

PLUTONIUM INVENTORY MEASUREMENTS DURING DECOMMISSIONING OPERATIONS

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ABSTRACT

The Sellafield plant of British Nuclear Fuels plc has handled radioactive materials for over 40 years and plutonium since the early 1950s. As part of the overall decommissioning program for the site BNFL has recently undertaken the dismantling of a mixed oxide fuel manufacturing plant which has yielded valuable information in terms of project management, manpower requirements and radiation uptake.

The project was deliberately chosen as a test bed for the development of decommissioning equipment and particularly methods of locating and measuring plutonium residues before and during dismantling using in situ assay techniques.

Prototype equipment has now been used over a two year period, for items ranging from glove boxes of 10M³ volume down to geometrically safe storage tanks made from 100mm pipe, and has proven invaluable. This review of experience highlights scope for improvements in the equipment and additionally considers means of applying in situ Plutonium assay in locations having access constraints or more complex chemical or plutonium isotopic compositions than was the case in this initial application.

INTRODUCTION

The Sellafield plant of British Nuclear Fuels plc has handled radioactive materials for over 40 years. Many plants dating from early years are now redundant and a previous paper to this conference has reviewed the overall decommissioning program for the site with reference to waste management and environmental protection issues (1).

Plutonium was first handled on the site in the early 1950s. The very earliest facilities have long been dismantled (2) but several plants which handled the element and its compounds remain in a shut down condition and this number will increase in future years.

Consequently to gain specific decommissioning experience of plutonium plant BNFL has recently undertaken the dismantling of a mixed oxide fuel manufacturing plant known as the "Co-precipitation Plant" (3, 4). The project was chosen as a test bed for equipment development and included in the Commission of the European Communities 1984-1988 Research Program on the Decommissioning of Nuclear Installations in this context.

A particular concern in the dismantling of plants which have handled large quantities of plutonium is the need to ensure nuclear (ie criticality) safety during the decommissioning. Consequently equipment was required for the location and measurement of plutonium residues in plant items before and during dismantling using in situ non destructive assay techniques.

SPECIFICATION AND PURPOSE

The outline requirement for this part of the project was therefore for equipment with a capability to:

- a) establish initial plutonium profile and inventory.
- b) monitor changes as clean out proceeded.
- c) assay plutonium content before packaging for storage of wastes containing significant residues.
- d) measure residual contamination levels to meet low level waste disposal criteria where appropriate.

As project planning developed and hands on dismantling commenced it became apparent that existing equipment largely satisfied the last of these and that the major new requirements were for:

- a) the location and measurement of significant fissile residues in plants which have handled very large quantities of plutonium.
- b) assuring that nuclear safety controls are met where clean out processes require any change in plant operating procedures or before there is any change in configuration of plant or equipment due to dismantling.

The Radiometric Physics Group of BNFL's Research and Development Department responded to this requirement by development of the prototype Decommissioning In Situ Plutonium Inventory Monitor (DISPIM) system. This currently consists of three items:

- a) Roving Gamma Counter,
- b) Roving Neutron Counter,
- c) Modular neutron detectors for passive coincident neutron counting.

The last of these is the principal subject of this paper.

MEASUREMENT TECHNIQUE

Of all the radiations from Plutonium, only neutrons are sufficiently penetrating to escape from process cabinets

without any significant signal losses due to attenuation within the Plutonium itself or the construction materials of the cabinet (glove box). This passive neutron emission originates from the spontaneous fission of the even mass isotopes (in which an average of 2.1 coincident neutrons per fission event are emitted) and from (alpha, n) reactions on light elements in chemical or physical contact with the plutonium. These depend respectively on isotopic, and on isotopic chemical and physical composition.

Two passive neutron counting methods are commonly used for Plutonium measurement: total neutron counting; and neutron coincidence counting. In total neutron counting all detected neutrons (from spontaneous and alpha, n events) are counted, whilst in neutron coincidence counting, time correlated neutrons from spontaneous fission events are counted separately.

