European Commission

Destructive Analyses for the Quality Checking of Radioactive Waste Packages

Review of methods within the Working Group on "Destructive Analyses" of the European Network of Testing Facilities for the Quality Checking of Radioactive Waste Packages.

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European Network of Testing Facilities for the Quality Checking of Radioactive Waste Packages

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EUROPEAN NETWORK OF TESTING FACILITIES
FOR THE QUALITY CHECKING OF RADIOACTIVE
WASTE PACKAGES

Working Group B

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FOREWORD

In October 1992, on the initiative of the European Commission, nine laboratories of the European Union Member States that are carrying out quality assurance programmes, founded an association to promote and facilitate collaboration in the development, application and standardisation of quality checking for waste packages. This organisation was named "European Network of Testing Facilities for the Quality Checking of Radioactive Waste Packages", or briefly " The Network".
Through the Network, destructive quality control procedures are facilitated, improved and harmonised.

Working Group B (WGB), reformed in April 1998 from previous working groups, comprises laboratories from Belgium, France, Germany, Italy, Spain, The Netherlands and United Kingdom.
The activity of the WGB is focused on the assessment and discussion on chemical, radiochemical and physical destructive analytical methods and techniques used in the treatment or conditioning processes, in the Quality Control, in the verification and characterisation of the waste forms and packages.

ABSTRACT

Conditioned radioactive waste has to meet the specifications and acceptance criteria defined by Regulatory and Management Authorities. An important parameter for a complete characterisation of the radioactive waste is the radionuclide inventory, which cannot always be determined by non-destructive methods, but often needs the application of destructive procedures. The radionuclides detectable only by destructive methods, include a group of “critical” nuclides, as they represent a long-term risk due to their long half-lives. They are pure α- and β emitting nuclides a second as some γ-emitters of low energy.
Every member state of the European Community routinely uses its own destructive methods based on various sample preparation processes and different counting techniques. Although a European Network of Testing Facilities for the Quality Checking of Radioactive Waste Packages was instituted, common destructive control procedures do not yet exist. The main purpose of Working Group B of the European Network is to select, compare, validate, and finally harmonise the destructive analytical methods applied to various types of waste streams in the different participating laboratories.
Taking into consideration the most important problems in destructive quality control related to sampling procedures, dissolution, separation, counting and measurement, an inventory of the traditional methods as well as of the new fast analytical techniques has been reported.
This review has highlighted a need for further research and co-operation in development of existing methods.

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1.0 Introduction

In the field of radioactive waste management, the subject of Quality Assurance (QA) is of great importance [1-2].
The handling and treatment of radioactive waste, and its subsequent conditioning, transport, interim storage and final disposal should be managed in such a way as to protect man and the environment, and to minimise hazards on future generations.
In general terms, the Quality Assurance can be defined as a series of planned and systematic actions aimed to assure adequate confidence in the processes and the products involved in any industrial system.

Quality Checking is considered as a part of the planned and systematic actions of Quality Assurance. When Quality Assurance was introduced in the field of radioactive waste management, quality controls were carried out only on the first steps of the management scheme, i.e. waste treatment and conditioning.
But it was recognised that the overall aim would be achieved if Quality Assurance took place in every phase of radioactive waste management: from the point at which the waste arises through to the point of ultimate disposal.

Each organisation which takes possession of the waste is responsible for verifying the accuracy of characterisation data and for performing additional characterisation for any aspect of the waste which may be altered by treatment, immobilisation, packaging or storage (changes to the isotopic composition owing to radioactive decay will also need to be taken into account). Ultimately, the organisation consigning the waste to disposal must provide for the final characterisation to the operator of the repository facility.

Process knowledge may be used in conjunction with sampling and analysis to determine if the waste contains radioactive, toxic or hazardous constituents, or exhibits hazardous characteristics, such as reactivity or corrosivity.
Once such a determination has been made, it is the responsibility of the waste management facility to:

- design and implement a treatment process that reduces the hazardous characteristics to an acceptable level, or destroy or immobilise the hazardous constituents;
- demonstrate that the proposed disposal methods will not result in an unacceptable release of hazardous waste to the accessible environment.

In order to accomplish these goals, it is necessary to characterise the wastes to demonstrate that either the selected processing has eliminated the hazardous characteristics or destroyed or immobilised the hazardous constituents (as applicable), or provided for sufficient analytical data on the waste form, that its behaviour in a possible disposal scenario can be predicted with a degree of confidence acceptable to those responsible for approval of the proposed disposal method and site.

For wastes that are retrieved from storage, such authoritative process knowledge may not be available.

In these instances, waste management organisations may perform initial investigative activities, e.g. interviewing waste generators, in order to reconstruct process knowledge for such wastes. This investigative data may be valuable for the ultimate waste characterisation, but the regulatory authorities do not always consider it valid for process knowledge, unless they are substantiated by actual sampling and analysis.

