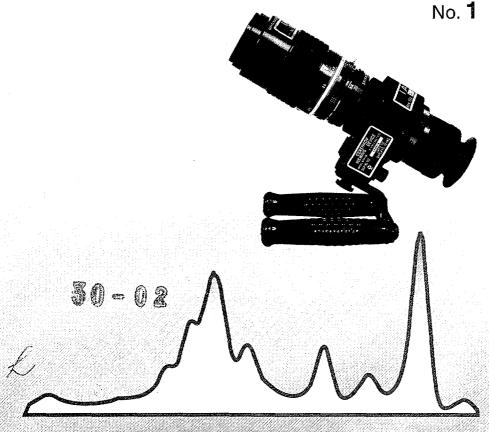




# SAFEGUARDS TECHNIQUESAND EQUIPMENT

INTERNATIONAL **NUCLEAR VERIFICATION SERIES** 



### SAFEGUARDS TECHNIQUES AND EQUIPMENT



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ZIMBABWE

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#### Foreword

In 1980 the IAEA Safeguards Information Series was launched, providing information on IAEA safeguards and related subjects. Between 1980 and 1985 six booklets were issued in the series and were widely used in Member States and within the IAEA itself. Those booklets described the safeguards system that had evolved since the 1960s.

The 1990s have seen significant non-proliferation related developments in the world, resulting in a new period of safeguards development. Over several years an assessment was made of how to strengthen the effectiveness and improve the efficiency of IAEA safeguards. In May 1997 this culminated in the adoption by the IAEA Board of Governors of a Protocol Additional to Safeguards Agreements which significantly broadens the role of IAEA safeguards. As a consequence, the IAEA safeguards system is entering a new era.

Together with the introduction of the strengthened safeguards system, the IAEA is beginning in 1997 a new series of booklets on safeguards, called the International Nuclear Verification Series. It is hoped that the new booklets will be of help in explaining IAEA safeguards, and especially the new developments in safeguards, particularly for facility operators and government officers involved with these topics.

The current booklet is intended to give a full and balanced description of the techniques and equipment used for both nuclear material accountancy and containment and surveillance measures, and for the new safeguards measure of environmental sampling. As new verification measures continue to be developed, the material in this booklet will be periodically reviewed and updated versions issued.

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#### INTRODUCTION

The IAEA has the task of providing continuing assurance to the international community that States that have entered into safeguards agreements with the IAEA are meeting their obligations. This requires that assurance can be given that any diversion of safeguarded nuclear material from civil use to a proscribed military purpose would be detected and that all nuclear material in the State has been declared. To this end, the IAEA must be able to verify the correctness and completeness of the statements that it receives from States concerning the nuclear materials included in safeguards agreements.

The basic verification measure used by the IAEA is nuclear material accountancy. In applying nuclear material accountancy, IAEA safeguards inspectors make independent measurements to verify quantitatively the amounts of nuclear material presented in the State's accounts. For this purpose, inspectors count items (e.g. fuel assemblies, bundles or rods, or containers of powdered compounds of uranium or plutonium) and measure attributes of these items during their inspections using non-destructive analysis (NDA) techniques, and compare their findings with the declared figures and the operator's records. The purpose of this activity is to detect missing items (gross defects). The next level of verification has the aim of detecting whether a fraction of a declared amount is missing (partial defect) and may involve the weighing of items and measurements with NDA techniques such as neutron counting or y ray spectrometry. These techniques are capable of measuring the amount of nuclear material with an accuracy of 1-10%. For detecting bias defects, which would arise if small amounts of material were diverted over a protracted length of time, it is necessary to sample some of the items and to apply physical and chemical analysis techniques having the highest possible accuracy, typically 0.1–1%. In order to apply these destructive analysis (DA) techniques, the IAEA requires access to laboratories which use such accurate techniques on a routine basis.

Containment and surveillance (C/S) techniques, which are complementary to nuclear material accountancy techniques, are applied in order to maintain continuity of the knowledge gained through IAEA verification, by giving assurance that nuclear material follows predetermined routes, that the integrity of its containment remains unimpaired and that the material is accounted for at the correct measurement points. They also lead to savings in the safeguards inspection effort, e.g. by reducing the frequency of accountancy verification. A variety of C/S techniques are used, primarily optical surveillance and sealing. These measures serve to back up nuclear

**TABLE I** MAIN TYPES OF FACILITIES SAFEGUARDED BY THE IAEA (data based on 1997 Safeguards Implementation Report)

Enrichment Plants	Fuel Fabrication Plants	Power Reactors and Separate Storage Facilities	Spent Fuel Reprocessing Plants
	Number of Facilities Sa	afeguarded in 1996	
11	40	229 reactors 51 separate storage facilities	6.
	Main Technique	es Deployed	
Materials: UF <sub>6</sub> Gamma ray spectrometry Weighing	Materials: U and Pu oxides, MOX Gamma ray spectrometry Neutron counting Destructive analysis Elemental Isotopic	Materials: Spent fuel Optical surveillance Fuel flow monitoring Cerenkov glow detection Gross γ ray and neutron detection	Materials: U and Pu nitrates Destructive analysis Elemental Isotopic
	IAEA Summary Statistics for 1	996 (approximate numbers)	
	3 000 inspection NDA measureme: 1 000 DA samples taken (2200 and 450 video cameras deployed for 20 000 seals detached and verified	lytical results)	facilities

material accountancy by providing means by which access to nuclear material can be monitored and any undeclared movement of material detected.

Unattended and remote monitoring is a special mode of application of NDA or C/S techniques, or a combination of these, that operates for extended periods of time without inspector access. In remote monitoring, the unattended equipment transmits the data off-site. For unattended and remote monitoring, additional criteria must be met, including high reliability and authentication of the data source. Data communication costs have dropped dramatically in recent years. Consequently, expanded deployment of unattended and remote monitoring systems has become an increasingly important element of IAEA safeguards in efforts to maintain or increase effectiveness without increasing inspector resources or overall costs.

Environmental sampling, which allows detection of minute traces of nuclear material, was added to the IAEA's verification measures in 1996. Environmental sampling and the subsequent highly sensitive laboratory analysis of the samples provide a very powerful yet unobtrusive means of contributing to the assurance that all nuclear material within a State has been declared. The non-detection of minute traces of a specific nuclear material indicates that no safeguards significant activities utilizing the material were located in the area where the environmental samples were taken.

The complexity and diversity of facilities containing safeguarded nuclear material require a correspondingly diverse set of verification techniques and equipment. Table I lists the main types of facilities where inspections are performed and the primary verification techniques that are implemented at these facilities.

Development of equipment and techniques for safeguards is continuing with the help of national support programmes which assist the IAEA in keeping pace with the evolution of new technology. The IAEA defines the safeguards needs, co-ordinates the support programmes and tests and evaluates the techniques developed and the equipment resulting from them. All aspects of equipment performance are evaluated, including compliance with specifications, reliability and suitability for transport and, most importantly, suitability for use by IAEA inspectors in nuclear facilities. As a result of these assessments, new equipment passes through different developmental categories until it reaches the status of a fully approved device for routine use in the field.

#### Safeguards Techniques and Equipment

The equipment and techniques highlighted in this booklet are those in frequent use for inspection purposes or in the late stages of development. The overall objective of this publication is to provide a comprehensive overview of the techniques and equipment underlying the implementation of IAEA safeguards.

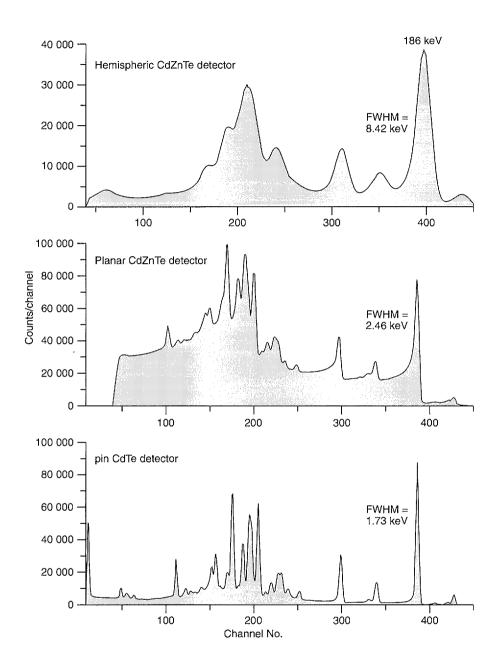
#### NON-DESTRUCTIVE ANALYSIS

#### Gamma Ray Spectrometry

Most nuclear materials of concern in IAEA safeguards emit  $\gamma$  rays and these can be used for NDA of the materials. Gamma rays have well defined energies which are characteristic of the isotopes emitting them. Determination of the  $\gamma$  ray energies serves to identify the isotopic composition of the materials. When combined with a measurement of the intensities they can provide quantitative information on the amount of material that is present. Enriched uranium fuel, for example, has a strong 186 keV  $\gamma$  ray associated with the  $\alpha$  decay of <sup>235</sup>U and the <sup>235</sup>U enrichment can be verified by measuring the intensity of this  $\gamma$  ray. Plutonium samples generally contain the isotopes <sup>238</sup>Pu, <sup>239</sup>Pu, <sup>240</sup>Pu, <sup>241</sup>Pu and <sup>242</sup>Pu as well as decay products, which give rise to a highly complex mix of characteristic  $\gamma$  ray energies. The date of discharge of irradiated fuel from a reactor can be verified by measuring the relative intensities of  $\gamma$  rays associated with fission and activation products. The 662 keV  $\gamma$  ray from <sup>137</sup>Cs is particularly important for this type of determination.

To detect  $\gamma$  rays the radiation must interact with the detector to give up all or part of the photon energy. The basis of all  $\gamma$  ray detector systems is the collection of this liberated electrical charge to produce a voltage pulse whose amplitude is proportional to the  $\gamma$  ray energy. In a  $\gamma$  ray spectrometer these pulses are sorted according to amplitude (energy) and counted using suitable electronics, such as a single- or multichannel analyser. With a multichannel analyser the  $\gamma$  rays analysed at different energies can be displayed or plotted to produce a  $\gamma$  ray energy spectrum which provides detailed information on the measured material.

The  $\gamma$  ray detectors most commonly used are either scintillators — usually activated sodium iodide (NaI) crystals — or solid state semiconductors — usually high purity germanium (Ge) or cadmium telluride (CdTe) crystals. The NaI detectors can be made with large volumes and generally have higher  $\gamma$  detection efficiencies than Ge detectors. Their uses in safeguards applications include, for example, the verification of fresh  $^{235}$ U fuel enrichment as well as the presence of spent fuel through detection of fission product  $\gamma$  radiation. Their ability to distinguish between  $\gamma$  rays of different energies, however, is relatively poor and of the three types of detector they have the lowest energy resolution.



**Figure 1** Comparison of  $\gamma$  ray spectrometric performance of various types of CdTe and CdZnTe detectors ( $^{235}$ U point source, 90% enrichment).

Germanium detectors have far superior energy resolution to NaI detectors and are better suited to the task of resolving complex  $\gamma$  ray spectra and providing information about the isotopic content of materials. The Ge detectors used range in size from small planar types to large (80–90 cm³) coaxial detectors. A disadvantage of these detectors is that they must be operated at very low temperature, which is usually achieved by cooling with liquid nitrogen.

Standard CdTe detectors (and CdZnTe detectors) do not need cooling and of the three detectors they have the highest intrinsic detection efficiency. Recent progress in fabrication techniques has substantially improved CdTe resolution. Until 1997 the standard volumes available were relatively small (20 and 60 mm<sup>3</sup>). Nevertheless, their portability and small size made them especially suitable for use in confined spaces, such as in verification measurements of fresh fuel assemblies whose design permits insertion of the detector probe

TABLE II GAMMA RAY SPECTROMETERS

Code	Equipment Name	Primary Applications
Low Resolution		
HM-4	Hand-held Assay Probe	Qualitative determination of U and Pu
Medium Resolution		
IMCN, IMCC	Inspector Multichannel Analyser (IMCA) with NaI or CdTe Detector	Verification of U enrichment and spent fuel
MMCN, MMCC	Miniature Multichannel Analyser (MMCA) with NaI or CdTe Detector	Verification of U enrichment and spent fuel
PMCN, PMCC	Portable Multichannel Analyser (PMCA) with NaI or CdTe Detector	Verification of U enrichment and spent fuel
High Resolution		
MCRS	Medium Count Rate Plutonium Isotopic Measurement Station	Measurement of Pu isotopic composition
PMCG, MMCG, IMCG	Multichannel Analyser with Ge Detector	Verification of UF <sub>6</sub> enrichment and spent fuel

into the assembly interior, and of spent fuel bundles stored underwater in closely packed stacks. Much larger CdZnTe detectors have recently been fabricated with volumes of up to 1500 mm<sup>3</sup>, providing a substantial increase in detector efficiency. Figure 1 illustrates the low, medium and high resolution capability of various types of CdTe and CdZnTe detectors.

A variety of  $\gamma$  ray spectrometers (multichannel analyser and detector) that differ mainly according to their resolution and analysing capability have been developed for safeguards purposes. These are summarized in Table II and further described below.

