Optimization of parameters of alpha spectrometry with silicon detector for low level measurements of actinides in environmental samples

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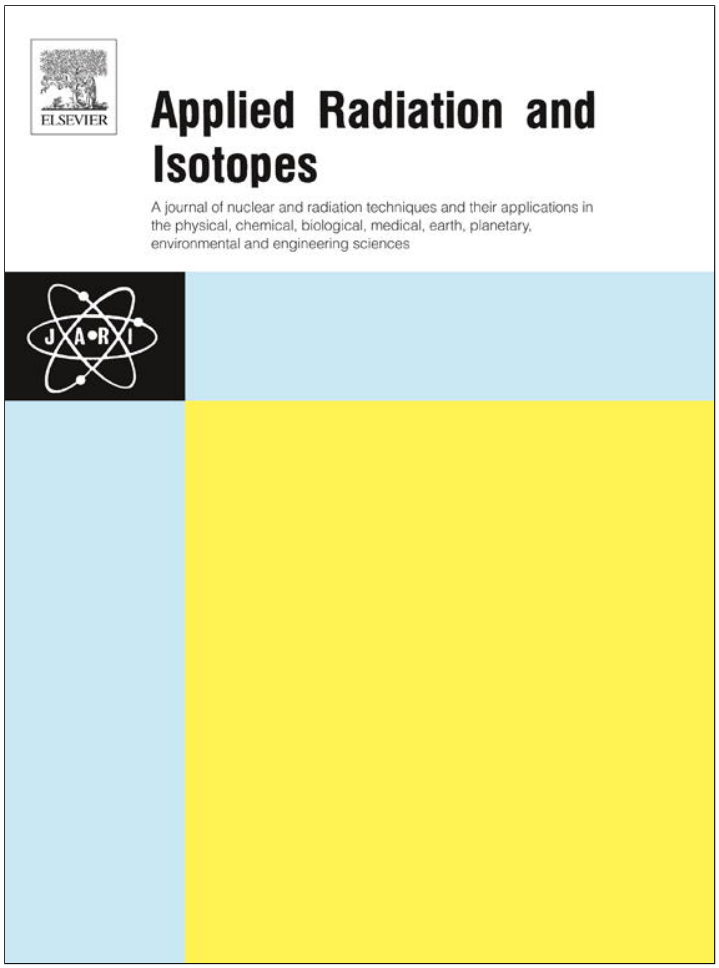

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Optimization of parameters of alpha spectrometry with silicon detector for low level measurements of actinides in environmental samples

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HIGHLIGHTS

- Several parameters relevant to low level alpha spectrometry have been investigated and appropriately optimized.
- The most important parameter has been the influence of chamber pressure on resolution when the chamber is in hold mode while the vacuum pump is electrically switched off for more than 40 h.
- Samples were counted for about 4 day for low levels of detection. Efficiency, tail length, detector size and other parameters were evaluated.

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ABSTRACT

Determination of actinides in environmental and biological samples is an important activity of radiation protection program at nuclear energy facilities. High resolution alpha spectrometry with passivated ion implanted Silicon detectors is widely used for the determination of actinides concentration. Low levels of activity concentrations in these samples often require long counting duration of a few days to obtain accurate and statistically significant data for further impact assessment. In alpha spectrometry, the chamber in which Si detector operated is a critical component and maintained at a desired vacuum for minimizing the alpha particle attenuation. Experimental evaluation of variations in energy resolution and tailing of alpha spectra was investigated under different chamber air pressures from about 6.7 Pa to more than 2700 Pa under the chamber hold mode and pump electrically switched off conditions. As part of validation, data collected on an IAEA inter-comparison exercise sample are presented under short and long counting durations with pump operating and switched off conditions respectively. It has been observed that the FWHM values do not significantly degrade, to impact the low and medium level concentration alpha spectra, for variations in vacuum chamber pressures from about 6.7 Pa to 2700 Pa.

