INTERNATIONAL STANDARD

ISO 19230

First edition
2020-11

Gas analysis — Sampling guidelines

Analyse des gaz — Lignes directrices pour le prélèvement des échantillons

Reference number
ISO 19230:2020(E)

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Published in Switzerland

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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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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.

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.

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.

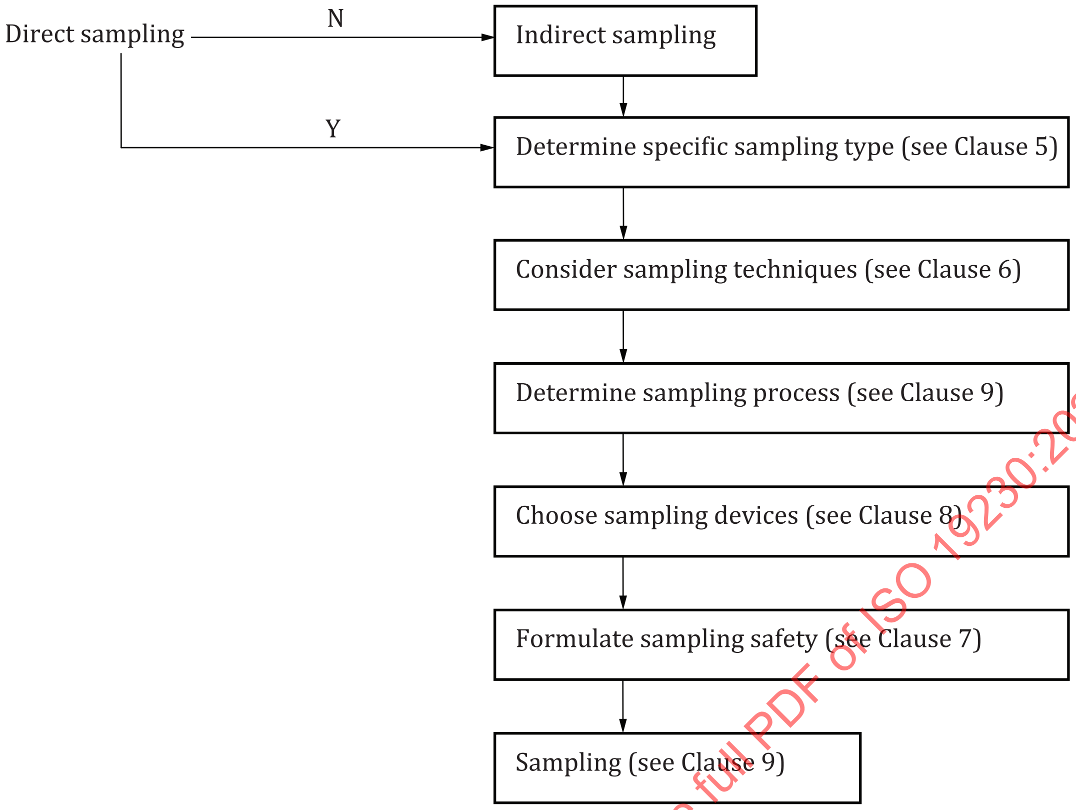


Figure 1 — Scheme of sampling plan

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.

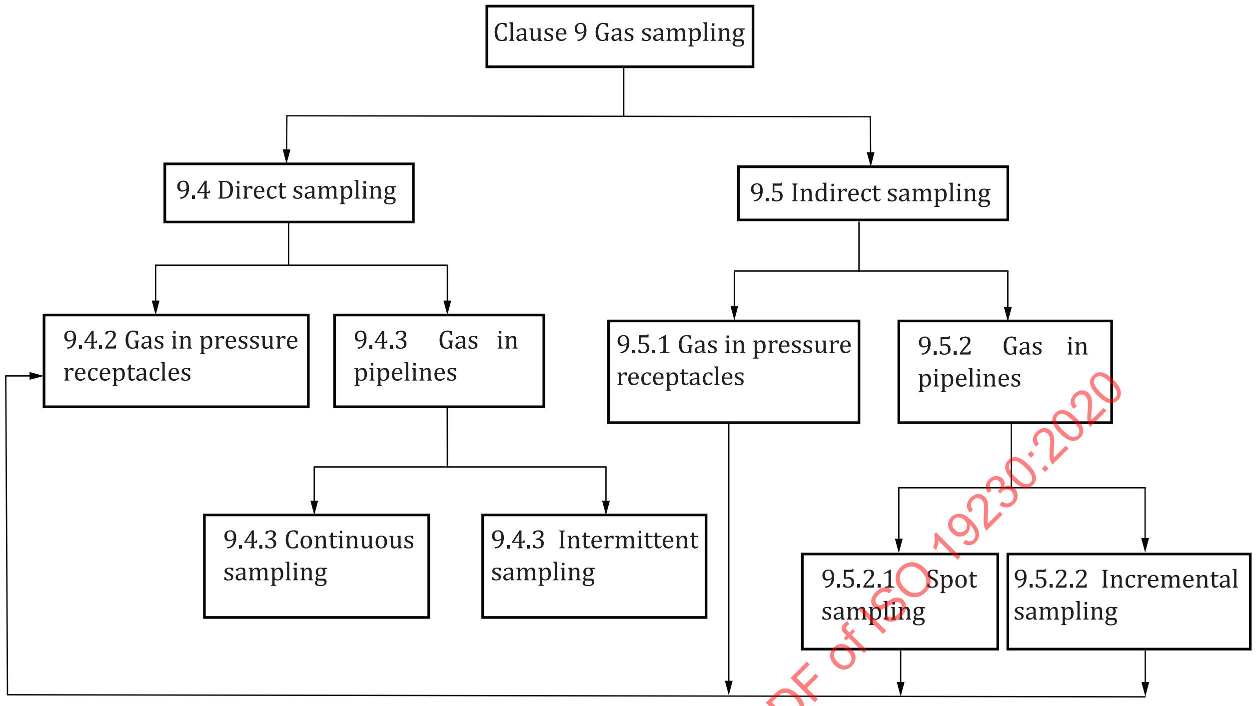


Figure 2 — Gas sampling classification

5.2 Sampling classification of liquefied gas

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.

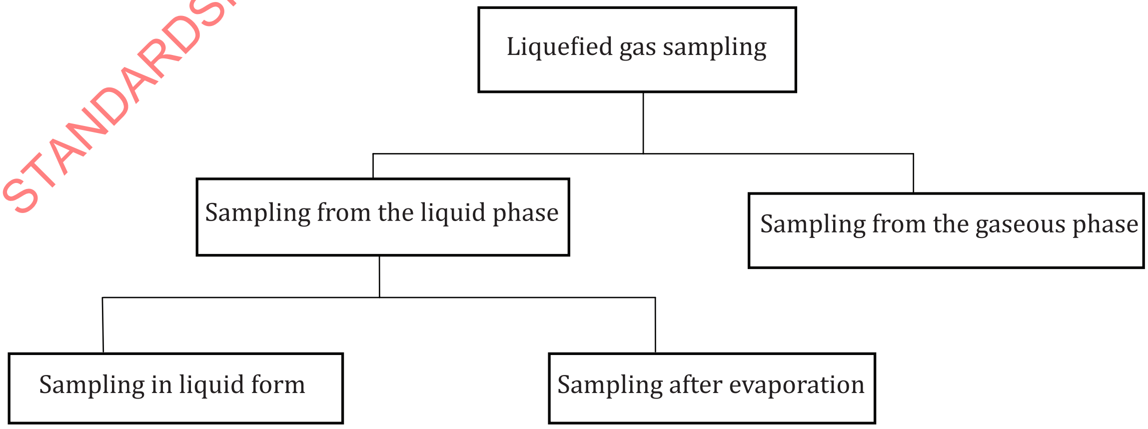


Figure 3 — Liquefied gas sampling classification

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):

$$x_{\mathrm{t}} = x_0 e^{-\frac{Q}{V}t} + x_{\mathrm{x}}\left(1 - e^{-\frac{Q}{V}t}\right) \quad (1)$$

where

x_t is a fraction of the component of interest in the sampling system after purging time t, in %;

x_0 is an initial fraction of the residual component in the sampling system, in %;

x_x is a fraction of the component in the gas the sample is taken from, in %;

Q is a continuous and constant purging flow rate of the sampling system, in ml/min;

V is a space volume of the sampling system to be purged, in ml;

t is purging time, in min.

A sampling relative error δ, %, is defined as in Formula (2):

$$\delta = \frac{x_t - x_x}{x_x} \times 100\ \% \tag{2}$$

and, by combining Formulae (1) and (2), we obtain Formula (3):

$$\delta = \left(\frac{x_0}{x_x} - 1 \right) e^{-\frac{Q}{V}t} \times 100\ \% \tag{3}$$

Formula (3) shows that when $x_0 > x_x$, δ is positive, when $x_0 < x_x$, δ is negative, and when $x_0 = x_x$, δ equals zero.