An example of general outline of this characterisation strategy is shown in Fig. 1.

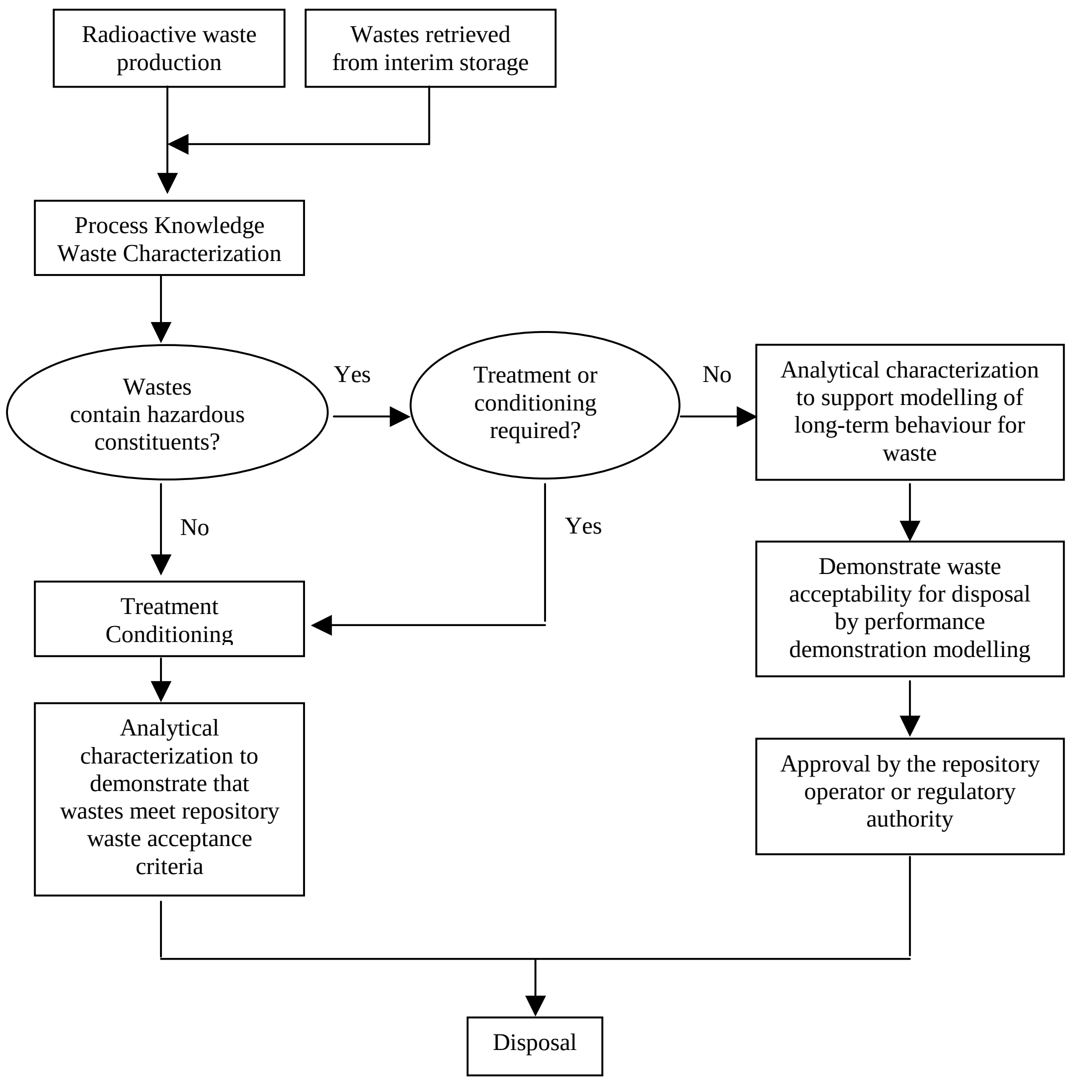

Figure 1: Example of general characterisation strategy.

The guidance on the Quality Assurance for radioactive waste packages is given not only to the operators of waste conditioning facilities and repositories, as well as to the consignees and consignor for the transport of radioactive waste, but also to the National or Regional Authorities in charge of the licensing and/or surveillance of conditioning processes or repositories.

The Quality Assurance programme should be developed in accordance with national and/or international legislation, and include the following elements: organisation and responsibilities, procedures and instructions, document control, control of purchased materials, process control, non-conformance control and corrective actions, records, audits and training of personnel.

The management of Quality Assurance has to be independent from the management of plant operation and all the activities are under the surveillance of the assigned national authorities or institutions.

On the initiative of the European Commission (EC) an association of several laboratories has been founded in 1992, in order to enhance the control of radioactive waste packages [3].