1. Introduction

Alpha spectrometry using passivated ion implanted planar silicon (PIPS) detectors is a sensitive and widely used detection and analysis system for the quantitative determination of isotopes of Pu, U, Am, Po etc., in several environmental and biological samples. Environmental samples such as soil, water, weeds, marine fish and sediments around nuclear power generation facilities and biological materials (urine and feaces) of personnel involved in nuclear fuel handling facilities are analyzed for the assessment of alpha emitting nuclides following a standard radiochemical separation as a part of radiation protection program (Manickam et al., 2008; Sansone et al., 2008; Shinohara and Kunihiko, 2004). These matrices generally have activity concentration of ^{239}Pu, ^{238}Pu, ^{240}Pu, ^{241}Am, ^{238}U and ^{234}U at very low levels of near the detection limit of measuring systems. The PIPS detector alpha spectrometry is also used for the evaluation of specific activities of Pu, U, Cm, Am and other isotopes at significantly high concentration in isotopes separation and characterization activities (Aggarwal et al., 1992). The challenges encountered in both situations of low level and high specific activity measurements are often extremely different from one another. The parameters such as energy resolution, detection efficiency and time of counting have contrasting importance in these types of measurements. Usually, small area detectors, low efficiency and high energy resolving power and short counting time of a few thousand seconds are required for high specific activity measurements. On the other hand, high efficiency, large area and long counting time and relatively less resolving power (can be tolerated) are the needs of low level environmental concentration measurements.

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The Silicon detector in an alpha spectrometry system is housed in a vacuum chamber and maintained at a certain vacuum pressure during operation. The vacuum pump used for evacuating the chamber is generally kept on all the time, for the entire counting duration (few days) to maintain the vacuum pressure. The spectroscopy systems commonly have a valve to operate the chamber in 'Hold' and 'Pump' mode. Pump mode for continuously evacuating the chamber and hold mode for holding the vacuum pressure when the pump is in off condition. However, in hold mode there are always certain leakages (or air entry paths) and pressure increases as the hold time increases. Martin and Hancock (2004) investigated the degradation of energy resolution with increase in the nitrogen pressure in the chamber. For counting of environmental samples, for a few days duration, to achieve significantly lower levels of detection, either the vacuum pump shall be kept on continuously for all the time to maintain constant chamber pressure or operate in hold mode. However, un-manned operation of pump during non-working hours of the day ($\sim$ 16 h d^{-1}; in our laboratory 17:00–9:00 h) is not recommended due to safety concerns of vacuum pump. In view of this, we investigated the changes in energy resolution and other parameters of alpha spectra as the pressure in vacuum chamber increases for more than 30 h by operating the chamber valve in hold mode. In this paper, we present the influence of vacuum chamber pressure on the energy resolution, tailing effect by operating the chamber in hold mode (Vacuum pump off) for more than 16 h, the source to detector distance on efficiency of detector, the surface area of the PIPS detector and the source diameter. The paper also presents, as an exemplification, the results of International Atomic Energy Agency (IAEA) inter-comparison exercise for ^{239}Pu, ^{238}Pu, ^{234}U and ^{238}U in spinach powder with radio tracers of ^{236}Pu and ^{232}U for short and long counting duration.

2. Instrumentation and materials

In the present measurements, a bench top eight chamber alpha spectrometer (model Octete plus) with Mastero basic alpha spectrometry software and a vacuum pump (Edward model RV8) were used. Each chamber has 6 level height adjustable source to detector distances of $\sim$5 mm each. In house prepared electroplated alpha standard sources of ^{239}Pu and ^{241}Am traceable to NPL standard ^{236}Pu (Standard Reference no. E05090391/01) and an AMR mixed source of ^{241}Am, ^{239}Pu and ^{244}Cm (AMR.43 No.9682A of Amersham Intl. Ltd.) were used for optimizing the parameters. The source strengths ranging from 5.4 Bq to 5550 Bq were used for checking the influence of varying concentrations. PIPS detectors of surface area 450 mm^2 and 600 mm^2 each with 100 μm depth were used. The laboratory prepared sources and also the samples have a plating diameter of 20 mm while that of AMR standard has a diameter of 5 mm. The energy resolution of an alpha peak was expressed as full width at half maximum (FWHM) and in the present measurements, it was evaluated using Mastero software and verified manually. The FWHM of PIPS detector is generally quoted in the range of 18 to 21 keV at 5486 keV of ^{241}Am under ideal conditions. However, it strongly depends on the condition of various parameters such as source to detector distance (STDD), source thickness, source diameter and the vacuum chamber pressure. Measurements for absolute efficiency and energy resolution were performed for such a counting duration so that each measurement had about 10% uncertainty. The chamber pressure was measured and displayed in the units of mTorr by the software (later in the manuscript SI unit, Pa will be used) and the sensors have a range of 10 m Torr to 25,000 m Torr, over which it shows overflow.

