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Introduction

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Date: xx/xx/20xx	Document: ISO/DIS xxxx
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1	2	(3)	4	5	(6)	7
MB	Clause No./ Subclause No./Annex (e.g. 3.1)	Paragraph/ Figure/Table/Note	Type of comment	Comment (justification for change) by the MB	Proposed change by the MB	Secretariat observations on each comment submitted
	3.1	Definition 1	ed	Definition is ambiguous and needs clarifying.	Amend to read '...so that the mains connector to which no connection...'	
	6.4	Paragraph 2	te	The use of the UV photometer as an alternative cannot be supported as serious problems have been encountered in its use in the UK.	Delete reference to UV photometer.	



DRAFT International Standard

ISO/DIS 16659-2

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

Part 2: Radioactive CH₃I method

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Contents

Page

Foreword	v
Introduction	vi
1 Scope	1
2 Normative references	1
3 Terms and definitions	2
4 Method	2
4.1 General	2
4.2 Principle of the method	2
4.3 Parameter affecting iodine removal	3
4.3.1 General	3
4.3.2 Effect related to the sorbent	3
4.3.3 Effects related to the operating conditions	4
4.4 Other specificities of the method	6
4.4.1 Use of the CH ₃ ¹³¹ I tracer	6
4.4.2 Toxicity	7
4.4.3 Sampling	7
5 Test equipment	8
5.1 Generation and injection devices	8
5.2 Sampling device	8
5.2.1 General	8
5.2.2 Sampling nozzle	10
5.2.3 HEPA filter	10
5.2.4 Activated charcoal cartridges	10
5.2.5 Air flow rate measurement	11
5.2.6 Release in the ventilation duct	11
5.3 Measurement device	12
5.3.1 Activity (γ-spectrometer)	12
5.3.2 Relative humidity (hygrometer) & temperature (thermometer)	12
5.3.3 Ventilation, injection and sampling flow rate	12
6 Safety of workers, members of the public and the environment	12
6.1 Main workers safety provisions	12
6.1.1 Radioactive considerations	12
6.1.2 Biological considerations	13
6.2 Main provisions for ensuring safety of the population and the environment	13
6.3 Management of radioactive sources and radioactive waste	13
7 Facility arrangements	13
8 Mode of performing the test	14
8.1 Test preparation	14
8.1.1 Calibrations/Periodic verifications	14
8.1.2 Na ¹³¹ I source preparation and calculation of the minimal activity	14
8.1.3 Facility arrangements	16
8.1.4 Sampling lines	16
8.1.5 Connections	16
8.2 Test execution	16
8.3 Test end	16
8.3.1 Disassembly	16
8.3.2 Radiation controls	16
8.3.3 Samples counting	17
9 Establishing the result	18
9.1 Expressing the result	18
9.2 Accuracy of the result	19

9.2.1	General formula of uncertainties.....	19
9.2.2	Influent parameters relative to the ventilation system & test conditions	19
9.2.3	Influent parameters relative to the equipment.....	20
9.2.4	Influent parameters relative to the method.....	21
9.3	Evaluation and test report.....	22
Annex A	(normative) Test method “In-situ generation method”	24
Annex B	(normative) Test method “Laboratory generation method”	28
Annex C	(informative) Examples of correlations	33
Annex D	(informative) Examples of activity of ^{131}I injected to test an iodine trap.....	35
Annex E	(informative) Example of typical test report for $\text{CH}_3^{131}\text{I}$ method.....	36
Bibliography		38

Foreword

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

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 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, to reduce iodine concentration in the air of facilities in case of air recycling, or to prevent radioactive iodine transfer to protected areas (such as control room for example) in accordance with the principles of ISO 17873^[3] and ISO 26802^[5] or other relevant documents (see References ^[9], ^[10], ^[11], ^[12] and ^[15]). 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)^[21] recommends testing periodically the filter system and demonstrating the efficiency of the adsorption material in iodine filters. There is no international standard in this regard.

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. high temperature for silver loaded zeolites).

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

This document is relative to an in-situ testing method for measuring the efficiency of iodine traps using radioactive methyl iodine ($\text{CH}_3^{131}\text{I}$) as a tracer.

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

Part 2: Radioactive CH₃I method

1 Scope

The present test method uses radioactive methyl iodide (CH₃¹³¹I) 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^[4] (or other national standards as ASTM D3803^[6]).

