RAP CONFERENCE PROCEEDINGS, VOL. 9, PP. 10–12, 2024
ISSN 2737-9973 (ONLINE) | DOI: 10.37392/RAPPROC.2024.03
RAP-PROCEEDINGS.ORG

NEW DEVELOPMENT OF RADIUM-226 ANALYSIS IN WATER SAMPLES USING MnO_2 RESIN AND ALPHA SPECTROMETRY

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Abstract. *A time-saving and optimum procedure for determining Radium in water has been implemented in Kuwait. This new development has been reached including radiochemical separation using manganese dioxide (MnO_2) resin and α spectrometry measurements. Radium is separated and retained by the MnO_2 resin, then it is extracted by 5 M HCL/15%H_2O_2 solution. Barium sulfate is precipitated onto the resolve filter followed by α measurement of Radium-226 (^{226}Ra), in addition to γ measurement of Barium-133 (^{133}Ba) to calculate chemical recovery%. IAEA proficiency test sample 2 was analyzed similarly, and the generated value of Radium-226 (^{226}Ra) was in agreement with the reference value as well as the γ result analyzed in the same laboratory. The newly developed radium analysis procedure is more efficient for water samples than other radio-analytical techniques due to the low detection limit of α spectrometry compared to γ spectrometry.*

Keywords: *alpha measurement, detection limit, manganese dioxide, radium*

1. INTRODUCTION

The need to develop a new optimized and time saving analytical method is of utmost importance specially in cases of emergency where a quick determination of radioactivity is required.^{226}Ra in a natural radioactive element produced by uranium and thorium decay chains in environment. It has 1,600 years half-life, 4784 keV α energy, and 186.2 keV γ energy. Measurement of Radium is important because it is carcinogenic if a human is exposed for long time, it acts like calcium and accumulate in bones (long and bone cancer) [1,2]. ^{226}Ra is commonly measured by γ spectrometry, α spectrometry, liquid scintillation counter, and mass spectrometry [2,3]. The most routine one applied is the γ measurements; however, due to some difficulties such as the low activity ^{226}Ra in environmental samples sometimes, another measuring technique is required such as α measurements which has low detection limit and background comparably. The routine and common time-consuming chemical separation has been improved in this study, a new optimization has been approached by implementing a rapid radioanalytical procedure for ^{226}Ra analysis in water samples using MnO_2 resin and measurements done by α spectrometry.

2. METHODOLOGY

Three different procedures have been applied sequentially as described below:

A) The first analytical method implemented was the Ba/Ra SO_4 co-precipitation [2,4]. Three deionized water samples (0.5 liter each) spiked with 0.4 Bq of ^{226}Ra have been radio-chemically separated and measured by α spectrometry (Figure 1).

Figure 1. 1st procedure applied

B) Second analytical method was the IAEA/AQ/39 procedure [3] where a radiochemical separation is applied for three deionized water samples spiked with 0.4 Bq of 226Ra followed by LSC measurement. However, first attempt included the similar separation followed by a measurement using α spectrometry instead (Figure 2).

C) Third attempt was done spiking 2.4 Bq of 226Ra into three 0.5 l deionized water, and measurements were done using LSC as applied in the IAEA/AQ/39 procedure. Verification was done by analyzing an additional deionized water sample spiked with 0.4 Bq of 226Ra (about 1/6 of the activity added to the first analyzed patch of samples).

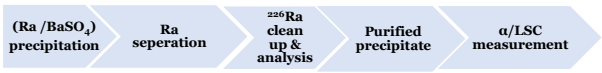

Figure 2. 2nd procedure applied (IAEA/AQ/39)

D) The last optimization was approached by a radiochemical separation of Ra/Ba pre-concentration using MnO_2 resin. One gram of this resin was merged in distilled water and then loaded into a glass column. The water samples were shifted to pH 6-7, then loaded into the resin. Radium was separated and retained by the

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resin, then it was extracted by freshly prepared 5 M HCL/15%H_2O_2 solution (resin speed 15 ml/min). Barium sulfate was precipitated onto the resolve filter followed by α counting for 20,000 seconds acquisition time, whereas the chemical recovery% of the procedure was calculated based on the γ measurement of ^{133}Ba tracer added at the beginning of the analysis. (Figure 3) [5,6].

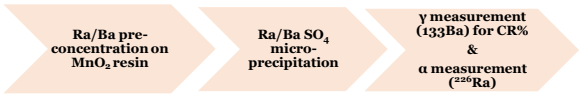

Figure 3. 3rd procedure applied.

3. Results and Discussion

The mentioned above several analytical methods have been practiced improving and simplify ^{226}Ra radioanalytical procedure including saving the time and achieving good statistics. The outcomes of each methodology were as described below:

A) Ba/Ra SO_4 co-precipitation: noise was observed in the three spectra as shown in Figure 4. Where defining the peak of ^{226}Ra was impossible.

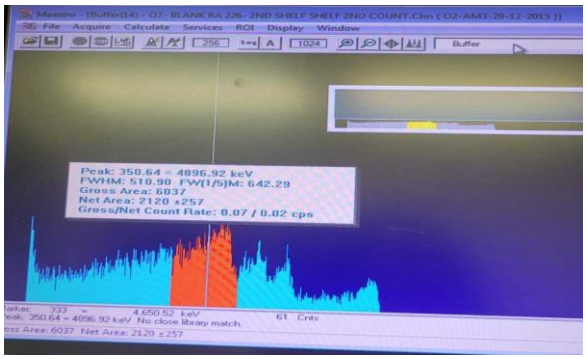

Figure 4. 1st procedure (Ra/$BaSO_4$ co-precipitation) spectrum

B) IAEA/AQ/39 followed by α spectrometry measurement: A good separation was observed in the spectrum (Figure 5) while the recovery was low, 20 % only.