For a given sampling error, δ_g, minimal necessary purging time t_{min} may be evaluated as in Formula (4):

$$t_{min} = \frac{V}{Q} \ln \left[100\ \% \times \frac{\left(\frac{x_0}{x_x} - 1 \right)}{\delta_g} \right] \tag{4}$$

Formula (4) shows that, with other conditions being fixed, the closer ratio x_0/x_x is to 1, the shorter purging time is required. By increasing the flow rate of the purge gas and reducing the system volume, the purging time can be reduced.

Formula (4) is meaningful, i.e. gives positive value for t_{min}, when $|(x_0/x_x) - 1| > |\delta_g/100\ \%|$, i.e. when δ at $t = 0$ is greater than δ_g, otherwise a sampling error is acceptable without purging.

6.2.4.3 Fill-empty cycle purging method

Generally, valves, lines and sample containers have a certain dead volume, especially for pressure reducers and sample containers, so continuous purging is not very effective. Fill-empty cycle purging is more effective by sequentially pressurizing and venting the system with the sample to be taken.

Annex D gives some sampling procedure examples using fill-empty cycle purging method.

It is important to open the gas source valve only partially and only for a very short time (i.e. 0,5 s), both for safety reasons and in order to avoid back contamination.

Both the initial pressure and the maximum pressure are the same for each cycle of fill-empty purging. After n cycles of fill-empty cycle purging, the residual components fraction x_n in the sampling system may be derived according to Formula (5), the ideal gas under the isochoric conditions:

$$x_\mathrm{n} = x_0 \left(\frac{p_0}{p}\right)^n + x_\mathrm{x}\left[1 - \left(\frac{p_0}{p}\right)^n\right] \quad (5)$$

where

x_n is a fraction of the residual component in sampling system after purging cycle n, %;

x_0 is an initial fraction of the residual component in the sampling system, %;

x_x is a fraction of the component in the gas the sample is taken from, %;

p_0 is an initial absolute pressure of the sampling system;

p is the maximum absolute pressure achieved when the sampling system is pressurized;

n is a number of purging cycles;

p_0/p is a dilution ratio.

A sampling relative error δ, %, is defined as in Formula (6):

$$\delta = \frac{x_\mathrm{n} - x_\mathrm{x}}{x_\mathrm{x}} \times 100\ \% \quad (6)$$

and, by combining Formulae (5) and (6), we obtain Formula (7):

$$\delta = \left(\frac{x_0}{x_\mathrm{x}} - 1\right)\left(\frac{p_0}{p}\right)^n \times 100\ \% \quad (7)$$

For a given sampling error, δ_g, minimal necessary number of purging cycles n_min may be evaluated as in Formula (8):

$$n_\mathrm{min} = \frac{\ln\left[100\ \% \times \dfrac{\left(\dfrac{x_0}{x_x} - 1\right)}{\delta_\mathrm{g}}\right]}{\ln \dfrac{p}{p_0}} \quad (8)$$

Formula (8) shows that, with other conditions being fixed, the closer ratio x_0/x_x is to 1, the less number of purging cycles is required.

Formula (8) is meaningful, i.e. gives positive value for n_min, when $|(x_0/x_x) - 1| > |\delta_\mathrm{g}/100\ \%|$, i.e. when δ at $n = 0$ is greater than δ_g, otherwise a sampling error is acceptable without purging.

6.2.4.4 Evacuation-gas purging cycles

Evacuation-gas purging cycles are more suitable for gas samples with a limited available sample volume and those that are toxic, expensive, ultra-pure, sensitive to oxygen and water, and with pressure equal to or lower than atmospheric pressure.

Make sure that the pressure reducers used are suited for evacuation and that the purging cycle starts with evacuation. And it is also important to open the gas source valve only partially and only for a very short time (i.e. 0,5 s).

B.1.1 gives a sampling procedure example using evacuation-gas purging cycles.

6.2.5 Homogeneity of gas

Inhomogeneity of the gas will result in sampling errors.

Liquefied gas is not effectively or quickly mixed by diffusion. If the sample in the receptacle is not homogeneous, it shall be homogenized before sampling.

Gas stratification across pipelines usually occurs at low flow velocities with large pipeline diameters. Turbulence caused by bends or obstructions is beneficial for gas mixing, and it is advantageous to sample downstream of a source of turbulence. Sampling at gas stagnation points should be avoided. In addition, gas stratification can be investigated with the aid of an adjustable sample probe that collects samples at different points in the pipeline cross section. Based on the results of these tests, the best sampling position representative of the average composition of the sample is determined, generally positioned one-third to halfway into the pipeline.

6.2.6 Inert-gas purging

For condensable, sensitive to oxygen and water, or toxic and corrosive gas sampling, inert-gas purging should be used (see Figure C.1). For gases that can condense, if condensation occurs due to some unexpected factors, inert-gas purging is required after sampling to remove any residual sample from the system before taking the next sample. For gases that are sensitive to oxygen and water, inert-gas purging is required before sampling to eliminate residual moisture and air (if a vacuum pump is available, inert-gas purging is not necessary). For toxic and corrosive gas sampling, inert-gas purging is used primarily for the purging of sampling system after sampling.

Inert-gas purging should be introduced downstream of the pressure reducer.

NOTE The inert gas mentioned in this document refers to gases that are not active and do not interfere with sampling, for example nitrogen, helium and argon are commonly used.

6.3 Possible condensation during compressed gas sampling

For the sampling of condensable gases, condensation is one of the main factors that can affect the collection of representative samples. Any of the following factors can cause condensation.

- Pressure and flow regulation by a pressure reducer or flow controller (needle valve, mass flow regulator, capillary, etc.) can cause a temperature decrease due to Joule-Thomson cooling.
- The venting process in the sample line and container through the fill-empty cycle purging method can cause condensation due to loss of pressure and flow rate.
- When sampling from a receptacle, condensation can occur inside the receptacle due to adiabatic expansion.

To avoid the possible condensation mentioned above, the pressure drop across the flow regulator should be minimized. Flow characteristics of a flow regulator are usually given by the manufacturer, and the information is sufficient to judge whether the required flow rate can be controlled by the chosen flow regulator. When calculating the pressure drop, tube sizing (inside diameter and length) shall also be considered.

Using more than one pressure reducer to drop the pressure in stages, adding flow (or pressure) control and/or indicator devices on venting lines, or adding an extension tube (see Figure D.1, typically 0,6 m to 1,2 m in length) to the outlet of sample containers and analysers, are advantageous to minimize the risk of condensation.

Heating the sampling system is the most effective method. In the case of decompression, it should be heated from upstream of the pressure reducer. In the case of indirect sampling, the sample container also needs to be heated for transport and storage. The temperature should ensure that no condensation of the condensable components is expected to occur. The required temperature depends on the gas composition, drop of pressure, pressure, temperature and flow rate, etc.

6.4 Main considerations for liquefied gas sampling

When sampling liquefied gas after evaporation, it should be noted that if the liquid cannot be completely vaporized, a representative sample cannot be collected.

For high pressure liquefied gases, the ambient temperature is sufficient to allow uniform evaporation. For low pressure liquefied gases, suitable vaporizers, such as water baths, flashers, etc., should be used to ensure the uniform and complete evaporation of all components.

During the process of sampling liquid directly, after the liquid sample is flowing out of the sample line, the pressure in the line should always be controlled to a higher pressure than the saturated vapour pressure of the sample so as to avoid partial evaporation of the components.

6.5 Samples that are not feasible in containers or cannot be used for analysis directly

If suitable sampling vessels are not available or direct sampling is not feasible, sorption tubes or impingers should be considered for sampling.

7 Safety guidance in sampling

7.1 Overview

Before taking samples, a risk assessment shall be performed to ensure that hazardous conditions are not created, and any potentially dangerous conditions are recognized and mitigated. This chapter is intended to provide safety warnings for gas sampling, subsequent sample preservation and transportation. Because of the complexity and diversity of the actual situation, only general guidance is given in this document. Users should be aware of all safety precautions to ensure sampling safety and perform risk assessments prior to beginning sampling.

