is water, the gas entering the meter should be saturated with water vapour at the temperature of the meter. This prevents loss of water from the meter and uncertainty as to the correction for gas volume. If the fluid is oil of low vapour pressure, no such precaution is necessary. A small amount of pressure is necessary to operate the meter and this is measured by a U-tube manometer at the meter inlet. Due to slight differences in volume between the compartments, the rate of gas delivery may vary slightly during a complete revolution and so, where possible, only whole numbers of revolutions

ISO 6145-1:1905 the meter should be used. For the same reason, the inlet standards.iteh.ai/catalog/standards/sip/ressure may/yary_during a complete revolution and the mean 38402c00347d/ize_61_pressure should be estimated.

> The meter should be kept level during calibration and use. The level of fluid in the meter is also critical as it defines the volumes of the compartments. This level can be most accurately adjusted with no gas flowing through the meter, after which calibration for each required value of flow rate is necessary. Calibration is independent of the gas used, provided that the fluid is given time to become saturated with the gas in use, and free from any previously used gas.

3.2.2.3.2 Temperature variations

Thermal expansion of the meter can be neglected, since the effect on the contained gas volume is much greater. Assuming that the temperature can be read during calibration and analysis to within 0,05 °C, and the appropriate correction applied, the maximum variation of 0,1 °C represents an error of $3,4 \times 10^{-4}$.

3.2.2.3.3 Influence of pressure

Atmospheric pressure can be measured with an uncertainty of 2×10^{-5} , giving a variation in the measured value between calibration and analysis of 4×10^{-5} . The excess pressure in the meter, which varies during the rotation cycle, can be measured with an uncertainty of 0,1 mbar, giving a variation between calibration and analysis of 2×10^{-4} . A total error of $2,4 \times 10^{-4}$ may be expected.

3.2.2.3.4 Differences in humidity

If the meter is filled with water then, in the range 18 to 22 °C, the 0.1 °C temperature variation will cause an uncertainty in the vapour pressure of water of about 0,14 mbar, and hence an uncertainty in the total gas volume of 1.4×10^{-4} . Also variation in saturator efficiency of 1 % relative humidity will cause a variation in gas volume of 3 \times 10⁻⁴. A total error of 4,4 \times 10⁻⁴ may be expected.

3.2.2.3.5 Error summation

The sum of the possible errors described in 3.2.2.3.2 to 3.2.2.3.4 is as follows:

	Water-filled	Oil-filled
temperature	3,4 × 10 ⁻⁴	3,4 \times 10 ⁻⁴
pressure	2,4 × 10 ⁻⁴	$2,4 \times 10^{-4}$
humidity	$4,4 \times 10^{-4}$	0
Total	$1,02 \times 10^{-3}$	5,8 × 10 ⁻⁴

3.2.2.4 Variable area flowmeter

3.2.2.4.1 Principle

glass tube mounted vertically with the smaller diameter at the

bottom. The tube contains a spherical or conical float, the $O = 6145 - 1 \frac{1986}{\Delta q} = 1 \Delta T$ largest diameter of which is slightly smaller than the bore at the standards 33840 e6f8-4cc6-b31b-bottom of the tube. Suitable stops at the top and bottom of the 347 d/so -6745 - 1298Ttube prevent the float from being lost.

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When gas enters the lower end of the tube, the float rises to a position at which its weight is supported by the pressure drop across the annulus between the float and the tube. The position of the float is read against graduation marks on the tube.

The flow rate, q, is given by

$$q = K_1 \cdot h \left[\frac{\varrho_{\rm f} - \varrho_{\rm g}}{\varrho_{\rm g}} \right]^{0.5}$$

where

 K_1 is a constant which includes the coefficient of discharge through the annulus;

h is the height of the float in the tube;

 $\rho_{\rm f}$ is the density of the float;

is the density of the gas in the same units as ϱ_{f} . ϱ_a

Since $\varrho_{\rm f} \gg \varrho_{\rm q}$

 $q = \frac{K_2 \cdot h}{(\varrho_{\rm q})^{0,5}}$

where K_2 is a constant including K_1 and the density of the float.

These equations suggest that different gases can be used, or the same gas at different pressures with appropriate conversion factors based upon the change in $\varrho_{\rm g}.$ In fact, this can cause serious errors as the coefficient of discharge can change; a variable area meter should, therefore, be used only with the gas with which it was calibrated and under the same pressure conditions.

Any individual meter will have a measuring range of about 10:1. The range of meters available should cover any flow rate needed for dynamic volumetric methods from 10 ml/min upwards.

3.2.2.4.2 Resettability of flow rate

Between calibration and use, the perceived variation in flow rate should not exceed 2 \times 10⁻² provided that the float is in the upper half of the scale.

