
**Iodine charcoal sorbents for nuclear
facilities — Method for defining
sorption capacity index**

*Pièges à iode pour installations nucléaires — Méthode pour définir la
capacité de rétention*

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Foreword

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

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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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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Introduction

Iodine sorbents are extensively used in nuclear facilities to remove radioiodine from gases and air in off-gas cleaning systems and ventilation installations. The sorbents are very important for protection of the members of the public and environment from iodine radionuclides radiation.

In the normal operation of nuclear installations, the main hazard comes from radioactive isotopes of iodine; as examples, for reactors ^{131}I and a minor extent ^{133}I , for fuel processing facilities ^{129}I , etc. Iodine is one of the main contributors of the radiation impact on the environment. Under abnormal and accident conditions, some other isotopes ^{132}I , ^{134}I and ^{135}I have also some significant effects on the total iodine dose (thyroid dose)^[3].

The volatile radioiodine forms can occur in the gaseous radioactive wastes as elemental iodine, the simplest organic compound methyl iodide, and some others such as hydrogen iodide under reducing conditions.

Radioactive iodine can create a serious danger to the members of the public and workers in abnormal and accident conditions at nuclear facilities as far as the exposure in these conditions could be much higher than the exposure due to the natural background radiation.

The need to prevent widespread dispersal of gaseous radioiodine from nuclear facilities is a major purpose of iodine sorbents. It is universally recognized that radioactive methyl iodide is the less readily removable radioiodine form. The removal of radioactive iodine from gaseous radioactive wastes at nuclear facilities is almost always performed with the help of impregnated activated charcoals that have become often accepted as the preferred iodine sorbents used in these facilities. Impregnated charcoals require a high efficiency especially from humid gases containing iodine in order to trap all the iodine gaseous compounds.

Two types of tests are considered^{[2][4]}: laboratory and *in situ* tests.

- Laboratory tests are done to establish the performance characteristics of the charcoal to be used in retention systems under specified operating conditions.
- *In situ* tests are done to obtain a measure of the performance of retention systems under appropriate operational conditions.

This document concerns only the laboratory tests. Laboratory tests of representative samples of charcoal (e.g. new charcoal, aged charcoal from iodine absorbers, etc.) are performed to establish their efficiency for a given test agent under specified conditions.

The quality of sorbents and its potential application at nuclear facilities can be estimated by means of a criterion that defines specifically the sorption capacity of the sorbent. Such criterion is called in this document the sorption capacity index.

The index is defined by the result of a laboratory test on the basis of radioiodine activity distribution inside the sorbent. This index characterizes the total kinetic sorption process for established test conditions and show whether the sorbent can be used as iodine filters for nuclear facilities. One example of criteria is given in [Annex D](#).

This document provides a method to determine the quantitative quality of a sorbent and also to compare the performance of different iodine sorbents at the specified conditions. It is useful for users of iodine sorbents (filter or sorbent manufacturers as well as operators).

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Iodine charcoal sorbents for nuclear facilities — Method for defining sorption capacity index

1 Scope

The scope of this document covers

- iodine sorbents for nuclear power plants, nuclear facilities, research and other nuclear reactors,
- iodine sorbents for laboratories, including nuclear medicine, and
- iodine sorbents for sampling equipment on sample lines.

This document applies to iodine sorbents manufacturers and operators in order to measure the actual performance of these sorbents and their sorption capacity for radioiodine.

