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Natural gas — Sampling guidelines

Gaz naturel — Lignes directrices pour l'échantillonnage

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 10715 was prepared by Technical Committee ISO/TC 193, *Natural gas*, Subcommittee SC 1, *Analysis of natural gas*.

Annexes A to J of this International Standard are for information only.

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Introduction

This International Standard provides guidance on all aspects of the sampling of processed natural gas. Unless otherwise stated, all pressures up to 15 MPa in this International Standard are given as gauge pressures.

The determination of the composition and the properties of the gas is highly dependent on the sampling technique. Also of great importance are the design, construction, installation and maintenance of the sampling system as well as the conditions of sample transfer and transport.

These guidelines cover sampling strategy, details of sampling methods, the choice of sampling method and sampling equipment.

This document is intended for use in those cases where sampling is not described as part of the analytical procedure.

This document concentrates on sampling systems and procedures. Analyses from the samples collected using these systems and procedures may be utilized in many different ways, including calculations to determine the calorific value of the gas stream, identification of contaminants contained in the gas stream, and compositional information to determine whether or not the stream meets contractual specifications.

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Natural gas — Sampling guidelines

WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability or regulatory limitations prior to use.

All sampling activities shall comply with local safety regulations.

1 Scope

The purpose of this document is to provide concise guidelines for the collection, conditioning and handling of representative samples of processed natural gas streams. It also contains guidelines for sampling strategy, probe location and the handling and design of sampling equipment.

It considers spot, composite (incremental) and continuous sampling systems.

This document gives consideration to constituents such as oxygen, hydrogen sulfide, air, nitrogen and carbon dioxide in the gas stream.

This document does not include sampling of liquid streams or streams with multiphase flow.

Traces of liquid, such as glycol and compressor oil, if present, are assumed to be intrusive and not a part of the gas to be sampled. Their removal is desirable to protect the sampling and analytical equipment from contamination.

This document can be used for custody transfer measurement systems and allocation measurement systems.

2 Definitions

For the purposes of this International Standard, the following definitions apply:

2.1 direct sampling:

Sampling in situations where there is a direct connection between the natural gas to be sampled and the analytical unit.

2.2 floating-piston cylinder:

A container which has a moving piston separating the sample from a buffer gas. The pressures are in balance on both sides of the piston.

2.3 flow-proportional incremental sampler:

A sampler which collects gas over a period of time and at a rate that is proportional to the flow rate in the sampled pipeline.

2.4 high-pressure natural gas:

Natural gas with a pressure exceeding 0,2 MPa.

NOTE — The maximum for this International Standard is 15 MPa.

2.5 hydrocarbon dew point:

The temperature, at a given pressure, at which hydrocarbon vapour condensation begins.

2.6 incremental sampler:

A sampler which accumulates a series of spot samples into one composite sample.

2.7 indirect sampling:

Sampling in situations where there is no direct connection between the natural gas to be sampled and the analytical unit.

2.8 liquid separator:

A unit, in the sample line, used to collect liquid fall-out.

2.9 low-pressure natural gas:

Natural gas having a pressure between 0 and 0,2 MPa.

2.10 purging time:

The period of time during which a sample purges a piece of equipment.

2.11 representative sample:

A sample having the same composition as the natural gas sampled when the latter is considered as a homogeneous whole.

2.12 residence time:

The time it takes for a sample to flow through a piece of equipment.

2.13 retrograde condensation:

Retrograde behaviour describes the non-ideal phase properties of hydrocarbon gas mixtures, such as natural gas. Retrograde condensation is the production of a liquid phase of heavy hydrocarbons at a particular pressure and temperature where, at that same temperature, the gas stays in a single phase at a higher pressure as well as at a lower pressure.

NOTE — See also 5.2.

2.14 sample container:

A container for collecting the gas sample when indirect sampling is necessary.

2.15 sample line:

A line provided to transfer a sample of the gas to the sampling point. It may include devices which are necessary to prepare the sample for transportation and analysis.

2.16 sample probe:

A device inserted into the gas line to be sampled and to which a sample line is connected.

2.17 sampling point:

A point in the gas stream where a representative sample can be collected.

2.18 spot sample:

A sample of specified volume taken at a specified place at a specified time from a stream of gas.

2.19 transfer line:

A line provided to carry the sample to be analysed from the sample point to the analytical unit.

2.20 water dew point:

The temperature, at a given pressure, at which water vapour condensation begins.

3 Principles of sampling

3.1 Sampling methods

The main function of sampling is to take an adequate sample that is representative of the gas.

The main distinction in sampling is between direct and indirect sampling methods.

In the direct sampling method, the sample is drawn from a stream and directly transferred to the analytical unit.

In the indirect sampling method, the sample is stored before it is transferred to the analytical unit.

The main classifications of the indirect sampling method are spot sampling and incremental sampling.

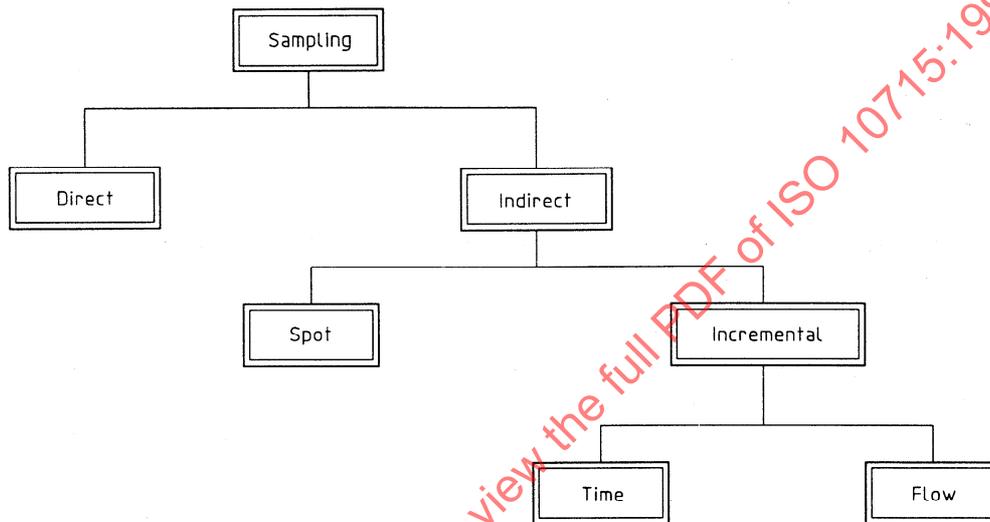


Figure 1 — Survey of direct and indirect sampling methods

The information needed from the analysis of natural gas falls into two basic categories: averaged and limit values.

3.1.1 Averaged values

A typical example is the calorific value. Custody transfer requires the time- or flow-averaged calorific value. Commercial agreements determine the period and method of averaging.

3.1.2 Limit values

Most gas custody transfer contracts contain specification limits on composition or on gas properties. Direct sampling can be applied, but often the requirements are such that also indirect sampling has to be applied.

3.2 Sampling frequency

This subclause gives guidelines for the establishment of the sampling frequency. Mostly the sampling frequency is a matter of common sense. Information on the properties of the gas stream in the past and about expected (systematic) future changes determines the sampling frequency.

Generally, pipeline gas composition will have daily, weekly, monthly, semi-annual and seasonal variations. Compositional variations will also occur because of gas treatment equipment and reservoir changes. All of these environmental and operational considerations shall be taken into account when selecting a sampling interval.

The statistical approach in this paragraph is only intended to support the common-sense approach.

In this context, the required sampling frequency is the number of samples to be taken in a certain period of time in order to obtain meaningful results.

The formula for calculating the number of samples is:

$$n^{\frac{1}{2}} = t \times \frac{s}{d}$$

where

d is the error margin required;

n is the number of samples;

s is the standard deviation;

t is Student's t -factor (see table H.1 in annex H).

This equation shall be solved by iteration: an initial value of t is estimated, and used to calculate a revised value of n , which is used, in turn, to give a new value of t . The error margin, the number of samples and the standard deviation shall be taken over the same period of time.

3.2.1 Error margin

There are two different cases of error margins. One case is related to the determination of averaged values. In most custody transfer contracts, these values are given as an indication of the accuracy.

The other is related to the determination of limit values. Custody transfer contracts specify the limits but rarely give an indication of the accuracy. In these cases, the difference between the last measured value, or the last year's average, and the limit value is the error margin.

3.2.2 Number of samples

The number of samples is the number of samples to be taken in a defined period. It is equivalent to the number of partial samples in incremental sampling.

3.2.3 Student's t -factor

Student's t -factor allows for the finite sample size, and is to be found in standard statistical tables. The value depends on the claimed certainty (typically 95 %) and the "degrees of freedom", here to be taken as the number of measurements minus one ($n-1$).

EXAMPLE 1

Determination of the monthly average caloric value

$d = 0,4 \%$ (error margin required from custody transfer contract for monthly averaged value)

$s = 0,6 \%$ (estimated variation over a one-month period)

First estimate, taking $n = 7$:

$t = 2,45$ for 6 degrees of freedom and a certainty of 0,975 single-sided (equals 0,95 double-sided)

$$n^{\frac{1}{2}} = 2,45 \times \frac{0,6}{0,4}$$

$$n = 14$$

First iteration, taking $n = 14$:

recalculate for

$t = 2,16$ for 13 degrees of freedom, and a certainty of 0,975 single-sided (equals 0,95 double-sided)

$$\frac{1}{n^2} = 2,16 \times \frac{0,6}{0,4}$$

$$n = 11$$

Second iteration, taking $n = 11$:

recalculate for

$t = 2,23$ for 10 degrees of freedom, and a certainty of 0,975 single-sided (equals 0,95 double-sided)

$$\frac{1}{n^2} = 2,23 \times \frac{0,6}{0,4}$$

$$n = 11$$

EXAMPLE 2

Total sulfur determination

Last measured concentration 20 mg/m³ and the contract limit value 50 mg/m³.

