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Development of a compact tritium activity monitor and first tritium measurements

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HIGHLIGHTS

- We report about experimental results of a new tritium activity monitoring system using the BIXS method.
- The system is compact and easy to implement. It has a small dead volume of about 28 cm^3 and can be used in a flow-through mode.
- Gold coated surfaces are used to improve significantly count rate stability of the system and to reduce stored inventory.

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ABSTRACT

To develop a convenient tool for in-line tritium gas monitoring, the TRitium Activity Chamber Experiment (TRACE) was built and commissioned at the Tritium Laboratory Karlsruhe (TLK). The detection system is based on beta-induced X-ray spectrometry (BIXS), which observes the bremsstrahlung X-rays generated by tritium decay electrons in a gold layer. The setup features a measuring chamber with a gold-coated beryllium window and a silicon drift detector. Such a detection system can be used for accountancy and process control in tritium processing facilities like the Karlsruhe Tritium Neutrino Experiment (KATRIN). First characterization measurements with tritium were performed. The system demonstrates a linear response between tritium partial pressure and the integral count rate in a pressure range of 1 Pa up to 60 Pa. Within 100 s measurement time the lower detection limit for tritium is $(143.63 \pm 5.06) \cdot 10^4$ Bq. The system stability of TRACE is limited by a linear decrease of integral count rate of 0.041 %/h. This decrease is most probably due to exchange interactions between tritium and the stainless steel walls. By reducing the interaction surface with stainless steel, the decrease of the integral count rate was reduced to 0.008 %/h. Based on the first results shown in this paper it can be concluded that TRACE is a promising complement to existing tritium monitoring tools.

1. Introduction

In various applications for tritium processing such as KATRIN [1] or future fusion power plants [2], in-line activity monitoring of a gaseous tritium source is indispensable for process control and accountancy. A precise determination of the stored/circulating inventory in a wide pressure and concentration range in various gas mixtures is vital for processes like supply, recovery, storage and waste treatment. To meet these requirements, beta-induced X-ray spectrometry (BIXS) is a promising measurement technique [3]. In contrast to other detection methods like ionization chambers [4], LARA [5] or mass spectrometry [6], BIXS is only sensitive to the activity content of a sample and not influenced by the sampling gas mixture as long as the pressure is low enough to avoid significant self-absorption of β-electrons in the gas sample. Moreover, a BIXS monitoring tool can be designed as a compact device that offers convenient integration in any tritium processing facility. Based on this, the TRitium Activity Chamber Experiment (TRACE) was set up at the Tritium Laboratory Karlsruhe (TLK) to develop a compact and robust BIXS device.

Previous work with gaseous tritium monitoring include experiments utilizing NaI(Tl)-scintillators and proportional counters in various configurations [4,7–9]. In TRACE a low-noise silicon drift detector (SDD) is used which is able to detect especially low-energy X-rays of a tritium BIXS-spectrum. Combined with the small geometrical footprint of such a detector the BIXS-system can be built very compact while having a rather high detection efficiency. Compared to a NaI(Tl)-scintillator the energy resolution of a SDD is about one order of magnitude better. This makes it possible to detect electronic drifts of the system by energy shifts

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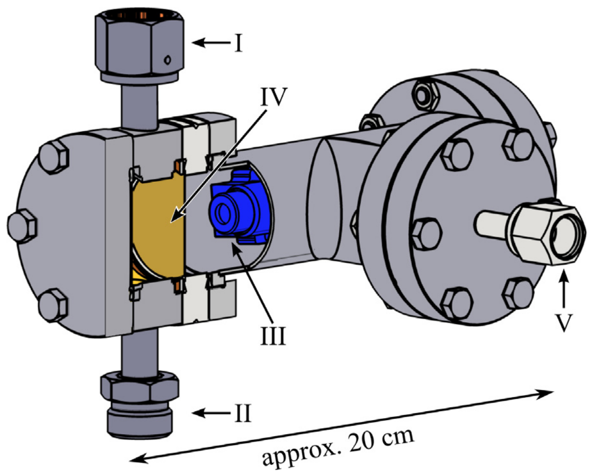

Fig. 1. CAD drawing of the measuring cell. The measurement cell is based on DN40CF parts. The flow-through sample volume is about 28 cm^3 and is gold coated to increase the detection efficiency and reduce the memory effect (I: Gas inlet, II: Gas outlet, III: SDD-capsule, IV: Au-coated Be window, V: Vacuum interface for evacuation of the detector electronics vessel).

