OPTIMIZATION OF A PASSIVE SAMPLING METHOD FOR TRITIUM MEASUREMENT IN INDOOR AIR OF NUCLEAR FACILITIES

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In terms of mobility in the environment, the atmospheric transport of tritiated water (TW) is important for understanding the behaviour of tritium. Therefore, knowing the amount of TW in the atmosphere is essential from the radioprotection point of view.

This paper presents the results obtained from experimental tests using the passive collector with calcium chloride developed at the Radiation Protection Laboratory of the Institute for Nuclear Research-Pitesti. The accuracy of the sampling method was assessed by performing tests in a controlled tritium-contaminated atmosphere. The dynamics of the water sorption process in the collector material has been studied with the scope of optimizing this method.

Keywords: passive collectors, water vapour, TW

1. Introduction

The radioisotope of hydrogen, tritium (^3H or T), is a low-energy beta-emitting radionuclide with a half-life of 12.33 years, which exists everywhere in the environment [1]. Its presence can have both natural causes because of the processes of interaction of gases and cosmic rays in the upper atmosphere, as well as artificial causes because of the atmospheric weapon tests and routine or accidental releases from the nuclear industry. Tritium is produced by neutron capture in nuclear power plants, mainly in the water coolant and moderator, in quantities depending on the reactor type [2]. In the case of Canadian-designed CANDU reactors, tritium is one of the radionuclides with the largest share in the radioactive emissions, because these types of reactors use heavy water (known as deuterium oxide) as a moderator and primary agent. The average production rate of tritium in the moderator is approximately 7.5 x 10^{10} Bq/kg of heavy water per year. According to the data published by the Canadian Nuclear Safety

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Commission, the total release from a CANDU power reactor is about 3.3×10^{14} Bq/yr, with releases occurring through both gaseous and liquid pathways. In all cases, tritium releases must be strictly controlled in order not to represent a risk to people's health or the environment [3]. Efficient management of tritium in a CANDU power plant is one of the most important issues to be resolved, given the fact that the production of this has a major impact on the conduct of current operational activities. Tritium has been identified as the main factor contributing to the dose received from internal exposure of workers; therefore, the implementation of specific radiation protection programs and, implicitly, the allocation of a budget necessary to manage the problems caused by tritium releases are necessary [4].

Most of the tritium in the power plant circuits is found in the form of the molecular compound DTO, which makes it closely linked to the circulation of heavy water in the plant. During the operation of the plant, any escape of heavy water from the heat-transport and moderator systems or their auxiliaries will involve a release of tritium. There are implemented a whole series of operations for the efficient recovery of technological heavy water containing tritium leaks. However, a significant proportion of heavy water leaks are lost, being released into the environment, together with tritium, as liquid or gaseous effluents [3], [5].

Tritium in the form of vapours can spread to adjacent areas. Since the installations of the two heavy water circuits are entirely located in the reactor building [6], leaks take place entirely inside technological rooms, which causes the air in these rooms to become radioactively contaminated by the presence of tritiated water (TW) vapour in equilibrium with the heavy water from the installation. Although the decontamination of the air inside these rooms is ensured by the ventilation and air conditioning systems in the reactor building, the presence of a quantity of free or adsorbed tritiated water on the surfaces of contaminated objects in the room will permanently contribute to air contamination. From the point of view of workers' exposure, tritium in the form of tritiated water vapour is more hazardous than the hydrogen gas, because it can be easily and rapidly incorporated into the body by inhalation, ingestion, or through the skin [7]. Therefore, the localization of the points in the installation where tritiated water leaks occur represents an efficient strategy for reducing both the volume of tritiated water released into the environment as liquid or gaseous effluents and the concentration of tritium in the air inside the room, and, implicitly, minimizing workers' exposure. Monitoring TW in the indoor atmosphere of nuclear facilities is a critical safety and operational practice, also representing a tool for assessing occupational exposure. One of the solutions available for monitoring TW in air consists of online tritium monitoring systems, which provide timely information in case of accidental leakage. However, these instruments are quite expensive, are not so efficient in terms of the detection limit and indicates only the level of radioactive tritium contamination of the air corresponding to the location where these are fixed, having limited possibilities to characterize the heterogeneity of TW present in the atmosphere of large enclosure [8].

