ASSAY OF PLUTONIUM CONTAMINATED WASTE BY GAMMA SPECTROMETRY

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ABSTRACT

The extreme toxicity of plutonium necessitates the segregation of plutonium contaminated materials (PCM) with extremely small (sub-μg) levels of contamination. The driver to measure accurately these small quantities of plutonium within (relatively) large volumes of waste is (in part) financial. In particular the cost of disposal (per unit volume) rises steeply with increasing waste-category.

Within the UK, there has been a historical reluctance to use low energy gamma radiation to sentence PCM because of the potential for self attenuation by dense materials. This is unfortunate because the low-energy gamma radiation from PCM offers the only practicable technique for segregating PCM within the various Low Level Waste (LLW) (>0.4Bq/g) and sub-LLW categories. Whilst passive neutron counting techniques have proved successful for assay of waste well into the Intermediate Level Waste (ILW) (>100Bq/g) category, a cursory study reveals that these techniques are barely capable of detecting mg quantities of plutonium – let alone the sub-μg quantities present in LLW.

This paper considers the use of two types of gamma detector for assay of PCM: the thin sodium iodide FIDLER (Field Instrument for the Detection of Low Energy Radiation) and the HPGe (High Purity Germanium) detector. Systems utilising these two types of detector can provide complementary information.

FIDLER measurements are conducted by careful, local, systematic monitoring of surfaces

By contrast a HPGe detector can be used to monitor entire walls, or even rooms, in one measurement. Thus, a HPGe detector placed in the centre of room (from which any radioactive hot-spots have previously been removed) could be used to demonstrate that the *average* activity remaining close to the surface of the walls/floor/ceiling is below a given limit.

The Monte Carlo Code MCNP 1 has been used to model both FIDLER probe and HPGe detector in the measurement geometries described above.

The MCNP simulations have been validated with experimental data.

Subject keywords: ASSAY; PLUTONIUM; WASTE; GAMMA; MCNP; MONTE CARLO; FIDLER; SODIUM IODIDE; HPGE; SIMULATED SPECTRUM

1. INTRODUCTION

Plutonium is a radioelement that has caused much debate. It is actually the heaviest primordial element, with the isotope Pu-244 having a half-life of 80M years and this isotope has been found in trace amounts on the Earth. Plutonium has two main uses which reflect the history of nuclear physics. Most of its isotopes (particularly Pu-239) undergo fission and it can thus be used firstly, to manufacture nuclear weapons and secondly, as nuclear fuel in reactors. The latter application has received a great deal of investigation over the last 40 years as plutonium is formed by multiple neutron capture on U-238 and this means that the non-fissionable U-238, which forms 99.3% by mass of uranium, can be transmuted into plutonium and thus nuclear fuel. This technology has proven difficult to engineer as it is necessary to surround a very small conventional U-235 powered fission reactor with a blanket of depleted (U-238 rich) uranium in order to obtain the correct reactor geometry and neutron physics conditions. This requirement has meant the nuclear core has had to be very small and has required cooling by materials with excellent thermal conduction and capacity properties, such as liquid sodium and potassium. These stringent requirements have thus led to complex chemical, material physics and thermal engineering problems associated with several world-wide fast (neutron) reactor programmes.

Several sites in the UK have processed plutonium for various purposes and as a consequence have generated contaminated nuclear waste materials. Although the various isotopes of plutonium decay by all of the four main processes – alpha, beta, gamma and neutron – it is in fact quite difficult to measure plutonium even at significant mass levels – a fact evidenced by some of the current Homeland Security programmes for covert nuclear material identification. This paper considers the historical assessments of plutonium contaminated wastes and proposes some new applications of existing technologies to improve measurement sensitivity.

2. HISTORICAL PROGRAMMES IN THE UK

Four main sites in the UK are associated with plutonium technologies.

These are:-

Sellafield – previously Windscale - which was the main site for extracting plutonium from spent nuclear fuel and U-238 blanket materials for the UK nuclear weapons programme. This process was responsible for the worst nuclear accident in the UK when the PILE 1 plutonium breeding reactor caught fire in 1957, with the resultant contamination of large parts of Cumbria. There are still major parts of the Sellafield site that are contaminated with plutonium – particularly some of the early chemical extraction buildings.

Dounreay – this site is in the north of Scotland and was the location of the UK fast reactor programme. This programme involved – as previously described – the use of fast reactor and sodium coolant technologies. The associated research studies and examination of spent fuel

and cladding materials in hot cells (thick walled shielded cells) led to a consequent contamination of these facilities.

