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Tritium and carbon-14 activity in gaseous effluents and gas discharges of nuclear installations —

Part 1:
Sampling of tritium and carbon-14

Activité du tritium et du carbone 14 dans les effluents gazeux et les rejets gazeux des installations nucléaires —

Partie 1: Prélèvement du tritium et du carbone 14

Reference number
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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 85, *Nuclear energy, nuclear technologies, and radiological protection*, Subcommittee SC 2, *Radiological protection.*

A list of all parts in the ISO 20041 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Discharges from nuclear installations are subject to regulatory requirements established by various regulatory bodies. Rigorous control of the discharges is implemented by the facility operations within the framework of water and air discharge permits. This control is carried out from commissioning of the installation and throughout its entire lifetime. In particular, this involves making measurements of the physical, chemical and radioactivity characteristics in the gaseous and liquid effluents. The decommissioning of these nuclear installations also generates liquid and gaseous effluents that should be characterized and quantified before their discharge.

Tritium and carbon-14 are usually present in the gaseous effluents of nuclear power plants and other types of nuclear installations. ISO 2889 presents the methods and provisions for sampling airborne substances from the exhaust stacks of nuclear facilities. The provisions defined therein cover all physical forms of the materials present in gaseous effluents: aerosol particles, vapours and gases. These provisions are more restrictive for radioactive aerosol particle measurements, given greater possibilities of losses in the transport lines.

Therefore, ISO 20041 goes further by addressing, in detail, the provisions specific to sampling methods, and sample preparation and calculations for determining the tritium and carbon-14 emissions. ISO 20041-1 covers the sampling methods or techniques for tritium and carbon-14. The future ISO 20041-2 will cover activity analysis of tritium and carbon-14 sampled by the bubbling technique and the future ISO 20041-3 will cover the activity analysis of tritium and carbon-14 sampled by molecular sieve.

Tritium and carbon-14 activity in gaseous effluents and gas discharges of nuclear installations —

Part 1:
Sampling of tritium and carbon-14

1 Scope

This document presents the methods and provisions for sampling tritium and carbon-14 in the gaseous effluents generated by nuclear facilities during operation and decommissioning. Specifically included are sample withdrawal location, extraction, transport flow measurement, and collection for later analysis.

This document doesn’t address to real time measurements of tritium activity and carbon-14 activity in the effluent air of stacks and ducts. Information about real time measurements can be found in ISO 2889:2021, Annex H.

Sample processing, analysis and calculations of tritium and carbon-14 emissions will be addressed in future parts of ISO 20041.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 2889, *Sampling airborne radioactive materials from the stacks and ducts of nuclear facilities*

ISO 5667-3, *Water quality — Sampling — Part 3: Preservation and handling of water samples*

ISO 10780:1994, *Stationary source emissions — Measurement of velocity and volume flowrate of gas streams in ducts*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at https://www.electropedia.org/

3.1
absolute humidity
mass of water per unit volume of moist air

3.2
activity
average number of disintegrations per second of a radionuclide

Note 1 to entry: The activity a is expressed in Becquerels (Bq).

Note 2 to entry: The discharged activity is calculated by multiplication of the activity concentration c and the discharged volume V.

3.3

activity concentration

activity (3.2) per unit volume of the *sample* (3.22)

Note 1 to entry: Activity concentration c is expressed in Becquerel per cubic meter (Bq·m^{-3}) or in Becquerel per litre (Bq·l^{-1} or Bq·L^{-1}).

3.4

adsorbent

material, generally a solid, that retains a substance contacting it through short-range molecular forces that bind the adsorbed material at the surface of the material

[SOURCE: ISO 2889:2021, 3.6]

3.5

aerosol

dispersion of solid or liquid particles in air or other gas

Note 1 to entry: An aerosol is not only the aerosol particles.

[SOURCE: ISO 2889:2021, 3.8]

3.6

coefficient of variation

C_V

quantity that is the ratio of the standard deviation of a variable to the mean value of that variable

Note 1 to entry: It is usually expressed as a percentage.

[SOURCE: ISO 2889:2021, 3.18]

3.7

collector

component of a *sampling system* (3.27) that is used to retain radionuclides for analysis

[SOURCE: ISO 2889:2021, 3.19, modified — The example was deleted.]

3.8

continuous measurement

real-time and in situ measurement of the characteristics of an *effluent* (3.11) without waiting and without conditioning and transport operations for the *sample* (3.22)

3.9

decision threshold

value of the estimator of the measurand, which, when exceeded by the result of an actual measurement using a given measurement procedure of a measurand quantifying a physical effect, is used to decide that the physical effect is present

Note 1 to entry: The decision threshold is defined such that in cases where the measurement result exceeds the decision threshold, the probability of a wrong decision, namely that the true value of the measurand is not zero if in fact it is zero, is less or equal to a chosen probability α.

Note 2 to entry: If the result is below the decision threshold, it is decided to conclude that the result cannot be attributed to the physical effect; nevertheless, it cannot be concluded that it is absent.

[SOURCE: ISO 11929-1:2019, 3.12]

3.10
detection limit

smallest true value of the measurand which ensures a specified probability of being detectable by the measurement procedure

Note 1 to entry: With the decision threshold, the detection limit is the smallest true value of the measurand for which the probability of wrongly deciding that the true value of the measurand is zero is equal to a specified value, β, when, in fact, the true value of the measurand is not zero. The probability of being detectable is consequently $(1 - \beta)$.

[SOURCE: ISO 11929-1:2019, 3.13]

3.11
effluent

exhaust (waste) stream beginning at the ventilated air space and flowing away from a process, plant, or facility to the environment

Note 1 to entry: A gaseous effluent is a fluid conveyed by a gas vector and may consist of gas, dust and/or particulates. In this document, the focus is on the gaseous and vaporous constituents of a gaseous effluent.

Note 2 to entry: An effluent can be subject to treatments, filtration, storage, recycling, controls and/or monitoring; and strict and regulated management. An effluent can be eliminated by dispersion into the receiving medium from a production site or within the immediate vicinity.

Note 3 to entry: The ventilated air space is the plane of entry of the effluent where the duct connects to a hood or snorkel or room vent.

[SOURCE: ISO 2889:2021, 3.30, modified — Modification in definition and additional Notes to entry.]

3.12
effluent sampling

action that involves taking one or more *samples* (3.22) of *effluent* (3.11)

3.13
flow rate

rate at which a mass or volume of gas (air) crosses an imaginary section in either a *sampling system* (3.27), tube, or a stack or duct

Note 1 to entry: The rate at which the volume crosses the imaginary area is called the volumetric flow rate; the rate at which the mass crosses the imaginary area is called either the mass flow rate or volumetric flow rate under standard conditions.

[SOURCE: ISO 2889:2021, 3.34]

3.14
hydraulic diameter

type of equivalent duct diameter for ducts that do not have a round cross-section

Note 1 to entry: Generally, it is four times the cross-sectional area divided by the perimeter.

[SOURCE: ISO 2889:2021, 3.38]

3.15
nozzle

device used to extract a *sample* (3.22) from an *effluent* (3.11) stream and transfer the *sample* (3.22) to a *transport line* (3.31) or collection device

Note 1 to entry: Within the nozzle, there is a transition zone where the sample stream adjusts to the conditions in the transport line. The nozzle is frequently a separate component and the nozzle exit plane is clearly defined as the downstream end of that component.

Note 2 to entry: The nozzle inlet corresponds to the imaginary cross-sectional inlet plane of a nozzle where the air stream first enters the transport system. In the special case of a shrouded nozzle, the inlet plane shall be taken as that of the nozzle and not that of the shroud.

Note 3 to entry: The nozzle exit (plane) is an imaginary plane across the cross-section of a transport system that divides the nozzle region from the transport line. If there is no separate component, the nozzle exit corresponds to the end of the transition zone of the nozzle air stream.

[SOURCE: ISO 2889:2021, 3.47, 3.48, 3.49]

3.16
off-line measurement

measurement of the characteristics of an *effluent* (3.11) *sample* (3.22) requiring either a waiting time before the measurement or conditioning and transport operations for the *sample* (3.22)

3.17
penetration

ratio of the concentration at the outlet of the *sampling system* (3.27), *transport lines* (3.31) included, to that in the duct or the stack

[SOURCE: ISO 2889:2021, 3.54]

3.18
probe

tubing or apparatus inserted into a stack or duct through which a *sample* (3.22) of the stream is withdrawn

Note 1 to entry: A probe usually refers to one or more nozzles and part of the transport line.

Note 2 to entry: Sometimes used colloquially to refer to the equipment inserted into a stack or duct for measurement of volumetric flow or amount of activity present.

[SOURCE: ISO 2889:2021, 3.57, modified — An additional Note 2 to entry was added.]

3.19
recording

action that fixes measurement data on a material support for their conservation

3.20
reference method

apparatus and instructions for providing results against which other approaches may be compared

Note 1 to entry: The application of a reference method is assumed to ensure correct results.

