
Soil quality — Sampling —
Part 7:
Guidance on sampling of soil gas

Qualité du sol — Échantillonnage —

Partie 7: Lignes directrices pour l'échantillonnage des gaz du sol



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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 10381-7 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 2, *Sampling*.

ISO 10381 consists of the following parts, under the general title *Soil quality — Sampling*:

- *Part 1: Guidance on the design of sampling programmes*
- *Part 2: Guidance on sampling techniques*
- *Part 3: Guidance on safety*
- *Part 4: Guidance on the procedure for investigation of natural, near-natural and cultivated sites*
- *Part 5: Guidance on the procedure for the investigation of urban and industrial sites with regard to soil contamination*
- *Part 6: Guidance on the collection, handling and storage of soil for the assessment of aerobic microbial processes in the laboratory*
- *Part 7: Guidance on sampling of soil gas*
- *Part 8: Guidance on sampling of stockpiles*

Introduction

ISO 10381-7 is one of a group of International Standards to be used in conjunction with each other where necessary. ISO 10381 (all parts) deals with sampling procedures for the various purposes of soil investigation. The stated soil-gas and landfill-gas measurements do not give any quantitative statement of the total quantity of material detected in soil gas or soil. The measurement results can be influenced by, e.g. temperature, humidity, air pressure, minimum extraction depth, etc.

The general terminology used is in accordance with that established in ISO/TC 190 and, more particularly, with the vocabulary given in ISO 11074-2.

In addition to the main components (nitrogen, oxygen, carbon dioxide), soil gas can contain other gases (methane, carbon monoxide, mercaptans, hydrogen sulfide, ammonia, helium, neon, argon, xenon, radon, etc.). It can also contain highly volatile organic compounds or inorganic vapours (mercury) which are of special interest within the framework of investigating soil and groundwater contamination.

Due to the different physical properties and ranges of concentrations of gases in soil and landfills as well as the wide variety of objectives for soil-gas sampling, this part of ISO 10381, after the general clauses 1 to 4, is subdivided into two sections:

- a) permanent gases of soil gas and landfill gas (Clause 5); and
- b) volatile organic compounds (VOCs) (Clause 6).

Thus it is inevitable that some details are repeated in both clauses in order to make the guidance comprehensive.

Soil quality — Sampling —

Part 7: Guidance on sampling of soil gas

WARNING — This part of ISO 10381 concerns on-site soil and sub-soil gas analysis requiring particular health and personal safety precautions.

1 Scope

This part of ISO 10381 contains guidance on the sampling of soil gas.

This part of ISO 10381 is not applicable to the measurement of gases from the soil entering into the atmosphere, the sampling of atmospheric gases, or passive sampling procedures.

2 Normative references

The following referenced documents are indispensable for the application 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 10381-1, *Soil quality — Sampling — Part 1: Guidance on the design of sampling programmes*

ISO 10381-2, *Soil quality — Sampling — Part 2: Guidance on sampling techniques*

ISO 10381-3, *Soil quality — Sampling — Part 3: Guidance on safety*

ISO 11074-1, *Soil quality — Vocabulary — Part 1: Terms and definitions relating to the protection and pollution of the soil*

ISO 11074-2, *Soil quality — Vocabulary — Part 2: Terms and definitions relating to sampling*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 11074-1 and ISO 11074-2 and the following apply.

3.1

active soil-gas sampling

sampling by extracting a certain volume of soil gas

3.2

biodegradation

the physical and chemical breakdown of a substance by living organisms, mainly bacteria and/or fungi

3.3

borehole

hole formed into soil or landfilled material into which may be installed a standpipe to enable gas monitoring to be carried out

NOTE A borehole is also used as a means of venting or withdrawing gas.

3.4

concentration/adsorption method

method in which substances to be determined are concentrated adsorptively on an adsorbent (e.g. activated charcoal or XAD-4 resin), subsequently desorbed and analysed

3.5

dead volume

volume which is present between the suction opening of the soil-gas probe and the sampling vial, including the volume of the sampling vial or of the absorption tube

3.6

direct method

direct measuring method

method of analysis where the soil-gas sample (aliquot) is directly introduced into a suitable equipment without first being concentrated and subjected to analysis

3.7

direct-reading detecting tube

glass tube filled with reagents which, after drawing through certain gaseous compounds, show concentration-dependent chromophoric reactions and which are thus used for qualitative and semi-quantitative analyses as well

NOTE It is important that attention be paid to cross-sensitivities.

3.8

gas migration

movement of gas and vapour from the wastes within a landfill or through the ground to the adjoining strata, or emission to the atmosphere

3.9

gas monitoring well

standpipe suitably installed inside a borehole from which gas samples can be taken to measure soil-gas concentrations and to monitor changes in composition of soil gas or soil-gas migration

3.10

gas sampling

collection of a proportion of material for testing such that the material taken is representative of the gas in the pore space of the location of sampling

3.11

landfill

deposition of waste into or onto the land as a means of disposal

NOTE It can eventually provide land which may be used for another purpose.

3.12

landfill gas

mixture of permanent gases (main components), dominated by methane and carbon dioxide, formed by the decomposition of degradable wastes within landfill sites

NOTE It can also include a large number of VOCs (trace components).

3.13**lower explosive limit****LEL**

lowest percentage (volume fraction) of a mixture of flammable gas with air which will propagate an explosion in a confined space at 25 °C and atmospheric pressure

3.14**one-stage soil-gas sampling**

sampling of soil gas directly from a soil-gas probe placed in soil, without pre-drilling

3.15**passive soil-gas sampling**

sampling based on the adsorption of soil gas on an absorbent placed in soil, without employing negative pressure

3.16**permanent gas**

element or compound with boiling point below – 60 °C at atmospheric pressure

3.17**sample volume**

volume of soil from which the soil-gas sample is taken

3.18**soil gas**

gas and vapour in the pore spaces of soils

3.19**soil-gas monitoring device**

borehole finished with suitable material for stabilisation of the borehole wall and/or for limiting the sampling area.

NOTE Depending on the type and stability of fitting, a difference is made between temporary (for single or short-term repeated soil sampling) and stationary (for long-term observations) soil-gas measuring points.

3.20**soil-gas probe****soil-gas sampling probe**

probe, generally a tube, which is installed directly into soil (one-stage soil-gas sampling), or in a borehole (two-stage soil-gas sampling) to take soil-gas samples.

NOTE By applying a negative pressure to the upper end of the soil-gas probe (head), the soil gas at the lower end (tip) is drawn through the suction opening(s) and transferred to a gas collecting equipment and online measurement equipment (direct measuring method) or to an absorbent (concentration method), which are installed either in or at the head of the soil-gas probe or subsequently used.

3.21**soil-gas suction test**

continuous soil-gas sampling from a borehole well over a controlled longer period of time (mostly several hours up to days) to observe the variations over time of the gas concentrations and of the pressure distribution in the soil

3.22**two-stage soil-gas sampling**

sampling done firstly through installation of a borehole with the aid of a drilling instrument or by small boring, and secondly by sampling of soil gas from a soil-gas probe installed in the borehole

3.23

volatile organic compound

VOC

compound which is liquid at room temperature (20 °C) and which generally has a boiling point below 180 °C

EXAMPLES single-ring aromatic hydrocarbons and other low boiling halogenated hydrocarbons, which are used as solvents or fuels, and some degradation products.

4 Preliminary points to be considered

The choice of sampling technique shall be consistent with the requirements of the investigation (including subsequent analytical procedures). Consideration should also be given to the nature of ground under investigation, as well as the nature and distribution of contamination, the geology and the hydrogeology. Every effort should be made to avoid cross-contamination and at no point should underlying aquifers be put at risk.

Before intrusive works begin, a comprehensive check should be made of the ground to ensure that no services or structures are at risk and no hazards are present. (For more information on sampling techniques and safety, see ISO 10381-2 and ISO 10381-3.)

When sampling soil gas close to the surface, the effect of ambient air penetration needs to be considered. The sampling depth is determined by the presence of impermeable cover over the ground surface, the soil type (porosity, clay content, etc.) and the depth of bedrock. It is considered unlikely that useful samples can be collected at depth less than 0,5 m. For routine monitoring of soil gas, a minimum depth of 1 m is recommended.

Circumstances in cold conditions make soil-gas sampling difficult in many ways. Ground frost greatly limits the mobility of gas in soil and should be considered in planning and carrying out sampling as well as in interpreting the measuring results. Similarly water saturated ground can limit mobility.

The main problem with soil-gas sampling below the frozen ground is the loss of air-filled porosity due to the high moisture content in the zone between frozen and unfrozen parts of the ground. Consequently the samples shall be taken from greater depths.

All buildings constructed on unfrozen ground act as pathways or barriers for upwards soil-gas migration. Underpressure and differences in concentration in the buildings can also assist gases to penetrate the basements of buildings.