Another solution is passive monitoring of tritiated water vapour in air, which represents a cost-effective and practical method for assessing tritium levels in nuclear facilities. Unlike active monitoring systems, which rely on powered instruments for real-time data, passive monitoring uses low-maintenance devices such as diffusion samplers or absorption materials to collect and measure TW concentration. These passive devices are capable of covering a larger spatial area, are easy to install, and can be placed in inaccessible or remote areas where power supply and active monitoring might be impractical [9, 8].

In the recent decades, a series of passive samplers have been developed for monitoring tritiated water vapour in the air, in response to the need for cost-effective methods, reliable and low-maintenance solutions for the long-term environmental and workplace safety monitoring. Examples of such passive devices presented in literature and whose performances have been demonstrated include: diffusion absorption samplers based on silica gel [8] or molecular sieves [10,11], and samplers based on chemical absorbers as calcium chloride or membrane-based samplers [12].

This study introduces and validates the passive collector with calcium chloride, developed by the Radiation Protection Laboratory of the Institute for Nuclear Research-Pitesti. This type of collector was designed for quantifying airborne tritium and represents a cost-effective, convenient, and easy-to-use tool. The aim of the paper was to optimize and demonstrate the performance of the tritium measurement method in atmospheric water vapour using passive collectors. The experimental tests conducted under controlled and real environmental conditions demonstrate the reliable performance of the passive method. The findings also confirm that the method achieves a good sensitivity, making it suitable for routine surveillance of occupational exposure.

2. Materials and methods

2.1. Equipment

The activity of tritium in samples was measured with a liquid scintillation counter (TriCarb 4910TR) calibrated with the NIST standard SRM 4947C. The weight of samples was measured using a calibrated analytical balance (AXIS model ACN220), and the temperature and humidity were measured by a calibrated thermo-hygrometer (PeakTech 5180).

2.2. The method for determining tritium in air using passive collectors

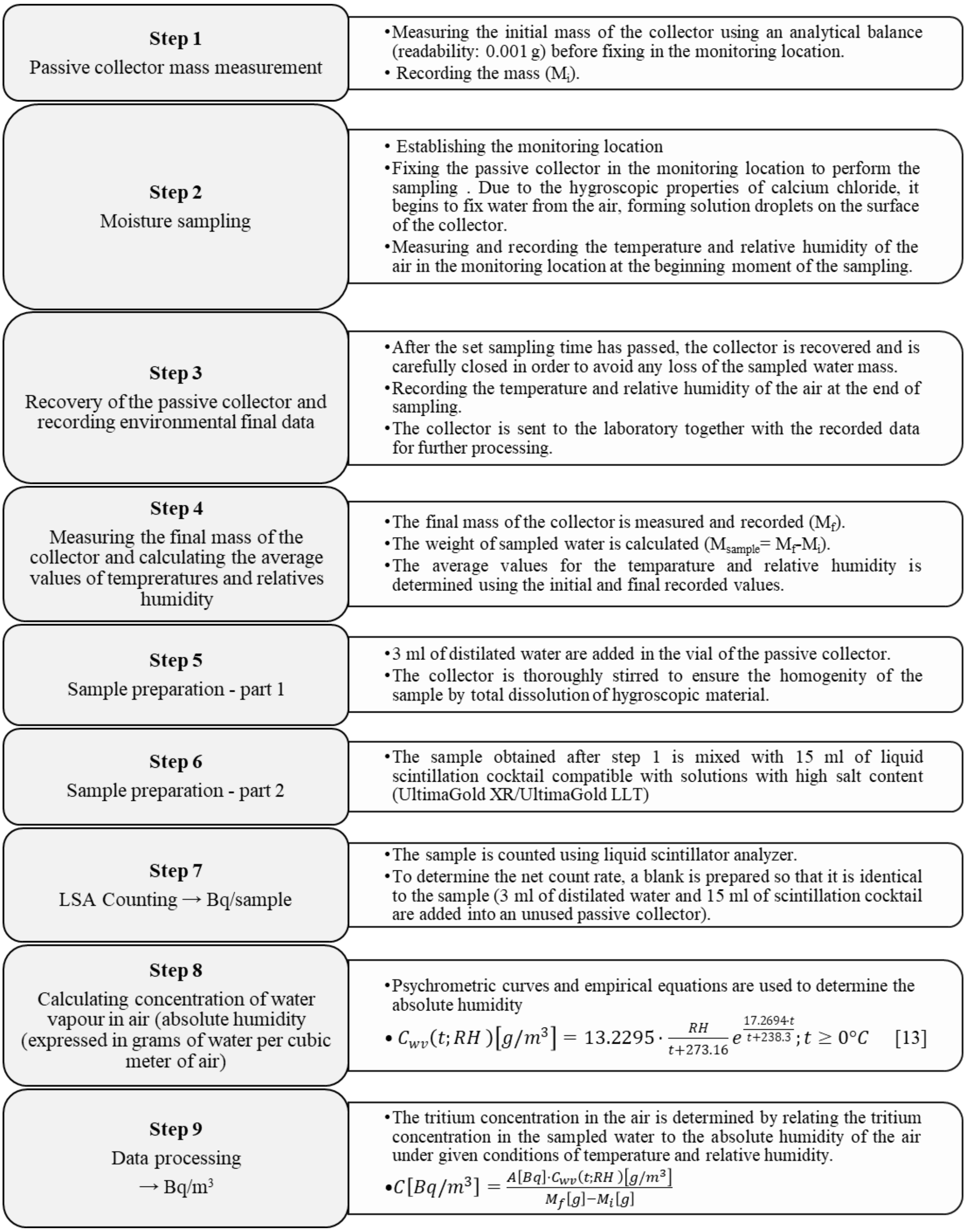
The passive collector with calcium chloride has been developed within the Radiation Protection Laboratory of the Institute for Nuclear Research Pitesti and is presented in Fig. 1. This has been patented, and the invention patent has been registered at the State Office for Inventions and Trademarks Bucharest, under the number RO 128555.