The association, officially called “European Network of Testing Facilities for the Quality Checking of Radioactive Waste Packages” (“Network”), is a non-profit organisation and its management and operation are conducted by a Steering Committee, who takes care of the organisation, objectives, decision of working programmes, policy issues and membership, and at present consists of three main Working Groups:

- WGA on non-destructive analysis;
- WGB on destructive analysis;
- WGC on Quality Assurance.

The WGB provides a *forum* for assessment and discussion on chemical, radiochemical and physical destructive measurements and techniques in the field of radioactive waste controls useful for:

- Treatment/conditioning processes
- Quality Control
- Measurement of volatile release from waste packages
- Verification and characterisation of the Waste Forms and Waste Packages

The Working Group will:

- Build-up an up-to-date a list of chemical and radiochemical methods and physical techniques in the frame of Network activities.
- Build-up an up-to-date a list of analytical procedures and techniques for measurement of gas releases in waste packages by process of internal irradiation, corrosion, biodegradation or others.
- Stimulate the investigation about modelling of gas release from waste packages.
- Stimulate the R&D programmes of the participating laboratories according to the requirements of the Network (e.g.: fast techniques, fingerprint analysis, etc.)
- Determine the reliability of measurements through the implementation and eventually the management of interlaboratory measurements programmes, promoting the systematic and correct use of *ad hoc* Reference Materials and the harmonisation of methodologies.
- Consider sampling problems, representativity and stability of the samples.
- Stimulate the co-operation and the exchange of information between laboratories and Working Groups.
- Provide assistance for the harmonisation of method used by WGB laboratories.

Information about the composition of the working group is given in appendix III.

For the characterisation of waste forms and waste packages, reliable and effective methods for measuring radiological, physical, chemical and biological properties have been developed and widely used.

Moreover, R&D work was started to develop new test methods and improve the available methods.

This report describes specific radiochemical destructive test methods applied in the phase of the characterisation of primary wastes, waste forms and packages.
These methods are effective and reliable to the satisfaction of waste management facilities, nuclear institutions and regulatory authorities in several States of European Community, which are represented in the Working Group B of the “Network”.

Technical terms, acronyms and abbreviations are explained in the glossary section, appendix II.

2.0 Background

Laboratories, which are members of the Working Group B of the "Network", originate from very different background. They perform various tests and analyses according to their own needs and those National Authorities.

In ***Belgium*** several nuclear activities are carried out by the Nuclear Research Centre SCK•CEN of Mol, and are subdivided in research areas.
Analysis of activities show that about half of them are allocated to statutory research areas of waste processing and decommissioning, waste characterisation and disposal, radioprotection and safety, reactor materials and fuel studies. Whereas the rest are devoted to the operation of the BR2 materials testing reactor (including the engineering studies of irradiation devices for isotope production, prototype fuel and materials irradiation's and semiconductor doping by nuclear transmutations) and organisational and management tasks (including public information, quality management, scientific support to the government and strategic studies, relevant for the use of nuclear energy in Belgium and the role of SCK•CEN as a research centre).

About the activities related to destructive radioactive waste analysis, SCK•CEN has developed analytical methods for the determination of critical nuclides in waste streams and also gives an analytical support to R&D programmes on waste management, particularly:

- The characterisation of solid and liquid wastes from nuclear power plants (effluents, water filters, spent resins, evaporator concentrates), with the aim of the determination of possible correlation between long-lived radionuclides (critical nuclides) and nuclides directly measurable by gamma measurements (^{60}Co, ^{137}Cs), is carried out in collaboration with regulatory authorities for waste management (ONDRAF/NIRAS). [4-5]
- In the frame of the own R&D program concerning the geological disposal of radioactive wastes in clay, development of separation methods for the determination of long-lived nuclides in conditioned wastes, studies of the long-term behaviour of conditioned waste (leaching tests) and studies of the diffusion of radionuclides in clay are carried out. [6]
- In the frame of R&D sponsored by the EC, it co-ordinates a research programme concerning the development of an approved method for the determination of actinides in conditioned and unconditioned wastes. [7]

In ***France***, the Commissariat à l'Energie Atomique (CEA) is pursuing research and development on radioactive waste in the frame of the 30 December 1991 law. Most of the studies concerning low and intermediate level waste are conducted within the Department of Nuclear Waste Storage and Disposal (DESD).

The major activities of the DESD consist of studying waste treatment and conditioning processes, methods for the characterisation and control of waste packages as well as their long-term behaviour in near-to-surface storage and deep disposal situations. This research work is performed in collaboration with French Regulatory Authorities (ANDRA) and nuclear waste producers (EDF, COGEMA). Additionally, collaboration with universities and European organisations is contributing to this work.