7.2 General recommendation

Generally, gas sampling shall be in accordance with the general recommendation given in ISO 3165 and the following.

a) Sampling personnel: The sampling personnel should be professionally trained, familiar with the nature of the sample to be collected, as well as any possible hazards involved, the protective measures and the precautions to be taken when sampling, and the actions and protective measures that should be taken in an emergency.

b) Sampling operation: Sampling should be performed strictly in accordance with the documented operating procedures. The nature of the material and the risks associated with it should be marked on the sample container. The sample container may be disconnected from the sampling system after the pressure is released to vacuum for toxic and hazardous samples or to atmospheric pressure for non-hazardous samples.

c) Sampling device: The sampling device should meet the conditions of the sample to be collected, such as temperature, pressure, flow rate, thermal expansion, etc. Sample containers for use under pressure should have been pressure-tested, and the maximum safe operating pressure should be marked on the container. The sample container used for liquefied gas sampling in liquid form should have a suitable reserved volume, and the position of the reserved tube [see Figure 5 c) and d)] and the desired fill level (see Figure 6) should be marked on the container and should not be overfilled. The method of connecting the sampling device should be compatible with the sampling pressure and sealing requirements.

d) Sampling system: The sampling system should have the necessary decompression, flow control, pressure relief, and venting gas (including the purging and ullaging venting gas) treatment or evacuation devices to avoid environment pollution or personal injury. The insertion of a shut-off valve after the pressure reducer (see Figure B.1, B.2 and C.1) is recommended.

e) Sampling site: The sampling site should have safe access to and from the place where the sample is taken, adequate light and ventilation as well as the necessary safety facilities (such as leakage alarm and explosion-proof safety measures when performing flammable gas and toxic gas sampling).

f) Preservation and transportation of samples: Precautions should be taken to protect the integrity of the container in storage and transportation. For example, the inlet and outlet of the sample container shall have suitable protective equipment (such as protective caps or valves, etc.), the samples should be kept away from heat (the liquefied gas sample container should always be kept cool) and exposure to sunlight.

7.3 Specific recommendation for sampling a certain substance

Sampling can potentially involve multiple hazards and for specific substances, safety when sampling shall be in accordance with the requirements detailed for hazardous materials given in ISO 3165.

8 Sampling devices

8.1 General provision

The main devices that comprise gas sampling systems include sample probe, sample line, sample container, sample pump, pressure reducer, flow controller, connectors and so on.

Depending on the sample to be taken, for example, the nature, the component to be determined, storage or transportation methods, composition, temperature, pressure, sampling error required and other factors, select appropriate sampling devices. The general principles are no infiltration, minimum adsorption, no reaction between sample and materials of sampling devices, and the materials used cannot catalyse reactions between the components in the sample. Also, the sampling device is required to be able to withstand the temperature and pressure of sampling, easily form leak-free connections with each other, no risk of explosion, corrosion, etc. The higher adsorption, toxicity, corrosivity and the lower the concentration of the assayed component and/or the gas to be collected give rise to the higher requirement for the sampling devices.

ISO 16664 and ISO 11114 (all parts)[11] specify material compatibility for some components and materials. When collecting the active components, the suitability of the sampling devices should be verified by proper methods. For example, after the gas has been continuously passed through a sampling system, the variation in concentration of the gas continuously sampled is observed. Concentration changes of the gas compared with expected concentration is monitored to test the suitability of sample container.

To prevent cross-contamination during sampling, devices should not be mixed while in use.

8.2 Sample container

8.2.1 Sample container material

8.2.1.1 Metal containers

Materials of metal containers commonly used include stainless steel, carbon steel, and aluminium alloy. When collecting active and trace components of gas samples, gas cylinders with special treatment of the inner wall (such as polishing, passivation, coating, etc.) are usually required. Samples with different components or different concentrations of the same components can require different treatments.

When selecting sample containers, the material of the valve and valve core, which might be in direct contact with the sample, should also be considered. Commonly used metal container valves are made of stainless steel and copper and the valve cores are made of stainless steel, polymer, rubber, nylon, etc.

For high-pressure gas, high and ultra-purity gases, corrosive gas, toxic gas, liquefied gas, large gas volumes and the detection of trace oxygen, nitrogen, water, carbon dioxide, and samples sensitive to oxygen and moisture, metal sample containers are recommended.

Material of the sample container for liquefied gas should also be able to withstand rapid changes in temperature.

8.2.1.2 Non-metallic containers

8.2.1.2.1 General provision

For gas sampling at atmospheric, sub-atmospheric and low-pressure, non-metallic sample containers may be used. But because of the poor pressure and temperature resistance and leak tightness, non-metallic sample containers are not suitable for situations where metal sample containers are required as described in 8.2.1.1 and the collection of samples with high temperature are required.

8.2.1.2.2 Glass containers

Glass containers have good chemical inertness and if necessary, the inner wall of these containers could be treated (such as silanization, etc.) to reduce any absorption.

Glass containers sealed with piston oil are not suitable for accurate sampling of organic components, because organic materials can partially dissolve in the piston oil. And, in future sampling, the organic matter can volatilize to contaminate the sample. If glass containers have to be used, other methods of sealing should be chosen.

Glass syringes and glass sample tubes are common glass sample containers.

There are two common forms of glass sample tube, dual straight stopcock sample tube [see Figure 4 a)] and double oblique bore stopcock sample tube [see Figure 4 b)].

a)   b)

Figure 4 — Diagram of glass sample tube

8.2.1.2.3 Composite membrane airbag

Composite membrane airbags are better than rubber airbags with respect to gas adsorption and permeability (see 8.2.1.2.5). The airbags produced by different manufacturers can have different kinds and concentrations of organic residue present. Therefore, before sampling for trace organic species, the air bags should be tested to determine whether they are satisfactory for their intended use.

8.2.1.2.4 Polymer airbags

Common polymer airbags are made of polyethylene, polyvinyl fluoride, polypropylene, polyester or copolymer, etc. Compared to composite membrane airbags, polymer airbags have better inertness and might be suitable for sampling of trace active components.

In all cases, the loss of component due to adsorption and diffusion shall be studied and quantified in the uncertainty budget.

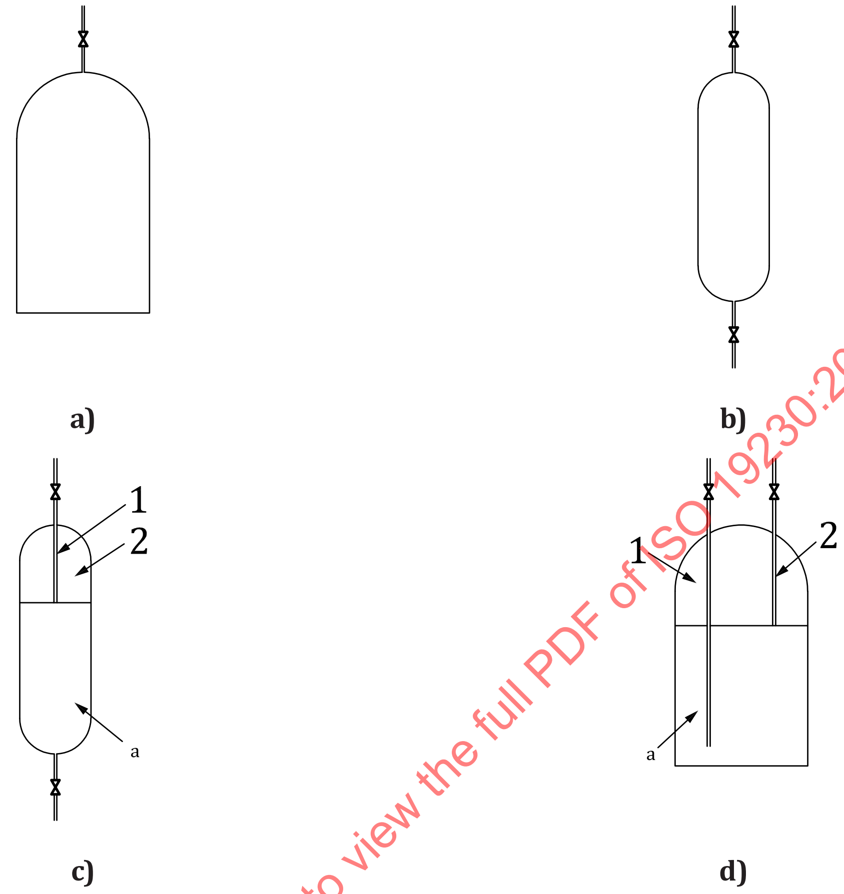
8.2.1.2.5 Rubber airbag

Rubber airbags are cheap and convenient to use but have strong permeability and adsorption for most gaseous components, which can cause sampling errors and safety hazards. Therefore, they are not recommended.

8.2.2 Structure of sample container

8.2.2.1 Commonly used sample containers

Commonly used sample containers for compressed gas, high pressure liquefied gas and low pressure liquefied gas are usually divided into single-valve container [see Figure 5 a), such as syringes, etc.] and dual-valve container [see Figure 5 b), c) and d)] according to the structure. Figure 5 c), d) apply to liquid sampling of high pressure liquefied gas and low pressure liquefied gas, and Figure 5 d) applies to sampling of toxic liquefied gas.



Key

1 reserved tube

2 reserved volume of the container

a The maximum volume may be filled with sample.