$d = 30$ mg/m³ (difference between limit value from custody transfer contract and last measured value)

$s = 10$ mg/m³ [standard deviation in spot sample results (in the past year)]

$t = 4,30$ $n - 1$ taken as 2, level of certainty 95%

$$\frac{1}{n^2} = 4,3 \times \frac{10}{30}$$

$$n = 2$$

Three samples are enough. Recalculation indicates that two samples are not enough.

4 Safety precautions

4.1 General

Sampling and sample handling shall follow all relevant national and company-related safety regulations.

In the case of inadequate regulations, those responsible for sampling shall establish detailed procedures. Specifications for equipment shall also be established.

Personnel involved shall be properly trained and educated to a level such that they are able to take necessary responsibility.

4.2 Personnel

The person responsible for the department/section which is to perform the sampling shall be satisfied that the sampling can be performed within relevant safety regulations.

Those performing sampling or installing sampling equipment shall have the necessary training and education to evaluate potential safety hazards in general.

The above personnel shall have the authority to prevent sampling or installation of sampling equipment which is unsuitable or unsafe.

4.3 Equipment

Equipment used in the sampling of high-pressure natural gas shall be inspected and recertificated if required regularly.

Documentation shall be available and up to date.

Equipment shall be designed to meet relevant sampling conditions, e.g. pressure, temperature, corrosivity, flow, chemical compatibility, vibration, thermal expansion and/or thermal contraction.

Glass cylinders shall not be exposed to pressure.

If provided for, end caps shall be installed on cylinders during transportation and storage.

Cylinders shall have volume, working pressure and test pressure permanently stamped.

Cylinders shall have a test pressure of at least 1,5 times the working pressure.

Cylinders shall be protected against damage during transportation and storage. Transportation boxes or cartons designed for the individual type of cylinder shall be available.

Cylinders shall be accompanied by labels or paperwork with relevant information protected against damage.

Cylinders and associated accessories shall be inspected and leak-tested periodically.

Permanent transfer and sampling lines shall be properly secured. Breakable connections shall have easy access for leak-testing. Outlets shall be equipped with double block and bleed valves. End caps shall be connected to fittings when the cylinders are not in use.

The use of flexible high-pressure tubing shall be limited and manufacturers' instructions for safe application shall be strictly followed. Transfer lines can be blocked by solid or liquid contaminants. Special precautions shall be employed when trying to "reopen" such lines. Only qualified personnel may do this.

Transfer lines shall have shut-off valves located as close to the source stream as possible. The sampling probe shall be equipped with a shut-off valve.

Electrical equipment shall be approved for the relevant sampling application.

Equipment which can create static electricity shall be avoided.

Use of equipment or tools which may create sparks shall be avoided.

4.4 Flammability

In order to prevent fire or explosions, the following restrictions shall be followed within areas where flammable concentrations of gas (about 4% to 16% for natural gas) may be found:

No open fire

No smoking

No use of equipment and tools which may create sparks

No use of equipment which operates at temperatures above the self-ignition temperature of gas mixtures, mostly above 400 °C (for natural gas)

No use of chemicals which can react vigorously with gas

No running spark ignition motors

Ventilation shall be sufficient to prevent the build-up of a flammable atmosphere.

Purging of transfer lines shall be directed to a "safe area" (e.g. flare). Release of gas during manual (spot) sampling shall be limited to a minimum at the sampling location.

Gas detectors shall be used at strategic locations relative to sampling locations.

Manual and/or automatic firefighting equipment shall be easily available.

Personnel performing sampling shall be trained to react appropriately in the event of a fire.

4.5 Personal protective equipment

Necessary personal protective equipment shall be available. The need for protective equipment will vary from place to place. The following factors shall, however, be considered:

Toxic or irritant components in the gas (H₂S, radon, Hg, aromatics, etc.) may require the use of breathing filters, a fresh-air supply, gloves and monitors for toxic components.

Sampling of high-pressure gas may require the use of goggles or face shields. Pressure indicators (gauges) shall be used to indicate the system pressure. Leak detector spray or a portable leak detection device shall be used to check that the system is leaktight.

For fire protection, personnel shall wear flame-resistant clothing (aprons, coveralls, lab dress). Personal smoke protection masks shall also be available.

4.6 Transportation

Sample cylinders containing gas under pressure shall be transported in accordance with relevant regulations.

Constant-pressure-type cylinders shall always be protected in some kind of transportation container. Damage to the cylinder itself and/or to valves, gauges, etc., may otherwise occur.

During transportation, the cylinder shall also be protected against conditions of temperature which could create overpressure or condensation of sample.

The container shall be properly labelled in accordance with applicable regulations.

5 Technical considerations

5.1 Flow characteristics

Flow in a pipeline may be laminar or turbulent. However, in the sampling system, laminar flow shall be avoided. It can be single-phase or multiphase. Most gas streams operate with turbulent single-phase flow. Two-phase turbulent flow may also be found in gas lines where the fluid is near saturated conditions.

For example, the flow from a gas/liquid separator will be near the gas dew point, and a reduction in line temperature will cause some condensation to occur, resulting in two-phase flow.

It can also happen that, after a mixing station, the combined gases are not completely mixed in the pipeline.

If the composition is not completely homogeneous, a static mixer will improve the homogeneity.

5.1.1 Laminar flow

Laminar flow will not normally occur in a gas line because the gas viscosity is low and the flow velocities are high enough to ensure that this will not happen. However, the design of the sampling system shall be such that laminar flow is avoided.

5.1.2 Turbulent flow

In general, turbulent flow is advantageous in a sampling system and in the gas line to be sampled because the turbulence creates a well-mixed fluid.

5.1.3 Two-phase flow

Sampling of two-phase (gas/liquid) mixtures is not covered by these guidelines and shall be avoided if at all possible.

Current technology of natural-gas sampling is not sufficiently advanced to accomplish this with reasonable accuracy.

5.2 Condensation and revaporization

The condensation behaviour of natural gas is rather complicated. Figure 2 gives an example of a pressure/temperature phase boundary diagram for a natural gas. The shape of the curve depends on the composition of the gas.

As figure 2 shows, the phase boundary is a complex function between the critical point and normal operating conditions. Retrograde condensation can occur when the phase boundary is encountered in an unexpected manner while adjusting the pressure or temperature of the gas.

Before starting the analysis, the sample shall be heated to at least 10 °C above the source temperature. If the source temperature is not known, the sample shall be heated to at least 100 °C. To ensure revaporization, this heating shall be applied for a period of 2 h, or longer if necessary.

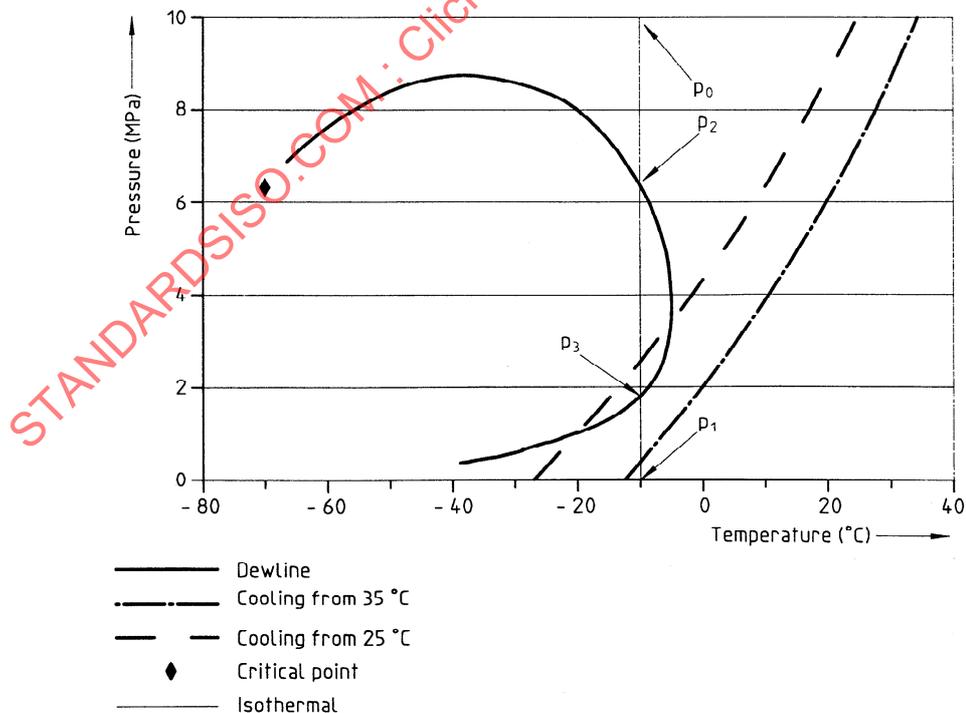


Figure 2 — Example of a pressure/temperature diagram for natural gas

5.2.1 Example of a condensation problem

An example of how this problem can occur is shown in figure 2. The pipeline contains gas at pressure p_0 . If the initial temperature is -10 °C , and the gas is expanded (i.e. has its pressure reduced) isothermally, it will follow the vertical line in the figure as it approaches the pressure at which it can be analysed, p_1 . The gas is a stable single phase at p_0 and continues to be so until it reaches pressure p_2 , which is on the boundary of the two-phase region.

Between p_2 and the lower pressure p_3 , both gas and condensed liquid are present. The relative quantities of the gas and liquid phases, and their compositions, vary continuously over this range. At pressures below p_3 , and down to the analysis pressure p_1 , a single-phase gas exists once more.

