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# Toroidal proportional gas flow counter for conversion X-ray Mössbauer spectroscopy

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ABSTRACT

This paper presents a new prototype of a toroidal proportional gas flow counter. This detector has been developed to be used in the Mössbauer spectroscopy for characterizing the surfaces of the iron bearing samples. The presented proportional counter allows to register both conversion X-rays (6.4 keV) and the backscattered gamma rays (14.4 keV) in the almost 2π geometry with the sufficient efficiency. This paper demonstrates the structure description of the detector and the optimization process of its use in the backscattering Mössbauer spectroscopy. The energy resolution of the detector is presented via the series of the multichannel analyses with different filters, where both energy peaks (6.4 keV and 14.4 keV) were observed. The measured Mössbauer spectra confirm the regularity of the energy assignment. The utilization of the aluminum filter enables to achieve a higher resonance effect, whereas the backscattered Mössbauer spectra were measured. The detector could be also used to measure in the transmission geometry with high efficiency.

## 1. Introduction

Mössbauer techniques are powerful tools in the material research, because of their ability to bring reliable information about the valence and spin states, phase composition, magnetic structure of materials etc. The typical transmission geometry provides the transmission Mössbauer spectroscopy (TMS) to analyze samples only in the form of the very thin layers or powders. On the other hand the backscattering geometry could be also used to analyze the surface of the bulk materials. The backscattering Mössbauer spectroscopy (BMS) covers a wide spectrum of different techniques, while only three the most used of them will be further discussed in this paper:

1) Conversion X-rays Mössbauer spectroscopy,
2) Backscattering gamma rays Mössbauer spectroscopy,
3) Conversion electron Mössbauer spectroscopy.

The conversion X-rays Mössbauer spectroscopy (CXMS) analyses the surface up to the depth 1–20 µm and the backscattering gamma rays Mössbauer spectroscopy (BGMS) up to 20–100 µm [1,2]. In general, scintillation detectors, proportional gas counters, and semiconductor detectors are the most frequently applied in the Mössbauer spectroscopy.

Since the proportional gas counters provide a good energy resolution, their utilization is very common in the area of the backscattering Mössbauer spectroscopy. They are widely used for detection of the conversion electrons (the conversion electron Mössbauer spectroscopy – CEMS) emitted from different shells of the atoms (K, L and M) on the surface of the sample. The penetration depth is approximately 300 nm for the K-shell [2]. Hence the electrons have a short mean free path in the air, the location of the samples is usually inside the detector [3]. In addition, a combination of two proportional gas counters allows the detection of the conversion electrons and the conversion X-rays at the same time [4]. The cylindrical geometry is usually used for the proportional gas counters, where the cathode is the body of the detector. The anode (typically a very thin tungsten wire below 100 µm in diameter) is mounted in the middle of the cylinder. This construction enables to achieve the high energy resolution. Another option is to use a toroidal shape, which enshrines the features of the cylinder. Moreover it allows to register emitted radiation from the studied surface almost in the 2π geometry. The anodes of the toroidal detectors presented in [5–8] are not located in the center of the detector, which is given by their shapes. They are not in the forms of circles, but in the forms of polygons. There are different choices of the filling gas for each type of the ionizing radiation detection. Despite the fact that the noble gases are very common for filling the proportional counters, they are usually

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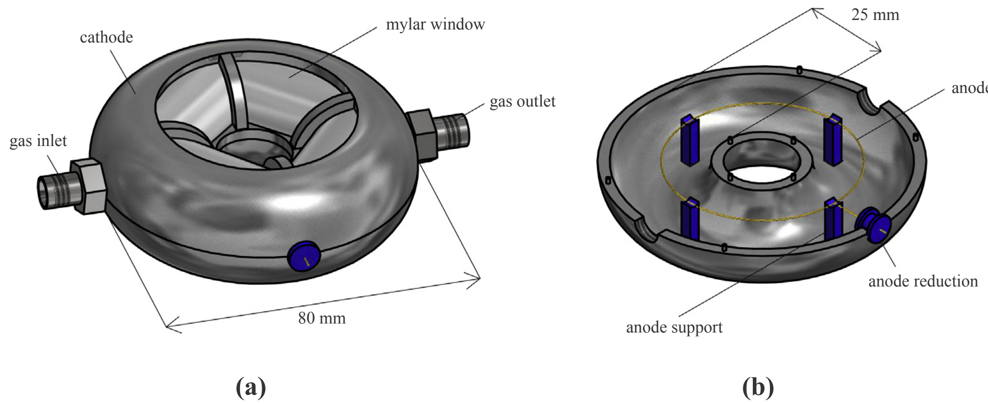


**Fig. 1.** (a) Layout of the proportional gas flow counter and (b) the anode configuration inside the counter.

used in mixtures with other more complex gases. The reason is that they do not offer large gains without entering into a permanent discharge regime. The quenching gases (like methane, propane, isobutane or carbon dioxide) are mostly added to the pure noble gas to transfer the charge from the noble molecule to the complex molecules. The most common ratio is 10% of the quenching gas to 90% of the noble gas [9].