Aldermaston – this is the main site associated with the production of nuclear weapons in the UK, and will thus have processed plutonium and generated contaminated waste materials in various forms.

UKAEA, Harwell – this was the major nuclear R&D site in the UK immediately after WWII and some of the original plutonium studies were conducted at this site before being transferred to the other sites (as they were created during the various nuclear power and defence programmes).

3. PROPERTIES OF PLUTONIUM

Plutonium is a complex element – it exists in six allotropic forms. Although it is a metal it has a high electrical resistance and it actually becomes denser as it melts from a solid to a liquid. Plutonium usually produces five isotopes from neutron capture processes in reactors – from Pu-238 to Pu-242 inclusive. The period of neutron irradiation dictates the isotopic mixture; thus a short irradiation period yields more of the lighter isotopes and a longer period enables more neutron captures of these isotopes to form the higher atomic weight isotopes. The length of this neutron irradiation is thus crucial in determining the isotopic fingerprint of the mixture which can range from that dominated by Pu-239 and Pu-240, known as 'A' or bomb grade plutonium, to mixtures with a relatively lower Pu-239 and Pu-240 concentration, known as 'O' or reactor grade material. Because of the smaller separation in atomic masses of the main plutonium isotopes Pu239 and Pu240 it is much harder to separate them by physical means such as gaseous diffusion or centrifuge; techniques which are used to enrich U-235 in a U-235/U238 mixture (which has an atomic mass number difference of 3 units). One possible fingerprint for plutonium material is given in Table 1 below.

Table 1 Fingerprint of plutonium

Isotope	% Activity all isotopes	Half life years
Pu-238	2	87
Pu-239	45	24,100
Pu-240	10	6,560
Pu-241	37	14.4
Pu-242	<0.01	376,000
Am-241	6	433

One important decay route - and for the purposes of this paper crucial - for plutonium is the β-decay of the shortest lived of these five radioisotopes, Pu-241 into Am-241. The importance of this process is that Am-241 has a significance gamma-ray decay emission of 60keV – and unlike all of the plutonium isotopes can be relatively easily detected by gamma spectrometry. A key

measure of this method is that although the activity of Am-241 can be measured, this still needs to be referenced to the actual plutonium content via a 'fingerprint. This will vary according to the 'burn-up', or the period of time in the reactor and its age. Figure 1 shows the ingrowth of Am-241 from Pu-241 as a function of time. For the purposes of this study a 'general' fingerprint ratio is assumed for total plutonium to Am-241 of 10:1. This will not be true in all cases – especially for bomb grade material when the plutonium is chemically processed to remove the Am-241 – but the correct assessment can be done with knowledge of the specific fingerprint of concern.

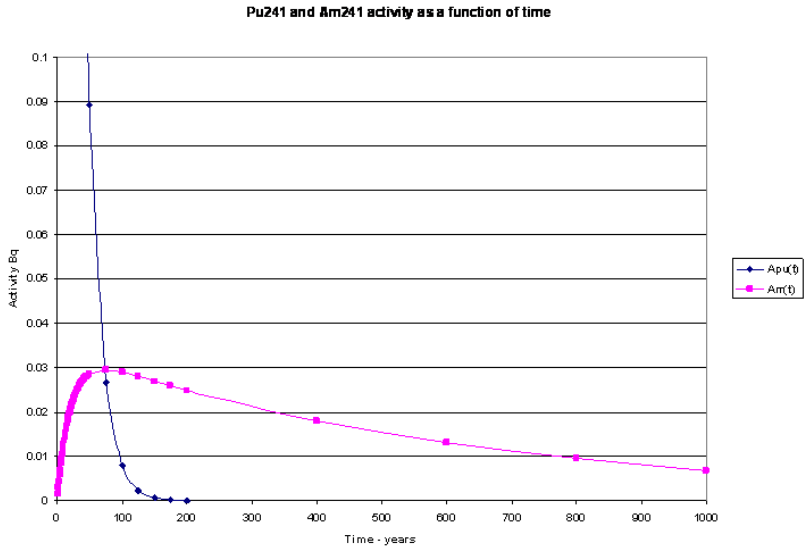


Figure 1 Am-241 ingrowth from Pu-241

4. WASTE MEASUREMENT METHODS FOR PLUTONIUM

One of the major issues with the assay of plutonium for the last few decades in the UK is that the emphasis has been placed on monitoring for significant quantities of plutonium – mainly because of the risk of a criticality event whilst processing the material. Thus the emphasis has been placed on various types of neutron monitoring systems, particularly passive neutron to measure even numbered spontaneous fission isotopes, such as Pu-240 and active neutron systems to monitor odd numbered fissionable isotopes, such as Pu239. In regard to waste assay management, criticality monitoring systems are not sensitive enough to detect plutonium at the low levels needed for the lower classifications UK waste categories. Table 2 highlights this issue in that criticality methods can only measure wastes above the 100Bq/g for PCM (plutonium contaminated material) with a probable disposal cost of at least £40,000 per m^3.

