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FLUORESCENCE SPECTROSCOPIC (TRFS) STUDIES**

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GOVERNMENT OF INDIA
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$^{248}\text{Cm(III)}$ का $^{252}\text{Cf(III)}$ से पृथक्करण और समय विभाजित प्रतिदीप्ति वर्णमापन अध्ययन में उपयोग

सारांश

इस रिपोर्ट में $^{252}\text{Cf(III)}$ के अवशिष्ट घोल से $^{248}\text{Cm(III)}$ के पृथक्करण क्रिया विधि का वर्णन है । सर्वप्रथम न्यूट्रॉन कूप संभावी गणक के द्वारा अवशिष्ट घोल में $^{252}\text{Cf(III)}$ का अनुमापन किया गया । तदुपरांत अवशिष्ट घोल से $^{248}\text{Cm(III)}$ का पृथक्करण ऋणायन तथा धनायन विनिमय द्वारा किया गया । पृथक्कृत क्यूरियम के अल्फा स्पेक्ट्रम में दो शीर्ष ^{246}Cm तथा ^{248}Cm की उपस्थिति के कारण दिखाई दिये, जबकि कैलिफोर्नियम घटक में $^{249, 250, 251, 252}\text{Cf}$ के शीर्ष दिखें । ^{249}Cf के 387keV गामा किरण पर आधारित बाकी किरणों की प्रचुरता का निर्धारण किया गया जो प्रकाशित आंकड़ों से मिलता है ।

इस पृथक्कृत $^{248}\text{Cm(III)}$ का उपयोग जलीय माध्यम में समय विभाजित प्रतिदीप्ति वर्णमापन के लिए भी किया गया ।

Separation of ^{248}Cm (III) from ^{252}Cf (III) and its use in time resolved fluorescence spectroscopic (TRFS) studies

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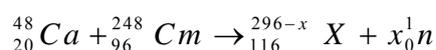
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Abstract

The present report gives a description of the methodology for the separation of ^{248}Cm (III) from decayed ^{252}Cf (III) waste solution. The waste solution was first assayed for ^{252}Cf content by neutron counting using a neutron well coincidence counter. The sample was subjected to the chemical separation of ^{248}Cm (III) from ^{252}Cf (III) following anion and cation exchange chromatography. The alpha spectrum of the separated curium fraction showed peaks due to ^{246}Cm and ^{248}Cm while the corresponding alpha spectrum of californium fraction showed 249 , 250 , 251 , ^{252}Cf . The gamma ray abundances of ^{249}Cf were determined with respect to its gamma rays of 387 keV and the data agreed well with that in literature. Separated Cm (III) was further characterized by recording its time resolved fluorescence spectrum (TRFS) in aqueous medium.

Introduction

^{248}Cm is a long-lived isotope ($t_{1/2} = 3.61 \times 10^5$ y) of curium and is important from two aspects: (i) Due to its higher N/Z, it is used as a target for production of super heavy elements [1], e.g.



and (ii) owing to its long half-life, ^{248}Cm is used as a fluorescence-probe for spectroscopic investigations of curium species present in aquatic environment. Its long half-life facilitates the

handling of micro gram amounts of curium compared to the commonly used isotope ^{244}Cm ($t_{1/2} = 18 \text{ y}$) in physico-chemical studies.

^{248}Cm is the daughter product of ^{252}Cf ($t_{1/2} = 2.645 \text{ y}$) which is extensively used as a neutron source for variety of applications including neutron activation analysis. One μg of ^{252}Cf gives about $2 \times 10^6 \text{ n/s}$.

Old, decayed source of ^{252}Cf is a rich source of ^{248}Cm . At Radiochemistry Division a $50 \mu\text{g}$ ^{252}Cf source was used for fission product chemistry during 1980s. Different fractions of waste solutions generated during the radiochemical separations of ^{252}Cf from Pd/Al alloy were counted using Neutron Well Coincidence Counter (NWCC) and the solution showing the major activity was selected for the separation of ^{248}Cm from the residual ^{252}Cf . For the same amount of ^{252}Cf and ^{248}Cm , the activity of ^{252}Cf is 10^5 times larger than that of ^{248}Cm . Obtaining a pure Cm target is a quite challenging task as both the elements have +3 as the most stable oxidation state and a range of fission products are present as a consequence of spontaneous fission of ^{252}Cf . In addition, there is a radiological concern while working with ^{252}Cf , which is associated with a high neutron dose and intense gamma dose of fission products. It is reported in literature that ion exchange methods [2, 3] could be used for separation of adjacent trivalent actinides. Initial attempts using only anion-exchange method [2] did not give satisfactory separation. Therefore, an alternative procedure was devised which involves the sequential use of an anion exchanger followed by a cation exchanger to achieve almost complete separation.