Fig. 1. Passive collector

The collector is for single use only and consists of a polyethylene vial for liquid scintillator counting (20 mL volume) with a modified cap. A polyethylene tube is attached to the cap, on which surface anhydrous calcium chloride is fixed using a water-free fixing agent.

The principle of the sampling method consists of the sorption of tritiated water vapour on a collector with calcium chloride, followed by the measurement of the tritium concentration in the sampled water. The tritium concentration in the air is determined by relating the activity concentration in the sampled water to the concentration of water vapour in the air.

The procedure for determining the tritium concentration in the air using the passive calcium chloride collector is presented in the form of a process diagram in Fig. 2.



Step 1 Passive collector mass measurement	•Measuring the initial mass of the collector using an analytical balance (readability: 0.001 g) before fixing in the monitoring location. • Recording the mass (M_i).
Step 2 Moisture sampling	• Establishing the monitoring location •Fixing the passive collector in the monitoring location to perform the sampling . Due to the hygroscopic properties of calcium chloride, it begins to fix water from the air, forming solution droplets on the surface of the collector. •Measuring and recording the temperature and relative humidity of the air in the monitoring location at the beginning moment of the sampling.
Step 3 Recovery of the passive collector and recording environmental final data	•After the set sampling time has passed, the collector is recovered and is carefully closed in order to avoid any loss of the sampled water mass. •Recording the temperature and relative humidity of the air at the end of sampling. •The collector is sent to the laboratory together with the recorded data for further processing.
Step 4 Measuring the final mass of the collector and calculating the average values of tempreratures and relatives humidity	•The final mass of the collector is measured and recorded (M_f). •The weight of sampled water is calculated (M_{sample}= M_f-M_i). •The average values for the temparature and relative humidity is determined using the initial and final recorded values.
Step 5 Sample preparation - part 1	•3 ml of distilated water are added in the vial of the passive collector. •The collector is thoroughly stirred to ensure the homogenity of the sample by total dissolution of hygroscopic material.
Step 6 Sample preparation - part 2	•The sample obtained after step 1 is mixed with 15 ml of liquid scintillation cocktail compatible with solutions with high salt content (UltimaGold XR/UltimaGold LLT)
Step 7 LSA Counting → Bq/sample	•The sample is counted using liquid scintillator analyzer. •To determine the net count rate, a blank is prepared so that it is identical to the sample (3 ml of distilated water and 15 ml of scintillation cocktail are added into an unused passive collector).
Step 8 Calculating concentration of water vapour in air (absolute humidity (expressed in grams of water per cubic meter of air)	•Psychrometric curves and empirical equations are used to determine the absolute humidity • $C_{wv}(t;RH)[g/m^3] = 13.2295\cdot\frac{RH}{t+273.16}e^{\frac{17.2694\cdot t}{t+238.3}}; t \geq 0°C$ [13]
Step 9 Data processing → Bq/m³	•The tritium concentration in the air is determined by relating the tritium concentration in the sampled water to the absolute humidity of the air under given conditions of temperature and relative humidity. • $C[Bq/m^3] = \frac{A[Bq]\cdot C_{wv}(t;RH)[g/m^3]}{M_f[g]-M_i[g]}$