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 cautions apply.

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

Second, 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). 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^{[24],[25]}).

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 10648 (all parts), — *Containment enclosures*

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

ISO 11933 (all parts), — *Components for containment enclosures*

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 using radioactive iodine covers all the physical phenomena involving for iodine retention during the test duration (physical adsorption, chemical adsorption, isotopic exchange, and short-term desorption). This method is so 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 it is generally not the most present gaseous iodine form in nuclear facilities, in particular in reactors where the major form is the molecular one (I_2). But the interest of using such a tracer is that methyl iodide is the most penetrating form for an iodine trap. This method is so relevant to support nuclear facility operators to compare to CH_3I but also I_2 reference values. This method is widely encountered in PWR even if different iodide 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 so leads to less worker contamination.

Concerning the test conditions (ambient conditions) which are generally different 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:

- 1) The injection of gaseous radioactive iodine-131 (CH_3I form) into the ventilation duct.
- 2) The measurement of the quantities of CH_3I present upstream and downstream of the iodine trap (sampling in ventilation duct, trapping radioactive iodine in test traps, gamma spectrometry counting of the test traps).

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

NOTE This document, as studies and research on iodine traps, is based on the iodine-131. The use of another iodine radioisotope (e.g. ^{123}I or ^{129}I) with this document is a priori possible (no known scientific barrier) with consolidated elements, particularly on trapping phenomena (e.g. impact of the difference between β^+/β^-). Some adaptations may occur, particularly for safety of workers.