[SOURCE: ISO 2889:2021, 3.62]

3.21
representative sample

sample (3.22) with the same quality and characteristics for the material of interest as that of its source at the time of *sampling* (3.25)

[SOURCE: ISO 2889:2021, 3.63]

3.22
sample

portion of an air stream of interest, or one or more collected constituents from a portion of an air stream of interest

Note 1 to entry: The volume of the portion of the air stream has to be known.

[SOURCE: ISO 2889:2021, 3.65, modified — Note 1 to entry was added.]

3.23
sample-extraction location
sampling location
location of extraction of a *sample* (3.22) from the air

Note 1 to entry: Location of inlet of the sampling system.

[SOURCE: ISO 2889:2021, 3.66, modified — Definition and Note 1 to entry were modified.]

3.24
sampler
device that collects or analyses constituents of the air *sample* (3.22)

[SOURCE: ISO 2889:2021, 3.67]

3.25
sampling
process of collecting constituents from the air stream of interest on media such as filters, absorbents or *adsorbents* (3.4) and transporting them to a *collector* (3.7) or monitor for analysis or a container

3.26
sampling environment
conditions of the gas and airflow within a stack that can influence the *sampling* (3.25) process

Note 1 to entry: Factors to take into account include pressure, temperature and molecular composition of the gas.

[SOURCE: ISO 2889:2021, 3.70]

3.27
sampling system
system consisting of an inlet, a *transport line* (3.31), a flow conditioning system and a *collector* (3.7) or monitor

Note 1 to entry: Depending upon the application, a flow conditioner might not be used in the sampling system.

Note 2 to entry: Record samples are usually analysed off-line in a laboratory.

[SOURCE: ISO 2889:2021, 3.72, modified — An additional Note 2 to entry was added.]

3.28
sensitivity
change in indication of a mechanical, nuclear, optical or electronic instrument as affected by changes in the variable quantity being sensed by the instrument

Note 1 to entry: This is the slope of a calibration curve of an instrument, when this calibration curve shows output values of an instrument as a function of input values.

[SOURCE: ISO 2889:2021, 3.74]

3.29
stack
duct, channel or chimney for the dispersion and dilution of discharges into the environment

3.30
standard conditions
temperature of 298 K (25 °C) and a pressure of 101 325 Pa

Note 1 to entry: Used to convert air densities into a common basis. Other temperature and pressure conditions may be used and should be applied consistently.

[SOURCE: ISO 2889:2021, 3.76]

3.31
transport line
part of a *transport system* (3.32) between the *nozzle* (3.15) exit plane and the inlet plane of a *collector* (3.7) or analyser

[SOURCE: ISO 2889:2021, 3.78]

3.32
transport system
all components of a *sampling system* (3.27), excluding the *collector* (3.7) or analyser

[SOURCE: ISO 2889:2021, 3.79]

3.33
vapour
gaseous form of materials that are liquids or solids at room temperature, as distinguished from non-condensable gases

Note 1 to entry: Vapours are gases but carry the connotation of having been released or volatilized from liquids or solids.

[SOURCE: ISO 2889:2021, 3.83]

3.34
velocity profile
distribution of the velocity values at a given cross-section in a stack or duct

[SOURCE: ISO 2889:2021, 3.84]

3.35
volatile
material having a high vapour pressure, which allows significant quantities of material to become gaseous at prevailing temperature

Note 1 to entry: In this document, the stack temperature is generally considered as the reference temperature.

[SOURCE: ISO 2889:2021, 3.85]

4 Symbols

Symbols that are used in formulae in this document are defined below.

A	Cross sectional area of a stack or duct, in m^2
A_i	Cross sectional area of a stack or duct at the midpoint of the i^{th} element, in m^2
C_{af}	Velocity-averaging correction factor for determining flow rate in a stack or duct from a line average velocity taken with an acoustic flow meter, dimensionless
C_{pt}	Velocity-averaging correction factor for determining flow rate in a stack or duct from a single-point reading with a Pitot tube, dimensionless
C_{ta}	Velocity-averaging correction factor for determining flow rate in a stack or duct from a single-point reading with a thermal anemometer, dimensionless
H_r	Relative humidity, dimensionless
M	Molar mass, in $kg{\cdot}mol^{-1}$
M_v	Molar mass of water vapour equal to 0,018 $kg{\cdot}mol^{-1}$

N	Number of intervals or elements, dimensionless
p	Pressure, in Pa
p_{std}	Standard pressure equal to 101 325 Pa
p_{SV}	Saturation vapour pressure at a known temperature, in Pa
q	Volumetric flow rate, in $\mathrm{m^3 \cdot s^{-1}}$
q_{a}	Volumetric flow rate at actual conditions, in $\mathrm{m^3 \cdot s^{-1}}$
q_{std}	Volumetric flow rate at standard conditions, in $\mathrm{m^3 \cdot s^{-1}}$
q_{t}	Tracer injection flow rate, in $\mathrm{m^3 \cdot s^{-1}}$
R	Individual gas constant for a particular gas (equal to R_{u}/M), in $\mathrm{J \cdot kg^{-1} \cdot K^{-1}}$
R_{a}	Gas constant for air equal to 287 $\mathrm{J \cdot kg^{-1} \cdot K^{-1}}$
R_{u}	Universal gas constant equal to 8,314 $\mathrm{J \cdot mol^{-1} \cdot K^{-1}}$
T	Temperature, in K or °C
T_{std}	Standard temperature equal to 298 K (25 °C)
t_{s}	Sampling period, in s
V	Volume of effluent that produced the sample at stream temperature, pressure, and gas composition, in $\mathrm{m^3}$
V_{a}	Total volume of gas (air) sampled at actual conditions, in $\mathrm{m^3}$
V_{std}	Total volume of gas (air) sampled at standard conditions, in $\mathrm{m^3}$
v	Velocity, in $\mathrm{m \cdot s^{-1}}$
v_i	Velocity at the midpoint of the i^{th} element, in $\mathrm{m \cdot s^{-1}}$
v_{std}	Equivalent velocity at standard conditions, in $\mathrm{m \cdot s^{-1}}$
$v_{\mathrm{std},\, i}$	Equivalent velocity at standard conditions at the midpoint of the i^{th} element, in $\mathrm{m \cdot s^{-1}}$
ρ	Density, in $\mathrm{kg \cdot m^{-3}}$
ρ_{std}	Density of air at standard conditions equal to 1,184 $\mathrm{kg \cdot m^{-3}}$
ρ_{v}	Absolute humidity, in $\mathrm{kg \cdot m^{-3}}$

5 Sampling location

5.1 General

The sampling location shall provide the possibility to extract a representative sample.

A representative sample is best extracted from a location where the radioactive materials of interest are well mixed within the free stream. The term "well mixed" addresses several criteria that are given in 5.3. The designer should design the ventilation system to provide a favourable location where the sample can be extracted from a well-mixed stream (see 5.4). In this case, the sampling probe may contain a single nozzle. In circumstances where the well mixed criteria are not achieved, a multi-nozzle probe may be used or can be required to get a representative sample. The design and operation of sampling probes are described in ISO 2889:2021, 7.3.

Following a careful evaluation (see ISO 2889:2021, 7.8), one or more of the following steps should be taken in circumstances where these criteria cannot be satisfied in effluent systems designed and constructed prior to the publication of this document.

a) Select another location for the sampling probe.

b) Install features that promote mixing.

c) Perform an in situ test demonstrating that a representative sample is being collected.

NOTE The 3rd option is usually the most used.

5.2 General selection of sampling locations

The stack or duct geometry and the airflow within should be fully understood. Usually, for a stack, the sample extraction location should be situated between the discharge of the fan(s) and the stack exit point, with the provision that the location should not be so close to the stack exit that wind effects can significantly influence the velocity profile at the sampling location. Typically, in a well-mixed airflow, successful sample probe locations are in the range of 5 to 10 hydraulic diameters downstream of a flow disturbance and 3 or more hydraulic diameters upstream of a flow disturbance. There can be instances where greater distances are needed. Particular attention should be given to the geometry of flow entry conditions. Any addition of a small secondary air stream close to the wall of the stack or duct should be avoided. Bends, fans, duct junctions and similar disturbances promote mixing, but can also produce distortions in the velocity and the contaminant concentration profile and angularity in the airflow in the first 2 to 3 hydraulic diameters downstream. Therefore, sampling locations too close to such disturbances should be avoided, even at the cost of longer transport lines.

In addition to the physics of obtaining a representative sample, there are other considerations in locating the probe and associated equipment. The location should be readily and safely accessible, it should not present a problem for sampler servicing and maintenance activities and it should accommodate analysis or collection equipment that does not compromise the quality of the sample. High radiation fields under post-accident conditions can present a problem with respect to worker safety at the sample-extraction location. High ambient temperatures or humidity can also be a problem in some cases. Either of these situations can dictate the requirement for transport lines longer than normally required to accommodate installation of the sample collection and analysis equipment.

