Internal Quality Control of the HPGe spectrometer stability

Aleksandra Samolov, Aleksandar Kandić, Igor Čeliković, Mirjana Đurašević and Tamara Milanović

***Abstract*— Ensuring confidence in the results of laboratory measurements is an essential assignment regarding quality control. Therefore, taking actions in obtaining valid results is crucial for such a task. In this paper we present the results of the internal quality control of the HPGe detector system. The results were obtained during 2022, in the Laboratory for Nuclear and Plasma Physics of the "Vinca" Institute for Nuclear Sciences, Belgrade, Serbia. Given results show proper use of the detector system and prove the validity of the results.**

***Index Terms*— gamma spectrometry, X-chart, quality control.**

I. Introduction

Concentrations of natural as well as anthropogenic radionuclides vary substantially at different locations and for different types of materials. Under certain conditions environmental samples can reach hazardous radiological levels and hence should be monitored. It is of great importance to provide accurate and precise analysis results of the environmental sample analysis as it is further used for the decision making [1]. In order to achieve so, quality control (QC) and quality assurance (QA) program are important to be established.

Quality control of a laboratory process has taken a significant place in daily laboratory practice, since a few decades ago. Demands for assuring valid results are strict and are given in a number of standards and guidelines [2, 3].

In order to ensure reliability of the results and to minimize number of measurements that should be rejected, QA program has to be defined and according to ISO/IEC/EN 17025 it should include different steps such as: proper use of certified reference materials, organisation and performing periodical internal laboratory quality control, taking part in Proficiency Testing Schemes, repeating testing using the same or different methods, repeating the examination of stored samples and making correlation of results for different sample characteristics [3].

Such QA/QC program is implemented in the Radionuclide Metrology Group of the Laboratory for Nuclear and Plasma Physics, "Vinča" Institute of Nuclear Sciences, Belgrade, Serbia.

Radionuclide Metrology Group on daily bases, among else, deals with gamma spectrometric measurement of radionuclide content in environmental samples. In accordance with national request and international recommendations, TCS No. 24, Quality System Implementation for Nuclear Analytical Techniques, permanent internal quality control of semiconductor HPGe spectrometer has been conducted. Internal control includes periodic checking of the characteristics of this device. The paper presents the control charts of the examined characteristics for the year 2022, based on which the long-term stability of the spectrometer was analyzed.

II. Methods

In order to maintain control over one testing method, every laboratory needs to plan the protocol for quality control. After that these results have to be recorded and analyzed so that misconduct of the examination process is spotted. Furthermore, ISO/IEC/EN 17025 demands that ensuring confidence in the obtained results has to be done in such a way that trends can be easily observed. One of the most useful ways of presenting quality control results is plotting so called control charts. Control charts are plotted using data obtained by certified reference material measurements. There are two basic types of control charts, X and R chart.

The simplest type of control chart is the Shewhart chart, which is the X type of the chart. Measured values are plotted on the y-axis, and time of consecutive measurements on the x-axis (ie daily, weekly, etc.). This type of chart can track record of the data on a daily basis, but if the measurement system is stabile enough a week period of time is sufficient [2,4-8].

As stated above, the Shewhart chart was used for the analyses. The mean value is used as a target value, while the standard deviation is used to set boundaries. The warning limits were set at ±2 standard deviations, while the action limits were set to ±3 standard deviations. Requirements are defined in such a way that when the measurement result is

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outside the warning limits, careful analysis is required, and if it is outside of the action limits, it is considered that there is a potential error. A signal that usually indicates a problem occurs in the following cases:

- two consecutive measured values are outside the limits of the action,
- four consecutive measured values are outside the warning limits,
- ten consecutive measured values are on the same side of the mean value and
- an upward or downward trend for several consecutive measured values are observed [2].

Internal quality control is carried out by gamma spectrometry system consisting of a HPGe detector (GEM30-70 Ortec, with relative efficiency of 37 %, resolution 1.66 keV and peak-to-Compton ratio of 70:1 at 1332.492 keV ^{60}Co) with standard supporting electronics.

Characteristics of the HPGe detector that were periodically checked were:

- counting of the background, which determines the possible contamination of the detector system,
- net peak area, which checks the stability of the measuring system
- the position of the peak, which additionally checks the stability of the electronics in spectrometer system and the influence of the environment (humidity, temperature) on measurement
- full width at half maximum (FWHM), which evaluates the quality system resolution. An increase in resolution may be caused by the increased electronic noise of the spectrometer, or may indicate vacuum problems in the system,
- full width at tenth maximum (FWTM), can show possible detection damage of the detector crystal, poor p/z ratio and the presence of current leaks,
- FWHM/FWTM ratio for different energies of gamma radiation, which monitors the quality of the spectrometer itself, [2].