This document applies to granulated and crushed iodine sorbents based on activated charcoal (hereinafter referred to as “sorbents”) used for trapping gaseous radioiodine and its compounds. This document establishes the method and conditions for defining sorption capacity index in a laboratory.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

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

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

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

3.1

gaseous radioactive wastes

wastes that contain radioactive material in gas form for which no further use is foreseen and have radionuclides at concentrations or activities greater than clearance levels as established by a regulatory body

3.2

discharge

planned and controlled release of (gas or liquid) radioactive material to the environment

3.3

mass transfer zone

defined zone (range) of sorbent volume in which the phenomena of substance mass transfer from gas to solid phases takes place

3.4

iodine sorbent

sorbent intended for trapping radioiodine in gaseous radioactive wastes

3.5

free volume

void between grains or crushed grains of the sorbent inside the whole volume containing the sorbent

Note 1 to entry: The free volume can be expressed as in [Formula \(1\)](#):

$$V_{\text{fr}} = \chi \cdot V_{\text{sorb}} \quad (1)$$

where

V_{fr} free volume;

χ fraction free volume in sorbent volume;

V_{sorb} sorbent volume.

3.6

contact time

gas flow transit time through sorbent layer

Note 1 to entry: Contact time τ is expressed using [Formula \(2\)](#):

$$\tau = \frac{V_{\text{fr}}}{Q_{\text{col}}} \quad (2)$$

where

Q_{col} the volumetric gas flow rate in the inlet section of column.

Note 2 to entry: The contact time defined here does not consider the geometric volume of the sample, but only its free volume.

3.7

bulk density

ρ_{sorb}

ratio between granulated or crushed grains sorbent mass (definite granulation) and the total volume containing the sorbent

3.8

sorption capacity index

ξ

criterion indicating the degree of reduction of gaseous radioiodine in the gas flowing through the sorbent under specified test conditions

Note 1 to entry: The index is valid only for a specific chemical form of the radioactive gas. In this document, the index has been calculated for radioactive methyl iodide. The index can also be calculated with other gaseous species, such as radioactive iodine, but the results cannot be compared with the ones obtained with radioactive methyl iodide.

4 Principles of the method

The main rationale for the method defined in this document are the following:

- radioactive methyl iodide ($\text{CH}_3^{131}\text{I}$) is the most difficult form of radioiodine to trap in nuclear facilities discharges;
- the distribution of radioactive methyl iodide along sorbent layer is exponential;
- there is an active mass transfer zone in the sorbent layer;

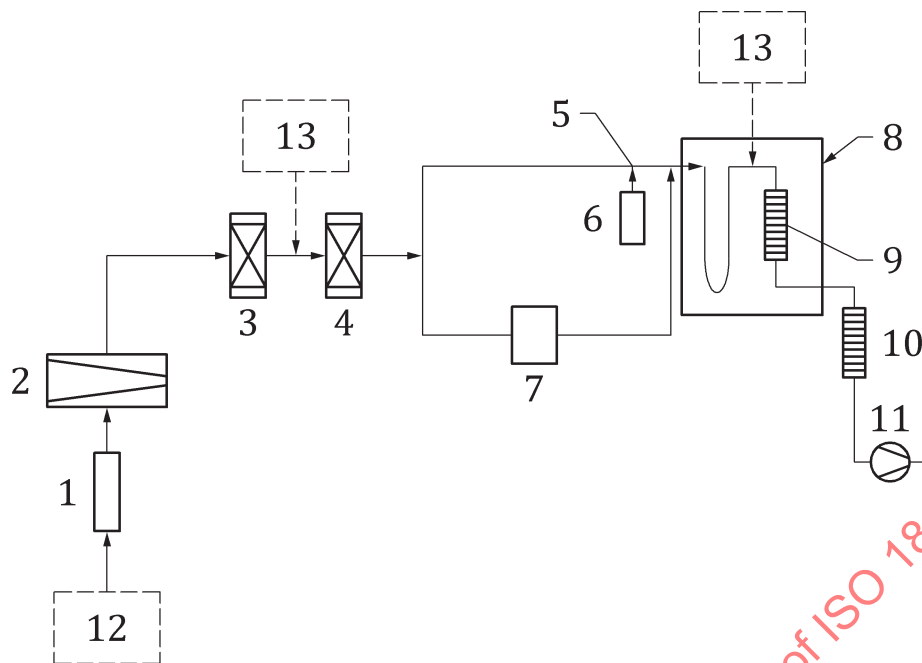
- the amount of sorbed radioactive methyl iodide depends on the contact time between the gas and sorbent.