#### Low Resolution Spectrometry

HM-4. The simplest  $\gamma$  ray detecting instrument presently in use by safeguards inspectors is the battery operated Hand-held Assay Probe, developed in the early 1980s (now approaching the end of its operating life). The HM-4 has a built-in NaI scintillation detector and photomultiplier tube. Two single-channel analysers can be set to count the 186 keV  $\gamma$  radiation emitted by  $^{235}$ U and an associated background region. A four digit display shows the counts in each channel and a corrected count obtained by multiplying the background count by a selected factor and subtracting this from the 186 keV count. The HM-4 can easily be set by the inspector to cover the range 300–450 keV to measure Pu  $\gamma$  rays or to give an integrated count in the approximate energy range 150–700 keV.

#### Medium Resolution Spectrometry

**PMCA.** The most widely used  $\gamma$  ray spectrometers over the past ten years consist of a battery operated Portable Multichannel Analyser coupled to either a NaI (Figure 2) or a CdTe detector. The microcomputer based PMCA incorporates a 4096 channel analog to digital converter, a pulse amplifier, a stabilizer circuit to compensate for gain shifts in the NaI detector, and high voltage supplies for the detectors. Gamma ray spectra are displayed on a cathode ray tube. Data analysis carried out by system firmware includes  $\gamma$  ray peak location, total peak counts, spectrum smoothing, assignment of multiple 'regions of interest' and integration of counts in each region, with or without automatic background subtraction. Spectral data can be saved onto minicassette tape or transferred to an external device through a serial port.

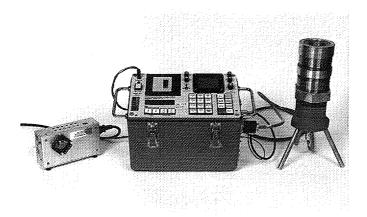


Figure 2 PMCN: Portable Multichannel Analyser (PMCA) with NaI Detector.

The PMCA can be used to carry out specific verifications whose procedural steps are installed in firmware on the microprocessor board. Two such procedures are available for <sup>235</sup>U enrichment, one for either a NaI (PMCN, low resolution) or a CdTe (PMCC, medium resolution) detector, and a second, high resolution measurement using a Ge detector (PMCG). Selection is made from a menu on the liquid crystal display. When the NaI option is chosen, the program automatically sets up parameter values for the NaI spectrometer using a table of settings stored in the memory, prompts the inspector through the measurement in a series of predefined steps and finally calculates the percentage enrichment. The same program is used with CdTe detectors (with appropriate parameter values for CdTe). Using built-in firmware for verification purposes ensures that different inspectors perform the same types of measurements in an identical manner. Application programs are also written for computers that can be connected to the PMCA.

MMCA, IMCA. The Miniature Multichannel Analyser (MMCA) and the Inspector Multichannel Analyser (IMCA) are the two successors to the PMCA. They are at the beginning of their operational life at the IAEA. They support all the detectors that had been used with the PMCA, including the NaI, CdTe and high purity Ge detectors. The MMCA (Figure 3) is significantly smaller and lighter than the PMCA and in operation the battery lifetime is three times longer (at least 12 h for CdTe and NaI detectors). The MMCA has the form of a palmtop computer and weighs 680 g, including the lithium ion battery. Combined with a palmtop computer and a CdTe detector it makes a powerful yet versatile system that fits into half a briefcase, which is very convenient for many inspection activities. The IMCA is chiefly being used at

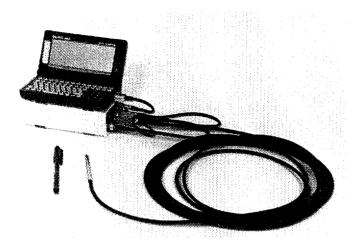


Figure 3 MMCC: Miniature Multichannel Analyser (MMCA) with CdTe Detector.

present in unattended monitoring applications, but will also serve all of the normal portable functions of the PMCA.

#### High Resolution Spectrometry

**PMCG**, **IMCG**. When coupled to a Ge detector the PMCA, MMCA or IMCA becomes a high resolution  $\gamma$  ray spectrometer. This type of spectrometer is often used to determine the  $^{235}$ U enrichment of uranium hexafluoride (UF<sub>6</sub>) in shipping cylinders. After selecting this procedure from the options menu in the firmware, the inspector is led through a series of predetermined steps to calculate the enrichment. The cylinder wall thickness must also be determined, so that corrections for  $\gamma$  absorption in the wall can be made. This measurement is made by means of an ultrasonic thickness gauge.

A new software code, MGAU, is available to assist the inspector in simplifying the measurement and analysing the high resolution spectra. MGAU can provide results with 1–2% accuracy provided that the container wall thickness is less than 10 mm of steel and the Th daughter activities are in equilibrium with the parent  $^{235}$ U and  $^{238}$ U activities. This code removes the need for a wall thickness measurement or calibration.

MCRS. The superior resolution of Ge detectors (now also possible with special cooled pin CdTe detectors, see Figure 1) enables them to isolate and



**Figure 4** MCRS: Medium Count Rate Plutonium Isotopic Measurement Station with Ge detector.

measure  $\gamma$  ray peaks in the complex spectra from Pu to determine the Pu isotopic composition. The isotopic fractions must be known so that, when combined with either a neutron coincidence counter or calorimeter measurement, the total Pu mass in a material can be determined. The Medium Count Rate Plutonium Isotopic Measurement Station (MCRS) employs a commercially available multichannel analyser for fixed station, high resolution measurements (Figure 4).

#### **Neutron Counting**

The IAEA uses a number of different types of neutron counting equipment (Table III). This section gives information on the source of the neutrons, the importance of neutron coincidence counting to obtain the mass of the fissile fuel, and a few examples of passive and active detector systems.

Neutron Emission and Detection for Non-irradiated Fissile Fuel

Neutrons can be emitted from non-irradiated nuclear fuel in three ways:

- Spontaneous fission from fissile isotopes such as those of Pu;
- Induced fission from fissile isotopes (typically by means of a low energy neutron source);

TABLE III COINCIDENT-NEUTRON DETECTOR SYSTEMS FOR NON-IRRADIATED FISSILE FUEL

Code	Equipment Name	Primary Applications
Passive 1	Neutron Coincidence Counters	
BCNC	Bird Cage Counter	Verification of Pu mass in special storage configurations
CMOX	CANDU MOX Bundle Counter	Verification of Pu mass in CANDU MOX fuel assemblies
CNCM	Compact Neutron Coincidence Counter	Verification (qualitative) of MOX fuel assemblies in shipping crates
DRNC	Drawer Counter	Verification of Pu mass in facility specific containers
FAAS	Fuel Assembly/Capsule Assay System	Verification of Pu mass in MOX fuel assemblies
FPAS	Fuel Pin/Pallet Assay System	Verification of Pu mass in MOX fuel pins in facility specific storage trays
GBAS	Glove Box Assay System	Semiquantitative determination of Pu hold-up in glove boxes
HBAS	Hold-up Blender Assay System	Semiquantitative determination of Pu hold-up in facility blenders
HLNC	High Level Neutron Coincidence Counter	Verification of Pu in 20–2000 g canned samples (pellets,
INVS	Inventory Sample Counter	powders, scrap) Verification of Pu in 0.1–300 g samples. Versions can be
LNMC	Large Neutron Multiplicity Counter	attached to glove boxes Verification of Pu in contaminated/impure items
MAGB	Glove Box Counter	Verification of Pu mass in facility glove boxes
PCAS	Canister Counter	Verification of Pu mass in MOX canisters
PLBC	Plutonium Bottle Counter	Verification of Pu mass in Pu nitrate storage bottles
PNCL	Plutonium Neutron Coincidence Collar	Verification of Pu mass in MOX fuel assemblies
PSMC	Plutonium Scrap Multiplicity Counter	Verification of Pu in 1–100 g canned samples of scrap
PWCC	Passive Well Coincidence Counter	Verification of Pu mass in CANDU MOX fuel bundles

TABLE III (continued)

Code	Equipment Name	Primary Applications
UFBC	Universal Fast Breeder Counter	Verification of Pu (up to 16 kg) in FBR fuel
UWCC	Underwater Coincidence Counter	Verification of Pu in MOX fuel assemblies underwater
Active N	eutron Coincidence Counters	
AWCC	Active Well Coincidence Counter	Verification of <sup>235</sup> U in high enriched U samples
UNCL	Uranium Neutron Coincidence Collar	Verification of <sup>235</sup> U in low enriched U fuel assemblies. Variety of collar configurations available
WDAS	Waste Drum Assay System	Interrogation of low level waste drums for <sup>235</sup> U mass

• Alpha particle induced reactions,  $(\alpha,n)$ , involving light elements such as oxygen and fluorine.

Fission neutrons in the first two categories are emitted in groups of two or more for each fission event. This signature is detected as a neutron coincidence. Nearly all the isotopes of U, Pu and other transuranium elements emit  $\alpha$  particles. These interact with light elements present in compounds (e.g. oxides and fluorides) or as impurities (e.g. B, Be and Li) to form an undesirable neutron background. Neutron coincidence counting discriminates against this background by processing the neutron pulses to select correlated (in time) detection events and eliminating the  $(\alpha,n)$  background neutrons, which are emitted singly and thus are uncorrelated.

Passive detector systems determine the mass of Pu on the basis of the spontaneous fission of primarily its even-numbered isotopes ( $^{238}$ Pu,  $^{240}$ Pu and  $^{242}$ Pu, with  $^{240}$ Pu the dominant contributor). The major fissile isotope,  $^{239}$ Pu, has a typical abundance in fuel of 60–80% yet it contributes insignificantly to spontaneous fission. Isotopic abundance must be known or verified — typically by a high resolution  $\gamma$  ray measurement. Using the isotopic abundance the coincident-neutron count rates can then be converted into a value for the total Pu mass in a sample. For uncontaminated samples, measurement accuracy is of the order of 1% or less.

The fissile isotope <sup>235</sup>U does not undergo sufficient spontaneous fission for practical passive detection. In this case an active system incorporating AmLi neutron sources is used to 'interrogate' the <sup>235</sup>U content by neutron induced fission. For low energy incident neutrons the <sup>238</sup>U in a sample contributes insignificantly to the measured coincident-neutron count rates even though <sup>235</sup>U may be enriched to only a few per cent (e.g. low enrichment fuels).

Neutron detectors employ various neutron capture reactions to function. The reactions produce energetic particles, which in turn ionize a gas and produce a charge pulse. The choice of detector (i.e. the capture material) is based mainly on the neutron detection sensitivity required and on the insensitivity to other radiation, e.g.  $\gamma$  rays. Nearly all detectors have the highest sensitivity for low energy neutrons. Consequently in many neutron detector systems the detectors are surrounded with a moderator material such as polyethylene to slow down the energetic neutrons to thermal energies.

#### Gross Neutron Counting

Gross neutron counting refers to the sum of all neutrons detected. Here the neutron source cannot be characterized since coincidence requirements are not applied. The presence of significant numbers of neutrons is often a sufficient indication that fissile nuclear material is present. All the neutron coincidence detection systems (discussed below) give total neutron count rates as well as coincidence count rates.

Other detector systems, such as the Fork Detector and the Unattended Fuel Flow Monitor, employ gross neutron counting as their primary function. These systems mainly measure spent fuel and will be presented in more detail in other sections.

#### Neutron Coincidence Counting

Neutron coincidence counting has evolved into a very stable, reliable and accurate technique to determine Pu and <sup>235</sup>U content. Modern, well designed neutron coincidence systems are capable of reliably processing pulses over a very large range of input count rates (i.e. over more than six orders of magnitude). This stability is achieved by judicious selection and placement of amplifier electronics to minimize noise interference. These electronics boards, when located at the detector head, amplify and shape the pulses, apply lower

level discrimination to remove  $\gamma$  pulses or noise, and feed out very narrow (50 ns wide) logic pulses to the external pulse processor (the electronics controller).

Reliable coincidence counting is also due to a sophisticated pulse processing circuit (shift register electronics) in the external electronics controller. Pulses within a specified time period (normally set at 64  $\mu s$ ) of one another may be termed correlated (i.e. 'coincident') neutron pulses. This correlation time is associated with the slowing down of neutrons in the moderator. The shift register electronics keep track of coincidences between pulses separated by about 1000  $\mu s$  (accidentals) and coincidences in the first 64  $\mu s$  (real coincidences plus accidentals) and subtract the former to give the real coincidences. Other small corrections are also automatically applied.

• Passive detector systems have one of two basic geometrical configurations: well detectors completely enclose the sample, and collar detectors encircle the sample (e.g. a fuel assembly). Well detectors have the preferred geometry since they have the capability to detect all the neutrons emanating from the sample. Collar detectors are an alternative detector design that is appropriate when the sample becomes too large for placement inside a well detector. Whereas calibrated passive well detectors measure the total mass of Pu in a sample, collar detectors measure Pu mass per unit length of the fuel assembly. This linear density must then be multiplied by an effective length to give the total Pu mass in the assembly.

About twenty passive detector systems are currently used in nuclear safeguards, with their design features optimized for specific sample sizes, shapes or Pu mass ranges. These passive detector systems are listed in Table III along with their primary applications. Two representative systems are described below.