3. Results and discussions

The measurements of energy resolution, expressed as FWHM, were performed with three sources: an AMR mixed source (^{241}Am, ^{239}Pu and ^{244}Cm) of diameter 5 mm on a 25 mm stainless steel (SS) planchet (5550 Bq total), a laboratory prepared source of ^{241}Am (40.72 Bq (diameter 20 mm on a 25 mm SS planchet) and another laboratory prepared mixed source of ^{239}Pu (12.07 Bq) and ^{241}Am (5.84 Bq) of diameter 20 mm on a 25 mm SS planchet. For the purpose of completeness of alpha spectrometry, Table 1 gives the variations of resolution and absolute efficiency against STDDs for ^{241}Am source at a chamber pressure of 6.7 Pa. The FWHM was found to decrease from about 65 keV (5 mm STDD) to about 38 keV (30 mm STDD) on a 450 mm^2 detector. The minimum STDD that can be utilized on the system is 5 mm. It is a standard practice that detector's resolution is measured at a STDD of 15 mm or higher. A difference of about 5–10 keV resolution can also be seen between the PIPS detectors of 450 mm^2 and 600 mm^2. The FWHM values at 20 mm STDD are found to be 42 and 47 respectively for 450 mm^2 and 600 mm^2 detector. These values are nearly 20 keV more than the technically specified FWHM and can be attributed primarily to source thickness. Vajda and Kim (2009) have also observed that FWHM in the range of 20–60 keV are possible depending on the quality of source, source to detector distance and the type of detector.

The changes in absolute efficiency for different STDDs are given in Table 1 for two PIPS detectors of 450 mm^2 and 600 mm^2 surface area. The maximum efficiency of 22.5% and 26% can be observed at 5 mm STDD for 20 mm source diameter and for 450 mm^2 and 600 mm^2 detectors respectively. A 5 mm increase in STDD results in a significant reduction in absolute efficiency which lowers the detection limit. A simplified solid angle expression for absolute efficiency (E) as defined by Jaffey (1954) in terms of STDD, source diameter and detector active area is given below for comparing the experimental efficiencies.

$$E = 0.5\left(1-\frac{h}{\sqrt{h^2+r^2}}\right)-\frac{3}{16}\left(\frac{a\times r}{h^2}\right)^2\left(1-\frac{h}{\sqrt{h^2+r^2}}\right)^5$$

where, a: radius of disk source (mm), h: source to detector distance (mm), r: radius of detector (mm).

The computed value of E for 450 mm^2 detector (r=12 mm), STDD of 5 mm (h=5 mm), and disk source of radius 10 mm (a=10 mm), is 0.27 (27%). The value from Table 1 is 22.5% and the difference is primarily due to the difficulty in measuring the accurate STDD. A STDD of 6 mm results in 23% efficiency which is close to the experimental value. Pollanen et al. (2011) had also observed that an error in STDD determination of about 0.3 mm can vary the efficiency by about 5%. The experimentally observed efficiencies agree well with the theoretical values for STDDs of 10 mm and above. They are 14.9% and 15.1% and 17.5% and 18.2% for 10 mm STDD for a 450 mm^2 detector and 600 mm^2 detectors respectively.

Table 1
Variations in experimental efficiency and energy resolution with respect source to detector distance (^{241}Am source, 40.7 Bq).

STDD (mm)	Efficiency (%)		Resolution–FWHM: (keV)	
	450 mm^2	600 mm^2	450 mm^2	600 mm^2
5	22.5	25.6	64.4	71.6
10	14.9	17.5	54.5	61.9
15	9.7	12.1	48.2	52.8
20	6.9	8.8	41.6	47.0
25	5.0	6.6	40.7	46.1
30	3.9	5.1	38.5	45.0

The influence of chamber vacuum pressure on energy resolution was investigated for several activity concentrations under increasing chamber pressure. This was one of the important investigations, as we required the operation of alpha spectrometry system in chamber hold mode while the vacuum pump was in electrically switched off condition. The energy resolution in FWHM was measured for chamber pressures from approximately ~6.7 Pa to more than 2700 Pa. Under the conditions of vacuum pump operating and the valve in pump mode, the chamber pressure remains stable at around 6.7 Pa. By turning the valve to hold mode and the vacuum pump switched off, the chamber pressure gradually increases towards atmospheric pressure due to ingress of air through minor leakage (entry) paths of manifolds and other rubber gaskets. Fig. 1 shows the variations of energy resolution (FWHM) as a function of increasing chamber pressure for three electroplated source planchets of pure Am, mixed Pu/Am and a mixed Pu/Am/Cm at a STDD of 15 mm. The chamber was kept in hold position for more than 48 h while the chamber pressure reached to more than 2600 Pa. The influence of increasing chamber pressure, on a ^{241}Am source of 40.7 Bq, for about 48 h hold position has resulted in FWHM values varying in the range of 48–58 keV. FWHM values varied from about 30 keV to 38 keV in 48 h of hold position for Pu/Am mixed source. The variation in FWHM during overnight counting (usually 16 h duration) was not significant, 30–33 keV for ^{239}Pu and 28–33 keV for ^{241}Am. The activity of ^{239}Pu and ^{241}Am in the mixed source were 12.07 Bq and 5.84 Bq respectively. Fig. 1 also shows the changes in FWHM against chamber pressure for an AMR

