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Nuclear Instruments and Methods in Physics Research A 484 (2002) 650–659

**NUCLEAR
INSTRUMENTS
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IN PHYSICS
RESEARCH**
Section A

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An alternative approach to tritium-in-water monitoring

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Received 21 May 2001; received in revised form 3 August 2001; accepted 3 September 2001

Abstract

A paradigm shift in the approach to tritium-in-water monitoring is considered and its viability demonstrated through a series of proof-of-principle experiments. The fundamental difference in the proposed detection system consists of changing the state of the tritiated water from the liquid phase to the gas phase and subsequently using any one of a variety of detection methods to detect tritium in the water vapour.

Proof-of-principle experiments using an un-optimized heated ionization chamber illustrate the viability of the proposed method. Operation of the experiment as a continuous in-line detection system shows a detection limit of $\sim 20 \mu\text{Ci/l}$, a detection time constant of 10 s, and simplicity and effectiveness of the overall system. It is projected that an optimized heated ionization chamber system using state-of-the-art current measuring circuitry, could yield a detection limit of $\sim 0.2 \mu\text{Ci/l}$. Furthermore, application of a more sensitive detection system (for example, the gas electron multiplier) is expected to result in additional gain in sensitivity.

The advantages of the proposed tritium-in-water monitoring system are: high sensitivity with a detection limit beginning to approach current day batch liquid scintillation detection systems; a truly continuous, on-line tritium-in-water monitor with response time of less than tens of seconds; a detection system potentially applicable for multiple stream monitoring; a system that does not generate any waste; and, a system that overcomes the fouling issues associated with current day on-line LSC systems. © 2002 Elsevier Science B.V. All rights reserved.

PACS: 29.40.Cs; 29.40.Ym; 28.52.Nh*Keywords:* Tritium; Water; On-line; Monitor; Ionization; Vapour

1. Introduction

A variety of techniques for monitoring tritium-in-water have been reported in the literature [1–9]. The simplest and most efficient of these methods is

liquid scintillation counting (LSC). ‘Grab sampling’ LSC routinely permits detection of tritium at concentrations of 10 nCi/l of water. An automated version of this is a quasi-continuous system that mixes a sample of pre-filtered water and a liquid scintillant, which is then directed to a photocell; this system yields a detection limit of the order of 10 nCi/l. Another less common method for tritium detection is solid scintillation counting (SSC). This technique is fundamentally less efficient than liquid scintillation counting

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because it relies on the principle of surface detection while LSC is based on the principle of volume detection. Automated solid scintillation counting detector systems have demonstrated detection limits around $1 \mu\text{Ci/l}$.

The automated liquid scintillation counting system while providing a low detection limit has the drawbacks of not being truly continuous, water filter fouling, production of tritiated liquid waste, and the associated significant cost of organic solvents, disposition of the tritiated solvents and attendant frequent maintenance requirements. The solid scintillation detection system, in contrast, has a much faster time response and is not as expensive a system to manage. However, this system has the disadvantages of needing a high surface-to-volume ratio, requiring prefiltration of the water, being vulnerable to fouling, and being subject to memory effects from tritium retention on the solid scintillant.

The common aspect in the two detection systems described above is that the state of the sampled tritiated water during the entire process of measurement is liquid. If alternatively one is prepared to consider the transformation of the water sample from the liquid phase to the gas phase, interesting consequences follow.

In the simplest case, tritiated water vapour¹ at an ambient pressure of 1 atm can be monitored for tritium content by using the principle of gas ionization. The betas produced as a result of tritium ($\text{HTO}_{(\text{g})}$) decay will traverse the water vapour and produce a number of ionizations which can be measured as a current signal in an ionization chamber. A functional system can be realized by the combination of a water vaporization sub-system and a suitable ionization chamber held at an elevated temperature. This is a continuous monitoring system with a fast time response principally determined by the quotient of the ionization chamber detection volume to the volumetric flow rate of the water vapour. Also, in comparison to the systems described above this system is cost effective and requires little to no

maintenance considering that the water vapour can be condensed and returned to the original water stream without any generation of waste and that the vaporization process² has none of the filtration and fouling issues. As for the detection limit of this system, it is determined by the detection limit of the ionization chamber and certain operating parameters; this is discussed and demonstrated in the subsequent sections.