In this context neutron coincidence counting is preferred because the total neutron counting signal is likely to contain significant background contributions from other cabinets in immediately adjacent areas which cannot be easily measured or estimated. In addition, the total neutron emission per unit mass of plutonium varies significantly with chemical composition due to the widely varying (alpha, n) emission rates. However, the "real" coincidence count rate from a cabinet is independent of this varying (alpha, n) emission, as only neutron pairs resulting from spontaneous fission events are recorded. The only significant effect of the uncorrelated neutron background radiation is to worsen the counting precision by adding to the accidental coincidence signal and not to the "real" coincidence signal.

The real coincidence counting rate (R) is given by:

$$R = \exp(-P/\tau) \cdot (1 - \exp(-G/\tau)) \cdot k \cdot Sf \cdot m \cdot \epsilon^2$$

Where

R = dead-time and bias corrected real coincidence count rate (s^{-1})

P = pre-delay time ($= 8\mu s$)

G = gate length ($= 80\mu s$)

τ = die away time ($= 64\mu s$)

k = a constant related to the multiplicity of Pu (dimensionless)

Sf = specific spontaneous fission rate of Pu ($s^{-1} \cdot kg^{-1}$)

ϵ = the total neutron counting efficiency (dimensionless)

m = the mass of Pu (kg)

The system die away time, τ (related to the average lifetime of a neutron in the counting system), is an important factor in Neutron Coincidence Counting (NCC). However

since process cabinets are largely non-moderating, the physical construction of the DISPIM modules becomes the dominant factor determining the die away time of the system. Varying the configuration of a number of DISPIM modules to measure a variety of cabinet sizes does not change this factor significantly, as each module is a discrete counting system isolated from the others by a cadmium layer.

Measurements of the die away time when assaying two process cabinets produced results of 72 and 67 μs and confirm the assumption that the system die away time is largely independent of the geometry of the cabinet being measured. As a result the die away time was not routinely measured in order to minimize assay times.

The values of k and Sf are a function of the Plutonium isotopic composition, however since the isotopic composition of Plutonium processed through the Co-precipitation Plant was well characterized and consistent they have been calculated as constants at 2.1 and 10550 $s^{-1} \cdot kgPu^{-1}$ respectively. (Other plants to be decommissioned may not have such a well characterized residual Pu content, in such instances the Plutonium isotopic composition may be determined by destructive analysis or by high resolution gamma spectrometry, see later).

The mean counting efficiency, ϵ , changes from measurement to measurement since the counting geometry is different each time the DISPIM system is set up around a process cabinet. It is therefore necessary to determine this parameter at every measurement. This is achieved using the "add-a-source" calibration method whereby the increase in total neutron count rate, induced by the addition of a source of known neutron emission rate at defined locations, is used to calculate detection efficiency values for Plutonium at those locations. Efficiency, ϵ , is calculated as the arithmetic mean. The range in efficiency values gives the potential geometric error (ie that error associated with the uncertainty in plutonium distribution):

The real coincidence rate, R, is measured by a series of long counts (usually about 12 hours) and corrected for dead-time effects as follows:

$$R_1 = R_0 \cdot \exp(4 \cdot \delta t \cdot T)$$

$$T = T_0 / (1 - T_0 \cdot \delta t)$$

where

- δt = total neutron counting dead-time (determined at commissioning)
- T_0 = observed total neutron count rate
- T = dead-time corrected total neutron count rate
- R_0 = observed real coincidence count rate
- R_1 = dead-time corrected real coincidence count rate
- R = dead-time and bias corrected real coincidence count rate

Biases in the coincidence counting electronics are then corrected for using the equation:

$$R = R_1 - B.A$$

where A = accidental coincidence count rate

B = real/accidental coincidence bias (determined at commissioning)