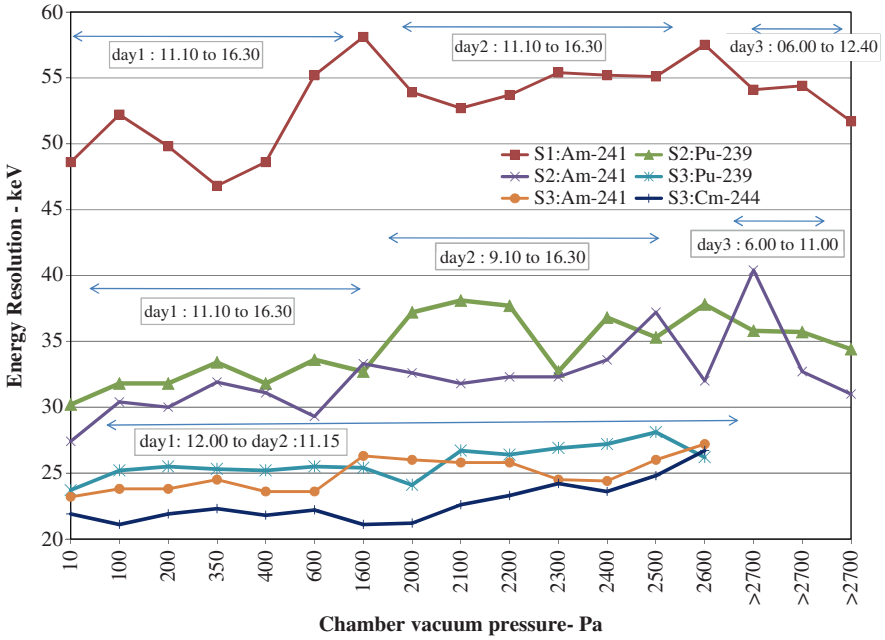

Fig. 1. Am-241 (*S1*), Pu/Am mixed source (*S2*) and AMR mixed source (*S3*) at STDD of 15 mm—in chamber hold mode for more than 24 h (time details are given above the graph lines).

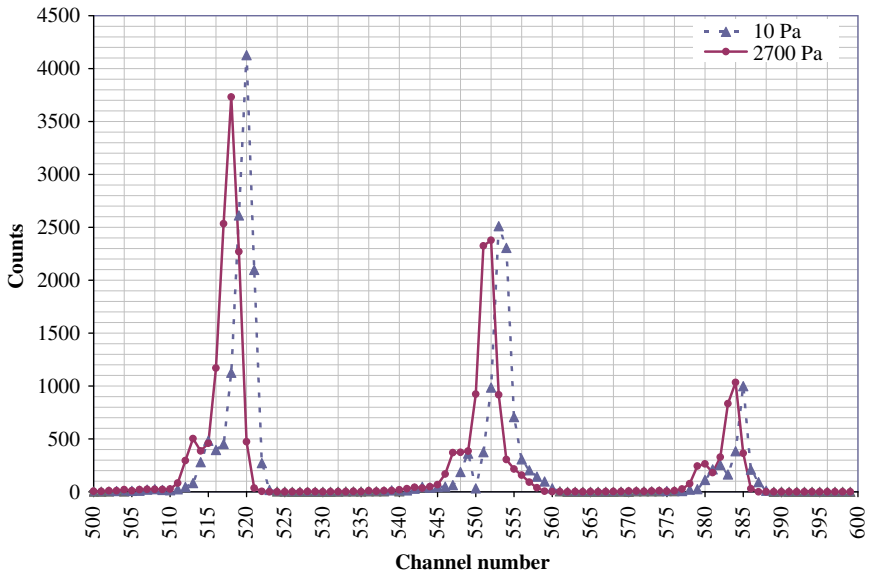

Fig. 2. Alpha particle spectrum of Pu, Am and Cm (AMR mixed source) at extreme chamber pressures.