Pressure effects caused by the rise of warm air within buildings can assist the entry of gases into buildings.

Some organic pollutants in the gas phase in soil and sub-soil can present toxicological risks of varying severity. Due to this possibility, personnel should be equipped, according to the potential toxicity (assumed or measured), with suitable protective material.

Certain organic fumes can form explosive mixtures with air. (Explosivity limits and self-ignition temperatures should be taken into account.). It is therefore appropriate to use electrical equipment and tools which are suitable for use in explosive atmospheres.

Health and safety issues should be considered at all times. Training should be given to ensure that personnel understand the necessary precautions. (For more information on safety, see ISO 10381-3.)

5 Permanent gases

5.1 Investigation objectives

5.1.1 Soil gas

The objectives of the investigation for permanent soil gases are

- analysis of soil-gas composition, and
- determination of the difference of concentration on a site.

5.1.2 Landfill gas

The objective of the investigation for landfill gases is

- analysis of landfill gas composition.

5.1.3 Further objectives

Further objectives may be

- assessment of possible reasons for plant growth inhibition,
- optimization or control of sealings or gas collecting installations,
- rough estimate of gas production potential and duration of gas production,
- detection of underground combustion,
- design of gas protection measures for buildings.

5.2 Basic principles

5.2.1 Physical and chemical principles

Wherever biodegradable material is present in landfill sites or within the soil matrix of the made ground beneath a brownfield site, microbial activity will produce landfill gas. Similar gas can also be produced in alluvial deposits and degrading natural organic material (see Annex B). Landfill gas consists primarily of methane and carbon dioxide (at a ratio of approximately 60:40). Depending on microbial activity, this ratio can change. A number of additional trace gases can be present.

Permanent gases can also originate from coal deposits, peat, natural deposits (e.g. chalk and alluvial deposits), from leaks of mains gas (natural gas) and from sewer gas. Information on techniques for identifying the origin of gas can be found in 5.2.3.

Methane is explosive at concentrations of between 5 % and 15 % (volume fraction) in air; below 5 % there is insufficient gas to support combustion and above 15 % (volume fraction) there is insufficient oxygen to support combustion. Carbon dioxide is an asphyxiant and can cause adverse health effects in concentrations greater than 0,5 % (volume fraction).

Landfill gas is usually saturated with moisture and is corrosive. It can cause vegetation to die back due to the elimination of oxygen from the plant's root zone or to the presence of phytotoxic compounds. Its density depends upon the ratio of carbon dioxide to methane: the higher the ratio of carbon dioxide the greater the density.

Gas pressure within the sub-surface is dependent on the gas generation rate, the permeability of the waste mass and the surrounding strata, and changes in the level of leachate or groundwater within the site. Other important factors are temperature and atmospheric pressure.

Depending upon site engineering and local geology, gas can migrate considerable distances and can present a hazard to nearby developments. In the case of mine gas, the cessation of water pumping can lead to a rise in water table levels which can increase the gas pressure, and consequently increase surface gas emissions. It is therefore important to gain an understanding of gas concentrations and flow rates to establish the potential for gas migration off-site or atmospheric emissions.

5.2.2 Ambient conditions

It is important during the monitoring of a site, that atmospheric conditions, for 3 to 4 days before and during the sampling, be recorded. Local climatic conditions at the time of monitoring should also be recorded. This information can help in the interpretation of the data. The most important parameters to record are

- atmospheric pressure, and
- rainfall.

Other useful parameters are

- temperature (ambient air and soil gas),
- wind speed/direction, and
- water table depth.

During dry periods the ground can crack, especially if clay is used to cover sites. This will lead to an increase in gas emissions at the surface. In periods of wet weather, the clay will become wet and swell, and cracks will be sealed. This will reduce surface gas emissions and can lead to increased gas concentrations and increased lateral migration. A measurement of soil permeability and moisture content can be helpful in assessing these effects.

A rising water table, caused by rainfall for example, can put the gas under pressure and force it to the surface; however, it can also block migration pathways. The saturation of superficial soils can restrict the venting of landfill gas to atmosphere. This can result in variations in gas pressure and concentrations.

Falling atmospheric pressure can increase emission rates. Rising atmospheric pressure can have the opposite effect. The magnitude of this effect depends upon the soil permeability and the rate at which the pressure changes.

In general, however, it can be difficult to establish the cause of changes in concentrations and emissions since they may be due to a combination of the above factors.

5.2.3 Identifying the source of gas

Identifying the origin of the gas is important when making decisions regarding its monitoring and control. The composition of a gas may help identify the source. Examples are given below.

- Gas from a geological source may have a higher proportion of methane than landfill gas.
- Geologically-derived gas generally contains up to 15 % ethane and higher hydrocarbons, while biogenic methane contains only trace amounts.
- It may be possible to distinguish mains gas from other gases if the exact composition of the local mains gas is known. Mains gas may have odour compounds such as sulfides and mercaptans added to give the gas a distinctive odour; it may also contain long chain hydrocarbons such as octane and nonane. Helium is often removed from mains gas.

Landfill gas may also contain higher than normal concentrations of higher hydrocarbons if the waste contains substances that generate or release such gases and vapours.

Identification of different components may, however, be limited as the components may be affected by chemical changes occurring in the ground during migration, by solution in groundwater and by adsorption onto clays, etc.

Biogenic (formed by microbiological activity) methane and thermogenic (formed by thermal degradation of organic matter at higher temperatures and pressures) methane have different proportions of carbon isotopes carbon 12 and carbon 13 which can be measured to identify the origin of the gas. The technique, however, requires specialist laboratories.

5.3 General considerations for sampling

The strategy should be site specific and should be based upon the particular conditions of the site in question as well as on the information obtained from the site investigation (see Annex C).

It should be considered that any invasive activity can affect migration patterns and will act as a pathway for the gas.

In addition to gas monitoring, boreholes are also useful for obtaining hydrogeological, geotechnical and contamination information and are therefore a useful multi-purpose tool.

If gas concentration measurements are required at different depths the use of multi-level boreholes is undesirable and multiple well installations are to be preferred.

When results shall be compared to others and especially when monitoring from standpipes, the technique used should be consistent to ensure comparable results between different operators, techniques and over different monitoring periods. To achieve this, quality assurance measures as given in 5.10 need to be followed.

Gas concentration measurements may be taken using portable equipment (see Table E.1) or samples may be taken for off-site laboratory analysis. It is advisable to collect gas samples, to be submitted for confirmatory analysis in a laboratory, in order to verify the on-site monitoring results.

5.4 Sampling requirements

5.4.1 Sampling options

Gas may be monitored using a range of different sampling techniques (see Table 1).

Although each technique has its uses, in situations where a detailed, long-term understanding of the site is required, monitoring wells installed in boreholes tend to be the most favourable option.

5.4.2 Borehole construction

During drilling of the borehole, the borehole atmosphere should be monitored with on-site equipment at 1 m intervals. Where ground water is encountered, useful information on the content of gas in the underlying ground can be obtained by measuring the gas concentrations immediately above the water level at 1 m intervals as the drilling progresses.

5.4.3 Location of sampling

The location and design of monitoring wells or other chosen technique should be planned well in advance, in accordance with the aims of the site investigation, the conceptual site model and considerations including health and safety, location of underground services, etc. (see Tables A.1 through A.5). A plan should be drawn up in detail and adhered to. Any changes to this plan should be noted.

In areas where contamination is thought to be severe, the drilling of boreholes can produce preferential pathways and as a result information should be sought on specialist drilling precautions.

The spacing of boreholes is dependent on the nature of the strata.

The depth from which samples are taken depends on the objectives. Information on concentrations at different depths is useful, as it allows for a better understanding of the propensity for the gas to migrate.

Table 1 — Options for sampling of permanent gases

Method	Description	Advantages	Disadvantages
Shallow probes	Hollow perforated pipe pushed into the ground and connected to a gas detector	Very quick Cheap Easy to install	Max. depth of 2 m Can become blocked Confirms gas presence but not absence
Auger	Hand-held auger is used to bore into the ground	Cheap and easy to use Allows unspecified sampling of solids Deeper than spiking/shallow probes	Physically difficult Cannot penetrate difficult ground Can be time consuming
Driven probes	Hollow tube with solid nose cone. Mechanically driven into the ground. Monitoring pipe installed inside tube. Tube extracted leaving behind nose cone	Minimal ground disturbance Easily portable thus access problems unlikely Max. depth of 10 m Allows soil-gas profile through the ground to be determined	Will not penetrate obstructions Can cause smearing in clayey soils which restricts gas ingress into the probe hole
Boreholes (without flushing device)	Cased borehole is sunk by cable percussion techniques into which a perforated standpipe is installed. The pipe is surrounded with gravel, and the casing withdrawn	Great depths attainable Minimal disturbance to ground Can install several standpipes in one borehole to measure at different depths Can take samples of strata at different depths during drilling Allows groundwater to be monitored Allows soil-gas profile through the ground to be determined	Relatively slow and expensive May have access problems Brings contaminated material to the surface Care is needed to avoid enabling contamination of an underlying aquifer
Boreholes (with flushing device)	Similar to above, but the hole is drilled by a rotary tool and flushed with air or water to aid rock penetration	As above but: — quicker than cable percussion — relatively mobile rig	As above but: — not intrinsically safe, sparks may be a hazard on a gassing site; — water flush can spread contamination — air flush can cause migration of soil gas Care is needed to avoid enabling contamination of an underlying aquifer Does not allow determination of soil-gas profile due to the effects of the flush

5.4.4 Sample volumes and sampling rates

In the case of point soil-gas sampling, a small volume is taken within the horizon layer (about 10 ml) for a detection, probably not affected by external parameters, of the pore volume filled with gas. When sampling larger volumes (up to several litres), the sampling area is diffuse and its location cannot be determined. Soil-gas sampling from a borehole with greater diameter than that of the probe is called "integrating", as the gas might be delivered over its entire length. In landfill gas sampling, larger sampling volumes should be taken to determine the gas components over a greater area.

