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Ventilation systems for nuclear facilities — In-situ efficiency test methods for iodine traps with solid sorbent —

Part 2: Radioactive CH_3I method

Systèmes de ventilation pour les installations nucléaires — Méthodes d'essai in situ de l'efficacité des pièges à iode à sorbant solide —

Partie 2: Méthode au CH_3I radioactif

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Foreword

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A list of all parts in the ISO 16659 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

In nuclear facilities, iodine traps are usually present within ventilation systems to limit radioactive gaseous iodine releases into the environment or to prevent radioactive iodine transfer to protected areas (such as control room for example) in accordance with the principles of ISO 17873[1] and ISO 26802[2] or other relevant documents (see References [3], [4], [5], [6] and [7]. The ability of these devices to trap gaseous radioactive iodine is primordial, particularly when they support the safety demonstration. The IAEA in paragraph 4,127 of Specific Safety Guide No. 53 (SSG-53)[8] recommends demonstrating the efficiency of the adsorption material in iodine traps and periodically testing iodine traps in-situ.

ISO 16659 series provides different in-situ test methods to determine the efficiency of radioactive iodine traps in ventilation systems of nuclear facilities. This series deals with iodine traps with solid sorbent, mainly impregnated activated carbon, the most usual solid sorbent used in ventilation systems of nuclear facilities, as well as other sorbent submitted to special conditions (e.g. silver loaded zeolites in case of high temperature).

ISO 16659–1 is the general part of this series and describes common general provisions applicable to all methods.

This document is about an in-situ testing method for measuring the efficiency of iodine traps using radioactive methyl iodide ($CH_3{}^{131}I$) as a tracer.

Ventilation systems for nuclear facilities — In-situ efficiency test methods for iodine traps with solid sorbent —

Part 2:
Radioactive CH_3I method

1 Scope

This document specifies a test method using radioactive methyl iodide ($CH_3{}^{131}I$) as a tracer to determine the in-situ decontamination factor of an iodine trap. An in-situ test allows to reach the global efficiency of the trap characterized by the sorbent efficiency but also by the implementation of the trap within the ventilation duct) while the intrinsic efficiency of a charcoal is characterized in a laboratory by ISO 18417[9] (or other national standards such as ASTM D3803[10]).

This document provides general and common requirements for this method to assess the efficiency of an iodine trap, but also, the tools requirements, accuracy and the provisions needed to ensure safety of the workers, public and the environment during the test.

This reproducible method can support nuclear facility operators as a reference method to compare the decontamination factor evaluated by this method to reference values (e.g. safety criteria, national legislation, etc.).

Because of the use of a radioactive tracer, some precautions should be applied.

Firstly, this method is usually used for ventilation systems with monitoring of gaseous iodine releases in environment in accordance with the national regulations.

Secondly, this method is not used to determine the decontamination factor of iodine traps used in ventilation systems with air release in rooms with potential presence of workers (e.g. control room). For those rooms, a non-radioactive method is preferred.

This document can apply to installations with low inventory of radioiodine equipped with iodine traps (e.g. small laboratories). In this case, some provisions can be adapted but always in accordance with the national regulations.

Finally, this document mainly deals with iodine traps using impregnated activated carbon. However, this method can be used with some adaptations to other solid sorbent as inorganic sorbent (e.g. zeolite – aluminium and silica base usually doped with silver nitrate – or impregnated catalytic supports[11][12]).

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 16659-1:2022, *Ventilation systems for nuclear facilities — In-situ efficiency test methods for iodine traps with solid sorbent — Part 1: General requirements*

ISO 20042, *Measurement of radioactivity — Gamma-ray emitting radionuclides — Generic test method using gamma-ray spectrometry*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in in ISO 16659-1 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/

4 Method

4.1 General

This method employing radioactive iodine encompasses all physical phenomena associated with iodine retention during the test (physical adsorption, chemical adsorption, isotopic exchange and short-term desorption). This method is relevant to determine a decontamination factor of a high efficiency iodine trap (a decontamination factor superior to several thousand is typically achievable for a new sorbent).