mixed source of Am/Pu/Cm with total activity of 5550 Bq. The FWHM values were in the range of 21–27 keV for chamber pressure of more than 2600 Pa. The FWHM values are close to the standard specified energy resolution of PIPS detector for the AMR source as compared to the other two laboratory prepared sources. Thus, the source diameter and thickness contributes significantly for poor resolution. The increased alpha activity of 5550 Bq under changing vacuum conditions also has not significantly affected the resolution. However, for low level measurements of environmental and bioassay samples, the activity concentration encountered are a few mBq per sample. Although there is no significant degradation of FWHM even up to 48 h hold position, a practice that pump will be kept on during the working hours of laboratory (9:00–17:00 h) and hold mode with pump off during non-working hours (17:00–9:00 h) is followed. The vacuum chamber pressure at 2700 Pa (the maximum up to which the FWHM measurements were performed) is providing a chamber vacuum of about 3% of normal atmospheric pressure (1 atm = 101.3 kPa) while the minimum maintained during the pump mode (vacuum pump switched ON) is equal to about 0.0098% of atmospheric pressure. Fig. 2 shows the part spectrum of AMR mixed source at 10 Pa and 2700 Pa. There is no significant deterioration in the quality of the spectra except that a small broadening can be visualized at 2700 Pa along with one channel shift towards lower energy. In a similar type of investigations with N_2 gas in chamber as absorber gas, Martin and Hankock (2004) observed that 25–30 μg cm^{-2} of absorber thickness do not significantly affect the FWHM and becomes poorer as the absorber thickness increases, up to 180 keV at 200–250 μg cm^{-2}. Vainblata et al. (2004) studied the influence of source coating with mylar film on resolution and efficiency and observed that FWHM varying from 22 keV to 58 keV at STDD of 17 mm for source coating of 0–0.85 mg cm^{-2}. Vargas (2001), in his experiments with Ba deposits on source has observed that *a* 0.3 mg of

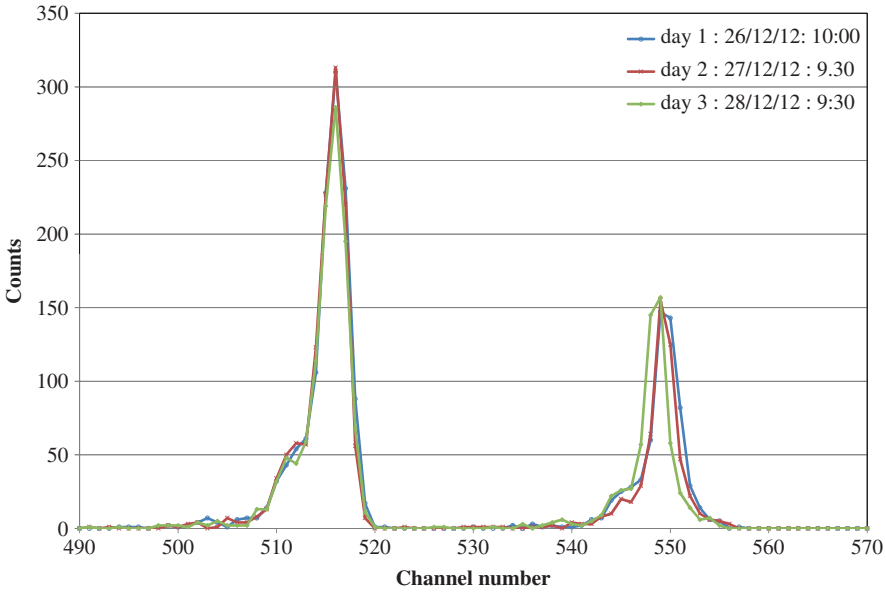

Fig.3. Spectra of Pu-239/Am-241 mixed source at STDD of 15 mm—chamber in 'pump mode' for 56 h.