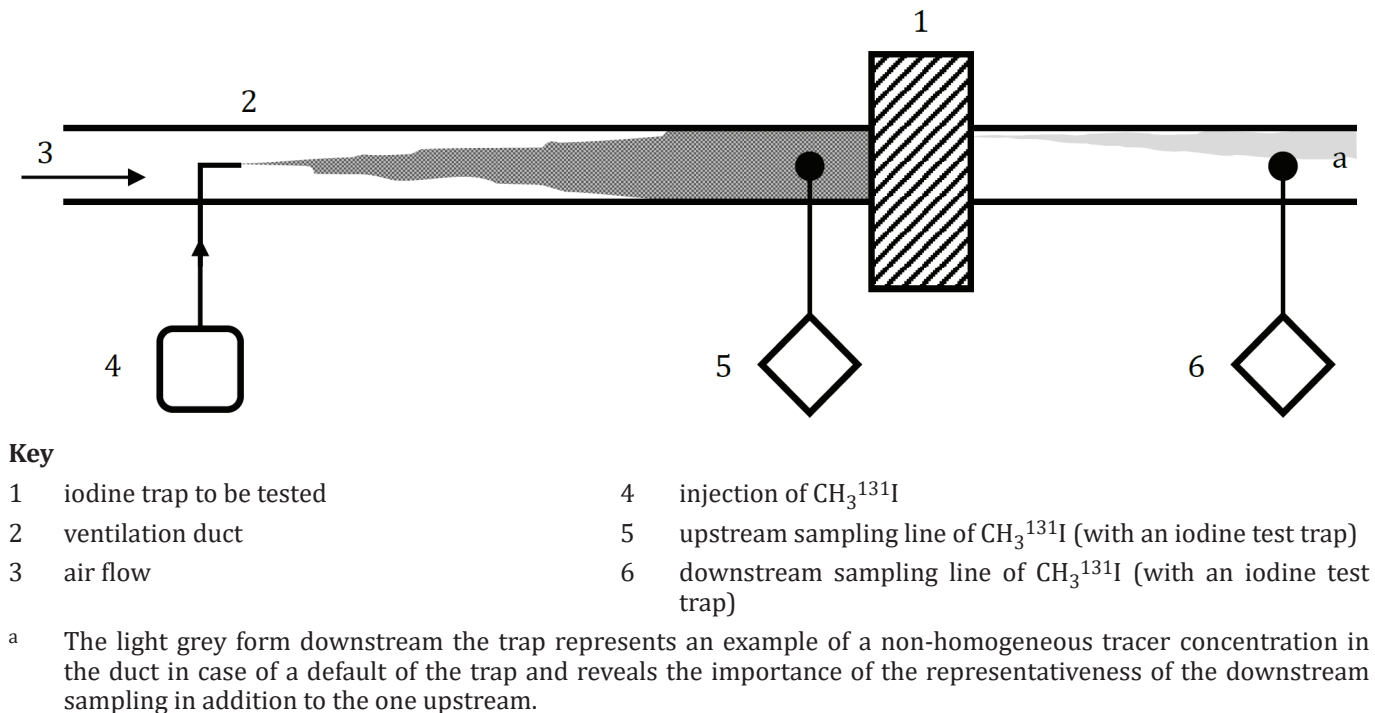


Figure 1 — General principle for the $\text{CH}_3^{131}\text{I}$ test method

4.3 Parameter 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 4.4 of ISO 16659-1, the following parameters have to be considered.

4.3.2 Effect related to the sorbent

The production method of activated charcoal (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 textural properties of activated charcoal (specific surface area, pores size distribution, pores volume) as well as its chemical characteristics (surface functional groups: type, molecules and quantity). And these physical and chemical parameters determine the iodine adsorption performances (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 Introduction

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 valorized 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 (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 & 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 filled 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.^[26] 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^{[18],[20],[28]}). This behavior of activated charcoal depending on the relative humidity and temperature is although established with the previous multiparametric work of Shiomi.^[16]

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

NOTE 1 Below a relative humidity of 40 %, an activated charcoal adsorbs few water molecules (activated charcoal is hydrophobic). Above 40 %, the water adsorption increases significantly (cf. adsorption isotherms of water for activated charcoal^[27]).

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

Considering this method supports nuclear facility operators to compare a result to reference values (e.g. safety criteria, national legislation, etc.), it is important to achieve the test with operating heaters when present to be as close as possible to conditions of use (conditions for which iodine traps are valorized in the safety demonstration).

Moreover, the achievement of the test with operating heaters leads to a characterized domain of temperature (service temperature of heaters) and relative humidity (under 40 %) which is favorable 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 hours are mentioned in ISO 16659-1:2022). This duration could 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 could 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. $\Delta T < 5\text{ }^{\circ}\text{C}$ and $\Delta RH < 5\text{ }\%$ measured by national standards as given in References [7] or [8]).

For tests carried out without heaters, this method is still valid and gives the real decontamination factor of the iodine trap at the test conditions. Nevertheless, first, the test conditions can be quite different from those encountered in accident conditions. Second, the test conditions can be different from one test to another and therefore the establishment of trend curves is more delicate.

Some correlations allow calculating a decontamination factor of an iodine trap at desired temperature or relative humidity from the result given at the test conditions. These empiric correlations must be used with extreme care regarding the domain validity (especially 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 report.

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, so the air flow rate becomes the key parameter for the contact time and shall be measured and registered in the report.

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 the air flow rate the closest 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 correlations given by R&D.

To ensure reproducibility of the tests, the air flow rate should be equivalent from one test to another (about 10 %–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 delicate.

4.3.3.4 Ageing phenomena

In addition to 4.4.5 of the ISO 16559-1:2022, regarding the quite short life of an iodine trap with high decontamination factor, the use of a radioactive tracer in this document involves attention when a high quantity of iodine is injected to test an old iodine trap (more than 4 years to 6 years). Thus, 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 should consider ageing phenomena. The operating experience feedback can give elements. Is it also possible to use correlations as the one developed by Taylor and Taylor.^[13]

4.4 Other specificities of the method

4.4.1 Use of the $\text{CH}_3^{131}\text{I}$ tracer

4.4.1.1 Advantages compared to the I_2 tracer

The radioactive gaseous iodine species involved in reactor severe accidents are generally mostly 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.]);
- limitation of iodine-131 activity injected since lower decontamination factor are expected with CH_3I than I_2 ;
- limitation of radioactive iodine contamination of test equipment limiting the risk of workers contamination and facilitating the evacuation of equipment from the nuclear controlled area.

4.4.1.2 Purity of the gaseous $\text{CH}_3^{131}\text{I}$

Gaseous $\text{CH}_3^{131}\text{I}$ is usually generated by the chemical reaction of dimethyl sulphate and radioactive sodium iodide [14], [23] but could 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 $\text{CH}_3^{131}\text{I}$ could release other iodide species (other organic forms, radioactive or not) with different physical and chemical properties (which could lead to a bias in the determination of the decontamination factor) or release undesired reagents (e.g. dimethyl sulphate for workers considerations).