Concerning the methyl iodide form (CH_3I) of radioactive iodine used as a tracer in this method, it is important to notice that it is generally not the most present gaseous iodine form in nuclear facilities, in particular, in reactors where the molecular form (I_2) is more common. But the interest of using such a tracer is that methyl iodide is the most penetrating form for an iodine trap.

So, because all the physical phenomena are tested and considering the use of methyl iodide as a tracer gives conservative decontamination factors, this method is relevant to support nuclear facility operators to compare the decontamination factor to CH_3I but also I_2 reference values (e.g. safety criteria). This method is widely encountered in PWRs even if different iodine species are present.

Moreover, due to its chemical properties compared to I_2, this CH_3I method is easier to implement in facilities (less contamination of the test equipment, less damage to equipment (corrosion), easier transport of test equipment, use of less fragile materials (stainless steel instead of glass)) and leads to less worker contamination.

Concerning the test conditions (ambient conditions) which can differ from those encountered during incidents or accidents (high temperature, high relative humidity, etc.), it is important to notice that the decontamination factor determined with this method may not be directly transposed as a reference value for the estimation of radiological consequences for population. Nevertheless, this document gives a reproducible method to precisely evaluate the decontamination factor of an iodine trap and thus to reveal its full ability to trap gaseous iodine when high decontamination factors are reached or its possible degradation over time due to ageing phenomena.

Finally, this method does not lead to a saturation of the iodine traps by the radioactive iodine tracer (the quantity injected is extremely low compared to the trapping capacity) and so is compatible with the safety of the nuclear facilities.

4.2 Principle of the method

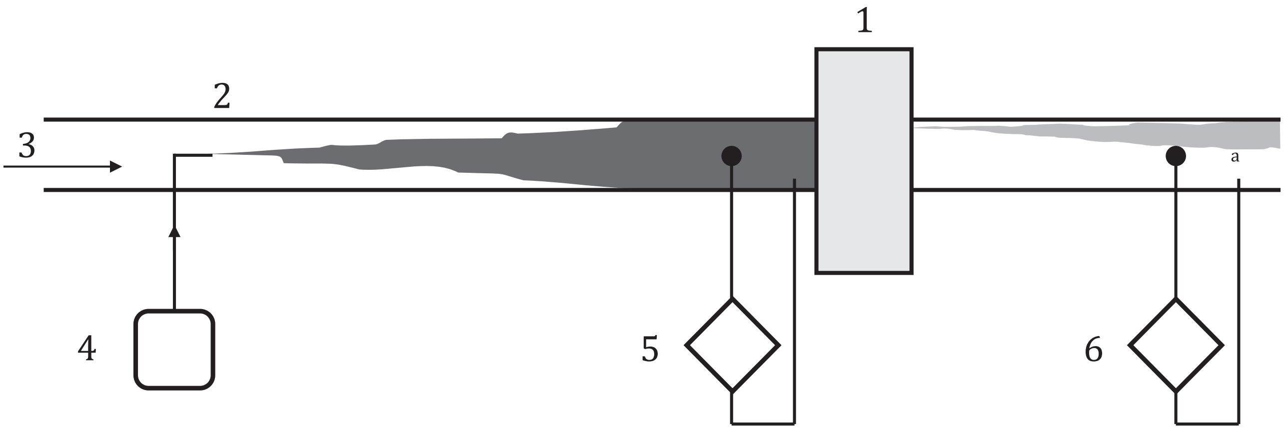
The principle of the method (see Figure 1) consists in:

a) the injection of gaseous radioactive iodine-131 (CH_3I form) into the ventilation duct;

b) the measurement of the quantities of CH_3I present upstream and downstream of the iodine trap (sampling in ventilation duct, trapping radioactive iodine in charcoal cartridges and gamma spectrometry counting of the charcoal cartridges);

c) the calculation of the decontamination factor and the comparison to a reference value.