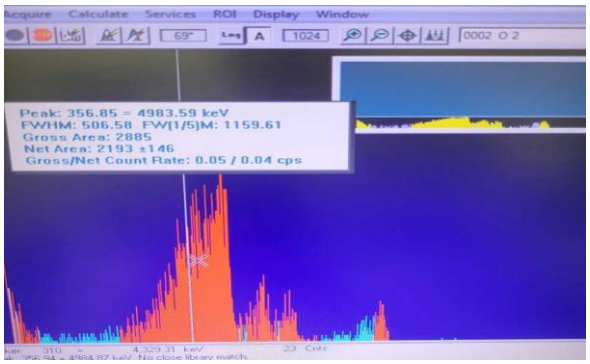

Figure 5. IAEA/AQ/39 procedure α spectrum

C) IAEA/AQ/39 followed by LSC measurement: Figure 6 presents the LSC spectrum for one of the three samples analyzed where ^{226}Ra peak were detected. However, no peak was detected in the spectrum of the verification sample that was spiked with 1/6 of activity (0.4 Bq). This is linked to the high detection limit of LSC (1.5 Bq/l). There was no problem collecting ^{226}Ra spectrum following IAEA/AQ/39 procedure where only LSC has lower CR% than α spectrometry.

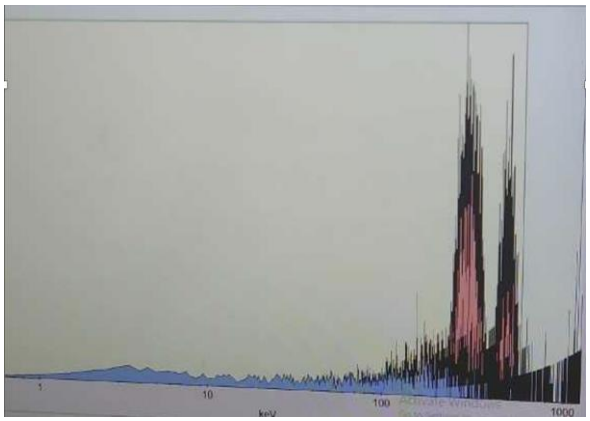

Figure 6. IAEA/AQ/39 procedure spectrum (LSC)

D) Ra/Ba pre-concentration using MnO_2 resin: good statistics and high resolution were achieved as shown in the spectrum (Figure 7), where 95% chemical recovery was calculated based on the added activity of ^{133}Ba.

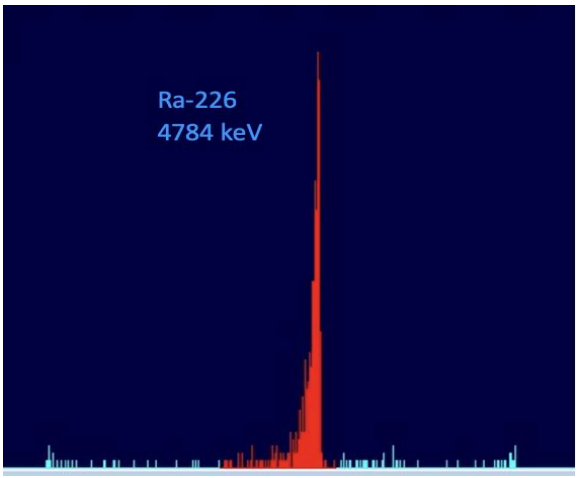

Figure 7. MnO2 resin separation of α spectrum.

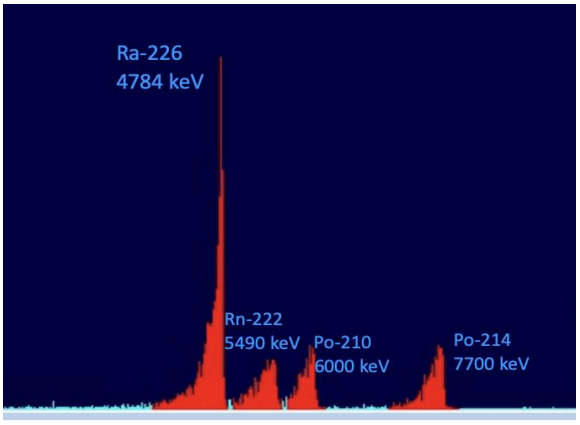

Figure 8. IAEA-proficiency test 2023-sample 2 α spectrum

The analytical results were validated by the analysis of IAEA-proficiency test-2023 sample 2. The experimentally obtained value of ^{226}Ra was in a good

agreement with the certified value of PT sample 2 as well as our own laboratory analyzed γ value. Our α value was calculated to be 7.34 ±0.5 Bq/kg and our gamma value was 6.30±0.30 Bq/kg, whereas the IAEA unknown sample certified value was 6.32±0.32 Bq/kg. The analyzed unknown sample spectrum is shown as Figure 8.

4. Conclusion

New improvement of ^{226}Ra rapid analysis has been achieved in Kuwait for water samples using MnO2 resin for the Ra/Ba separation while the measurements were done by α spectrometry. Verification of the chemical recovery% was approached via ^{133}Ba γ measurement. This approach is linked to the new need of rapid analytical procedures that are helpful for the emergency cases monitoring. Several procedures have been practiced till approaching the good statistical spectrum with highest chemical recovery. The presented work involved the ideal analytical practice where the procedure was confirmed by the analysis of IAEA proficiency sample for accuracy.

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