Fig. 2. The procedure for determining the tritium concentration in air

3. Performance tests and method validation

3.1. Experiments under simulated conditions

The accuracy of the method for determining the concentration of tritium in air was experimentally tested by collecting water vapour from the air that fills the empty volume of a vial containing tritiated water solution with known activity concentration. An equilibrium is expected to be established due to the fact that the tritium from the TW solution can pass into the vapour phase and mix with the air in the empty volume of the vial. Thus, the concentration of tritium in the tritiated water solution will be equal to the tritium concentration in water vapour existing in the air from the empty volume of the vial. To achieve this equilibrium quickly, a 250 ml vial was used, which contained approximately 30 ml of tritiated water with a concentration of 76.5 Bq/l. The solution was prepared gravimetrically using a standard solution. Also, the solution obtained was characterized by LSC. The vial thread was compatible with the passive collector thread, thus allowing the collector to be installed inside the vial, without significantly disturbing the tritium concentration of saturated water vapour in the vial. The experimental arrangement scheme is presented in Fig. 3.

Fig. 3. The experimental arrangement for determining the accuracy of the method

Eight passive collectors were used in the experimental tests, each of them being recovered after an exposure time of 10 minutes. Considering that the collectors are placed facing downwards, their handling must be done very carefully to avoid loss by leakage of the drops formed on the surface of the passive collector (drops of saturated calcium chloride solution). The samples collected were prepared according to the procedure presented in the previous chapter, and the activity concentration of the collected water was measured using the liquid scintillator analyser.

3.2. Experiments under real conditions

In order to verify the accuracy of the tritium determination method in the air by sampling humidity using the passive collector, experimental tests have been planned and carried out. These consisted of taking humidity samples to determine the tritium concentration in the air using 2 different methods:

- a passive method – the method in which the collection of water vapour is carried out using the passive collector
- an active method – the method in which the moisture sampling is carried out using the water vapour condensation device (a method whose performance has been evaluated and proven in other experimental tests within the laboratory).

The experiments were conducted within the Radioactive Waste Treatment Plant of the Institute for Nuclear Research Pitesti, in the area dedicated to the treatment of liquid radioactive waste. These wastes contain both gamma-emitting radionuclides as well as tritium. Tritium, due to its volatile nature, is released during the treatment process, leading to air contamination in the area. Four monitoring locations were established at different distances from the main tritium generating sources.

Through the condensation method, it was collected a considerably larger amount of water than the amount collected in the same time interval by using the passive method. This makes it more efficient but at the same time more sensitive to variations in temperature, humidity, or flow field.

Influenced by the source term and the exposure environment, the TW vapour level often presents significant variations over time, which makes it critical to carefully consider the method’s sensitivity when evaluating its practical value. Sampling using the two methods was carried out simultaneously under identical environmental conditions.

Some researchers have noted that the sampling rate of a passive sampler is affected by the surrounding wind speed, especially for passive collectors used to collect tritiated water vapor from the atmosphere, when these are placed in an open environment where the wind speed can vary significantly [10, 14]. Since the indoor environments of the nuclear facilities have less turbulence, the air flow speed in the vicinity of the collector wasn't measured during the testing. In order to prevent any variation in the movement of the air flow that could affect the sorption efficiency, during the sampling, the movement of people through the room and the opening of doors/windows were avoided.

Within the experimental tests carried out, for each monitoring location, the initial and final environmental conditions were determined (temperature – t °C and relative humidity – RH%. For both parameters, the initial values corresponding to each monitoring location were very close to those recorded at the end of the sampling, which demonstrates the constancy of the environmental condition during the sampling period.

After one hour (the time when the samples were taken), all the samplers were sealed and transported to the laboratory immediately after deployment.

The mean temperatures and relative humidities for each monitoring location, together with other necessary data for calculating the tritium concentration in the air, are shown in Table 1.