Table 2: Indicative costs for the disposal of PCM in the UK

Waste Category	α-activity limits	Cost per m^3 (GBP)
Exempt	< 0.4 Bq/g	£40
VLLW	0.4 – 4 Bq/g	£400
LLW (current UK repository)	4 – 100 Bq/g	£4 000
Other LLW	100 – 4kBq/g	£40 000 (?)
ILW	> 4kBq/g	£40 000

* Volume of all waste in category (i.e. not quantities of PCM).

As already stated plutonium is difficult to measure directly; although it does have a significant alpha decay signal, this is not easy to measure given the very limited range of the alphas (10 microns). It also has a very low energy beta decay, which again is not very penetrative. As stated above the neutron measurement techniques are insensitive for waste assay purposes and it only has low emission probability gamma-decay schemes. However, the decay of Pu-241 does yield Am-241, a radioisotope that can be readily detected by gamma methods, and even though its actual concentration is low, the resultant gamma signal is orders of magnitude greater than that from neutron and gamma detection methods based on plutonium itself. This is shown in Table 3, which compares the relative yields for neutron and gamma decay processes for A and O grade materials for plutonium with the gamma yields from Pu-239 and Am-241. It is evident from this table that the signal for the 60keV decay line from Am-241 is some seven orders of magnitude greater than for neutron methods.

Table 3: Neutron and gamma yields for assay of plutonium contaminated wastes (per gram Pu)

	SF† (n/s-g)	**α-n‡ (n/s-g)**	**Pu-239 (129 keV) (γ/s-g)**	**Am-241 (60 keV) (γ/s-g)**
'A' Grade	6.0×10^1	4.5×10^1	1.4×10^5	1.2×10^8
'O' Grade	3.6×10^2	2.0×10^2	8.7×10^4	3.2×10^9

† Neutron yield from spontaneous fission
‡ Neutron yield from α-n reactions (PuO_2)

It should be noted that although is has been stated that gamma methods can be used to monitor plutonium via Am-241 ingrowth, the actual energy of the Am-241 gamma decay line also has a limited range being only several centimetres in dense materials. Whilst this presents problems for heavy bulk items, and inhomogeneous dense wastes, gamma detection is still a practical solution in most circumstances.

The reservations about bulk monitoring methods for PCM led to large stockpiles of drummed wastes – typically many thousands of drums - on the four main UK plutonium sites. Only one of

these sites addressed the problem of these drummed wastes in detail in the 1990s and that was UKAEA Harwell. Here the Am-241 ingrowth technique was used to assay the wastes using high resolution gamma-ray spectrometry and the wastes sentenced to a low limit of LLW (Low Level <100Bq/g). The method was developed for UKAEA by Nuvia and a picture of the equipment is shown in Figure 2.

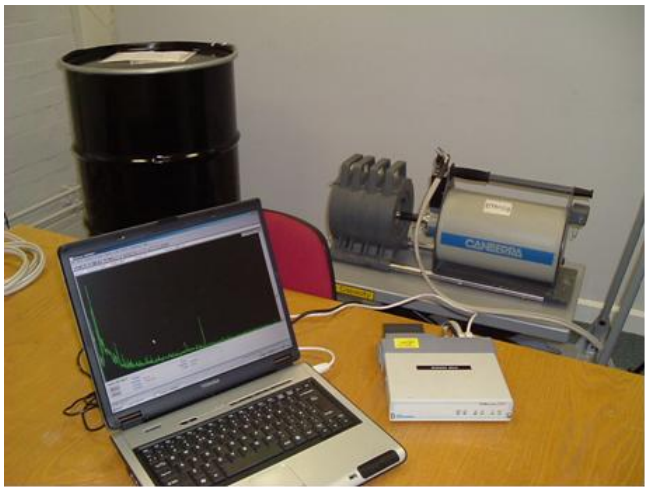


Figure 2 – High resolution gamma-ray spectrometry system for the measurement of PCM wastes

This system can - in general - sentence wastes to a level ~4Bq/g. In total some 2,500 drums were measured and reclassified by this method in 1996.