5.3 Criteria for the homogeneity of the sampling locations

Criteria for the homogeneity that signify a well-mixed location for sample extraction are given in ISO 2889:2021, 6.3. If the tritium or carbon-14 sampling probe is also used for sampling aerosol particles, then all the requirements of ISO 2889 shall apply. However, to the extent tritium and carbon-14 are considered as gases and vapours, the well mixed criterion would include only the gas concentration (tracer) uniformity, velocity profile, and flow angle.

A tracer gas (e.g. alcohol, sulphur hexafluoride, helium, nitrous oxide) is introduced into the airflow upstream of the sampling location. The tracer gas concentration is measured with the grid of measurement points using ISO 10780. The coefficient of variation of concentration at the measurement points is calculated. The coefficient of variation of the tracer gas should be less than or equal to 20 % across at least the centre two-thirds of the section of the stack or duct. Furthermore, at no measurement point should the concentration of the tracer gas differ by more than 30 % from the mean value for all of the points. The general method is outlined in ISO 2889:2021, Annex F.

Computational fluid dynamics (CFD) modelling may provide information supporting the selection of a well-mixed location and would generally be followed by physical measurements. CFD modelling requires a solution to the tracer gas profile using a boundary-fitted, three-dimensional flow. The CFD model is useful in visualizing and understanding bulk flow behaviour in the final exhaust system to assist in determining whether a more advantageous sampling location is feasible. CFD simulation is generally divided into the following steps: 1) simulation of the flow field, 2) particle tracking, and 3) post-processing to obtain particle deposition information[14]. Various models are available. The model should use a fully developed three-dimensional mesh of the full geometric detail and include the sections from the last disturbance to the exhaust point of the system. The CFD simulation should incorporate the appropriate aspects of the system and run long enough to achieve convergence. Output results from CFD are directly influenced by the quality of the model inputs such as the mesh model and running to convergence. Examples of methodologies for obtaining CFD data are given by[4][13][14][15].

If a gas container, also known as vessel, is used for collecting a huge volume of gas containing tritium and carbon-14 (see 6.4.2.4) the homogeneity of the gas inside the gas container should be as perfect as possible by distinguishing the internal mixing (gas injection mixing the effluent or internal recirculation) or the recirculation of the gaseous atmosphere by sampling the gas at one end of the vessel and reinjecting it into the other end. Homogeneity can be achieved by injecting a non-radioactive neutral gas with a predetermined volume according to the volume and initial pressure of the gas container. In the case of circulation in a non-pressurized closed gas container, the homogeneity of a gas containing tritium and carbon-14 can be obtained by using an internal mixing system. The use of a pressure-reducing valve/flow controller is recommended for pressurized gas containers. When the gas containers are pressurized, they are reputed to be subject to better homogenization.

5.4 Design of gaseous effluent discharge systems for sampler positioning

Extracting a representative sample from a stack or duct is best accomplished where the potential radioactive substances are well mixed with the airflow. When designing new stacks, or remodelling old stacks, the designer should also include provisions that promote mixing downstream of the fans, using abatement equipment as well as sampling downstream of all additions to the air stream.

The sampling probe (if this has a single nozzle) should be placed at the location where the measurement is the closest to the mean value of the gas concentration profile.

When changes to the air exhaust system are likely to modify the airflow pattern at the sampling location, the previous verifications should be repeated.

6 Sampling systems

6.1 General

In the following subclauses, the characteristics mentioned apply for the sampling of tritium and carbon-14 gaseous molecules. When the transport lines used for aerosol particle sampling are common to those of tritium and carbon-14, the requirements relating to aerosol particle sampling in these lines defined by ISO 2889 should be applied.

The penetration of gases and vapours of interest from the free stream to the sample collector shall be determined.

Performance criteria introduced in this document for the sampling of gaseous effluents may be considered as recommendations for sampling systems intended for monitoring only.

The following subclauses may not cover all possible situations and may be adjusted according to specific situations where the tests are neither appropriate nor practicable and according to the regulations in force. A risk-based graded approach may be used in the design of systems for sampling radionuclides in stacks and ducts of the nuclear industry. However, formulating such an approach is beyond the scope of this document.

6.2 Evaluation of an existing sampling system

If an existing gaseous effluent sampling system has not been designed according to the performance requirements and recommendations of this document, an evaluation of the performance of the system, including the location of the sampling nozzle, is recommended. If deviations are discovered, an optimization study should be performed to determine if a posteriori modification is useful and possible. Arriving at a suitable solution requires optimizing contradictory factors. The evaluation of existing systems should be undertaken using proven techniques described hereafter for the design and operation of new sampling devices.

6.3 Volumetric flow rate measurement

6.3.1 General

Accurate measurements of airflow in both the air sampling system and in the stack or duct being sampled should be provided because they directly impact the accuracy of emissions estimates. Errors are introduced into the calculation of emissions if the effluent and sample flow rate units are not based on the same gas density. This becomes significant where airflow is at either elevated or depressed temperature or pressure, for example if the facility is at an elevation of more than 300 m above sea level or if the sample flow meter is on the vacuum side of the sampling pump.

NOTE The regulations in force can specify the gas density conditions to apply for reporting discharges.

In calculating the amount of effluent air, the user should either adjust for the density differences in the air or use measurements based on a standard density. Typical conditions for standard density flow measurements are a pressure of 101 325 Pa and a temperature of 298 K (25 °C). Flow measurements at these conditions are reported in units of $m^3 \cdot s^{-1}$ and are represented by the symbol, q_{std}. The use of so-called mass flow meters in both the emission and sampler airflows, calibrated, can eliminate the need to make density adjustments.

6.3.2 Effluent flow rate measurement

The airflow of effluent streams sampled should be continuously measured if the flow rate is anticipated to vary by more than 20 % per year (if historical data are available, the 20 % value can be approximated by the standard deviation of the measurements.) Factors such as fan maintenance, the opening of doors and the variations in the number of fans should be taken into account in determining the requirement for continuously measuring flow rate.

If continuous measurement of flow rate is not needed, then periodic manual measurements of flow rate should be performed at least annually in accordance with ISO 10780. This standard method, as modified in Annex B, is denoted hereafter as the reference method.

For stacks and ducts that it is necessary to monitor continuously, the flow measurement and recording system should be capable of determining the mass or volumetric flow rate of the effluent stream with an accuracy that is within 10 % of that measured with the reference method.

Any continuous flow measurement device should be subjected to minimum annual accuracy audits. If the sensor of the continuous flow measurement device is based on electronic or acoustical principles, periodic checks of the instrument zero and span (or linearity) should be made, e.g. recommended by the manufacturer or regulatory bodies.

Overpressure ducts or stacks may sometimes be subject to a loss of flow rate between the fans, subject to pressurization, and the flow rate measurement point. In this case, any loss of flow rate should be identified so as to maintain an accuracy within 10 % on the flow rate measurement made with the reference method.

The uncertainty to be retained in the uncertainty calculations is that associated with the flow rate measurement method effectively implemented in the installation.

6.3.3 Sampling volume and flow rate measurement

The relative accuracy of the sample airflow rate measurement should be close to 10 % of the reference flow rate. The flow sensor should be placed in the sampling system so as to prevent any loss of aerosol particles or reactive radioactive gases. Consequently, the flow sensor is typically located more near and downstream of the sampling system or the sample analyser. Also, the density at the flow meter could be different from the density of the stack or extraction duct.

The sample flow rate should be displayed. If a mass flow meter is not used, pressure and temperature sensors should be added to enable calculation of the density of the sample at the flow meter. Usually, bubblers comprise a mass flow meter which eliminates the need for corrections related to pressure and temperature variations.

There can be differences between the effluent and the sampling flow rates. Therefore, the effluent flow rate and the sample flow rate should always be compared for minimizing the arising uncertainties.

If the sample flow rate is relatively constant during the sampling period, t_s, then as a minimum it should be recorded at the start, $t_{s,1}$, and the end, $t_{s,2}$, of each sampling period, in such a case, the total sample volume, V_a, may be calculated using the following Formula (1):

$$V_a = \frac{q_{a,1} + q_{a,2}}{2} \cdot t_s \qquad (1)$$

If the sampling flow rate is anticipated to vary by more than 20 % during the sample period, t_s, a flow meter should then be used continuously. In the case of continuous flow rate measurement, the values should be recorded at intervals not exceeding 10 minutes. The total sample volume corresponds to the integration of flow during the entire sampling period. If the time interval between recordings is Δt, and the sampling flow rate during this time interval is the mean of the two initial and final flow rates, $q_{a,i}$, the total sample volume V_a is calculated using Formula (2):

$$V_a = \sum_{i=1}^{N} q_{a,i} \cdot \Delta t \qquad (2)$$

NOTE Other integration modes may be used if the numerically induced errors do not exceed those that are implicit in Formula (2). The total sample volume, V_a, is based on the flow rates indicated by the flow meter.