The gas flow rate shall be ascertained. Several techniques for measuring gas flow rates, including their advantages and disadvantages, are listed in Annex D.

5.5 Technical equipment

5.5.1 General

Different instruments measure different gases over different concentration ranges, and each has its own advantages and limitations. It is important for the operator to obtain a good understanding of gas monitoring equipment and which type should be used in a given situation.

In Table 2, the operational advantages and disadvantages of several portable instruments, along with the gases analysed, are given. For more details, see Annex E.

The instruments required depend upon conditions at specific sites; it is therefore not appropriate to specify specific instruments.

Portable instruments which are to be used on gas-contaminated sites should be intrinsically safe; this is particularly so if the instrument is to be used within a confined space.

5.5.2 Installation of gas-monitoring standpipe

The borehole should reach 1 m into the natural ground or 6 m deep (whichever is the greater) or to a pre-determined depth as required by the specific site investigation. Where the investigation is related to a landfill, the borehole outside the landfill should reach at least 1 m beyond the maximum depth of the landfill. If this depth is not known, it should be established as part of the investigation.

A 50 mm diameter pre-slotted pipe should be installed to the base of the borehole. The pipe should, however, be un-slotted (plain) within 1 m of the ground surface. The pipe should comprise sections which can be fitted together with screw threads, as this avoids the need for organic compounds and solvents to seal the lengths together.

The annulus between the outer wall of the borehole and the slotted pipe should be filled in with pea-shingle or similar material.

The top of the hole (generally between 1 m and 0,2 m from the ground surface) should be sealed with an impermeable plug (bentonite grout/bentonite cement, etc.), while the upper 0,2 m from the ground surface should be sealed with cement which should support a cover. Where possible, pipes should terminate above ground level as this prevents flooding and makes them easier to identify. This may however not be possible on sites with public access. A lockable heavy-duty cover is worth considering to prevent vandalism and tampering.

A screw top should be fitted to the top of the standpipe to allow access into the pipe in order to take ground water level measurements. A stopcock should be fitted to the cap from which gas samples can be taken; the stopcock will allow the flow of gas from the standpipe to be opened or shut off as required.

Table 2 — Portable equipment to measure permanent gases

Instrument	Gases analysed	Advantages	Disadvantages
Infra-red spectro-photometer (IR)	Carbon dioxide, (methane) aliphatic hydrocarbons	— Specific gases can be analysed within pre-defined ranges — Wide detection range	— Reading may be affected by moisture
Sensor with catalytic oxidation	Flammable gases	— Sensitive	— Sensing element may deteriorate with age — Requires adequate oxygen — Not methane-specific — Not intrinsically safe — Sample is destroyed as part of measurement process
Thermal conductivity detector (TCD)	Carbon dioxide, flammable gases	— Wide detection range	— Not methane-specific — Errors can occur at low concentrations
Flame ionization detector	Flammable gases	— Very sensitive — Good for pinpointing emission sources	— Not methane-specific — Not always intrinsically safe — Requires oxygen — Possible errors if high levels of carbon dioxide — Sample is destroyed as part of measurement process
Indicator tubes	Most gases	— Very easy to use — Wide detection range — Used for large number of gases	— Limited precision and readability — high cross sensitivity
Electrochemical cells	Oxygen	— Simple to use	— Moisture can reduce sensitivity — Limited shelf-life
Paramagnetic cell	Oxygen		— Accuracy affected by changes in atmospheric pressure
Photoionization detector (PID)	Volatile aromatic and aliphatic hydrocarbons	— Detection of various ranges can be excluded — Different excitation energies possible	— Detection not specific — Detector signal depends on connection
Gas chromatograph (portable) (GC) equipped with appropriate detectors, e.g. flame-ionization detector (FID), photoionization detector (PID), heat conductivity detector (WLD)	Aromatic hydrocarbons, volatile halogenated hydrocarbons	— Elaborate	— Single component determination possible

5.6 Sampling plan

The sampling plan depends on the investigation objective and the local site.

To understand the conditions, the following parameters shall be considered:

- meteorological conditions;
- pressure differences;

- gas flow rates;
- gas concentrations.

Before measurements are taken, the following points shall be considered:

- understand exactly what is to be measured in order to ensure that the correct techniques and equipment are used;
- be aware of the limitations associated with the usage of the equipment chosen;
- whether the very act of setting up and making the measurement has affected the distribution equilibrium between the solid phase and the gas phase and hence affect the measurements taken;
- whether portable gas monitoring equipment is sufficient or whether off-site analysis is required.

It is good practice to decide upon a sampling protocol that can be used whenever monitoring is undertaken. This should help ensure that the techniques used by different operators are consistent, which should reduce uncertainties in data quality. A checklist should help to ensure that key activities are not overlooked. It is important that when planning a monitoring scheme the requirements of the investigation are kept in mind at all times.

The protocol given in Annex A is only an example; activities can be omitted or added depending upon the site and the requirements of the survey.

5.7 Sampling

5.7.1 Sampling for on-site measurements

Where portable instruments are used, they should be connected securely to the sample point and gas should be allowed to flow through the instrument until a steady reading is obtained. A reading should be taken of both peak and steady-state concentrations.

If more than one instrument is required to measure different gases, this procedure shall be followed for each of the gases. If the instruments are fitted with pumps, and provided that the sample is not destroyed in the measurement process, the instruments can be put in series, with the exhaust from one instrument going into the inlet of the next instrument. In this case it is advisable to fit non-return valves in the inlets to the instruments to prevent air being drawn back through them by the other instruments. Instruments shall be proved gas-tight. If an external pump is applied, then this shall be installed after the series of measuring instruments.

When taking a gas measurement from a standpipe, a length of sample line is usually required to connect the borehole to the gas analyser. This should be kept to a minimum, in general no more than 1 m. Care should be taken when selecting sample lines, as certain compounds can be adsorbed onto them. For example, poly(vinyl chloride) (PVC) absorbs water vapour and will release it in a dry air stream, while polyethylene is permeable to oxygen and carbon dioxide. Where possible, internally clean stainless steel should be used, or if flexibility is required, polypropylene is suitable for most gases. Silicone tubing, polyethylene and PVC should be avoided where possible.

A column of drying agent can need to be placed along the sampling line to prevent the moisture contained within the landfill gas from damaging the instrument. Consideration should be given to the type of drying agent used, as this can affect readings. Silica gel absorbs gases such as carbon dioxide, especially when wet. In most cases either calcium chloride or anhydrous calcium sulfate is recommended. For sensitive analysis, magnesium perchloride is probably the most suitable. Instrument manufacturers often supply proprietary hydrophobic filters, but care should be taken that only specified filters are used. Alternatively, a gas cooling device, e. g. with a Peltier element and fixed water separator, can be applied.

In some cases, during sampling, it can be beneficial to record the gas concentrations and flow rates observed in a borehole using a data-logging device. In most cases, the gas analyser will have a data logger connection. This can be used to log at pre-defined frequencies. When monitoring from a borehole, it is advisable to log every few seconds. This will show the steady-state concentrations, as well as the range of concentrations. In most cases the data logger can be downloaded onto a computer software package for further analysis and data storage.

5.7.2 Sampling for laboratory measurements

The laboratory chosen to carry out the analysis should be independent and competent in the work required, and preferably have an appropriate accreditation or notification.

Selection of suitable apparatus and sampling procedure shall be agreed between the analyst and the sampling staff.

A simple and widely used method of collecting gas samples is to use pressurised sampling cylinders, e. g. a Gresham tube. A hand pump is used to compress the sample into a small cylinder made from either aluminium alloy or preferably stainless steel. The cylinders can vary in capacity from 15 ml up to 110 ml.