NOTE This document, consistent with studies and research on iodine traps, is based on the use of iodine-131. The use of other iodine radioisotopes (e.g. iodine-123 or iodine-129) is, in principle, possible, as no scientific barriers are currently identified, provided that consolidated data are available, particularly regarding trapping phenomena (e.g. the impact of differences between β+/β- emissions). Certain adaptations may be required, for example due to the short half-life of iodine-123 (approximately 13,2 h), or with respect to workers safety.



Key

1	iodine trap to be tested
2	ventilation duct
3	air flow
4	injection of $\mathrm{CH_3}^{131}\mathrm{I}$
5	upstream sampling line of $\mathrm{CH_3}^{131}\mathrm{I}$ (with charcoal cartridges)
6	downstream sampling line of $\mathrm{CH_3}^{131}\mathrm{I}$ (with charcoal cartridges)
[a]	The light grey form downstream the trap represents an example of a non-homogeneous tracer concentration in the duct in case of a default of the trap and reveals the importance of the representativeness of the downstream sampling in addition to the one upstream.

Figure 1 — General principle for the $\mathrm{CH_3}^{131}\mathrm{I}$ test method

4.3 Parameters affecting iodine removal

4.3.1 General

The performance of iodine traps depends on many parameters which can be classified in two categories: parameters related to the sorbent and parameters related to the operating conditions. In addition to the ones mentioned in ISO 16659–1:2022, 4.4, the following parameters shall be considered.

4.3.2 Effect related to the sorbent

The production method of activated charcoal (e.g. carbonization, activation and further modification for example grain size selection) as well as the raw material nature have a key role in iodine removal because they determine physical properties of activated charcoal (e.g. specific surface area, pore size distribution, pore volume) as well as its chemical characteristics (e.g. surface functional groups: type, molecules and quantity). And these physical and chemical parameters determine the iodine adsorption performances (e.g. adsorption capacities and trapping stability).

To improve its iodine trapping efficiency, activated charcoal is generally impregnated in the nuclear context with a combination of potassium iodide (KI, generally 1 %) and triethylenediamine (TEDA, generally 5 %). The typical quantity of iodine collected in a trap is about 1 milligram of iodine per gram of activated charcoal.

Even if there are variabilities of the sorbent characteristics, this method is fully relevant to determine a decontamination factor of an iodine trap because it considers all the trapping phenomena.

4.3.3 Effects related to the operating conditions

4.3.3.1 Overview

In normal operation, iodine traps can be operated in different ways (bypassed, continuous service, with reduced air flow rate, etc.) with variable air conditions (relative humidity and temperature), which are controlled or not. In accident conditions (e.g. severe accident for reactor), these conditions may be very different. The efficiency test conditions may be therefore different from those for which iodine traps are evaluated in the safety demonstration, so it is important to measure these parameters to evaluate their impact on the decontamination factor.

Numerous studies have been conducted to determine the performance of impregnated activated charcoal in different conditions to trap methyl iodide. The most prominent factors are the relative humidity, the temperature, the frontal speed (i.e. face velocity) (and contact time) as well as the ageing phenomena.

This document gives recommendations on these influencing parameters to ensure reproducibility of the test in controlled conditions. It is important to notice that to compare test results to reference values, the test conditions shall be as close as possible to accident conditions.

Other parameters not developed below are considered second order in the influence on the decontamination factor.

4.3.3.2 Relative humidity and temperature

Relative humidity in the air has a negative effect on the efficiency of iodine traps to collect methyl iodide because of the reduction of available adsorption sites of activated charcoal. The water molecules on the surface of the activated charcoal create clusters bound in a network by hydrogen bonds and then fill the porous structure of the activated charcoal, leading to a decrease of available sites for iodine species. So, there is a competition between iodine species and H_2O for adsorption in the activated charcoal[13]. A simple way to reduce the relative humidity in the air and its effects on the efficiency of iodine traps is to increase the air temperature. However, the higher the air temperature is, the worse the physical adsorption is (because of high Brownian agitation). On the other side, the higher the air temperature is, the better reactions involved in chemisorption (with TEDA–impregnant) or isotopic exchange (with KI–impregnant) are.