3.2.2.4.3 Influence of temperature variation

By comparison with the meters described in 3.2.2.1, 3.2.2.2 and 3.2.2.3, variable area meters are relatively crude devices and as such are often used subject to variations caused by changes in ambient conditions without these changes being measured and corrections applied. The equations given in 3.2.2.4,1 show that flow rate varies inversely with the square A variable area flowmeter ("rotameter") consists of a tapered temperature so that

> Hence, for a variation of 3 °C between calibration and use, the variation in flow rate is 5 \times 10⁻³.

3.2.2.4.4 Influence of pressure variation

The variable area flowmeter is most likely to be used at atmospheric pressure. The pressure drop across the meter is low (up to 2 mbar) and so possible variations in atmospheric pressure are much more significant. Gas density is directly related to pressure so that

$$\frac{\Delta q}{a} \approx \frac{1}{2} \cdot \frac{\Delta p}{p}$$

Hence, for a variation of 20 mbar between calibration and use, the variation in flow rate is 1×10^{-2} .

3.2.2.4.5 Error summation

The sum of the possible errors described in 3.2.2.4.2 to 3.2.2.4.4 is as follows:

resettability		2	×	10-2
temperature		5	x	10-3
pressure		1	×	10-2
	Total	3,5	×	10-2

3.2.2.5 Thermal mass flow sensor

3.2.2.5.1 Principle

A thermal mass flow sensor relates an applied upstream/ downstream temperature difference to a mass flow rate.

A constant heat flux, Φ , given to a gas stream, causes a temperature rise, ΔT , determined by the heat capacity of the particular gas and the flow rate such that

$$\Delta T = \frac{\Phi}{c_p \cdot q_m}$$

Rearranging this equation gives mass flow rate

$$q_m = \frac{\Phi}{c_p \cdot \Delta T}$$

and volume flow rate

$$q_V = \frac{\Phi}{\varrho \cdot c_p \cdot \Delta T}$$

where

3.2.2.5.3 Influence of pressure variation

The values of $c_{\rm p}$ and ϱ are influenced by pressure variations to an extent which depends upon the gas. Table 3 gives the values for some gases at a temperature of approximately 290 K and a pressure of approximately 1 013 mbar.

Tab	le 3	3 —	Examples	of	influence of	pressure	variation
	_			_			

Gas	<i>c_p</i>	d <i>c_p/</i> d <i>p</i>	<i>₽</i>	d <i>ϱ/dp</i>
	(kJ⋅kg ⁻¹ ⋅K ⁻¹)	(kJ⋅kg ⁻¹ ⋅K ⁻¹ ⋅bar ⁻¹)	(kg⋅m ⁻³)	(kg⋅m ⁻³ ⋅bar ⁻¹)
N ₂	1,04	0,001 7	1,163	1,16
O ₂	0,92	0,000 8	1,335	1,30
CO	1,04	0,000 8	1,176	1,17
CO ₂	0,82	0,007 1	1,833	1,83
CH₄	2,21	0,006 3	0,666 7	0,67

3.2.2.5.4 Influence of the orientation

Some sensors are sensitive to changes in their orientation. There is very little quantitative information on the phenomenon.

In practice, the orientation should be the same during calibraiTeh STANDARDtion and use when this influence is not known.

 Φ is the heat flux which is fixed by the construction principal size 3.225.51 influence of variation of the heat flux ple of the device:

ISO 6145-1:19The influence of changes in the heat flux, Φ , appears directly in ρ is the density of the gas: https://standards.iteh.ai/catalog/standards/sistheydetermined flow rate values. It is therefore very important c_p is the specific heat at constant pressure, which only on the stability of this value.

depends only on the gas.

The temperature difference, ΔT , is determined by upstream and downstream temperature sensors which form the two arms of a Wheatstone-bridge network, balanced to zero reading when there is zero flow rate.

3.2.2.5.2 Influence of temperature variation

Variation of temperature leads to a variation in the value of c_n and ρ (in the case of q_V).

The temperature variations of c_p and ρ are not the same for different gases. Table 2 gives examples of c_p , dc_p/d_T , ρ and $d\rho/dT$ at a temperature of approximately 290 K and a pressure of approximately 1 013 mbar.

Table 2 - Examples of influence of temperature variations

0	c _p	dc_p/dT	Q	$d\varrho/dT$
Gas	(kJ-kg-1-K-1)	(kJ.kg ⁻¹ .K ⁻²)	(kg·m ⁻³)	(kg⋅m ⁻³ ⋅K ⁻¹)
N ₂	1,04	0,000 04	1,163	0,004 0
02	0,92	0,000 08	1,335	0,005 2
cõ	1,04	0,000 08	1,176	0,003 6
CO_2	0,82	0,000 8	1,833	0,006 1
CH ₄	2,21	0,002 1	0,666 7	0,002 3

Measurement of voltage and current shows that stabilities of better than 10^{-4} are obtainable.