HLNC. The High Level Neutron Coincidence Counter is shown in Figure 5. This detector system is typical of IAEA coincidence counting systems used with non-irradiated fissile material. The HLNC includes a detector head which houses the neutron detectors (<sup>3</sup>He gas proportional counters) connected to special amplifiers. Separately mounted are the electronics controller, which powers the amplifiers and processes the train of pulses to determine coincidence events; the portable computer, which automates measurement cycles and analyses and stores the data; and the printer, which presents results in a concise report format. This 60 kg detector features a large sample cavity and 18% neutron detection efficiency. By removal of the top

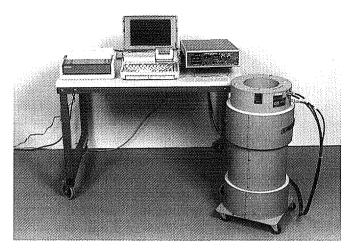


Figure 5 HLNC: High Level Neutron Coincidence Counter.

end cap, a can containing Pu (in pellet, powder or scrap form) can be centred in this large cavity. The sample is given an identification number in the computer, an appropriate calibration curve is chosen and a run time (typically 100 s) is selected. Upon initiation of the measurement, the IAEA neutron coincidence counting (INCC) program automatically runs through a sequence of measurements (typically three) which must pass all the criteria for acceptable results. The Pu mass is then calculated and compared with the declared value to provide a quantitative verification that for typical high purity Pu inventories is accurate to 1% or better.

INVS. For small samples (bagged Pu pellets, powders and solutions in vials) with much lower total Pu content than those measured with the HLNC, the Inventory Sample Counter is the detector system of choice. The INVS has nearly twice the neutron detection efficiency of the HLNC. Figure 6 presents one of four versions of this portable detector system. In another version, the INVS has an inverted geometry and is permanently attached to the floor of a glove box so that samples can be assayed for Pu content without the inconvenience and inefficiency of removal from the glove box. Although the cavity is typically only about 6 cm in diameter by 16 cm high, it is well matched to sample geometry at facilities such as fuel fabrication plants and provides very reliable Pu content verification (with an accuracy of 1% on individual measurements). Measurement procedures are again automated with the INCC program and are essentially the same as for the HLNC.

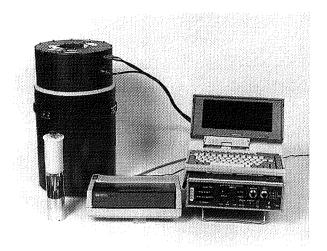


Figure 6 INVS: Inventory Sample Counter.

• Active detector systems use neutron sources (typically AmLi) to interrogate the <sup>235</sup>U in the sample. Again the well geometry is preferred but the collar geometry is the only practical solution when the sample is a fuel assembly. The active detectors in use in IAEA safeguards are listed in Table III. Details of an active well detector and an active collar detector are presented below. The full detector system includes the detector head, which detects the neutrons and houses the neutron source; the electronics controller, which powers the detector and determines the neutron coincidence rates; the portable computer for control and data analysis to determine <sup>235</sup>U content; and the printer for generating reports.

AWCC. The Active Well Coincidence Counter has a massive (150 kg) detector head permanently attached to a wheeled cart for transportability (Figure 7). The AWCC has 42 <sup>3</sup>He counters and thick polyethylene walls, which result in its high (nearly 30%) neutron detection efficiency. The <sup>235</sup>U content of the sample is interrogated with two AmLi neutron sources placed above and below to provide uniform neutron interrogation over the material volume. The cavity size is adjustable up to about 20 cm in diameter and 23 cm in height by removal of inserts and reflectors to accommodate samples at fuel fabrication facilities such as metal disks, canned metal oxide powders and fuel pebbles in carousels. The INCC program is used to automate the measurement procedure and analyse the data to assay the <sup>235</sup>U content to high accuracy.

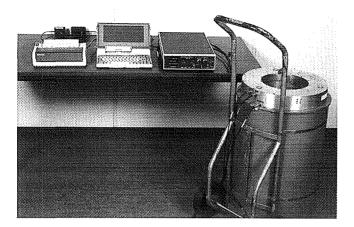


Figure 7 AWCC: Active Well Coincidence Counter.

**UNCL.** The Uranium Neutron Coincidence Collar is mounted on a cart. When used at a facility, the door containing the neutron source is removed and the collar is wheeled into position around the fuel assembly. Once the door with the AmLi source is reinserted in the slots, the measurement cycle is initiated. After a specific number of measurements have passed the acceptance criteria in the INCC program, the <sup>235</sup>U mass per unit length is calculated. This is combined with a measurement of the effective length to give the <sup>235</sup>U content of the entire fuel assembly.

#### Spent Fuel Measurement

Neutron Emission and Detection

Spontaneous fission of  $^{242}$ Cm and  $^{244}$ Cm is the major source of neutrons emanating from spent fuel. These isotopes are produced in the nuclear reactor fuel assemblies through multiple neutron capture events. The fission products in the spent fuel produce an extremely high radiation background in which the neutrons must be detected. This high radiation environment determines the techniques that can be deployed for spent fuel verification. One approach is to choose a detector which is basically  $\gamma$  ray insensitive. Another approach is to shield against the  $\gamma$  rays but allow the neutrons to pass

through the shield to the neutron detector. Spent fuel verification methods include not only neutron detection but also  $\gamma$  ray and ultraviolet light (Cerenkov radiation) detection.

Table IV lists the spent fuel measurement systems in use by the IAEA. The Fork Detector (FDET) incorporates both neutron and  $\gamma$  ray detectors to verify the irradiation history, the initial fuel content and the number of reactor exposure cycles of a fuel assembly. Several detector systems are available to measure the gross  $\gamma$  ray intensity (CPMU),  $\gamma$  ray energy spectra (SFAT, HSGM and GBUV), and  $\gamma$  ray intensity as a function of fuel bundle storage position (CBVB and CBVS). Cerenkov glow viewing devices (ICVD and ACVD) examine the ultraviolet light that appears in the water surrounding spent fuel. These measurement systems are described in more detail below.

TABLE IV SPENT FUEL MEASUREMENT SYSTEMS

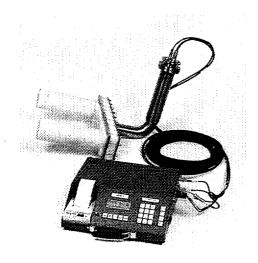
Code	Equipment Name	Description and Applications		
Cerenko	Cerenkov Glow Detectors			
ACVD	Advanced Cerenkov Viewing Device	Highly sensitive Cerenkov viewing device using an integrating video camera to increase sensitivity •		
ICVD	Cerenkov Viewing Device	Hand-held light intensifying device optimized to view Cerenkov light (near ultraviolet) in a spent fuel storage pond. System can be used in a lighted area. Primarily used to identify irradiated LWR fuel assemblies		
Spent Fuel Radiation Detectors				
CBVB CBVS	CANDU Bundle Verifier for Baskets CANDU Bundle Verifier for Stacks	Attended radiation monitoring systems that scan storage baskets or stacks of CANDU fuel bundles and record $\boldsymbol{\gamma}$ intensity as a function of detector position		
CPMU	High Range Underwater Monitor	General purpose monitor used underwater to detect the intense γ radiation from irradiated fuel assemblies		

**TABLE IV** (continued)

Code	Equipment Name	Description and Applications
FDET	Fork Detector Irradiated Fuel Measuring System	Detector system that straddles LWR fuel assemblies with pairs of neutron and $\gamma$ ray detectors. Gross $\gamma$ ray and neutron intensities and ratios of intensities can give specific information on the fuel assembly
GBUV	Gamma Burnup Verifier	Facility specific system used to make high resolution $\gamma$ ray measurements of spent fuel assemblies. Collimator in front of the Ge detector is built into the facility
HSGM	High Sensitivity Gamma Monitor	General purpose monitor used underwater to detect the intense $\gamma$ radiation from irradiated fuel assemblies
SFAT	Spent Fuel Attribute Tester	Gamma ray detector system that is positioned above stored fuel assemblies and measures the collimated radiation from an area below the detector

#### Gross Neutron and YRay Detection

**FDET.** The Fork Detector Irradiated Fuel Measuring System (Figure 8) includes the detector head, an extension pipe several metres long (not shown), a Gamma Ray and Neutron Detector (GRAND) electronics unit and a portable computer. The head incorporates  $\gamma$  ray insensitive neutron detectors (four gas filled fission chamber proportional counters) and  $\gamma$  ray detectors suitable for measuring extremely high  $\gamma$  ray intensities (two gas filled ionization chambers). The neutron and  $\gamma$  ray signatures measured by the detectors are used to verify the highly radioactive spent fuel assemblies stored underwater in spent fuel ponds. The head is positioned about 1 m above the tops of neighbouring assemblies. The irradiated fuel assembly is lifted so that the head straddles the fuel portion of the assembly with one pair of bare fission proportional counters and with a second pair of cadmium covered fission proportional counters. This arrangement is used to gain information about neutron capture due to boron in the water of the pool. In



**Figure 8** FDET: Fork Detector Irradiated Fuel Measuring System (detector head, GRAND electronics unit and portable computer).

addition the FDET can be used to measure the  $\gamma$  ray flux with two ionization chambers straddling the assembly. The ratio of neutron to  $\gamma$  ray counts, when combined with other, complementary information, is used to characterize a particular type of fuel assembly, giving information related to its neutron exposure in the reactor, its initial fissile fuel content and its irradiation history (e.g. the number of cycles for which the assembly was in the reactor).

#### Gross Y Ray Detection

Two other instruments, the High Range Underwater Monitor (CPMU) and High Sensitivity Gamma Monitor (HSGM), are available to provide qualitative confirmation of the presence of spent fuel or other irradiated items when other preferred methods, such as by means of the Spent Fuel Attribute Tester or Cerenkov viewing (both described below), cannot be used. In contrast to the use of other instruments, each item to be examined by the CPMU or HSGM must be isolated from neighbouring materials and so may involve the moving of items in the storage pond.

**CPMU.** The CPMU is an underwater survey meter with a battery operated electronics package connected by an 18 m long waterproof cable to one of two

ionization chamber detectors. The choice of detector depends on the radiation level. The system spans a dose rate range up to 258  $\text{C-kg}^{-1}\cdot\text{h}^{-1}$  (10<sup>6</sup> R/h).

**HSGM.** The HSGM has a choice of two Geiger–Müller probes used with a microprocessor based data processing unit having a liquid crystal display readout. The instrument can be operated from either a battery or an AC power source. Each probe is permanently connected to a 25 m long water-proof cable terminating in a connector to the processing unit. The HSGM measures  $\gamma$  and X rays in the 60 keV to 3 MeV range and together the probes cover a dose rate range up to  $7.74 \text{ C·kg}^{-1} \cdot \text{h}^{-1}$  (30 000 R/h).

#### Gamma Ray Energy Spectral Analysis

**SFAT.** The Spent Fuel Attribute Tester consists of a multichannel analyser and a NaI or CdTe detector specifically mounted to be used underwater in spent

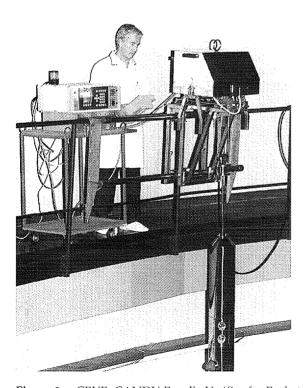
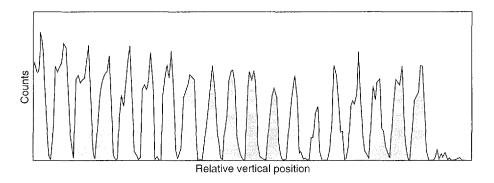


Figure 9 CBVB: CANDU Bundle Verifier for Baskets.

fuel ponds. The SFAT provides a qualitative verification of the presence of spent fuel through detection of particular fission product γ rays — either from <sup>137</sup>Cs (662 keV) for fuel which has cooled for longer than four years or from <sup>144</sup>Pr (2182 keV) for fuel with a shorter cooling time. The SFAT is particularly helpful in situations where Cerenkov viewing cannot provide verification, e.g. in the case of spent fuel that has a low burnup or that has cooled for a long period, where the Cerenkov radiation is too weak or where the water in the storage pond is insufficiently clear. The detector and its lead shielding are housed in a stainless steel watertight container, which can be submerged in a storage pond and positioned over the items to be examined. A watertight pipe is attached below the detector housing so that only radiation from the source rather than from adjacent materials reaches the detector. The multichannel analyser provides acquisition, recording and analysis of data as well as supplying power to the detector. The intensity of the selected  $\gamma$  rays from the fuel assembly is compared with that from the gap separating the assembly from its neighbour.