Inserting the known factors, the mass of plutonium, m , can be calculated:

$$m = R / [\exp(-P/\tau) \cdot (1 - \exp(-G/\tau)) \cdot k \cdot Sf \cdot \epsilon^2]$$

EQUIPMENT

The DISPIM system comprises:

Up to 6 counting modules, each initially containing two 50mm diameter, 5 bar pressure, 1000mm active length ^3He

neutron detectors (subsequently replaced by BF_3 detectors of similar dimensions);

- an EHT junction box to link all the detector cables into one amplifier/EHT supply;
- a single WE000C neutron detector pulse amplifier (developed at Sellafield);
- a microprocessor controlled neutron coincidence counting electronics rack (containing a Sellafield developed neutron coincidence board utilizing fast computer memory);
- Operator's terminal and printer; Interconnecting cables.

Also available for standardization and calibration measurements is a ^{252}Cf neutron source (chosen because it is non-fissile and has a neutron energy spectrum similar to Plutonium). The system is shown schematically in Fig. 1.

Two additional systems are also available:

- a) The Roving Neutron System (RNS) is a small and portable, total neutron counting system using a single 12cm active length ^3He detector surrounded by a layer of polythene clad with 0.5mm of Cadmium (polythene is a neutron moderator, and Cd is a thermal neutron

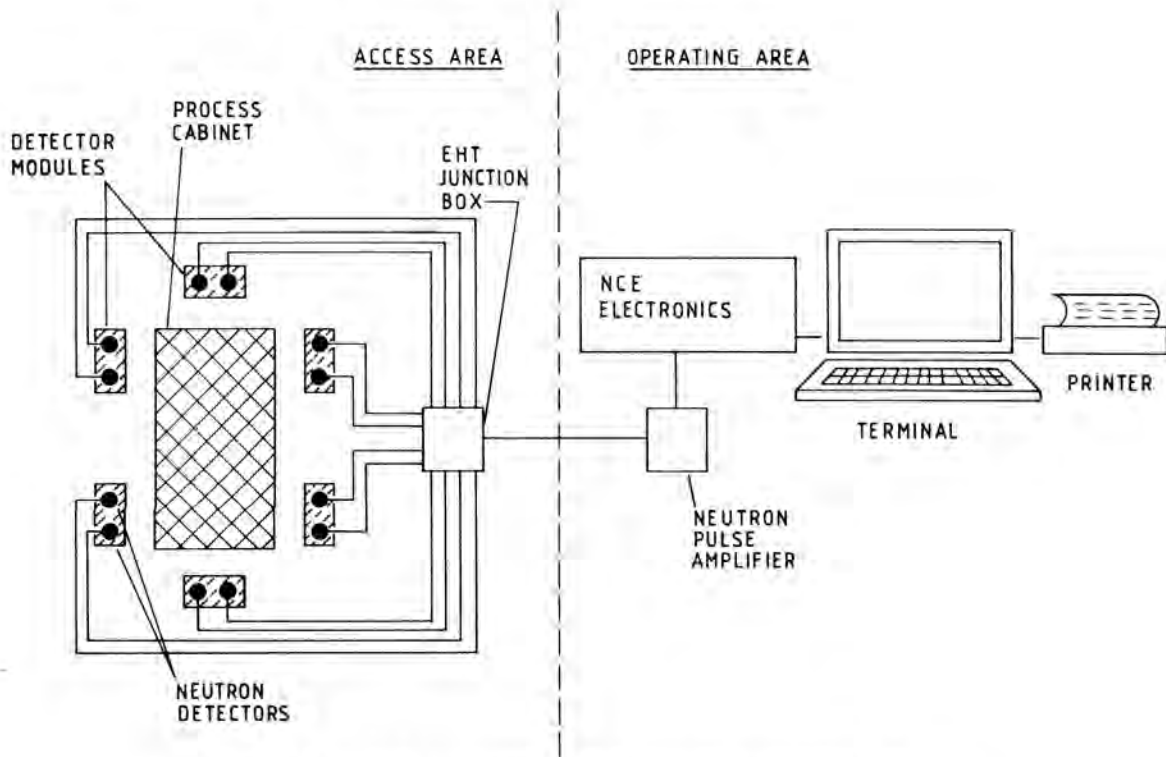


Fig. 1. Schematic Layout of DISPIM System Operating Procedures.

absorber). This system can be used to determine the relative abundance of Plutonium in various locations, either within a process cabinet or throughout the plant.