Table 1

Information characteristic of the sampling

Monitoring location	1st Location		2nd Location		3rd Location		4th Location	
Method used for sampling	Passive method	Active method	Passive method	Active method	Passive method	Active method	Passive method	Active method
Collector code	1P	1C	2P	2C	3P	3C	4P	4C
M_i (g)	8.1016	16.5160	8.7259	16.4930	8.0040	16.4930	8.3136	16.5160
M_f (g)	8.1624	19.6876	8.8012	21.7162	8.0382	22.4220	8.3548	23.1350
M_{sample} (g)	0.0608	3.1716	0.0753	5.2232	0.0342	5.9290	0.0412	6.6190
Mean temperature (°C)	26.95	26.95	24.90	24.90	26.60	26.60	24.70	24.70
Mean RH (%)	56.0	56.0	58.5	58.5	43.0	43.5	44.5	46.0
C_{wv} (g/m^3)	14.27	14.27	13.3	13.3	10.75	10.87	10.01	10.34
Air volumes from which the samples were taken (m^3)	0.0043	0.2222	0.0057	0.3926	0.0036	0.5453	0.0041	0.6399

3.3. Experimental test for method optimization

Calcium chloride is both hygroscopic and deliquescent. Thus, solid material will absorb moisture from the air until it dissolves, and the solution will continue to absorb moisture until an equilibrium is reached between the vapour pressure of the solution and that of the air. If the humidity of the air increases, more moisture is absorbed by the solution; if it decreases, water evaporates from the solution to the air [15].

The concentration of tritium in the air is determined by relating the concentration of tritium in the sampled water to the absolute humidity value of the air under given conditions of temperature and relative humidity. If the tritium concentration in the air in the monitoring room and the environmental parameters remain constant during the sampling time, the concentration of tritium in the sampled water is expected to be in equilibrium with the concentration of tritium in the tritiated water vapor in the air. If the collector is exposed beyond its calculated saturation time, the quantity of water captured will stop increasing and the dynamic exchange with the atmospheric water will continue without any change in mass of the sample. The desorption may occur only when the humidity in the air decreases significantly and it is due to the slow desorption of the captured water.

The rate at which moisture is absorbed by a given quantity of calcium chloride or its solutions depends on: the surface area of the calcium chloride exposed to the air, the rate at which air circulates over the calcium chloride and the water vapour pressure of the air to that of the calcium chloride [15].

These experimental tests aimed to determine the optimal length of the time interval for sampling water vapour from the air. For this purpose, several passive collectors have been prepared and used to study the dynamics of the sorption phenomenon by placing them in a controlled atmosphere in terms of relative humidity and temperature. The experimental tests were carried out both in free atmosphere and inside an enclosure to ensure constant environmental conditions throughout the experiment. The absolute air humidity varied between 2.45 and 22 g/m^3.

For each collector, the amount of water sampled was repeatedly determined over short time intervals (5-10 minutes), from the beginning of the sampling until the recovery efficiency was over 90%.

4. Results and discussion

4.1. Results of experimental tests conducted under simulated conditions

The tritium concentrations measured in the water samples collected using the eight passive collectors are graphically represented in Fig. 4. The black dots represent ^{3}H concentrations in the water collected, and the blue dots represent the recovery percentage calculated as the ratio between the ^{3}H concentration in the water sampled and that of the initial solution.

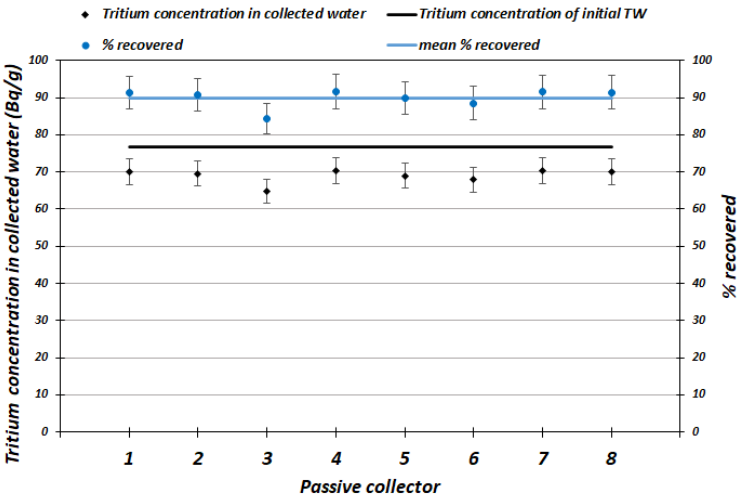


Fig. 4. The tritium concentration in water collected and the recovery performance

The measured tritium concentrations in the water samples had values between 64.7 and 70.3 Bq/g, being a maximum of 15.6% lower than the concentration of the tritiated water solution. These discrepancies could be justified by the fact that during the mounting of the passive collector inside the tritium controlled contaminated enclosure, it was briefly exposed to an uncontaminated atmosphere (maximum 3-5 seconds representing the time in which the collector was unscrewed from its vials and fixed inside the enclosure from which the sampling was made and the time in which the collectors was fixed back in its vials). During this time interval, calcium chloride absorbed uncontaminated water vapours from the atmosphere, implicitly leading to lower tritium concentration in the sampled water.

