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THE DETERMINATION OF PROMETHIUM-147 AND SAMARIUM-151 USING EXTRACTION CHROMATOGRAPHY

ABSTRACT

Methodology is described for the analysis of promethium and/or samarium in a variety of environmental and nuclear waste matrices. Lanthanides are separated from the bulk matrix by coprecipitation with calcium phosphate. Conditions for the use of extraction chromatography using a commercially available material (Ln Resin) were optimised and the elution profiles of a number of possible radiometric interferences and yield tracers determined. The use of ^{148}Gd as a radiometric yield tracer was investigated, however it was found to co-elute with europium. Hence it was inappropriate as a yield tracer for the analysis of the ^{147}Pm content of fission product mixtures. The use of natural samarium was however successful, therefore yield determination was undertaken by samarium measurement on a sample aliquot using Inductively Coupled Plasma - Optical Emission Spectrometry with the remainder of the ^{147}Pm fraction analysed by Liquid Scintillation Counting. The methodology was extended to allow the isolation of a pure samarium source from which the ^{151}Sm activity was determined. A variety of materials was analysed using this methodology and the results are reported.

1 INTRODUCTION

The radionuclides ^{147}Pm and ^{151}Sm are both pure beta emitting fission products with relatively high fission yields among the longer lived ($t_{1/2}$ > 1 year) radionuclide byproducts of the nuclear fuel cycle. The half-lives, emission energies and typical abundance (for fission of Magnox fuel) relative to ^{137}Cs for all major lanthanide fission products are shown in Table 1. Magnox plant is the first generation of nuclear power stations operating in the UK, using non-enriched uranium fuel, graphite moderation and CO_2 primary coolant.

Although low, the radiotoxicity[2] of ^{147}Pm (2.6E-10 Sv/Bq by ingestion (adult)) is comparable to that of ^{14}C and, due to its relatively long half-life, it is environmentally significant and is therefore measured in nuclear waste material.

Until recently anion exchange chromatography using a highly cross-linked cation exchange resin (BioRad AG 50W-X16) has been used successfully by this laboratory[3] for the determination of ^{147}Pm. Elution of promethium and its gravimetric yield determinant, neodymium, was effected selectively with ammonium α-hydroxyisobutyrate (α-HIBA). However, the resin is no longer available and alternative methodology was sought.

The lower abundance in discharges made under authorisation and the lower radiotoxicity of ^{151}Sm (9.8E-11 Sv/Bq by ingestion (adult)) have, until this time forestalled the requirement for the analysis of this species. However, as Table 2 shows, the calculated maximum theoretical activity of ^{151}Sm in waste consignments destined for final disposal approaches the *de minimis* level, above which formal reporting is required.

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2 SURVEY OF ALTERNATIVE METHODOLOGY

A survey of the literature revealed a diverse selection of alternative methodology. The use of cation exchange chromatography using less cross-linked resins (typically 8 % cross-linked) has been the most common technique. However the chemical similarity between the lanthanides required either: further chemical purification,[4] heating of the anion exchange columns in order to enhance the kinetics of the system[5-7] or use of long anion exchange columns of low particle-sized resin under considerably enhanced pressure.[8] More recently,[9] the latter methodology has been improved by high performance liquid chromatography enabling the determination of both ^{147}Pm and ^{151}Sm.

As early as 1961 bis(2-ethylhexyl) phosphoric acid (HDEHP) was used for the isolation of ^{147}Pm using multiple solvent extraction steps from nitric acid solutions of varying concentrations.[10] Peppard et al[11,12] reported that the distribution coefficient of this system is inversely proportional to the third power of the hydrogen ion concentration. HDEHP has been immobilised on a variety of inert supports in order to facilitate extraction chromatography.[13,14] However, recently a commercially available resin has reportedly been used in the determination of ^{147}Pm.[15,16] The material (Ln Resin™ SPS) consists of HDEHP loaded at 40 % w/v onto Amberchrom™ (particle size 80-160 μm). In laboratory studies Burnett et al[15] successfully determined ^{147}Pm with this material using both natural samarium and ^{148}Gd as gravimetric and radiometric yield tracers, respectively. Similarly, Sigg et al[16] reported the successful use of Ln Resin in the determination of lanthanides in nuclear waste. However, while there was no requirement for these workers to remove ^{154}Eu and ^{155}Eu from the promethium source the data in Table 1 show that considerable bias might be expected if europium radionuclides were not removed successfully from Magnox fission product mixtures.