Conversely, a cylinder with an initial pressure of p_1 , filled isothermally to p_0 , will, as the pressure passes through p_3 , contain two phases. These will in theory recombine at p_2 , but this process is slow, and any gas sampled from the cylinder while two phases are present will be unrepresentative, and furthermore its removal will alter the composition remaining in the cylinder.

The use of pressurized piston cylinders may be a way to avoid these problems, keeping the sample in a state where no fall-out will take place.

In fact, as a gas is expanded, its temperature falls due to the Joule-Thompson effect. The gas whose behaviour is shown in figure 2, starting from a temperature and pressure of 25 °C and 10 MPa , will cool to below -10 °C at p_3 , and hence suffer condensation. The initial temperature would need to be 35 °C to reach p_1 without encountering the two-phase region.

5.2.2 Condensation after sample has been collected

A gas sample could partially condense in the sample container when it is being transported or is awaiting analysis in a lab. High-pressure gas sample containers and the lines to an analytical unit shall always be heated prior to analysis (except for gas that will not pass through a phase boundary). Heating times and temperatures shall be sufficient to ensure that any condensed hydrocarbons are re-vaporized before an analysis is started.

5.2.3 Fall-out from the sampling probe

Liquid heavy hydrocarbons and condensation in the sample line which is returning into the main stream may reduce the measured calorific value of a gas. This will manifest itself in a day/night sine wave effect on the recorder chart, with the calorific value recording higher in the heat of the day and lower in the cool of the night.

5.2.4 Precautions by applying heating and insulation

In order to avoid condensation problems, the sample handling equipment temperature shall be kept above the gas dew point at any pressure in the sampling system. Also the gas may be pre-heated, as indicated in figure 2.

5.3 Adsorption and desorption

The process whereby some gas components are adsorbed on to or desorbed from the surfaces of a solid are called sorption effects. The force of attraction between some gas components and solids is purely physical and depends on the nature of the participating materials.

Natural gas may contain several components which exhibit strong sorption effects. Special attention shall be given to this in the case of the determination of trace concentrations of heavy hydrocarbons or impurities.

5.4 Leaks and diffusion

A regular check of the leaktightness of the lines and devices shall be carried out, in order to detect leaks. Minor leaks or diffusion would affect the composition in the case of trace determinations (water or atmospheric oxygen may diffuse into the tube or the container, even at high pressure: the partial-pressure difference for a constituent determines the direction in which it will diffuse). Take special care when hydrogen is present.

Leaks can be detected using detergent solutions, by pressuring the sampling line, or by more sophisticated methods such as portable leak detection equipment (e.g. mass spectrometers).

5.5 Reactions and chemisorption

Reactive components can combine chemically with the sampling equipment (e.g. by oxidation) or exhibit chemisorption. Also the materials used in the sampling equipment can catalyse reactions in the samples (e.g. in mixtures with traces of hydrogen sulfide, water and carbonyl sulfide).

5.6 Precautions using drip pots

Drip pots or gas/liquid separators in a sample line system are intended to remove troublesome intrusive liquids. Their application shall be considered carefully (see 8.4). Drip pots can accumulate liquid slugs and then continuously vaporise into the sample stream. There is a danger that their use may change the composition of the sampled gas. The concentrations of components which equilibrate between the gas and liquid phases are likely to be altered by removal of the liquid. Sample lines shall slope up from the sampling point, with no low spots that can accumulate liquids.

6 Materials used in sampling

6.1 General considerations

The suitability of materials used in a sampling system will depend on the gas being sampled. Generally, it is recommended that stainless steel be used for all surfaces with which the gas will come into contact (see however 6.1.1). Valve seats and piston seals shall be made of (elastic) material appropriate for the intended service. Sampling of wet or high-temperature gases, or gases containing hydrogen sulfide or carbon dioxide, presents additional material problems. These types of gas may require special materials and coatings in the sampling system. It is recommended that sample cylinders used in sour-gas service shall be either polytetrafluoroethylene (PTFE) coated or epoxy coated. Reactive components such as hydrogen sulfide and mercury shall be analysed on site using direct sampling methods when practical since even coated vessels may not eliminate absorption of these components.

The use of soft metals such as brass, copper and aluminium shall be avoided where corrosion and metal fatigue problems are likely to occur. Aluminium can, however, be used for sample containers in some applications where the sample container reactivity is critical.

Generally, materials coming into contact with samples or calibration gases shall have the following characteristics:

- impermeability to all gases;
- minimum sorption;
- chemical inertness to the constituents being transferred.

Because of the possible presence of small amounts of sulfur compounds, mercury, carbon dioxide, etc., in natural gas, all equipment and fittings shall preferably be made of stainless steel or, for low pressure, glass. However, possible alternative materials are listed in table 1.

6.1.1 Carbon steel

Carbon steel and other relatively porous materials may retain heavier components and contaminants such as carbon dioxide and hydrogen sulfide in the natural-gas stream and shall not be used in a sampling system.

Although stainless steel is generally a good material for use in sampling equipment, the user is recommended to consult corrosion experts before using it.

Stainless steel is not generally suitable for streams containing water. However, some stainless-steel materials, 4 CrNi 18 10 and 4 CrMo 17 12 2, have proven to be satisfactory.

Table 1 – Compatibility of sampling-system materials with gas components

| Material | Compatibility ¹⁾ with gas components: | | | | | | | |
|---------------------|--|------------------------|--------------------------------------|--------------------------------|------------------|----|----|----------------------|
| | C _n H _m | COS CO ₂ | CH ₃ OH O ₂ | H ₂ S RSH THT | H ₂ O | He | Hg | H ₂ CO |
| Stainless steel | a | a | a | b | b | a | b | a |
| Glass ²⁾ | a | a | a | a | a | a | a | a |
| PTFE ³⁾ | b | b | b | a | c | c | c | b |
| Polyamide | a | a | b | a | c | a | c | a |
| Aluminium | a | a | a | b | b | a | c | a |
| Titanium | a | a | a | a | a | a | a | a |

1) a= suitable
b= with reservations
c= not recommended

2) Glass is a highly inert material, but subject to breakage and unsafe for sampling above atmospheric pressure.

3) PTFE is inert but may be adsorptive. It is permeable to e.g. water, He and H₂. PTFE coatings may have imperfections, and parts of the interior surface may therefore not be protected.

6.1.2 Epoxy coatings

Epoxy (or phenolic) coatings will reduce or eliminate adsorption of sulfur compounds and of other minor constituents. It is not practical to coat small fittings, valves and other small areas. Losses of gas components from such unprotected areas may however be detectable and may be measured if concentrations are in the ppb or ppm range (see also 7.2).

6.1.3 Other polymers

The use of other polymers shall be limited to tubing or connectors joining items of equipment, where there is little or no direct contact with the sample. Special care shall be taken in the case of water or sulfur-compound analysis. However, good results may be obtained using polyamide material for short tubing lengths.

In some cases, soft PVC can be used at low pressures.

Before any new polymer material is used in a sampling system, it shall be tested using certified blends at expected concentrations to verify that it does not cause any change in the sample composition.

6.1.4 Rubbers

Rubber tubing or connections is not recommended, even at low pressure, because of the high reactivity and permeability of rubber.

Silicon rubbers are known for their high absorption and permeability for many components.

6.2 Bimetallic corrosion

Using dissimilar metals in contact with each other in a sample system may cause increased rates of corrosion and result in sampling errors and/or safety problems.

7 General preparation of equipment

7.1 Surface treatment

The sorption effects exhibited by some materials can be modified and often reduced by surface treatment. A clean, grease-free surface shows less absorption. Rough surfaces provide a nucleus for gases to adsorb and accumulate.

Polishing techniques are now available and can be used to minimize sorption effects and reduce the conditioning time required to bring the sampling equipment to equilibrium.

Other processes are also available to reduce sorption effects. Some materials can be electroplated with an inert material such as nickel to reduce adsorption.

Passivation of aluminium using proprietary techniques is available to inhibit adsorption.

7.2 Cleaning sampling systems

All parts of the sampling and transfer lines in contact with gas shall be free from grease, oil, mould or any polluting products. Sample containers shall be cleaned and purged prior to each collection of sample, unless they are special passivated cylinders used to sample streams containing highly reactive components (see also annex B). They shall be cleaned properly, e.g. with a volatile solvent, and dried to avoid absorption phenomena, particularly those caused by sulfur compounds and heavy hydrocarbons. Solvents, such as acetone, that do not leave a residue after drying are generally acceptable for removing heavy-ends contamination, although they may present hazards such as flammability and toxicity in some cases. Steam cleaning is generally acceptable only if the steam itself is clean and does not contain corrosion inhibitors, boiler water treating chemicals or other substances that may contaminate the sample cylinder.

Special care shall be taken in cleaning cylinders that contain deposits.

If analysis of sulfur components is intended, steam shall not be used to clean stainless-steel cylinders. Sulfur species will be readily absorbed by the cylinders and the analysis will dramatically underestimate sulfur levels. Samples to be analysed for their sulfur content need to be collected in special lined cylinders or passivated cylinders dedicated to that purpose. It is important to note that the entire wetted surface of the sample container and its secondary components shall be coated. Coating the cylinder, but not the valves, fittings, relief devices, etc., may not be sufficient protection. In certain cases, e.g. H₂S-containing gases, PTFE is the recommended coating.

7.3 Conditioning of sampling equipment

This can be achieved by purging the sampling equipment with the sample gas until gas samples taken in sequence show analytical consistency. Conditioning times may be reduced by the initial evacuation of the equipment prior to purging with the sample. Several sequences of evacuation and purging may be advantageous in reducing conditioning time and achieving equilibrium.

