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Gas analysis — Sampling guidelines

Analyse des gaz — Lignes directrices pour le prélèvement des échantillons

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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).

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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.

Introduction

The determination of gas composition, impurity concentration and physical properties depend, to a large extent, on sampling technique. The use of correct sampling techniques is an important safety and quality critical step in gas analysis. The design, construction and selection of the sampling equipment to avoid hazardous situations and sampling errors are important and directly influence the results obtained. Any slight carelessness, inexactitude or mistake will seriously influence safety and the results obtained.

Gaseous products are stored and transported in pressure receptacles in the form of compressed or liquefied gas or through gas pipelines. The sampling methods used differ depending upon the package, composition and delivery methods.

This document provides technical guidelines for the sampling of gases in pressure receptacles and pipelines for analytical purposes.

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73fee8b96e56/iso-19230-2020

Gas analysis — Sampling guidelines

WARNING — The use of this document can involve a number of hazards. This document does not specify all the safety issues associated with its use. Users of this document are responsible for establishing measures to ensure safety while gas sampling.

1   Scope

This document specifies the general provisions and gives the basic definitions of terms relating to sampling for gas analysis, including sampling devices, sampling methods, sampling technical considerations, and sampling safety.

This document applies to both direct and indirect sampling of gas in pressure receptacles and pipelines, including pure gases and gas mixtures. Compressed and liquefied gases are both considered.

This document applies to the sampling of processed gases and does not involve gas treatment processes.

The sampling procedures specified are not intended for the sampling of special products which are the subject of other International Standards, such as liquefied petroleum gases (see ISO 4257) and gaseous natural gases (see ISO 10715).

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 3165, *Sampling of chemical products for industrial use — Safety in sampling*

ISO 16664, *Gas analysis — Handling of calibration gases and gas mixture — Guidelines*

3   Terms and definitions

For the purposes of this document, the following terms and definitions 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/

3.1
gas
materials which are present completely in gaseous form at a temperature of 20 °C under the absolute pressure of 0,101 3 MPa

Note 1 to entry: The materials here include single mediums and mixtures.

3.2
compressed gas
gas (3.1) which, when packaged under pressure for transport, is entirely gaseous at all temperatures above −50 °C

Note 1 to entry: This category includes all gases with a critical temperature less than or equal to −50 °C.

[SOURCE: ISO 10286:2015, 705, modified — Modified to include all temperatures above −50 °C.]

3.3
liquefied gas
gas (3.1) which, when packaged under pressure for transport, is partially liquid at temperatures above −50 °C

[SOURCE: ISO 10286:2015, 706, modified — Modified to specify that gas is packaged under pressure.]

3.4
high pressure liquefied gas
gas (3.1) with a critical temperature between −50 °C and +65 °C

[SOURCE: ISO 10286:2015, 708]

3.5
low pressure liquefied gas
gas (3.1) with a critical temperature above +65 °C

[SOURCE: ISO 10286:2015, 707]

3.6
toxic gas
gas (3.1) which is known to be so toxic or corrosive to humans to pose a health hazard or which is presumed to be toxic or corrosive to humans because it has a *lethal concentration 50* (3.38) value for acute toxicity equal to or less than 5 000 ml/m^3 (ppm)

Note 1 to entry: Other risks, such as tissue corrosiveness, are sometimes associated.

[SOURCE: ISO 10286:2015, 716]

3.7
gas in pressure receptacles
gas stored in closed cylinders, tube, pressure drums, tanks and other pressure receptacles

3.8
gas in pipelines
gas delivered in pipelines during the production process

3.9
sampling device
components that comprise the *sampling system* (3.10) mainly includes *sample lines* (3.26), pressure regulators/reducers, flow controllers, connectors and sample containers

3.10
sampling system
gas transmission and control system constructed by gas storage container or sample point of *gas in pipelines* (3.8) and various *sampling devices* (3.9)

3.11
representative sample
sample assumed to have the same composition as the material sampled when the latter is considered as a homogeneous whole

[SOURCE: ISO 6206:1979, 3.3]

3.12
direct sampling
sampling in situations where there is a direct connection between the gas to be sampled and the analytical unit

[SOURCE: ISO 10715:1997, 2.1, modified — The word "natural" has been omitted from the definition.]

3.13
indirect sampling
sampling in situations where there is no direct connection between the gas to be sampled and the analytical unit

[SOURCE: ISO 10715:1997, 2.7, modified — The word "natural" has been omitted from the definition.]