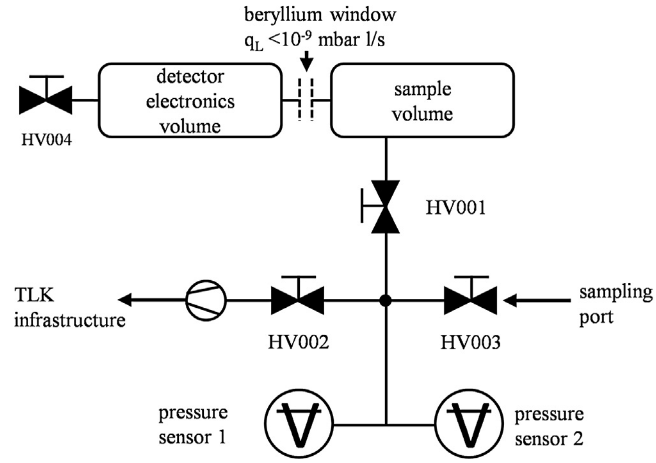

Fig. 2. Process diagram of the experimental setup for the characterization of TRACE. Manual valves are denoted with HV001-HV004.

of characteristic X-rays in the spectrum and correct the count rate accordingly. Furthermore, a SDD is easier to operate and significantly more rigid against external magnetic fields, vibrations and temperature variations which is especially important for the use with KATRIN. Based on previous experiments, TRACE is using a gold-coated beryllium window for X-ray generation and transmission [8,10].

The main work presented in this paper focuses on the construction of a compact BIXS activity monitor and first measurements with the TRACE experiment. In addition the sensitivity, stability, detection efficiency and minimum detectable activity (MDA) in the range of 1 Pa up to 60 Pa of TRACE is evaluated.

2. Experimental description

The concept of the TRACE experiment is strongly influenced by the experience which was made with a predecessor BIXS experiment [10] and the requirements for a monitoring tool for systems with high tritium throughputs as KATRIN [1] at TLK. The design criteria are:

1 inline measurement without waste production,
2 small dead volume to minimize stored inventory,
3 robust and easy to implement and
4 compact design.

To achieve costs competitive to other established methods and to make it easily serviceable, the system is mainly made of standard components.

2.1. Experimental setup

A schematic of the experimental setup is shown in Fig. 1. The sample volume consists of a DN40 Conflat® (CF) distance flange with two 1/2″ Swagelok® VCR® ports. One side is covered by a blind flange and the other side is connected to a beryllium window with a thickness of 125 μm and a diameter of 28.5 mm. A beryllium window is used to minimize X-ray absorption due to the low atomic number (Z = 4). For the same reason the thickness should be as low as possible while being able to withstand differential pressures of up to 10^5 Pa.

All the inner walls in the process chamber are sputter coated with 200 nm of gold except for the beryllium window which is sputter coated with 100 nm of gold. A titanium layer of 10 nm is used as an adhesion agent between the gold coating and the stainless steel/beryllium. Gold is chosen as an inner coating due to the high atomic number (Z = 79) which increases bremsstrahlung production during electron absorption. Furthermore, the memory effect is expected to be reduced [12]. The gold coating of the beryllium window of 100 nm is according to Monte Carlo simulations the optimal thickness. For a higher thickness, X-ray absorption in the gold coating would reduce the detector count rate and therefore the overall detection efficiency. For a lower gold thickness, the probability for beta electrons to traverse the gold coating without producing bremsstrahlung and being absorbed in the beryllium substrate is increased.

Since the beryllium window is specified for differential pressures of 10^5 Pa but only unidirectional loadable, the detector electronics volume (T-piece in Fig. 1) must be evacuated and the maximum process gas pressure is limited to 10^5 Pa. The detector volume houses an Amptek® OEM SDD with an active area of 25 mm. It has one 1/2″ Swagelok® VCR® port for evacuation. On the backside of the T-piece there is an DN40CF blind flange with electrical feed-throughs for the detector. Internally attached to this blind flange is a copper holder which fixes the position of the detector. The SDD is temperature stabilized and cooled to ≈−70 °C by Peltier elements, heat losses are transferred via the copper holder which also serves as a heat spreader. In case of a break of the beryllium window, the detector electronics housing serves as a first wall for tritiated gases and therefore fully complies with all rules and regulations applicable at TLK. Detector signals are acquired and processed by an Amptek® DP5 OEM digital pulse processor and MCA, which is placed outside of the detector volume.

2.2. Measurement procedure

In Fig. 2 a process flow diagram of the experimental setup is shown. Tritium gas mixtures are supplied by the CAPER facility [11] in a sample cylinder with a volume of 10 cm^3. The purity of these gas mixtures is analyzed by gas chromatography [11]. Sample cylinders are connected to the sample port (see Fig. 2) and for static tritium measurements the test gas mixture is expanded into the TRACE sample volume. Variations of the final pressure can be achieved by controlled expansion with the manual valve (HV001) or a step-wise pump-down of the system.