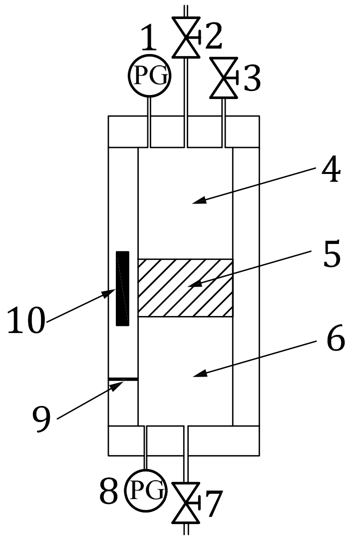
Figure 5 — Structure of sample container for gases

The longer tube of container d) should be inserted near the bottom of the cylinder, and the shorter one of container d) and the tube of container c) should be sized to ensure that the filling ratio (or reserved volume) of the container is kept.

Other configurations (such as a rupture disk on metal sample container), except for valves of the sample containers, are not shown here.

8.2.2.2 Floating piston cylinders

A typical structure of the floating piston cylinder is shown in Figure 6.



Key

1	sample gauge
2	sample valve
3	purge valve
4	sample
5	piston
6	precharge gas
7	precharge gas valve
8	precharge gas gauge
9	desired fill level
10	piston indicator

Figure 6 — Floating piston cylinder

Only some key configurations indicate the structure of the floating piston cylinder are shown in Figure 6.

The floating piston cylinder is designed to collect liquid samples with no vaporization by displacing a piston against a pressurizing fluid (precharge gas). The piston serves as a physical barrier between the sample and the precharge gas, at the sampling pressure. The position of the piston at the end of sampling indicates the percent fill of the sample container.

8.2.3 Volume of sample container

The volume of the sample container depends on the amount of sample required for all analyses to be performed (sometimes it can be necessary to collect multiple samples at the same time, such as sampling with a suitable cylinder that satisfies the sampling requirements for the different components). For the liquid sample volume of the liquefied gas, the volatilization of the liquid phase in the container after sampling should also be considered.

8.3 Sample probe

For sampling gas in pipelines, especially for larger size pipelines, the wall of the pipeline can adsorb some components. In addition, due to the high-speed flow of gas in the pipe, liquid impurities can accumulate at the inner wall of the pipeline. Without the use of a sample probe, the sample taken can contain too much water and other condensable and adsorption components, which are not representative of the gas in the pipeline.

In addition to meeting the general requirements in 8.1, the sample probe should also be easy to process and have good mechanical properties.

In general, for sampling gases containing particulate matter, a sample probe with a filter should be used. It is recommended that the sample probe itself has a replaceable filter and a backflush function to avoid the probe being blocked. For high-temperature gas sampling, sample probes with suitable temperature resistant materials should be selected, and cooling measures (e.g. air or water jacket) should be considered, especially for flammable gas sampling to reduce the possibility of chemical reaction during sampling. When collecting condensable gas, sample probes with heating should be selected.

8.4 Pressure reducer and flow controller

If the gas pressure of the sample to be collected is much higher than atmospheric pressure, it is recommended that the pressure be reduced by pressure reducers and/or flow controllers (such as needle valves, mass -flow controllers, capillaries, etc.) to a safe pressure and flow rate before sampling. When performing direct sampling, the pressure and flow rate of gas going through the analyser should be sufficient to meet the requirements of the analyser.

When selecting the regulating devices, the compatibility between materials and samples to be collected should be determined by the nature of the gas first. And then choose the regulating device according to the gas flow, the input and output pressure, the possible maximum pressure in gas sampling systems and the requirements of sampling temperature.

The material commonly used for regulators is stainless steel or copper, and the diaphragm material includes stainless steel or polychloroprene, etc. Both the valve body and diaphragm should be considered when selecting materials.

For example, for sampling a gas containing traces of absorbable components as well as oxygen, nitrogen, water, the dead volume of the regulating device chosen should be as small as possible. A fine control needle valve may be used in these situations, however the precaution of adding a safety valve between the needle valve and the analyser, should be taken so that a potential pressure increase in the system does not create a hazard.

8.5 Sample pump

Many types of pump are available, for example a diaphragm pump, a metal bellows pump, an ejection pump, etc. The choice depends upon the scale and duration of sampling operations, the facilities available on the site, and also upon the flow rate or degree of vacuum required.

8.6 Sample line

8.6.1 Material of sample line

The sample line might be a stainless-steel line, carbon steel line, copper line, aluminium line, metal hose, glass tube, quartz tube, polytetrafluoroethylene tube, polyethylene, rubber tubes and so on.

Where metal sample containers are required as described in 8.2.1.1, metal lines are preferred.

When using non-metallic sample lines, it is also important to consider their permeability by certain components and water vapour. For example, PTFE is extremely inert, but it is highly permeable to water vapour and therefore not suitable for the sampling of water-soluble gases.

Polymer lines are usually used only for short connections. If longer connections are required, experiments should be conducted to prove that the samples to be collected are not subject to adsorption or contamination due to permeation.

Due to the strong adsorption and permeability of rubber tubes to many components, rubber tubes are usually used for short sleeve connection only. To avoid the contact between rubber tubes and gas, two lines of the pipeline should be closely butted, and the interface should be fixed outside with a suitable diameter of the rubber sleeve.

For the sampling of trace and active components, if necessary, the inner surface of the line should be treated, by passivation, coating and polishing to minimize the adsorption and the purging time.

8.6.2 Length and diameter of sample line

The diameter of the tube should be selected to optimize sampling. A large diameter is advantageous for the rapid purging of a large number of samples. However, in view of laminar flow and rapid purging, a small diameter is more appropriate. For trace active components, oxygen, nitrogen, water, carbon dioxide and gases sensitive to oxygen and water, the diameter should be as small as possible.

The sample lines should be as short as possible. If long lines cannot be avoided, extending the purging time for the sampling of gas in pressure receptacles, and using a high flow bypass line helps to reduce the lag time for sampling of gas in pipelines (see Figure C.1).

8.7 Connecters and seals

Connector materials commonly used are stainless steel and copper. Seals include metal materials (such as copper and stainless steel) and non-metallic materials (such as rubber, etc.). Where metal sample containers are required as described in 8.2.1.1, metal seals are preferred.

When sampling trace active components particular care should also be taken with the selection of connectors and seals that come into direct contact with gases.

Try to avoid using dissimilar metals in contact with each other, as it can cause increased rates of corrosion and result in sampling errors or safety problems.

8.8 Cleaning and drying of the sampling device

Lubricants, grease, slag particles and other contaminants might be present in the sample container and sample lines and seals, which should be thoroughly cleaned using proper volatile solvents, then dried and the solvent residue in the container tested for prior to use. For a coated or passivated sampling device, the appropriate cleaning method should be selected after discussions with the manufacturer.

8.9 Connection of sampling devices

There are many kinds of connections between sampling devices, such as ferrule connections, connections with metal gaskets, welding, tower head-hose connections, quick connector connections and so on.

First, the connection is selected based on safety considerations. For example, metal lines and seals for connection or welding are recommended if long lines are used. For the determination and sampling of toxic and ultrapure gases is recommended that welded or connections with metal gaskets are used. High-pressure gas and liquefied gas sampling should use ferrule connection (the seals should be made of metal), welding, and special quick connector or connector with metal gasket. Tower head-hose connections and ferrule connections with non-metallic seals are only suitable for low-pressure gas sampling.

Second, the requirements for purging and the convenience of use should be considered. For example, a connector with metal gasket has been electropolished and cleaned and is provided with high leak tightness and no dead volume and is therefore especially suitable for ultrapure and toxic gas sampling that have strict sealing requirements.. Quick connector connections might connect or disconnect the sample lines without any tools, which is durable and especially suitable for applications requiring repeated disassembly, such as frequent indirect sampling. There are different types of quick connectors to choose from, depending on the type of gas sample to be taken.

9 Sampling

9.1 Sampling method block diagram

9.1.1 Overview

To facilitate the use of this document, based on the state of the gases (compressed gases and liquefied gases) and the classification of sampling in Clause 5, specific sampling methods of different gases are given in block diagrams in this section, allowing users to quickly select the appropriate sampling methods for the different gases.

In the block diagrams, only the more appropriate sampling method is given for each gas, and other methods might be used. For example, the sampling of high-pressure compressed gas may also be taken with a vacuum pump.

9.1.2 Block diagram of compressed gas sampling method

The block diagram of compressed gas sampling method is shown in Figure 7.