3.14
sampling plan
planned procedure of selection, withdrawal and preparation of a sample or samples from a lot to yield the required knowledge of the characteristic(s) from the final sample so that a decision can be made regarding the lot

[SOURCE: ISO 6206:1979, 3.1.5]

3.15
spot sampling
indirect sampling (3.13) from a specific part of the stream of material with a certain volume at a specific time

3.16
incremental sampling
indirect sampling (3.13) by collecting a series of spot samples into a combined sample

3.17
continuous sampling
direct sampling (3.12) taken continuously from a stream of material with a constant flow rate in a certain period of time

3.18
intermittent sampling
direct sampling (3.12) from a stream of material with predetermined intervals

3.19
ullage
outage
space in the container not occupied by the material, or the distance between the material surface and a fixed reference point at the top of the container

Note 1 to entry: This volume allows room for expansion.

[SOURCE: ISO 6206:1979, 3.3.14]

3.20
sampling error
part of the total estimation error of a characteristic due to known and acceptable deficiencies in the *sampling plan* (3.14)

[SOURCE: ISO 6206:1979, 3.4.10]

3.21
incremental sampler
sampler which accumulates a series of spot samples into one composite sample

[SOURCE: ISO 10715:1997, 2.6]

3.22
low-pressure gas
gases with a pressure between 0 MPa and 0,2 MPa at sampling temperature

Note 1 to entry: Except for special provisions, all pressures mentioned in this standard are gauge pressures.

3.23
high-pressure gas
gases with a pressure exceeding 0,2 MPa at sampling temperature

3.24
lag time
time taken for a *representative sample* (3.11) to enter the instrument

[SOURCE: ISO 11042-2:1996, 3.5.1.1]

3.25
sample container
container for collecting the gas sample when *indirect sampling* (3.13) is necessary

[SOURCE: ISO 10715:1997, 2.14]

3.26
sample line
conduit to transfer a sample of gas from the sample place to the analytical unit or *sample container* (3.25)

Note 1 to entry: Another word used for sample line is transfer line.

[SOURCE: ISO 14532:2014, 2.3.2.5]

3.27
sample probe
device inserted into the gas pipelines so that a *representative sample* (3.11) of the flowing gas can be taken

Note 1 to entry: The sample probe will have a conduit to convey the sample from the flowing gas to a point external to the pipeline.

[SOURCE: ISO 14532:2014, 2.3.2.6, modified — The second sentence has been formatted as a note to entry.]

3.28
sampling point
point in the gas stream where a *representative sample* (3.11) can be collected

[SOURCE: ISO 10715:1997, 2.17]

3.29
filling ratio
ratio of the mass of gas to the mass of water at 15 °C that would fill completely a pressure receptacle fitted ready for use

Note 1 to entry: Synonyms are filling factor and filling degree, often expressed in kg/l or similar.

[SOURCE: ISO 10286:2015, 747]

3.30
continuous purging method
purging method by continually purging the *sampling system* (3.10) with sample gases

3.31
fill-empty cycle purging method
purging method by sequentially filling and emptying the *sampling system* (3.10) repeatedly with the gas to be taken

3.32
evacuation-gas purging cycles
purging method by sequentially evacuating and pressurizing the *sampling system* (3.10) with the sample to be taken

3.33
sampling from the gaseous phase
process that takes a sample from the gaseous phase of the *liquefied gases* (3.3)

3.34
sampling from the liquid phase
process that takes a sample from the liquid phase of the *liquefied gases* (3.3)

3.35
sampling in liquid form
process that takes a sample in liquid form directly from the liquid phase of the *liquefied gas* (3.3)

3.36
sampling after evaporation
process that takes a sample in gaseous form by vaporizing the sample from the liquid phase of the *liquefied gas* (3.3)

3.37
liquid valve
device with an internal fixed sample loop, fitted to an analyser for the *direct sampling* (3.12) of *liquefied gas* (3.3) in liquid form, which can keep the liquefied gas to be collected completely in the liquid phase

3.38
lethal concentration 50
LC$_{50}$
concentration of a substance in air exposure to which, for a specified length of time, it is expected to cause the death of 50 % of the entire defined experimental animal population after a defined time period

[SOURCE: ISO 10298:2018, 3.1]

3.39
corrosive gas
gas (3.1) which, when dissolved in water or other liquid, causes corrosion of metal

[SOURCE: ISO 13703:2000, 3.1.4]

3.40
floating piston cylinder
sample container (3.25) that has a moving piston separating the sample from a precharge gas

Note 1 to entry: The pressures are in balance on both sides of the piston.