Experimental

Chemicals

The ion-exchange resins Dowex 1x8 (200-400 mesh) and Dowex 50x8 (200-400 mesh) were procured from Sigma-Aldrich and were converted to nitrate and ammonium forms respectively before using them in the column. Alpha-hydroxy isobutyric acid (α -HIBA) (>99%

purity, Fluka) and all other chemicals such as methanol, nitric acid, perchloric acid, ammonium chloride, liquor ammonia and FeCl₃ were of AR grade.

Apparatus and instruments

Neutron well coincidence counter: Indigenously developed neutron well coincidence counter was used for neutron counting. This counter comprises of an array of twenty four ³He detectors (with 2.54 cm dia and 50 cm sensitive length with 4 atm Fill gas pressure) placed in an annular geometry, with a well diameter of 36 cm dia x 52 cm depth. The absolute detection efficiency of NWCC for gross neutron counting is ~16.8%, providing a good flat response over the volume of the well.

Silicon surface barrier detector : Alpha spectra of the samples prepared by drying them on SS planchettes were recorded by silicon surface barrier detector. The energy calibration was carried out using a standard ²³⁹Pu-²⁴¹Am source. The efficiency of the detector was about 10%.

NaI(Tl) detector: Well type NaI(Tl) counter was used to measure gamma rays due to fission fragments (prompt) and short lived fission products produced (>99%) in the spontaneous fission of ²⁵²Cf present in the collected samples from the separation using ion-exchange column. These counts were used as a signature of Cf.

HPGe detector (γ spectrometry): Gamma ray spectra of the separated Cf fraction were acquired using HPGe based high resolution gamma spectrometer, having 4K channel analyzer. The resolution of the detector is ~2 keV at 1332 keV. The absolute detection efficiency of the gamma spectrometer was obtained using a standard ¹⁵²Eu source.

Edinburgh F-900 time resolved fluorescence spectrometer equipped with Xe-flash lamp: Photoluminescence investigations were carried out on an Edinburgh F-900 time resolved fluorescence spectrometer equipped with M 300 (R= 1nm) excitation and emission monochromators. Fluorescence life-time in microsecond range was measured using Xe flash

lamp. Time resolved emission spectra were obtained for the recovered Cm(III) complex with α -HIBA.

General: Glass columns of 0.4cm dia and 20cm length fitted with stopcocks and jacket were used for ion-exchange separations. Lauda Ecoline RE106 (Germany) instrument with water circulating facility at constant temperature with a precision of $\pm 0.01^\circ\text{C}$ was used to maintain the desired temperature by water circulation through a jacketed column during the cation exchange separation. The pH was measured using a Toshniwal pH meter with a precision of ± 0.01 units. Gamma tubes were used to collect the elution fractions while SS planchettes cleaned with acetone and water were used to prepare the samples for alpha spectrometry.

Procedure

An aged ^{252}Cf source was identified and assayed by NWCC and alpha spectrometry for ^{252}Cf and ^{248}Cm contents respectively. The ion-exchangers (both cation, Dowex 50x 8, 200-400 mesh and anion, Dowex 1x8, 200-400 mesh) were washed with distilled water and converted to nitrate form (anion exchanger) and to ammonium form (cation exchanger) respectively prior to their use. A glass column of 0.4 cm diameter and 5 cm height was used for anion exchange separation while another column having 0.4cm diameter and 11 cm height) with a jacket (for circulating water at a desired temperature) was used for cation exchange separation. In the case of anion exchanger, the eluent was initially 0.5M HNO_3 + 85% methanol and later 0.1M HNO_3 , while in the case of cation exchanger, the eluent was 0.4M alpha-hydroxy isobutyric acid (α -HIBA) at pH = 4.0. The Cf source was dissolved in a minimum amount of dilute nitric acid and precipitated with FeCl_3 solution and liquor ammonia. $\text{Fe}(\text{OH})_3$ precipitate carried with it Cf(III) and Cm(III) while most other fission product impurities remained in the supernatant. The precipitate was washed, collected and dissolved in concentrated nitric acid. The solution was evaporated under IR lamp and redissolved in nitric acid. These steps were repeated thrice and finally the mixture of Cf(III) and Cm(III) was taken in 0.5M HNO_3 + 95% methanol. This