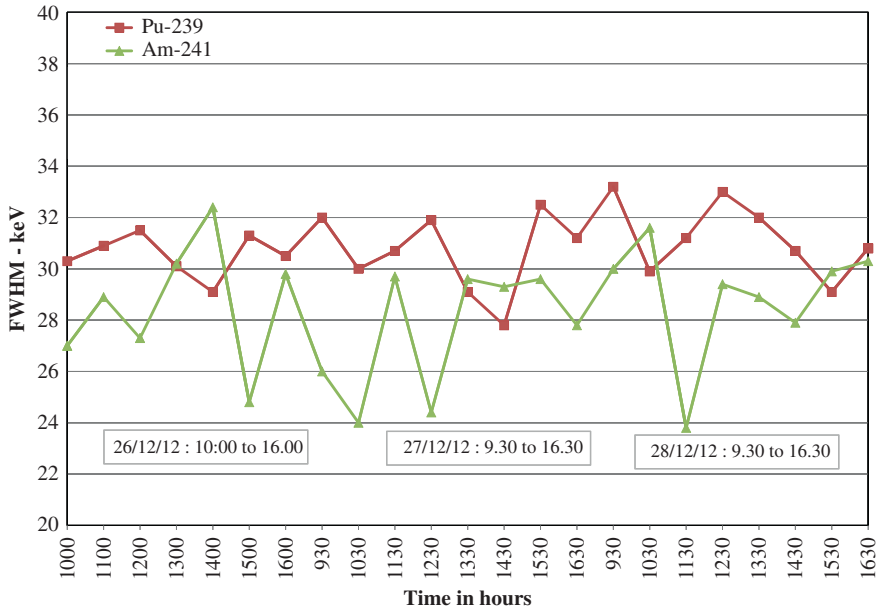

Fig. 4. Pu-239/Am-241 mixed source at STDD of 15 mm—chamber in 'pump mode' for 56 h.

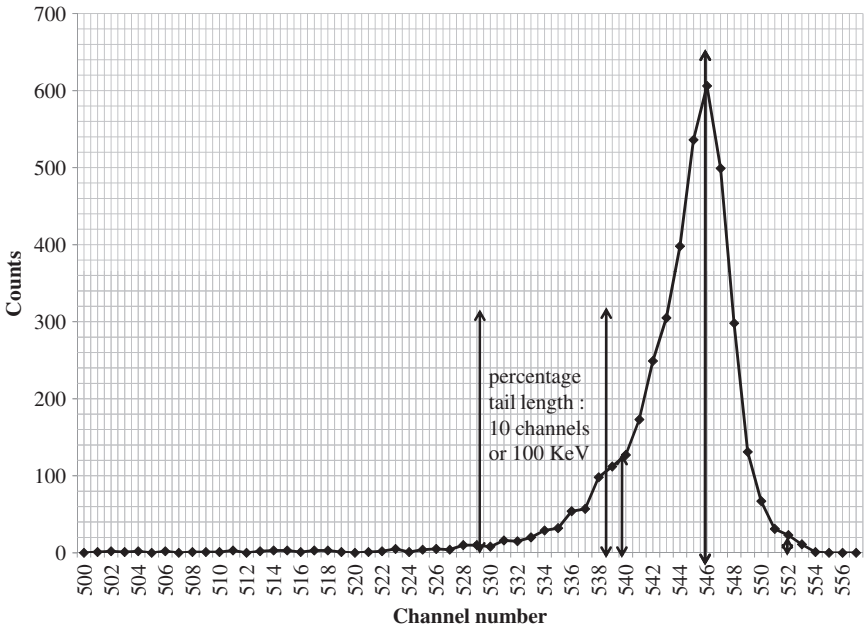

Fig. 5. Tail boundaries determination of an Am-241 peak.

Ba deposit resulted in the FWHM of 50 keV. In order to check the electronic stability of the alpha spectrometry system, measurements were performed for three days by keeping the chamber vacuum nearly constant at 6.7 Pa (Pump on mode for all the three days). Figs. 3 and 4 shows the part spectra and resolution variations on different days respectively. It can be observed that there are no significant changes in resolution except for statistical deviations and also no degradation in the quality of peaks including shift in peak channel. The present day electronics generally do not need much warm up time except for few minutes and also channel shifts are negligible particularly for 10 keV/channel energy bin size.

3.1. Peak tail

Individual energy peak of alpha spectra, in addition to peak information is also characterized by a limited tail formed on the lower energy part of the peak. The magnitude of the tail mainly depends on the sample source preparation and the resolution of the peak. The tail can interfere manly on the lower energy peak if any, particularly with radiochemical tracer peaks such as ^{242}Pu for ^{239}Pu and ^{243}Am for ^{241}Am. The tail magnitude is investigated under extreme vacuum chamber pressure conditions. A methodology for determination of tail boundaries has been formulated so as to maintain the consistency in comparison for peaks of different resolutions. The tail length of 100 keV (10 channel) is defined below the peak boundary on the left side, while the peak boundaries are set as $2.5 \times$ FWHM of that peak. In an example of ^{241}Am (40.7 Bq), the peak boundaries are 2.5×50 keV which is equal to 125 keV ($\sim$13 channels). Hence the peak boundaries are 6 channels on either side of peak channel. For other sources, (mixed Pu/Am/Cm and mixed Pu/Am) as the FWHM is around 25–30 keV, $2.5 \times 25 = 62.5$ or 70 keV or 7 channels are chosen as peak boundaries. Therefore, the peak boundaries are 3 channels on either side of the peak position. Fig. 5 shows the details of tail length determination. The magnitude of tail%, defined as the ratio of average counts of 100 keV (10 channels) tail energy band to the peak count multiplied by 100. The detailed data obtained for tail magnitudes for the extreme vacuum chamber pressures are given in Table 2. The % tail was not significantly different for low and high chamber pressures.