The total sample volume is often related to the standard conditions (see 3.31).

If a controller is used to maintain constant sampling, the controller should maintain the sampling flow rate within a range of more or less 15 % for conditions that correspond to an initial pressure drop across the sample collector to a value that is twice the initial pressure drop. The flow controller should be tested under conditions similar to the operating conditions.

If the effluent flow rate can vary more than 20 % on average over a sampling period, the associated uncertainties should be quantified or corrections or justifications corresponding to the specific discharge conditions should be made for the installation.

The ratio between sampling flow rate and gaseous effluent flow rate should be maintained within 20 % of a predetermined value under normal operating conditions. An exception may be made on this point for an air monitoring system with a real-time contamination sensor operating with a constant predefined flow rate.

6.3.4 Leak tightness checks

A leak in the sampling system or at the sample collector can produce an erroneous sampling flow rate and also incorrect functioning of the sample collector. The leak tightness of the sampling system should be checked during installation and, in particular, during modifications to the transport line; the verification or test methodology should be documented and easy to install. Additionally, periodic routine checks (quarterly, annually, or as appropriate and practicable) on the leak tightness of the system should be conducted.

For specific gas (tritium and carbon-14) activity flow rate measurements in the ducts and stacks, if the parameters for discharge flow rates, sampling flow rate and leak tightness cannot be specifically measured, a gas tracing test should be performed on the entire sampling device to qualify the sampling and quantify the overall uncertainty associated with the sampling.

NOTE Visual inspection and the observation of foreign materials on samples can identify large leaks. If the sampling system is strategically equipped with full-bore ball valves, then parts of the system can be isolated for vacuum or pressure decay measurements or a measuring flow through a blocked system. An explanation of the latter approach is to block flow through the nozzle, then apply vacuum to the transport line and measure the leakage rate. For example, a mass flow meter could be attached downstream of the collector or monitor and the vacuum source connected to downstream side of the mass flow meter. The pressure level in the tubing between the collector or monitor and vacuum source would be adjusted to the nominal value encountered during sampling (typically about 4 000 Pa for sampling systems that involve use of collection filters) by bleeding air into the line downstream of the flowmeter.

6.4 Gas and vapour transport and sampling

6.4.1 General

Tritium and carbon-14 are frequently present in the gaseous effluents of nuclear installations. Gaseous effluents can be sampled with different methods depending on whether:

- the gases and vapours are in the presence of radioactive aerosol particles also to be sampled;
- the gases and vapours are reactive with the materials of the sampling system.

When the gases and vapours are the only species sampled, the sampling recommendations are substantially simpler than those concerning the aerosols defined in ISO 2889.

6.4.2 General provisions

6.4.2.1 General

The laboratory in charge of analysis of the physico-chemical characteristics needs to be informed of the type of gaseous effluent sampled, so that it implements suitable measurement techniques, in particular in order to take into account possible interferences as X-rays, gamma and beta emitters in the case of tritium and carbon-14 during liquid scintillation counting.

6.4.2.2 Non-reactive gases and vapours

When non-reactive gases and vapours are the only species sampled, the recommendations for reducing particle line-loss are irrelevant. Condensation due to temperature changes in the line should be avoided (see 6.4.3.2). If the effluent air stream can contain only gaseous contaminants, the nozzle design is not critical, but the sampling should take place at a location where the air stream is well mixed and meets the criteria of 5.3. The nozzle design can be simply an open-ended or perforated tube. The extraction and transport requirements that apply aim to extract the sample from a well-mixed location and avoid water vapour condensation in the transport and collection system except when condensation is used as the collection method.

When non-reactive gases, vapours and particles are simultaneously sampled, the particle sample extraction and transport requirements should apply, which also ensures adequate transport of the gas and vapour sample. The last remaining consideration is the selection of appropriate collection devices. The gas or vapour collection device should be located downstream of a particulate filter to eliminate potential interferences of radionuclides bound on particles.

NOTE 1 Experience has shown (see Annex D) that an installation that complies with the sampling provisions defined in this document has a penetration of at least 90 % for non-reactive and non-condensable gases. The uncertainty on the measurement of each penetration with a coverage factor $k = 2$ is, in this case, estimated at 20 %.

NOTE 2 A demonstration of the acceptable character of the sampling installation (characterization and/or modelling of the sampling installation) specific to non-reactive gases should be carried out in order to assess its penetration with regard to an ideal value of 100 %.

6.4.2.3 Reactive gases and vapours

The minimum penetration for the transport of reactive gas and vapour samples from the free gaseous effluent stream to the collector or analyser should be optimized taking into account the following recommendations.

Particular attention should be given to the sampling system construction materials to minimize surface interactions which can lead to chemical transformations and condensation phenomena (also see 6.4.3.2). The construction material should have minimum reactivity with the gas: in particular, the use of organic materials shall be limited to avoid exchanges between tritium and carbon-14 and the sampling pipes.

The inner surfaces of the sampling probe and transport line should be constructed of, or coated with, non-reactive materials to minimize surface interactions with the sample. The possibility for conversion of a portion of the sample by chemical transformations induced by the sample transport process, such as conversion of molecular iodine gas into organic iodine vapour, should be understood and accounted for in the sampling system design.

Consideration should be given to the advantages of having a separate sampling system for the gases whenever the construction materials that are required for the transport of the particle and gas samples are incompatible. In situations where even a low level of reactivity cannot be avoided, the length of the transport line should be kept to a minimum. Penetration of the gas or vapour through the entire extraction and transport system should be documented.

If long transport lines are unavoidable, consideration should be given to the effect of transport and detection delay caused by deposition, chemical transformation and subsequent resuspension. Special attention should also be given to how significant the effect of delay has on the interpretation and usefulness of the data obtained. Although rapid emission variations can become attenuated over a long-time interval, this data can still be useful and its analyses give quantitative information on the discharge.

6.4.2.4 Collection of gas in gas containers

In some cases, a collection of gaseous effluents in a gas container may be of benefit towards direct sampling from transport lines and may be carried out in different ways:

- using a container in which a vacuum has been made beforehand and that is filled with part of the effluent stream to be sampled, then sealed and returned to a laboratory for measurement of the individual or overall activity of each of the constituents;
- passing part of the effluent stream through the container until the container is completely purged, then closing the inlet and outlet valves;
- pumping part of the effluent stream into deflated bags (manufactured from a non-adsorbing material) for subsequent compression and analysis;
- compressing the sample effluent stream into a bottle for subsequent analysis.

When the gas container is filled, it is important to distinguish the gas container with headspace that present risks of creating explosive atmospheres and to take measures for preventing deflagration risks (inerting, pressure vessels for preventing effluent intakes, flame-proof equipment, etc.).

A known volume of the collected gas is then used for the sampling described in 6.5.

NOTE Some closed gas containers can contain gases that present an explosion risk (e.g. presence of hydrogen at more than 4 %). Due to the inerting of these gas containers and the explosion risk, gas oxidation cannot be performed for sampling hydrogen gas (HT) and carbon-14 in organic compounds. In this case, a part of the volume of the gas container should be transferred in an intermediate container to eliminate the explosion risk by dilution of the gas in a larger volume before being analysed.

6.4.3 Special case of tritium

6.4.3.1 General

Tritium is a hydrogen isotope that has a behaviour similar to that of hydrogen. It is usually found in different chemical compounds in the effluent stream (gaseous effluent):

- hydrogen gas, HT,
- tritiated water vapour, HTO (boiling point slightly higher than 100 °C),
- methane or another volatile organic compounds, or
- sometimes bound on aerosol particles

The first step in selecting a suitable sampling system is to determine the chemical compound of tritium in the effluent stream. If it is present in several forms, several sampling techniques can be used.

6.4.3.2 Condensation phenomenon of tritium in vapour form

If tritium is present as water vapour, the temperature and humidity content of the effluent stream shall be carefully considered because condensation takes place when the temperature of air in the sample transport line is less than the saturation temperature of the vapour of interest. Therefore, sample transport lines, collectors and analysers should be designed to avoid condensation of vapour. It can be necessary to thermally insulate, and in some cases heat, the sample transport line to prevent condensation (heat tracing). For situations in which heating of the sampling line can result in unacceptably high temperatures at a collector or analyser, a dilution system should be considered; however, care should be exercised to ensure that the dilution process does not produce condensation at the mixing location. Furthermore, experimental or numerical analyses should be performed to demonstrate the effectiveness of any design provisions that are intended to minimize or preclude the formation of condensation in sample transport systems.

Several tritium sample-collection methods exist that depend either on absorption of water vapour into a medium or condensation in a condenser apparatus. If the temperature of the sample is maintained too high, the absorbing solution evaporates, only a proportion of the vapour condenses and a proportion of the absorbing solution in the bubblers risks being lost. All of these conditions lead to biased results.