A variety of alternatives to the system described in the preceding paragraph can be recognized by simply replacing the ionization chamber detector with another detector. One possibility is the use of a gas scintillation counting (GSC) detector [10] where the continuous stream of water vapour is mixed with a suitable scintillating gas in an ultraviolet³ optical detection chamber outfitted with one or more UV (ultraviolet) sensitive photomultiplier tubes. Yet another prospect is the use of a proportional detection chamber or/and an electron gas multiplication detector [11–13] where the gas ionizations are amplified by the placement of strategic electrodes creating high electric fields, thus leading to a lower detection limit. And yet another plausible system is a variation of the gas scintillation detector wherein suitable gas mixtures and the presence of high electric fields are used to amplify the measured UV signal.

From the above account it is evident that the fundamental implication of converting liquid water to water vapour is that the rarefied water ($\text{H}_2\text{O}_{(\text{g})}$ and $\text{HTO}_{(\text{g})}$) permits the tritium decay betas to now have a range of the order of millimeters as opposed to micrometers in liquid water. This in turn allows one to measure the number of disintegrations by application of the principles of gas ionization, gas scintillation and electron gas multiplication, all of which are based on the principle of volume detection. A corollary

¹Water vapour by definition is water in the gaseous state which is devoid of liquid water (droplets).

²A simple water vaporization system may consist of a liquid vortex centrifuge, from the centre of which water is aspirated into a hot pipe (containing appropriate baffles) and thus creating steam, followed by a gas vortex designed to knock-out any remaining water droplets and leading to the production of dry steam or water vapour free of liquid water droplets.

³Typical scintillating gases, such as nitrogen, argon and helium, emit in the UV.

of converting liquid water to dry steam, which can be easily accomplished for water streams containing particulate and biological elements, is simplification of water filtration requirements, no generation of contaminated organic waste, minimization of maintenance operations, and thus a reduction in the operating costs of the entire system.

With the foregoing as a framework for an alternative approach to tritium-in-water monitoring [14], this paper will present a theoretical description and proof-of-principle experimental results demonstrating the viability of using an ionization chamber for tritium-in-water monitoring.

2. Theory

According to the principle of gas ionization, the saturated current i_s resulting due to the presence of a tritium concentration of c_g in a detection volume V can be expressed as

$$i_s = \frac{c_g V \lambda E_m e}{W_{H_2O}} \quad (1)$$

where λ is the tritium decay rate constant, E_m is the mean tritium decay beta energy, W_{H_2O} , which is usually referred to as the W -value, is the mean energy expended by the emitted beta radiation to form an ion pair in water vapour, and e is the electronic charge. Using the definition

$$c_g \equiv \frac{N_{HTO}}{V} \quad (2)$$

where N_{HTO} represents the number of vaporized HTO molecules present in the detection volume V , Eq (1) can be expressed as follows:

$$i_s = \frac{N_{HTO} \lambda E_m e}{W_{H_2O}}. \quad (3)$$

Tacit in the above relation is the presence of water vapour in volume V at a pressure p which exceeds the threshold pressure above which the saturated current is unaffected by variations in pressure and less than an upper pressure limit beyond which charge recombination effects become significant. The above relationship represents a correspondence between the saturated current, which is

approximated by the net measured current, and the number or activity of vaporized HTO molecules in the ionization chamber detection volume. Defining the quotient of the saturated current and the number of vaporized HTO molecules as the specific saturated current, we obtain the following:

$$I_{HTO \text{ in } H_2O} \equiv \frac{i_s}{N_{HTO}} = \frac{\lambda E_m e}{W_{H_2O}}. \quad (4)$$