5. PROPOSED NEW MEASUREMENT METHODS FOR PLUTONIUM

The proposed new methods are really new adaptations of existing technologies and are more concerned with the overall approach of the measurement of PCM wastes. As already stated most of the measurement methods for assaying plutonium have a limited range – including the Am-241 assay technique and there has been a general approach to simply demolish facilities which have been plutonium contaminated – package the waste material into drums and store them for later assay. The weakness of this approach is that any waste assay would be most effectively conducted before decommissioning – when the plutonium will still be on the surface of walls, floors etc, rather than when it is distributed with a drum or other container. Thus the emphasis with these new methods is to assay for plutonium in-situ – before demolition of any facilities.

The first – and main - method is that of high resolution gamma-ray spectrometry – a technique already described for drum monitoring. In this case the proposed method is to monitor either individual walls or whole rooms using the Am-241 gamma measurement technique. This approach is best compared with – and was initially developed – by consideration of an Am-241 smoke detector in a room. These detectors typically contain 30kBq of Am-241 and can quite easily be located in a few seconds by a high resolution gamma-ray detector a few metres away.

This geometry is similar to the situation where the smoke detector is fixed to the ceiling of the room and the detector is positioned on a table in the middle of the room. A typical sized room has a surface area of ~100m^2 or 10^6cm^2. If the smoke detector source activity was distributed uniformly over the whole of the surface of the room this would yield an average activity of 0.03Bq/cm^2 – and assuming a (typical) Am-241 to plutonium fingerprint of 1:10 this would correspond to a surface plutonium contamination level of 0.3Bq/cm^2. UK legislation on waste is in terms of activity per unit mass or volume – thus an area activity needs some interpretation – but in general this measurement would probably be acceptable as corresponding to 0.3Bq/g. This approach would thus yield the assay of whole rooms as below the Exempt/LLW limit of 0.4Bq/g.

However, there is a further condition that if any contamination is shown to be present – or indeed if there are some areas of local contamination above the 0.4Bq/cm^2 limit – then this needs to be identified. This leads to a further measurement requirement – that of a wall monitoring instrument.

The second technology, the FIDLER probe, can provide more detail of local surface contamination and is effectively a contamination probe that can be used to scan the walls to identify contamination close to the surface. The design of the probe is that of a very thin crystal of sodium iodide. Although it is very thin it is also dense and very efficient at capturing the 60kev gamma-rays from Am-241. However, because it is thin it is not very efficient at detecting the higher energy natural background radiation. Therefore it has a low background signal and a high sensitivity for the detection of Am-241. This type of gamma sensor would also detect Am-241 under a paint layer – or even absorbed into plaster or brickwork – which conventional alpha monitoring techniques would not.

The next two sub-sections describe the technologies in more detail.

5.1 High resolution gamma-ray measurements

All analytical measurement techniques use either a comparison with a known, reference, national laboratory standard, or derive the analytical response from computer modelling. Gamma-ray spectrometry is one of the best examples of this dual analytical measurement arrangement in that the UK National Physical Laboratory produces reference standards for 200l drum intercomparison measurements. These drums are of known density, geometry, radionuclide content etc. However, practical measurement situations will involve different density drummed wastes, other radionuclides etc. To accommodate this variation in the waste materials the major radionuclide manufactures have produced modelling software which enables the effects of such variations in waste contents to be calculated. Nuvia has already used this drum measurement approach for over fifteen years to quantify plutonium wastes in drums and other fixed geometry containers.

The approach taken in this new study uses a specific computer code, MCNP, to calculate the response of a gamma-ray detector to Am-241 – and other background radionuclides – for various activity profiles on the inner surfaces of a room of other enclosed space such as a processing cell or glove box. This approach differs from the normal manufacturer's software, which only gives a

measure of the detection efficiency at a particular energy, in that the actual spectrum is generated for assessment by the analyst. The computer model is built in two sections. Firstly a surface contamination layer can be defined in units of Bq/cm^2 and secondly the general background activity in the brickwork etc. can be defined in units of Bq/cm^3. Three examples are given in Figures 3a, 3b and 3c. Figure 3a shows a spectrum due only to Am-241 for an activity of 0.04Bq/cm^3 (corresponding to a Pu activity of ~0.4Bq/cm^3). The data input conditions are defined in the two tables in the top and middle left of the figure, which allows the activity concentrations on the walls and 'in the walls' of the model to be set. The third table in the bottom left gives the nuclear data used in model. The upper figure show the spectrum over the range 0 to 1.5MeV – and the lower figure shows the expanded low energy region from 0 to 0.2 MeV in the lower figures. This model represents a room with all inner wall, floor and ceiling dimensions of 4m square and for a count time of 100secs. It is evident that this measurement situation would enable Am-241 to be easily detected at the sentencing limit for plutonium of 0.4Bq/g, because the peak can be clearly distinguished and thus an actual numerical assessment of activity can be performed.