#### Gamma Ray Intensity Scanning

**CBVB**, **CBVS**. The CANDU Bundle Verifier, for scanning either storage baskets (Figure 9) or storage stacks, includes a highly collimated and shielded CdTe detector suspended on a winch with settable speed. The detector is attached to an amplifier and a portable computer (neither is shown in the figure). The computer can be used either with an external analyser for high count rate conditions or with an internal multichannel analyser card for



**Figure 10** Vertical intensity scan of CANDU spent fuel bundles ( $^{137}$ Cs 662 keV  $\gamma$  ray peak) using the CBVB.

moderate count rate applications. The  $662 \text{ keV} \gamma \text{ ray line from}^{137}\text{Cs}$  stands out well above the background produced by the other radioactivity for spent fuel that has cooled for more than two years. For shorter times the 757 keV line from  $^{95}\text{Nb}/^{95}\text{Zr}$  is used to verify the presence of spent fuel. The particular  $\gamma$  ray line is selected in the SCANDU program. Then the detector head is moved at a selected speed vertically across the face of the stacked fuel and at the same time a scan sequence is initiated in the computer. The  $\gamma$  ray intensity is measured against vertical position and the high intensity peaks, indicating irradiated fuel bundles, are counted and compared with the declared information on the stored fuel bundles. An example of such an intensity scan is shown in Figure 10.

#### Cerenkov Radiation Detection

ICVD, ACVD. The Cerenkov Viewing Device (ICVD) and Advanced Cerenkov Viewing Device (ACVD) are image intensifier viewing devices that are sensitive to the ultraviolet radiation in the water surrounding spent fuel. The ICVD, which is hand-held, is shown in Figure 11. These viewing devices are capable of operating with facility lights turned on in the spent fuel pond area. They are optimized for ultraviolet radiation by filtering away most of the visible light and by having an image intensifier tube sensitive primarily to the ultraviolet. Cerenkov radiation is derived from the intense y radiation from spent fuel, which when absorbed in the water produces recoiling high energy electrons. In many cases these electrons exceed the speed of light and therefore must lose energy by emitting radiation (Cerenkov radiation). Furthermore, spent fuel also emits β particles (which are also energetic electrons), adding to the Cerenkov radiation. Spent fuel assemblies are therefore characterized by Cerenkov glow patterns that are bright in the regions immediately adjacent to the fuel rods. This variation in light intensity is apparent when viewed from an aligned position directly above the fuel rods. This behaviour can even be used to distinguish an irradiated fuel assembly from a non-fuel item which may look the same since non-fuel items do not display a strong intensity variation with alignment. Typically a row of fuel assemblies are viewed vertically from the bridge while the facility operator runs the bridge down the row. One inspector views the items in the row through the device and declares each as spent fuel, a vacancy or another item while a second inspector compares the measured results with the facility declarations. The ACVD is used in only a small number of locations for very old or very low burnup spent fuel.

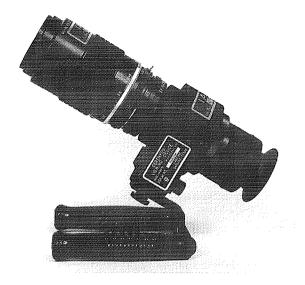


Figure 11 ICVD: Cerenkov Viewing Device.

#### Other NDA Techniques

Radiation Measurement

**KEDG.** The K-edge Densitometer is facility owned equipment used by the IAEA to determine the Pu concentration in solutions. The system consists of a high resolution Ge detector, a multichannel analyser and a portable computer. A  $^{57}$ Se/ $^{57}$ Co source of low energy  $\gamma$  rays is positioned for the  $\gamma$  radiation to pass through the solution. The absorption of this radiation gives a sensitive measure of the Pu in its path.

**TLDS.** The neutron sensitive Thermoluminescent Dosimeter is a compact portable device that measures neutron exposure (dose) and is made up of lithium fluoride crystals enriched in <sup>6</sup>Li. Dosimeters of this type are used to verify that the neutron level has not exceeded some threshold value, i.e. that spent fuel has not entered a particular area. Periodically the dosimeters are sent back to IAEA Headquarters, where the neutron dose is determined by measuring the amount of light emitted by the dosimeters as they are slowly heated.

#### Physical Property Measurement

The IAEA also uses equipment to measure such quantities as the weight of an object (LCBS), the wall thickness of a container (ULTG) or the liquid level in a tank (Electromanometer, ELTM).

LCBS. The Load Cell Based Weighing System (Figure 12) operates in two load ranges up to 5000 and 20 000 kg and provides a convenient and rapid means of determining the gross weight of bulky, massive objects such as UF<sub>6</sub> shipping cylinders. The load cell construction includes two shackles with a load supporting element between that is bonded to a strain gauge. As the load is lifted with the hoist, the strain gauge deforms and its electrical resistance changes. This change is converted to a weight, which is displayed on a digital readout unit attached by a cable to the load cell. Typically, gross weights are determined with this system to an accuracy of better than 1%.

**ULTG.** The Ultrasonic Thickness Gauge is a small hand-held device with a digital readout that measures wall thickness on the basis of the round-trip flight time of the ultrasonic waves reflected from the inner wall. This thickness information is needed to correct for radiation attenuation by container walls such as  $UF_6$  shipping containers and  $UO_2$  hoppers and cans. These

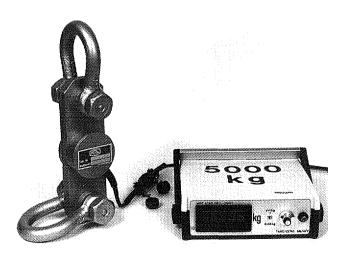


Figure 12 LCBS: Load Cell Based Weighing System.

corrections are particularly important where the container wall thickness varies. Using the standard probe, the typical measurement range for steel extends from 1.2 to 200 mm. In the standard mode of operation, the speed of the ultrasonic waves in the particular medium is stored in the unit so that the flight time can be internally converted directly to a wall thickness, which is displayed on the readout.

## DESTRUCTIVE ANALYSIS

Destructive analysis (DA) measurements for element assay and determination of isotopic composition can be made on all types of solid and liquid materials encountered in bulk handling nuclear plants. DA is used in the following ways:

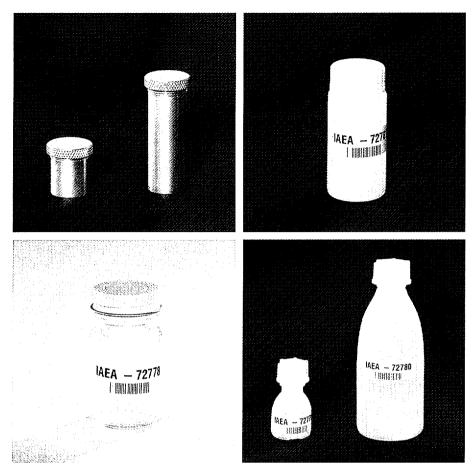
- To verify that protracted diversion of safeguarded nuclear materials has not occurred;
- To certify working standards used for the calibration of NDA and installed verification instruments;
- To provide assurance of the quality and independence of on-site measurements (e.g. validation of facility specific procedures);
- To carry out periodic verifications of the operator's measurement system.

DA verification measurements involve the following steps:

- (1) The taking of independent samples;
- (2) Their conditioning at the facility to ensure that they are in a chemical form adequate for maintaining their integrity during transport;
- (3) Their packaging, sealing and shipment to the IAEA Safeguards Analytical Laboratory (SAL) in Seibersdorf, near Vienna;
- (4) Their analysis by SAL or the Network of Analytical Laboratories (NWAL, consisting of laboratories in different States that have been certified to analyse safeguards relevant samples);
- (5) The statistical evaluation of the results of their analysis.

To obtain meaningful and sufficiently accurate results, it is necessary to apply optimized and validated procedures for each of these steps.

Bulk measurement is generally considered to be part of sampling. The sample related bulk data collected on-site by the inspector concomitantly with the sampling include the weights or volumes of the sampled items or batches as declared by the operator and verified by the inspector. In addition to the bulk data, the operator's declarations for the elemental and isotopic compositions of the material sampled are recorded in a working paper. This working paper provides instructions for the sample amounts to be taken and the most appropriate sample bottle to be used. Specific types of sample bottles have been selected and tested by the IAEA for taking and shipping samples of various types of materials (Figure 13).



**Figure 13** Sample bottles used for IAEA verification samples. Top left: vials for Pu, MOX or high enriched U powder. Top right: hard polyethylene bottle for hard or solid materials. Bottom left: glass bottle for depleted, natural or low enriched U powder. Bottom right: soft polyethylene bottles for U or Pu solutions (10 or 100 mL).

The main analytical techniques applied in DA measurements are summarized in Table V. The measurement precisions and accuracies reflected in the table by the random and systematic uncertainties, respectively, are values achieved in the analysis of materials of nuclear grade or similar chemical purity. They include the contributions of all uncertainties occurring after sampling. The effects of sampling, impurities and foreign components will vary with the type of material, to the extent that sampling uncertainties can become the dominant factor in the total measurement error.

**TABLE V** MAIN ANALYTICAL TECHNIQUES USED BY THE SAFE-GUARDS ANALYTICAL LABORATORY AND THE NETWORK OF ANALYTICAL LABORATORIES

	Analysed	Type of	Uncertainty (%rel.)	
Analytical Technique	for:	Material	Random	Systematic
Elemental Analysis				
NBL Davies and Gray titration	U	U, U–Pu, U–Th <sup>a</sup>	0.05	0.05
MacDonald and Savage titration	Pu	Pu materials <sup>a</sup>	0.1	0.1
Controlled potential coulometry	Pu	Pure Pu materials	0.05	0.05
Ignition gravimetry	U, Pu	U oxides, Pu oxides	0.05	0.05
K-edge X ray densitometry	U, Th, Pu	U, Pu, U–Pu, U–Th <sup>a</sup>	0.2	0.2
K X ray fluorescence analysis	Pu	Pu materials <sup>a</sup>	0.2	0.2
Wavelength dispersive X ray fluorescence spectrometry	Pu, U	Pure U and Pu oxides, and MOX <sup>a</sup>	0.3	0.3
Isotopic Analysis				
Isotope dilution mass spectrometry	U, Pu	Spent fuel input solutions and Pu and U-Pu materials	0.1	0.1
Thermal ionization mass spectrometry	U and Pu isotopes	All Pu and U materials, and spent fuel input solutions	0.05 <sup>b</sup>	0.05 <sup>b</sup>
High resolution $\gamma$ ray spectrometry (Ge detector)	Pu isotopes, Am, Np	Pure U and Pu materials	0.5–2.0	0.5–2.0
Gamma ray spectrometry (NaI detector)	235U	Low enriched U materials	0.2-0.5	5 0.2–0.5
Alpha spectrometry	<sup>238</sup> Pu	Pu materials	0.2	0.3
Laser fluorimetry	Np	Pu materials	2.0	2.0
Plutonium(VI) spectrophotometry	Pû	Pu, U–Pu <sup>a</sup>	0.2	0.2

a Except spent fuel.

# Elemental Analysis

Uranium by Potentiometric Titration

The New Brunswick Laboratory Davies and Gray titration is the basic method for the determination of U content in gram size samples of all types of

<sup>&</sup>lt;sup>b</sup> For ratios of major isotopes.

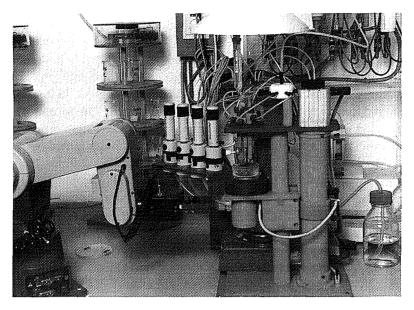


Figure 14 Automatic U titrator.

non-irradiated materials. An automated titration system (Figure 14), developed at SAL, achieves in routine operation measurement precisions and accuracies of 0.05%rel. or better. This method is applicable to samples of any U materials containing at least 50 mg U so that at least four replicate aliquots of the sample, containing at least 10 mg U each, can be titrated.

## Plutonium by Potentiometric Titration

The MacDonald and Savage titration is used for the determination of Pu content in gram size samples of non-irradiated nuclear materials. It provides precisions and accuracies of 0.1%rel. or better. This method has been designed to determine 2–4 mg Pu in nitric acid solutions. It is suitable for the direct determination of Pu in nuclear materials ranging from Pu from a reprocessing plant to fresh reactor fuel materials with a U:Pu ratio of up to 30.

## Plutonium by Controlled Potential Coulometry

Controlled potential coulometry (Figure 15) is used to determine 1–2 mg Pu after quantitative chemical separation from potential interfering species. The

separation procedure removes Np and other interfering elements to permit measurement precisions and accuracies of the order of 0.05%rel. with samples of small size.

Coulometry can be used also to determine Pu in samples of industrial materials provided that chemical separation is first carried out to remove potential interfering elements. The technique applies to gram size Pu samples, such as Pu product solutions, Pu metal and Pu oxide powders or

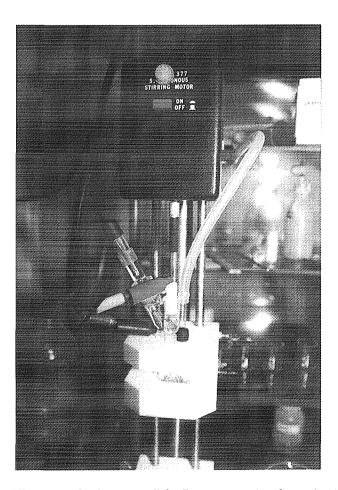


Figure 15 Coulometry cell for Pu concentration determination.

pellets, and to mixed U and Pu oxides, after dissolution of the solid sample. The standard separation procedure involves anion exchange chromatography.