The sorption capacity index indicates the degree by which radioactive methyl iodide concentration in gas phase is reduced during the contact of the gas flow with the sorbent.

The principle of the method is the following:

- indoor laboratory air is used as carrier gas;
- air is transferred in the test plant by means of extraction device (fan, vacuum pump, etc.);
- in order to remove indoor laboratory air pollutants, aerosols and humidity, air flow passes through an aerosol filter, an air conditioning system used for humidity (e.g. zeolite or dehumidifier) and volatile compounds removal (e.g. activated charcoal);
- the humidity level shall be maintained to a specified value and be controlled. One example for this humidity control is to split the total air flow and pass one of the flows through a second air condition system (e.g. humidifier);
- the air temperature and air flow are regulated and measured before introduction in the sample to test (e.g. the air temperature is control by heat insulation of the sectional column or to place it in a vessel with controlled temperature);
- radioactive methyl iodide is injected into the carrier gas from a generator and then it is trapped in the sectional columns containing the sorbent to be tested;
- before discharging the test gas, the air is passed through a protection column for radiation protection issues and for control of the penetration radioiodine and to ensure a low level of iodine active species in discharged air;
- each section test column is monitored for radioiodine content via gamma monitors (0,364 MeV for ^{131}I);
- the sorption capacity index is calculated using the formula given in [Clause 9](#).

[Figure 1](#) presents the principle of an example scheme of the test plant used for the method presented in this document.



Key

- 1 flowmeter
- 2 aerosol filter
- 3 dehumidifier
- 4 charcoal
- 5 injection of radioactive methyl iodide
- 6 generator of radioactive methyl iodide
- 7 humidifier
- 8 temperature controlled chamber
- 9 sectional column
- 10 protection column
- 11 vacuum pump
- 12 room air
- 13 controller humidity, temperature, absolute pressure and pressure drop

Figure 1 — Schematic of principle equipment and a process diagram of the test plant

5 Preparation of the test

5.1 General

Based on the typical arrangement proposed in [Clause 4](#), the test involves the following stages.

5.2 Removal of air impurity and humidity into the installation

Removing air pollutant and controlling humidity and temperature are important for the tests because these parameters can have a significant impact on the results. In order to clean the installation before a new test, the indoor air is passed through an aerosol filter, dehumidifier and charcoal filter to remove particulates, humidity and some volatile compounds. As an example, dehumidifier with zeolite warms at the temperature 350 °C to 400 °C in vacuum during 6 h and activated carbon to warm at temperature 300 °C to 350 °C in vacuum during 6 h in order to avoid degradation of the charcoal properties.

5.3 Radioactive methyl iodide used for sorbent testing

If radioiodide is supplied in ready-to-use form of radioactive methyl iodide ($\text{CH}_3^{131}\text{I}$) received from producer, a certificate date and activity shall be required. It shall be stored in a dark place at conditions that slows down the process of its decomposition into the molecular form (I_2).

Otherwise, methyl iodide labelled with ^{131}I isotope is produced by its isotopic exchange (e.g. with Na^{131}I or K^{131}I without carrier). Its activity shall be defined and specified with a date. For this purpose, 2 cm^3 of methyl iodide are injected into the container with Na^{131}I or K^{131}I and kept at a room temperature of $20\text{ }^\circ\text{C}$ for 48 h. To define the mass activity concentration (Bq/g), three portions of 5 g of activated carbon impregnated with triethylenediamine (TEDA) are weighed and placed in weighing bottles, the diameter of which is close to that of the test column. Three samples of $4\text{ }\mu\text{l}$ of radio-labelled methyl iodide are taken and placed into weighing bottles with activated carbon. Activities of resulting samples are measured and correlated with methyl iodide mass that is defined as the volume of samples taken and methyl iodide density at the temperature of its storage.