As the efficiency is a combination of all these antagonistic phenomena, the higher the air temperature is, the better the global efficiency in terms of decontamination factor of an iodine trap is (even if the adsorption of gaseous iodine is lower, the global gain on the efficiency is better due to the reduction of the relative humidity impact[14][15][16]). This behaviour of activated charcoal depending on the relative humidity and temperature is already established with the previous multiparametric work of Shiomi[8].

In a practical way, electric heaters are commonly encountered upstream of iodine traps in nuclear facilities to improve the efficiency of iodine traps. The service temperature of heaters is generally around 70 °C, which is sufficient to reduce relative humidity below than 40 %.

NOTE 1 Below a relative humidity of 40 %, an activated charcoal adsorbs fewer water molecules (activated charcoal is hydrophobic). Above 40 %, the water adsorption increases significantly (cf. adsorption isotherms of water for activated charcoal[18]).

NOTE 2 The service temperature can be higher but always lower than a range of 100 °C to 120 °C to avoid an increase of desorption phenomenon or degradation of the TEDA–impregnant which can lead to release of iodine previously collected (note that ignition of a new charcoal is expected for higher temperatures).

As this method enables nuclear facility operators to compare a decontamination factor to reference values (e.g. safety criteria, national regulations), it is important that the test is conducted with operating heaters, when present, to replicate conditions of use as closely as possible. These are the conditions under which iodine traps are evaluated in the safety demonstration.

Moreover, with operating heaters, hygrometric and thermal conditions (service temperature of heaters and relative humidity under 40 %) are controlled which is favourable to the reproducibility of the test.

Beyond the temperature and the relative humidity of the air upstream the iodine trap, as stated in the ISO 16659–1, it is important to reach the hygrometric and thermal equilibrium of the iodine trap to ensure a reliable comparison from one test to another. This equilibrium is generally reached after a quite long duration following the commissioning of the heaters (16 h are mentioned in ISO 16659–1). This duration can be shortened if the relative humidity of the air during the test is quite low (e.g. dry air) or the volume of activated charcoal in the iodine trap is quite small or when operating constraints exist (e.g. impossible commissioning of heaters for a long duration before test). The hygrometric and thermal equilibrium can then be considered reached, for example, if the air temperature downstream the trap is far enough from the dew temperature or if the temperature and the relative humidity of air upstream and downstream the iodine trap are close (e.g. $\Delta T < 5$ °C and $\Delta RH < 5$ % measured by national standards as given in References [19] or [20]).

For tests conducted without heaters, this method remains valid and provides the actual decontamination factor of the iodine trap under the test conditions. However, the test conditions may differ significantly from those encountered during accident scenarios. Second, the test conditions can be different from one test to another and therefore the establishment of trend curves is more delicate.

Certain correlations allow calculation of the decontamination factor of an iodine trap at a desired specified temperature or relative humidity, based on results obtained under the test conditions. These empirical correlations shall be applied with particular caution regarding their domain of validity, especially with respect to the nature of the activated charcoal or its impregnation (see Annex C).

Finally, due to the importance of relative humidity and temperature on the efficiency of iodine traps, these parameters shall be measured and registered in the test report (see Annex E).

4.3.3.3 Contact time between air and the sorbent

As stated in ISO 16659–1, the contact time (coupling of air velocity and bed depth) is a major parameter because the adsorption is not an instantaneous phenomenon. If the model of implemented iodine traps is always the same, the air flow rate becomes the key parameter for the contact time and shall be measured and registered in the test report (see Annex E).

NOTE Contact time for industrial iodine trap is usually comprised between 0,2 s and 0,4 s.

Also, to ensure the determination of a decontamination factor representing the one in accident (representativeness of the test), this current method requires to carry out the test at air flow rate as close as possible to the one in accident (and thus the contact time).

If test is carried out at a reduced air flow rate (which increases the contact time and thus the efficiency), the determination of the decontamination factor at nominal air flow rate should use appropriate correlations.

To ensure reproducibility of the tests, the air flow rate should be equivalent from one test to another (about 10 % to 15 % considering uncertainties). If periodic efficiency tests are carried out with variable air flow rates for one iodine trap, the comparison of one test to another and therefore the establishment of trend curves is more complex.