3.2.2.5.6 Error summation

The sum of the possible errors described in 3.2.2.5.2 to 3.2.2.5.5 is as follows for nitrogen:

heat flux	0 × 10-4
orientation	0
pressure (\pm 0,01 bar)	8×10^{-6}
temperature (± 1 K)	2×10^{-5}

3.2.3 Calibration of flow measurement devices

3.2.3.1 Bell prover

3.2.3.1.1 General

ISO 8959/2 specifies a volumetric method of gas flow rate measurement in closed conduits by measuring the gas volume displaced from a bell prover within a definite period of time. The standard deals with the measuring apparatus, the measuring procedure, the methods for calculating the flow rate and the measurement uncertainties.

This subclause gives an outline of the principle of operation and sources of error of a bell prover but it is not intended to replace reference to ISO 8959/2 for the operating conditions and requirements.

The error calculation presented in 3.2.3.1.8 uses the summation of maximum errors. This approach is different to the method given in ISO 8959/2 but has been adopted to be consistent with the assessment of errors presented in other International Standards produced on the preparation of calibration gas mixtures.

3.2.3.1.2 Principle

A schematic diagram of a bell prover is given in figure 4 and shows the bell (1) in a stationary tank (2) filled with the sealing liquid (3). The measuring scale (4) is used to take readings of the position of the bell, which is supported on a chain passing over rollers (5) and balanced by the counterweight (6).

The principle of operation is as follows:

a) The bell is raised and filled with air.

b) A definite volume of air is displaced from the prover by lowering the bell (1) into the stationary tank (3) while maintaining constant pressure in the conduits. The time interval over which the air is displaced is measured by a timer. The air flow rate is calculated using the measured values of volume and time interval.

3.2.3.1.7 Error due to the recalculation of flow rates to reference conditions

These are normally avoided by carrying out calibrations under the required conditions.

3.2.3.1.8 Error summation

The sum of the possible errors described in 3.2.3.1.3 to 3.2.3.1.7 is as follows:

capacity		1×10^{-3}
measuring rule		4×10^{-4}
timing		$2,5 \times 10^{-5}$
distribution		$7,5 \times 10^{-4}$
recalculation		0
	Total	$2,2 \times 10^{-3}$

This total is the maximum error on the mean flow rate and instability of the flow rate has not been taken into consideration.

3.2.3.2 Gravimetric method

3.2.3.2.1 Principle

g the measured **Statistical Gas from a cylinder** flows at a constant rate through the device to be calibrated. This is continued for a sufficiently long period <u>ISO 614for[_the6</u>loss of mass from the cylinder to be accurately https://standards.iteh.ai/catalog/standmeasured, The flow cate is then calculated from the mass of the 38492c99347d/gas and the time during which it flowed.

3.2.3.1.3 Error on prover capacity

The volume of the bell prover is determined at various points over the usable range and the error on each volume determination is assumed to be less than 0.5 cm^3 . A best-fit line is drawn through the volume determinations to provide a calibration graph having a maximum error of ± 0.05 %. The volume discharged from the bell prover is the difference in volume between the start and finish point, giving a maximum error of twice the calibration error which is ± 0.10 %.

3.2.3.1.4 Errors in the use of the measuring rule

The position of the bell prover is determined using a measuring rule which may be read to better than 0,2 mm. Assuming a change in position of 1 m, the maximum error is 0,4 mm in 1 m, or 0,04 %.

3.2.3.1.5 Error on displacement time interval

The time interval may be measured to better than \pm 0,001 s. Assuming a discharge time of 40 s, the maximum error is 0,002 5 %.

3.3.3.1.6 Error on the gas distributing device

Random variations in the speed of operation of the solenoid valve which starts and stops the gas discharge will not exceed \pm 0,03 s. On a discharge time of 40 s, the maximum error is 0,075 %.

ISO 7395 describes the use of this principle to make mixtures each of whose components are measured in this way. The mass dynamic method prepares mixtures for use at the moment of preparation and avoids non-additivity errors since it deals with masses. The same principle is used in the gravimetric method to calibrate individual flow rate measurement devices. This produces data in terms of mass flow, which can then be translated to molar flow rate or, with some degree of uncertainty, volume flow rate. When the flow rate measurement devices are used to prepare a mixture, non-additivity problems can arise as described earlier.

The gas cylinder and flow rate measurement device are set up as shown in figure 5. The cylinder (1) is fitted with a pressure regulator (2), on the outlet of which a precision needle valve (3) and shut-off valve (4) lead to the device to be calibrated (5). The dead volume between the needle valve outlet and the shutoff valve should be minimized by using the smallest size of tubing and fittings commensurate with the desired gas flow rate. The temperature and pressure of the gas are measured at the inlet to the device to be calibrated.