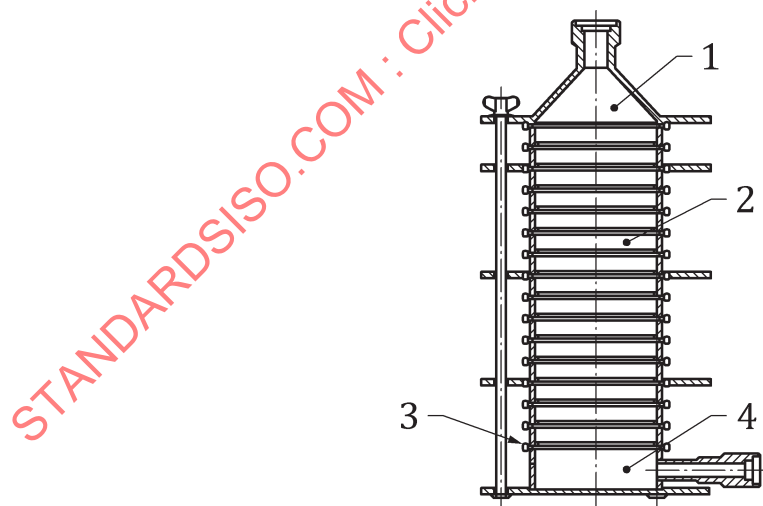
Mean value of mass activity concentration is calculated. The mass activity concentration (expressed in Bq/g) is used for calculation of the mass concentration of methyl iodide in the gas flow (see [Formula 8](#)).

5.4 Preparation of sorbent samples

Range of granulated/crushed grains sizes and bulk density ρ_{sorb} of the sorbent sample are determined if these data are absent. The methods used to determine grains size and the bulk density are defined according to a national or international standard.

When filling each section of the column, the filling shall be made uniformly in each section such that similar mass in each section is obtained, without specific compression or vibration.

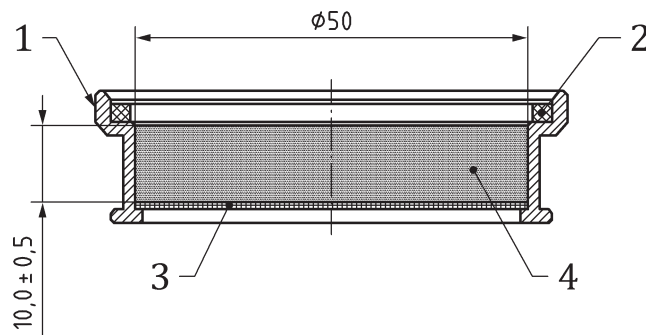
The sectional column is used to conduct the sorbent test. Examples of such column and of a small section inside the column are shown in [Figure 2](#) and [Figure 3](#).



Key

- 1 removable cover with inlet nozzle
- 2 sorbent-containing section
- 3 guide rod
- 4 bottom with outlet nozzle

Figure 2 — Column for sorbent testing



Key

- 1 vessel
- 2 sealing ring
- 3 perforated diaphragm
- 4 sorbent

Figure 3 — Section of sorbent-containing column

The number of sections has to be adapted with regard to the uncertainties of the measurement.

5.5 Measuring devices

The uncertainties of the following measurement devices shall be known:

- temperature sensors;
- pressure sensors,
- relative humidity sensors;
- gamma activity detectors;
- flow rate sensors.

Typical uncertainties are given in [9.3](#).

5.6 Conditions for achieving equilibrium during the test

Prior to testing the sorbent in the sectional column, test conditions shall be maintained such that the conditions for the equilibrium of water vapours inside the samples are achieved (humidifier conditions at gas flow velocity, Q_{col}). This is generally done in 16 h for around 15 cm thickness of sorbent.