- b) The Roving Gamma System (RGS) is a low resolution gamma spectrometry system using a 1" x 2" NaI(Tl) detector. This system can be used to determine the relative (or under some circumstances the absolute) abundance of Plutonium in various locations within a process cabinet.

General Approach

To a degree these have developed as measurement experience has been gained. They are now sufficiently well established that most cabinet measurements are routine with only occasional departures from the procedure described below.

The operator's knowledge of the original cabinet functions plus mechanical drawings, and HP&S radiation surveys are used to estimate the distribution of residual Plutonium in the cabinet. In addition, either the RNS or RGS may be used to confirm the distribution. Once determined, the positions shown to contain significant quantities of Plutonium are used to calibrate the DISPIM System. It is important that this stage is considered carefully before proceeding; too many non-essential calibration positions increase the geometric error estimate unnecessarily,

whereas too few positions will lead to an unrealistically low estimate of geometric error.

Up to 6 DISPIM modules are positioned around the cabinet in a suitable counting geometry. The counting geometry chosen must attempt to minimize variations in counting efficiency with plutonium location, whilst at the same time ensuring that sufficient access to the inside of the cabinet (usually via glove ports) is maintained for the "add-a-source" calibration. This often requires that platforms are constructed from scaffolding to support the modules in the preferred positions. Typical locations used during measurements of cabinets within the Co-precipitation Plant are shown in Fig. 2.

The system is calibrated each time it is set up around a process cabinet. The neutron source (²⁵²Cf) is introduced to the defined calibration positions inside the cabinet through a bagging port or glove port. The source is placed inside the glove or bag and manoeuvred into position for the count still inside the glove or bag i.e. outside cabinet containment. The increased count rates are recorded for all calibration positions. After the calibration the ²⁵²Cf source is returned to a storage location well away from the cabinet being measured.

The neutron coincidence count rate from the cabinet is measured by taking, typically, three x4 hour counts. This count time is long enough for a precise measurement of the