4.2. Results of experimental tests under real conditions

After sampling, the samples were prepared for determining the tritium concentration in the air. The activity values measured in the samples corresponding to the four monitoring locations using the 2 methods, together with the calculated values of the tritium concentration in the air, are presented in Table 2. The uncertainty budget of the activity concentrations was established considering the uncertainty arising from counting statistics (sample and background) and counting efficiency. The relative uncertainty arising from counting efficiency is computed taking into account the relative uncertainty of NIST standards used for equipment calibration (which is 1.6% according to the certificate of traceability) and the uncertainty arising from the counting statistic of standards (which is 1%).

Table 2

The concentration of tritium in the air determined using the two methods

Monitoring location	Method used for sampling	Collector code	^{3}H activity (Bq/sample)	^{3}H conc. in air (Bq/m^3)	Discrepancies (%)
1st Location	Passive method	1P	7.3 ± 0.5	1706.1 ± 103	13.8
	Active method	1C	439.9 ± 22.0	1979.2 ± 98.7	
2nd Location	Passive method	2P	8.7 ± 0.6	1525.8 ± 91.9	2.1
	Active method	2C	612.2 ± 30.6	1559.2 ± 77.8	
3rd Location	Passive method	3P	1.0 ± 0.1	294.4 ± 19.5	14.0
	Active method	3C	183.7 ± 9.2	336.9 ± 16.8	
4th Location	Passive method	4P	4.8 ± 0.3	1144.5 ± 69.8	-2.7
	Active method	4C	710.8 ± 35.5	1110.7 ± 55.4	

The results obtained in the experimental tests using the two methods described above were comparatively assessed by graphically representing the tritium activity concentrations in air determined using the passive collector method, depending on the tritium activity concentrations in air determined based on the condensation method (Fig. 5). The discrepancies between the results obtained using the two methods are less than 14% which is explained by the inhomogeneity of the distribution of tritium concentration in air in the room during sampling, with the existence of a concentration gradient towards the tritium-generating source.

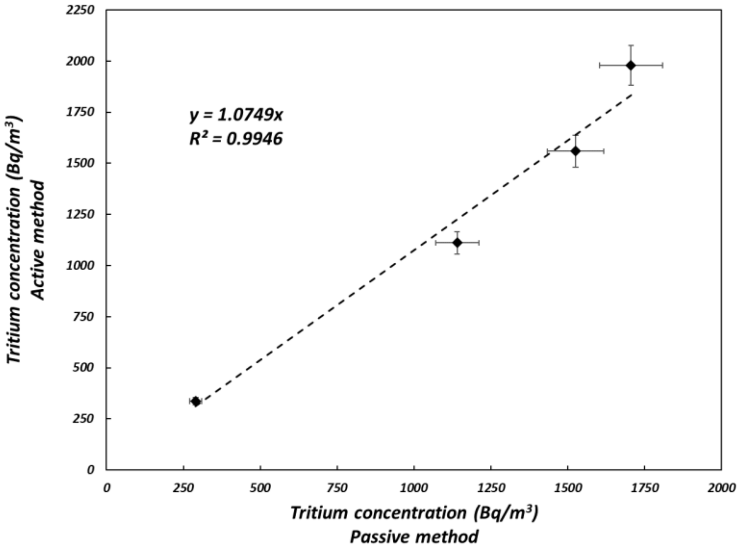


Fig. 5. Comparison of tritium concentration in air determined with active and passive sampling methods

The performance of the passive sampling method is limited by the amount of calcium chloride deposited on the surface of the passive collector, the environmental conditions, and the length of the sampling interval. The Minimum Detectable Activity (MDA) expressed in Bq/m^3 was calculated for the four monitoring locations, based on the real environmental conditions, the masses of collected water vapor after one hour exposure (Table 1), and assuming one hour counting time. The calculated MDA values fall within the range 17.1-29.8 Bq/m^3, the maximum and minimum MDA values corresponding to the samples with the lowest, respectively, the highest amount of water vapor sampled. The method indicates good sensitivity, allowing the determination of tritium concentration in air in a short time interval, compared to other methods in which sampling is done over periods of time from tens of hours to several tens of days [8, 10].