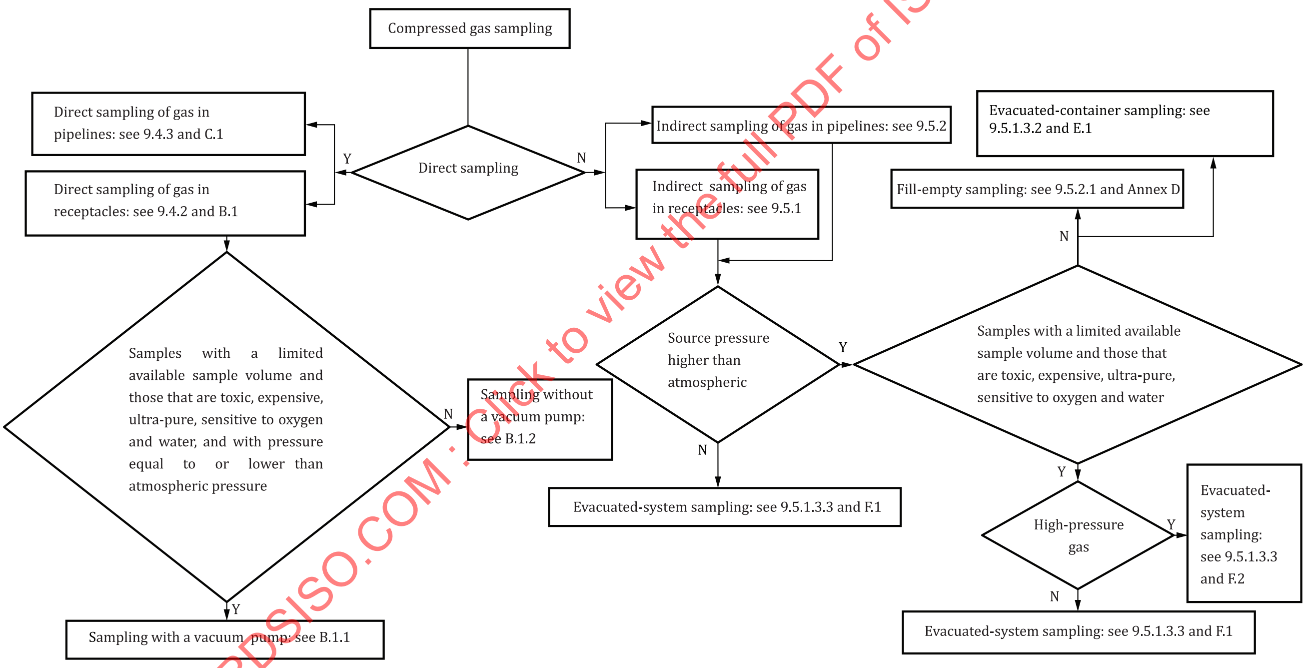


Figure 7 — Block diagram of compressed gas sampling method

9.1.3 Block diagram of liquefied gas sampling method

The block diagram of liquefied gas sampling method is shown in Figure 8.

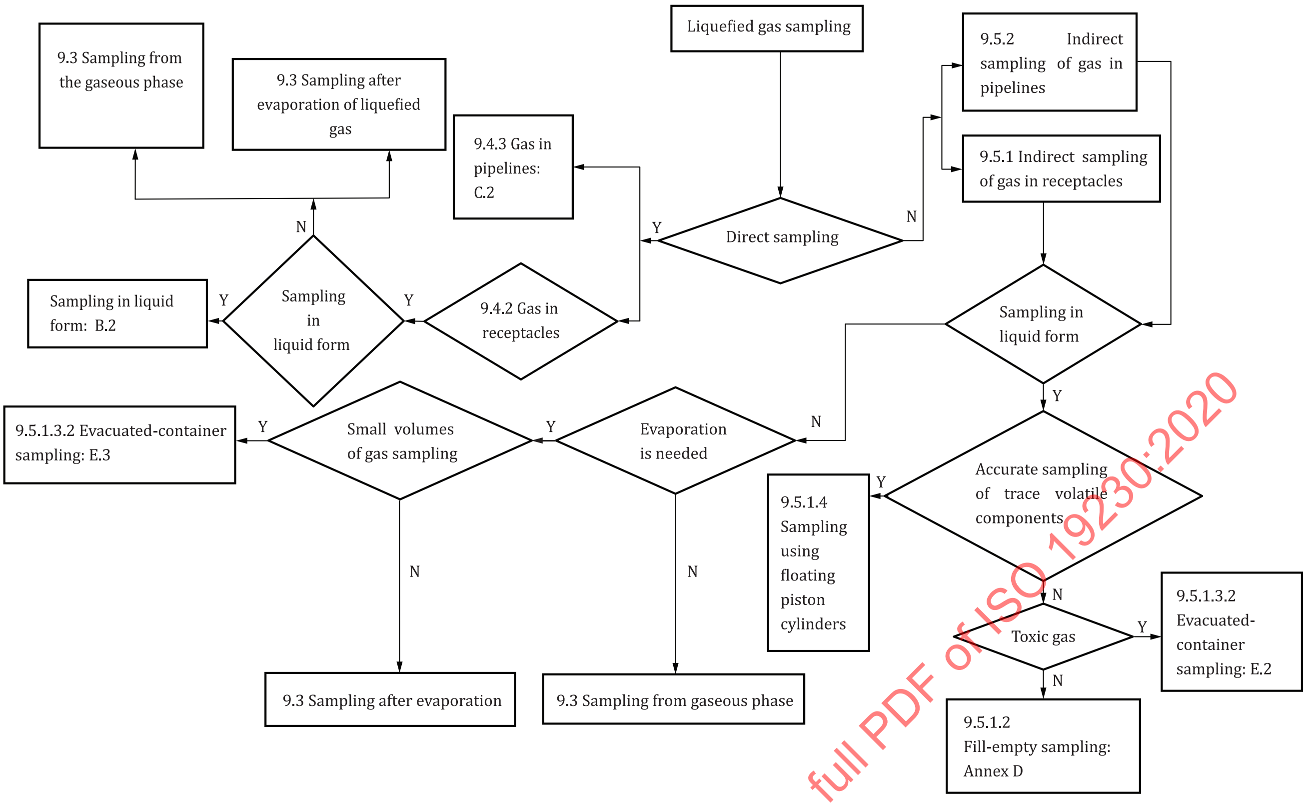


Figure 8 — Block diagram of liquefied gas sampling method

9.2 Quality assessment of the sampling system

To test the integrity of the sampling system, the assessment of the sampling system should be done before the first use.

The assessment of a direct sampling system shall be in accordance with ISO 16664 while an indirect sampling system shall be validated using a gas standard of known composition followed by analysis. The consistency of the analysis results, when compared with the concentration of the standard, demonstrates the integrity of the sampling system.

9.3 Sampling from the gaseous phase and sampling after evaporation of liquefied gas

If a vaporizer is required (for sampling after the evaporation of a liquefied gas), it should be installed after the sample probe (for gas in pipelines) or sample container outlet valve (for gas in pressure receptacles). If decompression is required, the pressure reducer should be installed at a suitable position after the vaporizer, or a vaporization pressure reducer may be used.

Whether for direct or indirect sampling, sampling of gas in pipelines or in pressure receptacles, the sampling method and flow chart of liquefied gas after evaporation are the same as that of the corresponding condensable compressed gas (for example, the direct sampling method after evaporation of liquefied gas in pipelines is the same as the direct sampling method of condensable compressed gas in pipelines).

The method and flow chart of the sampling in the gaseous phase of liquefied gas are the same as that of the corresponding condensable compressed gas.

9.4 Direct sampling

9.4.1 General provisions

A direct sampling system should ensure that the sampling repeatability meets the expected requirements.

If a calibration gas is used, it should be introduced into the analyser under the same conditions as the sample gas. Measures should be taken to ensure that there is no interference between the calibration gas and the sample.

To ensure the repeatability of the direct sampling system, the flowmeter (as shown in Figure B.2) or pressure gauge should be specified in the sampling system depending upon the requirements for the analyser. In this way, a constant flow or pressure shall be maintained for all sampling.

It is recommended the flowmeter is placed in the outlet of the analyser for sampling in pressure receptacles (as shown in Figure B.2) for rapid purging.

For direct sampling, the design of the sample line also depends on how the sample is introduced into the analyser. There are usually two methods of sample introduction, either using extractive analysers or non-extractive analysers. The flow chart in this standard is based on a system using non-extractive analysers. For extractive analysers, an excess-flow line shall be included in the sampling system to prevent over pressurization of the analyser and sampling pump. The excess-flow line shall be located at the end of the sample line and before the analyser in a T orientation.

9.4.2 Direct sampling of gas in pressure receptacles

Examples of direct sampling methods and flow chart of gas in pressure receptacles are shown in Annex B.

For compressed gas sampling, depending on whether a vacuum pump is available, there is sampling with and without a vacuum pump. In general, for the gases suitable for evacuation-gas purging cycles as described in 6.2.4.4, sampling with a vacuum pump is more suitable. For other gases, either sampling method may be used.

In addition, sampling with a vacuum pump or without a vacuum pump also depends on the design of analyser.