## Uranium or Plutonium by Ignition Gravimetry

Ignition gravimetry is applied for determining U and Pu concentrations in nuclear grade U and Pu oxides. An accurately weighed sample is converted to stoichiometric  $\rm U_3O_8$  by ignition in air to a constant mass at 900°C  $\pm$  10°C for U and to stoichiometric  $\rm PuO_2$  at 1200°C  $\pm$  10°C for Pu. The amount of U or Pu in the sample is calculated using a gravimetric conversion factor for  $\rm U_3O_8$  to U or  $\rm PuO_2$  to Pu, depending on the isotopic composition of the sample. The precision and accuracy for nuclear grade oxides containing less than 200 ppm of impurities are of the order of 0.05%rel. or better.

The presence of non-volatile impurities (the more frequent and abundant being Am, Np, Fe, Si, Al and Ca) requires a correction, based on the impurity content determined by inductively coupled plasma–mass spectrometry.

## Uranium, Thorium or Plutonium by K-edge X Ray Densitometry

K-edge X ray densitometry is applicable to all U, Th and Pu materials and to mixed U–Th or U–Pu samples containing a sufficient amount of the analyte: a precision and accuracy of about 0.2%rel. may be achieved when the concentration of the analyte ranges from 80 to 150 g/L. The method is very selective, but the determination of the major actinide, such as Pu, may be biased by the presence of a minor actinide element of lower atomic number, such as U. The software used at SAL for dual element samples corrects most of the mutual bias effects.

#### Plutonium by K X Ray Fluorescence Analysis

K X ray fluorescence analysis is applied to samples of PuO<sub>2</sub> and Pu nitrate solutions containing at least 3–4 mg Pu with the addition of known amounts of U as an internal standard. It is also used for Pu assay in samples of MOX and U–Pu nitrate solutions in combination with a determination of the U content by titration or by K-edge X ray densitometry. A precision and accuracy of 0.2%rel. are achievable.

Plutonium and/or Uranium by Wavelength Dispersive X Ray Fluorescence Spectrometry

X ray fluorescence spectrometry is used in conjunction with a commercial high frequency furnace for fast analysis of Pu and mixed U–Pu oxides (Figure 16). About 0.3 g of the sample is melted in a lithium borate flux and the melt cast into a platinum dish, producing a borate disk of very homogeneous composition. The concentrations of Th, U, Np, Pu and Am can be determined simultaneously by measuring the fluorescence of the  $L_{\alpha}$  lines. The calibration curves are linear over at least a tenfold change in concentration. A complete analysis can be performed in about 15 min, with a reproducibility of about 0.3% for the concentrations of the major heavy elements.

X ray fluorescence spectrometry is also used to determine semiquantitatively in various types of samples the concentrations of major, minor and trace elements with atomic masses from 9 (fluorine) through to 89–103 (the actinide elements).



Figure 16 X ray fluorescence spectrometer.

# Isotopic Analysis

Uranium or Plutonium by Isotope Dilution Mass Spectrometry

Isotope dilution mass spectrometry is applied for U or Pu determinations in all samples of spent fuel input solutions, but also for samples of low content, such as milligram size U–Pu samples and wastes.

For U and/or Pu determinations in high burnup spent fuel input solutions, an aliquot of the sample solution is spiked with a known amount of a certified tracer containing enriched <sup>235</sup>U and <sup>239</sup>Pu. For pure U materials, a spike of <sup>233</sup>U is used; for pure Pu materials or low burnup spent fuel, a spike of <sup>242</sup>Pu or <sup>244</sup>Pu is used. Spiked solutions of Pu-bearing materials are chemically treated to attain an isotopic equilibrium of Pu. Two spiked aliquots and an unspiked aliquot are separately purified by reversed phase chromatography in order to provide pure fractions for thermal ionization mass spectrometry (see below). The chemical treatment of spent fuel samples is performed with a fully automatic, robotized system (Figure 17). The resulting U and Pu

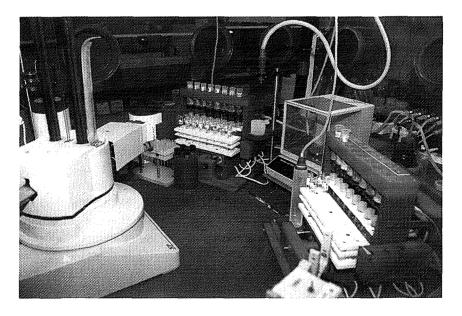


Figure 17 Robotized system for separation of spent fuel input solution samples.

fractions are then evaporated to dryness and redissolved in nitric acid to yield solutions containing about 1  $\mu g$  U and 50 ng Pu per microlitre. The isotopic ratios of both the spiked and unspiked aliquots are measured by thermal ionization mass spectrometry and the U and Pu contents are calculated accordingly.

When the original sample can be spiked directly and total evaporation mass spectrometry measurements are done, the elemental assays have a precision and accuracy of 0.1%rel. or better.

Uranium or Plutonium Isotopic Composition by Thermal Ionization Mass Spectrometry

Thermal ionization mass spectrometry, employing two multidetector mass spectrometers, each equipped with nine Faraday cups, is used to measure the U or Pu isotopic composition of all samples of nuclear materials submitted to SAL. Comprehensive new software, developed in co-operation with the Institute of Reference Materials and Measurements in Geel, Belgium, and the Los Alamos National Laboratory in the United States of America, includes routines for basic calibration steps, such as the cup linearity test, relative cup efficiency measurements and a system calibration of mass fractionation effects.

Isotope ratios of 0.05–20 can be measured with a precision and accuracy of 0.05% rel. using a data collection procedure involving total evaporation of the sample loaded on the filament. This procedure greatly reduces the mass fractionation effects.

Plutonium Isotopic Composition by High Resolution γ Ray Spectrometry

High resolution  $\gamma$  ray spectrometry is used to screen all Pu samples as they are received at SAL. The Pu content of samples containing Np is analysed by isotope dilution mass spectrometry rather than by potentiometric titration. SAL has thereby acquired considerable experience in isotopic analyses using a multipurpose  $\gamma$  ray spectrometry analysis program called MGA ('Multi-Group Analysis').

Samples containing Pu are placed, in their original packaging, on a planar Ge detector and a spectrum is acquired in the energy range 0-614 keV. The

spectrum is then analysed using MGA, which calculates the abundances of  $^{238}$ Pu,  $^{239}$ Pu,  $^{240}$ Pu and  $^{241}$ Pu. The isotope  $^{242}$ Pu is estimated from isotopic correlation. The abundances of  $^{235}$ U and  $^{237}$ Np, if present in the Pu sample, as well as that of  $^{241}$ Am, are determined simultaneously. Typical precisions and accuracies range between 0.5 and 2%rel. for all isotope abundances except  $^{242}$ Pu.

## Uranium-235 in Solution by γ Ray Spectrometry

*Gamma ray spectrometry* with a NaI detector is used as a backup procedure for mass spectrometry in the determination of <sup>235</sup>U enrichment of U samples, which are dissolved and analysed for U concentration by potentiometric titration.

In this procedure, 5 mL aliquots of a U solution containing about 80 g/L U in 1M nitric acid are weighed into identically shaped glass tubes and the tubes closed with a stopper. A set of five standard solutions, containing known concentrations of NBS standards (U-010, U-015, U-020A, U-030A and U-050), is used for calibration. The number of counts at the 185.7 keV energy peak of <sup>235</sup>U is calculated and related, in weight per cent, to the total U content of the sample which has been assayed by titration. In the absence of radioisotope interferences, the results have a precision and accuracy ranging between 0.5%rel, for natural and 0.2%rel, for enriched U.

# Other DA Techniques

*Alpha spectrometry* is applied for the measurement of <sup>238</sup>Pu abundance with Si(Li) or ion implanted detectors. This method is used in parallel with isotope dilution mass spectrometry for the determination of <sup>238</sup>Pu abundance or for the measurement of Pu in spent fuel samples.

Laser fluorimetry is used for the determination of Np in solutions. This method is applied to the same types of materials which are analysed by the MacDonald and Savage titration.

*Plutonium(VI)* spectrophotometry is applicable to the determination of milligram amounts of Pu in small samples of products, with accuracies similar to those of titration.

## CONTAINMENT AND SURVEILLANCE

Containment and surveillance (C/S) techniques are extensively deployed by the IAEA owing to their application flexibility and cost effectiveness. The two main C/S categories are optical surveillance and sealing systems.

Optical surveillance is most effective in storage areas (such as spent fuel storage ponds) with relatively few activities that could be interpreted as the removal of nuclear material. A typical application would consist of two or more cameras positioned to completely cover the storage area. The field of view of the cameras is such that any movement of items that could be the removal of nuclear material is easily identified. This means that items have to be sufficiently large in the field of view to be identified and that one or more images have to be recorded during the movement. The image recording may be set at a periodic frequency (significantly shorter than the fastest removal time) or the motion (i.e. scene change) may trigger the recording. Optical surveillance is intrinsically an unattended operation that may be enhanced by the remote transmission of image data or system operation data (i.e. the status of the surveillance system). Unattended and remote monitoring systems are discussed in the next section.

Seals are typically applied to individual items containing nuclear material. A seal can help to indicate that material was neither introduced into nor removed from a container and, at the same time, provides a unique identity for the sealed container. Unattended IAEA monitoring equipment is also sealed. Most IAEA seals are applied for extended periods of time, typically several months. These seals may be either single-use seals that are replaced when checked or seals that are verifiable in situ, i.e. they can be checked for integrity and identity in the field. If the seals are in situ verifiable then the verification activity must be efficient (to limit radiation exposure to the inspector) and extremely reliable. The in situ verification activity must consist of checking the item containment as well as the seal and the method of its attachment to the item.

# Optical Surveillance Systems

IAEA optical surveillance equipment has undergone a transition from analog video systems to digital surveillance systems. Digital surveillance systems were essentially mandated by the strong commercial industrial trend in the manufacture of low cost digital components and systems with significant improvements in system reliability. Other inherent benefits for IAEA safeguards include the enhancement of data evaluation and automated review, the facilitation of remote surveillance and improved authentication and encryption capability. A list of optical surveillance systems employed by the IAEA is given in Table VI. Three of the camera systems (EMOS, GDTV and VDIS) are digital systems extensively field tested for routine use.

## Single-camera Systems

GDTV. The Gemini Digital Video System (Figure 18) consists of two independent single-camera units. The cameras are connected to the control unit via a data and power cable (transmitting a digital signal generated in the camera module). Data are recorded on removable hard disks located in the control unit along with the backup power supply (for over three days of operation) and other set-up and control functions. The storage capacity is sufficient for three months of operation. Extensive testing was performed in 1996–1997 to check the system for the types of environment encountered by the IAEA.

## Multi-camera Systems

**VDIS.** The VXI Digital Image Surveillance System (Figure 19) has been developed to replace the MXTV and MOSS. The VDIS is mainly based on the digital camera module DCM14 with authentication, encryption, image compression, connection to the VACOSS seal, connection for remote communication through wire or satellite, battery backup and local storage using the international PC card standard.

The modular structure of the VDIS facilitates the construction of four different surveillance systems:

- System 1: The DCM14 can be used as a standalone system, particularly in remote monitoring applications.
- System 2: One-channel VDIS, self-contained and built into the standard camera housing used for the PHSR (twin Minolta film cameras) and MIVS. This system can serve as a direct replacement for the PHSR. However, a mains supply is required with a battery backup for five days if the picture taking interval is 5 min.