5.7 Safe conditions for workers and members of the public

The tests use a radioactive form of iodine. Thus, the tests shall be performed following national radiation protection regulations (for workers and members of the public) in force in the country. All the handling with radioactive iodine solutions and iodine vapours shall be performed under protective glove boxes. The glove box should be in a negative pressure enough to avoid to spread contaminated flows to the rooms.

The test installations shall be equipped with specific protection devices allowing to efficiently reduce the iodine releases towards the environment (e.g. protection columns represented in the equipment 10 of the [Figure 1](#)), connected to the air ventilation system of the building. Examples of protection columns are made with silver impregnated zeolites, silver impregnated aluminium oxide and qualified activated charcoal.

6 Test conditions

In order to ensure accurate and comparable measurement of sorbent performance, the following conditions shall be met.

The following parameters shall be specified with their accuracy:

- specified temperature;
- specified gas relative humidity;
- gas flow transit time through the sorbent layer (contact duration);
- concentration of $\text{CH}_3^{131}\text{I}$ in gas;
- specified CH_3I mass concentration in gas.

The absolute pressure shall be measured for the test, and the accuracy of the measurement shall be known.

The relative humidity and the gas flow of the test bench are set to achieve nominally each relative humidity (RH) conditions for the sorbent on the scale 30 % RH to 90 % RH.

The following parameters are fixed, within range intervals, for the purpose of estimating the potential to use sorbents under high relative humidity (abnormal) conditions and comparing the charcoal performance index:

- specified gas relative humidity: $(90,0 \pm 3) \%$ in order to cover accidental conditions;
- specified temperature: $(30,0 \pm 0,5) ^\circ\text{C}$;
- gas flow transit time through the sorbent layer (contact duration) — contact time in the range from 0,20 s to 0,40 s;
- concentration of $\text{CH}_3^{131}\text{I}$ in gas: from 10^3 Bq/m^3 (for the efficiency of the test) to a higher concentration to be defined with regard to worker safety;
- mass concentration of methyl iodide (mostly the stable one) in gas flow: 1 mg/m^3 to 20 mg/m^3 (adsorption versus temperature is a linear function for methyl iodide concentration in this region).

Additionally, the total tested sorbent layer shall be sufficiently thick for the formation of an active mass transfer zone.

Other comparisons at other humidity levels (e.g. 30 % RH, 40 % RH, 70 % RH) or test temperature (e.g. room temperature) shall also be possible, but exclusively with the same specified conditions.

NOTE The experience at a laboratory scale has shown good repeatability of the results with these parameters.

7 Sorbent testing

Each section of the sectional column (see [Figure 3](#)) is weighed, the sections are filled with the sorbent, after which each section is once again weighed. Based on the difference in weights and bulk density, the sorbent volume, V_i , in each section is determined.

Sorbent volume in the column, V_{sorb} , is calculated using [Formula \(3\)](#):

$$V_{\text{sorb}} = \frac{G_{\text{sorb}}}{\rho_{\text{sorb}}} \quad (3)$$

where

G_{sorb} sorbent mass in the column;

ρ_{sorb} sorbent bulk density.

The sectional column is assembled and hermetically connected to thermostat gas lines. Radioactive methyl iodide is placed into the generator. The humidifier is filled in with demineralized water or filled with normal water with a specific demineralization system.

The humidifier temperature and gas flow velocity are set to achieve nominally the specified humidity conditions for sorbent.

Approximate volumetric gas flow rate, Q , is calculated on the basis of the values of fraction free volume in the sorbent volume and chosen contact time, τ , using [Formula \(4\)](#):

$$Q = \frac{V_{\text{fr}}}{\tau} \quad (4)$$

The free volume V_{fr} is calculated using the formula:

$$V_{\text{fr}} = \chi \cdot V_{\text{sorb}} \quad (5)$$

where

χ fraction free volume in sorbent volume.