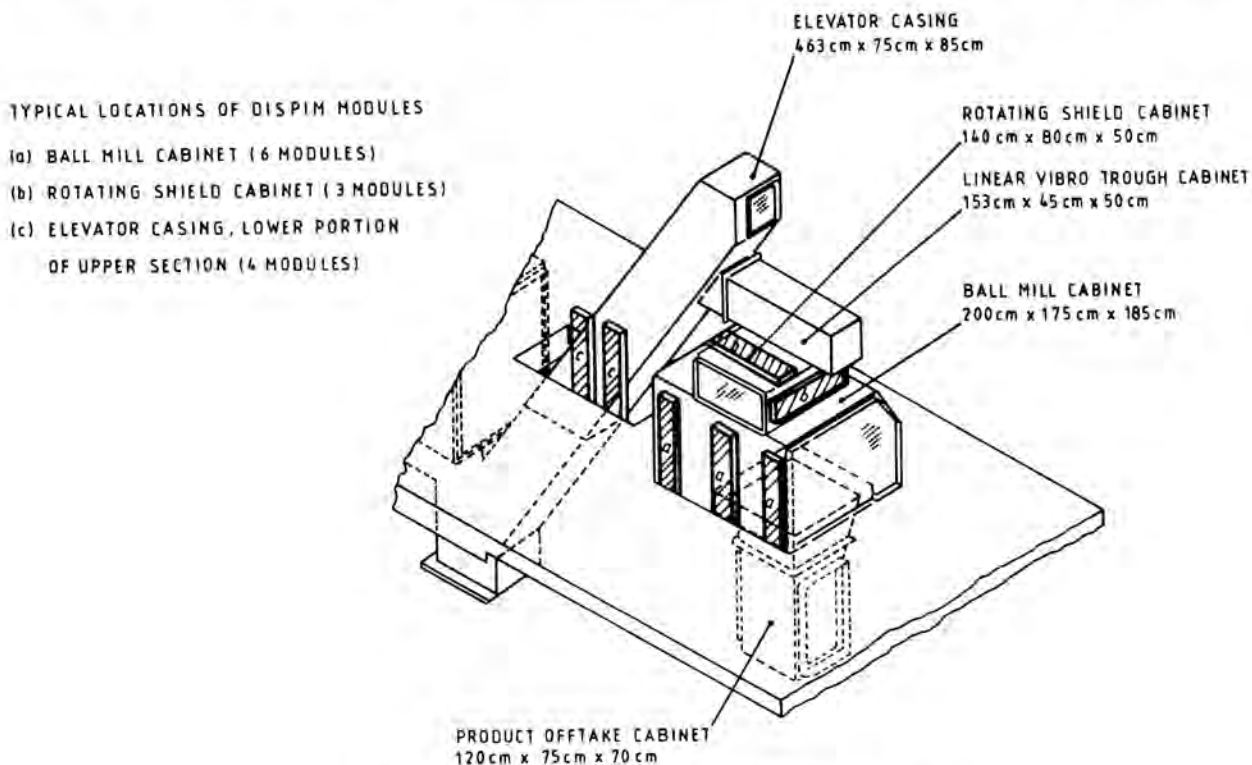


Fig. 2. Typical Locations Of DISPIM Modules.

real coincidence rate from most process cabinets (ie three standard deviations < 10%).

The calibration and measurement data are analyzed off-line to determine the estimated $^{240}\text{Pu}_{\text{eq}}$ mass content (and hence total Plutonium content from knowledge of isotopic composition) of the cabinet together with geometric and statistical errors.

For nuclear materials accountancy and for the information of operators continuing to clean out the plant results are reported as a "Best Estimate" figure. For nuclear safety (ie criticality) assessment however the maximum statistical and geometric errors are added to this figure.

Measurement Control

For any coincidence count, under control of the micro-processor, the overall count time is subdivided into a number of smaller segments and the real coincidence count rate for each recorded as a separate element. Upon completion of the total count, the full set of count rates are analyzed to determine if all elements fall within the distribution expected from counting statistics. If any elements lie outside this distribution they are removed from the data set (these are usually the result of external interference, eg RF welding, or EHT tracking within the system). In this case the mean count rate is recalculated from the remaining elements. However, if a large number (> 10%) of elements are rejected, then the whole count is discarded and repeated.

In addition to the statistical measurement control described above, standardization measurements verify that the system operates reliably throughout the cabinet measurements. The ^{252}Cf source is placed against the rear of each module in turn (at a marked location) and the total neutron count rate from the system is recorded. Each count rate is compared against the others and any significant difference noted as a possible indication of a module malfunction. These standardization measurements are performed routinely after set up, and again after all measurements are complete.

OPERATING EXPERIENCE

General Comments

The DISPIM system has now been used for some 40 measurements, providing necessary plutonium data for criticality safety requirements and for the cabinets to be dismantled and size reduced.

There are currently 6 DISPIM modules available for use when measuring any cabinet. This is adequate for small to medium sized cabinets (ie < 2m x 2m x 2m), and good counting precision (< 10%, 3σ) and reasonable geometric accuracy are achievable ($\pm 50\%$). However, attempts to measure larger cabinets have resulted in poor counting efficiencies and larger geometric errors. Therefore, these

items have had to be measured in two halves, and the results summed. It is intended to provide any future DISPIM systems with a greater number of counting modules to improve this situation.