Count time	100	seconds

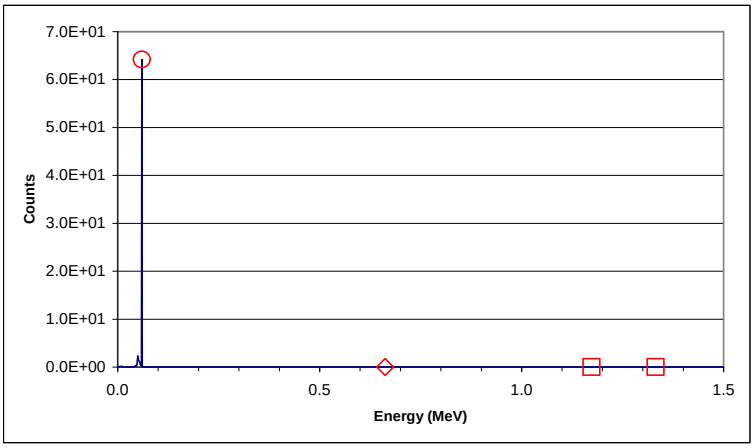
RA material evenly destributed over all surfaces of the box.

Nuclide		Activity		Dose rate (Sv/h)
K-40	0	Bq/cm^2		0.00E+00
Ra-226	0	Bq/cm^2		0.00E+00
Th-232	0	Bq/cm^2		0.00E+00
Cs-137	0	Bq/cm^2		0.00E+00
Co-60	0	Bq/cm^2		0.00E+00
Am-241	0.04	Bq/cm^2		3.11E-11

RA material evenly destributed throughout volume of walls/floor/ceiling.

Nuclide		Activity		Dose rate (Sv/h)
K-40	0	Bq/cm^3		0.00E+00
Ra-226	0	Bq/cm^3		0.00E+00
Th-232	0	Bq/cm^3		0.00E+00
Cs-137	0	Bq/cm^3		0.00E+00
Co-60	0	Bq/cm^3		0.00E+00
Am-241	0	Bq/cm^3		0.00E+00

Nuclide	Energy	Gross counts
Cs-137	661.6	0.00E+00
Co-60	1173	0.00E+00
	1332	0.00E+00
Am-241	59.5	6.42E+01



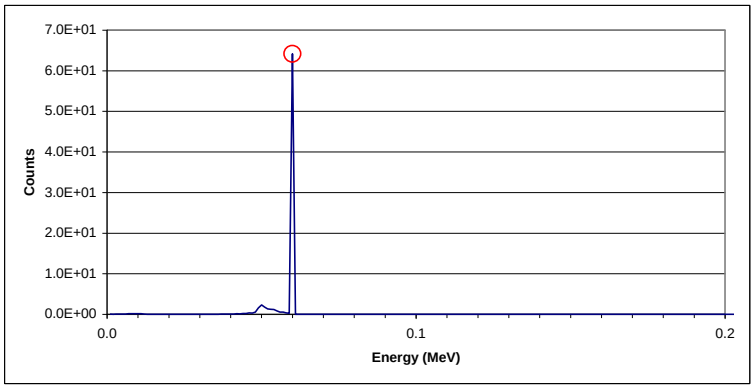


Figure 3a – MCNP, Monte Carlo generated spectrum of Am-241

Figure 2b is more realistic in that it also includes all of the contributions from the naturally occurring background radiation from uranium isotopes, thorium and radiopotassium. These have been added to the model at typical background concentrations. It can be seen that there is now a substantially higher background under the Am-241 peak – note that its height (area) is still the same as in Figure 1 – but the clarity of signal is reduced. Even so it is still apparent that it would be possible to determine the concentration of Am-241 by this method.

Count time	100	seconds

RA material evenly destributed over all surfaces of the box.

Nuclide		Activity		Dose rate (Sv/h)
K-40	0	Bq/cm^2		0.00E+00
Ra-226	0	Bq/cm^2		0.00E+00
Th-232	0	Bq/cm^2		0.00E+00
Cs-137	0	Bq/cm^2		0.00E+00
Co-60	0	Bq/cm^2		0.00E+00
Am-241	0.04	Bq/cm^2		3.11E-11

RA material evenly destributed throughout volume of walls/floor/ceiling.