 TABLE VI
 OPTICAL SURVEILLANCE SYSTEMS

Code	Equipment Name	Description and Applications
Photogr	aphic System	
PHSR	Photo Surveillance Unit	Twin and triple Minolta camera unit used for general surveillance. Being replaced by digital video systems (see EMOS, GDTV and VDIS)
Video: S	Single-camera Systems	
CSMS	Compact Surveillance and Monitoring System	COSMOS video surveillance system comprising battery operated camera, recorder and control electronics.  Designed to fit as a direct replacement into the camera housing of the earlier PHSR
GDTV	Gemini Digital Video System	Digital video system capable of replacing film cameras, CSMS and MIVS
MIVS	Modular Integrated Video System	Integrated video surveillance system consisting of camera, twin recorders and control electronics
SIDS	Sample Identification System	Consists of HLNC with special head and a video unit, which is triggered by neutrons above a certain level, allowing identification of the sample. Used for unattended verification of MOX in a fuel fabrication plant
STVS	Short Term TV System	Single camera and recorder from MXTV for temporary use
UWTV	Underwater TV	Commercial underwater TV system used for identification of fuel assemblies
Video: N	Aulti-camera Systems	
EMOS	Euratom Multi-camera Optical Surveillance System	Digital video system (up to four cameras) capable of replacing film cameras, CSMS and MIVS
FTPV	CCTV System	Facility specific closed circuit TV system. Used at fuel transfer ponds

**TABLE VI** (continued)

Code	Equipment Name	Description and Applications
MOSS	Multi-camera Optical Surveillance System	Multi-camera video surveillance system consisting of cameras, recorders and control electronics
MXTV	Multiplex TV Surveillance System	Multi-camera closed circuit TV system used for general surveillance. Consists of up to 16 cameras and a central recording station
VDIS	VXI Digital Image Surveillance System	Multi-camera digital image surveil- lance system capable of replacing MXTV and MOSS. System is based on digital camera module DCM14 with authentication, encryption, image compression, connection to VACOSS seal, connection for remote communi- cation through wire or satellite, battery backup and local storage. Single-camera versions of VDIS have been designed
VSPC	Video System	Facility specific closed circuit TV system consisting of four cameras on a split screen
Video R	eview Systems	
GARS	Gemini Advanced Review Station	Allows automated review of images from GDTV
MARS	MIVS Advanced Review Station	Video review station specific to MIVS only
MORE	Multi-system Optical Review Station	Generic video review station for all tape based systems (MOSS, MXTV, CSMS, MIVS)

- System 3: One-channel VDIS version with a DCM14 installed in the standard camera housing; a second DCM14 used for recording is in a separate compartment, reusing the MIVS console. This system can serve as a direct replacement for the MIVS.
- System 4: Multi-camera version (up to 32 cameras) built into a VXI environment and therefore capable of integration with other VXI based

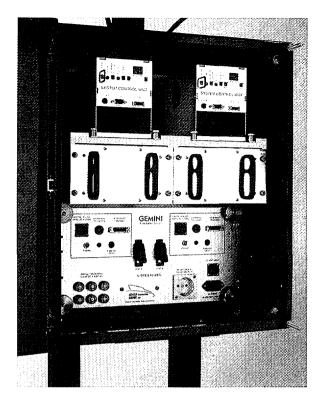
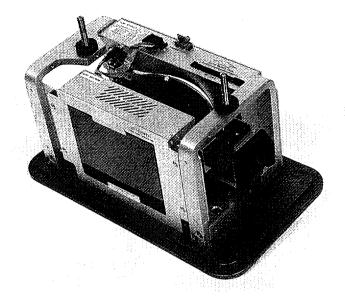


Figure 18 GDTV: Gemini Digital Video System.

equipment such as the CANDU Spent Fuel Bundle Counter and CANDU Core Discharge Monitor for both front end triggering and data review. The functionality and flexibility of the multi-camera VDIS far exceed those of MOSS, discussed below.

MOSS. The Multi-camera Optical Surveillance System uses authenticated, individually timed analog cameras for long periods of unattended surveillance. Since the system uses a modular design, the exact configuration (numbers and types of modules) varies according to the individual installation. Determining factors include the number of channels (cameras) used and the presence or absence of a redundant VCR recording capability. The system uses a video buffer memory to store scenes from various cameras, with each camera having its own specific timing sequence. When the buffer is full the stored scenes are recorded ('dumped') onto a single 3 h



**Figure 19** VDIS: VXI Digital Image Surveillance System (one-channel version built into standard camera housing).

Super VHS videotape. To meet stringent safeguards requirements MOSS uses a sophisticated video image authentication module, the Tamper Resistant TV Link (TRTL), to ensure that the signal sent from any remote camera over unsecured cable corresponds to the signal received by the receiver unit.

The image sequence recorded on tape depends on the individual timing programs of the cameras used. Therefore, to each frame collected, MOSS adds coded information to establish the identity of the camera used for that frame. Other information such as authentication of the image is also encoded in each frame. MOSS reviewing software uses the coded information in each frame to sort and assemble scenes from any one of the cameras and allows these to be viewed in a smooth, continuous presentation. The review software can also be used to create from any single camera a standalone VCR tape that can be viewed on a conventional VCR system. All the system status data stored in each frame are simultaneously collected in the MOSS network controller 'notebook'. This notebook is normally downloaded to a PC as part of the

system inspection. The notebook is printed out and used as a guide prior to and/or during a visual review of the tape.

MOSS is made up of two components, the recording station and the review station. A single review station can be used to service many recording units. Although a recording station can be reconfigured into a review mode, this is normally done only during system maintenance. MOSS tapes are typically reviewed off-site (i.e. away from the data collection station) on the MORE review station, discussed below.

#### Video Review Systems

MORE. The Multi-system Optical Review Station is a tool to aid in reviewing CSMS, MIVS, MXTV and MOSS tapes. Each MORE unit consists of an IBM compatible PC running the MORE software (with built-in DAT drive for archival of digitized images), a video monitor for the PC, a black and white video monitor with automatic CCIR/EIA-170 standard detection, three VCRs and a printer. A variety of tape formats can be reviewed. To perform an automated review it is first necessary to create set-up files. Regions of interest are defined in image areas that correspond to possible nuclear material removal pathways. The MORE system is the generic review station for all IAEA analog surveillance systems. An enhanced version is able to review VDIS digital surveillance data as well.

GARS. The Gemini Advanced Review Station performs a review of digital images created on the GDTV. GARS provides a flexible interface that is simple to use. An efficient motion detection algorithm and a cross-platform application are two of the many advantageous features of the software package. Review of surveillance data can be implemented either from the removable hard disk of the GDTV or from a CD-ROM archive disk.

# Sealing Systems

A sealing system comprises the containment enclosing the nuclear material to be safeguarded, the means of applying the seal (e.g. a metal wire) and the seal itself. All three components must be examined in order to verify that the sealing system has fulfilled its function of ensuring continuity of knowledge of the identity and integrity of the nuclear material concerned. A list of sealing systems in current use is given in Table VII.

# TABLE VII SEALING SYSTEMS

Code	Equipment Name	Description and Applications
Single-i	use Seals	
CAPS	Metallic Seal	Cap seal applied to a wide range of containments for continuity of knowledge of the contents. Verified after removal at IAEA Headquarters
VOID	Improved Adhesive Seal	Commercial multilayer sealing tape that cannot be removed without causing both the seal and the surface to be marked with the word "VOID"
In Situ	Verifiable Seals	
ACIV	Automatic COBRA Image Verifier	Automatic verifier for COBRA seals
FBOS	Fibre Optic General Purpose Seal (COBRA)	In situ verifiable fibre optic seal
ULCS	Ultrasonic Seal (ARC)	Seal used for underwater stack sealing of fuel bundles. It uses a random coil, which gives the seal a unique signature. An automated reader compares the signature with a stored value of the seal in situ
USSB	Ultrasonic Sealing Bolt	General purpose bolt seal primarily used underwater to seal the lids of spent fuel assembly containers
VCOS	VACOSS-S Electronic Seal (Variable Coding Seal System)	Reusable seal consisting of a fibre optic loop and electronic seal. Light pulses monitor the loop and every opening and closing of the seal is stored in the seal. A palmtop computer reads the seal
VMOS	VACOSS-S/MOSS System	Unattended system that records the closing (or opening) of VACOSS electronic seals by means of a specially adapted MOSS

Single-use Seals

CAPS. The Metallic Seal is extensively used for sealing material containers, material cabinets and IAEA safeguards equipment. Typically, 20 000 of these metal cap seals are verified each year. The seal is detached in the field and brought to IAEA Headquarters for identification. The primary advantages of CAPS are that it is simple, inexpensive and easily attached or detached by the inspector. Attachment and detachment efficiency is important to limit the radiation exposure of the inspector. Unique identification of each seal is obtained by imaging random scratches on the inside surface of the metal cap and by comparing the installation and removal images (Figure 20).

**VOID.** The Improved Adhesive Seal is made of special plastic material and contains on one side a special multilayer glue coating. When affixed to a surface and subsequently removed, a part (layer) of the glue remains on the surface to which it was bonded. The part remaining on the sealed surface as well as that on the seal itself exposes the word "VOID" printed many times, indicating that the seal has been removed. As for all adhesive seals, the VOID seal is intended only for temporary applications (24 h or less).

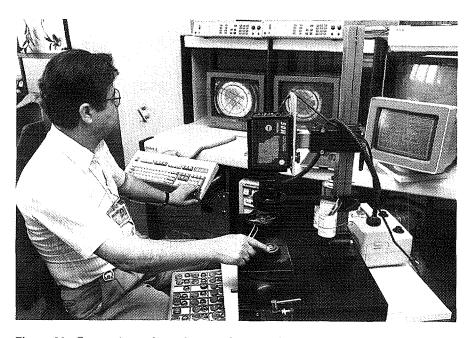


Figure 20 Comparison of metal cap seal images for seal validation.

In Situ Verifiable Seals

In situ verifiable seals are a kind of seal that is uniquely identifiable and verifiable in the field. They fall into the three main categories of fibre optic, ultrasonic and electronic seals.

FBOS. In the Fibre Optic General Purpose Seal the seal wire as used in CAPS is replaced by a multi-strand plastic fibre optic loop with its ends enclosed in a seal in such a way that a unique random pattern of fibres is formed. This can be verified by shining a light into the ends of the loop and observing the magnified pattern of the fibre ends either photographically or by means of a digital recording of the image pattern. The COBRA Seal System II (FBOS) employs this technology. Immediately after it is installed, the seal is inserted into a verification assembly that records a reference image of the seal signature pattern. The verifier consists of a verifier head, a still video camera and a liquid crystal display monitor. The verifier head holds the body of a COBRA seal while an image of the seal signature is recorded by the video camera. The image can then be printed and compared with the reference image of the same seal.

A much more logistically convenient COBRA seal verifier has recently been developed which stores digital images and is able to compare the patterns. This procedure enables the inspector to automatically verify the seal identity and integrity in situ and to conveniently store the pattern in a computer. Figure 21 illustrates the Automatic COBRA Image Verifier (ACIV) with a COBRA seal.

ULCS, USSB. The Ultrasonic Seal (ULCS) and Ultrasonic Sealing Bolt (USSB) are constructed to contain a unique random pattern of, for example, inclusions of metal pieces in a lighter substrate, or a randomly oriented coil of wire. They are applied in a variety of ways, with special designs for different applications. Verification is accomplished by transmitting ultrasonic pulses through the seal with a suitable transducer and observing the unique pattern of reflections. Verification consists of comparing the pattern obtained when installed with that obtained during subsequent in situ checks. These types of seals have proven particularly effective for underwater applications such as for stacks of CANDU fuel bundles (ULCS) or for bolts closing shipment and storage containers of LWR spent fuel assemblies (USSB).

**VCOS.** Electronic seals will be used with increasing frequency in IAEA applications as remote monitoring becomes more universally applied and as

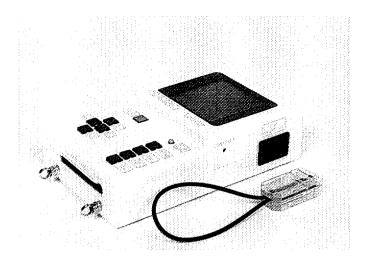
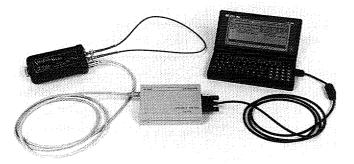


Figure 21 ACIV: Automatic COBRA Image Verifier with COBRA seal.

seal manufacture becomes less expensive. The first IAEA electronic seal, originally designed in the late 1970s, was the Variable Coding Seal System (VACOSS-S), shown in Figure 22. This seal uses electronic encoding methods in conjunction with fibre optic loops. The VACOSS-S Electronic Seal is intended for high reliability, long duration surveillance in applications that require periodic access. The time, date and duration of openings and closings of the loop are recorded internally for later retrieval. The fibre optic loop is interrogated with a light pulse every 250 ms for continuity of the light path. There is no known method of splicing the fibre in the interval between interrogations. The internal batteries have a two year operational lifetime. For installations with multiple seals in proximity, the seals may be connected in series. All seals connected in this fashion can be read in sequence without changing the connection. The electronics are potted in an X ray resistant compound of epoxy and ceramic particles to frustrate any possible attempt at reverse engineering. A tamper switch detects any opening of the seal housing. The seal housing is opened only to replace the internal batteries and openings are recorded as tamper events.

VMOS. Recently, the use of VACOSS-S seals has been integrated with existing video surveillance systems to allow a facility operator to attach or detach seals under video surveillance, thus eliminating the need for an inspector to be present during such an operation. In addition there is



**Figure 22** VACOSS-S Electronic Seal with fibre optic loop, interface box and palmtop computer.

redundant data collection in that the seal information is also stored on videotape for ease of review, and the built-in computer guides the operator to correctly install the seals. One such integrated system is the VACOSS-S/MOSS interface system. For this system a MOSS terminal has been modified to allow the user to communicate with the seal and MOSS. At each sealing activity the terminal verifies that the seal status is satisfactory, the seal box has not been opened (or tampered with), the seal battery status is satisfactory and the fibre optic cable is in its proper state. If any checks fail, the system indicates a problem. A typical VACOSS-S/MOSS system has seven camera/recorder channels for normal video surveillance and one camera/recorder dedicated to the monitoring of the seal handling operations.

## UNATTENDED AND REMOTE MONITORING

The use of unattended safeguards instrument systems has always been a requirement for IAEA safeguards. Optical surveillance systems, for example, are inherently unattended systems since their prime function is to survey an area for safeguards relevant activities over extended periods of time. Contemporary unattended monitoring systems employing radiation detection sensors are increasingly being used to detect the flow of nuclear material past key points in the facility process area. For complex nuclear facilities where the plant is automated (remotely operated), unattended assay and monitoring techniques are an integral part of a practicable safeguards implementation approach.

