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TRitium Activity Measurements with a PhotomultipliEr in Liquids–The TRAMPEL experiment

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HIGHLIGHTS

- We have set up a new test device for measuring of tritiated water samples.
- The device is very compact and easy and reliable in operation.
- Easy integration in flow-through systems is possible.
- The device has been operated at Tritium Laboratory Karlsruhe for several months.
- The lower detection limit has been improved with regard to predecessors experiments.

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ABSTRACT

A common technique for the determination of the activity of tritiated water (HTO) is liquid scintillation counting (LSC). This implies sample taking, sample preparation and radioactive waste processing afterwards. When handling highly tritiated water special care has to be taken because of possible harmfulness of the sample. Furthermore, LSC devices are mostly large, heavy and expensive. The TRAMPEL experiment aims at measuring the activity of tritiated water in-line without sample taking. The device is intended to be easy to use and operate, quite inexpensive and compact. The measurement principle is based on electrons from β-decay which induce light in commercially available scintillation fibres. The light is detected by a small photomultiplier tube (PMT). A proof-of-principle was set up for static measurements using standard stainless steel parts. The complete device has a volume of less than 0.5 l.

1. Introduction

For the determination of the tritium content of a liquid sample, a very well established method in the laboratory environment is liquid scintillation counting (LSC). This method promises to have the lowest detection limits because scintillator and sample are mixed together for a high yield. Because tritium β-electrons have a very low mean free path in water (O(μm) [1]) this is more convenient than using solid scintillators with limited interaction surface. Drawbacks of the LSC method are sample taking (no in-line or real time measurements) and the production of waste.

Especially when handling highly tritiated waste water with $>10^8$ Bq/ml like in water detritiation systems for future fusion plants [2,3], LSC is not the perfect tool. A more convenient solution would be a device placed at any process critical point in-line for near to real-time process control. Another application is the monitoring of the tritium content in cooling water of heavy water fission reactors as reported in [4,5].

Systems for in-line measurement of tritiated water (HTO) based on solid scintillators have been reported in the past [4–8]. Despite these systems working well, they were not as simple, rigid, compact and inexpensive as the device described in the following.

2. Experimental setup

The TRAMPEL experiment (TRitium Activity Measurements with a PhotomultipliEr in Liquids) was designed right from the beginning as an inexpensive system with a small geometrical footprint. Only commercially available parts are used to decrease costs and speed up assembly. Fig. 1 visualises the principle and shows the ready-to-measure assembled cell.

Conversion of β-electrons to visible light is maintained by three halogen free scintillating fibres of type BCF20 made by Saint Gobain. The fibres have a 2 mm × 2 mm square cross section with a

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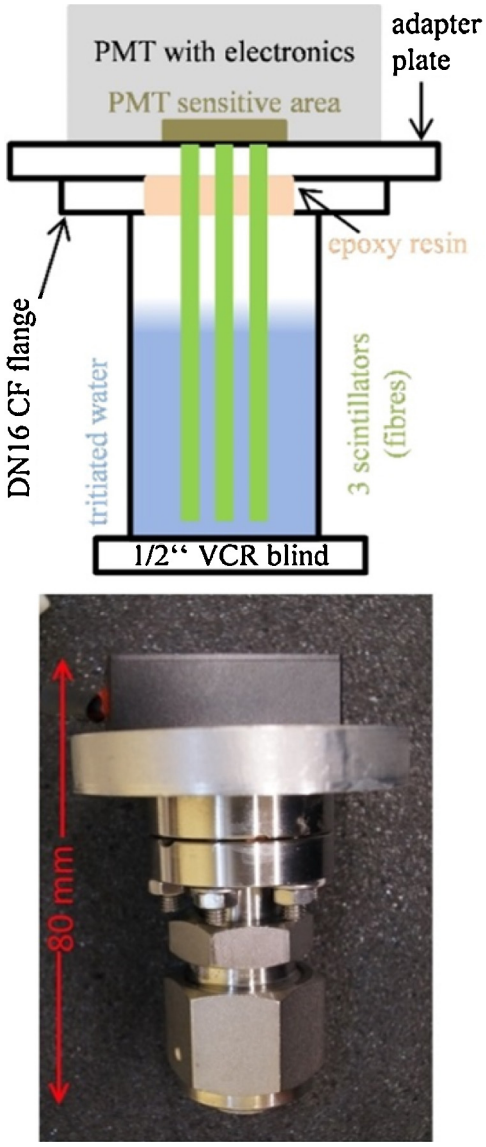

Fig. 1. Sample cell. The picture on top shows a principle sketch of the cell with three scintillating fibres (green) inside the stainless steel tubing. The lower picture shows the assembled cell with PMT on top and CF16 to 1/2″ VCR adapter with VCR blind flange. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

single clad made of acrylic and a core material made of polystyrene. The length of each fibre is 60 mm with 43 mm dipped into tritiated water (HTO). This leads to a surface in contact with HTO of 3.4 cm^2/fibre (“active surface”). The emission maximum of the fibres is reached at 492 nm.