Nuclide		Activity		Dose rate (Sv/h)
K-40	0.2	Bq/cm^3		4.53E-09
Ra-226	0.04	Bq/cm^3		1.07E-08
Th-232	0.04	Bq/cm^3		8.10E-09
Cs-137	0	Bq/cm^3		0.00E+00
Co-60	0	Bq/cm^3		0.00E+00
Am-241	0	Bq/cm^3		0.00E+00

Nuclide	Energy	Gross counts
Cs-137	661.6	2.08E+00
Co-60	1173	9.75E-01
	1332	1.95E-01
Am-241	59.5	7.78E+01

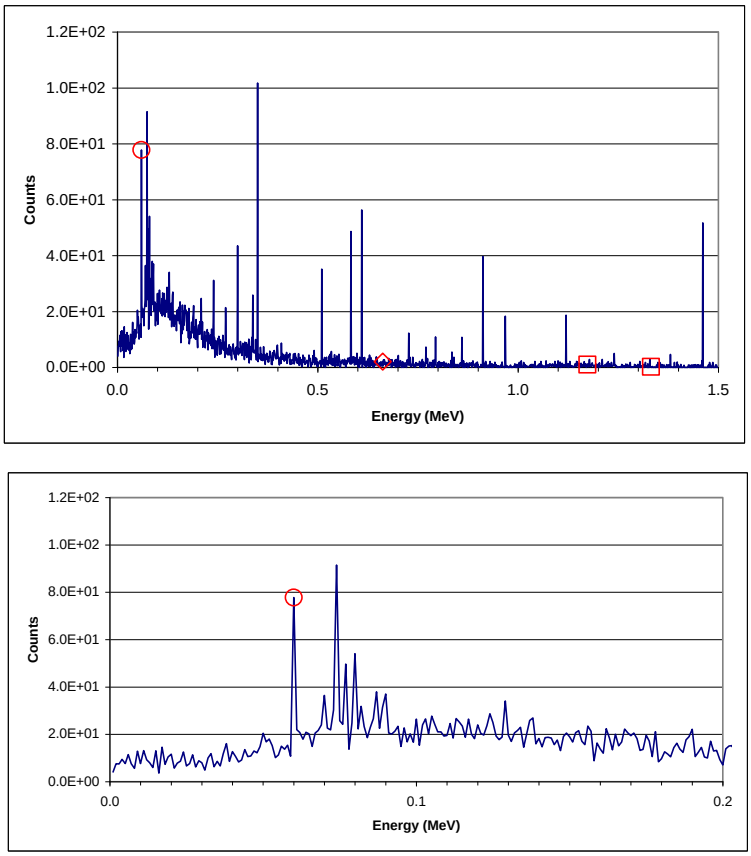


Figure 3b – MCNP, Monte Carlo generated spectrum of Am-241with natural background

A third figure, 3c, is more representative of sites such as Sellafield, where other contaminants such as Co-60 and Cs-137 are also present. This shows the large spectral contributions from Cs-137 (diamond legend) and Co-60 (square legends) to the spectra – with a consequent further reduction in the peak to background ratio from gamma downscatter for the low energy Am-241 line. Even in these circumstances it would still be able to quantify the Am-241 content from this spectral information. It should be noted that this 'room' monitoring technique has already been used by Nuvia in support of delicensing and clearance measurements building contaminated with Co-60, Cs-137 and Ra-226 at various nuclear sites already.

The essential conclusion form these high resolution computer studies are that – in general – contaminated rooms can be monitored to below the UK's Exempt level for plutonium of 0.4Bq/g

(0.4Bq/cm^2). It should also be noted that such measurements would also be a significant support in statistical clearance assessments using techniques such as VSP [2].

Count time	100	seconds

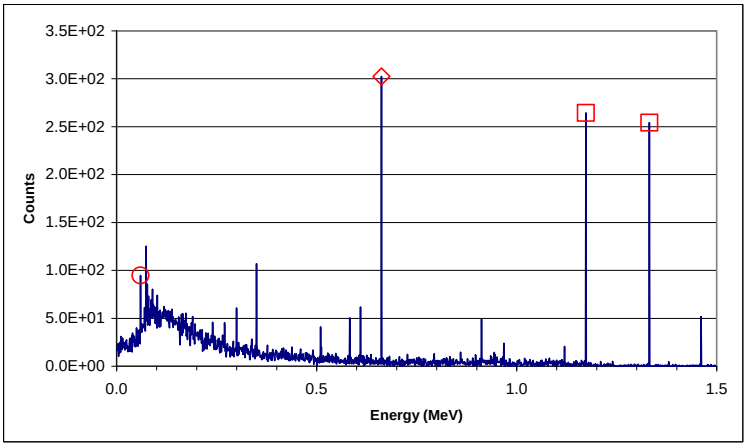
RA material evenly destributed over all surfaces of the box.

