The 56th International October Conference on Mining and Metallurgy
22-25 October 2025, Bor Lake, Bor, Serbia
www.ioc.tfbor.bg.ac.rs

DOI: 10.5937/IOC25593D *Original scientific paper*

INTERNAL QUALITY CONTROL OF ICP-MS METHOD FOR WATER ANALYSIS

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Abstract
Reliability of analytical results in determining the content of metals in water samples depends not only on the instrument performance, but also on the implementation of the internal quality control measures. The confirmation of results validity is one of the fundamental principles of the ISO/IEC 17025 standard and represents a key requirement for the accredited laboratories. The aim of this study is to demonstrate the application of certified reference material (CRM) in combination with X- control charts to assess the stability and accuracy of the ICP-MS method. The obtained results showed that the values for As, Cd, and Mn were within the specified limits, thereby confirming the stability of the ICP-MS method. It was concluded that integrating CRMs with graphical tools for monitoring results represents an efficient and practical approach to ensure the reliability of ICP-MS analyses, and that internal quality control is of essential importance when analyzing environmental samples.

***Keywords**: Control Charts, CRM, ICP-MS*

1. INTRODUCTION

The Chemical Testing Laboratory of Mining and Metallurgy Institute in Bor has a tradition of more than half a century and holds a significant position in the field of analytical chemistry. In addition to the analysis of geological samples and finished metallurgical products, an important segment of its work today also involves the testing of environmental samples.

The reliability of these analytical results is particularly important for environmental monitoring. As an accredited laboratory, the Institute is obliged to meet the requirements of ISO/IEC 17025, among which is ensuring the validity of results. This is carried out through internal quality control, which includes the use of certified reference materials (CRMs), blanks, and standard solutions [1].

One of the most powerful tools for statistical process control is the control chart. Its application enables simple and efficient monitoring of result stability, provides a clear visual overview of process status, and facilitates the timely detection of deviations. The basic elements of a control chart are the center line and the lower and upper warning and action limits, whose application indicates when a process correction is required or when the effects of implemented measures should be confirmed. In this way, the continuous validity of ICP-MS analyses in routine work is ensured [2,3].

The aim of this paper is to present the application of certified reference materials (CRMs), in conjunction with X-control charts, to assess the stability and accuracy of the ICP-MS method for

the analysis of water samples, specifically for the determination of arsenic (As), cadmium (Cd), and manganese (Mn).

2. EXPERIMENTAL

In addition to routine water samples, the certified reference material *WP Trace Metals VHG-QWPTM-15* (VHG Labs, USA) was analyzed. The CRM was prepared according to the manufacturer's instructions, with a 10-fold dilution using ultrapure water. The certified values for As, Cd, and Mn were 637, 497, and 1680 µg/L, respectively.

The contents of arsenic (As), cadmium (Cd), and manganese (Mn) were determined by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) using a NexION 1000 instrument (PerkinElmer, USA). Instrument parameter control and data processing were carried out with Syngistix software. Before each analytical run, instrument performance was optimized using a *Tuning solution* (Be, Ce, Fe, In, Li, Mg, Pb i U, 1 µg/L, PerkinElmer, USA).

Calibration was performed using a multielement standard solution containing As, Cd, and Mn (AccuStandard, USA), while single element solutions of rhenium (Re) and rhodium (Rh) (AccuStandard, USA) were used as internal standards. The calibration range comprised seven points: 0, 1, 5, 10, 50, 100, and 200 µg/L.

The control charts were constructed based on the certified values of the CRM. The central line represents the certified value, while the warning limits were defined according to the QC acceptance limits and the action limits according to the Performance Acceptance Limits issued by the manufacturer.

3. RESULTS AND DISCUSSION

During CRM analysis, the measured value is entered into the control chart and interpreted. The method is considered out of control if: (1) one value lies beyond the action limit (red line); (2) two consecutive values lie beyond the warning limits (orange line); or (3) 10–11 consecutive values fall on the same side of the center line (green line) [2].

Figure 1 shows the X-control chart for As; since all values fell within the warning limits, we conclude that the method was in control and that the obtained analytical results are valid.

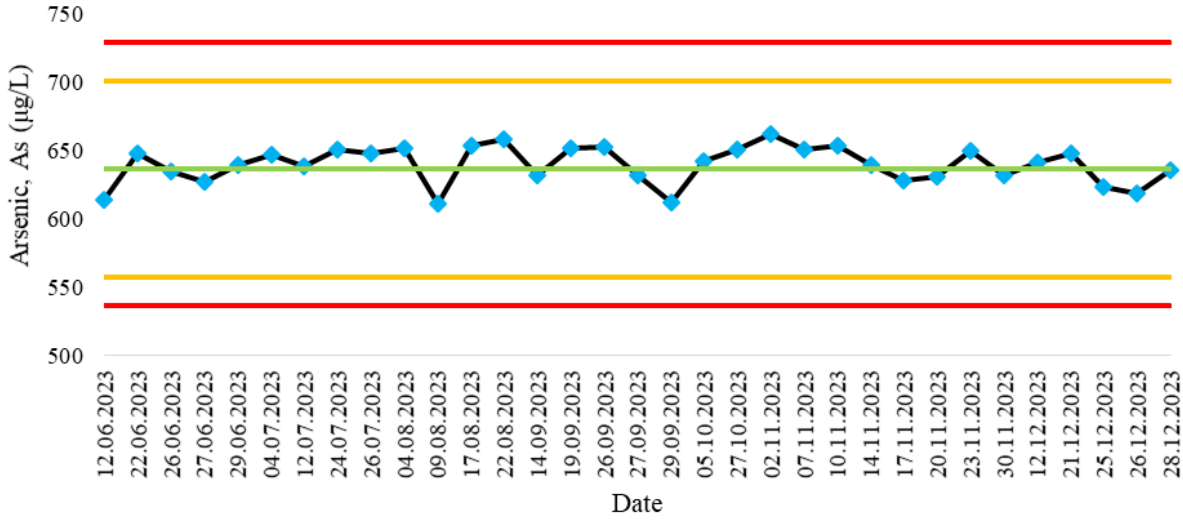

Figure 1. X-control chart for As

Figure 2 shows the X-control chart for Cd. Two values on 27 June and 7 November 2023 exceeded the warning limits; however, since the preceding and subsequent values were within the warning limits, the method remained in control.