The development of destructive analytical methods for waste characterisation is carried out in the Laboratory of Radiochemical and Chemical Analyses (LARC), with the following objectives:

- the characterisation of liquid and solid waste from nuclear power and reprocessing plants, especially the measurement of critical radionuclides and complexing organic species;
- the development of very sensitive methods for the determination of long-lived nuclides in waste packages addressed to surface disposal, in order to define desirable detection levels in relation to dose impact to the environment;
- the determination of correlation between critical nuclides (long-lived β emitters) and directly measurable β–γ emitters;
- the study of the long-term behaviour of different kinds of waste matrices (leaching tests).

In ***Germany,*** several institutions are involved in the quality checking and management of radioactive waste packages.

The Institut für Radiochemie as an institute for higher education and research and development belongs to the Technischen Universität München (TUM/RCM). It had been founded in 1963, near the research reactor (FRM) in Garching, near Munich.
The institute is equipped with radiochemical laboratories (type A, B, C) and glove boxes for alpha, beta, gamma nuclide preparation, hot cells for remote handling of high radioactive samples up to the 1E10-fold of the handling of gamma emitters, a ^{60}Co source for gamma radiation experiments with an activity of 15 TBq is installed. Until now a diversified area of radio spectrometric and conventional analytical instrumentation has been installed.
Depending on the activity of the samples, the investigations are performed in hot cells, glove boxes or in low level counting rooms.

The TUM/RCM has got a lot of experience from conducting research and development projects in the fields of nuclear reprocessing or in the behaviour of actinide elements in the environment.
Since 1988 investigations concerning destructive and non-destructive methods for the characterisation and the quality control of radioactive waste forms became more and more important. In this field besides non-destructive investigations many invasive inspection techniques have been developed and improved. New preparation methods for the separation and identification of many different nuclides have been specified.

Beyond R&D activities, the institute is tied up in the praxis of official quality control of radioactive waste in Bavaria. A cooperation exists with the Bavarian technical inspection company (TÜV Sueddeutschland) acting as expert and consultant for the local authorities in the field of radioactive waste management. Based on this cooperation a lot of contracts have been performed for nuclear plants in Germany, Switzerland or Russia. Furthermore the institute works in contract with the National companionship for storage of radioactive wastes in Switzerland (Nationale Genossenschaft fuer die Lagerung Radioaktiver Abfaelle, NAGRA) as expert laboratory. Within these cooperations the institute provides its experience with all practical aspects of waste characterisation and places its personnel, laboratories and instrumental equipment at disposal.

The experience gained at TUM/RCM is used in close co-operations with other research institutes like FZ Karlsruhe and FZ Jülich or with commercial companies like the TÜV Süddeutschland.
The "Quality Control Group for Radioactive Waste" (PKS - Produktkontrollstelle) was installed in 1985 by the Federal Office for Radiation Protection (BfS - Bundesamt für Strahlenschutz) at the Research Center Jülich (FZJ), Germany. PKS is associated to the "Institute for Safety Research and Reactor Technology" (ISR) of the FZ Jülich as independent organisation.

It works on behalf of the BfS in the following sectors:

- documentation checking;
- characterisation of waste packages by non-destructive/destructive methods and;
- process qualification for conditioning plants including inspections.

The waste characterisation division of the ISR was founded some years later. Beside the development of non-destructive and destructive methods the ISR was responsible for the characterisation of radioactive waste packages characterisation for commercial organisations.
The ISR is equipped with radiochemical laboratories, glove boxes and hot cells.

In 1999 the characterisation group of the PKS was integrated into the ISR whereas documentation checking and process qualification remains at PKS.
Beside routine analysis for different organisations the main work of the ISR was related to the characterisation of old waste packages with insufficient or non-prevail declarations. From 1994 to 1998 the ISR investigated nearly 1000 old 200 l drums with non-destructive methods. Further the gas phase of each drum has been analysed. With respect to the results of the non-destructive analysis about 10 % of these old drums had to be checked by destructive methods.

In future one of the main subjects of the ISR will be the analysis of waste generated from the decommissioning of the AVR, a prototype high temperature gas cooled graphite-moderated reactor, located at Jülich.

In ***Italy***, since its creation, ENEA (Ente per le Nuove tecnologie l'Energia e l'Ambiente) operated in the area of nuclear power research, reactor development, fuel fabrication, spent fuel reprocessing and industrial promotion. When the ENEA activities were reoriented toward the closure of nuclear activities a specific branch for nuclear plant dismantling and radioactive waste management was established and in this frame the National Laboratory for the Radioactive Waste Characterisation early 1993 was created.

The ENEA Laboratory (RAD-LAB), closely connected with the Italian Nuclear Regulatory Authority (ANPA), performs its activities observing the main guidelines issued from the national and international Regulatory Bodies, in order to assess the necessary requirements to consider conditioned wastes acceptable for disposal.

The main tasks of the Laboratory in this field are:

- the determination of the radionuclide inventory in the primary wastes and packages by destructive and non-destructive methods and techniques;
- the determination of the chemical and physical properties of radioactive primary wastes and packages;
- the Quality Control on the treated and conditioned radioactive wastes;
- the formulation of matrices suitable to the immobilisation of radioactive wastes;
- the verification of the conditioning processes for radioactive wastes;
- the development of processes for the minimisation of volume of radioactive wastes;
- the analyses of non-characterised wastes or suspect materials.