Nuclide		Activity		Dose rate (Sv/h)
K-40	0	Bq/cm^2		0.00E+00
Ra-226	0	Bq/cm^2		0.00E+00
Th-232	0	Bq/cm^2		0.00E+00
Cs-137	0	Bq/cm^2		0.00E+00
Co-60	0	Bq/cm^2		0.00E+00
Am-241	0.04	Bq/cm^2		3.11E-11

RA material evenly destributed throughout volume of walls/floor/ceiling.

Nuclide		Activity		Dose rate (Sv/h)
K-40	0.2	Bq/cm^3		4.53E-09
Ra-226	0.04	Bq/cm^3		1.07E-08
Th-232	0.04	Bq/cm^3		8.10E-09
Cs-137	0.1	Bq/cm^3		9.26E-09
Co-60	0.1	Bq/cm^3		3.75E-08
Am-241	0	Bq/cm^3		0.00E+00

Nuclide	Energy	Gross counts
Cs-137	661.6	3.02E+02
Co-60	1173	2.64E+02
	1332	2.54E+02
Am-241	59.5	9.46E+01



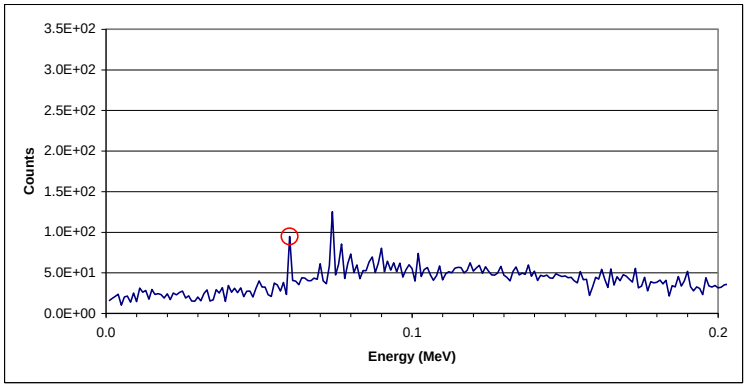


Figure 3c – MCNP, Monte Carlo generated spectrum of Am-241with natural background and contributions from Cs-137 and Co-60

5.2 FIDLER measurements

The FIDLER (**F**ield **I**nstrument for the **D**etection of **L**ow **E**nergy **R**adiation) is not a new technology, but is some 20 years old. Again the importance is in its application to low level clearance-type measurements – and the synergy of the combined high resolution/FIDLER approach.

The FIDLER probe is a 1.6 mm thick x 127mm diameter sodium iodide detector which is optimised for the detection of low energy gamma and X-rays (10-100 keV). This type of detector is ideal for making rapid surveys of relatively large areas to identify 'hot-spots.' The FIDLER has a number of advantages over use of an alpha detector in this application:

- Detection over a wide area;
- Less sensitivity to detector-surface distance;

- Can operate in presence of surface coverings (e.g. paint);
- Will detect material concealed in crevices.

A major investigation has been initiated by Nuvia into the performance of the FIDLER probe for measuring Am-241. This necessitated the manufacture of a specially manufactured test rectangular flat source of known activity. This planar source, together with a point Am-241 source, were used to assess the performance of the FIDLER probe for the detection of Am-241.

A picture of the uncased FIDLER probe positioned on the planar source is shown in Figure 3a, and the full GROUNDHOG Insight packaged system in Figure 3b.

Figure 4a FIDLER probe on Am-241 planar source and 4b cased FIDLER probe (Groundhog Insight detector) on Am-241 planar source.

A comparison of the two Am-241 spectra (intensity as a function of energy keV) for the sensor – with and without the casing are given in Figure 5. It can be seen that there is little attenuation from the carbon fibre case of the Groundhog Insight system. A corresponding background trace in shown in the lower part of the figure.