Another method is the use of a gas sampling vessel, which can be sealed at both ends by taps or valves. The vessel is connected to the sample point with a vacuum pump or hand aspirator in-line to provide suction. Gas should be drawn through the vessel until at least three to five changes of the vessel volume have passed through.

Landfill gas samples can be taken using a variety of containers.

It is advisable to flush all containers with an inert gas, such as argon, before the sample is taken. It is important to be consistent in the methods of sampling, the apparatus used and the analytical and measurement techniques employed. It is important to consider the possibility of absorption onto the surfaces of containers and, where necessary, treated containers should be used.

5.8 Storage and transport of samples for laboratory analysis

Storage characteristics (time and conditions) of the vessels shall be determined using mixtures of gases of defined concentrations of the analytes. Once collected, samples should be analysed as soon as possible within the determined time period.

The collected samples shall be clearly labelled with the date, time, location and, where possible, the approximate concentration of at least one major component. If samples cannot be analysed immediately the sample should be kept under temperature conditions identical to those prevailing at the time of collection. An insulated box, fitted with a lid and a maximum/minimum thermometer, is recommended for this purpose.

5.9 Sampling report

The requirements outlined in ISO 10381-1 apply in general to the soil-gas sampling report.

For each sample taken, the following information should be given:

- location;
- description of soil profile;
- photos or video when appropriate;
- date and time of sampling/measurement(s);
- pressure differences;

- sampling depth;
- gas flow rates;
- gas concentrations;
- instrument(s) and technique(s) used;
- atmospheric pressure on the day of sampling and for the three preceding days;
- meteorological conditions at the time of sampling;
- ground conditions, e.g. wet, water logged, dry with vegetation showing no signs of stress, cracks in the soil;
- expected gas concentrations (as information for subsequent laboratory analysis) or results of direct measurements;
- for laboratory analysis, time and conditions of transport and storage shall be recorded.

5.10 Quality assurance (see also 6.10)

5.10.1 General

A series of quality control procedures should be integrated within the sampling plan. The minimum quality assurance procedures that should be performed are

- a) collection of quality control samples,
- b) use of standardized field sampling forms,
- c) documenting calibration and use of field instruments, and
- d) testing of sampling and storage equipment for gas-tightness.

Instruments used shall be checked regularly to avoid, e.g.

- exhaustion of steam-filters,
- decreasing electric power from batteries influencing readings,
- possible influences on readings caused by inclination of instruments while in use, and
- undesired turning of calibration screws.

Actions to be taken, when deviations from predetermined procedures are observed, should be considered prior to sampling.

5.10.2 Quality control samples

5.10.2.1 General

Quality control samples are taken to indicate the quality of the sampling programme, in addition to the samples taken from pre-determined sampling points. . They provide information which ideally discounts any errors due to possible sources of cross-contamination, inconsistencies in sampling and checks on the analytical techniques used. In accordance with the data quality objectives of the sampling and investigation

programme, consideration should be given to implementing each of the quality control procedures described below.

5.10.2.2 Blind replicate samples

These samples can be used to identify the variation in analyte concentration between samples collected from the same sampling point and/or the repeatability of the laboratory's analysis. For every 20 samples taken, one set of blind samples should be collected. The blind samples should be removed from the same sampling point in a single operation and divided into two vessels. These samples should be submitted to the laboratory as two individual samples and no indication given that they are duplicates.

5.10.2.3 Split samples

These samples provide a check on the analytical proficiency of the laboratories. For every 20 samples, one set of split samples should be taken. The samples should be removed from the same sampling point in a single operation and divided into two vessels. One sample from each set should be submitted to a different laboratory for analysis. The same analytes should be determined by both laboratories, using identical analytical techniques.

5.10.2.4 Trip blanks

These blanks are used to detect cross-contamination of samples during transport. A container or sorbent cartridge or other collection medium, identical to the ones being used for the samples, is sealed as for a real sample, placed with the samples and transported back to the laboratory. The blank is then analysed along with the collected samples.

5.10.2.5 Other quality control samples

Other quality control samples to be considered include field blanks, field spikes, rinsate blanks and resubmission of a previously analysed sample to the same laboratory or to a different laboratory.

5.10.3 Evaluation of quality control sample results

The analytical results and quality control data should be evaluated following recognized procedures to allow the interpretation of accuracy, precision and representativeness of the data. Typical variations which can be expected from acceptable quality control samples are shown in Table 3.

Table 3 — Acceptance criteria for quality control samples

Quality control samples	Minimum number of samples	Typical RPD for quality control samples ^{a, b}
Blind replicate sample	One per 20 samples collected	30 % to 50 % of mean concentrate of analyte
Split sample	One per 20 samples collected	30 % to 50 % of mean concentrate of analyte determined by both laboratories ^c
Trip blank	One per box used to contain samples	The significance of the trip blank results should be evaluated in terms of those obtained for the actual field samples

^a The relative percent difference (RPD) is [(Result 1 minus Result 2) divided by the mean result] times 100.

^b The significance of the RPDs of the results should be evaluated on the basis of sampling technique, sample variability and absolute concentration relative to criteria and laboratory performance.

^c This variation can be expected to be higher for organic analytes than for inorganic and low concentration analytes.

5.10.4 Chain of custody

This process details the links in the transfer of samples between the time of collection and their arrival at the laboratory. Several transfers can take place in this process, but details of each transfer should be recorded on a chain of custody form. The minimum information that shall be included on the form is the following:

- a) name of the person transferring the samples;
- b) name of the person receiving the samples;
- c) time and date that samples are taken;
- d) time and date that samples are received by the laboratory;
- e) name and contact details of the client;
- f) analytes to be determined;
- g) the set of samples to be composed for the analysis, if the use of composite samples is required. This set
 - 1) should contain no more than four samples, and
 - 2) the analysis should be carried out in the laboratory;
- h) other specific instructions in the handling of the samples during analysis, e.g. special safety precautions;
- i) samples that are expected to contain high levels of the analyte in question or other substances that can interfere with the analysis.

5.10.5 Calibration of instruments

All equipment should be calibrated and demonstrated to meet the calibration specifications prior to use. There are a number of reasons for this.

- a) gas analysers have a tendency to drift over time;
- b) some instrument types have a limited life and can fail suddenly without warning;
- c) landfill gas contains trace gases that can “poison” the monitoring equipment.

The instrument should be calibrated using a span gas with appropriate concentration and a zero gas such as nitrogen. The frequency of calibration will depend on how regularly the instrument is used. Advice on how to carry out calibration should be provided with the instrument.

Regular servicing should be carried out according to the manufacturer's recommendations.

Regardless of whether the calibration is performed off-site or on-site, all calibration data should be recorded and made available on request. Off-site calibrations are not suitable for instruments recording changes during transportation.

5.11 Interferences

Soil-gas examinations can be influenced by various interferences, which can lead to errors or misinterpretations at large sample volume and with cohesive soils (see 6.11).

6 Volatile organic compounds (VOCs)

6.1 Objectives

The main objectives of soil-gas analyses for VOCs are as follows:

- qualitative determination of substances present in the unsaturated soil layer (inventory);
- determination of the magnitude of local concentrations and concentration differences;
- determination of the place(s) of input and location of the contamination centres (= areas of highest concentration measured);
- determination of the horizontal and vertical distribution;
- mapping of groundwater contaminations (pollutant plumes);
- observation of spatial distribution over the time;
- measurements to confirm the effectiveness of remediation measures.

Usually the distribution of VOCs in soil gas does not reflect the distribution in the soil (see 6.2.1).

6.2 Basic principles

6.2.1 Physical and chemical principles

Depending on the pressure and temperature conditions, VOCs enter into soil pore space as either a liquid or a gaseous phase. They are present in soil as gaseous and liquid phases, dissolved in soil water, adsorbed on solid (organic and inorganic) soil particles or enclosed in capillary cavities.

Dynamic distribution equilibria are established according to the prevailing conditions and bound forms of the pollutants. Owing to the diversity of the possible substance distributions and the time-dependent effects on the equilibrium, each determination of the contaminant concentration can provide only a description of the 'as is' status. Every interference with the soil affects the distribution equilibrium in a different way, which is difficult to assess.

A saturation equilibrium between the liquid and gaseous phases is established within a small area independent of the amount of substance present. A soil-gas saturation concentration of a VOC develops in the immediate surroundings of the polluted area, irrespective of whether it is a very small drop of the substance or a large deposit. The concentrations measured in the soil gas shall not be used as an index of the actual amount of substance present in the soil. VOCs disperse in soil gas convectionally (i.e. in the direction of the pressure gradient) and diffusively (i.e. in the direction of the concentration gradient). VOCs in the soil can be transported as flowing non-aqueous-phase liquids (NAPLs), or together with another flowing liquid phase (e.g. groundwater, or dissolved in mineral oil), from which they can be transferred back into the soil gas.

6.2.2 Ambient conditions

See 5.2.2 and 5.11.