9.4.3 Direct sampling of gas in pipelines

For most direct sampling of gas in pipelines, only a small proportion of gas enters the analyser, and the remainder should be diverted in the bypass line and returned to the main pipeline (as shown in Figure C.1) or vented to a safe location.

Annex C gives detailed examples of direct sampling (continuous sampling and intermittent sampling).

9.5 Indirect sampling

9.5.1 Indirect sampling of gas in pressure receptacles

9.5.1.1 General

There are two methods for indirect sampling of gas in pressure receptacles, namely fill-empty sampling and evacuation sampling.

9.5.1.2 Fill-empty sampling

Fill-empty sampling is mainly based on the fill-empty cycle purging of the sampling system, which is suitable for compressed gas sampling where the source pressure is higher than the atmospheric pressure and for liquefied gas sampling in the liquid phase with a double-valve container as shown in Figure 5 c). Special attention should be paid to gases with a pressure only slightly higher than the atmospheric pressure. This is because, if the temperature of the source gas is high, the temperature of the sample line and container shall be such that the gas pressure is still higher than the atmospheric pressure after cooling, to prevent air ingress into the samples.

For compressed gas sampling, sample containers should be double-valve as shown in Figure 5 b) to enable effective purging.

Annex D gives detailed examples of this method.

9.5.1.3 Evacuation sampling

9.5.1.3.1 Overview

Evacuation sampling is mainly based on the purging of the sampling system by evacuation-gas purging cycles, this mainly includes evacuating the sample container only (evacuated-container sampling method) and evacuating the entire sampling system (evacuated-system sampling method).

9.5.1.3.2 Evacuated-container sampling

In this method, the sample container is evacuated to a suitable pressure that does not interfere the sample to be collected before connected to the sampling system.

This method is suitable for high-pressure and low-pressure compressed gases sampling, toxic liquefied gas sampling in liquid form using a double-valve container shown in Figure 5 d), as well as small amounts of liquefied gas sampling after evaporation. The sample container for compressed gas sampling and small volumes of liquefied gas sampling after evaporation usually has a single valve structure, however a double valve structure is also applicable.

Annex E gives detailed examples of this method.

9.5.1.3.3 Evacuated-system sampling

This method is not limited by the pressure of the gas source and is especially suitable for sampling of gases described in 6.2.4.4.

Detailed examples of this method are shown in Annex F.

9.5.1.4 Sampling using floating piston cylinders

The use of floating piston cylinders is encouraged to minimize or eliminate the volatilization of volatile components into the headspace. It gives a highly accurate sampling of low pressure liquefied gas that might contain trace volatile components, such as ethane. In addition, it is recommended whenever highly accurate determination of light components is required.

Detailed examples of this method are shown in Annex G.

9.5.2 Indirect sampling of gas in pipelines

9.5.2.1 Spot sampling

The method of spot sampling is the same as indirect sampling of gas in pressure receptacles (see 9.5.1).

9.5.2.2 Incremental sampling

Incremental sampling is mainly used for gas sampling in pipelines where the sample composition changes significantly with time.

The key to effective incremental sampling is setting the sampling time and interval appropriately and keeping a constant flow rate during sampling to ensure the collection of representative samples.

Incremental sampling may be controlled manually or automatically by a commercial incremental sampler.

The method of incremental sampling is the same as indirect sampling of gas in pressure receptacles (see 9.5.1).

9.5.3 Leakage test of sample container

After the sampling is completed, the sample container should be tested for leaks by a suitable method, such as using the leak detector or leak detection liquid described in 6.2.3, or by immersing the container in water. If a leak is detected, discard the sample, repair or replace the sample container, and resample.

After the test is completed, the inlet and outlet of the container should be capped immediately to prevent contamination.

9.5.4 Storage of samples

Analysis should be completed as soon as possible after sampling, and there should be experimental data to prove that the required storage period does not cause changes in the composition of the sample. The factors that can cause changes in the composition of the sample include the adsorption and permeation related to the sample container, the reaction of the components in the sample under the storage conditions, etc.

9.6 Sampling records

Sample records should include at least the following:

- the sampling method;
- the sample description;
- the sampling container number;
- the sampling date and time;
- the sampling point details;
- the sampling pressure or volume;
- the gas source temperature;
- the sampling personnel;
- the sampling conditions;
- any abnormal conditions found during sampling.

Annex A
(informative)

Examples of estimation of the purging time and purging cycles for sampling system

A.1 Examples of estimation of the purging time needed for continuous purging

EXAMPLE 1 The residual sample contains a component to be determined.

To sample and detect O_2 and N_2 with both mole fraction x_x of 0,001 % in H_2. The purging flow rate Q is 50 ml/min, purging volume V is 20 ml, the required sampling error δ is 10 %, the sampling system is exposed to air with oxygen/nitrogen mole fraction $x_{0\text{-O2}}/x_{0\text{-N2}}$ are approximately 21 % and 79 % respectively. The time t for continuous purging is estimated using Formulae (A.1) and (A.2) according to Formula (4):

$$t_{O_2} = \frac{20}{50}\,\text{min}\,\ln\left[100\,\% \times \frac{\left(\dfrac{21}{0{,}001}-1\right)}{10\,\%}\right] = 4{,}90\,\text{min} \tag{A.1}$$

$$t_{N_2} = \frac{20}{50}\,\text{min}\,\ln\left[100\,\% \times \frac{\left(\dfrac{79}{0{,}001}-1\right)}{10\,\%}\right] = 5{,}43\,\text{min} \tag{A.2}$$

The reference purging time is subject to the larger one (nitrogen in this example).

EXAMPLE 2 The residual sample does not contain any component to be determined.

To sample and detect CO with mole fraction x_x of 0,001 % in H_2. The purging flow rate Q is 50 mL/min, purging volume V is 20 ml, the required sampling error δ is 10 %.

The residual sample does not contain the component to be determined (CO), so x_0 is 0, $x_0 < x_x$, δ is negative, that is −10 %. The time t for continuous purging is estimated using Formula (A.3) according to Formula (4):

$$t = \frac{20}{50}\,\text{min}\,\ln\left[100\,\% \times \frac{\left(\dfrac{0}{0{,}001}-1\right)}{-10\,\%}\right] = 0{,}92\,\text{min} \tag{A.3}$$

A.2 Example of estimation of the purging cycles needed for fill-empty cycle purging

The initial pressure p_0 is 0,1 MPa, the maximum pressure p is 10 MPa in purging, and the initial mole fraction of oxygen in the sampling system $x_{0\text{-O2}}$ is 21 %. If the mole fraction of the component to be determined x_x is 0,001 %, and the sampling error is 2 %, the purging cycles estimation process is as shown in Formula (A.4) according to Formula (8):

$$n_{\min} = \frac{\ln\left[100\,\% \times \dfrac{\left(\dfrac{21}{0{,}001}-1\right)}{2\,\%}\right]}{\ln\dfrac{10}{0{,}1}} = 3{,}01 \tag{A.4}$$

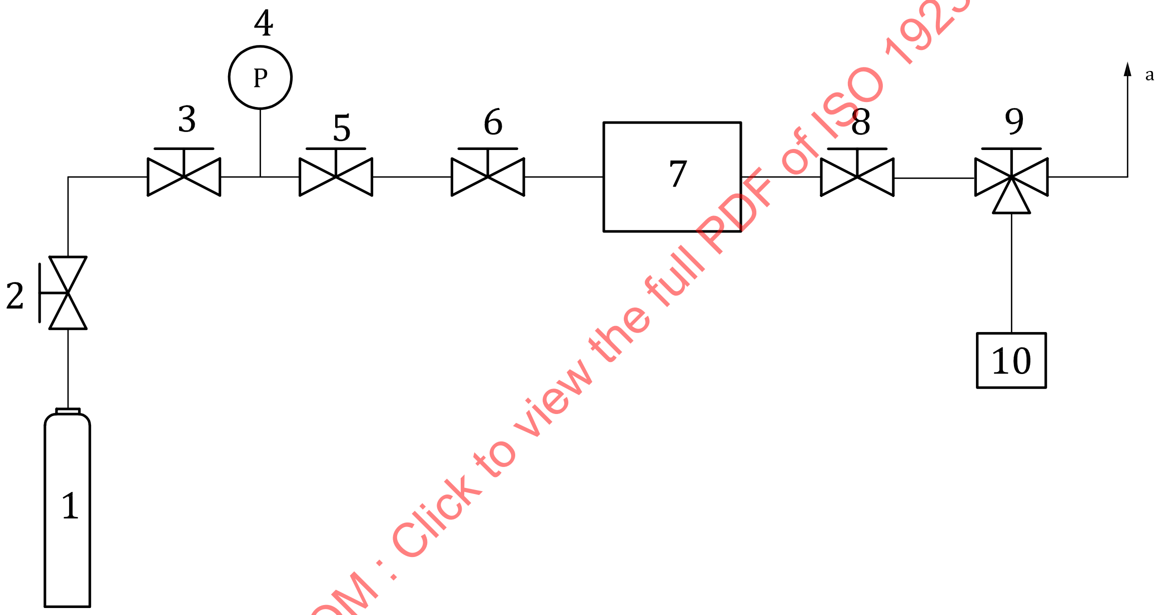
Annex B
(informative)

Direct sampling for gas in pressure receptacles

B.1 Direct sampling for compressed gas in pressure receptacles

B.1.1 Sampling with a vacuum pump

If a vacuum pump is available, the sampling process is shown in Figure B.1.