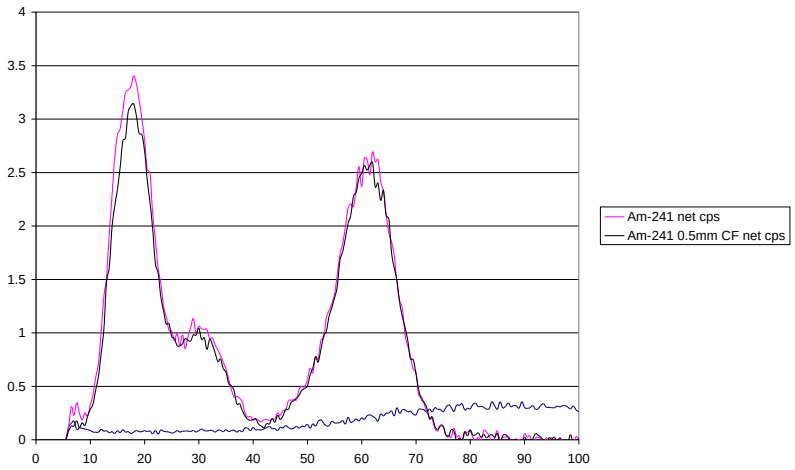


Figure 5 – FIDLER Am-241 probe spectra from the planar source

The spectra obviously have a distinct structure, essentially consisting of two peaks – one X-Ray generated (with a higher energy shoulder on the peak) and one from a gamma decay process. It is possible from a knowledge of the source activity and measured spectra to calculate the detection limit for these spectra, for all the counts below 80keV and for the Am-241 gamma peak between 40keV and 80keV and for all the counts below 40keV. The figures for a 100 second count are given in Table 4.

Table 4 – Limit of detection for the FIDLER Insight system

	Background type	<80keV	>40<80KeV	<40keV
Am-241	Brick wall	0.09	0.17	0.08
Am-241	Concrete floor	0.07	0.14	0.07

In general it would be difficult to achieve the 0.04 Bq/cm^2 limit for Am-241 on the walls – but in practice there are certain conditions which relax this limit. Firstly, as with many assay situations the legislation out performs the actual physical measurement capability. Other techniques are either not viable in-situ – for example alpha measurement of plaster walls – or require a sampling programme which in truth will only represent a very limited investigation at specific sample points. Secondly, even if the detection limit cannot be met – the measurement limit does approach the actual required limit. Thus this approach will identify areas a factor 2 or so above the detection limit. Thirdly, a more subtle interpretation of the data from the survey from a whole wall can be made in so far as if every reading shows activity level below the detection limit, then a statistical argument can be made to show that the mean of all of these readings may be below the detection limit [3].

Further investigation of the method is required as there is a differential attenuation of the two peaks in the FIDLER spectrum – and further insight into the contamination can be gathered from this information. A plot showing the attenuation for PVC and layers of paper is shown in Figure 6.

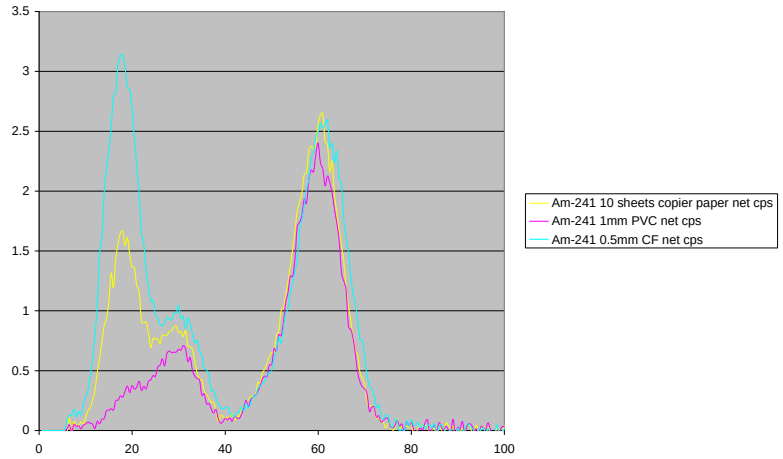


Figure 6 – FIDLER Am-241 probe attenuation with absorbers

A little more experimental and computer modelling work is still required on this subject before a full assessment of the performance of the Groundhog Insight probe can be made, but it is apparent that it is a useful tool in the identification of plutonium contamination. At the moment efforts are also being made to find a suitable contaminated site on which to evaluate these technologies

6. SUMMARY

A description of the current approaches to dealing with plutonium contaminated wastes has been described together with some of the limitations. Two new methodologies – using existing technologies - have been proposed to enable accurate and rapid in-situ activity assessments of PCM wastes to be made.

7. REFERENCES

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