Point sources of ^{60}Co ($T_{1/2}$ = 5.271 years; 1173.228 keV and 1332.492 keV; 11.87 kBq activity at reference date: 27th November 2009) and ^{137}Cs ($T_{1/2}$ = 30.05 years; 661.657 keV; 41.81 kBq activity at reference date: 25th November 1977) were used as control sources for checking peak positions, FWHMs, FWTMs and FWHM/FWTM ratios.

For checking the net peak area of ^{137}Cs, a decay correction was included. Measurements duration were 1000 s and corresponding spectra were recorded and analyzed using Canberra's Genie 2000 software [9].

III. Results and Discussion

In the period from January to December 2022, 52 measurements were carried out and these results are presented in the Fig. 1-10.

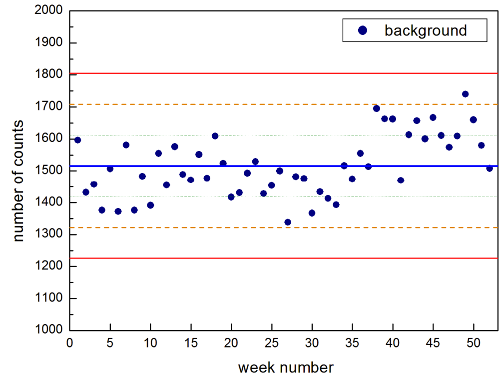

Fig. 1. Control chart of background measurements

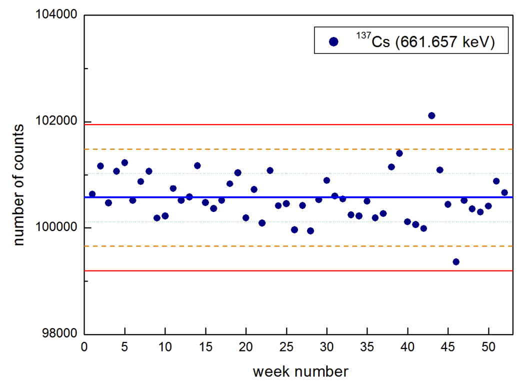

Fig. 2. Control chart of net peak area for ^{137}Cs

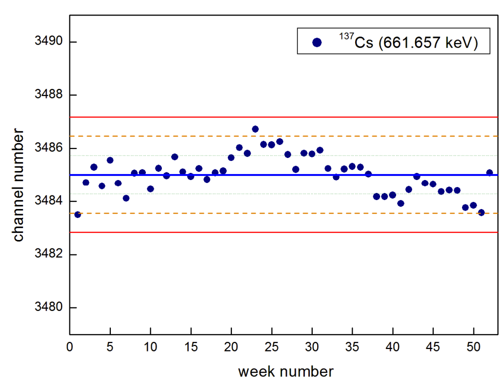

Fig. 3. Control chart of the peak position for ^{137}Cs

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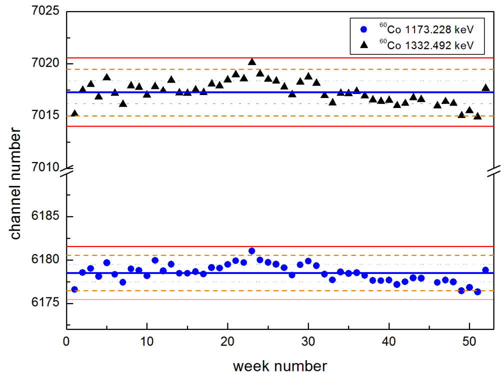