solution was loaded on preconditioned anion exchange column of Dowex 1x8, at ~ 1drop/min and washed with 5-6 column volumes of the same solution. The effluent and washings did not show the presence of either Cf(III) or Cm(III). The eluent was changed to 0.5M HNO₃ + 85% methanol and elution fractions of 0.3-0.4 mL were collected and counted in a well type NaI(Tl) detector. Since the contribution of the gamma counts from ²⁵²Cf fission (from fission fragments and short lived fission products) constitute relatively large (>99%) percentage, the gross counts were taken as the signature of Cf. The counts increased with successive fractions and reached a maximum and decreased thereafter. After collecting a total of ~20 samples, the column was eluted with 8-10 column volumes of 0.1 M nitric acid. The samples were analyzed using NWCC and alpha spectrometry to ascertain the content of Cf and Cm respectively. Based on the above analyses, fractions were grouped as (i) 1-8 which contain pure Cf(III), (ii) the fractions 9-16 containing the mixture and (iii) fraction 17 onwards containing pure Cm(III). Fractions, 9-16 containing reasonable activities of both (as seen by alpha spectrometry) were mixed together and used for the next run of separation by cation exchange column.

The fractions 9-16 collected above (Cf(III) + Cm(III)) were evaporated to dryness and made into a minimum volume of 0.5M nitric acid and loaded on to the cation-exchange column at flow rate of 1-2 drops /min, followed by washing with 0.5M nitric acid. The column was maintained at 85.5 °C by circulation of water by a constant temperature water bath. The elution was carried out with 0.4M α -HIBA at pH=4.0. The elution fractions were collected as outlined earlier and analyzed for γ and α activity. Plots of activity vs. number of volume fractions were drawn in both the cases. This procedure was standardized and validated for the separation of Cm(III) using a small aliquot spiked with ²⁴⁴Cm. The final qualitative and quantitative analyses were made by alpha (silicon surface-barrier) and gamma (HPGe) spectrometry. The separated Cm in α -HIBA can be converted in to nitric acid medium (by repeatedly evaporating with

mixtures of perchloric and nitric acids for destroying the α -HIBA and finally with nitric acid). ^{248}Cm in α -HIBA solution was used to record the fluorescence spectrum.

Results and Discussion

The alpha spectrum of the sample in duplicate prior to separation was recorded and the results are given in Table 1. The following calculation shows the relative amounts of ^{252}Cf and ^{248}Cm likely to be present as estimated using alpha spectrometry by taking a small aliquot (20 μL) from a 10 mL stock, containing Cf and Cm .

$$t_{1/2} \text{ of } ^{252}\text{Cf} = 2.645 \text{ y} \qquad t_{1/2} \text{ of } ^{248}\text{Cm} = 3.61 \times 10^5 \text{ y}$$

$$\text{Specific activity of } ^{252}\text{Cf} = \frac{6.02 \times 10^{23} \times 10^{-6} \times 0.693}{252 \times 2.645 \times 365 \times 24 \times 60 \times 60} = 2.00 \times 10^7 \text{ dps}/\mu\text{g}$$

From Table 1, the average counts of Cf present = 2600 dps

Amount of Cf present in 20 μL = 2600/2.0 $\times 10^7$ μg , therefore in 10 mL = 0.065 μg

$$\begin{aligned} \text{If the source is obtained in 1985, then after 20 years, the amount of Cf} &= N \times e^{0.693 \times 20 / 2.645} \\ &= 0.065 \mu\text{g} \times 198.35 = 12.9 \mu\text{g} \end{aligned}$$

$$\text{Specific activity of } ^{248}\text{Cm} = \frac{6.02 \times 10^{23} \times 10^{-6} \times 0.693}{248 \times 3.61 \times 10^5 \times 365 \times 24 \times 60 \times 60} = 148 \text{ dps}/\mu\text{g}$$

From Table 1, the average counts of Cm present = 2.325 dps

Therefore, amount of Cm present in 20 μL = 2.325/148 = 0.0157 μg

Amount of Cm present in 10 mL stock = 0.0157 $\times 50 \times 10$ = 7.8 μg

Thus it is inferred that ^{248}Cm could be present to the extent of $\sim 8 \mu\text{g}$ in 10 mL stock solution. Out of 10 mL stock, 5 mL was taken for actual separation. Table 2 gives the gamma counts of the fractions (~ 20 , of $\sim 0.3 - 0.4 \text{ mL}$) collected during anion and cation exchange column runs. Later, these fractions containing Cf(III) and Cm(III) were measured by alpha spectrometry. The detailed analysis by alpha spectrometry resulted in a typical plot shown in