The specific saturated current, $I_{HTO \text{ in } H_2O}$, in short I , is a constant determined by the decay rate and mean beta energy of tritium and the ionization property of water vapour. The measurable signal current, i , which approximates the saturated current, can now be simply expressed as

$$i = I c_g V. \quad (5)$$

In order to establish a correspondence between the current signal from an ionization chamber and tritium activity in pre-vaporized liquid water, let c_w be the concentration of HTO in liquid water. The mass of water vapour at pressure p and temperature T in a detection volume V can be expressed simply as

$$m = \frac{p V A_{H_2O}}{RT} \quad (6)$$

where R is the ideal gas law constant and A_{H_2O} is the molecular weight of water. Using the preceding equations we obtain the relationship for the tritium concentration in water c_w in terms of the measured current i

$$c_w = \frac{c_g V}{m} = \frac{i}{I} \frac{RT}{p V A_{H_2O}}. \quad (7)$$

The preceding relationship assumes that the vapour/liquid partition of tritium in tritiated water is negligible [15].

The detection limit for a bakeable ionization chamber is a current signal of 1 fA⁴. For a detection volume of 11 (10⁻³ m³), water vapour pressure of 1 atm, monitor and gas temperature of 250°C, and W_{H_2O} value of 29.6 eV per ion pair [16], one obtains a corresponding detection limit of tritium in water of 2.1 μCi/kg or 2.1 μCi/l. From observation of Eq. (7) it is evident that the detection limit for tritium in water can be

⁴ 1 fA = 1 femtoampere = 10⁻¹⁵ A.

improved by increasing the detection volume and water vapour pressure and by lowering the measurable current signal. For example, a ten-fold increase in the detection volume will result in a tritium in water detection limit of $0.2 \mu\text{Ci/kg}$ while a doubling of the water vapour pressure would result in a further improvement to $0.1 \mu\text{Ci/kg}$. Recently, advances in current measurement circuitry suggest that a detection limit of 0.5 fA is achievable which would imply a calculated tritium in water detection limit of $0.05 \mu\text{Ci/kg}$. One might conservatively presume that such a detection system in reality might not do better than a factor of 5 of the calculated detection limit, which then suggests a practical detection limit of $0.25 \mu\text{Ci/kg}$. It should be noted that one other change which could also improve the detection limit is an increase in the specific saturation current or alternatively introducing an electron multiplying gas in the water vapour, such as methane, which will effectively reduce the W -value and thus improve the sensitivity of the detector.

It is now worth considering the influence of the presence of any liquid water droplets in the water vapour sample on the measured signal. The beta particles produced via tritium decay have a range of about 6 mm in gaseous media such as air while the range in liquid water is about $3\text{--}5 \mu\text{m}$. It is known that the presence of liquid water droplets in water vapour at temperatures significantly above

the boiling temperature of water is not uncommon unless special measures have been taken to remove the water droplets. Further, a typical water droplet present in water vapour has a diameter of the order of microns. Therefore, it is not unreasonable to expect that some of the beta particles emitted by tritium atoms in the droplet will not escape the droplet (to enter the gas phase). Hence, these betas do not contribute to the production of gas ionizations, which in turn leads to an attenuation in the current signal. From this argument, it is seen that minimization, if not avoidance, of the presence of water droplets in water vapour is quite desirable.

3. Experiment

A schematic of a simple experimental system designed to demonstrate the viability of using an ionization chamber [17,18] for tritium-in-water detection is shown in Fig. 1. The system consists of a bakeable ionization chamber tritium monitor, a water boiler to vaporize liquid water, a septum on the boiler to permit injection of $\text{HTO}_{(l)}$ with a needle and syringe, a water cooled condenser to condense the dry steam, and a weigh scale to measure the rate of flow of water. The ionization chamber tritium monitor and the flow lines between it and the boiler and condenser are heated