If the physical state of the tritium in the exhaust stream is a gas or vapour and the effluent stream contains a high humidity content, then heat tracing may be necessary to avoid condensation of the sample in the transport lines and in the sampling chamber. Condensation risks causing the sample collector to plug.

6.4.3.3 Collection support protection

A particulate filter should be installed on the sampling system upstream of the collection support to protect the flow meter located at the sampling system inlet and limit the accumulation of any other radioactive particles on this support.

Annex C contains the ranges of use for tritium measurement devices on different collection media.

6.4.4 Special case of carbon-14

Carbon-14 is an isotope of carbon with behaviour similar to that of stable carbon isotopes. It has been identified in the gaseous effluents of nuclear installations in the form of:

- particulate ^{14}C,
- gaseous carbon dioxide $^{14}CO_2$ and
- other gases (non-$^{14}CO_2$), e.g. carbon monoxide ^{14}CO or methane $^{14}CH_4$.

When selecting a suitable and appropriate sampling medium for carbon-14, it is important to take into account the presence of other contaminants (both radioactive and non-radioactive) in the sample air stream in addition to the physical and chemical forms of carbon-14 to be collected.

For example, in the case of the trapping of carbon-14 in the form of CO_2 on a molecular sieve, water contained in the gaseous effluent is also retained by the molecular sieve.

Annex C contains the ranges of use for carbon-14 collection devices.

6.5 Sampling techniques

6.5.1 General

In general, sampling with extraction and collection of specific constituents requires detailed knowledge of the chemical and physical properties of the radioactive material under investigation and also detailed knowledge of the materials that can interfere with the sample, such as particulate matter and non-radioactive gases (e.g. acids and organic compounds) that accompany these specific constituents. The many possible combinations of the properties of the constituents to be measured and the accompanying airborne materials require careful study to select the optimum collector. Gaseous and vapour components can be soluble in water, can be extremely reactive with certain solutions, can dissolve in specific non-aqueous solvents, or can be retained on certain specific adsorbents or in other specifically prepared media. In general, continuous or long-term sampling is carried out when separation or trapping of a constituent is essential. The characteristics of the sampling device (sampling flow rate and sampling time, trapping support retention container) should be adapted to the characteristics of the gaseous effluent sampled to ensure sensitivity compatible with the measurement method chosen and to prevent saturation of the sampling support.

The main sampling methods are based on the trapping of the radionuclide sought on solid (e.g. zeolite, silica gel, metal beds) or liquid (e.g. water, sodium hydroxide solution) supports, with or without catalytic or chemical reaction. The condensation technique can also be used specifically for tritiated water vapour.

In the following sub-clauses, the principles mentioned apply for the sampling of tritium and carbon-14 gaseous molecules. When the transport lines used for aerosol particle sampling are common to those of tritium and carbon-14, the requirements relating to aerosol particle sampling in these lines defined by ISO 2889 should be applied. This document also defines the measurement techniques for the flow rate in stacks and ducts (see Annex B) and the demonstration of the homogeneous character of the gaseous stream discharged necessary for the establishment of representative sampling (see Annex C).

6.5.2 Sampling by bubbling technique (tritium and carbon-14)

6.5.2.1 General

The bubbling technique is a method commonly used for performing tritium and carbon-14 activity concentration measurements in gaseous effluents.

6.5.2.2 Principle

Gaseous effluent sampling is carried out continuously by pumping either from the transport line or a gas container.

After filtering the aerosol particles of the gaseous effluent, the air sample bubbles through a unit of a first series of at least two bubblers containing an appropriate trapping solution in order to collect tritiated water vapour or gaseous $^{14}CO_2$. Then it passes an oven in which the elementary form of tritium HT or non-CO_2-gases are oxidized to water vapour HTO or gaseous $^{14}CO_2$ in the presence of a catalyst and oxygen. The oxidized compounds are trapped in a second series of at least two bubblers containing the same trapping solution (see Figure 1).

Commonly two trapping units are used, one for trapping tritium and one for trapping carbon-14. The most used trapping solutions are respectively water for tritium, and a sodium hydroxide solution for carbon-14.

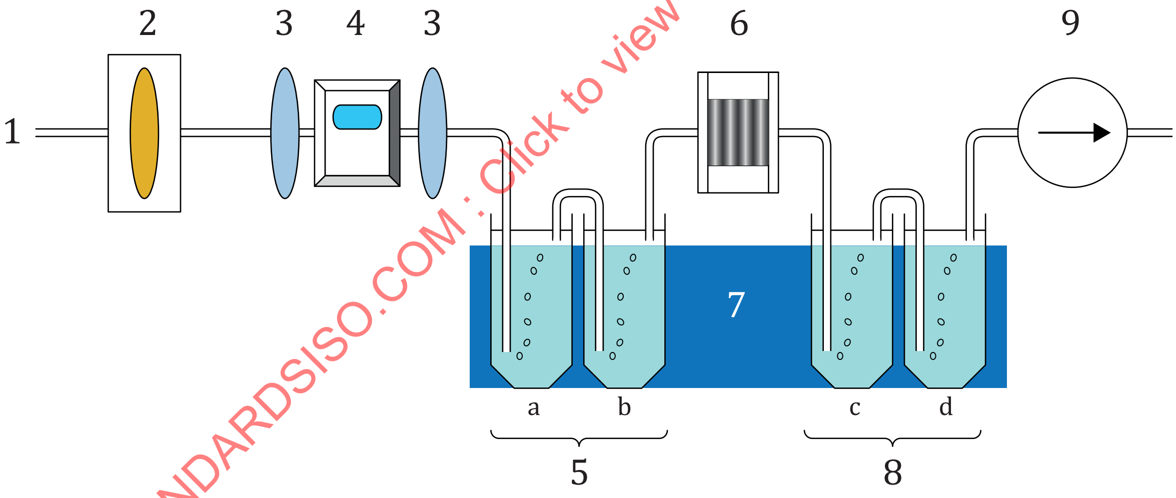
The trapping solution of each bubbler is collected and then the activities of tritium and carbon-14 are measured by liquid scintillation counting.

This technique with off-line measurement in a laboratory is used to determine effluent activity concentrations that may be low (approximately 1 Bq·m^{-3} for tritium and carbon-14) in stacks.

Annex C presents the associated measurement ranges for various sampling techniques.

6.5.2.3 Apparatus

Figure 1 shows the example of a bubbler unit with four bubblers intended for the sampling of tritium or carbon-14 depending on the trapping solution.



Key

1 transport line or gas container
2 a particulate filter, e. g. anti-dust filter
3 hydrophobic filter
4 flow meter
5 bubbler unit (a, b) with appropriate trapping solutions for HTO and gaseous $^{14}CO_2$
6 catalytic conversion oven
7 cold unit
8 bubbler unit (c, d) with adapted trapping solutions for HTO and gaseous $^{14}CO_2$ after catalytic conversion
9 pump

Figure 1 — Block diagram for sampling tritium or carbon-14 by the bubbling technique

The apparatus used includes:

- a sampling head with dust-retaining filter;
- a mass or volumetric flow meter, associated with a pump flow rate control, protected by hydrophobic filters positioned upstream and downstream;
- a volumetric counter upstream of the bubblers to avoid taking into account any leaks at the bubblers;
- a connection pipe between the sampling head and the first set of bubblers that is as short as possible, made of a material the nature and condition of which reduce the retention of water vapour and isotopic exchanges with hydrogen;
- a temperature heating or maintenance device that prevents condensations;
- a series of at least two bubblers to trap tritiated water vapour or gaseous $^{14}CO_2$;
- a pump located downstream of the bubblers.

If required, the apparatus can be completed by:

- a cooling system for the bubblers to reduce the evaporation;
- a catalytic conversion oven ("oxidation oven") with a catalyst, which is a finely divided metal such as copper, nickel, platinum or palladium.

  NOTE 1 A platinum catalyst is usually used for oxidation of the organic forms of tritium. The efficiency of the catalytic conversion oven should be determined (see 6.5.2.4.1).

  NOTE 2 The oven temperature shall be adjustable and permit oxidation of the various forms of tritium and carbon-14 found in the gaseous effluents of the installation.

  NOTE 3 A cooling system downstream of the oven obtains a relative humidity close to 100 % and prevents the liquid receiving medium in the bubblers from drying.

- a second series of at least two bubblers to trap tritiated water vapour or gaseous $^{14}CO_2$ after oxidation.

6.5.2.4 Sampling environment and precautions for use

6.5.2.4.1 General

Many quantities may influence the sampling or the measurement. Special precautions shall be taken to limit as much as possible the influence of the parameters that distort the sampling and that make the measurement results non-representative of the situation of interest.