3 METHOD DEVELOPMENT

3.1 Use of ^{148}Gd as a Radiometric Yield Tracer for ^{147}Pm

In the absence of a suitable elemental yield tracer for ^{147}Pm the utility of ^{148}Gd was investigated, as described by Burnett et al.[15] They had transferred a sample solution to 1 g of Ln Resin and rinsed with a volume of 0.2 M nitric acid to remove most interferences. Co-elution of promethium and gadolinium with 1.0 M nitric acid allowed radiometric yield determination of the gadolinium alpha emission (3.27 MeV) by liquid scintillation counting with α/β-discrimination. Adaptation of this chromatographic system was undertaken based on the studies of Sigg et al.[16] in order to satisfy our requirement for the removal of europium species, however it was observed that the behaviour of gadolinium on Ln Resin was unfortunately analogous to that of europium rather than promethium. Figure 1(a) shows the elution profile of a mixture of ^{241}Am, ^{147}Pm, ^{152}Eu and ^{148}Gd on 0.7 g of Ln Resin. The unsuitability of ^{148}Gd as a yield tracer for ^{147}Pm is apparent as it was not possible to separate europium radionuclides from the ^{148}Gd yield tracer. This observed similarity in the separation factors of gadolinium and europium as a consequence of the so-called 'double-double' effect has been documented.[13]

3.2 Use of Natural Samarium as a Radiometric Yield Tracer for ^{147}Pm

In the same manner as the Gd-Eu pair, the electronic structures of samarium and promethium are such that their properties are extremely similar, thus samarium should be a more suitable choice than gadolinium as a yield tracer for promethium. Elemental samarium contains two naturally occurring long-lived isotopes of which ^{147}Sm (abundance 14.97 %, half-life 1.06E11 years by α-emission) dominates the specific activity at a level of 127 Bq.g^{-1} over ^{148}Sm (abundance 11.24 %, half-life 7E15 years by α-emission). Our methodology utilises an initial spike to the sample of 1 mg of samarium with its

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recovery determined by ICP-OES. As this addition is at such a low concentration the samarium alpha activity can be readily eliminated by α/β-discrimination during liquid scintillation counting (LSC).

Figure 1(b) shows the performance of a 1.6 g (6 cm) Ln Resin column in the separation of a mixture containing samarium, [241]Am, [147]Pm and [152]Eu. Although a degree of speciation is apparent between samarium and promethium a fraction containing only these elements may be separated from the most problematic interferences, americium and europium under the following conditions. Americium was removed in the initial rinse of 0.15 M nitric acid, together with most other species. The Sm/Pm fraction was then eluted in 0.25 M nitric acid and europium, should it be required may be removed in 0.4 M nitric acid. ICP-OES determination of an aliquot of the Sm/Pm fraction gives the chemical yield and the [147]Pm content of the sample is determined by LSC of the remaining fraction.

3.3 Isolation of Pure Samarium from a Sample Mixture Containing Promethium, Europium and Americium Radionuclides

It is also apparent from Figure 1(b) that while promethium is completely removed from the column in the first 40 cm^3 of the 0.25 M nitric acid rinse, less than 40 % of the samarium content is removed at this stage. Thus [151]Sm was determined by using the same column geometry and conditions as for [147]Pm, but rather than collecting the whole 120 cm^3 of 0.25 M nitric acid rinse, the first 40 cm^3 of eluate was discarded and the remaining 80 cm^3 collected as the samarium fraction. The samarium yield was determined (as above) and a liquid scintillation source was prepared in Ultima Gold AB™ and counted in a low energy window using α/β-discrimination in order to exclude the natural samarium alpha activity. In the absence of a certified standard the counting efficiency of [151]Sm (E_{max} 76 keV) was determined using a [63]Ni standard (E_{max} 66 keV) and an allowance was made for any discrepancy introduced in this manner in the uncertainty budget.

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3.4 Ln Resin Capacity Studies

Burnett et al[15] reported saturation of an Ln Resin column (0.7 g) by less than 10 mg of samarium, therefore we sought to determine the actual limit for samarium. A 0.7 g column was conditioned in 0.15 M nitric acid and spiked with successive additions of samarium (1 mg) and ^{147}Pm (7500 dpm), followed by a rinse of 0.15 M nitric acid (10 cm^3). Successive rinses were collected and counted by LSC. Breakthrough was observed after loading with 6-7 mg samarium per 0.7 g of Ln Resin. Our methodology utilises 1 mg of samarium as the yield determinant in a procedure utilising 1.6 g of extractant. Gravimetric yield determination of the samarium yield tracer is possible in the absence of ICP-OES, with up to 15 mg of samarium capable of being retained by 1.6 g of Ln Resin..

Calcium is a common bulk matrix interference for the separation of lanthanides in the majority of environmental and waste materials and it was hoped to utilise the presence of calcium by inclusion of a calcium phosphate pre-concentration step. However, the possibility of extractant saturation by calcium was a concern which was addressed by a similar study to that detailed above. It was determined that up to 600 - 800 mg of calcium may be tolerated by 1.6 g of Ln Resin in the absence of rare earth salts.

In a similar experiment it was determined that approximately 20 mg of iron (III) led to loss of promethium retention by a 1.6 g Ln Resin column, the effect of ascorbic acid reduction of iron (III) to iron (II) was negligible. It is therefore due to concerns over the possibility of iron saturation that the methodology detailed herein concentrated on characterisation of the larger 1.6 g column geometry.