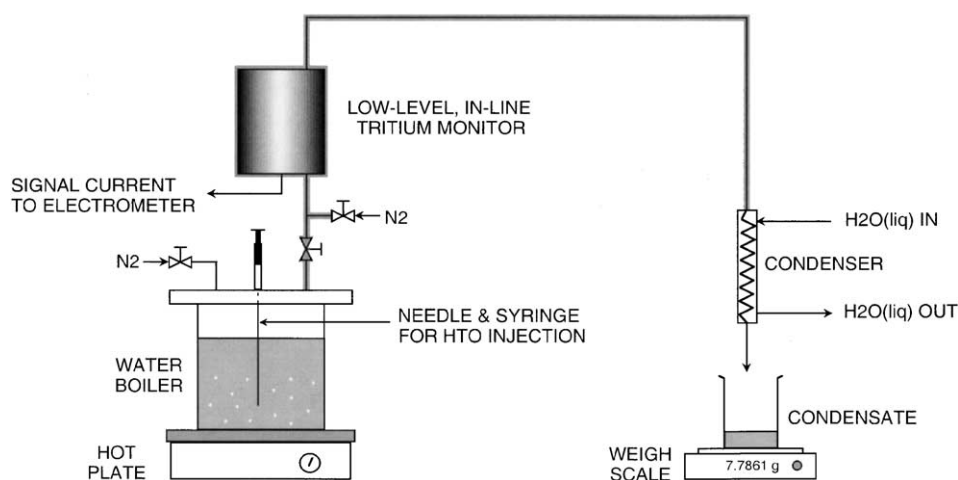


Fig. 1. Schematic of the tritium-in-water monitoring proof-of-principle experiment.

to $\sim 250^\circ\text{C}$, thus ensuring the presence of dry steam and avoiding any condensation of water within the ionization chamber. The current signal from the ionization chamber is measured with a Keithley 617 electrometer wherein the collector is held at a bias of -100 V DC ; the current-time data is collected by a data acquisition system.

A typical experiment involved pre-heating of the ionization chamber and the flow lines leading to and leaving the chamber⁵ to a temperature of $\sim 250^\circ\text{C}$, and heating of tritium-free deionized water in the boiler to a steady boil. Upon achieving a steady-state condition, the background current signal is noted and the corresponding condensate is analyzed for background tritium activity using a liquid scintillation counter. Subsequently, a small volume of tritiated water is injected via a syringe and needle in the septum into the boiling water and the ensuing response of the ionization chamber is observed. Once again, upon achieving a steady-state condition, the current signal is noted and the corresponding condensate is analyzed for tritium activity. This procedure is repeated for each additional injection of tritiated water. During the course of each experiment the rate of mass increase on the weigh scale is noted to obtain the flow rate of water.

It is noteworthy that the ionization chamber monitor background current signal is negative for all the tests and it diminishes progressively with each test (-95 , -25 and -3 fA for Tests 1, 2 and 3, respectively). The negative current represents a combination of effects due to tritium contamination on ionization chamber surfaces and offset current in the amplifier circuitry of the electrometer. The progressive decrease in the background signal is due to tritium decontamination of internal surfaces of the monitor.

4. Results

Experimental results for three experiments with progressively lower concentrations of tritium in

water are shown in Figs. 2–6 and in Tables 1–3. The figures show the ionization chamber current-time plots while the tables summarize the steady-state data for each test. The flow rates of water in the three tests are as follows (tests 1, 2, and 3): $3.5\text{ ml H}_2\text{O}_{(l)}/\text{min}$ ($4.41\text{ H}_2\text{O}_{(g)}/\text{min}$), $3.1\text{ ml H}_2\text{O}_{(l)}/\text{min}$ ($3.91\text{ H}_2\text{O}_{(g)}/\text{min}$), and $4.1\text{ ml H}_2\text{O}_{(l)}/\text{min}$ ($5.11\text{ H}_2\text{O}_{(g)}/\text{min}$).

A total of five separate tritium injections were carried out in Test 1 such that the first three injections resulted in tritium-in-water concentrations of the order of a few tens of $\mu\text{Ci/l}$, while the last two injections had concentrations of the order of tens of mCi/l . The current signals due to the first three injections (Figs. 2 and 3) while being rather noisy show hints of a non-zero signal. The current signals due to injections four and five are observed very clearly. The large spikes in the current-time plots corresponding to the injection of tritiated water are due to physical disturbances of the cable carrying the signal from the monitor to the electrometer. The steady-state data in Table 1 shows that following injections 3, 4 and 5 the tritium-in-water concentration as predicted by the ionization monitor signal is in good agreement with the tritium activity in the condensate as measured by LSC.