Fig.1 as activity vs. volume fractions. It can be seen from the figure that the plot has three portions, namely, (i) the pure Cf(III), up to the fractions 8-9, (ii) pure Cm(III) from fraction 17 onwards and (iii) a middle portion (fractions 10 to 16) wherein the mixture of Cf(III) and Cm(III) was present. Fig.2 gives a similar plot to Fig.1 giving the elution profile of cation exchange column. It is clearly seen from the figure that improved separation was obtained as compared to that in anion exchanger run. Figure 3 and 4 give the alpha spectra of Cm and Cf from where it can be deduced that apart from ^{252}Cf and ^{248}Cm , there were other isotopes of Cf and Cm present in the fractions. The Cf fraction was shown to contain isotopes, 249 , ^{250}Cf and ^{251}Cf apart from ^{252}Cf while the Cm fraction contained ^{246}Cm in addition to ^{248}Cm .

The californium fractions were counted for gamma activity on an efficiency calibrated HPGe detector in a standard geometry. Fig. 5 shows the gamma spectrum due to ^{249}Cf and ^{251}Cf . Some of the gamma lines of ^{249}Cf were resolved sufficiently well enabling the estimation of its relative gamma abundance with respect to 387 keV line. The absolute activity of ^{249}Cf was determined from the alpha spectra taken in a standard geometry. The efficiency calibration of the HPGe detector in the same geometry as that of the ^{249}Cf source was carried out using a standard ^{152}Eu source. The data on relative intensities of the gamma rays of ^{249}Cf are given in Table 3, and are in good agreement with the literature data [4]

TRFS studies with ^{248}Cm

In view of its long half life, ^{248}Cm is an excellent fluorescence probe to investigate and analyze the complexation behaviour of trivalent actinides. It was of interest to use ^{248}Cm ($t_{1/2} = 3.61 \times 10^5$ y) isolated in the present work and was employed to have better insight into the complexation mechanism of trivalent actinides using TRFS.

The fluorescence emission spectrum of Cm-HIBA was recorded using Edinburgh F-900 unit equipped with M 300 monochromators and the spectrum is given in Fig.6. The long-lived

component obtained after the delay time of 160 μs showed the emission at 605 nm. The peak at 605 nm, arises due to the transition from the lowest excited state, A (having significant contributions from ${}^6\text{P}_{7/2}$, and ${}^6\text{D}_{7/2}$), to the ground state, Z (${}^8\text{S}_{7/2}$) and is attributed to the long-lived component (with a fluorescence decay time of 147 μs); whereas the short-lived component having featureless emission observed in 590-610 nm region is expected to arise due to emission from the organic species. Since the fluorescence of Cm(III) in aqueous solution was reported at 594 nm with a life-time of 68 μs based on laser induced fluorescence studies, the shift in the emission peak towards the higher wavelength in the present case, could be due to the complexation with α -HIBA and the increase in the lifetime can be attributed to the exclusion of hydrated water molecules from the first coordination sphere of Cm(III) by α -HIBA.

Fluorescence lifetime measurements provide information on the composition of the first coordination sphere of Cm(III). A linear correlation between the decay rate and the number of H_2O molecules in the first coordination sphere of Cm(III) was found by Kimura and Choppin [5]. The Cm(III) aquo ion is characterized by a measured lifetime of $68 \pm 3 \mu\text{s}$ corresponding to $\text{Cm}(\text{H}_2\text{O})_9$. Increasing lifetimes of Cm(III) species reflect the exclusion of water molecules out of the first coordination sphere of the Cm(III) due to complex formation reactions as given in equation (1) [5].

$$n(\text{H}_2\text{O}) = 0.65k_{\text{obs}} - 0.88 \quad (1)$$

where k_{obs} [ms^{-1}] is the reciprocal lifetime of the excited state.

The value of $n_{\text{H}_2\text{O}} \cong 3$ suggests the replacement of 6 water molecules by α -HIBA indicating that α -HIBA acts as a bidentate ligand and three molecules of α -HIBA are bound to Cm(III).

Conclusions

Using ion exchange procedure ${}^{248}\text{Cm}$ was separated from aged ${}^{252}\text{Cf}$ solutions. The detailed analysis showed the presence of other isotopes of Cf and Cm viz. ${}^{249-251}\text{Cf}$ and ${}^{246}\text{Cm}$.

The separated ^{248}Cm was used to record the fluorescence spectra of the Cm(III) complex with alpha-HIBA. Nuclear data on the abundance of various gamma emissions of ^{249}Cf were ascertained.