The Laboratory is situated at Saluggia's ENEA Centre for the Destructive testing facilities and Casaccia's ENEA Centre for the Non-Destructive testing facilities and works in collaboration with the major Italian scientific organisations, universities, industries, national and local government agencies.

In ***Spain***, the Nuclear Fission Department (DFN) is a section of the Energy, Environmental and Technological Research Centre (CIEMAT: Centro de Investigaciones Energéticas Medioambientales y Tecnológicas"), whose aim is to improve the use of existing and alternative energy generation resources and systems, paying special attention in both cases to the environmental impact they cause.

The purpose of DFN is to carry out R&D projects in the nuclear field regarding operation, maintenance and enhancement of Nuclear Power Plant safety and availability and management of radioactive wastes.

The management of radioactive wastes (L&MLW) in Spain, under the responsibility of ENRESA, has given rise to the development of technology in order to carry out its radiological characterisation. The work developed in CIEMAT/DFN about this subject has originated the formation of a working group whose aim is the characterisation, homologation and radiological control of radioactive wastes and materials.

According to Annex IV of the CIEMAT-ENRESA Association Agreement, the Analysis and Characterisation of the Radioactive Waste Unit, belonging to the DFN, has developed and applied the work to the radiological characterisation of low and medium level radioactive wastes and its main aims are:

- Development and application of non-destructive methods: the non-destructive methods based on gamma spectrometry with semiconductor detectors allow suitable techniques in order to know both qualitative and quantitative composition of gamma-emitting radionuclides present in conditioning radioactive wastes. This technique has a broad field of application, both waste packages of different characteristics, heterogeneous solid wastes without conditioning and characterisation of radioactive lightning rods “in situ” [9].
- Development and application of destructive methods, in order to have available different sample preparation methods and to radiologically characterise the following radioactive wastes and materials: spent ion exchange resins, evaporator concentrates, graphite, deposit of corrosion products on nuclear steam-generator tubing, metals, resins or concentrates immobilised in cement and so on [7].
- Establishment of correlation factors for critical nuclides (long-lived radionuclides) to easily measurable key nuclides (^{60}Co and ^{137}Cs).

DFN works in collaboration with Nuclear Power Plants and other firms and organisations of the Spanish Nuclear Sector such as ENRESA, CSN, ENUSA, TECNATOM, ENSA, LAINSA, etc. It also participates in numerous research projects with countries of the European Union and the OCDE.

On the other hand, El Cabril Disposal Facility, constituted and operated by ENRESA, is an important part in the management of radioactive waste program in Spain. It has been designed incorporating the last advances on storage technology and has capacity enough for storing the medium and low level wastes generated in Spain until the first decade of next century.

In order to ensure compliance with the Quality Objectives required of the waste packages, which are to form part of the Disposal Units, ENRESA has developed a Characterisation Laboratory at the El Cabril. This laboratory is an essential tool in the development of the methodology applied by ENRESA for the acceptance of waste packages at the Facility and is equipped with all the resources and equipment required for the performance of the tests described as follows:

- Verification Tests on historic packages (Non-Typified)
- Characterisation Tests on current packages (Typified)
- Technical Verification Tests or Supercontrols.
- Determination of Non-conditioned wastes.
- R&D Activities.

Inside these tests are included total and segmented gamma spectrometry (non-destructive tests), mechanical testing, leaching, separation and chemical-radiochemical measurements on samples arising from waste packages (destructive tests) or non-conditioned wastes.

ENRESA works in collaboration with Nuclear Power Plants and organisations as CIEMAT. Nowadays, it participates with OCDE-AEN and EU in several projects.

In ***The Netherlands*** radioactive waste has to be transported to the Central Organisation for Radioactive Waste Management (COVRA) for treatment and storage.
As a result of all activities, NRG produced both liquid and solid waste streams, whereof the major part of the liquid waste is disposed to the sea after treatment. The remainder of the liquid waste is packed into drums or containers for transportation to the COVRA. Basically, the solid waste streams are classified by the supplier before packing to avoid the sampling of waste packages and to avoid transporting of non-radioactive waste to the COVRA. The waste is packed into 100 litre drums for transportation.
The philosophy of the COVRA regarding quality checking of radioactive waste is based upon the licensing system in The Netherlands and the price system of the COVRA. For practices with radioactivity the licensee has the obligation to install a bookkeeping system for the purchase and disposal of radioactivity. Calculation the fee for radwaste by the COVRA is by the volume of the radwaste (and not by the radioactivity). This guarantees that under-estimation of the radioactivity by customers is unlikely.
Good characterisation and controlling of the conditioning process of radwaste is a prerequisite for good radwaste management. The parameters of the conditioning process are monitored by methods of concrete technology (compressing strength and leaching). Drums with solid radwaste are measured at the producers site by radiation protection monitors (rpm's) of COVRA's Health Physics department. They perform swap tests, dose-rate measurements and in case of non-compliance, gamma-spectrometry measurements.