3.5 Removal of Plutonium Interference

Early studies indicated considerable plutonium interference in the liquid scintillation spectrum of the

isolated sources, with ^{241}Pu contamination of both Pm/Sm and Sm fractions. Therefore a further clean-up step using Eichrom TRU Resin™ was incorporated, see Figure 2. *- incorporated after the separation*

4 RESULTS AND DISCUSSION

4.1 Determination of ^{147}Pm and ^{151}Sm in Estuarine Sediment and Nuclear Waste Samples

In order to fully validate the separation procedure summarised in Figure 2 a range of samples of different matrix and activity level were analysed:

4.1.1 Intertidal Sediment Natural Matrix Reference Material. Although its radionuclide composition has been widely characterised[17] analysis for ^{147}Pm or ^{151}Sm has not, to our knowledge, been undertaken on this material from the Cumbrian coastline. Approximately 1 g aliquots of ashed material were microwave digested and the liquor analysed using the new procedure.

4.1.2 Low Level Waste Quality Control (QC) Digestion Liquor. A set of QC solutions is available within the laboratory for use in the routine work programme of low level waste assay. Four solutions have previously been prepared using a blank swab dissolution liquor and a blank filter paper liquor, each spiked (using calibrated radionuclide standards of ^{45}Ca, ^{54}Mn, ^{60}Co, ^{55}Fe, ^{63}Ni, ^{90}Sr, ^{137}Cs, ^{147}Pm, ^{238}Pu, ^{239}Pu, ^{241}Pu and ^{241}Am) at levels corresponding to either a typical Advanced Gas-Cooled Reactor (AGR) radionuclide composition or a typical Magnox mixture.

4.1.3 Intermediate Level Waste (ILW) Dissolution Liquor. Two samples of Active Effluent Treatment Plant (AETP) Sludge dissolution liquor corresponding to approximately 0.25 g each were

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analysed. This material originated from the back-washings of sand filters on a Magnox plant. Additionally a sample of Caesium Removal Ion-Exchange Resin (Lewatit DN) corresponding to approximately 0.5 g of dry sample was analysed.

4.1.4 Magnox Final Monitoring Delay Tank (FMDT) Liquid Effluent Annual Bulk (1997). A volume of 50 cm^3 of liquid effluent representative of the annual discharge from a Magnox station was analysed in six-fold replicate.

The results of these analyses, shown in Tables 3-5, demonstrate that the methodology is applicable to a wide range of sample matrices. Furthermore, its use for the determination of ^{147}Pm and ^{151}Sm in relatively fresh (effluent) and aged (ILW) fission product mixtures demonstrates that the decontamination from other interfering species is sufficient for their isolation from a broad range of radionuclide mixtures. Matrix problems were however the cause of low chemical recoveries in the analysis of the intertidal sediment and all results on this sample were below the limit of detection.[18] The minimum detectable activity (MDA) obtained for the sediment samples was in the region of 20 Bq.g^{-1}.

4.2 Promethium-147: Chemical Recovery, Accuracy, Precision and Limit of Detection

The mean chemical recovery for ^{147}Pm in the low level waste QC digestion liquor was 80 % (7 replicates), however it was observed that when the yield was below 50 % discrepant (low) results arose. It is likely that early breakthrough of both natural samarium and ^{147}Pm occurred, however due to the differing elution properties of these species losses of promethium occurred which were unaccounted for by the samarium yield tracer. Therefore an acceptance criterion requiring a minimum 50% yield was set, below which results were rejected. Use of a yield tracer of a differing element has always been the main weakness in ^{147}Pm analysis and it is believed that on occasion differences in

column packing caused the elution profile to slip, leading to unmonitored losses of promethium activity. It is for this reason that when this methodology is conducted on routine samples a minimum of duplicate concordant analyses may be required.

Of the accepted LLW QC results (n = 7), see Table 3, all data were within 10% of the target activity and were satisfactorily within the 95 % confidence limits, confirming the accuracy of the methodology for [147]Pm. The standard deviation of the data set from the known activity was calculated as 5.4 %. This quantifies the precision attainable with the methodology and includes such variables as speciation between analyte and tracer (at yields greater than 50 %). This precision component, together with the certified uncertainty associated with the [147]Pm standard, the yield determination and the counting uncertainty dominate the uncertainty budget for [147]Pm determination. Furthermore, it should be noted that if [147]Pm is determined in aged material it may be necessary to compensate for the presence of [151]Sm. Such a correction has been made for all samples analysed in this study.

The MDA obtained for [147]Pm in the ILW samples was in the region of $0.02 Bq.g^{-1}$ and in liquid effluent was approximately $2 Bq.L^{-1}$.

