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Investigations of the applicability of a new accountancy tool in a closed tritium loop

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HIGHLIGHTS

- We have set up a new test device for measuring of tritiated gas samples.
- The device is very compact and easy and reliable in operation.
- Easy integration in flow-through systems.
- 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 commonly used activity monitoring method for tritium accountancy and process monitoring in tritium technology is ionization counting. Despite the wide use of ionization chambers (IC), they have several drawbacks like a strong gas species and pressure dependency. Furthermore, if compact systems are needed, there is also the necessity for process gas pressures >10 kPa. To encounter these drawbacks, the TRitium Activity Chamber Experiment (TRACE) has been developed at the Tritium Laboratory Karlsruhe (TLK) as a compact tritium monitor based on the beta induced X-ray spectrometry (BIXS) principle.

TRACE can be used as an accountancy tool in tritium-processing facilities like the KArlsruhe TRItium Neutrino (KATRIN) experiment. In contrast to ICs TRACE shows a linear response to pressure changes up to approx. 1 kPa. The results of performed flow-through measurements confirm that TRACE is a complement for ICs in the low-pressure regime. Furthermore the gas species dependency of TRACE is investigated both with tritium measurements and with Monte Carlo simulations.

1. Introduction

For process monitoring and accountancy of gaseous tritium sources in the KArlsruhe TRItium Neutrino (KATRIN) experiment [1] or in future fusion plants like ITER [2] various tools are available. Commonly used tools in the Tritium Laboratory Karlsruhe (TLK) are calorimeters [3], mass spectrometers [4] and ICs [5]. Since KATRIN and future fusion plants have unprecedented requirements on tritium analytics tools, alternative methods are part of the R&D task of the TLK [6]. Typical requirements are in-line and real-time measurement of tritiated gases in a wide pressure (1 Pa–1 kPa) and concentration (1 ppm – 95%) range with high precision and accuracy. Furthermore, the used tools should be designed as compact as possible. None of the enumerated tools fulfills all the requirements. For the measurement with calorimeters it is necessary to take samples, with mass spectrometers the compact construction is not possible due to the demand of low pressures (lower than 0.1 Pa, requires separate pumping unit) in the measurement volume. ICs need pressures higher than 10 kPa when designed compact. Also, there is a lack of commercially available tritium compatible ICs, in which only metal or ceramic surfaces are exposed to the process gas. To encounter these drawbacks a monitoring tool, based on beta induced X-ray spectrometry (BIXS), is developed at TLK [7]. The TRitium Activity Chamber Experiment (TRACE) has been designed as a compact tritium accountancy and process monitoring tool, which fills the analytical gap in a pressure range below 10 kPa.

The focus of this paper is set on the response of the system to activity changes in flow-through mode and on the comparison with an IC to [5]. Additionally, the results of the measurements with TRACE are compared with performed Monte Carlo (MC) simulations.

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2. Experimental description

2.1. Experimental setup

TRACE uses as detection principle BIXS, which is based on the measurement of X-rays produced by absorption processes of tritium beta electrons in a dense medium [8].

A schematic overview of the TRACE setup and an illustration of the measurement principle is shown in Fig. 1.

The setup of TRACE consists of a sample volume which contains the tritiated gas mixture and a volume which hosts a silicon drift detector (SDD) for the detection of the produced X-rays. The two chambers are separated by a beryllium window with a thickness of 125 μm. To optimize the detection efficiency of and to reduce the X-ray absorption in the system, the beryllium window is sputter-coated with 100 nm of gold. The inner walls of the sample volume are sputter-coated with a gold layer of a thickness of 200 nm to increase the bremsstrahlung production in and to reduce the memory effect of the system [9]. With two 1/2″ VCR ports the sample volume can directly be integrated in process gas streams. A more detailed description and further information about the setup and the working principle of TRACE can be found in [7]. In [7] also a comparison to previous work with the BIXS method is given.

2.2. Measurement procedure

Currently TRACE is integrated in the TriToP experiment [10], which is used to circulate tritiated gas streams through the sample volume. In Fig. 2 the process flow diagram of TriToP is shown. TriToP features an IC for activity measurements and a mass spectrometer for gas analysis. In the current installation location it is possible to achieve pressures between approx. 1 Pa and 1 kPa in the sample volume of TRACE.

Tritiated gas mixtures are provided by the CAPER facility [6] in a sample cylinder with a volume of 50 cm^3. The purity of the provided gas mixtures is analyzed either by gas chromatography [6] or with the mass spectrometer of TriToP. The sample cylinder is connected to the sampling port of TriToP and the gas is transfered into BV001 (see Fig. 2). During measurements the scroll pump circulates the tritiated gas sample through the complete setup. Variations of the pressure in the TRACE sample volume are achieved by different set points of a flow controller and by partly closing the manual valve HV007.