The standard procedure has not been suitable for monitoring certain cabinets or equipment, in these cases the measurement procedure being tailored for the particular purpose. For example, the measurement of the residual Plutonium content of 4 geometrically safe (harp) tanks required a special procedure. Each tank to be measured consisted of a series of long lengths of steel pipe connected together and occupied a large area. Some locations were difficult to reach with parts of the tanks at heights over 3 metres from floor level. The DISPIM NCC modules could not be arranged to monitor an entire tank at once, and monitoring the tank portion by portion (the approach taken when measuring other large items) was not practical due to the problems of positioning 35kg counting modules at heights in excess of 3M. The procedure designed for this situation was to use the RGS to determine the relative distribution of Plutonium along the entire length of the tank, and then the DISPIM NCC system to determine the absolute Plutonium content of one section of the tank. The measured Plutonium mass for the one section was then scaled up, using the RGS based relative distribution data, to provide an estimate of the total Plutonium content of the tank.

Electronic Equipment

The neutron amplifiers currently used on both the Roving Neutron and DISPIM systems are of the WE000 C type (Sellafield developed). This amplifier can be susceptible to interference from sources of electrical noise - such sources may include components of the monitoring systems that contain electronic oscillators such as printers, terminals etc. This type of noise may significantly increase the total count rate, but more importantly, due to its time correlated nature, it can also increase the real coincidence rate by several orders of magnitude. This is potentially a serious inaccuracy in DISPIM operations. It is obviously important to take particular care when setting up the DISPIM system to avoid such sources of noise (careful positioning of system cables and components can completely avoid the effects of interference). A more satisfactory solution for the future will be to use better screened components, especially detector cables and amplifiers. A new version of the WE000C amplifier has recently entered production, and has shown much better noise immunity properties when tested together with super-screened detector cabling. It is planned to replace the existing amplifiers and detector cables, and make further system improvements, in the near future.

Each neutron detector is connected to the single junction box via a SHV-SHV cable and that junction box is in

turn connected to a WE000 C Amplifier via one SHV-SHV cable. This means that for a 6 module system, with 12 detectors, 13 cables (each in excess of 1m) are connected to one amplifier. This amounts to a considerable length of cable and a large number of SHV connectors (26), each a potential problem via EHT tracking or breakdown. The fitting quality of some connectors has also proved inadequate to withstand constant connecting and disconnecting. In addition, the long total length of cable can make the system prone to RF interference detection. Careful positioning and handling of cables, and control of environmental equipment (eg RF welders), is therefore essential for successful monitoring.

Detector Equipment

During the latter part of 1988 and the beginning of 1989, the DISPIM system suffered from persistent amplifier failures (typical of EHT breakdown) and consequently, a high system down time. Much effort was put into locating the source of these problems resulting with the high efficiency ^3He detectors being replaced with BF_3 detectors. This has reduced the counting sensitivity from, typically, 3%-4% to 1%-2%. The reliability of the ^3He detectors is now being investigated but it is not yet possible to say whether mechanical shock, a poor operating environment, or faulty manufacture was the cause of the failures.

Module Portability

Handling the DISPIM NCC modules (each weighing about 35kg) can sometimes be difficult, especially in situations where space is limited, or access restricted. To date this has not given rise to any insurmountable problems during measurements on the Co-precipitation Plant. However this may not be the case in future when monitoring locations may be extremely restricted or difficult to reach.

There are currently no facilities incorporated for the modules to be rigidly secured in position. Quantities of adhesive tape or cord are used to fasten the modules to the supporting framework of the cabinet being measured. This method, although effective, will be replaced by a better system.

Equipment Contamination

The DISPIM modules are routinely covered in protective heavy duty paper and the EHT detector cables are all placed inside plastic sleeving. Occasionally, the paper covering on the NCC modules, or the sleeving on the interconnecting cables, has been contaminated whilst in the access area, this has not caused any difficulties as the coverings are designed to be stripped off, leaving the equipment uncontaminated.