[SOURCE: ISO 14532:2014, 2.3.2.1, modified — The container has been specified as a "sample container" and the second sentence was formatted as a note to entry.]

4   Sampling plan

A feasible and complete sampling plan should be developed before sampling as shown in Figure 1.
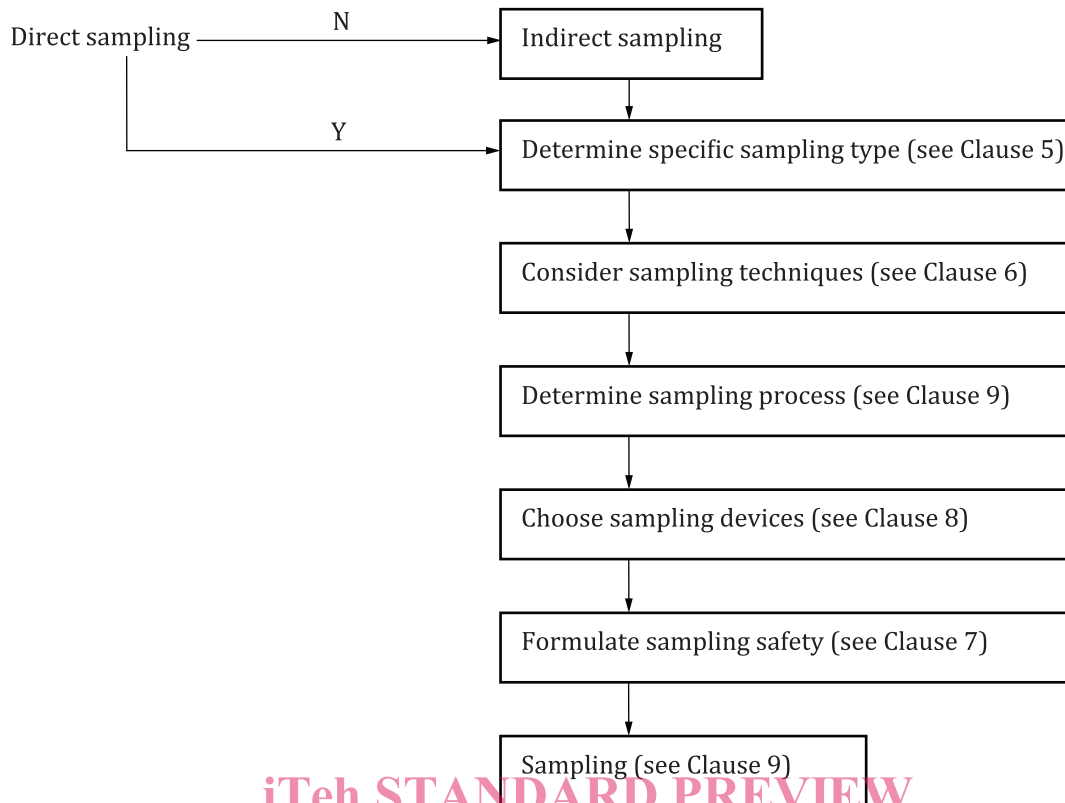
Figure 1 — Scheme of sampling plan

5 Sampling classification

5.1 Sampling classification of gases

In this document, the sampling classification is based on whether the gas is directly fed to the analyser or not. In addition, the sampling methods vary among different gas packaging, storage methods and sampling purposes.

Direct sampling, if possible, is strongly recommended. In the case of indirect sampling, the potential loss of component during the time between sampling and analysis should be studied and incorporated in the uncertainty budget.

In general, for gas in pressure receptacles, the internal composition is relatively uniform and constant.

For gases in pipelines, the purpose and controls required dictate the type of sampling used. The design of a sampling plan should consider whether the objectives of sampling are to:

— determine the instantaneous gaseous composition;

— determine an average composition over a specified time interval;

— establish changes in concentration by repeated sampling over a specified time;

— pass continuous samples into the analyser to measure both limit and average composition.

