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

Part 2: Determination of tritium and carbon-14 activities sampled by bubbling technique

National foreword

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ISO 20041-2

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

**Part 2:
Determination of tritium and
carbon-14 activities sampled by
bubbling technique**

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

*Partie 2: Détermination de l'activité du tritium et du carbone 14
prélevés par la technique du barbotage*

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Foreword

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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 within the framework of water and air discharge permits. 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^[1] 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 measurements, given greater possibilities of losses in the transport lines. However, in the gaseous effluents of nuclear facilities, tritium and carbon-14 are present in gas or vapour forms as multiple chemical compounds, which requires adapting the provisions of ISO 2889^[1]. Furthermore, ISO 2889:2023^[1] only deals with tritium and carbon-14 sample collection in Annexes H and K.

Therefore, the ISO 20041 series go further by addressing, in detail, the provisions specific to sampling methods, sample preparation and calculations for determining the tritium and carbon-14 activities. ISO 20041-1^[2] covers the sampling methods or techniques for tritium and carbon-14. This document covers activity analysis of tritium and carbon-14 sampled emissions by the bubbling technique.

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

Part 2:

Determination of tritium and carbon-14 activities sampled by bubbling technique

1 Scope

The objective of this document is to characterize the gaseous effluents tritium and carbon-14 generated by nuclear facilities during operation and decommissioning and occurring in the same chemical species as hydrogen and carbon, e. g. as water vapour (HTO), hydrogen gas (HT, TT), carbon dioxide ($^{14}\text{CO}_2$), carbon monoxide (^{14}CO), methane (CH_3T , $^{14}\text{CH}_4$). It concerns measurements on samples that are representative of a certain volume stream or volume of discharge during a given period of time and of the corresponding volume discharged. The result is therefore expressed in becquerels.

This document applies to samples that were obtained by sampling methods according to ISO 20041-1^[9] and describes

- analysis methods for the determination of tritium and carbon-14 activities by liquid scintillation counting, and
- calculation methods to determine the tritium activities discharged as tritiated water vapour (HTO) and tritium in other chemical compounds (non-HTO) as well as carbon-14 activities discharged as carbon dioxide ($^{14}\text{CO}_2$) and carbon-14 in other chemical compounds (non- $^{14}\text{CO}_2$).

This document does not apply to tritium and carbon-14 activity concentrations in the environmental air, e.g. in the vicinity of nuclear installations.

The accountability rules of the activities discharged necessary for the establishment of regulatory reports do not fall within the scope of this document and are the responsibility of the regulatory bodies.

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 19361, *Measurement of radioactivity — Determination of beta emitters activities — Test method using liquid scintillation counting*

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

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 2889:2023, 3.8]

3.2

aliquot

ideally representative part of the trapping solution or part of the carbonate precipitate used for the *LSC sample* (3.6)

3.3

bubbler

container for the trapping solution used

3.4

discharge

dispersion of *effluents* (3.5) into a receiving medium

3.5

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 20041-1:2022, 3.11]

3.6

LSC sample

mixture of an *aliquot* (3.2) of the trapping solution or carbonate precipitate and an appropriate scintillation cocktail for liquid scintillation counting (LSC)

3.7

non-HTO/non-¹⁴C₂

other chemical compounds which contain tritium (³H, T) and/or carbon-14 (¹⁴C) instead of hydrogen and/or carbon-12 or carbon-13, include elemental forms (e.g., HT, T₂), hydrocarbons (e.g., ¹⁴CH₄, ¹⁴C₂H₆), carbonates (e.g., ¹⁴CO₃²⁻), and other compounds that contain combinations of either H-3 or C-14 and at least one other atom.

Note 1 to entry: Methane (¹⁴CH₄) and tritiated hydrogen (HT, T₂) are usually the predominant non-HTO/non-¹⁴C₂ chemical forms in the gaseous effluent.