The final assessment that equilibrium has been achieved and the sampling equipment conditioned can initially be determined by analysis using a known standard.

7.4 Pre-charging

Nitrogen, helium, argon and dry, instrument-quality air are good examples of gases that may be used to dry or purge cylinders which are free of deposits and heavy-ends contamination. In order to avoid interference, the drying or purging gas used shall not contain any of the constituents to be analysed. Many laboratories leave a blanket of nitrogen, helium or other gases in sample cylinders in order to protect the cylinder from air contamination. The blanket gases and gases used to recharge or back-pressure sample cylinders shall be carefully selected so that, if leakage does occur within the cylinder or the sample is contaminated by these gases, the analytical system will not interpret the contamination by these gases as being a part of the sample being analysed. For example, chromatography using helium as a carrier gas will not detect helium left over from the recharge of a single-cavity cylinder or helium leaking past the piston in a floating-piston cylinder.

8 Sampling equipment

8.1 Sample probes

The design of the probe shall take into account the possibility of resonant vibration being induced in the probe by high flow velocities in the pipeline. Gas lines with streams free of entrained liquids and at flow conditions well above their dewpoint temperatures may be sampled with any probe design. However, lines that are operating at or near

the gas stream dewpoint require a special probe designed to overcome the problems of condensation and liquid particle entrainment in the gas.

8.1.1 Straight-tube probe

The most basic sample probe design is the straight-tube probe shown in figure 3. The end may be flat or angle-cut.

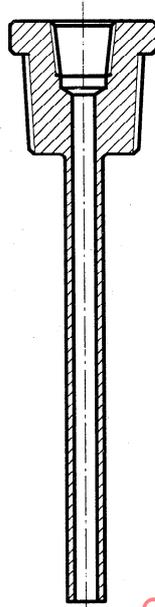


Figure 3 – Straight-tube probe

8.1.2 Regulated probe

The other type of probe design in common use in the gas industry is the regulated probe. These probes are commonly used with continuous analyser systems and are designed to deliver the gas to the system at reduced pressure. The diaphragm and control spring are mounted externally to the pipe wall, and connected by an internal rod to the point at which the pressure reduction occurs, which is at the lower end of the probe which is inserted into the gas stream. This lower end is often finned, so that the temperature drop on expansion is compensated for by the thermal mass of the gas stream. An illustration of a typical regulated probe is shown in figure 4.

8.1.3 Location and installation

The probe shall be located directly in the gas stream in such a way that problems of aerosols and dust are eliminated.

It is recommended that the probe be located a minimum of 20 pipe diameters downstream from any flow-disturbing elements such as elbows, headers, valves and tees.

The location of the probe shall be on the top of a horizontal part of the pipe. The inlet shall be located so as to withdraw gas from the centre one-third of the pipeline diameter.

The probe shall be externally equipped with adequate valving. This makes it possible to disconnect the sample line from the process line. The probe may be of a stationary or removable type depending on location and operating conditions.

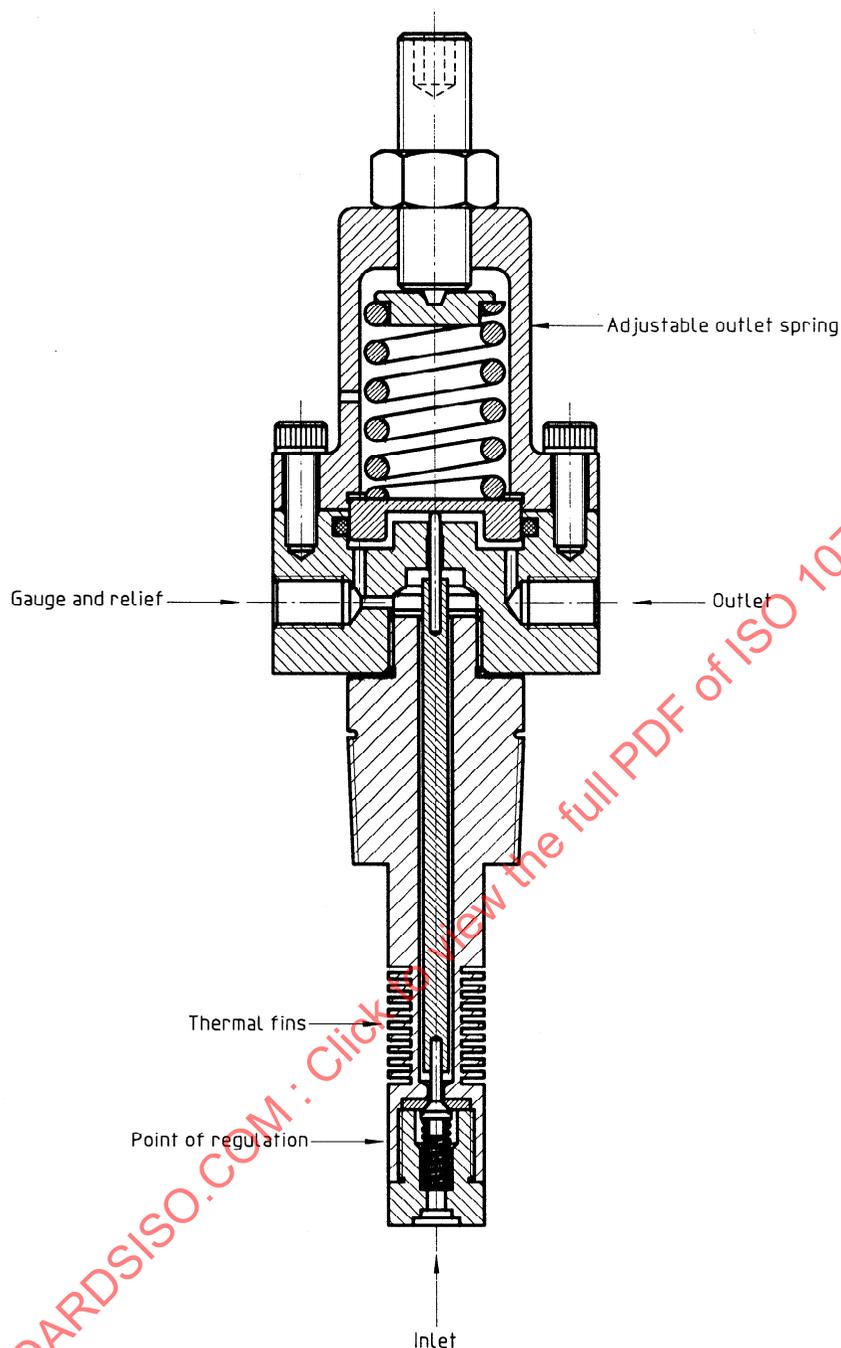


Figure 4 — Regulated probe

8.2 Sampling and transfer lines

Generally, sampling lines shall be as short and as small in diameter as possible, but not less than 3 mm in diameter, to decrease the residence time.

Sample lines venting to the atmosphere shall be minimized. In addition, high-pressure drops may cause cooling and condensation, which will affect the representative nature of the sample.

The purging time for spot samples shall be at least 10 times the residence time. Annex G gives guidelines for the calculation of the residence time.

All connections between the sample point and the sample container shall be such that sample contamination cannot occur. Where necessary and allowed, threaded connections shall be made using PTFE tape. Pipe thread sealing

compounds shall not be used. These products may contaminate the sample and/or absorb components from the sample, resulting in erroneous results.

8.2.1 Pressure drop in a sample line

Proper operation of a sample line requires a pressure differential from the collection point to the discharge.

This pressure drop may be provided by an orifice plate, regulator or other appropriate device in the flow line.

8.2.2 Dimensions of sampling lines

The flow rate through the sampling line is chosen to ensure a fast response time. However, each application has to be considered on its own merits.

8.3 Bypass constructions

When using a bypass, closed loops are preferred due to environmental and safety considerations.

8.3.1 Bypass loop

The bypass loop, also known as a "fast loop" or "hot loop", shall be of the closed configuration; it shall return to the process line.

3 mm to 10 mm stainless-steel tubing should preferably be used. The loop requires a pressure differential, from collection point to discharge, to ensure a constant and steady flow rate through the sampling equipment located in the loop.

8.3.2 Bypass line

Where it is impractical to provide a sufficient pressure differential, thought can be given to the use of an open-ended bypass line which will ultimately vent to the atmosphere or to a flare.

The flow rate and pressure loss in an open-ended line will need to be controlled to limit any cooling and condensation which will affect sample integrity.

8.4 Aerosol and/or dust traps

It might sometimes be necessary to control some characteristics of the gas at the outlet of process units (for example, water content after dehydration, hydrogen sulfide content after desulfurization, dewpoint after compression). Some units, because of the nature of the process, may release some contaminants in the form of liquid, aerosols or froth (glycol, amine, oils, etc.). In that case, it is necessary to protect the pressure reducer and also the analytical units from contact with any liquid sampled with the gas. If the probe cannot be installed downstream of a gas/liquid separator in the line, the devices presented in figures 5 and 6 may be used to stop non-gaseous materials.

8.4.1 Separators

Separators (or "drip pots") are generally not recommended in sampling systems. They may however be used to ensure that any free liquids that may have been collected by the sample probe do not enter the analyser or sampling cylinder. Use of this apparatus can create serious inaccuracies if no precautions are taken to ensure that the sample is taken at line temperature. Ideally, a separator would not be required in a single-phase line. Care shall be taken whenever one is used. Heat tracing or insulation may be useful on lines out of separators or drip pots to eliminate condensation. If ambient conditions are cooler than the stream to be sampled and the stream is near the dewpoint, the entire sampling system may need to be insulated to ensure representative samples. If possible, no mechanical devices, filters or absorptive materials that promote condensation or absorption shall be allowed.