A photo multiplier tube (Hamamatsu μPMT type H12402) with its maximum sensitivity at 420 nm is used for the detection of scintillation light. The benefit of using the μPMT is its small size and that it only needs a single 5 V operating voltage. The high voltage (500 V – 100 V) for the dynodes is generated internally by applying a control voltage ranging from 0.5 V to 1 V. All measurements presented here were made with a control voltage of 0.9 V (= 900 V PMT supply voltage). The control voltage is monitored all the time and has a deviation <0.5%.

TRAMPEL uses a standard CF16 to 1/2″ VCR adapter with CF/VCR blind flange as sample cell. The CF blind flange has a milled slit for gluing the fibres with epoxy resin (leak tested to $<10^{-9}$ mbar l/s (helium leak rate)). The whole assembly shown in Fig. 1 is enclosed by a CF63 pipe for light-tight operation of the PMT.

The current-signal-output is measured with a Keithley 617 electrometer amplifier (20 pA and 200 pA full scale ranges used). Its output signal (0–2 V) is digitalized by a Meilhaus USB scope type UM202 (16 bit sampling).

With the setup described there are as few losses as possible since the scintillation light can pass the sample cell straight to the PMT without any further window in between (no light loss due to reflections).

Table 1
Summary of measurements. The measurements >11 MBq/ml were performed with the 200 pA FS range of the Keithley 617 while all others were made with the 20 pA FS range. *Duration* gives the total time of each measurement and *N* the number of digitized values.

Activity (MBq/ml)	Signal (pA)	Variance (pA)	Duration (h)	N ($\cdot 10^7$)
Empty	0.29	0.16	68.38	24.6
0.0215	0.72	0.15	71.52	25.7
0.066	0.88	0.29	16.88	6.08
0.2	0.94	0.25	30.38	10.9
0.63	1.84	0.34	21.35	7.69
0.9	2.46	0.41	19.11	6.88
2	4.40	0.51	18.06	6.50
6.5	14.30	0.86	17.82	6.42
11	22.15	0.83	28.14	10.1
20	40.27	1.55	13.93	5.02
24	54.06	1.62	17.82	6.42

3. Experimental procedure and results

Table 1 gives a summary of the measurements presented in this paper. They cover a wide range in the specific activity from 0 (empty cell) to 24 MBq/ml. A typical single event from the PMT is shown in Fig. 2.

For a measurement, the sample cell is filled with 3.7 ml HTO, closed with a VCR blind flange and placed inside a CF63 pipe sealed with two blind flanges to shield the PMT from ambient light. The PMT signal is allowed to stabilize for several hours (3 h–15 h) and the output is recorded during this time, too. This allows for finding a region where the output is no longer influenced by a saturated photocathode from a recent exposition to ambient light (Fig. 3). A procedure of rinsing with demineralized water and drying after each measurement ensures that the next sample of HTO is not altered in its specific activity. Measurements with a LSC device have been made to check the specific activity of the samples regularly. There is a relatively large noise on the signal in all measurements despite using shielded cables (Fig. 2). This is considered as the main source of uncertainty causing a relatively large standard error while processing the data.

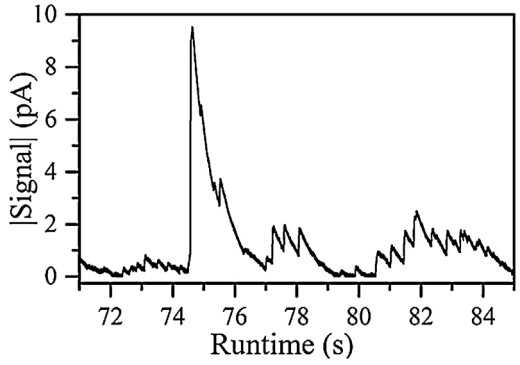

Fig. 2. Typical event signature from PMT. Shown is an event from the 0.0215 MBq/ml measurement with a sharp rise and a long exponential decay due to the amplifier used (input resistance too high for fast PMT signal).

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