According to Publication 30 of the International Commission on Radiological Protection (ICRP) [16] and using the doses coefficients and derived limits for occupational intakes of radionuclides presented in Publication 119 [17], the annual limits on intake (ALI) for tritiated water is 1×10^9 Bq and the derived air concentrations (DAC) for tritiated water (in a working year of 2 000 h) is 5.5×10^5 Bq/m^3. The comparison between the obtained MDA values and the DAC demonstrates that the proposed passive sampling method is suitable for accurately determining tritium concentrations in air at levels significantly lower than 1% of the DAC.

4.3. Results of the experimental test for method optimization

The mass of water collected is graphically represented as a function of time (Fig. 6), and the data were fitted with the following exponential function:

$$m_{(t)} = a \cdot (1 - e^{b \cdot t}) \tag{1}$$

Where $m_{(t)}$ represents the total amount of water sampled at time t, t is the sampling time, b is defined as the hydration reaction constant, and a is the mass of water at saturation. Under the test conditions, this constant can have different values, based on which the "half-life time" can be calculated using the equation (2):

$$T_{1/2} = -\frac{ln(2)}{b} \tag{2}$$

After approximately seven "half-life time" the collector reaches saturation.

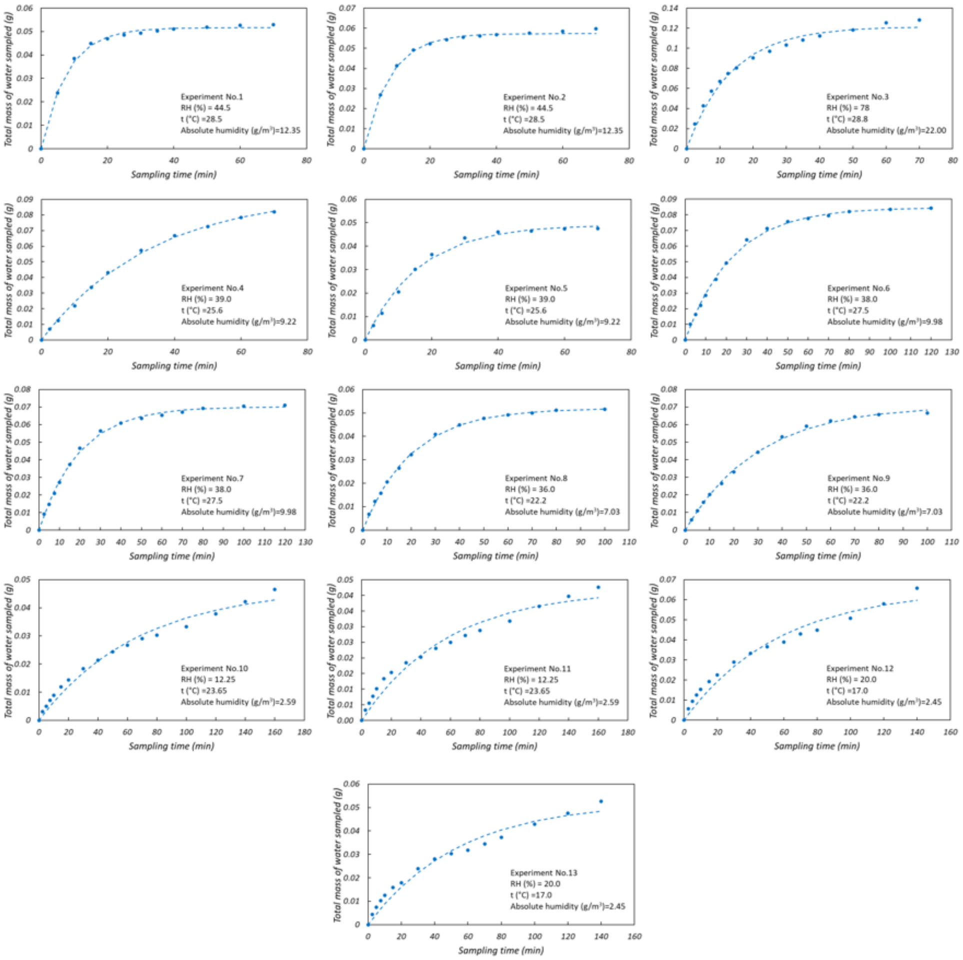


Fig. 6. The dynamics of the adsorption process for different environmental conditions

Following the evaluation of the obtained results, it was found that the time at which the collector reaches saturation depends both on the environmental conditions (absolute humidity calculated using the relative humidity and temperature) and on the amount of calcium chloride contained in the passive collector.