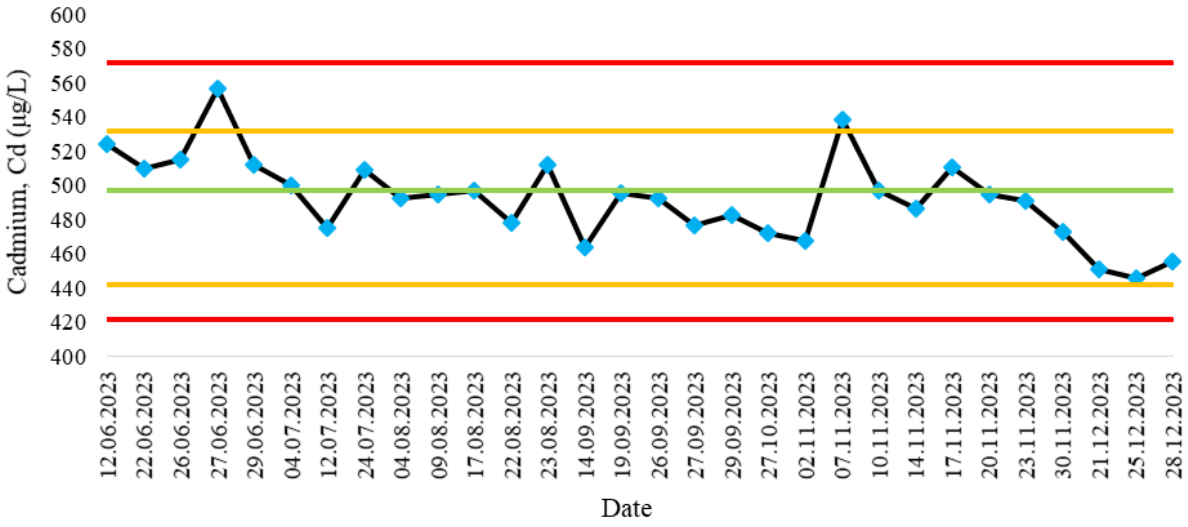

Figure 2. X-control chart for Cd

Figure 3 presents the X-control chart for Mn; since all values were within the warning limits, we concluded that the method was in control and that the obtained analytical results are valid.

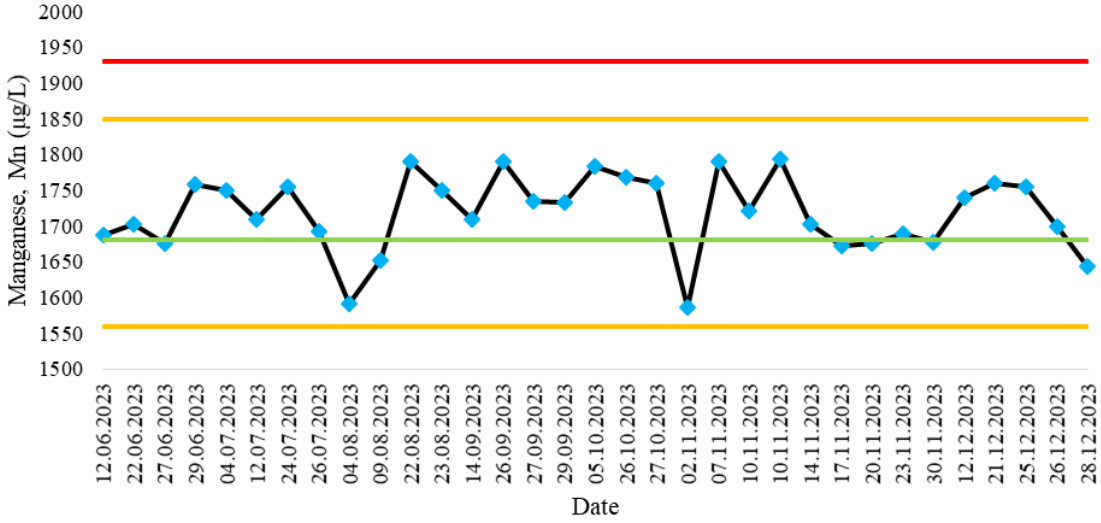

Figure 3. X-control chart for Mn

4. CONCLUSION

The results of six months of monitoring showed that all CRM values for As, Cd, and Mn were within the permissible limits defined by the manufacturer. This confirmed the reliability of the ICP-MS method and ensured the conditions for issuing valid analytical results, enabling the laboratory to report results with confidence in accordance with ISO/IEC 17025 requirements.

ACKNOWLEDGEMENTS

This paper was financially supported by the Ministry of Science, Technological Development and Innovation of the Republic of Serbia, contract numbers: 451-03-136/2025-03/ 20052 and 451-03-65/2025-03/200124.

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