Table 2
The percentage of tail length under extreme chamber air pressure.

No.	Source	Activity (Bq)	Vacuum (Pa)	% of tail	FWHM (keV)	Detector number: No. of 'hold' hours
1	^{241}Am	40.7	6.7	7.9	48.66	D3: 0 h
1	^{241}Am	40.7	2600	7.3	57.5	D3: 30 h
2.	^{241}Am	1850	11.3	3.34	23.2	D8: 0 h
2.	^{241}Am	1850	2730	4.9	26.0	D8: 22 h
2.	^{239}Pu	2775	11.3	3.36	23.7	D8: 0 h
2.	^{239}Pu	2775	2730	3.74	28.1	D8: 22 h
2.	^{244}Cm	925	11.3	4.07	21.9	D8: 0 h
2.	^{244}Cm	925	2730	6.37	24.8	D8: 22 h
3	^{241}Am	5.84	3.3	4.44	27.4	D4: 0 h
3	^{241}Am	5.84	1280	6.64	31.0	D4: 24 h
3	^{239}Pu	12.07	3.3	5.75	30.2	D4: 0 h
3	^{239}Pu	12.07	1280	7.92	34.4	D4: 24 h

3.2. Minimum detectable activity

Minimum detectable activity (MDA) for environmental/biological samples is defined to set a limit for the analytical and measurement technique, that the activity concentration above such value is positive/true with a confidence of 95% and is generally expressed with following equation (Boecker et al., 1991).

$$\mathrm{MDA} = \frac{4.65\sqrt{B} + 2.7}{t \in RW}$$

where, B is alpha spectrometer background counts in the appropriate energy band, t is counting time, $\in$ is absolute efficiency (≤ 1), R is radiochemical recovery (≤ 1) and W is the weight of sample or sample size correction factor.

The MDA for the system used in the present study is estimated to be 0.0024 Bq kg^{-1} or Bq d^{-1} for a 1 kg of an environmental sample or for a one day urine excreta sample based on an average background counts of 6 for 345,000 s counting time, 0.23 efficiency and radiochemical recovery of 0.75. It is necessary to count the samples at closest possible STDD (1 rack of$\sim$5 mm) to obtain

Table 3
Results of IAEA Spinach powder for chamber pump and hold mode operation.

Analyte	IAEA Value (Bq kg^{-1})	Lab Value (Bq kg^{-1})	Relative bias (%)[a]	Precision (%)[b]	Trueness	Precision	Final score
[c]							
^{234}U	1.02 ± 0.07	1.06 ± 0.21	3.92	20.97	Passed	Failed	Warning
^{238}U	0.95 ± 0.05	0.89 ± 0.20	−6.32	23.08	Passed	Failed	Warning
^{238}Pu[d]	0.023 ± 0.013	Not detected	–	–	–	–	–
^{239}Pu[d]	0.049 ± 0.028	0.12 ± 0.04	–	–	–	–	Passed
[e]							
^{234}U	1.02 ± 0.07	0.95 ± 0.05	6.86	8.64	Passed	passed	Passed
^{238}U	0.95 ± 0.05	0.99 ± 0.05	−4.2	7.29	Passed	Passed	Passed
^{238}Pu[d]	0.023 ± 0.013	0.04 ± 0.01	–	–	–	–	Passed
^{239}Pu[d]	0.049 ± 0.028	0.05 ± 0.01	–	–	–	–	Passed

[a] Maximum acceptable bias (MAB%)=20%.
[b] Least acceptable precision (LAP%)=20%.
[c] Counting time 25,000 s for U and 86,400 for Pu isotopes—Evaluated by IAEA.
[d] Pu isotopes were evaluated with upper level evaluation (ULE), precision and bias were not estimated, ULE for ^{238}Pu=0.05 Bq kg^{-1} and ^{239}Pu=0.11 Bq kg^{-1}.
[e] Counting time 270,000 s for U and Pu isotopes—Recounting with new methodology of chamber hold mode.