8.5 Pressure reducers

In order to feed the analytical unit with sample gas at the appropriate pressure, a pressure reduction device is often required.

Depending on the pressure in the pipe and the pressure drop along the transfer line, it may be more appropriate to reduce the pressure at the beginning of the line, at the end, or not at all.

Pressure reducers should preferably be made of stainless steel and PTFE.

Pressure regulators shall have a pressure rating that exceeds the maximum expected line pressure of the gas-sampling system.

Due to the Joule-Thompson effect, the temperature will fall by about 0,5 °C/0,1 MPa during pressure reduction, and consequently there is a potential for condensation of heavy ends.

If this occurs, the sample is no longer representative, and shall consequently be rejected. The normal way of preventing this fall-out is by heating to compensate for the temperature drop. The heat is applied upstream of the pressure reduction device. The system (see figure 7 for an example) shall be designed so that no condensation is taking place at any point. The amount of heat energy required will depend on the gas composition, pressure reduction, pressure and temperature, flow rate, etc.

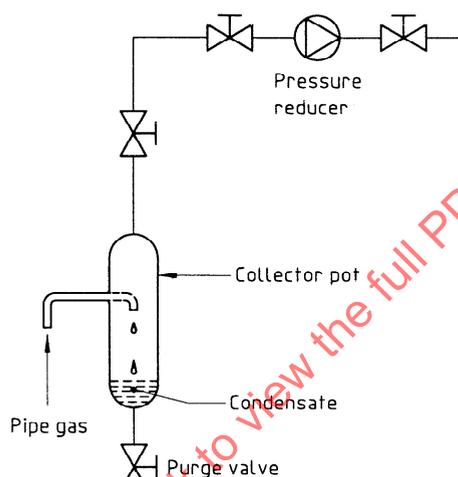


Figure 5 — Drip pot installation

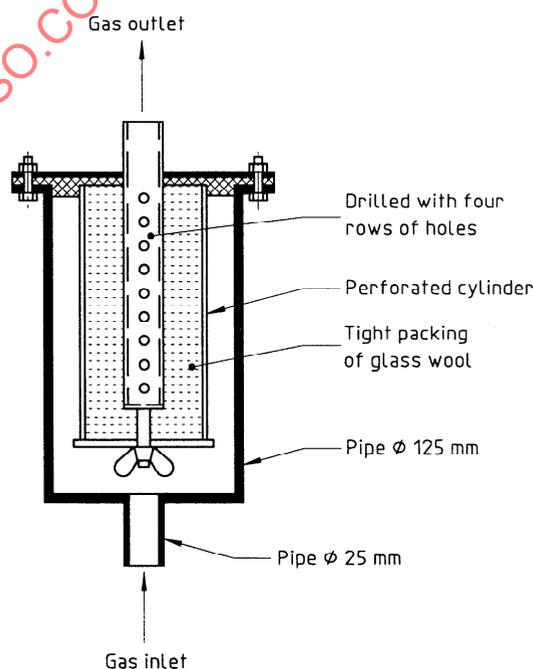


Figure 6 — Separator

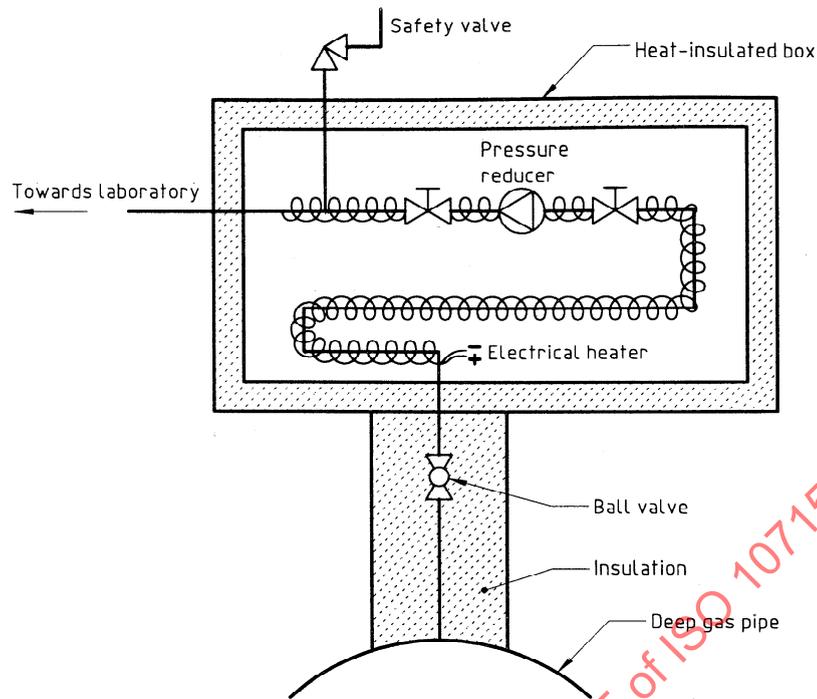


Figure 7 — Heat box for pressure reduction in sampling

8.6 Heating devices

Heating elements may be installed on the sample probe and sample lines. In some cases, heating the sample cylinder is also required.

Electrical heating elements shall be of the self-limiting type. They shall also meet the requirements of electrical codes for the area in which they are used. These requirements are needed to ensure that a heating element does not overheat if a failure occurs in the electrical components.

8.7 Sample containers

8.7.1 General considerations

The sample container shall not alter the gas composition in any way or affect the proper collection of the gas sample. The materials, valves, seals and other components of the sample container shall all be specified with this main purpose in mind.

Containers for sampling are usually made of glass (for very low pressures, overpressure below 0,2 MPa), stainless steel, titanium alloy or aluminium alloy. Special internal coatings for metallic containers ensure minimal reactivity with sulfur compounds. These internal coatings shall be specified. Unless the containers are vacuum-sealed, they shall be equipped with at least two valves, allowing purging of the gas sample. The container surface in contact with the gas shall be free from grease, oil or any other polluting product. They shall be carefully cleaned to avoid absorption phenomena. Annex B describes a cleaning procedure.

Soft-seated valves are recommended over those having metal-to-metal seats.

8.7.2 Floating-piston cylinders

The container required for this method is constructed of metal tubing, honed and polished on the inside surface. The cylinder is preferably closed with removable end caps to provide access for removal and servicing of the moving piston. The end caps are drilled and tapped for valves, gauges and relief valves. Figure 8 gives an example of a floating-piston sample cylinder.

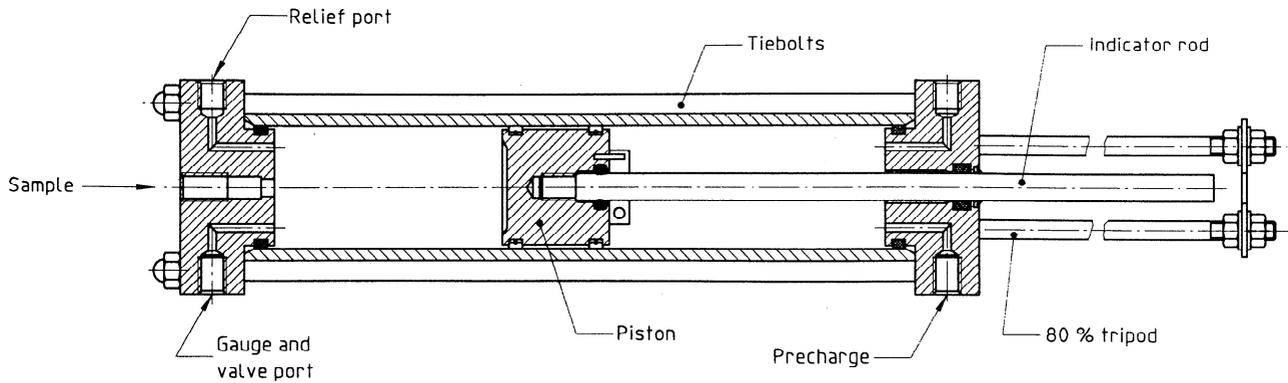


Figure 8 — Floating-piston cylinder

8.8 Incremental samplers

There are two general classes of commercial incremental sampler:

8.8.1 Regulator samplers

A specially designed pressure regulator increases the pressure of the collected sample in a sample cylinder from zero to a maximum of line pressure during the sample period. Such samplers are not recommended for low-pressure lines or widely variable flow rates.

8.8.2 Displacement samplers

The precharge gas in a floating-piston cylinder is displaced stepwise by the sample pumped into the cylinder at constant line pressure during the sampling period.

9 Spot sampling

This clause specifies a method of indirect sampling in which a suitable cylinder is filled with the sample. The sample is subsequently transported to the place of analysis.

Annex C on low-pressure sampling describes a method of obtaining spot samples from a low-pressure natural-gas distribution system using a glass vessel.

Methods suited for high- and low-pressure spot sampling are:

- fill and empty
- controlled rate
- evacuated container
- helium pre-fill
- floating-piston cylinder

9.1 Fill-and-empty method

This method is applicable when the sample container temperature is equal to or greater than the source temperature. The source pressure shall be above atmospheric pressure. A detailed example procedure is given in annex D.

9.2 Controlled-rate method

In this method, a needle valve is used to control the sample flow rate.

This method is applicable when the sample container temperature is equal to or greater than the source temperature. The source pressure shall be above atmospheric. Annex E gives a detailed example of this method.

9.3 Evacuated-cylinder method

In this method, a previously evacuated cylinder is used to gather the sample.