The second experiment, which was essentially a repetition of the first experiment, was carried out more carefully in order to re-observe the current response of the ionization chamber during the various stages of the test. Also, the temperature of the flow line between the ionization chamber and the condenser was elevated to 250°C as opposed to 170°C in Test 1; the temperatures of the ionization chamber and the flow line between the boiler and the ionization chamber remained unchanged at 250°C .

The current-time plot in Fig. 4 shows that as the water in the boiler comes to a boil the current signal begins to drop, followed by a sharp drop in the current to a minimum as the water begins to boil, and then the current signal begins to rise until it reaches a steady state value which corresponds to the steady-state current signal prior to the heating of the water. This result indicates a momentary occurrence of a leakage current at the electrical feedthrough in the ionization cham-

⁵ An exception to this is experiment one where the flow line between the ionization chamber and the condenser was held at 170°C .

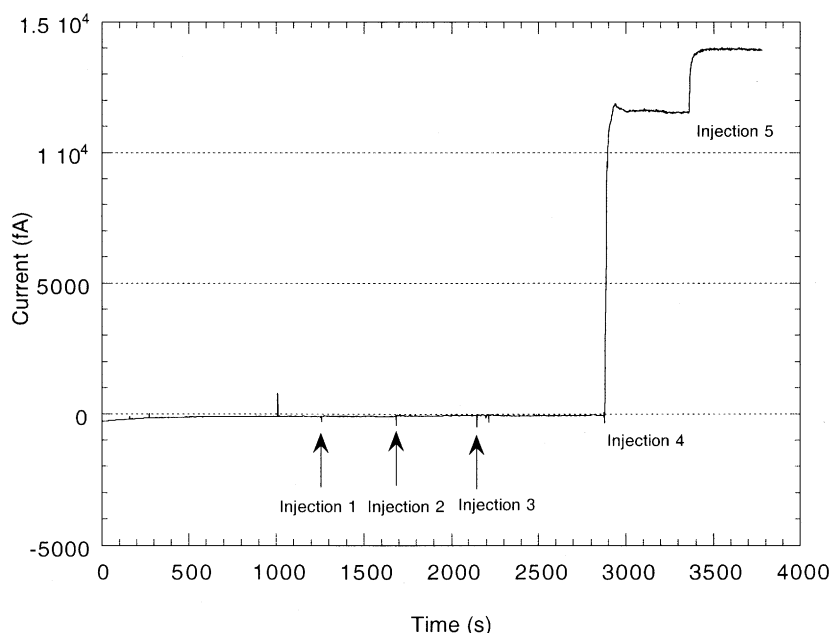


Fig. 2. Test 1. Tritium-in-water monitoring using water vapour and a heated, in-line, ionization chamber.

ber as the system comes to a steady-state operating condition. The explanation for this result is that while the monitor is at a temperature of 250°C , the high purity, glazed alumina ceramic feedthrough is at a lower temperature⁶ and therefore is a site for the condensation of the vaporized steam which leads to the observed leakage current. However, as the water vapour continues to flow through the monitor, the feedthrough is convectively heated and in due course the condensed water on the surface of the feedthrough evaporates and the leakage current diminishes.

The current–time plot of Fig. 4 also shows that, with the exception of injection 1 (where the signal cable was disturbed), there are no large current spikes as observed in the previous test. Upon introduction of tritium into the boiling water a monotonic increase in the current signal is observed for each of the injections. The current–

time plot along with the corresponding data in Table 2 show that changes in tritium concentrations of less than $70\text{ }\mu\text{Ci/l}$ are clearly measurable.