Key

1	storage container
2	outlet valve of storage container (gas source valve)
3	pressure reducer
4	pressure gauge
5	flow controller
6	shut-off valve
7	analyser
8	shut-off valve
9	three-way valve
10	vacuum pump
a	Vent.

Figure B.1 — Flow chart of direct sampling with a vacuum pump for gas in pressure receptacles

The sampling method is as follows.

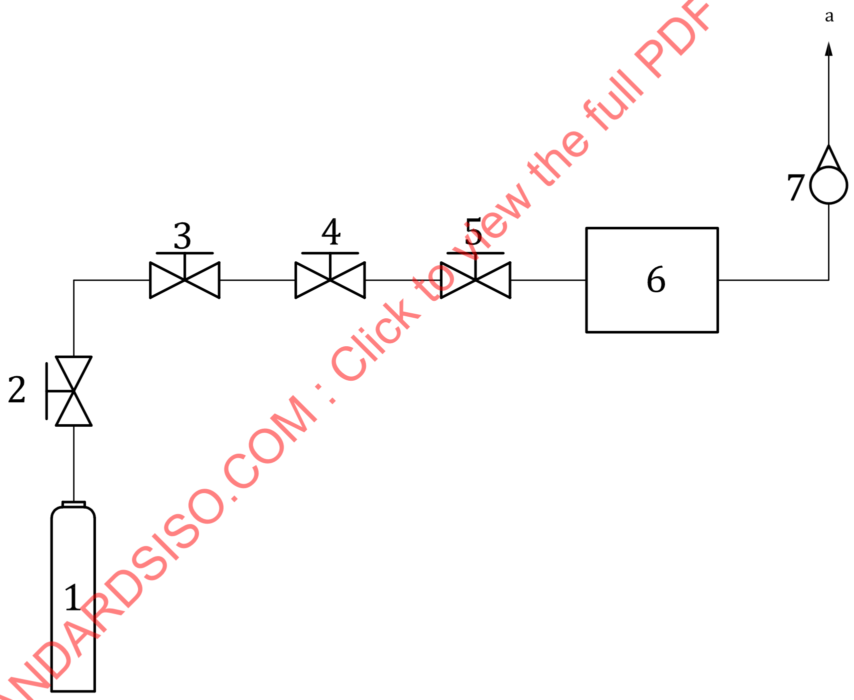
a) Purge the sampling system thoroughly by evacuation-gas purging cycles with the following steps.

  - Keep the outlet valve of the storage container (gas source valve) closed, valve 9 connected to the vacuum pump, open the other valves and start the vacuum pump to evacuate the sampling system.
  - Stop the vacuum pump after evacuation.
  - Open the gas source valve briefly and then close it.
  - Repeat the prior steps.

b) After the last cycle, close valve 8 and then open outlet valve 2, the injection pressure is adjusted to the pressure required to perform sampling.

B.1.2 Sampling without a vacuum pump

If a vacuum pump is not available, the sampling process is shown in Figure B.2. The sampling system should be purged by fill-empty cycle purging method first and continuous purging method after. After the purging is completed, the flow rate is adjusted to the flow rate required by the analyser to perform sampling.



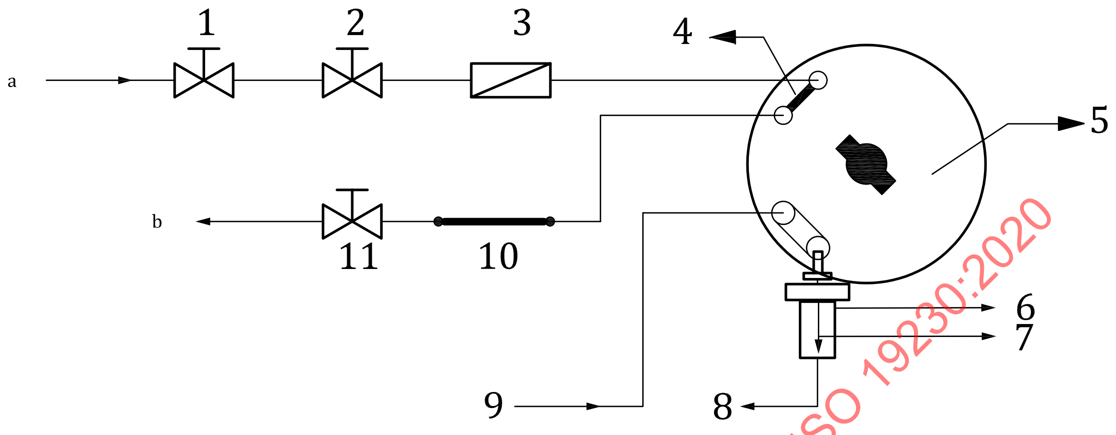
Key

1	storage container
2	outlet valve of the storage container
3	pressure reducer
4	flow controller
5	shut-off valve
6	analyser
7	flow meter
[a]	Vent.

Figure B.2 — Flow chart of direct sampling without a vacuum pump for gas in pressure receptacles

B.2 Direct sampling in liquid form from the liquefied gas in pressure receptacles

Direct sampling in liquid form for the liquefied gas is a commonly used method for analysing the liquefied gas, especially for low pressure liquefied gas nowadays. The sampling flow chart is shown in Figure B.3 and purging is done by continuous purging method.



Key

1	gas source valve
2	control valve
3	filter
4	internal fixed sample loop
5	liquid valve
6	injection inlet
7	needle
8	connect to the chromatographic column
9	auxiliary gas
10	fused silica capillary
11	restrictor valve
[a]	Gas in.
[b]	Vent.

Figure B.3 — Flow chart of liquefied gas sampling in liquid form

In Figure B.3, a small piece of fused silica capillary 10 is installed at the outlet of the liquid valve and may be replaced by other transparent tubes for bubble detection in the liquid. Behind the fused silica capillary, a suitable restrictor valve 11 is fitted to maintain the pressure of the sample, which might avoid evaporation of the sample effectively. The needle 7 ensures the liquid from the restrictor valve is connected to the analyser inlet as closely as possible to prevent the sample from vaporizing before it enters the analyser. Other connection methods are also available.

The sampling procedure is as follows.

With valve 11 closed, open valve 1 and valve 2. Then slowly adjust valve 11 until no bubbles are observed and a steady liquid flows out evenly. Continuously purging the system thoroughly through sample loop 4. Then rotate the liquid valve 5 to sample.

Annex C
(informative)

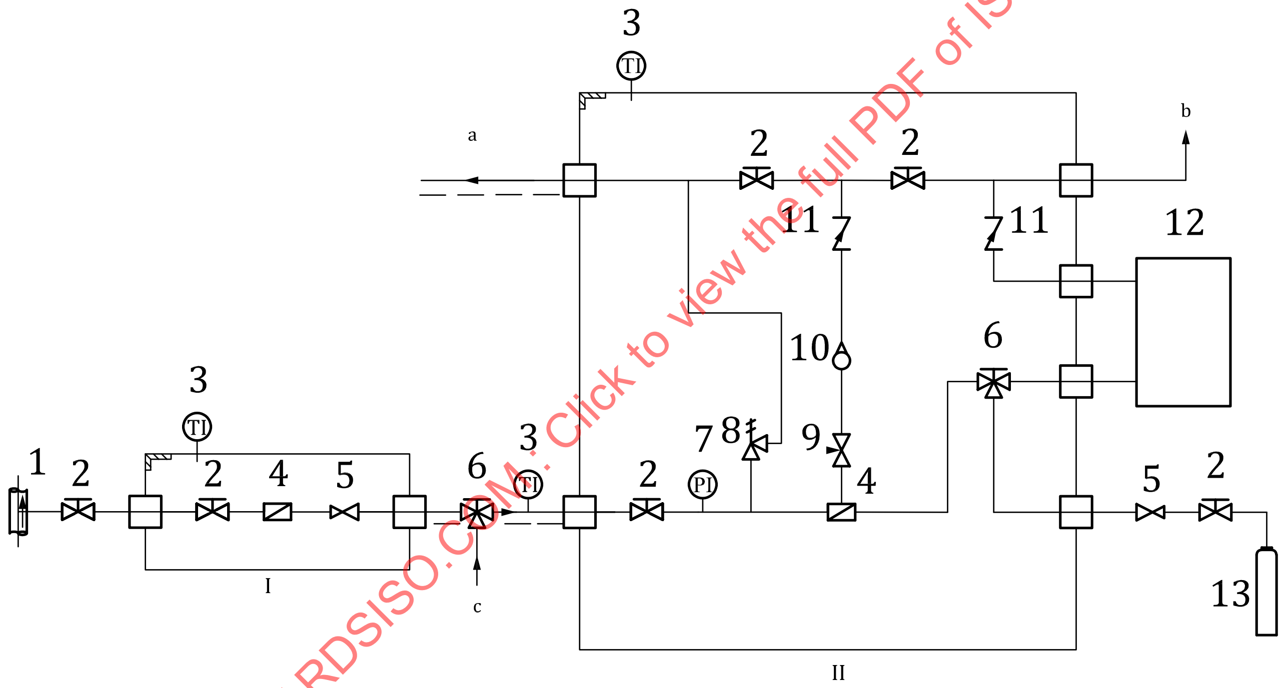
Direct sampling of gas in pipelines

C.1 Direct sampling of compressed gas in pipelines

A flow chart example of direct sampling of condensable compressed gas in pipelines is shown in Figure C.1.