This method is applicable when the source pressure is above or below atmospheric pressure and the source temperature is greater or less than the sample container temperature.

The valves and fittings on the sample cylinder shall be in good condition and there shall be no leaks. Annex F gives an example of a detailed procedure for the evacuated-cylinder method.

9.4 Helium pre-fill method

This is similar to the evacuated-cylinder method except that a helium pre-fill is used to keep the container "air free" prior to sampling. It is used in those cases when helium is not to be measured, and preferably can be ignored, for example analysis by gas chromatography with helium carrier gas.

9.5 Floating-piston cylinder method

It is generally agreed that a sample drawn into a floating-piston cylinder at pipeline pressure and with heat-traced sample lines, where appropriate, gives analytical results that agree closely with a proper on-line analysis.

10 Direct sampling

10.1 General lay-out

Figure 9 shows an example of a direct-sampling system as described in this International Standard.

10.1.1 Automatic drainage

Automatic drainage is generally not acceptable, as it could bias the sample.

10.1.2 Reducing the pressure

In gas analysis, some measurements are carried out at line pressure (e.g. potential condensate or dewpoint measurements), and some measurements are carried out at reduced pressure. If pressure reduction is necessary, a pressure reducer is installed. The reducer is sometimes equipped with two valves, one on each side.

10.1.3 Inert-gas purging

Downstream of the pressure reducer, provision is made for inert-gas purging.

10.1.4 Transfer line

In the case of trace component analysis or dewpoint measurement, the transfer line shall be heated to a temperature equal to or higher than the gas line temperature. The sampling line is equipped with a temperature indicator upstream of the analyser house to sense the temperature. In cold environments, a drain may also be installed to detect any condensation.

10.2 Purging

The system shall be equipped with facilities for inert-gas purging. Purging of the sample system may be required if, for some reason, condensation has taken place and also to remove air (oxygen) from the system prior to introducing process gas to avoid a hazardous condition.

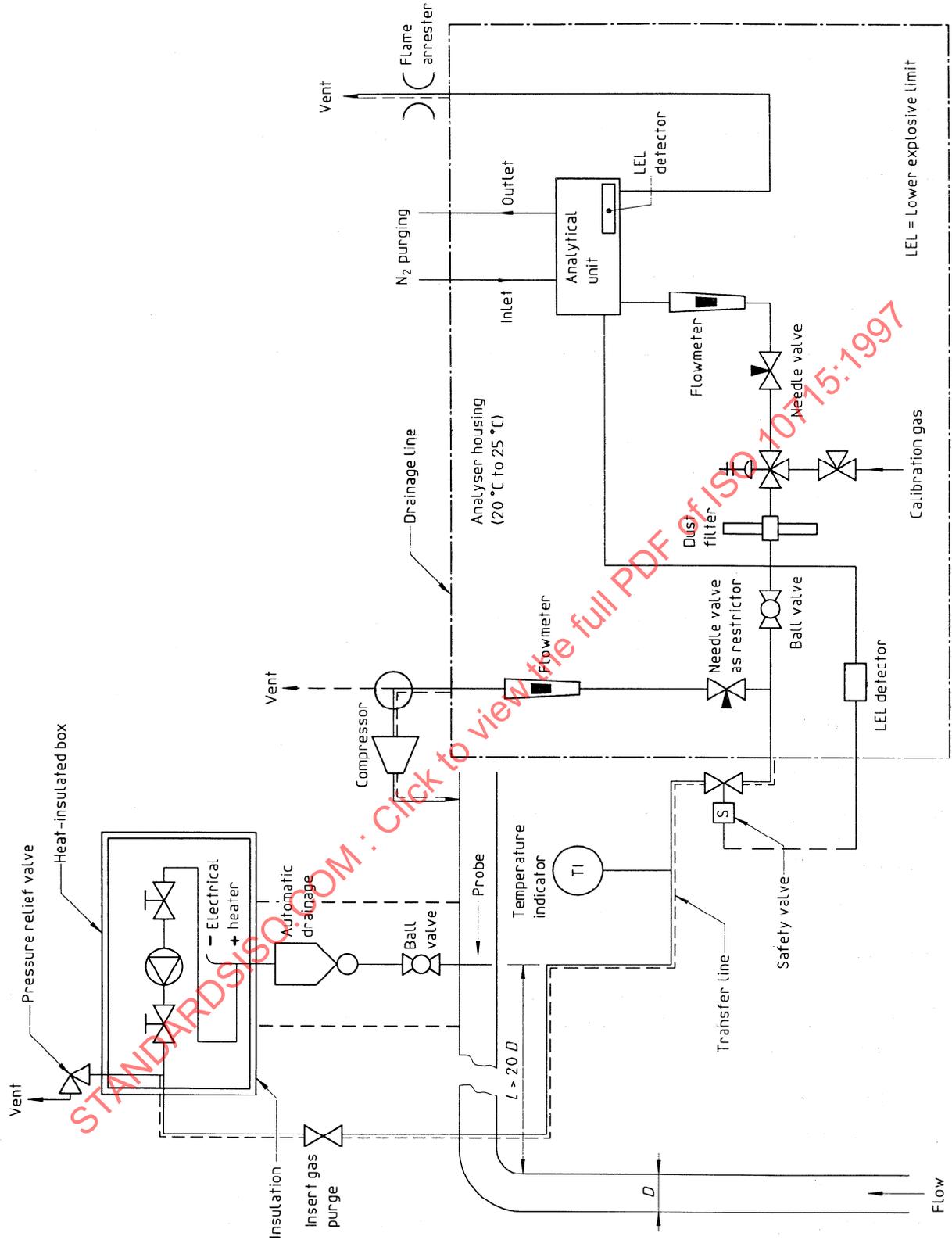


Figure 9 — Continuous sampling using a pressure reducer

10.3 Safety valve

A pressure relief valve shall be installed downstream of the pressure reducer, in order to protect the analyser from an uncontrolled increase in pressure should the reducer fail.

10.4 Heating of transfer line

As described in 9.6, the transfer line shall be heated if formation of liquid or adsorption of compounds may occur. The transfer line shall always be heated to a temperature at least 10 °C above the condensation temperature.

10.5 Transfer feed line

The line for the analytical unit shall be equipped with a ball valve for carrying out maintenance work. Downstream of this valve, a fine-dust filter will normally be included. Special attention shall be paid to ensuring that this filter does not change the gas composition. Calibration gas is introduced upstream of the dust filter. For sampling/analysing at reduced line pressure, a needle valve with a flowmeter shall be installed upstream of the analyser, and also in the calibration gas feed line.

In the case of sampling/analysing at line pressure, the flow rate regulation device needs to be installed downstream of the analytical unit. To control upstream pressure and also to avoid venting high-pressure gas, a pressure reducer with upstream pressure regulation shall be installed at the vent line of the analytical unit.

10.6 Security of analyser housing

NOTE — As an example, the following measures could be taken: A lower explosive limit (LEL) detector could be installed in the analyser housing. In the event of gas leakage, the detector will give a signal to close the safety valve upstream of the housing and also to shut off the electric power to the analytical unit. The detector may be adjusted for instance to 10 % or 20 % of the LEL value in accordance with local safety regulations.

Another safety precaution is to have a flame arrester downstream of the analytical unit, avoiding an explosion outside the analyser house. This is a possibility if air is introduced in the gas by mistake.

The design of the analyser housing and its equipment shall comply with local safety regulations.

11 Incremental sampling

11.1 General considerations

The sampling system shall provide a sample representative of the gas flowing in the pipeline. Flow rates and compositions may vary with time, so the interval between the sampling increments shall be carefully chosen so that the collected sample reflects these changes.

11.2 Intervals

If possible, flow-rate-proportional sampling shall be used for incremental-sampling systems. It is especially important to use flow-proportional sampling if both the flow rate and the composition change. For example, if the flow is stopped and the sampler continues to collect a sample, then the composite sample will have some part of its gas collected when no gas flow was present. If the composition during this period is different from the average composition, the sample will not be representative.

Time-proportional sampling may be used and will provide representative samples only if the flow rate is steady over the sampling interval or if the composition is stable over the sampling interval.

There are several incremental samplers commercially available. Such units may be controlled by a timer or a flow-proportional signal from the flow computer.

11.3 System considerations

A recommended incremental sampler is the displacement type which pumps a sample into a floating-piston cylinder at constant line pressure.

The sample line between the sampling device and the collection cylinder shall be of minimum length. Except for very dry gases, the sample line and sampler shall be heat-traced and insulated to avoid sample condensation.

Samplers shall be designed so that they allow a continuous and uninterrupted flow of gas through them and thus pump a representative sample increment into the sample container.

11.4 Monitoring the filling process

The filling process shall be monitored daily.

11.5 Cylinder tracking

All information important to the laboratory shall be on a label with the cylinder. Labels shall be securely attached to the sample cylinders, but shall not interfere with the utilisation of the cylinder.

Information attached should preferably include:

the cylinder number

the cylinder type

the location of sampling

all details necessary for identification of the pipe sampled

the date and time, or period, of sampling

the method of sampling

the actual destination of the cylinder

any need for maintenance on the cylinder (e.g. leakage)

any information relevant to the analytical lab concerning the sample

the sample pressure, if a pressure gauge is not an integral part of the sampling cylinder

the line static pressure

the temperature of the gas stream

the differential pressure

any field remarks

Annex A (informative)

Use of a block valve in direct sampling

A difficult point in sampling is the installation of calibration gas manifolds in the sample line. Single-valve connections are avoided, because a leak across the valve when in the closed position cannot be detected, and calibration gas leaking into the sample or *vice versa* is unacceptable.