3.8

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 20041-1:2022, 3.22]

3.9

stack

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

[SOURCE: ISO 20041-1:2022, 3.29]

3.10

tritiated water

HTO

tritium (^3H , T) present in gaseous effluent in the form of water vapor in which one or two hydrogen atoms are substituted by tritium atoms

4 Symbols

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

Symbol	Definition	Unit
A	Activity of a radionuclide discharged with the gaseous effluents during the sampling period	Bq
A_m	Tritium or carbon-14 activity discharged as water vapour ($m = \text{HTO}$) or carbon dioxide ($m = ^{14}\text{CO}_2$) during the sampling period	Bq
A_{std}	Activity of a standard solution	Bq
A_m^*	Decision threshold of the tritium or carbon-14 activity discharged as water vapour ($m = \text{HTO}$) or carbon dioxide ($m = ^{14}\text{CO}_2$) during the sampling period	Bq
$A_m^\#$	Detection limit of the tritium or carbon-14 activity discharged as water vapour ($m = \text{HTO}$) or carbon dioxide ($m = ^{14}\text{CO}_2$) during the sampling period	Bq
$A_m^<$	Lower limit of the probabilistic coverage interval of the tritium or carbon-14 activity discharged as water vapour ($m = \text{HTO}$) or carbon dioxide ($m = ^{14}\text{CO}_2$) during the sampling period	Bq
$A_m^>$	Upper limit of the probabilistic coverage interval of the tritium or carbon-14 activity discharged as water vapour ($m = \text{HTO}$) or carbon dioxide ($m = ^{14}\text{CO}_2$) during the sampling period	Bq
A_n	Tritium or carbon-14 activity discharged in other compounds ($n = \text{non-HTO}$ or $n = \text{non-}^{14}\text{CO}_2$) during the sampling period	Bq
A_n^*	Decision threshold of the tritium or carbon-14 activity discharged in other compounds ($n = \text{non-HTO}$ or $n = \text{non-}^{14}\text{CO}_2$) during the sampling period	Bq
$A_n^\#$	Detection limit of the tritium or carbon-14 activity discharged in other compounds ($n = \text{non-HTO}$ or $n = \text{non-}^{14}\text{CO}_2$) during the sampling period	Bq
$A_n^<$	Lower limit of the probabilistic coverage interval of the tritium or carbon-14 activity discharged in other compounds ($n = \text{non-HTO}$ or $n = \text{non-}^{14}\text{CO}_2$) during the sampling period	Bq

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$A_n^>$	Upper limit of the probabilistic coverage interval of the tritium or carbon-14 activity discharged in other compounds ($n = \text{non-HTO}$ or $n = \text{non-}^{14}\text{CO}_2$) during the sampling period	Bq
$A_{n,a}$	Tritium or carbon-14 activity discharged in other compounds ($n = \text{non-HTO}$ or $n = \text{non-}^{14}\text{CO}_2$) during the sampling period if no activity concentration is found in the second bubbler b of bubbler unit 5	Bq
$A_{n,a}^*$	Decision threshold of the tritium or carbon-14 activity discharged in other compounds ($n = \text{non-HTO}$ or $n = \text{non-}^{14}\text{CO}_2$) during the sampling period if no activity concentration is found in the second bubbler b of bubbler unit 5	Bq
$A_{n,a}^\#$	Detection limit of the tritium or carbon-14 activity discharged in other compounds ($n = \text{non-HTO}$ or $n = \text{non-}^{14}\text{CO}_2$) during the sampling period if no activity concentration is found in the second bubbler b of bubbler unit 5	Bq
$c_{A,i}$	Activity concentration in the trapping solution of bubbler i (with $i = a, b, c, d$)	$\text{Bq}\cdot\text{l}^{-1}$
$c_{A,i}^*$	Decision threshold of the activity concentration in the bubbler i	$\text{Bq}\cdot\text{l}^{-1}$
$c_{A,i}^\#$	Detection limit of the activity concentration in the bubbler i	$\text{Bq}\cdot\text{l}^{-1}$
f_{A^*}	Correction factor for determining the decision threshold of the activity in presence of an HTO or $^{14}\text{CO}_2$ contribution during the sampling period	Bq
f_A	Correction factor for calculating the activity discharged	dimensionless
f_q	Quenching factor	dimensionless
k	Quantile of a standardized normal distribution, if: $k_{1-\alpha} = k_{1-\beta}$	dimensionless
k_p	Quantile of a standardized normal distribution for a probability p , e.g. $p = 1 - \alpha$, $p = 1 - \beta$ or $p = 1 - \gamma/2$	dimensionless
k_q	Quantile of a standardized normal distribution for a probability q , e.g. $q = 1 - \omega\cdot\gamma/2$	dimensionless
m_{aq}	Mass of the aliquot of precipitate used for measurement	kg
m_p	Total mass of precipitate after precipitation	kg
n	Numbers of measurement repetitions	dimensionless
P	Penetration	dimensionless
p	Probability for “success” in a binominal distribution	dimensionless
p_{std}	Standard pressure, equal to 101 325 Pa	Pa
Q	Quench parameter	dimensionless
q	Probability for “failure” in a binominal distribution	dimensionless
$\bar{q}$	Average volumetric flow rate	$\text{m}^3\cdot\text{h}^{-1}$