Unattended use necessitates that special considerations be included in the instrumental system design if the system is to be reliable and cost effective in providing credible, independent data. This means that the system must operate without failure over extended periods, including times when the facility power supply is interrupted. The unit should automatically record its status periodically. If data are to be sent over unsecured transmission pathways then the data must be authenticated. And if data are to be shipped off-site then they must be encrypted to meet the requirements of the facility and the State for confidentiality of information. Because of the stringent design considerations unattended and remote monitoring equipment typically has to be flexible, modular and highly reliable.

Unattended safeguards instruments are often deployed in facility areas with limited personnel access, such as areas with a high radiation level. Depending on the facility and the process being safeguarded, the optimum placement of appropriate instruments, even though custom designed, more than justifies the initial high cost when the long term overall economics are considered.

Remote monitoring in the safeguards context is generally considered to mean that data are shipped off-site to IAEA Headquarters or to an IAEA office. Cost effectiveness is a prime justification for adding this feature to unattended monitoring systems. If data can be sent to the inspector then the frequency of inspection visits to the facility can be reduced, thus saving both time and expense. In principle, a remote monitoring system with 'state of health' reporting can also function significantly more reliably than an unattended system that is serviced on a set frequency. Some events that would ultimately lead to a failure of the system can be remotely detected and reported in time for appropriate action to be taken. Limited IAEA

human resources, an ever growing stockpile of nuclear material and economics are likely to accelerate implementation of remote monitoring in the near future.

In summary, the primary advantages of unattended and remote verification techniques are:

- Reduced inspection efforts
- Reduced radiation exposure of inspectors
- Reduced level of intrusiveness in the operation of nuclear facilities.

Unattended and remote monitoring systems in use are listed in Table VIII.

TABLE VIII UNATTENDED AND REMOTE MONITORING SYSTEMS

Code	Equipment Name	Description and Applications
Unatter	ided Monitoring Systems	
ACSP	Advanced C/S System	Facility specific C/S system, custom designed for a MOX fuel fabrication plant
CONS	Input Flow Verification System	Radiation monitoring system that tracks the movement of irradiated fuel at a large scale reprocessing plant (CONSULHA)
ENGM	Entrance Gate Monitor	Radiation monitoring system that monitors non-irradiated fuel assemblies containing Pu that are brought into the reactor facility
FCPM	FCA Portal and Penetration Monitor	Monitoring system that monitors the entrance portal and containment periphery of a fast critical reactor (FCA, Japan)
REPM	Reactor Power Monitor	Neutron monitoring system placed outside the reactor biological shield to monitor the power level of the reactor

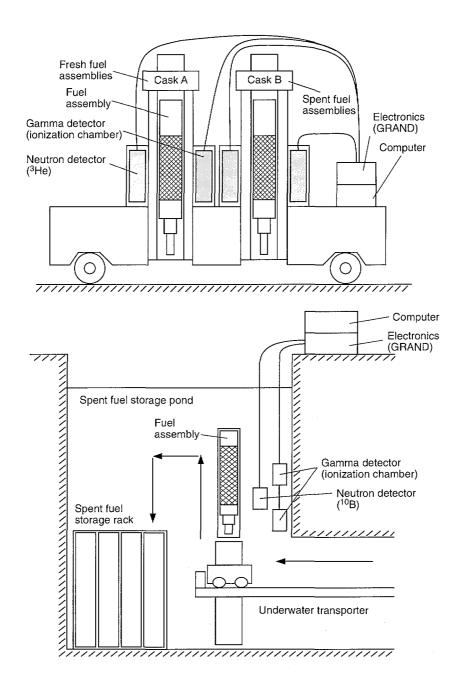
**TABLE VIII** (continued)

Code	Equipment Name	Description and Applications
UFFM	Unattended Fuel Flow Monitor	Generic radiation monitoring system that monitors the flow of fresh and irradiated fuel in a reactor facility. Placement and details of radiation detectors are facility dependent
VIFB	CANDU Spent Fuel Bundle Counter	Radiation monitoring system that counts irradiated fuel bundles as they are discharged to the spent fuel storage pond of an on-load refuelled power reactor
VIFC	CANDU Core Discharge Monitor	Radiation monitoring system that monitors the discharge of spent fuel bundles from an on-load refuelled power reactor core (reactor may be on-power or shut down)
Remote	Monitoring System	
SRMS	Safeguards Remote Monitoring System	Sends surveillance and electronic seal data via a satellite or ISDN connection to IAEA Headquarters or an IAEA office for review and archiving

# Unattended Radiation Monitoring

**UFFM.** The Unattended Fuel Flow Monitor consists of separate neutron or  $\gamma$  ray detector assemblies permanently installed in a reactor facility. The detectors monitor the movement of fresh fuel assemblies to the reactor (at breeder reactors only), of spent fuel assemblies from the reactor to the fuel storage pond and of spent fuel assemblies out of the storage pond. Neutron detection in the UFFM is contingent on shielding the neutron detectors from the intense  $\gamma$  radiation from the spent fuel. The sealed detector systems may be attached to massive transport vehicles or to the storage pond wall near the underwater entrance (Figure 23).

A typical transfer sequence involving several UFFM units could include a fresh fuel assembly brought to the reactor core and a spent fuel assembly



**Figure 23** UFFM: Unattended Fuel Flow Monitor. Top: detector assembly mounted on facility transport shielding casks. Bottom: stationary detector assembly mounted at spent fuel storage pond.

retrieved and brought to the storage pond. The combination of neutron and  $\gamma$  ray signatures at the successive units characterizes the transferred material as fresh fuel, spent fuel or another material (e.g. neutron irradiated blanket material at a breeder reactor facility). Although individual systems are facility specific, the neutron detectors on the transport vehicles tend to be  $^3{\rm He}$  proportional counters and the  $\gamma$  ray detectors are NaI scintillators or ionization chambers. Underwater in the storage pond the neutron detector is usually a  $^{10}{\rm B}$  lined gas filled proportional counter and the  $\gamma$  ray detector is a gas filled ionization chamber. UFFMs are designed to monitor neutron and  $\gamma$  ray counts continuously but store only data that are significantly above background levels. This ensures that the memory in the portable computer is adequate for at least one month. Surveillance cameras normally complement a UFFM over the fuel transfer route.



Figure 24 VIFB: CANDU Spent Fuel Bundle Counter electronics.

**ENGM.** The Entrance Gate Monitor is included at Pu fuelled reactor facilities which incorporate the UFFM. This is a permanently installed passive neutron coincidence collar detector (PNCL). Fresh fuel assemblies entering the reactor facility must pass through the ENGM so that their Pu content can be verified. Therefore the ENGM is the system which verifies the amount of fresh fissile fuel in an assembly and serves as the first detector in a sequence of detector systems which follow the movement of fuel assemblies within the reactor facility.

**VIFB.** The CANDU Spent Fuel Bundle Counter (Figure 24) is an unattended monitoring system that monitors a strategic location in the spent fuel bundle pathway of an on-load refuelled power reactor. Collimated  $\gamma$  ray detectors detect the fuel bundle as it passes. The proper placement of detectors and the use of the appropriate algorithm for the facility enable the device to count the

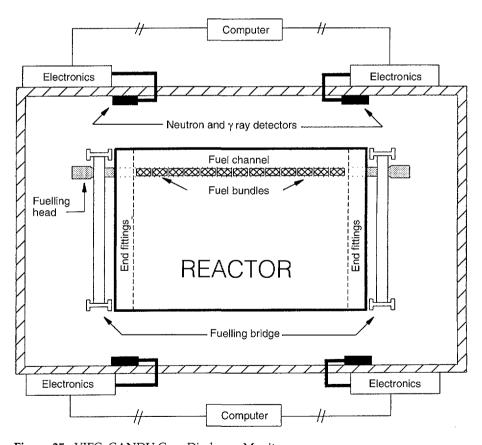


Figure 25 VIFC: CANDU Core Discharge Monitor.

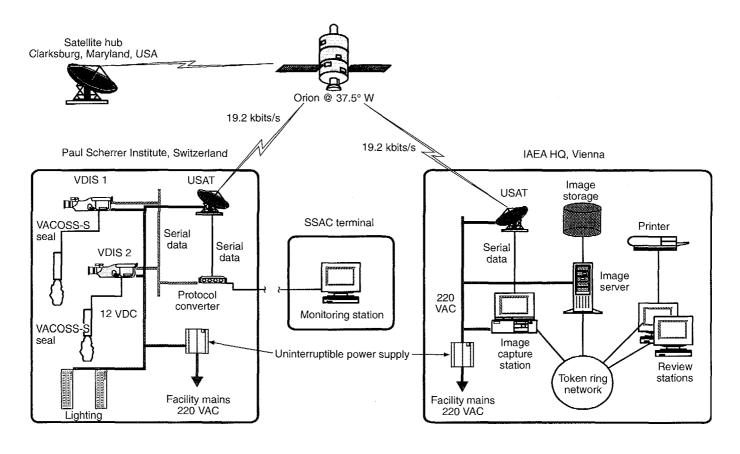


Figure 26 SRMS: Safeguards Remote Monitoring System.

bundles as they pass and record the direction in which they are moving, even when two bundles are moving together. High operational reliability, large dynamic detection sensitivity (to include all operational possibilities) and insensitivity to power outages are some of the important features of the bundle counter.

VIFC. The CANDU Core Discharge Monitor is a typical unattended monitoring system operating in an inaccessible area. The VIFC detects irradiated fuel upon discharge from the core face of a CANDU reactor (Figure 25).

Both neutron (normal on-power discharge signal) and  $\gamma$  ray intensities are continuously monitored. The inspector, upon reviewing the data, is able to identify in a straightforward, unambiguous manner the abrupt but characteristic change in count rate associated with fuel bundle discharge. The review technique is valid for irradiated fuel discharge both when the reactor is on-power and when it is shut down. Because of the linear increase in background signal, the system can also track the operating power level of the reactor.

The VIFC was designed to be fail-safe. Sufficient redundancy was built in to accommodate individual component failures without compromising operation (failure of the VIFC would be exceedingly difficult to recover from in safeguards terms). The detection modules are designed to last the lifetime of the reactor, since their location inside the containment area limits possibilities for maintenance and repair. Automatic performance monitoring and failure announcement have been incorporated in the VIFC.

# Remote Monitoring

SRMS. Field testing of a new Safeguards Remote Monitoring System (Figure 26) was completed in 1996 and the system has since been transmitting regular images and data directly back to IAEA Headquarters. Remote monitoring not only improves inspector efficiency and safety but also greatly improves the availability to the inspector of C/S data. At the facility there are two self-contained DCM14 units with digital video cameras (the same electronics and camera modules as in the VDIS), providing authenticated and encrypted surveillance images and data, and two verifiable VACOSS-S electronic seals. Sophisticated power monitoring and control, backup battery power and scene change detection are all integral features which contribute

to the overall system reliability. In the event of a loss of facility mains power each camera can independently operate on battery backup for up to five days. The images and data are transmitted to data storage computers at IAEA Headquarters via communication satellite and ultra-small aperture terminal (USAT) satellite transceivers. Subsequently, the images and data are periodically transferred to a local area network for review, upon demand, by authorized inspectors.

The improved safeguards efficiency and system reliability provided by remote monitoring, together with its potential for reduced costs, are expected to lead to an increase in its deployment. The full cost effectiveness and system advantages will become even more evident with the inclusion of additional review stations in an international network of safeguarded facilities covered by IAEA remote monitoring.

## ENVIRONMENTAL SAMPLING

Environmental sampling was introduced in 1996 as one of the new IAEA safe-guards measures which contribute to the confirmation of the absence of undeclared nuclear material or nuclear activities. Collection of environmental samples at or near a nuclear site combined with ultrasensitive analytical techniques, such as mass spectrometry methods, particle analysis and low level radiometric techniques, can reveal signatures of past and current activities in locations where nuclear material is handled. Initial implementation of environmental sampling for safeguards is focused on the collection of swipe samples inside enrichment plants and installations with hot cells. It is anticipated that implementation will be extended to other types of nuclear facilities and may include the collection of other types of environmental samples (e.g. vegetation, soil and water) exterior to facilities and sites.

Samples are analysed in either bulk or particle mode, depending on the sampling objectives and the activity levels of the swipes. Bulk analysis involves the analysis of an entire sample, usually by  $\gamma$  ray spectrometry or isotope dilution thermal ionization mass spectrometry; the analytical measurements represent average results for the material contained. Particle analysis relies on the detection and analysis of individual particles in the micrometre size range and provides as results isotope ratios of U and/or Pu in these particles.

# IAEA Clean Laboratory for Safeguards

The IAEA Clean Laboratory for Safeguards (Figure 27) was inaugurated in December 1995 with the goal of providing a Class 100 clean-room capability for the provision and certification of sampling kits and for the receipt, screening and distribution of environmental samples from safeguards inspections. This facility significantly reduces the risk of cross-contamination that might lead to incorrect safeguards conclusions. The Clean Laboratory consists of over 200 m² of laboratory space, with approximately 100 m² at Class 100 cleanliness level (Figure 28). The laboratory is equipped with a suite of analytical techniques, including  $\alpha$ ,  $\beta$ ,  $\gamma$  and X ray fluorescence spectrometry, scanning electron microscopy with electron probe analysis and high sensitivity thermal ionization mass spectrometry.