4.3.3.4 Ageing phenomena

The iodine trap has a relatively short service life when expecting a high decontamination factor. When a radioactive tracer is used, attention shall be paid if a high quantity of iodine is injected to test an iodine trap that has been in service for more than four to six years. To calculate the iodine-131 activity to be injected (see 8.1.2), the determination of the expected decontamination factor of the iodine trap to be tested shall consider ageing phenomena. The operating experience feedback can provide elements for estimating degradation of the decontamination factor. It is also possible to use correlations as the one developed by Taylor and Taylor[21].

4.4 Other specificities of the method

4.4.1 Use of the $CH_3{}^{131}I$ tracer

4.4.1.1 Advantages compared to the I_2 tracer

The radioactive gaseous iodine species involved in reactor severe accidents are commonly I_2 and in a smaller proportion CH_3I (other gaseous iodide molecules exist but they are not considered because of their negligible quantities). A method with CH_3I rather than I_2 is however more relevant because of the quite low deposition/adsorption properties of the methyl iodide compared to the molecular form. Indeed, this document takes advantage of this characteristic with:

- avoiding pre-injection of non-radioactive iodine into the ventilation ducts which artificially ages ventilation systems because I_2 is corrosive (this pre-injection is usually done to discriminate the iodine collected in the iodine trap, so the decontamination factor, from the one collected by equipment of the ventilation system [ducts, valves, etc.]);
- limiting the iodine-131 activity injected, since lower decontamination factors are expected with CH_3I than I_2;
- limiting radioactive iodine contamination of test equipment, thereby reducing the risk of worker contamination and facilitating the evacuation of equipment from the controlled area of the facility.

4.4.1.2 Purity of the gaseous $CH_3{}^{131}I$

Gaseous $CH_3{}^{131}I$ is usually generated by the chemical reaction of dimethyl sulfate and radioactive sodium iodide[22][23] but can also be generated by isotopic exchange between stable methyl iodide and radioactive sodium iodide (see 8.1.2).

Whatever the reaction, if parameters are not well-controlled, the generation of $CH_3{}^{131}I$ can release other iodide species (other organic forms, radioactive or not) with different physical and chemical properties (which can lead to a bias in the determination of the decontamination factor) or release undesired reagents (e.g. dimethyl sulfate for workers considerations).

Unfortunately, there is no real time method to verify the composition of iodide species released particularly the $CH_3{}^{131}I$ form. So, it is therefore essential to respect the $CH_3{}^{131}I$ generation procedure in order to perform the reaction correctly. The procedure shall include verification of parameters such as temperature or bubbling and mixing conditions, and the purity of the reagents used, for example dimethyl sulfate. Respecting these procedures shall contribute to the reproducibility of efficiency test.

NOTE 1 Feedback and know-how from nuclear operators are key elements of proven procedures that contribute to reproducible methods.

NOTE 2 For development of new procedures or optimization of existing ones, in a laboratory phase and before introducing radioactive materials, such methods as the infrared spectroscopy (e.g. FTIR: Fourier transform infrared spectroscopy) or the gas chromatography can be used to determine optimal values of parameters for these reactions of generation of gaseous $CH3^{131}I$.

4.4.1.3 Desorption of the $CH_3{}^{131}I$ in an iodine trap

Due to the reversible trapping phenomena of gaseous iodine on charcoal (physical adsorption and isotopic exchange, but not chemisorption), desorption can occur. The typical time of the desorption peak appears several hours after injection[24][25]. This timing depends on parameters related to the sorbent, such as the nature of impregnant, the specific surface and the thickness of the charcoal bed, as well as on operating conditions, including relative humidity and contact time.

To take into account the desorption of methyl iodide, the current method requires that the duration of the samplings, in particular the downstream sampling, be longer than the injection duration. Nevertheless, a long duration of sampling may not be compatible with industrial constraints. In general, the in-situ duration of sampling is short, for example about one hour, which is a usual industrial practice. It is important to