The 6000 series DISPIM NCC electronics rack is normally sited outside the access area, connected to the modules via long cables, and therefore does not risk contamination. However, the RGS and RNS electronic racks are operated inside the area close to the detectors, and can not be covered by plastic sheet since it would be difficult to operate the front panel controls. This makes these electronics racks vulnerable to contamination, and the variety of front panel controls make them difficult to decontaminate successfully. The RNS counting rack has been contaminated in this way and is now confined to use in the Co-precipitation Plant access area where respiratory protection is routinely worn.

Roving Neutron and Roving Gamma Systems

Operational pressure has limited the use of the roving systems to determine the Plutonium distribution within a cabinet - using these systems adds to the monitoring and data analysis times. Instead, the Pu distribution has been routinely estimated using mechanical drawings, operator knowledge, and HP&S radiation surveys wherever this could be regarded as adequate.

RESULTS

Some 40 measurements of the plutonium content of process cabinets and equipment have been made at various stages during the clean out and dismantling of the Co-precipitation Plant decommissioning project. The equipment has also been used to assess the plutonium content of several cabinets (ie glove boxes) in other facilities, although with due regard for differences in chemical and isotopic composition in this case.

Table I contains results from the Co-precipitation Plant work, selected both to illustrate specific points and to show the range of equipment measured and results typically obtained.

The table shows several items of interest:

- a) DISPIM NCC measurement of the Ball Mill and Casing (Pu mass = 680g) and another measurement of the empty Casing (Pu mass = 148g) made after the Ball Mill was removed, provided an estimate of the Pu content of the Ball Mill (680 - 148 = 532g Pu) which was in good agreement with the separate measurement by total neutron counting of 486 Pu.
- b) The die away time measurements on two cabinets are in adequate agreement with expectations (64 μs).
- c) Because of its shape and size the Drier Sample Cabinet was measured in two parts. The results show the progress of cleaning, reducing levels as this pro-

TABLE I
 Typical DISPIM Results from Co-Precipitation Plant Decommissioning

Item	Best Estimate of Plutonium	Including Statistical and Geometric Errors Rounded Up To Nearest 50g	Comments
Ball Mill Cabinet containing Mill	680	-	1.1x1.3x1.6m approx
Empty Ball Mill Cabinet	148	-	
(Ball Mill by difference)	532	-	0.7x0.6x1.0m approx
Ball Mill	486	-	By total neutron counting
Filter Segment Treatment Cabinet	14	< 50	Die away time 72 μ S
Linear Vibro trough Cabinet	4	< 50	Die away time 67 μ S
Drier Sample Cabinet			Measured in two halves because of shape & size
Upper half 5.10.88	254	< 550	
Lower half 10.10.88	281	< 750	
Upper half 30.1.89	176	< 350	After cleaning & removal of drier rotor
Lower half 31.1.89	237	< 400	
Upper half 24.4.89	69	< 250	After more cleaning & moving away from drier
Lower half 3.5.89	50	< 200	
Geometrical safe storage (HARP) Tanks (4)	5 to 16	all < 50	Procedure described in text
Rotating Shield Cabinet	87	< 300	0.5x1.0x1.0m approx
Drier rotor	112	< 350	About 2m long
Scrubber column	9	< 50	More than 3m long

gressed, as the rotor was removed from the Drier itself (which protruded into the sample cabinet), and finally as the cabinet was moved to a lower background area.

- d) The geometrically safe tanks, constructed of nominal 4" stainless steel pipe to approximately 150 litre capacity each, were measured by roving gamma counter and only the highest area of each, estimated at over 90% of the Pu content, was measured by DISPIM.
- e) The remaining three items are typical examples, the rotating shield cabinet being measured in situ and the others after lowering to a horizontal position.