The cylinder is turned on, the pressure regulator is set to a value of, say, 1 bar gauge, and the needle valve is adjusted to the desired flow rate. When conditions are seen to be steady, the shut-off valve is closed and the pipework is disconnected at the outlet of this valve. The cylinder, regulator, needle valve and shut-off valve are weighed as a single unit. The pipework is reconnected and the shut-off valve is opened to re-start the

flow at the same rate. After the gas has flowed for long enough for the mass used to be measured accurately, the shut-off valve is closed and the cylinder, regulator, needle valve and shut-off valve weighed as before. The time during which the gas flowed is accurately measured. The volume of gas flowed is calculated from the change in mass, and the flow rate from the volume and the time.

3.2.3.2.2 Weighing error

Gravimetric preparation of mixtures is described in ISO 6142. Using the procedures described in this part of ISO 6145, it may be assumed that the mass of gas used in a test can be weighed with an error of 2×10^{-4} (i.e. 20 g of gas taken from a 10 kg cylinder whose mass before and after the test can be measured with an error of 2 mg, giving a total uncertainty of 4 mg in 20 g).

3.2.3.2.3 Transient flow changes

No error is involved with the cylinder and its flow rate control devices as both are pressurized with gas to the same degree for both weighings. However, when the gas is shut off before weighing, the pipework between the needle valve and the shut-off valve becomes pressurized to the value set on the regulator, and this will cause a surge when the gas flow rate restarts. The maximum relative error caused by this surge is the amount of gas required to pressurize the volume between the needle valve and the shut-off valve relative to the amount of gas flowed. If 2 ml of dead-space is pressurized to 1 bar gauge in a test in which 20 g of methane flows, the maximum error is 7 \times 10.45-1:10 follows:

3.2.3.2.7 Error summation

The sum of the possible errors described in 3.2.3.2.2 to 3.2.3.2.6 is as follows:

weighing	2×10^{-4}
flow transients	7×10^{-5}
mass to volume	3,7 × 10 ⁻⁴
flow rate variation	2×10^{-3}
timing	2×10^{-4}
Total	$2,8 \times 10^{-3}$

3.2.3.3 Volume measurement by weighing the water content

3.2.3.3.1 Principle

The volume of the device is determined by filling it with water and weighing the volume expressed between the upper and lower points where the photoelectric detector cells are sited. This procedure provides a calibration of the volume of the meter. In the case of the soap-film flowmeter, this may not necessarily correspond to the volume swept out by the soap bubble. The apparatus shown in figure 6 is used for the determination and the sequence of operations for calibration is as follows:

https://standards.iteh.ai/catalog/standards/sist/5038a0eb-e6f8-4cc6-b31b-

3.2.3.2.4 Error on conversion of mass to volume

The temperature, pressure, compressibility (κ) factor and relative molecular mass of the gas all affect the error on conversion of mass to volume. Measurement of temperature to 0,05 °C and pressure to 0,1 mbar represent errors of 1,7 × 10⁻⁴ and 10⁻⁴ respectively. Compressibility factors are commonly quoted to four places of decimals, which implies an error of 10⁻⁴, and relative molecular masses are known with sufficient accuracy not to contribute significantly to error. The total uncertainty is therefore not greater than 3,7 × 10⁻⁴.

3.2.3.2.5 Error due to flow rate variation

If the device to be calibrated measures either instantaneous flow rates or volumes which are small by comparison with the volume taken from the cylinder, then variations in flow rate will be a source of error. A high quality pressure regulator and needle valve should ensure a flow rate constancy of 0,2 % relative, apart from the initial flow surge (see 3.2.3.2.3), but should be checked for each installation. This level of flow rate control represents an error of 2×10^{-3} .

3.2.3.2.6 Error on time measurement

The time during which the gas flows from the cylinder can be measured by an electronic timer with an error of no greater than 2 \times 10⁻⁴.

38492c99347d/iso-6145-1a) 9{nitially ensure that the flowmeter assembly is carefully cleaned and freed of grease.

b) Fill burette A, which should have a larger capacity than the meter, with air-free, triple-distilled water. Ensure that the burette is firmly clamped in position.

c) Weigh a container and cap of sufficient volume to hold the water discharged from the flowmeter.

d) With the detector system in operation, slowly open tap 1 to allow water into the flowmeter system until detector 1 is activated. Allow 2 cm of water above this level. Close tap 1.

e) Carefully open tap 2 to displace air from the tap assembly and then allow water to run to waste until detector 1 responds to the meniscus level. Immediately close tap 2.

f) Place the container without its cap under tap 2.

g) Open tap 2 and allow water to flow slowly from the flowmeter assembly until detector 2 operates. Immediately close tap 2 and cap the container to prevent loss of water by evaporation. Check to ensure that drainage in the flowmeter raises the level above the set point and re-adjust if necessary.

h) Weigh the capped container on an analytical balance and determine the mass of water.