In the ***United Kingdom***, the Waste Quality Checking Laboratory (WQCL) established in 1985, is operated by NNC Ltd. NNC was awarded the contract in 1997 to quality check solid low level radioactive waste (LLW) on behalf of the Environment Agency, as part of the a routine monitoring programme which is managed by the National Compliance Assessment Service (NCAS). WQCL is required to determine and record the content of selected LLW disposals in accordance with agreed criteria. The Agency undertakes these monitoring programmes to act as both a check on site operators' results and to provide independent data on the exposure of the public.

WQCL carries out independent checks on solid LLW in the UK. Consignments of LLW seized by the Agency inspectors are transported to the WQCL at Winfrith for analysis by destructive and non-destructive methods. The results of quality checking campaigns are compared with the waste consignor's declaration and authorisations and reported back to the Agency.

Limits and conditions on the disposal of radioactive waste are detailed in site specific Authorisation and Transfer Certificates. Over 1100 sites in England and Wales are currently authorised, and include hospitals, universities and industrial research or manufacturing centres. The most significant amount of radioactive waste comes from a relatively small number of nuclear licensed sites. These include nuclear fuel fabrication and reprocessing plants, nuclear power plants, atomic research establishments and isotope production centres.

More than 99% of the solid LLW produced originates from these nuclear sites and is destined for disposal at the UK landfill site operated by British Nuclear Fuels plc (BNFL) at Drigg in Cumbria. Historically waste was tipped into trenches cut into clay to a depth of about 8 metres. This method of disposal ceased in 1994 and now suitable wastes are compacted and placed into half height ISO-containers at the Waste Monitoring and Compaction (WAMAC) facility at Sellafield. Compacted waste is conditioned in concrete grout and the containers are placed in a concrete lined vault. Waste accumulated in 200 litre drums is inhomogeneous and generally consists of trash from areas of low contamination such as discarded protective clothing overalls, overshoes and gloves as well as refurbishment and decommissioning waste.

3.0 Capabilities of the Network

3.1 The Partners

Each Laboratory of the WGB offers its experience and knowledge in the field of waste forms and waste packages characterisation.
Different procedures have been developed and qualified to analyse the waste streams such as ion exchangers, reactor filters, cements, liquids and solids coming from fuel fabrication plants, waste forms and packages. Combination of NDA methods, mainly segmented gamma scanning and passive neutron counting, along with low-level waste assay destructive techniques, are currently used.

Destructive methods are generally based on traditional analytical techniques, including different sample preparation methods and counting techniques.

New technologies are under development or qualification, mainly within the Framework Programmes of the European Community, such as the application of the chemical separation technique based on ion and liquid chromatography (HPIC, HPLC) combined with mass spectrometry and Liquid Scintillation Counting [10]. Experimental studies have been conducted on dissolution and sample preparation procedures, as part of α nuclide control project [7].
The results obtained are exchanged and compared among laboratories.

The determination of the critical nuclides inventory and the identification of possible correlation between those isotopes and key nuclides is one of the common concerns of the laboratories.

Since physical, chemical and radiochemical properties of the waste product and its components (matrix, radionuclides, container, etc.) have to meet the specific criteria for the transport, interim and final disposal, many member state of the European Community have developed analytical methods on behalf with national regulatory bodies and waste management authorities.

An overview of the Network capabilities is reported below. In table 1 are reported and compared techniques and instrumentation currently used or actually under development. In table 2 is reported a summary of isotopes analysed in each laboratory. More details are summarised in appendix I.

SCK•CEN (***Belgium***) has been involved for many years in the characterisation of radioactive wastes. These involve as well vitrified, cemented and bitumised wastes. With the aim to assess the safety of waste storage, long-term leaching experiments are developed to study the behaviour of conditioned wastes exposed to water or interstitial clay water.

Methods for the determination of long-lived nuclides are also worked out. They have especially focused on the analysis of reactor concentrates, ion exchangers and filters.
Appropriate dissolution methods followed by dedicated separation schemes have been extensively studied. The measurement of the nuclides is performed by using different chemical and radiochemical techniques.