Percolating water from precipitation or rising ground water can leach the pore space and thus remove some of the VOCs.

Decreasing soil temperature affects the volatility of organic compounds and lowers both the vapour pressure of the contaminants and diffusion rates, resulting in lower observed concentrations. Soil frost strongly limits the mobility of gases and vapours; it hinders the emission into the atmosphere, and can lead to accumulation in the coldest areas.

If the soil gas is being trapped directly, it is recommended that soil-gas samples be taken only if the temperature of the ambient air is above that of the soil. Sampling of soil gas at temperatures which are significantly lower than that of soil, can lead to condensation in cold spots in the sampling system. Subsequently, the vapour pressure and the concentration of VOC in the sample will be lowered. However if the soil gas is being collected on an adsorbent, this is generally less of a problem although low soil temperatures will depress the rate of collection. Because of these considerations, the conditions at the time of sampling should be documented in detail in the report, irrespective of the method of sampling.

6.3 General considerations for sampling

6.3.1 General

The strategies and procedures for soil-gas sampling for VOCs should be formulated taking into account the specific conditions on the site and the objectives of the site investigation.

There is no single strategy which can be generally applied. Therefore some typical examples are given, and reference should also be made to Annex C.

6.3.2 Initial explorations (field screening)

The aim of an initial exploration is to determine the substances present. At this stage data on concentrations are generally not known, so there is the risk that the measuring range selected may be not appropriate. Detection limits can be too high, raising the possibility of breakthrough, in the case of adsorbents. In the case of contaminants present at low levels, the extraction of large volumes (e.g. 20 l) allows gathering of information on a more comprehensive range of substances.

When effectively used, screening methods can provide a comprehensive overview of substances present and thus result in considerable cost saving as regards the subsequent detailed investigation.

The use of a photoionization detector (PID) without prior use of a gas chromatograph, as well as direct-reading detector tubes, serve for labour protection, workplace monitoring and for rough mapping of pollutant plumes (e.g. for determining effective starting points for establishing groundwater or soil-gas measuring sites). They do not supply exact concentrations of single VOCs.

6.3.3 Known contamination centre

If the contamination centre is known, the extent and limits of the contamination can be investigated using point screening. The screening is carried out on selected profiles both horizontally and vertically according to the nature of the site and the objectives. Starting with large distances between sampling locations and increasing sample density stepwise, if appropriate, will provide a cost-effective strategy. Sampling the contamination centre itself is of limited use for the results of soil-gas analyses, for VOCs can not be used for evaluation and risk assessment (see 6.2.1).

6.3.4 Unknown contamination centre

If the contamination centre is not known or when there is doubt about the presence of contamination, screening is carried out using a coarse regular grid pattern. All available information from historic enquiries or previous investigations should be taken into account. The use of integrated sampling techniques, large soil-gas volumes and concentration methods, as appropriate, to reduce the risk of not finding contamination which is present. Once a contamination centre has been found, follow the strategy of 6.3.3.

6.3.5 Determination of concentration maxima and places of input

The determination of contamination maxima and places of input should in general follow the strategy described for unknown contamination centres in 6.3.4. The difference is that the contamination as such is already known, so the sampling is confined to predefined areas.

6.3.6 Determination of horizontal and vertical distribution of VOCs

This is essentially the same process given in 6.3.2. Screening or collection of soil samples for headspace examination over horizontal and vertical profiles is carried out to establish the distribution of the contaminant in the soil gas.

Point sampling (small sampling volumes taken from defined sampling depths) along profiles can also be used to determine spatial distribution. Care shall be taken that ambient conditions (see 6.2.2) and consistent sampling techniques and instruments are achieved to enable comparable measurements.

On-site analysis can help to minimize interferences and can allow an effective intensification of the sampling point screen.

6.3.7 Observation of spatial distribution of VOCs in the course of time

Repeated sampling under almost the same conditions is possible only for homogeneous and well defined ground conditions. Therefore, it is useful to establish permanent soil-gas monitoring devices for permanent continuous observations.

The disadvantage of such monitoring devices is that they present a limited picture of the situation as regards soil gas on the site. In particular, the geometry and surface of the monitoring device, as well as the potential for gas exchange, are significantly different from the corresponding properties of soil pores.

Particular attention shall be paid to the ambient conditions (variations in temperature, humidity, air pressure, soil saturation, etc.).

6.4 Sampling requirements

6.4.1 Working ranges of measurement methods

The aim of spatially localizing or delineating soil contamination provides for a relative quantitative comparability of the concentration values determined. The soil-gas concentration values range according to the corresponding substance and method-specific detection limits (several micrograms per cubic metre, $\mu\text{g}/\text{m}^3$) up to the corresponding saturation concentrations (in some cases, up to $> 100\,000\,000\ \mu\text{g}/\text{m}^3$, i.e. $> 100\ \text{g}/\text{m}^3$). In order to cover the whole concentration range, sampling techniques and measuring methods adapted to the relevant problem shall be used.

NOTE When using adsorption methods, the capacity of adsorbents can readily be exceeded. The volumes at which breakthrough occurs differ from chemical to chemical. In the case of high concentrations in soil gas, direct measurement methods are more practical as they provide reliable concentration values without needing additional handling and measurements.

6.4.2 Sampling options

Figure 1 shows typical techniques used for sampling VOCs in soil gas (in accordance with VDI 3865-2^[14]).

The following techniques are illustrated.

- a) Sampling technique 1 [see Figure 1 a)]: soil-gas sampling using a probe with small opening at its lower end.

A limited volume of soil gas (e.g. 1 l) is pumped from a more or less point source.

- b) Sampling technique 2 [see Figure 1 b)]: two-stage soil-gas sampling using a probe with small opening at its lower end.

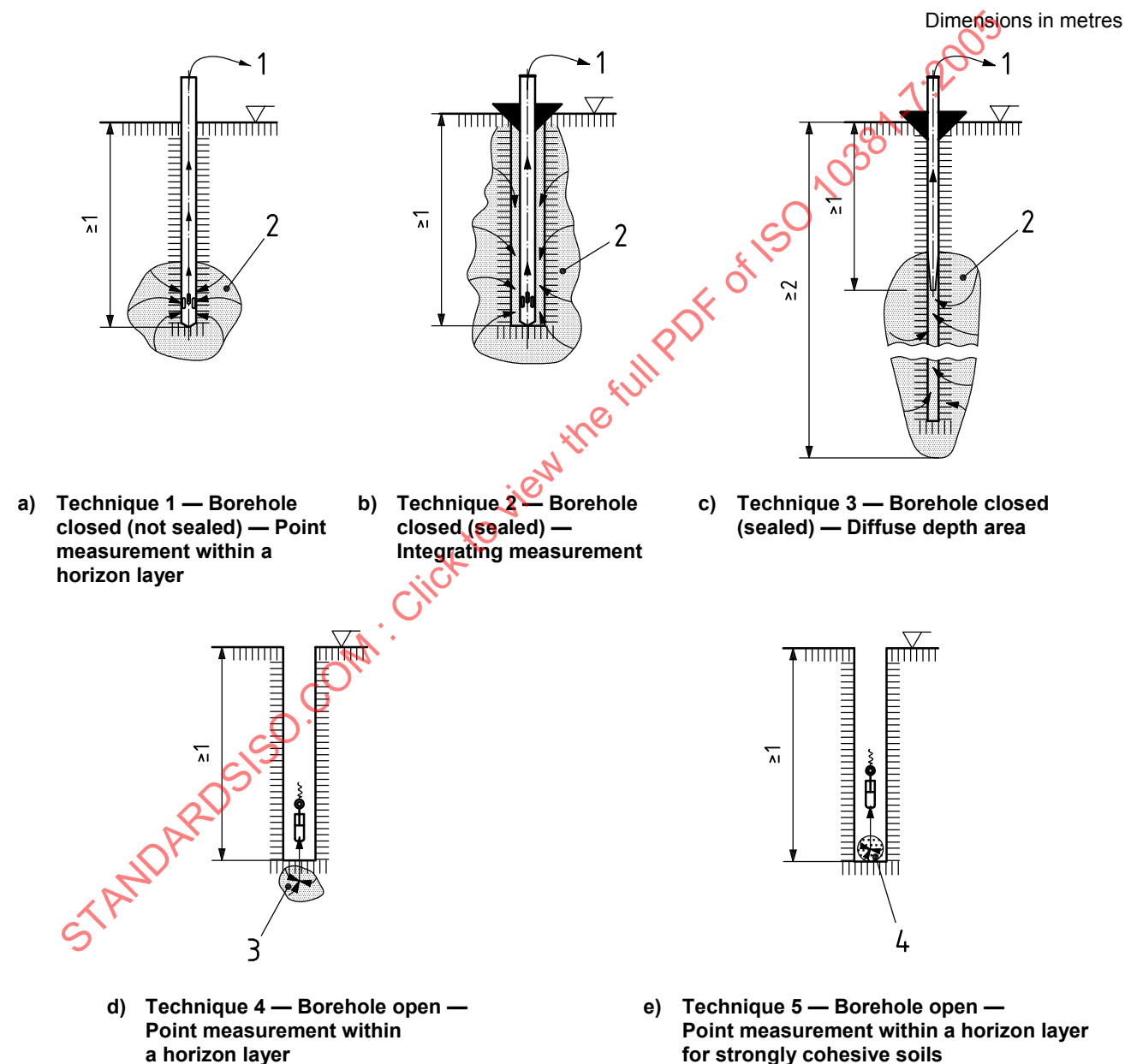
A limited volume of soil gas (e.g. 1 l) is pumped. The more the diameter of the open borehole exceeds the diameter of the probe, the more the soil-gas sample differs from a point source sample, and becomes an integrated sample.