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Table 1: Estimated amounts of Cm and Cf by alpha spectrometry in original samples

Sample of 20µL from 10mL was planchatted in duplicate

Activity of std source = 236000 dpm

Alpha spectrometry gave the activity = 125392/300s

$$\frac{125392}{300}$$

Efficiency of the detector = ----- = 0.1062 = 10.6%

$$\frac{236000}{60}$$

Sample	Nuclide	Time s	Energy (MeV)	Accumulated counts	dps	Mean dps
Sample 1	²⁴⁸ Cm	16200	5.07	4729	2.748	Cm=2.325
	²⁵² Cf	16200	6.13	4622338 (cum)	2686.71	
	²⁵⁰ Cf	"	6.03			
Sample 2	²⁴⁸ Cm	15425	5.07	3570	2.179	Cf = 2600
	²⁵² Cf	15425	6.13	4117140 (cum)	2513.3	
	²⁵⁰ Cf	"	6.03			

Table 2: Fission gamma counting by NaI(Tl) to follow Cf by anion and cation exchangers
 Background (bkgd) : 4612/444s = 606/ min.

Anion exchanger		cation exchanger	
Fractional effluent	counts	Effluent	counts
Loading L1	bkgd	Loading L1	bkgd
L2	bkgd	L2	bkgd
L3	bkgd	L3	bkgd
L4	bkgd	washings W1	bkgd
Washing W1	bkgd	W2	bkgd
W2	bkgd	Elution E1 to E8	bkgd
W3	bkgd	E9	834
Elution E1	712	E10	936
E2	1889	E11	1006
E3	1609	E12	1555
E4	1117	E13	1481
E5	972	E14	1092
E6	938	E15	845
E7	833	E16	836
E8	723	E17	825
E9 onwards to E20	10-80 above bkgd	E18 onwards to E25	10-80 above bkgd

Table 3: Relative abundance of gamma rays of ²⁴⁹Cf with respect to 387 keV line

Energy (keV)	Relative abundance (Present work)	Relative abundance (Literature) [4]
333	4.14	4.25
252	25.39	24.17
266	90.61	89.19
387	100	100

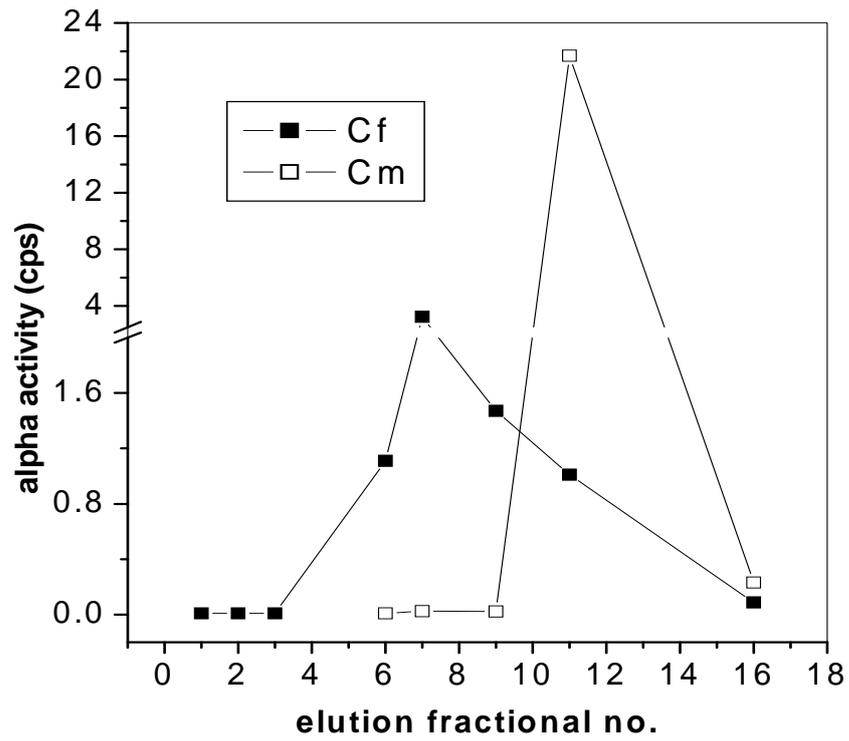


Fig.1: Typical plot based on alpha spectrometry using anion exchange resin for Cm-Cf separation
eluent: 85% methanol in 0.5M HNO₃