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$\bar{q}_{e,std}$	Average volumetric flow rate of the effluent air in a stack or duct at standard conditions	$m^3 \cdot h^{-1}$
$\bar{q}_{s,std}$	Average volumetric flow rate in the sampling system at standard conditions	$m^3 \cdot h^{-1}$
r	Count rate of the LSC sample without internal standard solution added	s^{-1}
r_I	Count rate of the LSC sample with internal standard solution added	s^{-1}
$r_{g,std}$	Gross count rate of the standard source	s^{-1}
$\bar{r}_{g,i}$	Average gross count rate of n measurements of an aliquot of the trapping solution i	s^{-1}
$\bar{r}_0$	Average background count rate of n measurements of the blank sample	s^{-1}
T_{std}	Standard temperature, equal to 298 K (25 °C)	K
t_C	Counting time of the LSC sample	s
t_I	Counting time of the LSC sample with internal standard solution added	s
t_{std}	Counting time of the standard source	s
t_0	Counting time of the blank sample	s
t_s	Sampling period	s
$u(A_m)$	Standard uncertainty of the tritium or carbon-14 activity discharged as water vapour ($m = HTO$) or carbon dioxide ($m = {}^{14}CO_2$) during the sampling period	Bq
$u(A_n)$	Standard uncertainty of the tritium or carbon-14 activity discharged in other compounds ($n = \text{non-}HTO$ or $n = \text{non-}{}^{14}CO_2$) during the sampling period	Bq
$u_{rel}(A_m)$	Relative standard uncertainty of the tritium or carbon-14 activity discharged as water vapour ($m = HTO$) or carbon dioxide ($m = {}^{14}CO_2$) during the sampling period	dimensionless
$u_{rel}(A_n)$	Relative standard uncertainty of the tritium or carbon-14 activity discharged in other compounds ($n = \text{non-}HTO$ or $n = \text{non-}{}^{14}CO_2$) during the sampling period	dimensionless
$u_{rel}(A_{std})$	Relative standard uncertainty of the activity of the standard source	dimensionless
$u_{rel}(c_{A,i})$	Relative standard uncertainty of the activity concentration in the bubbler i	dimensionless
$u_{rel}(f_A)$	Relative standard uncertainty of the correction factor for the activity discharged	dimensionless
$u_{rel}(m_{aq})$	Relative standard uncertainty of the mass of the aliquot of precipitate used for measurement	dimensionless

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$u_{\text{rel}}(m_p)$	Relative standard uncertainty of the total mass of precipitate	dimensionless
$u_{\text{rel}}(P)$	Relative standard uncertainty of penetration	dimensionless
$u_{\text{rel}}(\bar{q}_{\text{e,std}})$	Relative standard uncertainty of the average volumetric flow rate in a stack or duct at standard conditions	dimensionless
$u_{\text{rel}}(\bar{q}_{\text{s,std}})$	Relative standard uncertainty of the average volumetric flow rate in the sampling system at standard conditions	dimensionless
$u_{\text{rel}}(V_{\text{aq}})$	Relative standard uncertainty of the volume of the aliquot of the trapping solution used for measurement	dimensionless
$u_{\text{rel}}(V_i)$	Relative standard uncertainty of the volume of the trapping solution in the bubbler i	dimensionless
$u_{\text{rel}}(w_i)$	Relative standard uncertainty of the calibration factor for the bubbler i	dimensionless
$u_{\text{rel}}(y_{\text{qa}})$	Relative standard uncertainty of the fraction of the quaternary amine solution used for measurement	dimensionless
$u_{\text{rel}}(y_V)$	Relative standard uncertainty of the fraction of the trapping solution used for sample preparation	dimensionless
$u_{\text{rel}}(\varepsilon_{\text{I}})$	Relative standard uncertainty of the detection efficiency for the internal standard method	dimensionless
$u_{\text{rel}}(\varepsilon_{\text{cv}})$	Relative standard uncertainty of efficiency of the catalytic conversion oven	dimensionless
$u_{\text{rel}}(\varepsilon_{\text{p}})$	Relative standard uncertainty of the efficiency of the sample preparation	dimensionless
$u_{\text{rel}}(\varepsilon_{\text{q}})$	Relative standard uncertainty of the detection efficiency at a quenching parameter Q	dimensionless
$u_{\text{rel}}(\varepsilon_{\text{t}})$	Relative standard uncertainty of the efficiency of the trapping solution	dimensionless
V_{aq}	Volume of the aliquot of the trapping solution or distillate used for measurement	l
V_i	Volume of the trapping solution in the bubbler i at the end of sampling	l
w_i	Calibration factor for the bubbler i , depending on the preparation method	l ⁻¹
y_{qa}	Fraction of the quaternary amine solution used for measurement, less or equal 1	dimensionless
y_V	Fraction of the trapping solution used for sample preparation, less or equal 1	dimensionless
z	Auxiliary quantity used for determining the decision threshold of the activity discharged of non-HTO or non- ¹⁴ CO ₂ in presence of an HTO or ¹⁴ CO ₂ contribution	dimensionless
α	Probability of the false positive decision	dimensionless
β	Probability of the false negative decision	dimensionless