- c) Sampling technique 3 [see Figure 1 c)]: two-stage soil-gas sampling using a probe that completely fills the borehole but does not reach its lower end.

A greater volume of soil gas (e.g. 20 l) is pumped through the opening at the lower end of the probe. The soil-gas sample becomes an integrated sample from an undefined soil volume of diffuse depth.

- d) Sampling techniques 4 and 5 [see Figures 1 d) and e)]: two-stage soil-gas sampling techniques using a syringe to take a small volume of soil gas from the subsoil of the end of the borehole or of gas from within the borehole.

Both techniques enable point measurements.



Key

- 1 extraction
- 2 sampled volume, e.g. 1 l pore air
- 3 sampled volume, e.g. 0,01 l pore air
- 4 sampled volume, e.g. 0,01 l borehole air

Figure 1 — Typical sampling techniques

6.4.3 Location of sampling

Depth of soil-gas sampling depends on the information required and on site conditions such as structure of the ground, location of the groundwater table, migratory pathways, etc. These conditions shall be established prior to planning of soil-gas sampling. The bores performed during two-stage sampling can serve this purpose. Whether soil profiles of all of the soil-gas sampling bores shall be recorded in detail depends on the heterogeneity of the ground and the objectives of the investigation.

Owing to increasingly uncontrolled influence of ambient air at shallow depth the minimum sampling depth should not be less than about 1 m below ground surface. If there is good reason for sampling from closer to the surface (e.g. below sealed sites), this should be indicated in the sampling report.

Maximum sampling depth is limited by the accessibility, by the availability of techniques to secure gas-tightness of the probe system and limit dead volumes, and by the level of the water saturated soil zone. Regarding the capillary rise of groundwater, it is recommended that the sampling point be at least 1 m from the water table.

If concentration gradients have to be investigated (e.g. when delineating hot spots), point sampling is necessary. In this case techniques have to be used that allow collection of soil-gas samples from small sampling volumes (i.e. from holes less than 10 cm in diameter).

6.4.4 Sampling volumes and sampling rates

The extraction of soil gas during sampling reduces pressure in the pore space and hence influences phase and solution equilibria. To limit these influences, especially when taking point samples, volumes of < 1 l and flow rates up to a maximum 2 l/min are recommended. For soils exhibiting a low gas permeability and a small gas pore volume, even lower flow rates should be used.

When a qualitative determination of the substances present is required (e.g. during preliminary investigations), a large soil-gas volume (e.g. 20 l for XAD-resin as adsorbent) should be extracted. This will provide a more volume-related diffuse sampling area. The incorporation of atmospheric air shall be avoided. Appropriate controls (e.g. by simultaneous, continuous observation of typical soil gases such as CO₂ and methane and oxygen) are essential. It shall be kept in mind that point-related information (e.g. to determine concentration distribution) will not result from using this technique.

In the case of point soil-gas sampling, a small volume is taken within the horizon layer (about 10 ml) for a possibly non-influenced detection of the pore volume filled with gas. When sampling larger volumes (up to several litres), the sampling area is diffuse and its location cannot be determined. Soil-gas sampling from a borehole with diameter greater than that of the probe is called "integrating", as the gas might be delivered over its entire length.

Instruments for the measurement of gas flow rate are listed in Annex D.

6.5 Technical equipment

The standard equipment for soil-gas sampling for VOC generally includes the instruments given in Table 4.

Each part of the sampling system which is in contact with the soil-gas sample should be properly cleaned between each sampling.

Table 4 — Standard equipment used for soil-gas sampling for VOC

Equipment and purpose	Requirements, limits of application
Electric hammers for driving down soil-gas probes and installing boreholes using window sampler	Possible contamination due to exhaust air, petrol and oil from petrol-fuelled engines shall be avoided
Generator for operating electrically driven equipment	Installation of generator in sufficient distance and suitable wind direction
Jack (hydraulic or mechanical)	
Electrically driven hammer or core drilling instrument for boring through sealed or solidified surfaces	
Soil-gas probes	Stable, temperature resistant, inert (stainless steel), easy to operate, gas-tight extendible, easy to clean, known dead volume, defined close opening area Sampling head with septum, if necessary Connection of gas dosage valve to probe head to make direct measurements using a gas chromatograph, if necessary
Pump for extraction of soil gas	Controllable according to volume flow, possibly with continuously adjustable desired soil volume (alternatively, gas meter) When using direct-reading detector tubes – the appropriate bellows vacuum tube for the detector tube type
Manometer (if not integrated in pump)	For measuring the negative pressure developing during soil-gas sampling
Volume flow meter	e.g. gas meter to measure sampling volume variable area flow meter to control volume flow
Water separator, if necessary	e.g. Wulff flask
Sampling devices	e.g. Pasteur pipettes, gas mice, rimmed vials with PTFE-lined septa (head-space glasses, volume 10 ml or 20 ml, these septa shall self-seal after insertion and withdrawal of syringe) Sampling devices and, if required, closures shall be heated prior to use to avoid the carry over of contamination So-called air-bags are not suitable for analysing VOCs
Cartridge (butane) with burner	To melt end of sample-filled Pasteur pipettes
Closure indicator for rimmed vials	For gas-tight fastening of septa on ampoules using aluminium seal rings
Adsorption tube with protective tube	Filled with activated charcoal or XAD-4 Gas-tight sealable protective tube for transport and storage
Decontaminants or devices	
Glass syringe	Gas-tight
Evacuation equipment (negative pressure pump or large volume syringe)	
Connection hoses	Possibly from inert material (stainless steel)
Timing device to determine starting time of sampling or to measure duration of sampling	
Barometer to observe (several measuring) air pressure at sampling location	
Thermometer to measure ambient and soil-gas temperature	
Psychrometer to measure relative atmospheric humidity	

6.6 Sampling plan

To ensure that soil-gas analyses provide an optimum result, soil-gas sampling should be planned by taking into account the objectives and the cost-effect relationship. The choice of the appropriate procedures is determined by the type of the suspected soil contamination, available information, soil structure and the location of the groundwater table. In particular, underground lines and underground parts of a building shall be considered in the sampling plan.

If samples cannot be taken according to the plan (e.g. because of obstacles on the site or in the ground, higher groundwater level than expected) this shall be recorded in the sampling report. It is wise to define certain procedures for such contingencies (e.g. relocate the sample, leave it out, etc.). In the absence of properly document procedures, sampling personnel can make wrong decisions, rendering the investigation useless.

6.7 Sampling

6.7.1 General

Depending on the objectives and the local conditions, different sampling procedures are used. Therefore, a universally applicable description cannot be presented in this part of ISO 10381. After giving a short overview about possible variants, the general requirements are defined and the procedures used for some variants are shown.

For soil-gas sampling, either the one-stage or the two-stage procedure is used (from open or sealed borehole or from a soil-gas measuring location). The soil layer to be sampled is either explored directly or within several steps by separate sampling (soil-gas profile).

Soil-gas sampling is carried out with the aid of a syringe or soil-gas sampling probe. The samples are transferred into collecting containers (septum vial, hermetically-sealed glass ampoule), concentrated on adsorption materials (activated charcoal or adsorbent resin), or delivered in a closed measuring system for direct measuring in a gas chromatograph or direct-reading detector tube. Using evacuated sampling vessels always leads to diluted concentrations, because a complete vacuum is not reached during evacuation.

For two-stage sampling, the probe diameter is equal to or smaller than the diameter of the borehole or sample measuring location. The borehole shall be stable up to the completion of soil-gas sampling. The installation of the soil-gas probe may not lead to ingress of unstable soil material into the borehole, which contribute to a relocation of soil gas and might contain volatile substances.

The start of the individual sampling procedure during the pumping period should preferably be controlled by the development of the concentration of characteristic soil gases in the gas flow or, alternatively, controlled by time or volume rather than uncontrolled.

During soil-gas sampling, the following principles shall generally be considered independently of the sampling variant chosen.