The current–time plot for Test 3 (Fig. 5) shows similar results to those observed in Test 2 with the exception that the incremental concentrations of tritium-in-water are smaller. It is also interesting to note that in this experiment the water in the boiler at the start of the experiment was not found to be free of tritium as is evident from the non-zero net current signal. In fact, LSC analysis of the condensate confirmed this result; that is, a predicted background tritium-in-water concentration of $44\text{ }\mu\text{Ci/l}$ compares favourably to the condensate activity of $37\text{ }\mu\text{Ci/l}$ (see Table 3). As before, Test 3 shows good agreement between the ionization chamber predicted concentrations and the condensate concentrations. Also, in this experiment it is evident that a tritium-in-water concentration approaching $\sim 20\text{ }\mu\text{Ci/l}$ is measurable. Furthermore, in Fig. 6 the instrument time response is observed to be of the order of less than 10 s ($\sim 20\text{ s}$ to realize a change of $> 90\%$ of the steady-state change in signal).

⁶The assumption being that the entire monitor is not at a uniform temperature. Further, the lower thermal conductivity of the ceramic permits it to be at a lower temperature than its surroundings.

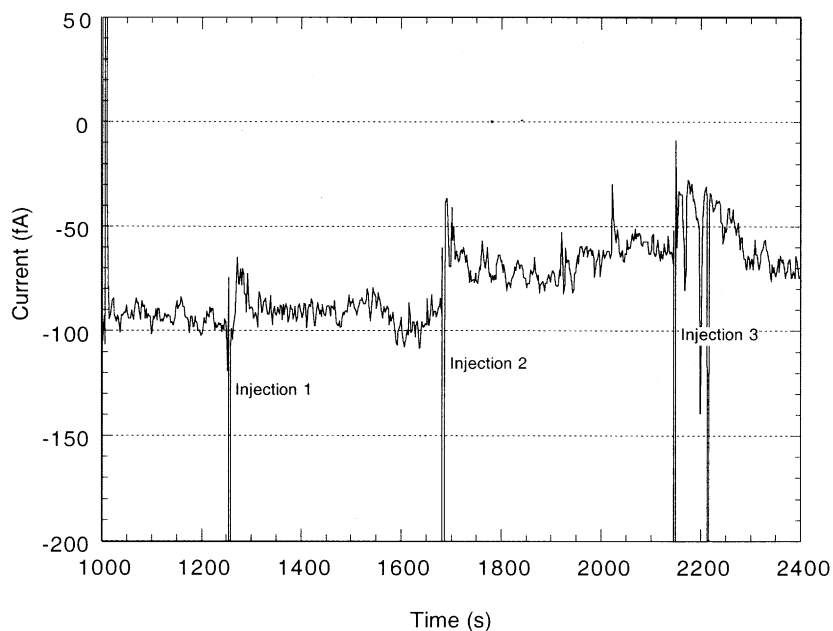


Fig. 3. An expanded view of the response during the first three injections in test 1.

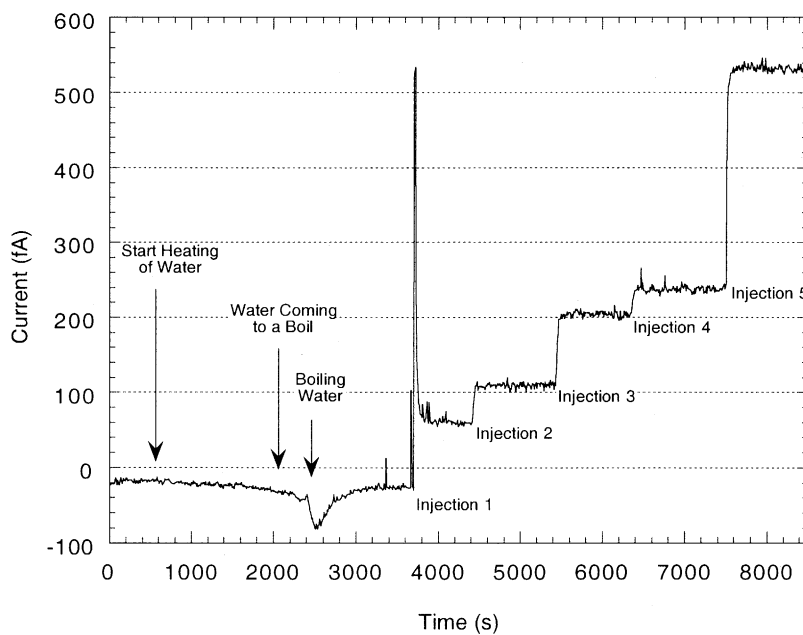


Fig. 4. Test 2. Tritium-in-water monitoring using water vapour and a heated, in-line, ionization chamber.