When data related to free volume is absent, a nomogram (see [Figure 4](#)) can be used to determine the free volume, based on experimental values [\[2\]](#) [\[5\]](#) [\[6\]](#).

In the example of [Figure 4](#), ℓ is the granule size (mm) and $(\ell_{\text{min}} - \ell_{\text{max}})$ corresponds to a granules range of 93 % to 98 % of the granules number.

Draw a straight line connecting corresponding values of minimal and maximal granule size through the plenum fraction scale. The point of intersection shows the desired value.

The uncertainty related to the free volume shall be established.

For the example of [Figure 4](#), the uncertainties are linked on the χ fraction free volume on the nomogram and to the data used to establish the nomogram are 8 %.

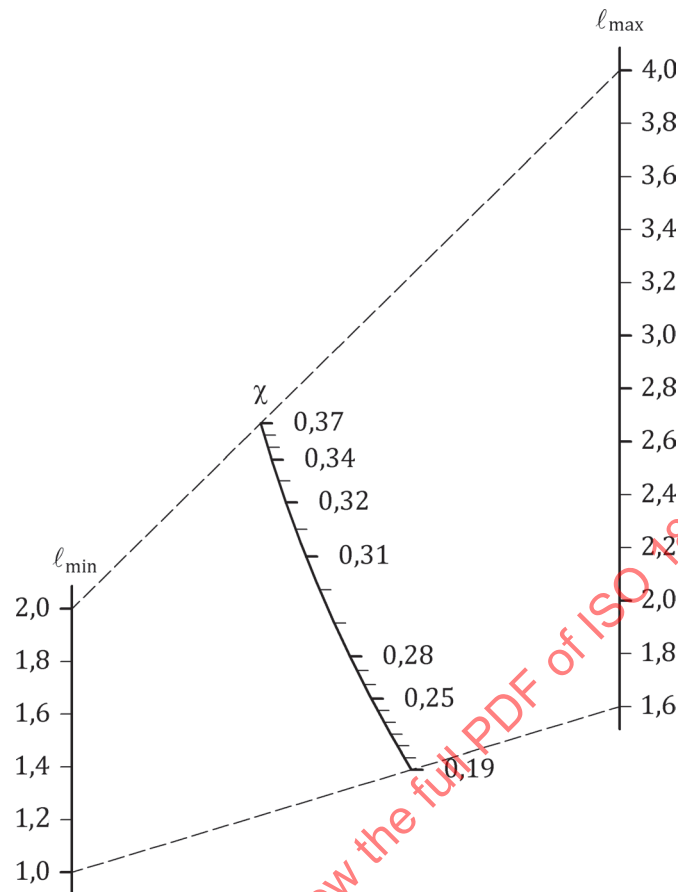


Figure 4 — Nomogram for defining the fraction of free volume in the entire volume containing granulated activated non-compressed charcoal to be used when free volume data are absent

8 Test performance

The test procedure is as follows.

- The specified conditions (as identified in [Clause 6](#)) are maintained stable during all the test.
- $\text{CH}_3^{131}\text{I}$ vapour injection into the gas flow is started, and gas mixing continues for enough time to identify a distribution along the sections (typically 3 h for around 15 cm thickness, to be adapted depending of the mass transfer zone). After this time, the injection is stopped and sorbent test is then terminated.
- The columns are removed from the test equipment and disassembled into separate sections (if any).
- ^{131}I activity is measured in each section. ^{131}I activity is measured in terms of gamma radiation line with 0,364 MeV energy and converted into Becquerel's. The measurement uncertainties shall be known.
- The environment protection column is removed from the plant and ^{131}I activity is also measured in the sorbent.
- Gas activity concentration is defined as the ratio of the activity measurement for the whole sectional columns and protection columns divided by the total volume of gas that passed through the sorbent.
- Mass concentration is defined as the ratio of all the activity measurement for sorbents in test and protection columns divided by the specific activity of $\text{CH}_3^{131}\text{I}$ and the total volume of gas that passed through the sorbent.