Each tritium measurement campaign starts and ends with background measurements. For the background measurements the sample volume of TRACE is evacuated to a total pressure of ≤5 Pa. After the background measurements the pressure in the sample volume of TRACE is varied between 7 Pa and 0.8 kPa and tritium measurements are performed. During gas analyses the TMP of the setup achieved pressures ≤6 mPa for the operation of the mass spectrometer.

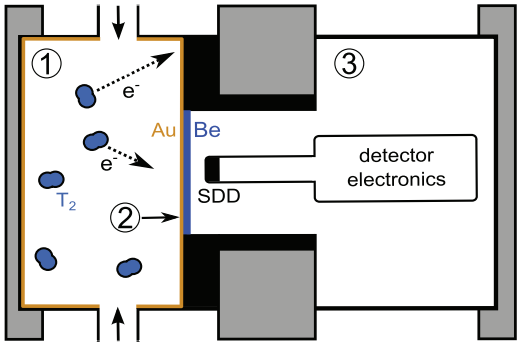

Fig. 1. Schematic of the TRACE setup (not to scale). TRACE consists of two volumes, which are separated from each other through a gold-coated beryllium window (2). The sample volume (1) contains the tritiated gas sample, the detector volume (2) hosts a SDD.

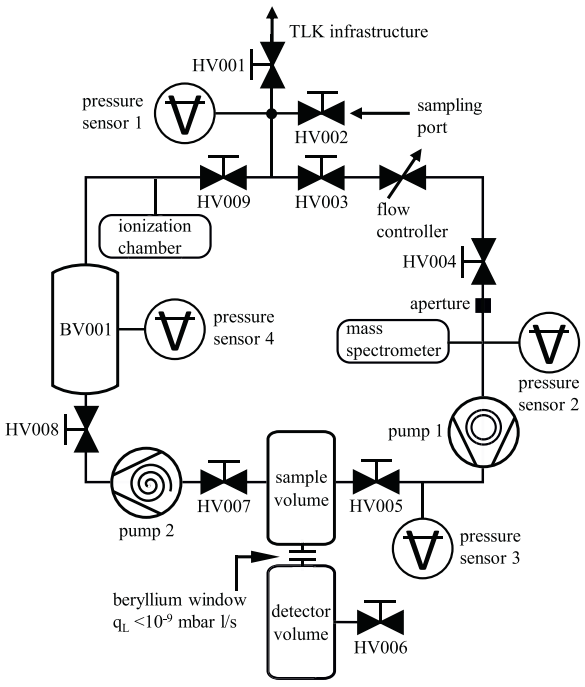

Fig. 2. Process diagram of the experimental setup for flow-through measurements. The manual valves are termed as HV001–HV009. Pump 1 is a TMP, pump 2 a scroll pump. The buffer vessel is denoted as BV001.

3. Experimental results

3.1. Flow-through activity monitoring of tritiated gases

Figs. 3 and 4 show the results of the measurements in flow-through mode with different pressures in the TRACE sample volume. There is a linear relationship between the integral count rate and the total pressure in the sample volume of TRACE in an area of 7 Pa–0.8 kPa. At pressures higher than 1 kPa variations from the linear response are expected due to self-absorption effects in

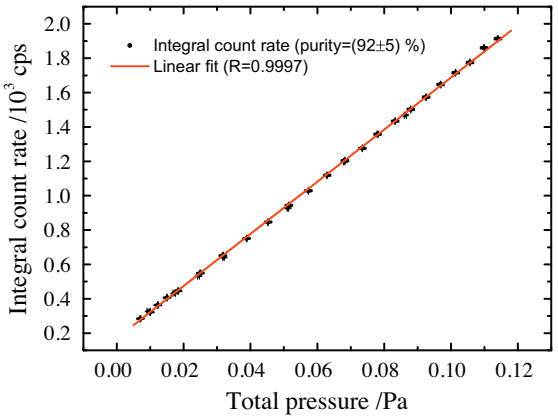

Fig. 3. Integral count rate over total pressure in flow-through mode. The total pressure in the sample volume of TRACE was varied between 7 Pa and 0.1 kPa in order to determine the linearity of the system in a low-pressure range. The purity of the gas mixture was (92 ± 5)% (measured with gas chromatography) and the measurement time for each data point was at least 100 s. The error bars indicate 1σ statistical uncertainties. The line of the best fit is $y = (15.17 \pm 0.01) \cdot x + (170.74 \pm 2.33)$.

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