Fig. 4. Control chart of peaks position for ^{60}Co

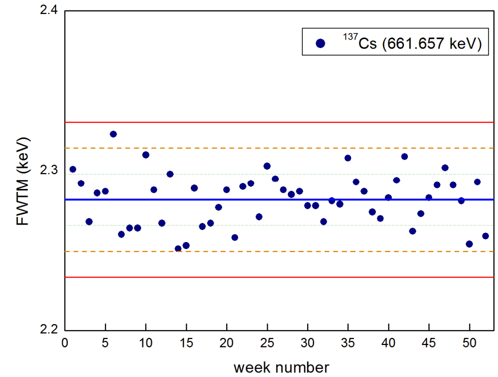

Fig. 7 Control chart of ^{137}Cs FWTM measurements

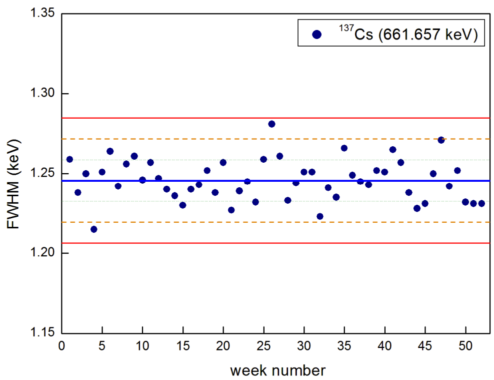

Fig. 5 Control chart of ^{137}Cs FWHM measurements

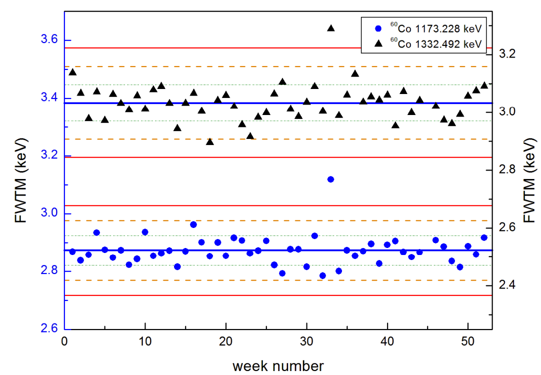

Fig. 8 Control chart of ^{60}Co FWTM measurements

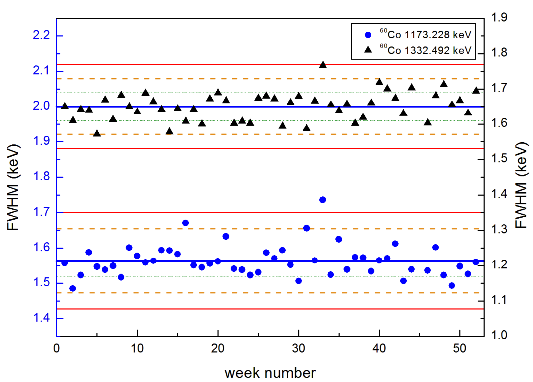

Fig. 6 Control chart of ^{60}Co FWHM measurements

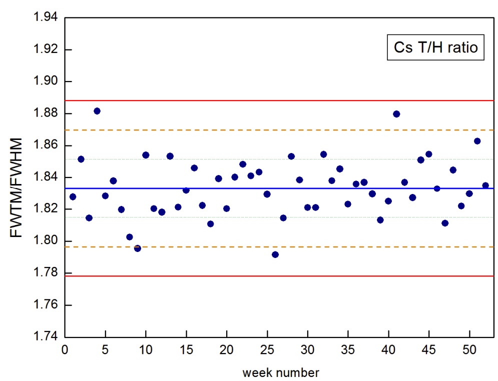

Fig. 9 Control chart of ^{137}Cs FWTM/FWHM ratio

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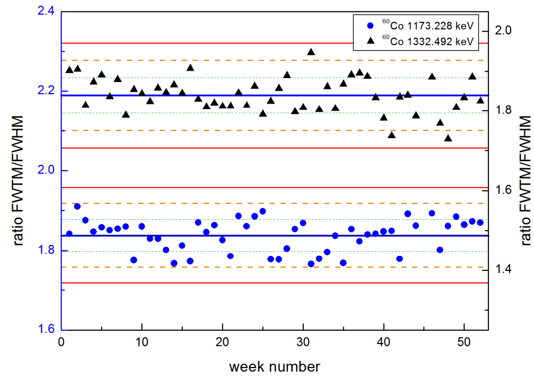

Fig. 10 Control chart of ^{60}Co FWTM/FWHM ratio

Analysis of the presented charts is performed according to the requirements given in [2] as described in Section II.

Control charts show that most of the measured points are within the allowed range and distributed around the mean values. One measurement point value of FWHM for ^{60}Co exceeded the action limit as well as one measurement point of net peak area for ^{137}Cs. Since there were no two consecutive measured values outside the limits of the action, there was no need for correction measures.

Four consecutive measured values outside the warning limits were not observed.

Measured number of counts in background were on the same side of the mean value for more than ten consecutive measured values. This was explained as natural variation of background due to variation of radon and its progeny.

Peak positions for both ^{60}Co and ^{137}Cs transitions varied within 2 channel numbers only. Therefore, observed trend of more than ten consecutive measured values that are on the same side of the mean values could be neglected.

IV. CONCLUSION

As seen from this paper internal quality control includes periodic checking of the device's measuring characteristics. Based on the displayed control charts, it can be concluded that the work of the spectrometer system is stable and that the results obtained can be considered reliable, i.e. the work of this laboratory is in accordance with international standards and recommendations.

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