4.3 Samarium-151: Chemical Recovery, Accuracy, Precision and Limit of Detection

The mean chemical recovery of [151]Sm in FMDT liquid effluent was 87% using the extraction chromatography separation outlined in Figure 2, however in two of the six replicates the liquid scintillation spectra were dominated by traces of residual [147]Pm. The remaining spectra appeared free of interference but in order to accomodate any systematic uncertainty due to the presence of promethium impurity the uncertainty budget for these data included a component accommodating the standard deviation of the satisfactory analyses.

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The method was modified for the analysis of the ILW samples in order to include a repeat of the Ln Resin separation, thereby doubling the effectiveness of the decontamination from [147]Pm. Figure 3 shows the liquid scintillation spectrum of the resulting [151]Sm, together with those of [63]Ni and [147]Pm. The similarity in E_{max} between the [63]Ni and [151]Sm spectra demonstrates the validity of using [63]Ni to determine the counting efficiency of [151]Sm. The resulting liquid scintillation spectra (see Figure 3) were devoid of any traces of [147]Pm and this repeat separative step will be used in future [151]Sm analyses. Although the improved purity was at the cost of the chemical recovery (see Table 5) in all future [151]Sm analyses the repeated column separation step will be included.

The uncertainty budget for [151]Sm includes components for the use of [63]Ni as a counting efficiency standard for [151]Sm (nominally 5 %), sample and standard measurement uncertainties, yield determination and counting uncertainty. Results clearly in excess of the MDA were obtained for the effluent and ILW samples.

The lowest MDA obtained for [151]Sm in the ILW samples was in the region of 0.02 Bq.g[-1] and in liquid effluent was approximately 2 Bq.L[-1].

4.4 Samarium-151 Activity Levels

Reports of [151]Sm determination are rare in the literature,[9] however we determined the activity concentration of this radionuclide in aged intermediate level waste (between 8 and 34 years decayed) and in relatively fresh liquid effluent (approximately 1 year from discharge). Samarium-151 was determined in all of the intermediate level waste samples at levels of approximately 80% of the [147]Pm activity. The origin of this material was a power station currently undergoing decommissioning and which ceased operation in 1990. Since generation of the fission products the proportion of [151]Sm activity

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activity. The origin of this material was a power station currently undergoing decommissioning and which ceased operation in 1990. Since generation of the fission products the proportion of ^{151}Sm activity relative to ^{147}Pm has increased due to the more rapid decay of the latter.

Samarium-151 was also determined in the FMDT liquid effluent annual bulk. The ratio of ^{147}Pm to ^{151}Sm in this sample was 75 ± 24. The FISPIN data in Table 1 indicate that this ratio at a decay period of 4 years would be 25, reflecting the more recent genesis of this material.

4.5 Further Work

The methodology described herein has enabled time savings of the order of 70 % to be introduced into the analysis of ^{147}Pm. Additionally, novel methodology for the determination of ^{151}Sm has been validated. In theory however it is possible to conduct both ^{147}Pm and ^{151}Sm determinations on a single sample using a sequential procedure: if the methodology described for ^{151}Sm analysis is followed, but the 40 cm^3 rinse containing ^{147}Pm is retained and its ^{151}Sm yield determined and summed with that of the second (pure samarium) fraction the overall Sm/Pm yield would be evident. Each fraction would then be determined by liquid scintillation counting. The uncertainty associated with this methodology would however be greater and it is unlikely that the methodology would be as robust. However, if a large throughput of ^{151}Sm determinations was necessary, or if the fraction of ^{151}Sm present in an aged sample too large to be accommodated as a systematic uncertainty element in the uncertainty budget, further modification of the method in this way may be justified.

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5 CONCLUSIONS

Methodology for the assay of ^{147}Pm and ^{151}Sm has been successfully validated. It utilises natural samarium as a yield tracer with chemical recovery determined by ICP-OES. The use of ^{148}Gd as a radiometric yield determinant was found to be unsuitable for the determination of ^{147}Pm in the presence of europium radionuclides typically present in fission product mixtures. The capacity of the commercially available extraction resin was determined for samarium (9 mg.g^{-1}), calcium (400 mg.g^{-1}) and iron (12 mg.g^{-1}). *check figures of Webster etc using ICP-MS to see if they are above tolerance of method.

Decontamination of a Pm/Sm containing fraction from mixtures of activation and fission products using extraction chromatography gave rise to radiochemically pure sources, however in aged nuclear material it was found to be particularly necessary to correct the ^{147}Pm activity for the presence of ^{151}Sm.

Pure samarium was itself isolated via a repeated extraction chromatography procedure, with its yield also determined by ICP-OES. Liquid scintillation counting of low levels of ^{151}Sm activity necessitates the use of α/β-discrimination by pulse-shape analysis. Use of ^{63}Ni as an analogue for ^{151}Sm enabled determination of the liquid scintillation counting efficiency and a factor accounting for additional uncertainty due to its use was included in the uncertainty budget.

The ^{151}Sm content of waste arisings has been characterised. The ratios of ^{147}Pm to ^{151}Sm in recent liquid effluent discharges to the environment and in aged intermediate level waste are 75 ± 24 and 1.3 ± 0.2 respectively. These data are consistent with the radionuclide decay profile of the materials.