This problem is solved if every calibration gas line, and the sample gas line itself, has a double shut-off valve with an in-between bleed to the atmosphere. This ensures that no cross-contamination can occur between the calibration gas and the sample gas. This method is usually referred to as the double-block and bleed (DBB) system.

Application of this principle for one calibration gas is given in figure A.1.

When valves A and B are closed and C is open, any gas leaking across A will pass through C to vent.

A single-calibration-gas, single-sample manifold is given in figure A.2. A multigas manifold is given in figure A.3.

All valves are mounted directly in the tees in order to avoid extra dead volume in the unused branch of the T-piece.

All supplementary lengths of tubing in such an arrangement are only purged through diffusion and are therefore to be avoided.

The recommended valves are toggle valves, for which air actuators are available as standard, so that the manifold can readily be automated.

With a proper choice of "normally open" and "normally closed" actuators, a logical gas transmission to the analyser can be selected, with full fail-safe provision.

A non-venting, spring-loaded, pressure safety valve is incorporated in the sample line after the last calibration gas connection and before the analyser.

This safety device protects the analyser against the malfunctioning of pressure reducers on high-pressure calibration gas cylinders.

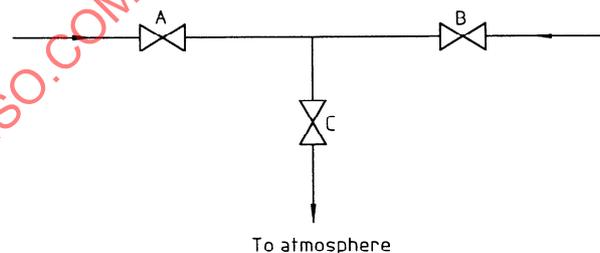


Figure A.1 — Principle of double block and bleed

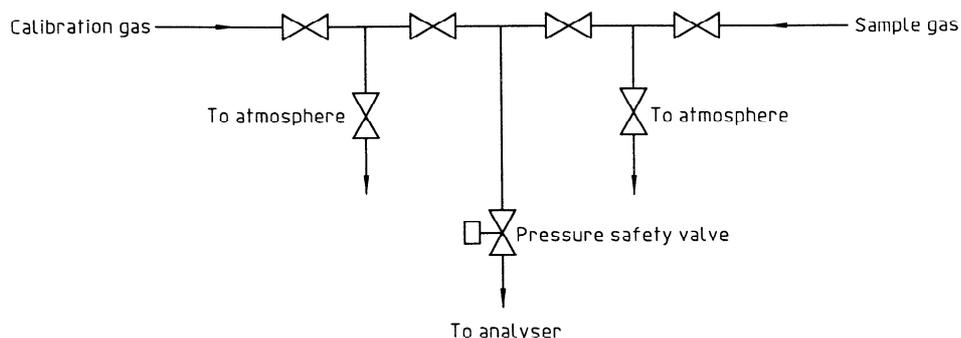


Figure A.2 — Single-sample, single-calibration-gas manifold

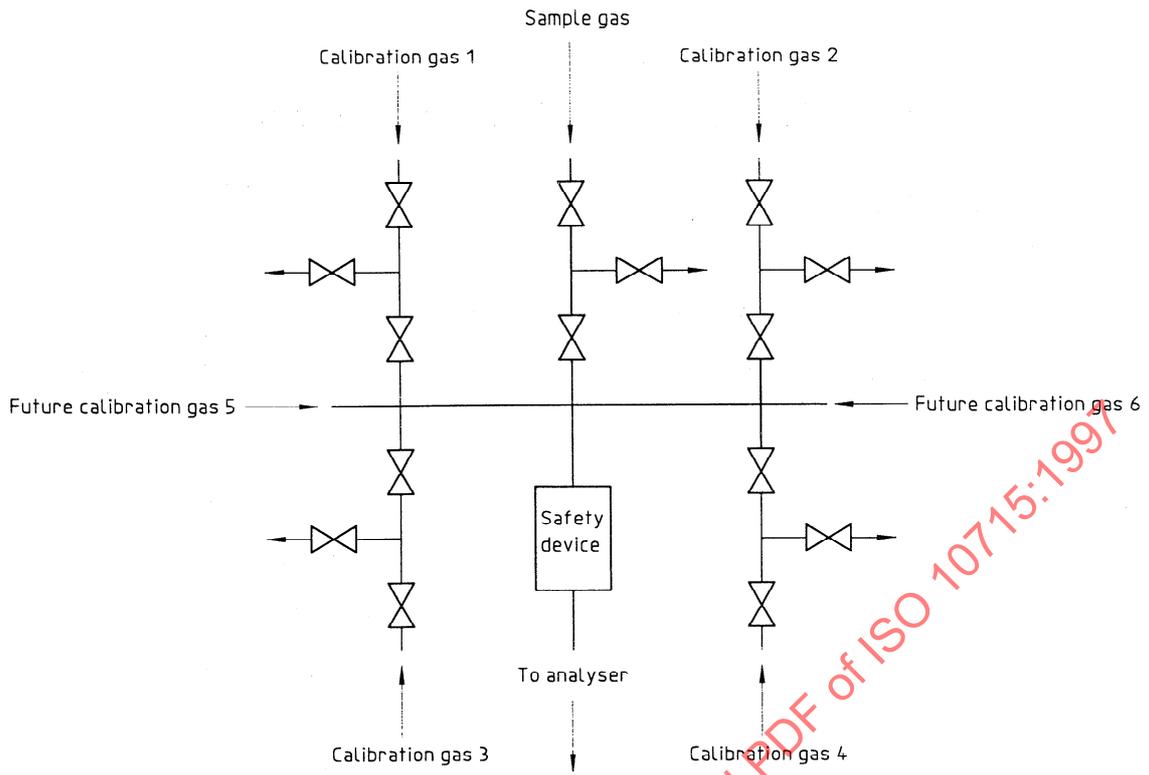


Figure A.3 — Multigas manifold

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Annex B (informative) Cleaning of steel sampling cylinders

An example of a very thorough cleaning procedure is given below:

Vent off any residual sample gas.

Evacuate or purge with nitrogen.

Fill the cylinder with a cleaning product, e.g. acetone.

Shake the cylinder on a shaking machine for 2 h.

Transfer the acetone to a suitable receptacle.

Fill again with fresh acetone and replace the cylinder on the shaking machine for 2 h.

Remove the acetone, drying with nitrogen or dry air.

Dry the cylinder further in a hot-air oven at 90 °C. If the cylinder is equipped with only one valve, evacuate the cylinder during the drying operation. If it is equipped with two valves, purge it with nitrogen during the drying. The drying operation takes approximately 12 h.

After cooling, fill the cylinder with nitrogen and empty it three times.

Afterwards, fill the cylinder with nitrogen to a pressure of 1 MPa.

Wait for 2 h and check by chromatography for the presence of acetone and other impurities.

Keep the corresponding chromatogram with the cylinder documents.

WARNING — Acetone is a highly flammable liquid and must therefore be handled carefully.

NOTE — The amount of chromatographic checking may be reduced by using a statistical approach.

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Annex C (informative)

Procedure for low-pressure sampling into glass cylinders

C.1 Specific safety precautions

Check that the sample cylinder (see figure C.1) has no cracks. It is recommended that a flexible sleeve be used around the sample cylinder. Normally, this is not necessary for a line pressure of 0,5 kPa to 10 kPa, but there is always a risk that the line pressure will be somewhat higher than 10 kPa.

Use safety goggles during sampling.

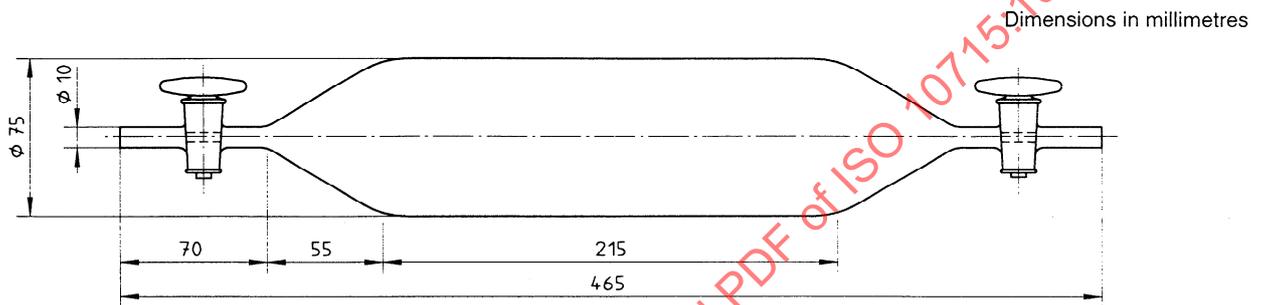


Figure C.1 — Glass sample container (1 litre)

C.2 Preparation of the glass cylinder

Lubricate the stopcocks of the sample cylinder with silicone grease.

Clean the sample cylinder using a potassium hydroxide soap solution.

Rinse afterwards with distilled water.

Dry the cylinder with hot air (water- and oil-free).

When hydrogen sulfide is to be analysed in the gas sample, rinse the sample cylinder with 0,01 mol/l H_2SO_4 .

C.3 Sampling

For a typical sampling arrangement, see figure C.2. (Figure C.3 shows a set-up for sampling from sub-atmospheric pipelines.)

Measure the pressure at the sampling point. Make sure that the sample transfer line overpressure does not exceed 0,2 MPa.

Connect the sample transfer line to the sample cylinder.

Connect the inlet of the cylinder as close as possible to the sampling point, using the sample transfer line.

Connect the outlet of the sample cylinder to the inlet of a flowmeter.

Connect the outlet of the flowmeter to a vent or a flare pipe.

Open the two stopcocks of the sample cylinder in the order of the direction of the gas flow.