The ratio of the tritium activities as predicted by the tritium monitor to that measured in the condensate is of the order of unity $\pm \sim 15\%$.

Vapour/liquid partition of tritium in tritiated water will only account for a variation of a few (~ 3) percent [15]. However, closer observation of

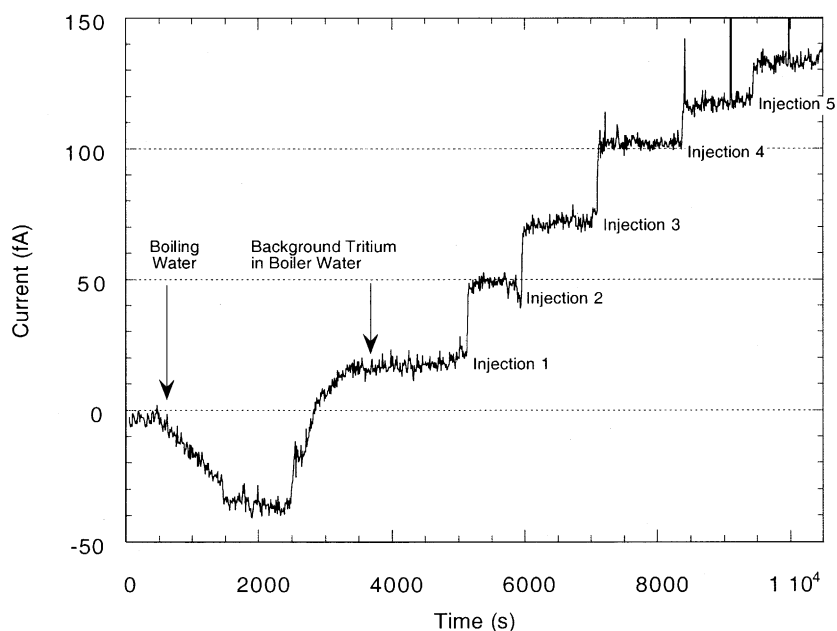


Fig. 5. Test 3. Tritium-in-water monitoring using water vapour and a heated, in-line, ionization chamber.

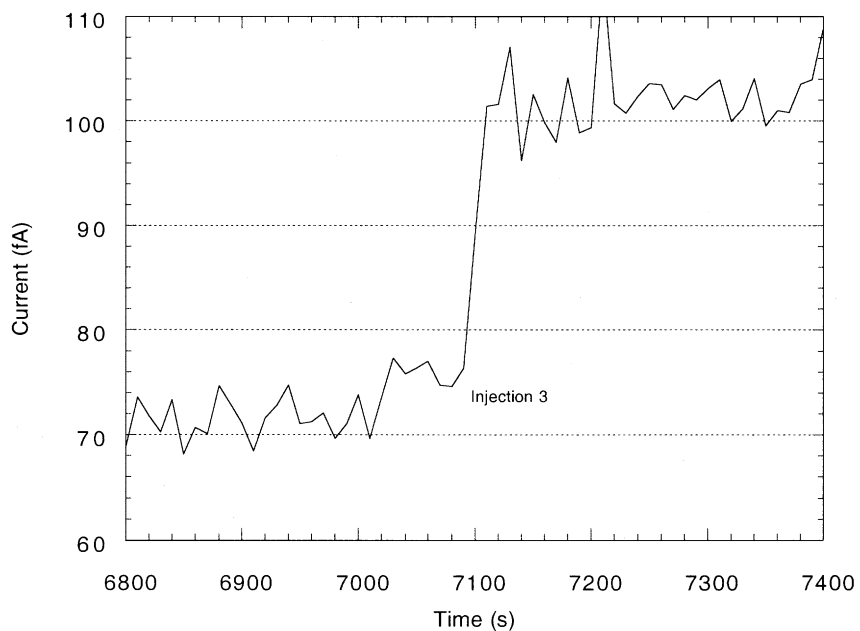


Fig. 6. An expanded view of injection 3 in test 3 illustrating the rapid time response of the tritium-in-water monitoring system.