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Table 1. *Nuclear Decay and Fission Product Inventory Data for Lanthanide Fission Products.*

Radionuclide	Half-life (years)	beta E_{max} (MeV)	Relative abundance by activity[†]
Ce-144	0.78	0.318 (+Pr-144 at 2.996)	0.23
Pm-147	2.6	0.225	0.79
Sm-151	88.7	0.076	0.0031
Eu-154	8.59	0.580	0.019
Eu-155	4.85	0.160	0.026

[†] *Ratioed to ^{137}Cs and based on FISPIN code calculations[1] at a Magnox fuel burn-up of 5 GWD/T and a decay period of 4 years.*

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Table 2. *Assessment of 'Low Abundance' Isotopes Compliance with de minimis Criteria for Disposal of a Theoretical Waste Consignment* [‡]

Radionuclide	Maximum Theoretical Activity Per Consignment (MBq)	*de minimis* (MBq)
Se-79	0.0075	13
Y-91[††]	**10300**	**13**
Zr-93	0.0047	13
Nb-93m	0.029	13
Tc-99	0.37	13
Pd-107	0.002	13
Sn-121m	0.28	13
Sn-126	0.019	13
I-129	0.00069	0.05
Cs-135	0.019	13
Sm-151	**6.3**	**13**

[‡] *Where a consignment is defined as 14.4 m^3 of waste contained in 72 drums (each nominally of 200 litre volume) packed in an ISO container.*

[††] *[91]Y has 58.5 day half-life so after 2.5 years will be below de minimis.*

Table 3. Determination of ^{147}Pm in spiked blank low level waste dissolution liquor.

Sample ID	Blank matrix description	Yield (%)	Activity (Bq.L^{-1})	Certificated activity	Deviation (%)
1	Magnox: filter paper	91	278 ± 26	302	–8.1
1	Magnox: filter paper	54	317 ± 33	302	5.0
1	Magnox: filter paper	68	312 ± 30	302	3.3
2	AGR: filter paper	91	57 ± 7	60	–4.9
3	Magnox: Tak cloth	94	286 ± 26	302	–5.4
3	Magnox: Tak cloth	88	299 ± 28	302	-1.0
4	AGR: Tak cloth	85	55 ± 7	60	–8.9

Table 4. Determination of ^{147}Pm and ^{151}Sm in FMDT liquid effluent.

Promethium-147 Analysis		Samarium-151 Analysis	
Yield (%)	*Result*	*Yield (%)*	*Result*
81	*704 ± 50*	*86*	*10.8 ± 3.2*
77	*721 ± 51*	*85*	*8.76 ± 2.72*
58	*669 ± 47*	*88*	*8.02 ± 2.52*
90	*706 ± 49*	*87*	*9.57 ± 2.91*

Table 5. Summary of Results.

Sample description (and reference date)	Promethium-147 Analysis		Samarium-151 Analysis		Units
	Yield (%)	Result	Yield (%)	Result	
FMDT liquid effluent annual bulk (15/06/97)	72 [a](4)	698 ± 49	87 [a](4)	9.28 ± 2.83	Bq.L^{-1}
AETP sludge dissolution liquor - A	76	2.40 ± 0.20	15	1.92 ± 0.22	Bq.g^{-1}
AETP sludge dissolution liquor - B	62	1.35 ± 0.15	46	0.949 ± 0.099	Bq.g^{-1}
ILW resin sample (Lewatit DN)	35	18.3 ± 1.4	14	13.0 ± 1.0	Bq.g^{-1}
LLW QC spiked solution	82	(-2.8 ± 10.8) % [b]	-	-[c]	-

[a](n) Denotes mean data (number of data points, n)

[b] Deviation from known activity ± 2σ sample standard deviation.

[c] No ^{151}Sm standard available.