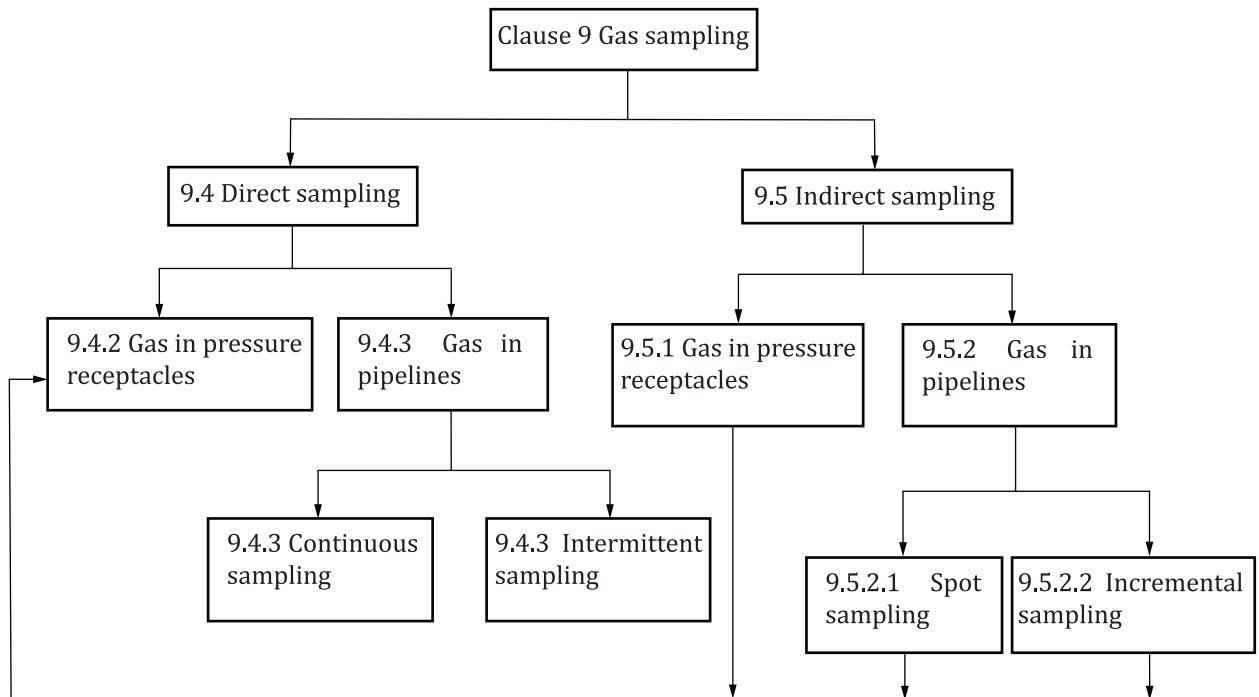A flow chart detailing the gas sampling classification is shown in Figure 2.

Figure 2 — Gas sampling classification

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5.2 Sampling classification of liquefied gas (standards.iteh.ai)

When sampling liquefied gases, a representative sample is obtained by sampling from the liquid phase, however, it can also occasionally be necessary to sample the vapour phase.

Sampling from the liquid phase is further subdivided into sampling in liquid form or sampling after evaporation. The method of sampling used is normally determined by a review of the physical properties of the liquefied gas such as vapour pressure, etc. Generally, high pressure liquefied gases require evaporation whereas low pressure liquefied gases may be sampled in liquid phase.

For the liquefied gas sampling classification, see Figure 3 which details the sampling methods that should be used. Then, follow 5.1 to determine the specific sampling type.

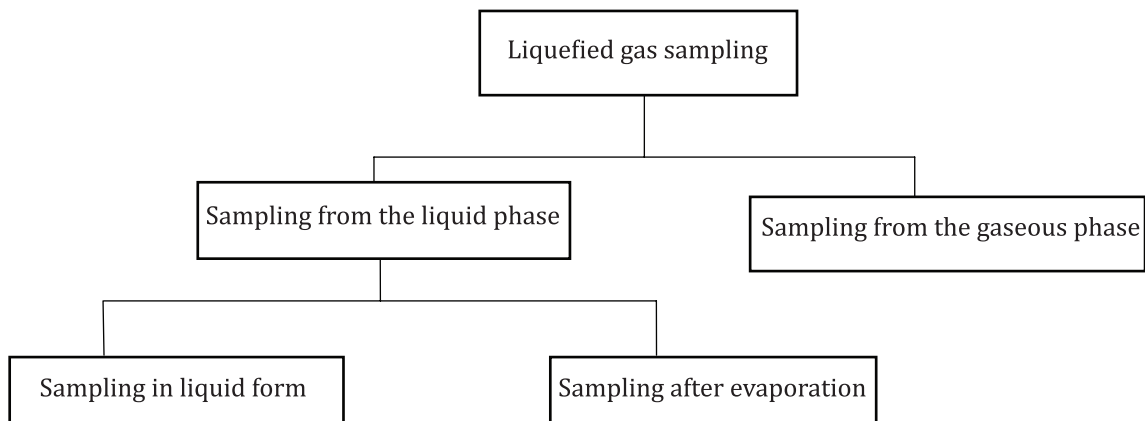**Figure 3 — Liquefied gas sampling classification**

6 Technical specifications

6.1 Overview

For gas sampling, attention should be paid to but not limited to the following technical aspects in order to collect sufficient representative sample.