ε	Detection efficiency of an unquenched sample or standard	dimensionless
ε_I	Detection efficiency for the internal standard method	dimensionless
ε_{cv}	Efficiency of the catalytic conversion oven	dimensionless
ε_p	Efficiency of the sample preparation	dimensionless
ε_q	Detection efficiency at a quench parameter Q	dimensionless
ε_t	Efficiency of the trapping solution used for sampling	dimensionless
$\Phi(t)$	Distribution function of the standardized normal distribution	dimensionless
ω	Auxiliary quantity used for the distribution function	dimensionless
$1 - \gamma$	Probability for the coverage interval of the measurand	dimensionless

5 Principle

The following procedure describes the sample preparation, the measurement by liquid scintillation counting and the evaluation of tritium or carbon-14 activities in representative samples obtained from sampling of the effluent air in nuclear installations by bubbling technique according to ISO 20041-1[9].

Usually, samples for analysing are taken from each bubbler of the sampling device (see [Figure 2](#)). The bubbler unit before the catalytic conversion oven is used for the determination of tritium as water vapour (HTO) or carbon-14 as carbon dioxide ($^{14}\text{CO}_2$) and the bubbler unit after the catalytic conversion oven is used for the determination of tritium or carbon-14 in other chemical compounds (non-HTO or non- $^{14}\text{CO}_2$). In the case that total amount of tritium (HTO and non-HTO) and total amount of carbon-14 ($^{14}\text{CO}_2$ and non- $^{14}\text{CO}_2$) are subject to regulatory requirements, the sampling device is configured only with a bubbler unit after a catalytic conversion oven.

NOTE 1 When sampling carbon-14 with sodium hydroxide trapping solution, tritium can also be collected in the trapping solution. Therefore, it is necessary to pay attention to the choice of a suitable sample preparation (see [8.4](#)) and measurement (see [8.6](#)).

NOTE 2 The temperature of the catalytic conversion oven is a key parameter to the cracking phenomena for measuring tritium. If the temperature is not too high (e.g. around 170 °C), only HT is converted to HTO and not other chemical forms such as $\text{C}_x\text{H}_y\text{T}_z$ (T being tritium) which will pass through without being trapped (or subsequently measured). Hence, a higher catalytic conversion oven temperature (e.g. around 450 °C) is necessary to convert any $\text{C}_x\text{H}_y\text{T}_z$ into HTO (and CO_2).

Tritium, ^3H , and carbon-14, ^{14}C , are pure beta emitters. The maximum beta energy of tritium is equal to 18,591 keV and its radioactive half-life is equal to 12,312 years[2]. Carbon-14 has a maximum beta energy equal to 156,476 keV and radioactive half-life equal to 5 700 years[2]. Both tritium and carbon-14 can be measured directly from the samples by liquid scintillation counting.

If other pure beta emitters or beta/gamma radionuclides, such as sulphur-35, ruthenium-106 and iodine-129, are present in the samples, these should be separated using a suitable method to prevent interferences during liquid scintillation counting (see [8.6.4.2](#)). Examples of sample preparation methods are described in the [Annexes A](#) to [D](#). The choice of the analytical procedure depends on the aim of the measurement and the sample characteristics.

The general principles of measuring beta emitters by liquid scintillation counting are given in ISO 19361 and in Reference [3].

For liquid scintillation counting, an aliquot of the sample – either with or without previous sample preparation – is mixed with a suitable scintillating cocktail in a counting vial to obtain a homogeneous medium or considered as such.