9 Determination of the sorption capacity index

9.1 Sorption capacity index determination

The determination procedure is as follows.

Determine the activity of sorbent for each section of column and the activity of the protection column.

Determine the activity relative to mass of sorbent for each section column A.

Calculate total sum activity A, Bq, using [Formula \(6\)](#):

$$A = A_{\text{col}} + A_{\text{pr}} \quad (6)$$

where

A_{col} activity of sectional column;

A_{pr} activity of protection column.

If injected activity is known, compare it with the total sum activity A. If the difference is higher than the accuracy of the tests measurement, the results of the test cannot be used and the installation shall be checked.

Calculate air flow rate in the inlet of the column containing tested sorbent Q_{col} (cm³/s) using [Formula \(7\)](#):

$$Q_{\text{col}} = \frac{(p_0 - \phi_1 \cdot p_1^0) \cdot Q_{\text{in}} \cdot T_2}{(p_2 - \phi_2 \cdot p_2^0) \cdot T_1} \cdot \left(1 + \frac{\phi_2 \cdot p_2^0}{p_2} \right) \quad (7)$$

where

p_0 barometric air pressure, Pa (kPa);

ϕ_1 relative humidity of atmospheric air inlet, before conditioning, fraction;

p_1^0 saturated water vapour pressure at T_1 , Pa (kPa);

Q_{in} air flow rate in the plant inlet, cm³/s;

T_1 atmospheric air temperature, K;

T_2 operating temperature of the column, K;

p_2 absolute pressure gas flow measured in the input to the column with tested sorbent, Pa (kPa);

ϕ_2 measured relative humidity of gas flow passing into the column with tested sorbent, fraction;

p_2^0 saturated water steam pressure at the column operating temperature with tested sorbent, Pa (kPa).

Mass concentration of methyl iodide in gas flow during performance testing sorbent is calculated using [Formula \(8\)](#):

$$C = \frac{A}{I \times V} \quad (8)$$

where

C mass concentration of methyl iodide, kg/m³ (mg/m³);

I mass activity concentration of methyl iodide labeled by isotope of ¹³¹I, Bq/g;

V total volume of gas passed through column during performance testing sorbent, m³.

Concentration shall be between 1 mg/m³ to 20 mg/m³.

Further to construct plot ($\ln A_i$) as function along layer of sorbent (L).

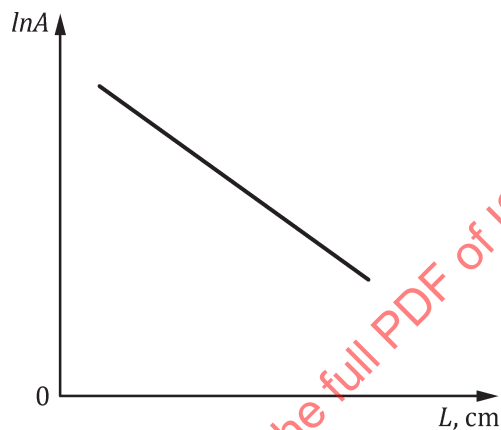


Figure 5 — Logarithmic activity distribution along sorbent layer

If the experimental line is linear (Figure 5) it means the distribution of radioactive methyl iodide is circumscribed exponential law and on the basis results of measurement can calculate sorption capacity index, if not the sorption capacity index cannot be calculated.

9.2 Calculating sorption capacity index

Determine volume of sorbent in each section column using Formula (9):

$$V_i = \frac{g_i}{\rho} \quad (9)$$

where

V_i volume of sorbent in section, cm³;

g_i mass of sorbent in section, g;

ρ bulk density, g/cm³.

Calculate $\ln \frac{A}{A - \sum_{i=1}^m A_i}$ for the layers of sorbent in column.