6.2 General considerations for gas sampling

6.2.1 Adsorption, reaction and permeation of sampling system

Such problems can be minimized by choosing sampling devices of suitable materials (see Clause 8 for details).

However, some slight adsorption is difficult to overcome. In this case, the sampling system should be heated or continuously purged for a long period of time. Quality assessment of the sampling system should be carried out according to 9.2. The adsorption shall be considered in the uncertainty budget.

6.2.2 Leaks and atmospheric diffusion in the sampling system

Leaks in the sampling system not only result in a loss of gas from the system but also allow air to diffuse into the system (the partial pressure of the component determines the direction of the diffusion) thereby affecting the composition of the sample.

The sampling system should be leak tested (see 6.2.3) prior to use to ensure the sample will not be contaminated, the composition changed, or hazardous conditions created by the ingress of air. Furthermore, the back-diffusion of air into gas venting lines should be avoided by, for example, using longer venting lines.

6.2.3 Leak testing of the sampling system

All connections and welds shall be tested prior to first use. During subsequent re-use of the sampling system, re-connected parts should be retested for leaks. Other parts of the system should be regularly retested, this is particularly important for corrosive gases. When sampling toxic gases, leak testing shall be performed before each use of the sampling system. The integrity of the sampling containers and their connection with the sampling system should also be tested.

The following test methods may be used.

a) Pressurization of the system, followed by monitoring of the static pressure with respect to time. A pressure drop indicates a leak.

b) Evacuation of the system and monitoring the vacuum achieved. A deterioration in the vacuum resulting in an increase in pressure indicates a leak.

c) Pressurize the system and check all connections with a leak detection solution. Following the use of leak detection solution, the system should be purged out to ensure dryness prior to use.

d) Use of a leak detector (e.g. mass spectrometry, where the system is filled with helium and the presence of helium outside the system is detected with the mass spectrometer).

The correct selection of a leak detection method depends upon the system requirements. For example, leak detection solution might not detect small leaks, however, instrumentation such as a leak detecting mass spectrometer having a high sensitivity can be used to determine the precise location of the leak(s). It is common practice to use a combination of the methods specified to establish the integrity of the sampling system, i.e. leak tightness.

Sampling systems for use in toxic gas service shall be thoroughly leak tested. A sensitive method of leak detection such as evacuation followed by the monitoring of the vacuum or the use of a leak detecting mass spectrometer should be used.

When leak detection solution as well as pressurization are used in the test, if pressurization by an inert gas is required, the molecular size of the chosen inert gas should not be larger than that of samples to be collected and the gas pressure shall not be less than the maximum filling pressure of the sample to be collected.

For samples to be collected which require the analysis of trace water or water-soluble components, leak detection solution cannot be used to avoid contamination of the sample.

6.2.4   Purging of the sampling system

6.2.4.1   Overview

To avoid contamination from previous samples or residual air in the sampling system, thorough purging before use should be completed. Inadequate purging can result in inaccurate results being obtained.

Commonly used methods of purging include:

— continuous purging;

— evacuation-gas purging cycles;

— fill-empty cycle purging.

A combination of these methods may be used depending upon the design of the sampling system and gas service.

For non-reactive components, 6.2.4.2 and 6.2.4.3 gives a detailed explanation of the methods used. Annex A gives examples of calculations. Purging cycles and the times for effective purging should be experimentally verified.

For reactive components, where adsorption can occur, more purging cycles or time is required.

6.2.4.2   Continuous purging method

Continuous purging is usually used for sampling systems with a small dead volume. It should be used in combination with fill-empty cycle purging or evacuation-gas purging cycles for systems with larger dead volumes.

The residual component concentration in the sampling system using continuous purging may be described by the mathematical model for exponential dilution.

There are two factors determining the component of interest amount fraction change with time in course of continuous purging: a decreasing amount due to removing residual analyte and an increasing amount due to incoming sample containing this component. The purging time may be estimated using the mathematical model shown in Formula (1):

$$x_t = x_0 e^{-\frac{Q}{V}t} + x_x \left(1 - e^{-\frac{Q}{V}t} \right) \tag{1}$$