The purpose of this paper is to present a new toroidal proportional gas flow counter, which is able to register soft X-rays (6.4 keV) and the low-energy gamma rays (14.4 keV) utilized in the $^{57}$Fe Mössbauer spectroscopy.

## 2. Design of the detector

The presented detector (see Fig. 1(a)) was constructed from the stainless steel EN 1.4404 (AISI 316L) utilizing the 3D printing technology (LaserCUSING, layer thickness 30–45 µm). The detector surface polishing was not required, because the printed inner surface was smooth enough as-made (except the created support for reaching this non-standard shape, which was removed by the sandblasting method). As the detector was primarily designed for bulk sample surface analysis, the outer diameter of the detector was 80 mm. The cathode (the ground state) was the shell of the detector and had 25 mm diameter for reaching optimal cathode and anode radius ratio. This parameter affects the gas multiplication factor of the detector [10]. The anode formed a circle (gold plated tungsten wire with 50 µm diameter) and was supported by 4 ABS plastic columns located in the center of the detector. Despite being a very fine wire, the anode was able to keep the circle shape very well. However, an attempt with a thinner wire (10 µm diameter) proved that at least 50 µm one was required. The anode was carried out through ABS plastic reduction from the detector and soldered to the coaxial cable with SHV connector.

There are 4 curved aluminized mylar windows (see Fig. 2, bending mylar did not cause any wrinkles), which allow the X-rays or gamma rays penetrate into the detector. These windows were sticked using cyanoacrylate and sealed with epoxy resin (TS-10 Epoxy Vacuum, THORLABS). There is a gas inlet and outlet to fill the detector and for continuous exchange of the mixture of the working gas. The body of the detector was compounded from two parts and was sealed with the same epoxy resin as the windows.

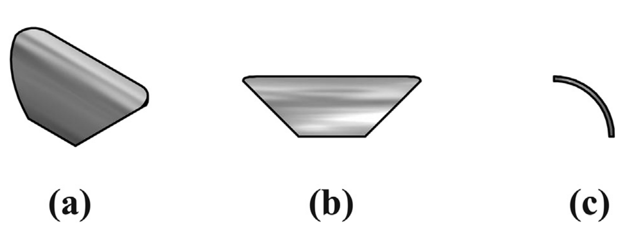


**Fig. 2.** Specific shape of mylar window: (a) layout, (b) front view and (c) side view.

## 3. Experimental setup

The proposed experimental setup (see Fig. 3) was designed to determinate the features of the detector, where the gas management with the precise signal processing and data acquisition were provided. The detector was supplied by the programmable high voltage supply HP2.5PAA025 (Applied Kilovolts). The current pulses with low amplitudes from the detector were converted to the voltage signal with a high signal to noise ratio by the 142PC preamplifier (ORTEC). The voltage signal was amplified by the 572A amplifier (ORTEC) with the shaping time of the pulses 1 µs. The fast high-resolution digitizer NI PCI-5124 (National Instruments) was used to record data and the LabVIEW™-based user application [11] to process the data. Corresponding to the shaping time of the pulses the sampling rate of the detector signal was set to 2 MSa/s (MegaSamples per Second) with the vertical range of the input channel ± 0.5 V. The gas mixture was prepared by the gas mixer KM 20-200-3ME (WITT). In this study a mixture of 90% of argon and 10% methane was used. The gas pressure inside the detector was approximately 0.1 MPa.

In the Mössbauer spectrometer the transducer and the analog PID regulator [12] were utilized in the setup to register Mössbauer spectra. The velocity signal and the synchronization signal for the digitizer were generated by the function generator 33521A (Agilent). The detailed layout of the detection part is shown in Fig. 4.

## 4. Results and discussion

### 4.1. Energy resolution

Crucial feature of the ionization detector is its energy resolution, because the soft X-rays (6.4 keV) and low-energy gamma rays (14.4 keV) have to be distinguished for the purpose of the $^{57}$Fe Mössbauer spectroscopy. The detector energy resolution was presented with the help of several multichannel analyses, where relevant energies were filtered or not. It was verified that 1400 V is the optimal value of the high voltage for the detector performance.

The multichannel analysis spectra in both geometries were measured:

1) Without filters – all the ionization radiation was detected.

2) With the aluminum filter – the aluminum filter shields the soft X-rays. The thickness of the filter was approximately 40 µm.

3) With the copper filter – the copper filter shields the soft X-rays as well as the low-energy gamma rays. The thickness of the filter was approximately 40 µm.

The multichannel analysis spectra in the transmission geometry (see