FUTURE DEVELOPMENTS

Operating Requirements

The Co-precipitation Plant, for which the DISPIM equipment was initially developed, handled only mixed plutonium and uranium material, from nitrate through to oxide. It operated only from 1969 to 1976, with well documented operational history, and handled only well characterized plutonium of essentially the same isotopic composition throughout (typically 18% ^{240}Pu). This was confirmed by NDA and laboratory analysis of residual material. Gamma and neutron emission from residues can therefore be related to plutonium hold-up in a relatively straight forward manner as described above.

This will not be the case in future Sellafield decommissioning work, because BNFL is currently planning for the dismantling of plutonium plants which have operated for up to 30 years. These have processed plutonium from 5% ^{240}Pu to 25% ^{240}Pu content and handled plutonium as oxide, nitrate, metal, fluoride and in both aqueous and non-aqueous solutions. Additionally some facilities have access constraints due to physical restrictions, high contamination levels and/or high gamma dose rates.

Consequently further development of in situ plutonium measurement techniques is required to cope with more extreme physical conditions and more complex radiometric situations. Work is in hand in these areas.

DISPIM Design Improvements

Through experience gained with measurements on the Co-precipitation Plant the weaker elements of DISPIM design have been identified. These have been considered and new designs are being produced with improvements in terms of mechanical design, electronic design and reliability. The requirements of future decommissioning operations have also been considered and the second generation of DISPIM equipment will have: improved radiofrequency screening on the detector cabling and amplifiers; a lower module weight and reduced module size to increase portability; an improved mechanism for fastening modules in their measurement locations; and lower operating voltage.

Components for production of the first such modules are already on order.

Improved Techniques

Further work is ongoing, to develop measurement systems to assay plutonium of unknown isotopic composition and high (alpha, n) neutron emissions by high resolution gamma spectrometry and fast neutron coincidence counting.

To relate the $^{240}\text{Pu}_{\text{eq}}$ measurement supplied by the DISPIM NCC system to total plutonium mass requires a knowledge of the isotopic composition. This information is available for the Co-precipitation Plant but may not be available for future measurements, since the residual plutonium may not be entirely from the last campaign but will probably be contaminated by material of different isotopic composition from earlier campaigns. However, it is possible to determine the plutonium isotopic composition of residual plutonium via High Resolution Gamma Spectrometry (HRGS). Consequently, suitable HRGS equipment has been bought and tested and a development program defined. The development program will investigate: plutonium isotopic composition measurements; measurement of the PuF_4/Pu ratio; matrix attenuation correction methods, and will eventually lead to a HRGS system to operate, on plant, in parallel with a neutron coincidence counting system.

Samples with very high (alpha, n)/spontaneous fission ratios (eg PuF_4) when measured with the long coincidence gates required for thermal neutron coincidence counting, produce a high accidental coincidence rate which can result in a poor measurement precision. Therefore, development equipment for a liquid scintillator based fast neutron coincidence counting system has been purchased, assembled and tested. A development program has been defined, and is now underway. Initially, it is envisaged that the system will be capable of determining the plutonium content of packaged plutonium contaminated materials (PCM) rather than items of cabinet size.

CONCLUSIONS

The objective of the Co-precipitation Plant decommissioning project was "to pilot the development of technology for the decommissioning of facilities used in the fabrication of mixed oxide fuels". Clearly the work has gone some way towards this particularly in the area of in situ plutonium assay.

The prototype equipment has now been used on a wide range of items, from 10M^3 glove boxes down to geometrically safe storage tanks made from 100mm stainless steel pipework, over a two year period. It has proven invaluable in dismantling operations both on the Co-precipitation Plant and elsewhere for indicating the progress of clean out

operations and for assuring nuclear safety (ie criticality) requirements.

Finally the work has highlighted inadequacies, both in equipment design and in the technology, as far as future decommissioning is concerned, and has shown the direction for appropriate development. This will use our previous experience to develop current equipment, and will also utilize alternative counting techniques in the production of new monitoring systems. The result of these developments will be a monitoring capability suitable for performing in situ Pu assay measurements in a wide variety of situations.

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