The equation that can be used to determine the time at which the collector reaches saturation is:

$$T = a + b \times \frac{M_{CaCl_2}}{C_{wv}} \tag{3}$$

where M_{CaCl2} is the mass of calcium chloride (g), and C_{wv} is absolute humidity (g/m^3), a and b are fitting constants.

The obtained results are presented in Table 1, and Fig. 7 illustrates the comparison between the experimental and calculated times at which the collector reaches saturation.

Table 2

The experimental and calculated times at which the collector's sorption capacity is reached

Experiment number	Mass of calcium chloride (g)	Absolute humidity (g/m^3)	Experimental time in which the collector reaches saturation (min)	Calculated time in which the collector reaches saturation (min)
Exp. No.1	0.0393	12.41	38	56
Exp. No.2	0.0444	12.41	38	61
Exp. No.3	0.0807	22.00	65	62
Exp. No.4	0.0934	9.22	162	135
Exp. No.5	0.0443	9.22	78	75
Exp. No.6	0.0798	9.98	112	111
Exp. No.7	0.0669	9.98	96	96
Exp. No.8	0.0486	7.03	98	99
Exp. No.9	0.0658	7.03	146	126
Exp. No.10	0.0712	2.60	329	332
Exp. No.11	0.0576	2.60	280	272
Exp. No.12	0.0599	2.88	276	298
Exp. No.13	0.0513	2.88	264	258

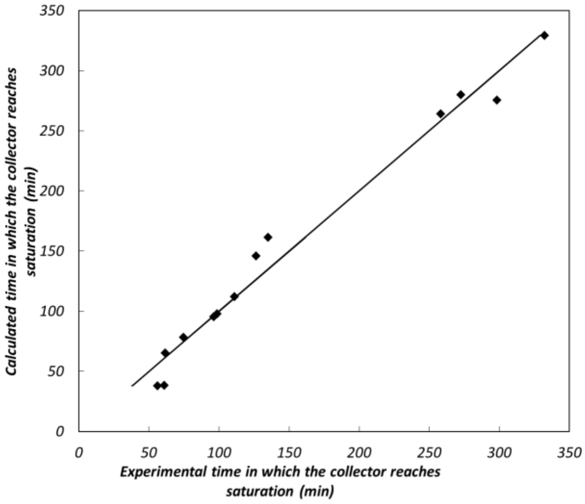


Fig. 7. Experimental time in which the collector reaches saturation vs. calculated time

By analysing the values corresponding to the time in which the collectors reach saturation, it can be observed that passive collectors containing an average of 0.04 g of calcium chloride reach saturation after approximately 60 minutes if sampling is carried out from an environment characterized by an absolute humidity greater than 9 g/m^3. The time required to reach saturation increases considerably for the collectors exposed to environments whose absolute humidity is less than 3 g/m^3.

6. Conclusions

To determine and optimize the parameters of the method, the dynamics of the water vapour sorption process in the collector material was studied. The performance of the sampling method is limited by the amount of calcium chloride deposited on the collector surface, the environmental conditions, and the length of the sampling interval. The results obtained showed that the time at which the collector reaches saturation as a function of absolute humidity value and of the amount of calcium chloride contained in the passive collector could be estimated with enough accuracy. The experimental and theoretical results are comparable for values of absolute humidity between 2.6 and 22.0 g/m^3.

The method optimization tests show that under given temperature and humidity conditions, which are close to common environmental conditions encountered in technological spaces (RH ~ 40% and temperature ~ 20°C), the collector's sorption capacity is reached within a time frame of approximately one hour, which makes efficient sampling possible through short-term collector exposures. The experiments conducted in the atmosphere with controlled contamination of tritium, in the form of tritiated water vapour, demonstrate the method’s accuracy.

The experimental tests performed in real field conditions demonstrate the robustness of the passive method. The tritium activity concentration values in the air obtained with both methods tested (the passive collector and the active method) are similar. The passive method can be applied both for detecting accidental tritium leaks from technological installations and for quantifying tritium in the air, being a simple and inexpensive method that can be easily implemented even in places that are difficult to access or without energy sources.

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