- Prior to sampling the system shall be checked for gas-tightness (see 6.10).
- Meteorological boundary conditions shall be measured before, during and after sampling.
- The sample shall be taken in front of the pump or the adsorbent used before pump.
- It shall be ensured that the soil-gas sample is not influenced by atmospheric air or soil gas from soil horizons, which are not to be sampled.
- Prior to sampling or inserting the adsorbent, at least five times the dead volume shall be extracted.
- The time between establishing the borehole or installing the probe and the sampling shall be kept constant for each investigation point.

- The flow rate shall be adapted to the underground properties and be kept as low as possible (0,2 l/min to 1,0 l/min) and constant at low negative pressure; when using adsorbents the manufacturer's instructions should be followed and recorded.
- All parts of the sampling system which came in contact with soil gas shall be used only once or be decontaminated prior to re-use (from measuring point to measuring point). Suitable decontamination methods are, e.g. torching probes with a hot-air blower, boiling off syringes with water, torching small material in an oven. If the decontamination methods are not carried out on-site, it shall be ensured that sufficient probes, etc. are available.

6.7.2 Direct measuring method

Different methods can be used for delivering soil gas in the sampling equipment. In general, it shall be ensured that all samples are taken under the same boundary conditions. Each intermediate step represents a potential source of errors.

Two different types of container are widely used for direct sampling of soil gas, i.e. Pasteur pipettes and rimmed vials (headspace glasses). They are considered below in more detail. In comparison, other sample containers have many disadvantages as regards handling or storing samples to be analysed for VOC, and thus are not further considered here.

Direct sampling using rimmed vials with PTFE-lined septa should involve the following steps:

- insertion of the gas probe;
- turning on pump, pumping and extracting the dead volume of the probe twice;
- closing the transfer from the pump to the gas-tight probe;
- turning off the pump;
- allowing the negative pressure in the probe to decrease (control with the aid of a glass syringe);
- taking the soil-gas sample from the sampling head (septum) using a gas-tight glass syringe with a millilitre-reading that has been decontaminated previously by heating. Before separating the syringe from the sampling head, compensation with ambient pressure should have occurred.

The soil-gas sample is then transferred into the rimmed vial that is sealed by a septum. This procedure is sensitive to leaks and shall be performed very carefully.

This sampling can be done in different ways, but one of the simplest and most secure is as follows:

- sealing the rimmed vial (which now is full of ambient air);
- “evacuating” the vial using a vacuum pump to about 50 hPa (Although this is not a complete vacuum, it is sufficient for the purpose of this work), or alternatively lowering the pressure inside the vial by extraction of a defined volume using a large glass syringe (The extracted volume shall be known and recorded in order that a known volume of soil-gas sample is collected. The greater the volume extracted, the lower the detection limit that can be reached with the analysis.);
- inserting the syringe containing the sample into the “evacuated” vial. (The negative pressure in the vial is compensated by transfer of a certain volume of the soil-gas sample. As this portion will be analysed, the volume shall be accurately recorded together with the total volume of the vial to allow calculation of the concentrations.)

The sample transfer by pressure compensation provides good proof of the gas-tightness of the vial. It is not gas-tight if almost the total volume of the vial has not been evacuated by vacuum pump. It is not gas-tight if almost the calculated volume has not been extracted using the large volume syringe. In these cases another sample container can be used immediately.

Before proceeding with the sampling operation, confirmation shall be made that none of the substances of interest is present in the ambient air where the vial is sealed. The use of blank samples is recommended (see 5.10). Sealing of the vial and transfer of the soil-gas sample and laboratory analysis should take place under similar atmospheric pressures.

6.7.3 Sampling using adsorbents

If low concentrations of VOCs are expected and no point sampling is required, concentration methods using adsorbents may be appropriate in order to reach lower detection limits.

During the sampling operation, information given by the manufacturers of the adsorbents [see quality control (5.10)] should be taken into account.

6.8 Storage and transport of samples for laboratory analysis

Samples containing VOCs shall be transported under the exclusion of any light.

Samples should arrive at the laboratory no more than 24 h after sampling and analysis should commence within the following 24 h.

If it is not possible to fulfil the above requirements, the laboratory should provide evidence that reliable results can nevertheless be achieved. It shall be remembered that observing the above holding times does not guarantee correct results and use shall be made of quality assurance procedures (see 5.10) to demonstrate effectiveness of the procedures employed.

6.9 Sampling report

The sampling report that should be collected for each sample is given in 5.9.

6.10 Quality assurance

(See also 5.10.)

Following are listed some essential elements of quality assurance for the sampling of soil gas.

- While checking for leaks, the suction opening of the sample probe shall be sealed gas-tight. The entire extraction system is evacuated with the pump which will be used for sample extraction. Then, the system is shut off in front of the suction opening of the pump and the negative pressure is measured. The negative pressure shall be constant for the same amount of time of the sampling process. Then the apparatus is gas-gas tight according to this standard.
- During sampling, transport and storage of the samples it is important to ensure that they do not become contaminated, e.g. through exhausts, cigarette smoke, solvents or materials that contain volatile organic compounds (e.g. polystyrene products, pesticides, sunscreen products, skin care products).
- Blank sampling of ambient air at the borehole shall be performed regularly, e.g. before and after each examination phase or at the beginning of each sampling day.
- The dead volume in the sampling probe shall always be exchanged before sampling.
- The carry-over of contamination shall be avoided. Parts made of rubber or plastic (e.g. packer, PTFE insets, sealant rings) can carry over substances into other boreholes/samples.
- The drilling instruments shall be cleaned thoroughly with clean water and then dried in order to avoid carry-over of contaminated soil.
- The sampling apparatus shall be cleaned regularly.

- The imperviousness of the sampling apparatus shall be checked regularly (see 5.4).
- An instrument notebook shall be kept for the sampling apparatus. In this notebook, the projects, the time periods of use, as well as the substances examined shall to be recorded. Additionally, maintenance work, leak checks, date and type of cleaning performed on the parts of the apparatus shall be recorded.
- The adsorption tubes to be used in sampling shall be checked regularly according to their type with respect to their rate of leakage, e.g. per batch.
- The samples shall be exactly and permanently labelled directly after sampling to avoid later errors.
- The shelf-life of loaded adsorption tubes or filled sampling containers under typical storage conditions (temperature, light conditions, location, container, duration) shall be checked for each substance.
- If septum-sealed vials are used as gas sampling containers, it may be necessary to use external standards to correct the analysis results (adsorption losses ^[3]).
- The influence of typical transport conditions (temperature, light conditions, location, container, duration) for loaded adsorption tubes or filled sampling containers on sampling results shall be examined for each substance. The use of trip blanks is recommended (see 5.10.2.4).
- If general conditions change considerably during the sampling series (e.g. strong rain storms), an additional sample shall be taken within the suspected area of contamination. This shall be recorded in the sampling report.
- A reference sample should be taken outside of the suspected area of contamination.
- A complete sampling record shall be kept for each sample.

Listed in Annex A are several sampling parameters and other general conditions which can have an effect on the sampling result. The user can compile a report for the specific measurement task with this list. The completeness of the sampling report depends on the type of sampling and can be different from case to case.

The quality of the adsorption materials shall be tested at least once per working/analysing day by taking a blank sample.

Actions to be taken, when deviations from predetermined procedures are observed, should be considered prior to sampling.

6.11 Interferences

6.11.1 General

Soil-gas measurement can be influenced by various interferences, which can lead to errors or misinterpretations.

6.11.2 Large sample volume

The larger the sample volume, the more likely is the ingress of ambient air, especially when sampling close to the surface. In such instances, there is insufficient protection against the influence of ambient air especially in highly permeable ground. As the sample volume can have a significant influence on the measurement results, techniques that employ small sample volumes are generally recommended.

6.11.3 Cohesive soils

With strongly cohesive soils it may not be possible to achieve the required sample volume. In such conditions soil-gas measurements are not predicative.

6.11.4 Soil moisture

Soil moisture can interfere with the adsorption of the organic substances onto the collection matrix. If the pore spaces at the bottom of the borehole are saturated with soil moisture (e.g. due to an impermeable layer), sampling from the subsoil of the borehole is not possible. The pump is turned off for some sampling systems when the moisture content is too high.

6.11.5 Low ambient temperatures

The sampling of soil gas at ambient temperatures, which are clearly lower than that of the soil gas (e.g. frost), can lead to spuriously low estimates of volatile organic substances due to condensation in cold spots in the sampling system.

6.11.6 Heterogeneous stratigraphy

The adherence to a constant sampling depth without taking into account the orientation of strata yields different results, irrespective of the effective pollutant contents in the soil. Therefore, only measuring results from the same soil layers should be related to each other if heterogeneous stratigraphy is discovered.

6.11.7 Seepage front

After strong rains or floods, soil layers may have been washed through, leading to lowered recoveries in soil-gas samples taken above the seepage front.