Figure 1. *(a) The Elution Profile of Promethium and Gadolinium in the Presence of Americium and Europium on a 0.7 g Ln Resin column (above) and,(b) the Elution Profile of Promethium and Samarium in the Presence of Americium and Europium Under Volume-Optimised Conditions on a 1.6 g Ln Resin Column (below).*
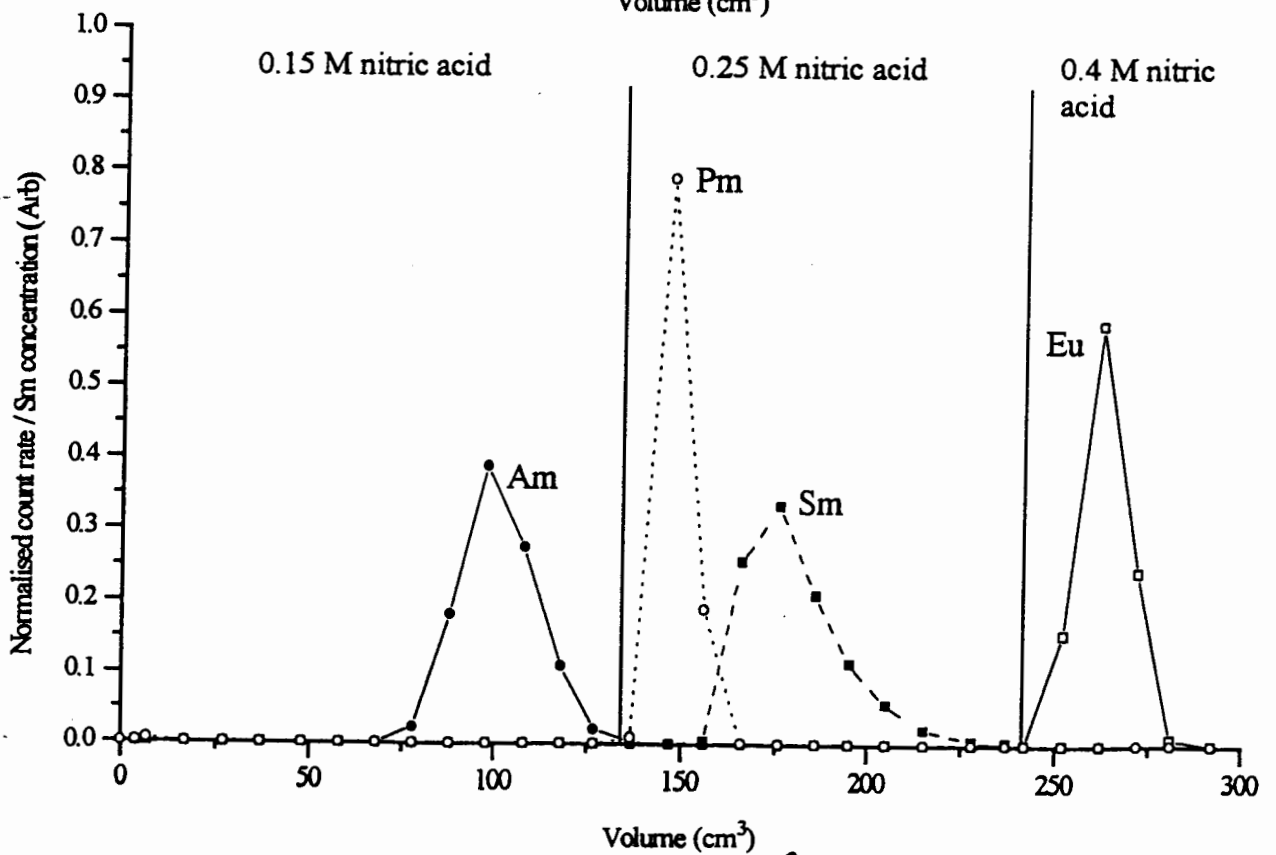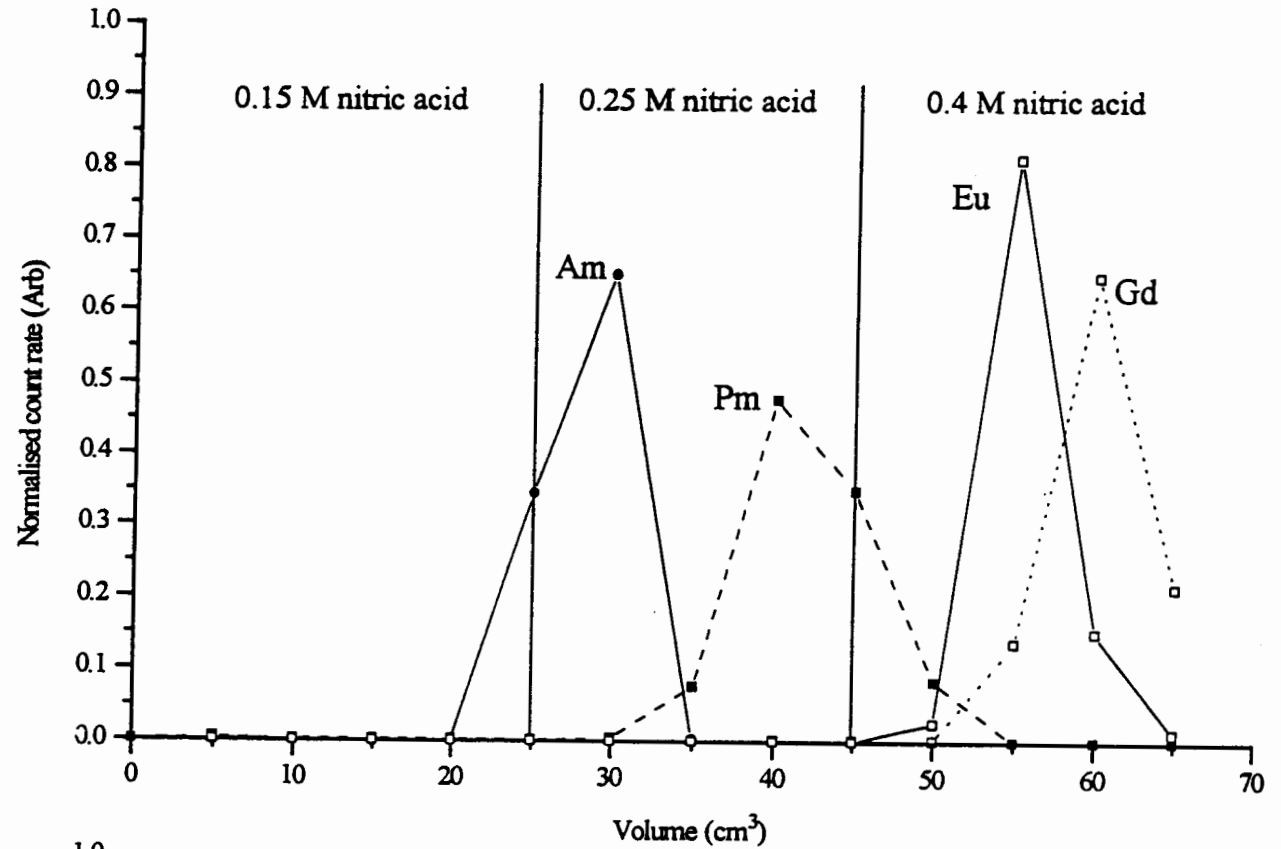