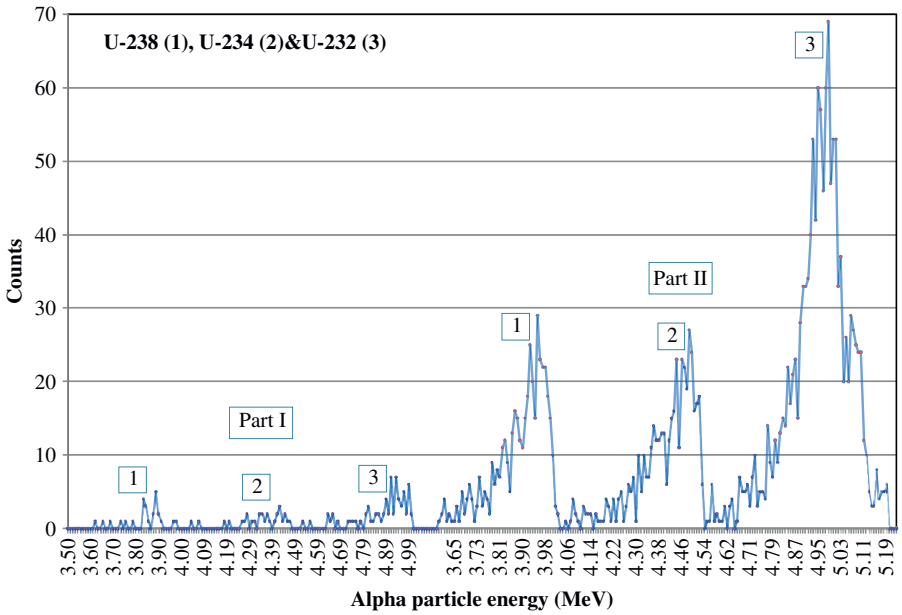

Fig. 6. Spectra of U-isotopes of an IAEA-SPINACH powder counted for 25,000 s in 'pump mode' (Part I) and 270,000 s in 'chamber hold mode' (Part II).

lowest MDA as the efficiency at next available rack (STDD 10 mm) is nearly 10% lower and efficiency being invariant for 4–8 MeV energy alpha particles IAEA (2009). Samples of high activity concentration can be counted at larger STDD to benefit from improved energy resolution.

3.3. *Measurements on an IAEA spinach powder sample under pump and hold modes*

In order to demonstrate the quality assurance of chamber hold mode, the results obtained for an IAEA inter-comparison exercise sample for long counting time, with vacuum pump electrically switched off (chamber valve in hold mode for 16 h during non-office hours) are presented. A sample of spinach powder of IAEA-CU-2007-03 (IAEA 2009) world wide open proficiency test (Sample code IAEA334), was analyzed and the electrodeposited SS planchets of U and Pu isotopes were counted as per normal practice of keeping the pump in switched on condition for full counting duration and the results reported. The U- source planchet was counted for 25,000 s and Pu source planchet was counted for 84,600 s. The results are summarized in Table 3. Fig. 6 shows the spectra of U-isotopes (^{238}U, ^{234}U and ^{232}U) for both the counting conditions. Both the electrodeposited planchets were re-counted for 270,000 s by adopting the present methodology of operating the chamber in hold mode. Under these conditions, although accuracy was good in both the modes of counting, the precision statistics have improved for all the four isotopes and also the ^{238}Pu was detected, which was reported as below detection at the time of reporting the results. The details of the evaluation (our Lab code-124) are available in IAEA-CU-2007-03 report (IAEA, 2007).

4. Conclusions

The operation of alpha spectrometry system with PIPS detector for long counting time under increasing vacuum chamber air pressure was examined. Following conclusions are drawn from the investigations:

- The influence of chamber air pressure from 6.7 Pa to more than 2700 Pa has not significantly affected the resolution (FWHM) of alpha peaks, to impact the low level measurements of actinides. The increase in pressure was implemented

by keeping the chamber valve in 'hold' mode and the vacuum pump electrically switched off.

- The time taken for chamber air pressure to drop down to 2700 Pa from 6.7 Pa in 'hold' mode was generally 30–40 h.
- On an average, the resolution became poorer by 20% from the observed FWHM at 6.7 Pa over a 40 h duration. In normal practice, the detectors are operated in 'hold' mode for only 16 h during non-office hours.
- The resolution (FWHM) was close to technically specified value of $\sim$20 keV under the conditions of 15 mm STDD and for a small diameter (5 mm) thin AMR source.
- The resolution and the alpha peak shapes have not changed even when the source activities were increased from about $<$ 50 Bq to 5550 Bq under increased chamber air pressures up to 2700 Pa.
- The tailing remained nearly un-influenced by the increase in chamber air pressure, thus providing minimum interference on the lower energy side peaks.
- The experimental efficiency and empirically estimated efficiency for different source to detector distances were in good agreement.
- The estimation of activity concentration of Pu and U isotopes in an IAEA inert-comparison exercise for long counting duration in chamber hold mode has validated the experimental investigations discussed in this paper.

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