Calculate real contact time for each section using [Formula \(10\)](#), where $m \leq n$:

$$\tau_i = \frac{\chi \cdot \sum_{i=1}^n V_i}{Q_{\text{col}}} \quad (10)$$

where

Q_{col} air flow rate as calculated in [Formula \(7\)](#);

χ fraction free volume in sorbent volume.

Construct plot $\ln \frac{A}{A - \sum_{i=1}^m A_i}$ as a linear function of τ (see [Figure 6](#)).

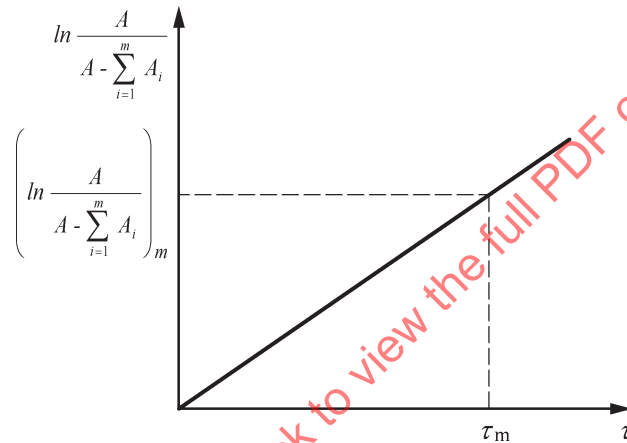


Figure 6 — Logarithm activity as function contact time

For any contact time, find the value logarithm ratio activities using the plot.

The sorption capacity index of the sorbent ξ , is calculated according to [Formula \(11\)](#) for the contact time 0,2 s to 0,4 s:

$$\xi = \frac{1}{\tau} \ln \frac{A}{A - \sum_{i=1}^m A_i} \quad (11)$$

For an example using sorption index, see [Annex D](#).

9.3 Uncertainties of measurements

All the uncertainties shall be consistent with the range of parameters to control defined in [Clause 6](#), and in any case shall be less than the ones described in the [Table 1](#).

Table 1 — Measurement accuracy requirement for instrumentation

Measuring parameters	Limiting error for the maximum sensor range
Atmospheric air	
— absolute pressure	$\pm 1,0$ kPa
— temperature	$\pm 1,0$ °C
— relative humidity	$\pm 1,5$ %
— gas flow speed	± 5 %
Inlet sectional column	
— absolute pressure	$\pm 1,0$ %
— temperature	$\pm 0,5$ °C
— relative humidity	$\pm 3,0$ %
— pressure differential in the column	$\pm 1,0$ %
Test sample	
— weighting sample	$\pm 0,01$ g
Radiometric unit	
— sample activity	$\pm 3,0$ %
Fraction of free volume	± 8 %

All the uncertainties values in [Table 1](#) are expanded relative uncertainties (denoted U) of ± 1 (66 % confidence interval).

The global uncertainty value is expressed at an enlarged confidence interval of 2 sigma (95 %).

10 Documenting test results

The test results shall comprise the following elements:

- test report (see [Annex A](#));
- internal test report (see [Annex B](#));
- measurement results (see [Annex C](#)).

For the purpose of solving potential controversial issues, the tested samples may be stored in the laboratory for a given period with a specified date for the sample.

Annex A (normative)

Format of test report (for the customer)

Laboratory

Title
Address
Phone (fax) No.
E-mail address

Date of testing

The person who conducted the test
Name
Position
Signature

Customer:

Title
Address
Phone (fax) No.

Rated test conditions (to be expressed with measurement uncertainties):

Sorbent (sample) reference

Temperature _____ K (°C)
Relative humidity _____ %
Gas flow rate _____ m³/s (cm³/s)
Column diameter _____ m (mm)
Sorbent layer thickness _____ m (mm)
Contact time _____ s
Injected activity (if known) _____ Bq

Conclusion:

1 Sorbent sorption capacity index (ξ) _____ s⁻¹;

2 Comments

_____ (approver name and signature)