Chemical techniques:
- Inductively Coupled Plasma - Atomic Emission Spectrometry
- Inductively Coupled Plasma - Mass Spectrometry
- Thermal Ionisation Mass Spectrometry
- Ion Chromatography

Radiochemical techniques:
- Alpha-spectrometry
- Gamma-spectrometry
- Liquid Scintillation Counting
- X-Ray Low Energy Spectrometry

Besides the destructive techniques, NDA methods are also available for the measurement of conditioned waste drums:
- Neutron Measurements
- Q2 (Low-level Waste Assay System)
- SGS (Segmented Gamma Scanner)

The characterisation of radioactive wastes is performed on unconditioned as well as conditioned waste forms. Unconditioned wastes samples are taken by the producer.
Nuclear power plants deliver homogenised samples of evaporator concentrates and mixed bed ion exchange resins. However, the amount of primary sample is too important to be handled as such. To reduce the exposure to radiation, sub-samples need to be taken before their treatment in the laboratory.
Resins are simply divided in smaller portions which are totally dissolved.

Large amounts of concentrates (up to 30 litres) are previously filtered to separate the solution from the precipitate. A homogeneous part of each fraction is then dissolved and proportionally recombined.
Coolant filter cartridges are dissolved in a special shielded vessel and sub-samples are taken whilst continuously mixing the solution.
Ash samples are milled and mixed for a long period before sampling.

The sampling of conditioned wastes has been performed on vitrified and bitumized radioactive waste. The sample is extruded by core drilling. The homogeneity is evaluated by optical and electron microscopy and by thermal analysis. The distribution of the nuclides is investigated by gamma scanning.

The inventory of radionuclides in primary the waste and the cemented waste has been compared. The data were evaluated and used as input data for a mathematical model and calculation code developed by the Belgian authorities to predict the inventory of critical nuclides produced in the main PWR streams.
An interlaboratory intercomparison campaign organised by the EC allowed to validate the procedures.

Great importance has been given to the participation to an interlaboratory measurement evaluation programme whose objective was to validate the results obtained by different methods and laboratories in safety studies related to the disposal of low and intermediate level wastes

because of the long term risk of a series of critical nuclides which are not easily measurable in the waste packages for their low content, their low specific activity or the particular characteristics of their radiation. Furthermore measurements have been continued on all samples for those isotopes whose procedures have been already qualified. Therefore limited progress has been made in the development of the procedures for ^{99}Tc, ^{129}I and transplutonium elements. Moreover some procedures already developed and qualified had to be adapted again with regard to the particular characteristics of some of the samples. For ^{129}I apparently the measurement at the low energy gamma ray of 39.6 keV is more sensitive than the beta ray measurement, even when differences in counting times are taken into account. The beta measurements lasted about 1000 minutes, the gamma spectra were taken during 2 10^5 seconds. The detection limit for ^{129}I was decreased by a factor of 6 to 8. The limit of 0.14 Bq in a separated fraction corresponds to 1.8 Bq per gram of resin dried at 105 °C.
The comparison of the results show satisfactory agreement between the different laboratories within a range of a few percent for the key nuclides up to 30 % for very low levels of critical nuclides.
Non-destructive and destructive analysis methods were applied for the characterisation of historical waste packages. Techniques have been developed for the inspection and sampling of raw waste, ashes and conditioned waste packages up to 1000 kg. Core drilling equipment has been installed for the safe sampling of cemented waste containers over their full height allowing for contact radiation doses over 10 mSv and alpha emitters in the range of 10 kBq/g.
Calibrated segmented gamma scanning of waste drums up to 500 kg is installed to measure the activity distribution over the package, to define appropriate sampling plans and to establish total nuclide inventories in the waste package.
Grinding, homogenisation, sub-sampling and dissolution techniques have been developed for most types of raw and conditioned historical waste.
Specific measurement methods have been developed to verify compliance with storage site requirements, in particular with respect to total alpha activity and fissile material content.
Cross checks and calculation models have been developed and implemented to correlate different measurement types (alpha, beta, gamma, dose rate, weight, density, humidity content etc.) for underpinning the isotopic inventories and characteristics in the final waste package documentation.

The principal types of nuclear waste, the **CEA/DESD** in ***France*** has been working on for the last years, are high salt containing solutions (effluents), spent ion exchangers, reactor filters, cements, graphite, hulls, and to a lesser extent evaporator concentrates [11].

The research has focussed on the determination of long-lived nuclides considered to be critical for the safety of surface storage in France: ^{3}H, ^{14}C, ^{36}Cl, ^{59}Ni, ^{63}Ni, ^{79}Se, ^{93}Mo, ^{93}Zr, ^{94}Nb, ^{99}Tc, ^{107}Pd, ^{129}I, ^{135}Cs, ^{151}Sm, ^{237}Np, together with major and trace elements.