Thus, consideration should be given to the influence of:

- the humidity of the effluent on the sampling;
- the temperature of the effluent on the sampling;
- the sampling flow rate;

  NOTE 1 If the flow rate is too high, it can cause losses of tritium and carbon-14, respectively, and reduce the oxidation and/or efficiency of the collection medium;

- the leak tightness of the bubbler unit;
- the presence of other radionuclides;
- the efficiency of the collection medium;

NOTE 2 To determine or verify the efficiency of the collection medium, various methods can be used. Among these, a conventional sampling device with four bubblers can be used whose catalytic conversion oven is either switched off to determine the collection efficiencies of bubblers a, b and c or switched on to determine the collection efficiencies of the bubblers a and c. After sampling, the measurement in each of the four bubblers is used to quantify the efficiency of the collection medium as well as its associated uncertainty, e.g. standard deviation of reproducibility on a population of data. Under these conditions, the efficiency of the collection medium for HTO is usually (86 ± 7) % with $k = 1$ for each bubbler[6]. If the first two bubblers are considered the efficiency is (96 ± 5) %. For CO_2, the efficiency of the collection medium is determined by calculating the mass of collected carbon from the mass of the barium carbonate precipitate obtained. Commonly, the efficiency of the collection medium for CO_2 is (87 ± 4) % with $k = 1$ for each bubbler[12]. Regarding two bubblers, the combined efficiency is (98 ± 8) % with $k = 2$. These values are highly dependent of the sampling time, climatic conditions, sampling flow rate, chemistry and volume of the trapping solution, etc.

— the efficiency of the catalytic conversion oven;

NOTE 3 To determine or verify the efficiency of the catalytic conversion oven, various methods can be used. Among these, two conventional sampling devices with four bubblers in series can be positioned, with ovens switched on, and carried out in the same way. A measurement in each of the bubblers b and c of the two sampling devices in series quantifies the efficiency of the catalytic conversion oven as well as its associated uncertainty, e.g. standard deviation of reproducibility on a population of data. Under these conditions, the efficiency of the catalytic conversion oven for the conversion for HT is usually (98 ± 3) % with $k = 1$[6]. For CO_2, the efficiency of the catalytic conversion oven is determined by calculating the mass of collected carbon from the mass of the barium carbonate precipitate obtained. Commonly, the efficiency of the catalytic conversion oven is (94 ± 3) % with $k = 1$. These values are highly dependent of the sampling time, oven temperature, type of catalyst, sampling flow rate, chemistry and volume of the trapping solution, etc.

— the evaporation of the trapping solution;

NOTE 4 There is a risk of reduction of the saturation and efficiency of the collection medium distorting the measurements.

— the saturation of the trapping solution;

NOTE 5 In the event of precipitation, sampling cannot be used and the sampling times are readjusted and/or the trapping solution is adapted.

The precautions retained to minimize the influence of these parameters are given in the following subclauses.

6.5.2.4.2 Sampling room

The temperature of the room should be limited to reduce the evaporation of the trapping solution, which shall not exceed that recommended by the bubbler manufacturer, a fortiori in the case of uncooled bubblers.

To limit the exchanges between the bubbler and the atmosphere of the room, a chilled bubbler should be used, placed in a room whose atmosphere is well ventilated.

6.5.2.4.3 Flow rate, sampling time, volume and detection limit

In the case of integrated sampling, it is desirable to continuously sample a known volume of effluent at a constant sampling flow rate to ensure representativeness over time.

The volume of effluent is determined from the flow rate measurement. If the flow meter is volumetric, the volume of effluent shall be brought to normal pressure and temperature conditions.

The measurement quality of this volume sampled is ensured by verification of the calibration of the flow meter and its connection to the internal system. This volume shall be known with its associated uncertainty.

The sampling time and flow rate shall be suitable for the activity of the medium and for its foreseeable fluctuations, and in agreement with the regulatory requirements.

The volume of the effluent sampled is suitable for the detection limit sought and for the apparatus used.

The effluent flow rate shall be compatible with good absorption of the tritiated water vapour or gaseous $^{14}CO_2$ in the trapping solution.

NOTE For example, an amount of effluent sampled of 1,5 m^3 achieves a detection limit of approximately 1 $Bq \cdot m^{-3}$ of effluent, for a flow rate of approximately 30 $l \cdot h^{-1}$, which is suitable for a sampling time of 24 h to one week.

6.5.2.4.4 Trapping solution

The bubblers shall contain an appropriate trapping solution in sufficient amount for the requirements of the measurement (e.g. 150 cm^3):

- for tritium:

  Any type of water with a known content of low tritium activity selected in agreement with the laboratory responsible for the measurement (e.g. deionized water, mineral water, mains water, groundwater) can be used. This same solution shall necessarily be identical to that used for the measurement and preparation operations (example of the completion of blank tests) in a laboratory;

  NOTE When the equipment is intended to operate under low temperature conditions below zero, a few percent of anti-freeze liquid may need to be added to the trapping solution. However, too high a proportion of glycol in the water produces a significant chemiluminescence phenomenon during the liquid scintillation measurement. The addition of this additive can have an influence on the efficiency of the collection medium that is quantified.

- for carbon-14:

  A sodium hydroxide solution (e.g. NaOH, 1 $mol \cdot l^{-1}$ or 2 $mol \cdot l^{-1}$) with a low carbon-14 content selected in agreement with the laboratory responsible for the measurement is used. The molarity of the basic solution shall be adapted according to the flow rate, sampling time and volume of solution to prevent its saturation. This same solution shall necessarily be identical to that used for the measurement and preparation operations (example of the completion of blank tests) in a laboratory.

The bubblers should be filled so as to ensure a sufficient efficiency of the collection medium (e.g. a bubble path in the receiver of approximately 7 cm for a flow rate of 20 $l \cdot h^{-1}$ to 30 $l \cdot h^{-1}$); the apparatus should be equipped with devices giving rise to micro bubbling.

The bubblers shall have a sufficient gap to prevent one bubbler from overflowing into the following bubbler.

The amount of trapping solution in each bubbler needs to be measured with a known accuracy, e.g. by weighing or graduated test specimen, at the start and end of the sampling period.

To quantify the uncertainty associated with the sampling, the efficiency of the collection medium, the efficiency of the catalytic conversion oven and their respective uncertainties, provided by the manufacturer from experimental bases, need to be considered.

6.5.2.4.5 Risk of contamination of the equipment

Precautions shall be taken to avoid contamination of the equipment; by way of example, these precautions may be:

- systematic cleaning of the bubblers (e.g. dishwasher) or use of single-use bubblers;
- regular changing of the bubblers;
- systematic changing of the bubblers following a higher discharge episode;

- dummy operation of the apparatuses (in non-tritiated ambiance) to verify their radiological cleanliness, etc.

NOTE Glass bubblers have the advantage of not salting out the tritium with the trapping solution after the first use.

6.5.2.5 Conditioning, transport and preservation of samples

At the end of sampling, the trapping solutions are removed from the apparatus then conditioned as quickly as possible to be transported to the laboratory, directly in the bubblers or in specific bottles. In the latter case, the dead volume should be limited to minimize gaseous transfers.

The transport and preservation of samples shall be carried out in accordance with ISO 5667-3.

Therefore, this concerns:

- identifying and labelling the trapping solution containers;
- limiting sample temperature variations (e.g. transport in cold boxes);
- protecting the sample from light

  NOTE Multiparametric studies of storage stability of tritiated water in weakly tritiated mineral water, e.g. dark and refrigerated versus day-/night-cycles at room temperature and storage time, were carried out in order to assess the impact of the conditions of conservation. Due to these tests, it appears that tritiated water solutions can be stored at room temperature not exceeding 30 °C for up to 60 days[10].

- paying attention to the risk of "inter-sample" contamination in the event of transfer of trapping solutions from bubblers into specific bottles;
- verifying the integrity of the sample;
- limiting the transport time, etc.

The chemical risk related to using a caustic solution as a trapping solution should also be taken into account during the sample transport and handling phases (e.g. use of personal protection equipment suitable for operators).

6.5.3 Sampling using molecular sieves (tritium and carbon-14)

6.5.3.1 General

The molecular sieve method is used in this document for the simultaneous sampling of tritium and carbon-14 in gaseous form, especially long-term sampling (e.g. monthly).

The molecular sieve, an adsorbent inorganic crystalline structure (e.g. zeolite), is capable of selectively adsorbing the molecules to which it is exposed, according to various parameters:

- critical molecular diameter

  NOTE The cavities in which adsorption occurs are accessible via pores of diameter suitable to that of CO_2 molecules or H_2O molecules.

- polarity (polar molecules are more significantly adsorbed);
- polarizability (polarizable molecules are better adsorbed);
- degree of saturation in hydrogen atoms (non-saturated compounds are preferably adsorbed);
- molecular mass (heavier compounds are better adsorbed).

6.5.3.2 Principle

Gaseous effluent sampling in ducts and stacks is carried out by continuous sequential pumping through a buffer tank, either from the transport line or a gas container.