the ratios shows that the ratio is usually less than unity, implying that the ionization chamber is underestimating the actual concentration of

tritium as measured by LSC. It is believed that this attenuation in current signal is likely due to the presence of some liquid water droplets in

Table 1

Test 1. Steady-state tritium-in-water activities as per the LSC measurement of the condensate and the tritium-in-water monitor, and the ratio of these activities^a

Injection	Activity in condensate as per LSC ($\mu\text{Ci/l}$)	Net ionization chamber tritium monitor signal (fA)	Activity in water as per tritium monitor ($\mu\text{Ci/l}$)	Ratio of activities: tritium monitor to LSC
1	5.5	10	21	3.8
2	14.0	35	73	5.2
3	50.0	35	73	1.5
4	22 420.0	11 745	24 550	1.1
5	27 120.0	14 055	29 380	1.1

^a Note: monitor background: -95 fA.

Table 2

Test 2. Steady-state tritium-in-water activities as per the LSC measurement of the condensate and the tritium-in-water monitor, and the ratio of these activities^a

Injection	Activity in condensate as per LSC ($\mu\text{Ci/l}$)	Net ionization chamber tritium monitor signal (fA)	Activity in water as per tritium monitor ($\mu\text{Ci/l}$)	Ratio of activities: tritium monitor to LSC
1	241	83	177	0.74
2	358	134	286	0.80
3	584	231	491	0.84
4	692	263	560	0.81
5	1554	557	1187	0.76

^a Note: monitor background: -25 fA.

Table 3

Test 3. Steady-state tritium-in-water activities as per the LSC measurement of the condensate and the tritium-in-water monitor, and the ratio of these activities^a

Injection	Activity in condensate as per LSC ($\mu\text{Ci/l}$)	Net ionization chamber tritium monitor signal (fA)	Activity in water as per Tritium Monitor ($\mu\text{Ci/l}$)	Ratio of activities: tritium monitor to LSC
none	37	20	44	1.17
1	130	52	111	0.86
2	192	76	161	0.84
3	250	106	226	0.90
4	300	121	257	0.86
5	333	138	293	0.86

^a Note: monitor background: -3 fA.

the water vapour stream flowing through the detector (see discussion in Section 2). It is expected that application of a good water vaporization system, which includes a gas vortex that “knocks-out” water droplets, will reduce or eliminate this effect.

5. Conclusions

An alternative approach to tritium-in-water monitoring is considered by changing the state of the water from the liquid phase to the gas phase. The fundamental implication of this change is that

the mean free path of the tritium-decay-beta in water vapour is of the order of millimetres, as opposed to micrometres in liquid water. The detection of tritium in water vapour can result in a much improved on-line tritium-in-water monitoring system. Various detection systems can be adapted for this purpose so long as the detection system is maintained at a sufficiently high temperature so as to maintain the tritiated water in the gas phase.

Application of a heated ionization chamber for tritium-in-water monitoring using a water boiler as a means of vaporization is investigated. Proof-of-principle experimental results demonstrate the viability of this method of detection of tritium-in-water to concentrations as low as $\sim 20 \mu\text{Ci/l}$ and time constants of less than 10 s. However, with present day improvements in current measuring circuitry, detection limits of less than a $\mu\text{Ci/l}$ are deemed achievable.

Acknowledgements

The assistance of Mr. Clive Morton and Mr. Nick Fragiadakis in carrying out the experiments is gratefully appreciated. Valuable technical discussions with Dr. Phil Lichtenberger and Dr. Bob Dyck are also acknowledged.

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