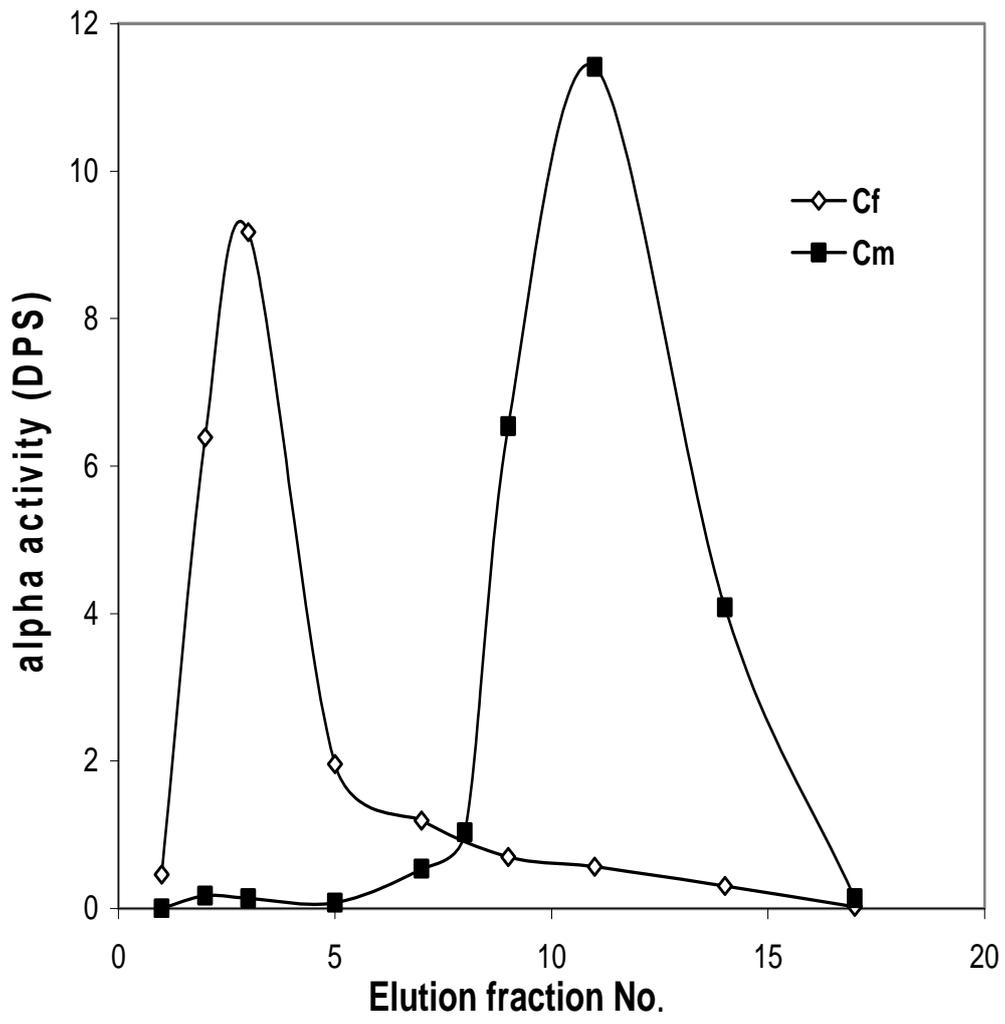


Fig.2 Cf-Cm separation by cation-exchange column

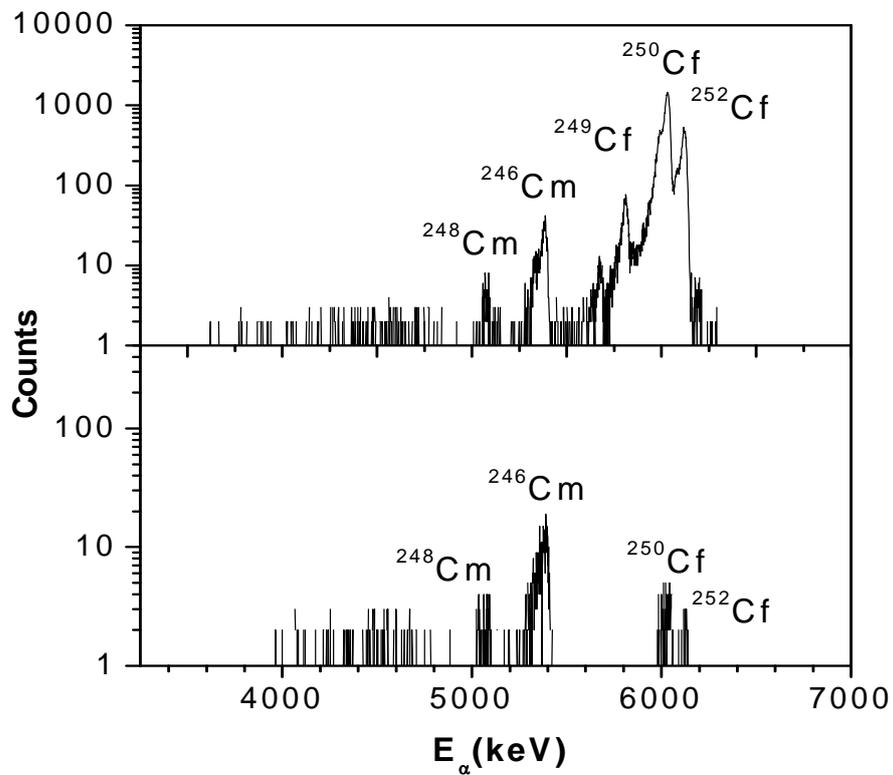


Figure 3. Alpha spectrum of original (top) and separated (bottom) Cm fraction

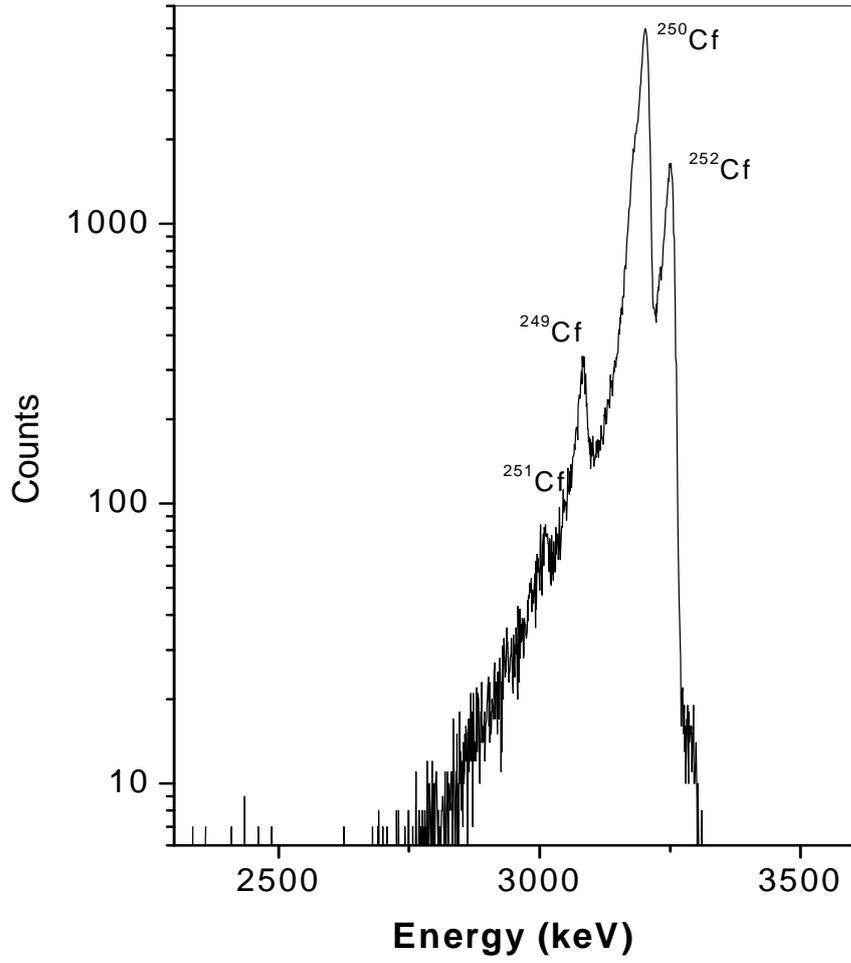


Fig.4 Alpha spectrum of the Cf fraction

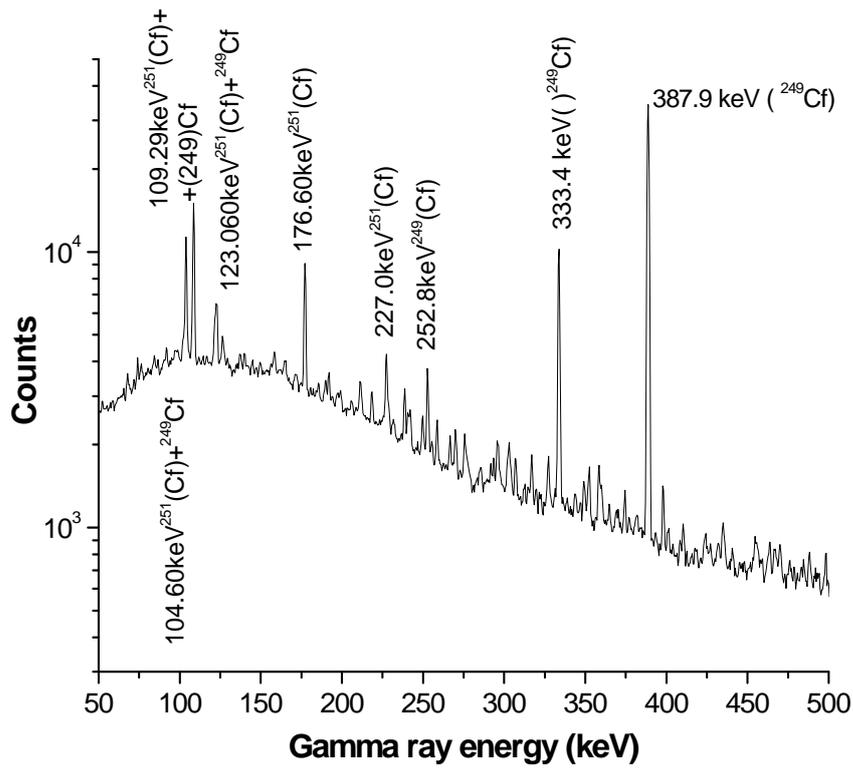