Carbon in CO_2 form and tritium as water vapour, present in the effluent to be controlled, are adsorbed on an activated molecular sieve contained in a first cartridge. If the elementary form of tritium (HT) or non-CO_2-gases shall also be measured, the effluent passes an oven after the first cartridge in order to oxidize these compounds both into water vapour and CO_2 in the presence of a catalyst and oxygen. Upon exiting the oven, water vapour and CO_2 are trapped in a second cartridge containing the same kind of molecular sieve (Figure 2).

NOTE In the case where it is not necessary to make a distinction between the various chemical forms of carbon-14, a single molecular sieve cartridge can be used downstream of the conversion oven. This cartridge therefore traps all CO_2 and non-CO_2-compounds.

The molecular sieve of each cartridge is then collected.

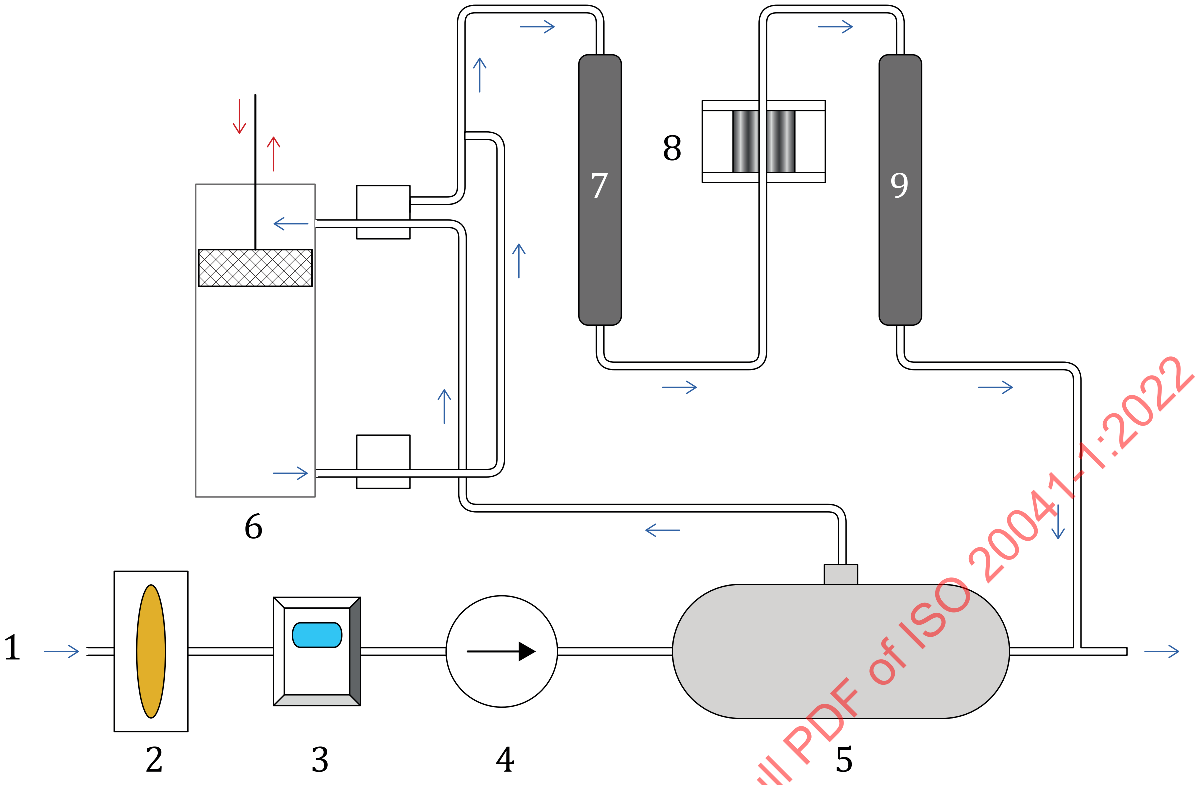
The trapped compounds on the molecular sieve are desorbed at high temperature and under nitrogen stream. Water vapour is condensed, and CO_2 is retained in a trapping solution. This treatment regenerates the molecular sieve and allows it to be reused.

Both the activities of tritium and carbon-14 in the trapping solution and the condensed water are measured by liquid scintillation counting.

This technique with off-line measurement in a laboratory is used to determine effluent activity concentrations that may be low in stacks, approximately 1 $Bq \cdot m^{-3}$ for tritium and carbon-14.

6.5.3.3 Apparatus

Figure 2 shows the example of a sampling device with a molecular sieve, intended for the sampling of carbon-14 and tritium.



Key

1	transport line or gas container
2	particulate filter, e. g. anti-dust filter
3	flow meter
4	pump
5	tank for stabilizing the pressure
6	piston pump
7	cartridge with molecular sieve for HTO and gaseous $^{14}CO_2$
8	catalytic conversion oven
9	cartridge with molecular sieve for HTO and gaseous $^{14}CO_2$ after catalytic conversion

Figure 2 — Block diagram for simultaneous sampling of tritium and carbon-14 by molecular sieve

The apparatus used includes:

- a sampling head containing a dust protection filter;
- a buffer tank, after the particulate filter;
- a pump, e.g. a piston pump;
- a device for measuring the volume sampled upstream of the molecular sieve cartridges;
- a connection pipe between the sampling head and the first cartridge;
- a molecular sieve cartridge (e.g. zeolite).

If required, the apparatus can be completed by:

- a catalytic conversion oven ("oxidation oven") with a catalyst, which is a finely divided metal such as copper, nickel, platinum or palladium (see 6.5.2.3);

— a molecular sieve cartridge placed at the exit of the oven in series to trap the tritiated water vapour or gaseous $^{14}CO_2$ after oxidation.

6.5.3.4 Sampling environment and precautions for use

6.5.3.4.1 General

Many quantities may influence the sampling or the measurement. Special precautions shall be taken to limit as much as possible the influence of the parameters that distort the sampling and that make the measurement results non-representative of the situation of interest.

Thus, consideration should be given to the influence of:

— the sampling flow rate (see 6.5.2.4.1);

— the presence of other radionuclides;

— the efficiency of the collection medium;

NOTE 1 The efficiency of the collection medium can be verified for example by placing two sampling devices in series.

— the efficiency of the catalytic conversion oven

NOTE 2 The efficiency of the catalytic conversion oven can be verified for example by placing two sampling devices in series.

— the saturation of the molecular sieve by the CO_2 or water that would come into competition with the retention of the CO_2.

The precautions retained to minimize the influence of these parameters are given in the following subclauses.

6.5.3.4.2 Flow rate, sampling time, volume and detection limit

In the case of integrated sampling, it is desirable to continuously sample a known volume of effluent at a constant flow rate to ensure representativeness over time.

The volume of effluent sampled is determined from the sum of the volumes sampled.

The measurement quality of this volume sampled is ensured by periodic verification of the sampling device. This volume shall be known with its associated uncertainty.

The sampling time and flow rate or the volume sampled shall be suitable for the activity of the medium and for its foreseeable fluctuations, and in agreement with the regulatory requirements.

The volume of the effluent sampled is suitable for the decision threshold sought.

The sampling flow rate shall be compatible with good adsorption of CO_2 or water vapour by the molecular sieve.

6.5.3.5 Conditioning, transport and preservation of samples

At the end of sampling, the molecular sieve cartridges are removed from the apparatus, preserved hermetically, to be transported to the laboratory.

The molecular sieve cartridges are identified and labelled, having measured their mass before and after the sampling.

6.5.4 Sampling by condensation (only tritium)

Sampling by condensation allows the tritium present in the air in the form of water vapour (HTO) to be measured without loss or dilution effect. With a high inertia cooling device, a sufficient amount of water (several tens of ml) can be condensed within a few tens of minutes to then the activity of tritium be analysed by liquid scintillation.

To link the activity measured in the water vapour to the corresponding volume of gaseous effluents, the absolute humidity of the effluent in which the sampling was performed needs to be known. This determination is performed by reading the atmospheric pressure, humidity and temperature (in K) parameters and using the following Formula (3):

$$\rho_\mathrm{v} = \frac{M_\mathrm{v}}{R_\mathrm{u} \cdot T} \cdot H_\mathrm{r} \cdot p_\mathrm{sv}(T) \quad (3)$$

Thus, the activity concentration can only be determined if the absolute humidity of the air varies little during the sampling. Consequently, this technique is difficult to apply for continuous long-term sampling. Therefore, it can only be used reliably for short sampling and therefore condensation periods less than one hour in large-volume discharge ducts or containers (several m^3) such that the sampling does not influence or influences little the absolute humidity of the air sampled.

Due to the absence of dilution of the sampling whose efficiency is total (equal to 100 %), devices with condensation of the water vapour of the air allow atmospheric tritium to be measured from the natural level (0,01 $\mathrm{Bq \cdot m^{-3}}$ to 0,02 $\mathrm{Bq \cdot m^{-3}}$) when liquid scintillation analysis presents a detection limit of approximately 1 000 $\mathrm{Bq \cdot m^{-3}}$.