Adjust the flow through the sample cylinder by means of the flowmeter.

Vent gas.

Note the presence of liquid fall-out during venting.

Discard the sample in the event of considerable amounts of liquid occurring.

Close the sample cylinder after 30 min of purging, closing the stopcocks in the order opposite to the direction of the gas flow so that the pressure builds up.

Disconnect the sample cylinder.

Secure the stopcocks with clamps against unintended opening and check for leaks.

C.4 Preparation for transport

Use a suitable box for transporting the sample cylinder.

Note whether grease, hydrocarbon condensate, dust or rust and/or water drops have been entrained in the gas flow.

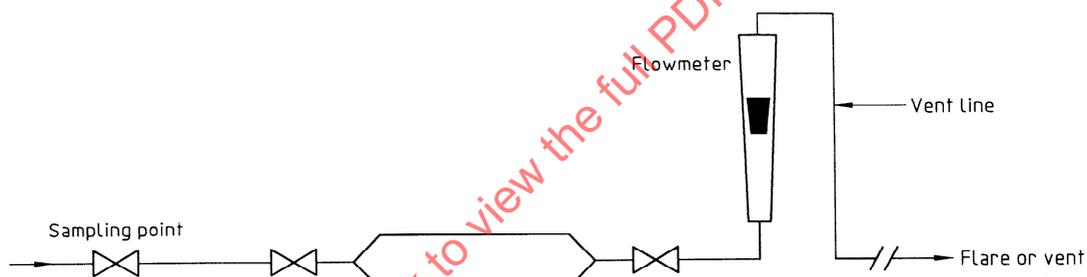


Figure C.2 — Sampling into glass cylinders

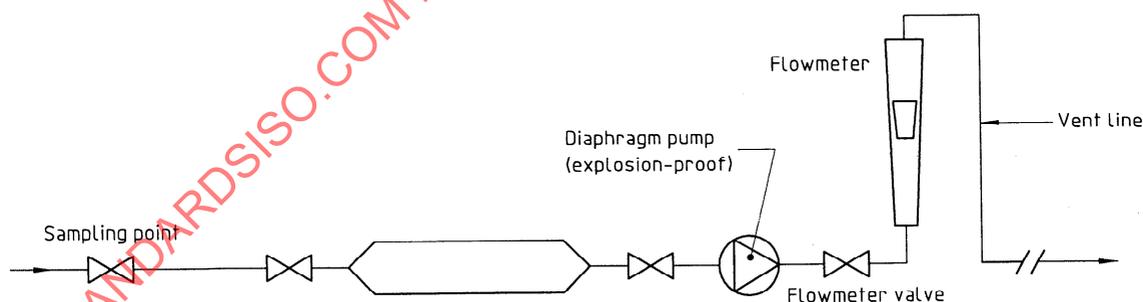


Figure C.3 — Sampling into glass cylinders from a sub-atmospheric pipeline

Annex D (informative)

Procedure for sampling by the fill-and-empty method

The equipment is arranged as shown in figure D.1. The extension tube has a length of 0,6 m to 1,2 m. All materials, including the tubing, are of stainless steel. The extension tube may be coiled to allow the sampling apparatus to be more compact. This extension tube is needed to prevent heavy-hydrocarbon condensation in the sample container outlet valve.

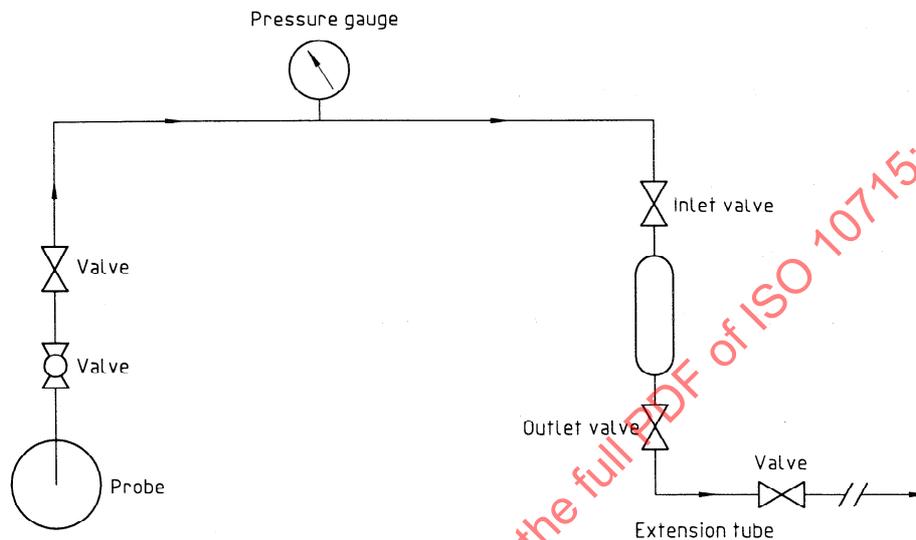


Figure D.1 — Fill-and-empty method

The procedure for sampling by this method is as follows:

Install the sample probe.

Connect up the sample line.

Open the valve at the sampling point and thoroughly blow out any accumulated material.

Connect one end of the sample container through the sampling system to the gas source.

Purge the line and container slowly with gas to displace the air.

Close the extension line valve and allow the pressure to build up rapidly to the selected container pressure.

Close the inlet valve and slowly vent the container through the extension tube valve until it reaches atmospheric pressure.

Open the inlet valve.

Repeat the last two steps for a number of cycles (see table below) to effectively purge the container of the original gas in the container.

Observe for traces of liquid at the discharge tube end.

After the last cycle, first close the extension tube valve and, after the pressure has built up to the selected container pressure, also close the sampling valve.

Note the container pressure.

Note the source temperature.

Close the container inlet and outlet valves.

Depressurize the sample line.

Remove the sample container.

Check for leaks by immersion of the valves in water, if possible, or use leak detector soap solution.

Plug the valves.

Final pressure in cylinder

MPa

0,1 to 0,2

0,2 to 0,4

0,4 to 0,6

0,6 to 1

1 to 3,5

≥34

Number of purge cycles

13

08

06

05

04

03

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Annex E (informative)

Procedure for sampling by the controlled-rate method

The following precautions are to be observed when sampling by this method:

- a) The source pressure must be sufficient to produce stable flow conditions in the flow tube. The pressure in the extension tube has to be 0,1 MPa or higher when venting to atmospheric.
- b) The valves and piping in the sampling apparatus are sized large enough to allow sufficient flow upstream of the flow tube.

The sampling arrangement is shown in figure E.1

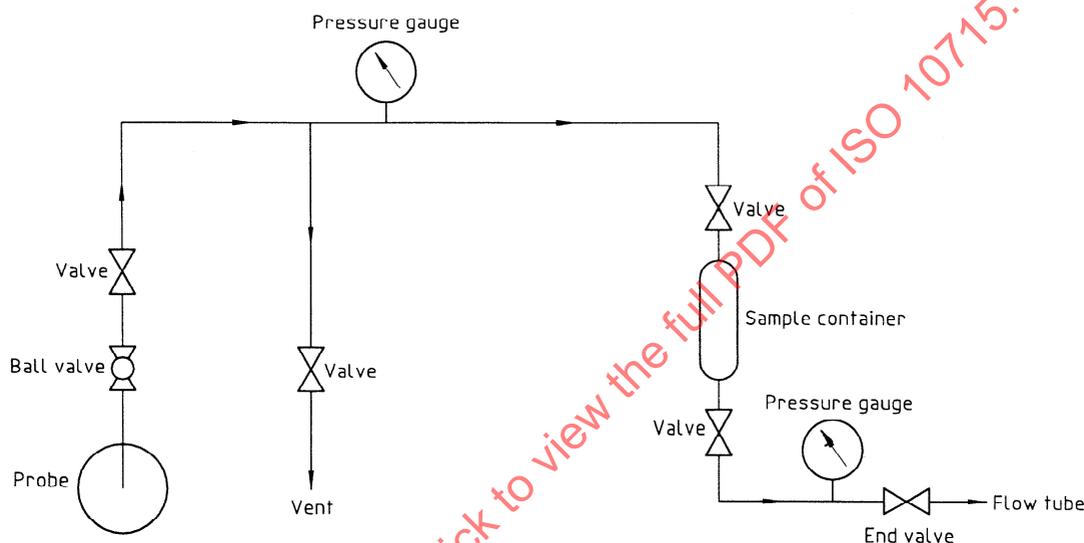


Figure E.1 — Controlled-rate method

The procedure for sampling by this method is as follows:

Install a sampling probe and purge the probe with the natural gas.

Choose the most suitable sampling unit. This depends on the sampling pressure. In most cases, a set suited for 0,8 MPa to 3 MPa, or a set suited for 3 MPa to 7 MPa may be applied.

Now install the sample container.

Open the sample valve and purge the sample container.

Close all the valves.

Open the ball valve and sample valve slowly.

Slowly open the vent valve a little.

Close the sampling valve and wait until the pressure in the sampling line is near atmospheric. Repeat this purging procedure three times.

Close the vent valve.

Open the inlet valve slowly and then bring the sampling container up to the prevailing pressure.

Open the container outlet valve.

Open the end valve.

Purge for at least 1 min.

Note, during purging, the temperature of the natural gas and the inlet and outlet pressure of the gas stream through the container.

Close the end valve.

Close the outlet valve.

Close the inlet valve.

Close the sample valve.

Note the container pressure and the service temperature.

Open the vent valve and wait till the pressure falls to atmospheric.

Remove the sampling unit and probe, and check the sample container for leaks by immersion of the valves in water, if possible, or by using leak detector solutions.

Plug the valves.

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