6.11.8 Perched water table horizon

The perched water table horizon above cohesive soil layers can make the pollutants contained in the deeper soil layers unreachable for soil-gas examinations. A strongly contaminated perched water table can feign contamination of the soil through lateral transport of soluble substances, thereby leading to misinterpretations of the contamination of the non-saturated soil zone.

6.12 Interpretation of soil-gas analyses for VOCs

The results obtained from different sampling methods are not directly comparable due to variations in sampling conditions.

Owing to the physical and technical boundary conditions presented in 6.2 the results of soil-gas investigations for VOCs are suitable only for exploratory works and not for the assessment of dangers and risks from soil contamination.

Soil-gas analyses for VOCs always produce relative results, and concentrations in soil or groundwater cannot be determined quantitatively on the basis of the soil-gas analyses. Immediate conclusions cannot be drawn about the actual contamination in soil or groundwater in terms of the compliance with a regulatory value. High concentrations of VOCs do not categorically mean high concentrations in the surrounding mineral ground. Therefore, soil-gas analyses for VOCs are suitable only for establishing roughly the spatial distribution of substances, but not for assessing dangers or evaluating risks.

Soil-gas investigations performed only once (and assuming that all necessary aspects have been taken into account) represent the conditions available at the time of sampling. A change in the concentration and phase equilibria in soil over time will result in changes in the distribution and intensity of substance concentrations in soil gas. In the case of soil-gas components that are not bound to liquid medium in soil (in particular groundwater), their relative distributions over time in response to variables (such as direction, range, intensity, composition) shall also be considered.

Annex A (informative)

Sampling protocol

Table A.1 — Pre-site checks

Action	Complete	Comments/faults found
Equipment check – power supply, pumps, displays		
Servicing up to date		
Calibration check (note any drift) (see below)		

Table A.2 — Calibration chart

	Concentration (volume fraction)	
	Zero	Span
Methane		
Carbon dioxide		
Oxygen		

Calibration should be carried out at the beginning and end of every day of monitoring wherever possible. Any calibration drift should be noted and then corrected for.

Table A.3 — Meteorological conditions

	Entering site	Leaving site
Atmospheric pressure (pascals) (falling or rising)		
Weather conditions (dry, rain)		
Air temperature (° C)		
Wind speed and direction		

Table A.4 — Actions at each sampling point

Date	
Time	
Sample point number	
Location	
Monitoring equipment	
Exchange of dead volume, times	
Suction to steady state condition	

Measurements from boreholes should be carried out in the following order: pressure difference, flow rate, gas concentration and water table depth.

Table A.5 — Monitoring results during sampling

	Borehole concentration (volume fraction)		Technique used
	Peak	Steady	
Methane			
Carbon dioxide			
Oxygen			

Flow rate (ml/min)		Technique used	
Water table depth (m)			
Soil-gas temperature (° C)			
Pressure difference			

Annex B (informative)

Anaerobic degradation and the formation of methane and carbon dioxide

B.1 General

The formation of methane gas from organic matter is not a new process; it has been acknowledged since the discovery of methane in marsh gas in the eighteenth century. The process, known as anaerobic degradation, is the decomposition of organic matter by bacteria in the absence of air, with the resultant formation of, principally, methane and carbon dioxide.

Natural sources of methane include the following:

- natural gas – formed by anaerobic decomposition of prehistoric plant material, now trapped beneath the earth's crust. This gas is usually associated with coal measures and oil deposits.
- marsh gas – formed by plant material decomposition in the absence of air. The anaerobic process can occur where there are significant concentrations of organic material, for example, in alluvial deposits.

A major source of methane and carbon dioxide due to anaerobic decomposition is landfilled wastes. In landfills there are a number of factors which influence gas formation. These include depth and age of the waste, types of waste contained within a site, moisture content, temperature, landfill pH as well as waste management practices.

B.2 Anaerobic degradation process

The anaerobic degradation process is briefly described below.

- a) Biodegradable organic matter consists of an assortment of molecules whose chief constituent is carbon, associated with other elements such as hydrogen, nitrogen, sulfur and oxygen. Within landfilled wastes particularly, there are three important molecular types:

- 1) proteins (which contain nitrogen and sulfur);
- 2) fats; and
- 3) carbohydrates.

Initial biodegradation will occur by aerobic bacterial activity until there is insufficient oxygen in the waste to sustain this process further. There are then several stages of anaerobic decomposition before landfill gas, with major methane and carbon dioxide components, is produced.

- b) The initial anaerobic phase consists of three stages.

- 1) The first stage involves the hydrolysis and fermentation of fats, proteins and carbohydrates by both facultative aerobic and anaerobic bacteria to produce simple organic acids, such as volatile fatty acids, as well as carbon dioxide, hydrogen and ammonia.
- 2) In the second stage, the volatile fatty acids are further degraded to acetic acid, carbon dioxide and hydrogen. This stage also enables the establishment of methanogenic bacterial populations.
- 3) The establishment of methanogenic bacterial populations results in a third stage of steady production, with the generation of a gas mixture, containing methane and carbon dioxide as its main components. The state of steady gas evolution can last for a considerable time before gas production starts to fall.

B.3 Changes in gas composition

The composition of gas associated with a landfill will vary with both the time and the stage of the degradation process.

Initially when waste is deposited, the gas composition will be similar that of ambient air (80 % nitrogen, 20 % oxygen and traces of carbon dioxide). The aerobic decomposition phase will result in the formation of carbon dioxide, with a reduction in the oxygen content and a lowering of the proportion of nitrogen.

The onset of anaerobic conditions, with the formation of volatile fatty acids, will result in further production of carbon dioxide (up to 70 %) and also the formation of hydrogen which can be as high as 10 %. These gases contribute to the replacement of nitrogen.

The onset of the methanogenesis leads to the formation of methane and a reduction in the hydrogen and carbon dioxide concentrations. As the methanogenic stage stabilises, the presence of hydrogen is eliminated by reaction with carbon dioxide to form more methane, and the production of methane and carbon dioxide stabilises to give a "steady" gas composition (typically of the order of 60 % methane and 40 % carbon dioxide).

Once methane generation diminishes, atmospheric air affects the gas composition and other effects (such as solution in water and reaction with surrounding material) can result in very varied gas compositions.

Decomposition within a landfill can commence very quickly, dependant on how conducive the conditions are to the process, and steady state gas production can be maintained for 20 years to 30 years. Even when gas formation has started diminishing, alteration of the conditions of the landfill, e.g. compression, can cause an increase in activity.

In alluvial materials, the content of methane can be quite significant, but the rate of gas production is likely to be low compared with an actively gassing landfill.

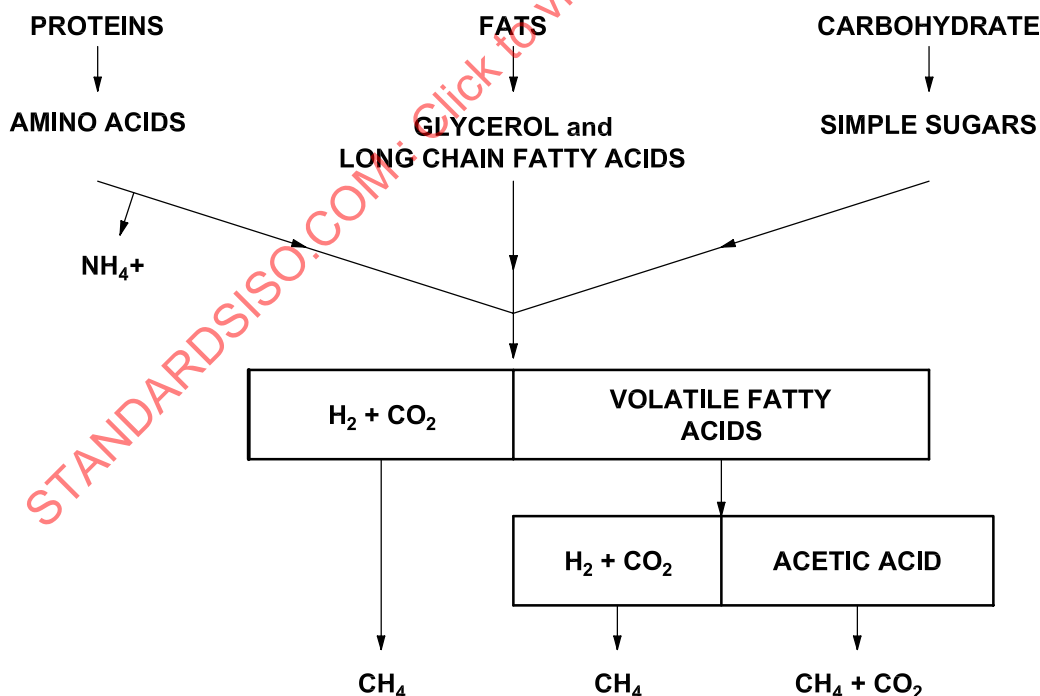


Figure B.1 — Decomposition of domestic waste