Unfortunately, there is no real time method to verify the composition of iodide species released particularly the $\text{CH}_3^{131}\text{I}$ form. So, it is primordial to respect the $\text{CH}_3^{131}\text{I}$ generation procedure to well perform the reaction by verifying such parameters as temperature or bubbling/mixing, but also to pay attention to the purity of the different reagents used (e.g. dimethyl sulphate). The respect of these procedures will contribute to the reproducibility of the efficiency tests.

NOTE 1 Feedback and know-how from nuclear operators are key elements of proven procedures for 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 $\text{CH}_3^{131}\text{I}$.

4.4.1.3 Desorption of the $\text{CH}_3^{131}\text{I}$ 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. Typical time of the desorption peak is about several hours after injection, depending on many parameters related to the sorbent (e.g. specific surface and thickness of the charcoal bed) or the operating conditions (e.g. relative humidity and contact time).

To take into account the desorption of the methyl iodide, this current method requires a duration of the samplings (in particular the downstream sampling) 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 (e.g. 1 hour which is a usual industrial practice). It is important to keep this duration for all the tests of the same iodine trap to ensure reproducibility. It is also important to keep in mind that it is

a limit of this method (a short duration of sampling does not take into account the complete phenomenon of desorption).

4.4.1.4 Half-life of the ^{131}I

The half-life of the ^{131}I shall be considered for the activity of the source at the time of the test but also for the time lapse between the sampling and the counting.

4.4.2 Toxicity

The generation device must be in a containment enclosure (e.g. glove box) due to the chemical and radiological toxicities (dimethyl sulphate, liquid Na^{131}I and gaseous $\text{CH}_3^{131}\text{I}$) as well as the volatility of the solutions to be used and the produced gas.

4.4.3 Sampling

4.4.3.1 Representativeness

Sampling shall be representative of ^{131}I concentration in the ventilation duct upstream and downstream the iodine trap to be tested to determine its decontamination factor. For this purpose, general requirements defined in ISO 2889, which describes criteria for sampling at a well-mixed location, shall be respected. Isokinetic sampling is however not required due to injected methyl iodide is a gas.

For upstream sampling, the representativeness of sampling is relative to the well-mix of CH_3I within the ventilation duct from the injection which is usually centred in the ventilation duct.

For downstream sampling, the representativeness of sampling shall be reached considering a non-centred emission due to a default of the iodine trap to be tested, as drawn in [Figure 1](#) (e.g. default of the flange or gasket of the trap).

Moreover, because the goal of the method is to determine an in-situ decontamination factor, it is important that the tracer should be dispersed within the whole section of the ventilation duct to reach all areas of the iodine trap to be tested (including contact zones with the ventilation duct) and not only following recommendations of ISO 2889 relative to the quality of sampling. For this purpose, the concentration of tracer over the major part (more than 2/3) of the cross-sectional area in front of the iodine trap to be tested shall be measured to verify the well-dispersion of the tracer (no measure with a too low concentration).

The representativeness of samplings, but also the concentration of the tracer within the ventilation duct in front of the trap to be tested, shall be characterized once and each time the ventilation configuration changes significantly (air flow rates, geometry, etc.).

Finally, to ensure reproducibility and precision of the method, the use of two identical sampling lines with identical parameters (see [5.2](#)) is preferred to reduce the uncertainties.

4.4.3.2 Sampled volume

Using sampling in this method requires a well estimation of the sampled volume upstream and downstream the iodine trap to ensure low uncertainties on the $\text{CH}_3^{131}\text{I}$ concentrations.

This current method requires calibrated devices (volumetric flow meter or sampling air flow indicator coupled to sampling time).

4.4.3.3 Activities of the iodine test traps

A sufficient thickness of activated charcoal in the iodine test traps (i.e. charcoal cartridges) for each sampling line is necessary to ensure a maximum trapping of the $\text{CH}_3^{131}\text{I}$ to obtain a relevant decontamination factor and to limit radioactive iodine releases downstream sampling lines for workers considerations (see [5.2](#)).

This current method requires the use of two cartridges in series for each sampling line (or at least two separate activated charcoal layers in series inside a bigger cartridge).