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Figure 2. *Schematic of the method for the determination of ^{147}Pm and ^{151}Sm using Ln Resin.*
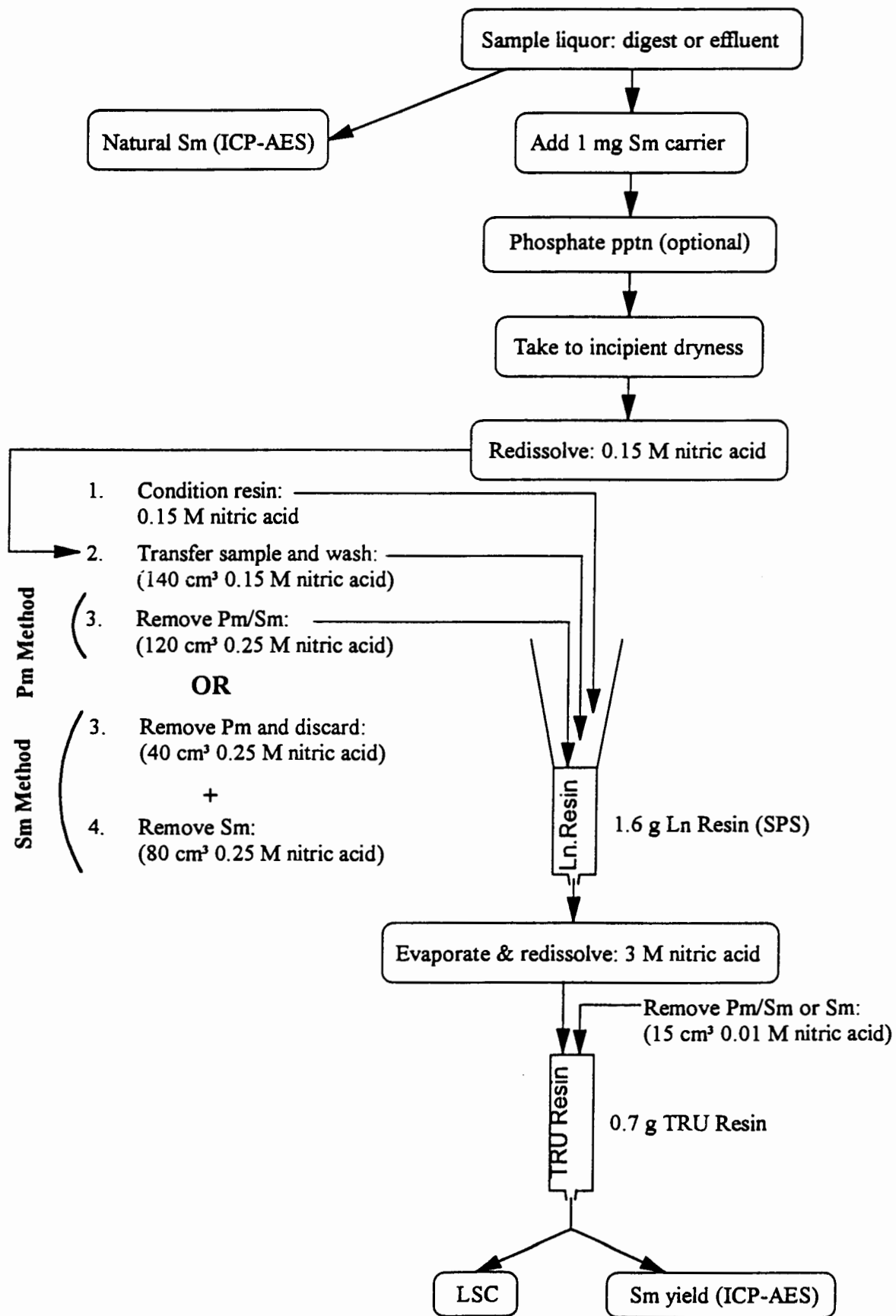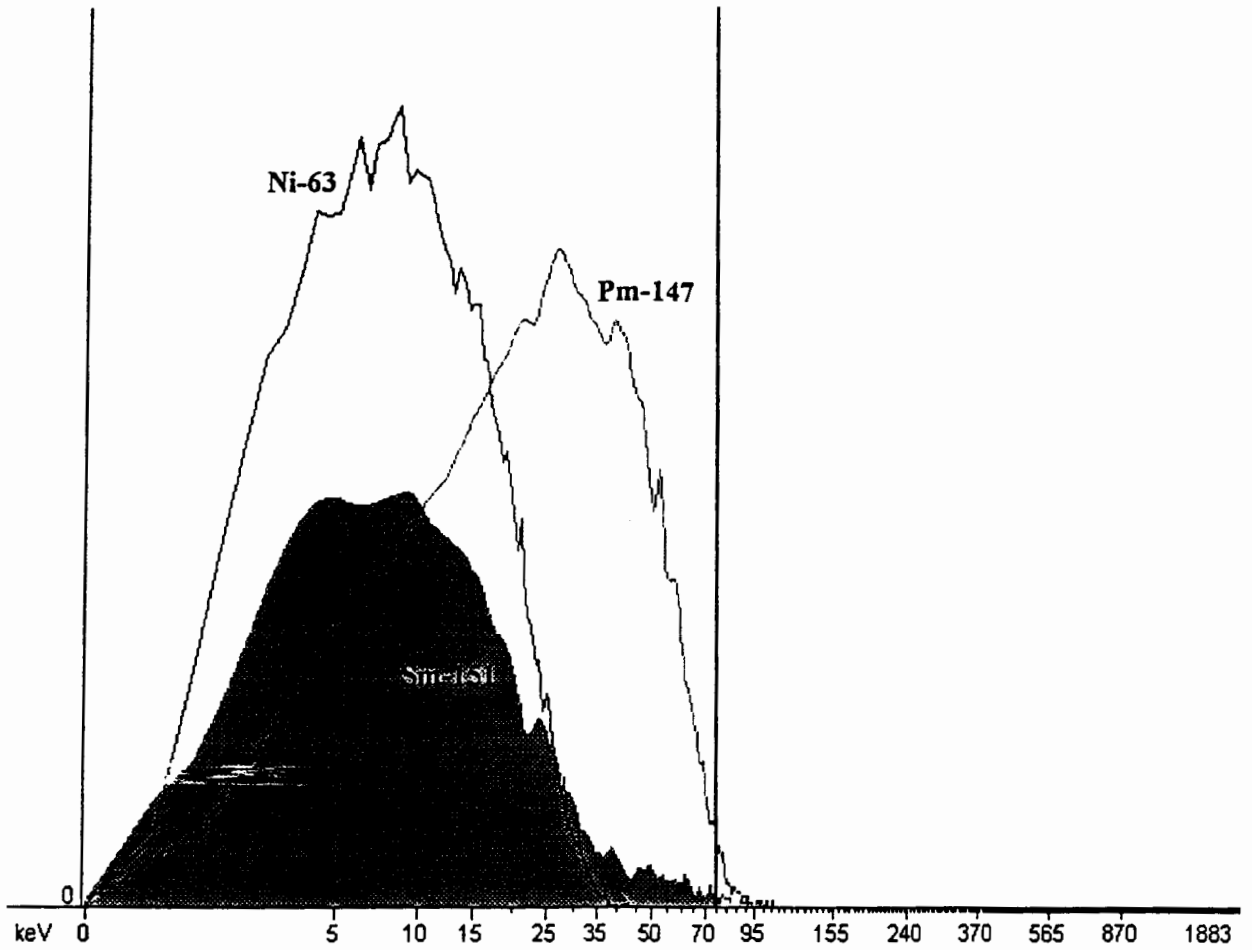
Figure 3. *Liquid scintillation spectra of ^{147}Pm and ^{63}Ni standard solutions and the ^{151}Sm spectrum from AETP sludge dissolution liquor A.*

^{147}Sm   x   1.96 MeV   abundance   14.97 % $t_{\frac{1}{2}}$ 1.06×10^{11} y

^{148}Sm   x   2.223 MeV   "   11.24 % $t_{\frac{1}{2}}$ 7×10^{15} y

^{147}Sm   sp. activity   127 Bq g^{-1}

^{148}Sm   "

18 µg Sm