Environmental swipe samples received at the Clean Laboratory are given a code number to maintain confidentiality about their origin. The samples are

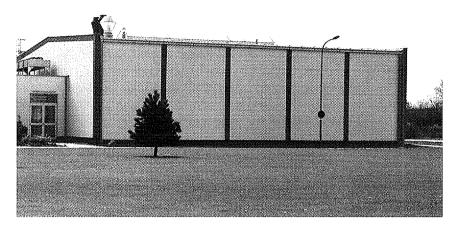


Figure 27 The IAEA Clean Laboratory for Safeguards.

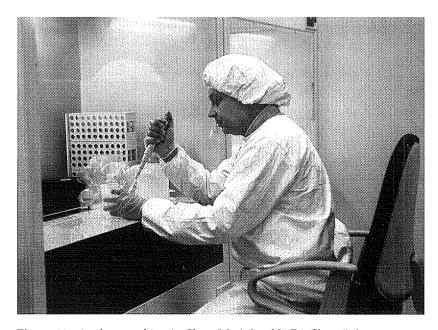


Figure 28 Analyst working in Clean Module of IAEA Clean Laboratory.

then measured by low background  $\gamma$  ray spectrometry to detect the presence of actinide elements (primarily U and Pu) and fission or activation products (such as  $^{60}$ Co,  $^{137}$ Cs and  $^{106}$ Ru); the samples are then measured by radioisotope excited X ray fluorescence spectrometry to detect the presence of U, Pu or other important elements. Alpha/beta counting is then applied to the samples to detect actinides or  $\beta$  emitting isotopes such as  $^{3}$ H,  $^{90}$ Sr or  $^{99}$ Tcm. Scanning electron microscopy is used to examine small particles removed from environmental samples. The size and morphology of these particles can be examined at high magnification and their elemental composition measured by X ray fluorescence spectrometry using an electron probe attachment.

Following the screening measurements, subsamples are distributed to laboratories of the NWAL for more detailed analyses. Selected samples are chosen for measurement in the Clean Laboratory by isotope dilution thermal ionization mass spectrometry, using a highly sensitive instrument equipped with pulse counting detection. The ultimate sensitivity of this method is in the  $10^{-15}$  g range for U and Pu.

One of the main activities of the Clean Laboratory is the preparation of clean sampling kits for collecting environmental samples. A kit for the collection of swipe samples is shown in Figure 29. This consists of all the supplies needed by an IAEA inspector in the field: clean swipe cloths, plastic minigrip bags, clean-room gloves, a sample data form, a pen and labels. A roll of aluminium foil is provided to establish a clean working surface. A different type of swipe sampling kit is required for sampling inside hot cells, where the subsamples must be taken with remote manipulators and shipped back to the IAEA in a special lead-lined container because of their higher radiation level.

# Screening of Samples

*Low Level γ Ray Spectrometry* 

Immediately after receipt, environmental samples are measured with a low background  $\gamma$  ray spectrometer system. The spectrometer is based on a 90% efficient coaxial Ge detector enclosed in a high purity lead shield of 10 cm thickness. The samples, in special beakers, are placed in a 15 position sample changer and counted for 1 h each to provide a  $\gamma$  ray spectrum in the energy range from 5 keV to 3 MeV. The total  $\gamma$  activity, corrected for background, is

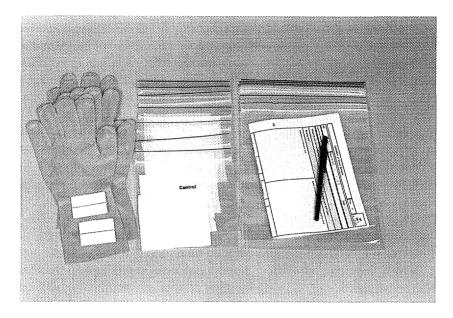


Figure 29 Cotton swipe kit for environmental sampling.

obtained by this method and if sufficient activity is detected, an evaluation of the spectral peaks can be performed to estimate the activity in the sample of individual  $\gamma$  emitting isotopes such as  $^{60}\text{Co}$ ,  $^{95}\text{Zr}$ ,  $^{106}\text{Ru}$ ,  $^{134}\text{Cs}$ ,  $^{137}\text{Cs}$  and  $^{241}\text{Am}$ . Depending on the number of counts collected, the precision and accuracy of these measurements are in the range 2–5%rel. The absolute activity of individual radioisotopes is not as important as the relative activity compared with a selected isotope such as  $^{137}\text{Cs}$ .

## *X Ray Fluorescence Spectrometry*

X ray fluorescence spectrometry is used to detect microgram amounts of U, Pu or other elements of interest on the surface of swipe samples. The sample is placed on the spectrometer and irradiated by X rays from a 7.4 MBq (20 mCi) <sup>109</sup>Cd source, which cause the emission of fluorescent X rays from elements present on the swipe. The fluorescent X rays are detected using a 100 mm<sup>2</sup> Si(Li) detector placed under the radioisotope source. Counting is performed for 10 min and the spectrum is then evaluated to determine the amount of the element present. The detection limit for U is approximately

 $1 \mu g/cm^2$ , with an accuracy of about 10–20% rel. This screening method is completely non-invasive because the subsample can be measured inside its double plastic bagging.

## Alpha/Beta Proportional Counting

A gas flow proportional counting system is used to screen environmental swipe samples for the presence of  $\alpha$  or  $\beta$  emitting isotopes. Each sample is removed from its plastic bags and placed in a cassette for introduction into the counting chamber using a 50 position sample changer system. The chamber is filled with counting gas and data are collected for 1 h. The final result is the total number of  $\alpha$  and  $\beta$  counts, corrected for background. This system has high collection efficiency and has a sensitivity in the millibecquerel range. Alpha emitting nuclides such as  $^{210}$ Po and  $\beta$  emitters such as  $^{3}$ H,  $^{90}$ Sr and  $^{99}$ Tc<sup>m</sup> can be measured in this way much more sensitively than by  $\gamma$  or X ray methods.

## Isotopic and Elemental Analysis

Pulse Counting Thermal Ionization Mass Spectrometry

Screening measurements are used to decide which samples should be sent for more detailed analysis. Thermal ionization mass spectrometry is used to measure U and Pu concentrations and isotopic compositions in environmental samples. The basic technique was described in the section on destructive analysis. However, for measurements of environmental samples a much higher sensitivity is needed, extending into the  $10^{-9}$  and  $10^{-12}$  g ranges. This is achieved by the use of special sample treatment procedures, electrodeposition of the sample elements on the mass spectrometer filament and use of a pulse counting detection system with high detection efficiency. The mass spectrometer is shown in Figure 30.

Isotopic spikes (<sup>233</sup>U, <sup>242</sup>Pu or <sup>244</sup>Pu) are added to the samples during chemical processing to ensure the recovery efficiency of U or Pu. Isotopic spikes are also added to allow U or Pu concentrations to be determined using thermal ionization mass spectrometry. Isotope ratios are measured for all isotopes of U or Pu relative to the spike isotope and the isotopic composition of the sample is estimated by subtraction of the known isotopic composition

# Safeguards Techniques and Equipment

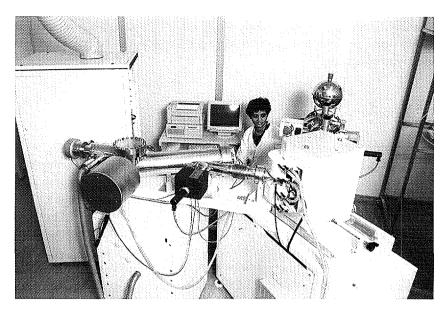


Figure 30 Mass spectrometer for isotopic analysis.

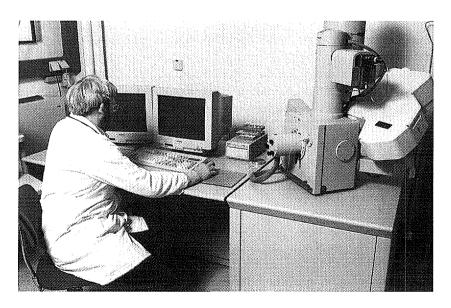


Figure 31 Scanning electron microscope for particle analysis.

of the spike. The accuracy and precision of this technique are about 1-10% for a U or Pu concentration in the  $10^{-9}$  g range and for the ratios of the major isotopes in the sample.

## Scanning Electron Microscopy with Electron Probe Analysis

The Clean Laboratory is equipped with a scanning electron microscope with wavelength and energy dispersive X ray fluorescence detectors (Figure 31). Particles of interest are removed from the sample by ashing or ultrasoneration and deposited on a conducting substrate for introduction into the electron microscope. Under high magnification (1000–5000×) the particles are examined and the backscattered electron signal is used to search for particles containing heavy elements. Heavy particles can then be measured by energy dispersive X ray fluorescence spectrometry to give a semiquantitative elemental analysis. Particles containing U or Pu can be identified in this way; their size and morphology, as well as other elements present, will give information about the process that created them. This type of analysis forms a part of the classical 'particle analysis' approach which is applied in certain NWAL laboratories in conjunction with thermal ionization mass spectrometry.

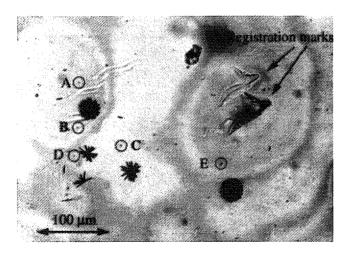
#### Fission Track Method

Traditional particle analysis involves first a step in which particles of interest, containing fissile isotopes such as <sup>235</sup>U or <sup>239</sup>Pu, are selected by the fission track method. These particles are then placed on a substrate for scanning electron microscopy and electron probe examination to measure the elemental composition. The particles are then mounted on the filament of a thermal ionization mass spectrometer for measurement of the isotopic composition of the U and Pu present.

The fission track method involves removal of particles from the environmental sample by ashing (for vegetation or swipe samples) or physical removal by ultrasoneration in an inert solvent. The particles are then spread onto a plastic track etch film (e.g. Lexan) in a layer of collodion (nitrocellulose). The film is then irradiated in a reactor with thermal neutrons with a total dose of 10<sup>14</sup> neutrons. Particles containing fissile isotopes leave damage tracks in the film, which can be etched to make them visible under a light microscope (Figure 32). An experienced analyst can compare the size and appearance of the particles with the number of fission tracks to decide

which particles should be measured further. The analyst can then pick up each particle of interest and mount it either onto a substrate for scanning electron microscopy and electron probe measurements or directly onto a filament for thermal ionization mass spectrometry.

In the case of thermal ionization mass spectrometry, the particle is held in a rhenium metal filament and heated in the ion source of the mass spectrometer at 1500–1800°C to produce ions of U or Pu, which are counted by a pulse counting detection system. The mass spectrometer steps between the isotopes of U or Pu to accumulate a mass spectrum. The abundance of the various isotopes can be estimated from the collected ion counts with a precision and accuracy of better than 1%rel. for isotopes of 1–90% abundance in particles with a diameter of 1–5  $\mu$ m. Particles with diameters down to 0.1  $\mu$ m can be measured, but with less precision and accuracy.



Particle	Size (µm)	Tracks	% <sup>235</sup> U	Compound
A	1.2	100	3.0	UO <sub>2</sub>
В	1.0	19	0.5	UO,
C	1.5	40	0.5	$UO_2$
D	0.7	8	0.5	UO,
E	1.5	600	91.8 % <sup>239</sup> Pu	$PuO_2$

Figure 32 Lexan film showing fission tracks.

## Secondary Ion Mass Spectrometry

Another technique for measuring the isotopic composition of micrometre size environmental particles is secondary ion mass spectrometry. The particles are mounted on a conducting substrate and placed in the vacuum system of the instrument, where they are bombarded with energetic ions of oxygen, gallium or caesium. The ion bombardment results in sputtering of the sample and the ejection of secondary ions which are representative of the particle under examination. The secondary ions are accelerated and mass analysed by the spectrometer and counted with either an imaging detector or a pulse counting ion multiplier. In the ion microscope mode of operation, an image is generated using secondary ions of a given mass (e.g. <sup>235</sup>U<sup>+</sup>). Another image can then be taken using a different secondary ion signal (such as <sup>238</sup>U+) and the two images merged to obtain the  $^{235}U/^{238}U$  ratio for each particle in the field of view (typically 150 µm in diameter). By scanning 100–200 fields in one session, it is possible to interrogate several thousand particles by this method, thus giving a distribution of the <sup>235</sup>U enrichments found in the particles from a sample.

Once an interesting particle has been identified in the ion microscope mode, it can be measured to completion by focusing the primary ion beam on it and stepping between the isotopes of interest. This will result in a complete isotopic composition of the particle, including the minor isotopes such as <sup>234</sup>U and <sup>236</sup>U. Depending on the size of the particle, the precision and accuracy of this approach can be 1%rel. for isotopes at the 1–90% abundance level and up to 10%rel. for minor isotopes.

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