The switch valves may be controlled manually or automatically.

The sample lines within and between I and II need to maintain the proper temperature to avoid condensation (see 6.3). Incondensable compressed samples do not require heating.



Key

1	sample probe	8	pressure relief valve
2	valve	9	flow controller
3	temperature indicator	10	flow meter
4	filter	11	check valve
5	pressure reducer	12	analyser
6	three-way valve	13	calibration gas cylinder
7	pressure gauge		
a	To the pipe.		
b	Vent.		
c	Inert gas.		

Figure C.1 — Flowchart of direct sampling of condensable compressed gas in pipelines

The sampling method according to Figure C.1 is as follows.

a) Open the gas source valve, purge the sampling system with fill-empty method, discharge the gas via bypass line.

b) Adjust the appropriate flow of the sampling and bypass line, purge the sampling system with continuous purging method sufficiently and start sampling.

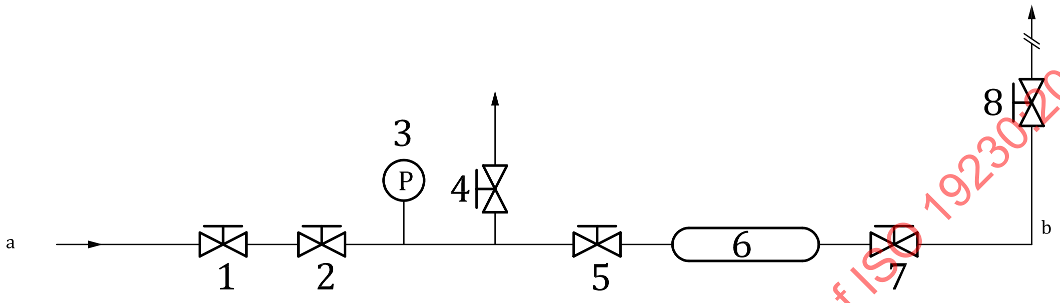
C.2 Direct sampling of liquefied gas in pipelines

It is easy to form a two-phase gas-liquid flow for sampling of liquefied gas to go through a long line and decompression process. So, sampling after evaporation (see 9.3) is generally used instead of liquid sampling for direct sampling of the liquefied gas in pipelines.

Annex D
(informative)

Fill-empty sampling method

The flow chart of fill-empty sampling method for compressed gas is shown in Figure D.1.



Key

1	gas source valve
2	pressure reducer or flow controller
3	pressure gauge
4	vent valve
5	inlet valve of the sample container
6	sample container
7	outlet valve of the sample container
8	needle valve
a	Gas in.
b	Extension tube.

Figure D.1 — Schematic of fill-empty sampling method

For compressed gas, the sampling method is as follows.

a) Purge the sample line thoroughly by fill-empty method with the following steps.

- Open gas source valve 1 briefly and then close it.
- Open valve 2, and then vent valve 4 to vent the gas.
- Close valve 4 after venting.
- Repeat the prior steps to purge the sample line upstream of the inlet valve 5.

b) Purge the sample container thoroughly by fill-empty cycle purging method as follows.

- Open valve 1 and valve 7.
- Open valve 5. When the pressure builds up to the selected pressure, close inlet valve 5 and open valve 8 slowly vent the container to atmospheric pressure. Then close valve 8.
- Repeat the last step for a number of cycles to effectively purge the sample container.

c) Sample as follows.

  - After the last cycle, open valve 5. After the pressure has built up to the selected container pressure, close gas source valve 1.
  - Close inlet valve 5 and outlet valve 7.
  - After depressurizing the sample line through vent valve 4 and valve 8, remove the sample container.

Fill-empty sampling method of liquefied gas should comply with the following steps.

a) Purge the sample line continuously: open the gas source valve 1, vent valve 4, and slowly open valve 2, using the sample to purge the sample line and then close vent valve 4.

b) Purge the sample container by fill-empty cycle purging method as follows.

   The sample container is placed upright, the valve 7 with an ullage tube at the top. Open the inlet valve 5 and vent valve 8 completely. Slowly open the outlet valve 7. After an appropriate amount of gas is filled into the sample container, close gas source valve 1, and allow part of the sample to escape in vapour phase through valve 7. Close valve 7 and invert the upright sample container several times, return the container to the upright position with valve 7 on the top, then release the remainder of the sample in the liquid phase by opening vent valve 4.

c) Repeat step b) until purged thoroughly, usually no less than 3 times.

d) Gas collection: close vent valve 4 and open the gas source valve 1 to start sampling. Open inlet valve 5 of the sample container completely, and slowly open the outlet valve 7 of the sample container. After filling the gas into the sample container, close all the valves.

e) Sample ullage: Place the sample container in an upright position with valve 7 at the top and immediately open valve 7 and vent valve 8 slightly. Allowing the excess liquid to escape from valve 7 and close valve 7 at the first sign of vapour.

WARNING — For safety, sample ullage should be done immediately after the sample has been obtained. The container reserved volume when sample ullage is stopped at the first sign of vapour is the designed reserved volume of the container (see Figure 5). Sampling at extremely low ambient temperatures or from cryogenic sources may require additional precautions (such as discharging more liquid than the reserved volume to prevent the sample containers from becoming filled completely with liquid when warming up the sample to high ambient temperature).

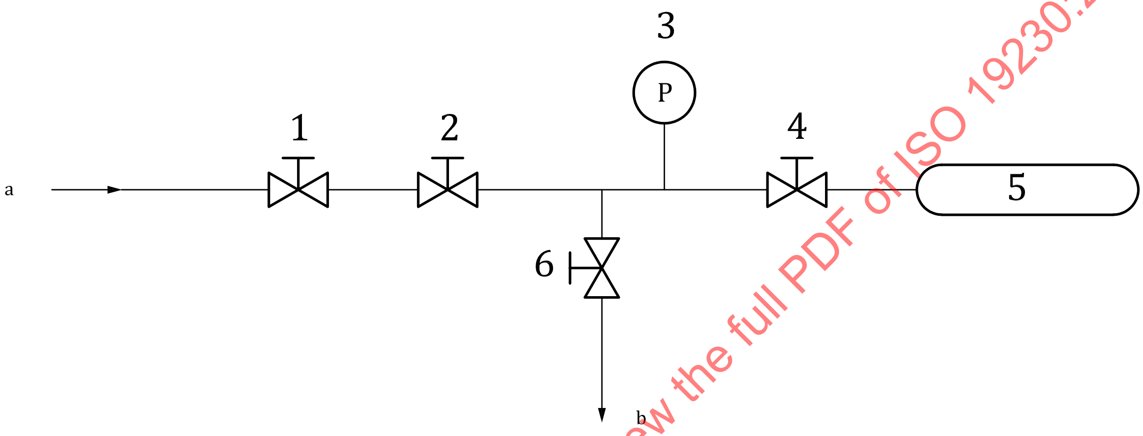
f) Remove the sample container: remove the sample container after the sample system is fully released.

Annex E
(informative)

Evacuated-container sampling

E.1 Evacuated-container sampling of compressed gas

For compressed gas, a flow chart example of the evacuated-container sampling is as follows in Figure E.1.



Key

1	gas source valve
2	flow controller or pressure reducer
3	pressure gauge
4	inlet valve of the sample container
5	sample container
6	vent valve
a	Gas in.
b	Vent.

Figure E.1 — Schematic of evacuated-container sampling for compressed gas

For compressed gas sampling, purge the sampling system thoroughly by fill-empty method via the vent valve 6, then fill the sample container to the desired pressure.

For low-pressure gas sampling, double oblique bore stopcock sample tube [see Figure 4 b)] also may be used as a sample container. A flow chart example of evacuated-container sampling is shown in Figure E.2.