Fig.5 Gamma ray spectrum of ^{249}Cf and ^{251}Cf

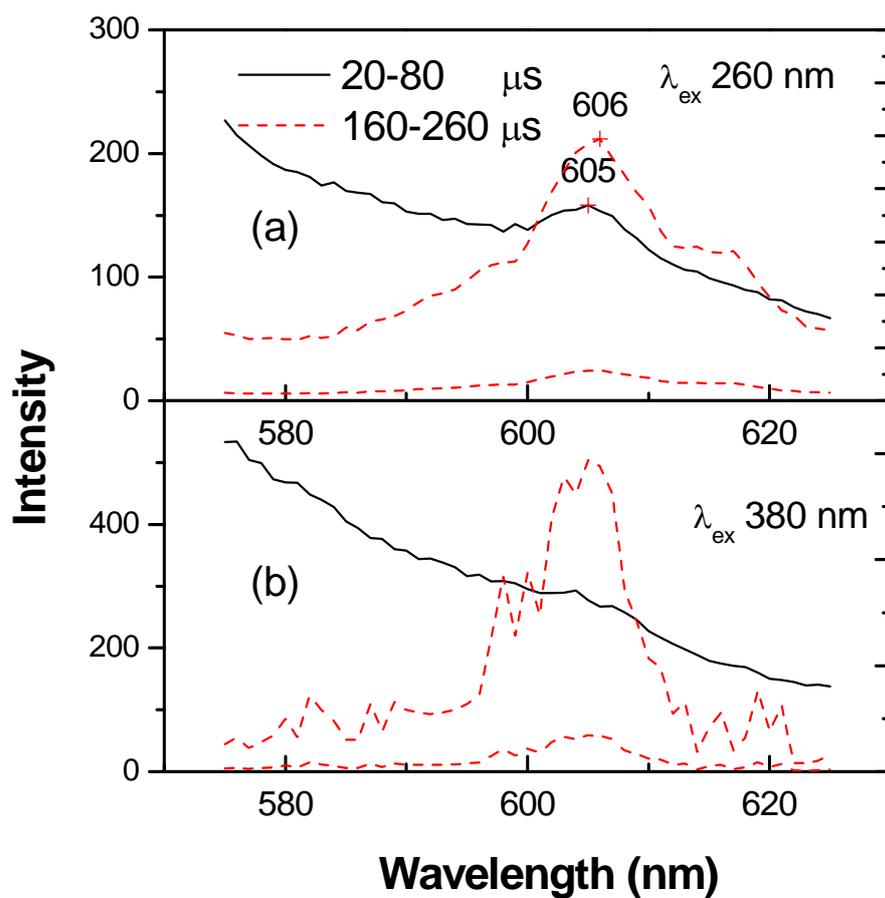


Figure 6. Emission spectra of Cm-HIBA complex (a)excitation at 260 nm (b) excitation at 380 nm