To prevent aerosol particles present in the sample from disturbing the measurement, a filter should be installed upstream of the condensation device that is suitable for the particles that may affect the quality of the condensate.

7 Sampling sheet and follow-up sheet

7.1 General

The necessary data for the expression of results is explained in the following and shall be the subject of a follow-up sheet. An informative presentation of this necessary data is given in Annex A.

7.2 Sampling sheet

These elements are subjects of a record, depending on the sampling technique:

- the identification of the sample (sampling location reference);
- the date and time of the start and end of sampling;
- the nature of the collection medium;

  NOTE The collection medium is identical to that used in a laboratory for the measurement.

- the volume or the mass, respectively, of the collection medium in each collection unit, at the start and at the end of the sampling period;
- the total volume of effluent sampled during the sampling period at standard conditions;
- the average volumetric flow rate in the sampling system during the sampling period at standard conditions;
- the homogenization technique and the associated parameters at the collection in a gas container;
- the dilution factor, if the sample is subject to a physico-chemical treatment prior to the measurement.

7.3 Follow-up sheet

Furthermore, the following information shall be tracked in the follow-up sheet:

- the operating parameters specific to the sampling system (oven temperature, operational chiller unit, effective bubbling, etc.);
- the average volumetric flow rate of the effluent air in the stack or duct during the sampling period at standard conditions;
- the total volume of effluent that passed through the stack during the sampling period or was inside a gas container at standard conditions;
- the efficiency of the collection medium and the associated uncertainty;
- the efficiency of the catalytic conversion oven and the associated uncertainty;
- the penetration (representing the sampling quality) and the associated uncertainty;
- the absolute humidity and the associated uncertainty, if necessary.

All these uncertainties shall be expressed without coverage factor ($k = 1$).

The errors related to the sampling quality and losses in the transport line (penetration), and the associated uncertainties, shall be tracked in the expression of results sheet.

Annex A
(informative)

Example of a sampling sheet (bubbling technique/molecular sieve method)

NUCLEAR INSTALLATION: **OPERATOR:**

GENERAL INFORMATION				
Sampling	Stack		Gas container	
Sampling device				
Nature of the collection medium	Water (H-3)			
	NaOH (C-14)			
	Molecular sieve (H-3/C-14)			
Nature of the catalyst				
SAMPLING PARAMETERS of the collection medium				
Bubbling technique				

Bubbler		**Mass of the trapping solution, in g**		**pH of the NaOH (≥ 8)**	**Sample identification**
No.	**type of nuclide (e. g. HTO, CO_2)**	**at sampling start**	**at sampling end**	**after sampling**	
1					
2					
3					
4					

Uncertainty of the used scale in g:

Molecular sieve

Cartridge		**Mass of the cartridge in g**		**Sample identification**
No.	**type of nuclide**	**at sampling start**	**at sampling end**	
1	HTO/CO_2			
2	non-HTO/non-CO_2			

Uncertainty of the used scale in g:

SAMPLING PARAMETERS of the sampling device

START of sampling		**Date (yyyy-mm-dd)** **Time (hh:mm)**				
Flow rate, in $m^3 \cdot h^{-1}$	Setpoint		Read		Uncertainty	
Oven temperature, in °C	Setpoint		Read		Uncertainty	
Effective bubbling in each bubbler	Yes		No (Nr.)			
Piston volume, in ml	per stroke			Uncertainty		
END of sampling		**Date (yyyy-mm-dd)** **Time (hh:mm)**				
Flow rate, in $m^3 \cdot h^{-1}$	Setpoint		Read		Uncertainty	
Oven temperature, in °C	Setpoint		Read		Uncertainty	
Effective bubbling in each bubbler	Yes		No (Nr.)			
Piston strokes	Number					
Total volume at standard conditions, in m^3	Sampler			Uncertainty		
Observations during sampling						

ADDITIONAL INFORMATION			
Average flow rate in the stack/duct, in $m^3 \cdot h^{-1}$		Uncertainty	
Total volume in the stack/duct/gas container at standard conditions, in m^3		Uncertainty	
Penetration, in %		Uncertainty	
Efficiency of the collection medium, in %		Uncertainty	
Efficiency of the catalytic conversion oven, in %		Uncertainty	
Dilution factor		Uncertainty	
Homogenization technique			

Annex B
(informative)

Techniques for measurement of flow rate through ducts and stacks

B.1 General

In general, to measure a flow rate in a duct, a global method is preferred whenever possible. However, for the ducts or for most of the stacks given their large dimensions, a flow meter cannot be inserted.

Also to access the flow rate in the ducts or stacks, most of the time continuous measurement of the velocity of the fluid is carried out at a location or averaged over one or more diameters, using a Pitot tube or, a single or multi-point thermal anemometer. This type of reading can also be performed by integral measurements on a duct (using an acoustic flow meter).

A correction factor should be applied to this data to take into account the shape of the velocity profile in the measurement section. This correction factor is determined by comparing the flow rate values obtained using the single location technique with those obtained using the reference method for this document (see ISO 10780) or a gas tracing technique.

The reference method described in ISO 10780 is based on a reading of velocities at various locations of the measurement section according to a grid of points defined, then calculating the flow rate by integration. This method of exploring a velocity field is often only used occasionally within the context of the calibration, periodic verification or modification of the installation giving rise to a change in the flow rate.

B.2 Reminder on the ISO 10780:1994 reference method

The volumetric flow rate, q, flowing in a duct or stack is defined by the following Formula (B.1):

$$q = \int_{\text{section}} v \cdot \Delta A \qquad \text{(B.1)}$$

A method for determining q involves measuring the velocity at a finite number of points in a duct, where each point is chosen as the centre of an area element. The relationship that defines q is the following:

$$q = \sum_{i=1}^{N} v_i \cdot \Delta A_i \qquad \text{(B.2)}$$

The cross-sectional area of the stack or duct is divided into N elements. In practice, the N elements are chosen such as to have the same area. The approach described in ISO 10780 serves as the reference method in this document. The requirements for the absence of cyclonic flow specified in ISO 10780:1994, Annex D, are also included in the reference method.

The flow rate, q, is related to the air density, ρ, in the stack or duct. The density is defined as given by the ideal gas Formula applied to dry air, i.e.:

$$\rho = \frac{p}{R_a \cdot T} \tag{B.3}$$

At the end of testing and analysis, the volumetric flow rate under standard conditions, q_{std}, shall be calculated; the relationship between the standard volumetric flow rate and the actual volumetric flow rate, q, is the following:

$$\rho \cdot q = \rho_{std} \cdot q_{std} \tag{B.4}$$

The flow rate under standard conditions, q_{std}, can be expressed from Formula (B.4) in the Formula (B.5):

$$q_{std} = q \cdot \frac{T_{std}}{T} \cdot \frac{p}{p_{std}} \tag{B.5}$$

In practice, q is determined from measurement of the velocity at the transverse points specified in ISO 10780, with the value calculated from Formula (B.2). The temperature and pressure in the stack or ducts are measured in accordance with the requirements specified in ISO 10780.

B.3 Special considerations for use of ISO 10780 in sampling stacks and ducts of the nuclear industry

B.3.1 General

The reference method for determining air flow rate through a duct or stack of this document is an adaptation of ISO 10780, originally developed for flow rate determination in non-nuclear stacks and ducts. Therefore, it is necessary to take into consideration several differences between sampling from non-nuclear and nuclear stacks and ducts.

B.3.2 Pitot tubes

An S-type Pitot tube should be used to limit risks of dust plugging the ports of the Pitot tube when measurements are made in dusty environments. The accumulation of dust during determination of the flow velocity in the case of ducts and stacks of the nuclear industry is not usually of particular concern. Consequently, Prandtl-type Pitot static tubes (Type L in ISO 10780) should be used as the reference apparatus for sampling in accordance with the requirements specified in this document. In the rare cases where the accumulation of dust shall effectively be taken into consideration, an S-type Pitot tube may be suitable.

B.3.3 Mean molar mass of stack discharges

In the industrial applications for which ISO 10780 was developed, the gas being tested often contains combustion products or a high water vapour content due to drying operations. However, in the nuclear industry, the gas that flows in the stacks and ducts is ventilation air. In this industry, the mean molar mass, M, of the gas flowing in the stacks and ducts therefore need not be determined. However, the mean molar mass of the gas shall be determined if the water vapour content is likely to exceed 10 % in mass or gases other than air are likely to be present in the stack or duct and change the mean molar mass by more than 4 % from the value for dry air (28,96 g·mol^{-1}). In this case, the mean molar mass needs to be measured, as the result obtained is then used to calculate the gas constant, as given in Formula (B.6), i.e.:

$$R = \frac{R_u}{M} \tag{B.6}$$