Many procedures have been developed for this purpose, including specific chemical separation schemes and different measurement strategies. The general strategy for radiochemical analyses includes three main steps: 1- Extraction of the analyte from the matrix; 2- Separation of the analyte from potentially interfering nuclides; 3- Measurement. The extraction of the analyte from the matrix is currently performed using acid dissolution methods. In some cases, thermal methods such as pyrohydrolysis or combustion can also be used. The separation from interfering isotopes uses very different means, including precipitation, liquid-liquid extraction, ion exchange chromatography, reversed phase liquid chromatography, electrodeposition, electrothermal vaporisation, hydride generation, etc. Measurement techniques are selected according to selectivity and detection capabilities for each isotope. In general, radionuclides with half-lives longer than 10^4 years (^{93}Zr, ^{99}Tc, ^{107}Pd, ^{129}I, ^{135}Cs, ^{237}Np) are measured by Inductively Coupled Plasma – Mass Spectrometry whereas radionuclides with shorter half-lives are measured using radiometric techniques, i.e. alpha, gamma and X-ray spectrometry, or liquid scintillation counting.

The instrumental techniques involved in this field are:

- Inductively Coupled Plasma - Mass Spectrometry, with Flow Injection Analysis, Electro-Thermal Vaporisation and Laser Ablation Systems [12-14]
- Total Reflection and Wavelength Dispersive X-Ray Fluorescence Spectrometry
- Thermal Ionisation Mass Spectrometry
- Inductively Coupled Plasma - Atomic Emission Spectrometry (with SCD detectors)
- Atomic Absorption Spectrometry
- Alpha Spectrometry (with electro-deposition)
- Liquid Scintillation Counting
- Gamma Spectrometry
- X-Ray Low Energy Spectrometry
- Liquid Chromatography (HPLC, HPIC) [15]
- Capillary Electrophoresis
- Potentiometry, Spectrophotometry.

Sampling procedures are generally undertaken by the partners of the DESD who select and send discrete sub-samples to the laboratory.

For the destructive characterisation of the different waste matrices as there are resins, evaporator concentrates, core material, crud, etc., **TUM/RCM**, the Institut für Radiochemie der Technischen Universität München (***Germany***), can refer to an extensive analytical equipment:

- Alpha Spectrometry
- Liquid Scintillation Counting and Spectrometry (LSC)
- Gamma Spectrometry (coincidence and anti-Compton techniques)
- Neutron Activation Analysis (NAA)
- Scanning Electron Microscopy and Energy Dispersive X-ray Analysis (SEM-EDXA)
- Accelerator Mass Spectrometry (AMS)
- High Performance Ion Chromatography (HPIC)
- High Pressure Liquid Chromatography (HPLC)
- Inductively Coupled Plasma Mass Spectrometry (ICP-MS)
- Atomic Absorbance Spectrometry (AAS)
- Gas Chromatography coupled with Mass Spectrometr (GC-MS)
- Differential Thermal Analysis (DTA), Thermal Gravimetry (TG), and others.

It is also involved in analyses of environmental samples (Low Level): analysis of alpha, beta, gamma emitters down to 10^{-6} to 10^{-2} Bq/g, analyses of samples from nuclear power plants: evaporator concentrates, ion exchange resins, core reactor components with activities up to 10^{13} Bq/g, testing of conditioning procedures (laboratory scale) and radiolysis behaviour of radioactive compounds and components.

TUM/RCM uses appropriate techniques for all kinds of destructive and/or invasive sampling procedures (core drilling without liquid cooling, cutting, etc) and sample preparation (grinding, milling, etc.).

FZJ-ISR (***Germany***) is involved in the non-destructive and destructive characterisation of all types of radioactive waste, e.g. cemented wasted, ion exchange resins, evaporator concentrates, graphite, metals and crud. The FZJ-ISR performs this work as subcontractor of the "Produktkontrollstelle".

Due to the chemical and physical properties of the samples different pre-treatment methods had been develop:

- High temperature combustion
- Microwave dissolution
- Leaching device
- Salt fusion
- Liquid-Liquid extraction
- Solid phase extraction
- Ion exchange chromatography
- High performance liquid chromatography
- Distillation
- Gas Chromatography

Due to the activity of the waste, the analysis will be performed in hot cells, glove boxes or low level laboratories, with the following analytical equipment:

- α-spectrometry
- γ-spectrometry
- x-ray spectrometry
- Liquid Scintillation Counting
- Total α-, β-counting
- Radio Gas Chromatography

Additional equipment, only for special demands, are:

- Inductively Coupled Plasma - Mass Spectrometry
- Inductively Coupled Plasma - Atomic Emission Spectrometry
- Wavelength Dispersive X-Ray Fluorescence Spectrometry
- Gas Chromatography equipped with Mass Spectrometry
- Differential Thermal Analysis, Thermal Gravimetry
- UV-VIS Spectrophotometry
- Infrared Spectrophotometry
- Electron microscopy and Energy Dispersive X-ray Analysis
- Nuclear activation analysis

Further a gas sampling system had been developed for 200 l drum. This device allows gas sampling without destroying the drum sealing.

The sampling is mostly undertaken by the waste producers as part of qualified waste treatment processes and sample aliquots are send to the ISR laboratories. Further a sampling device for 200 l drums is installed in a glove box. This device allows the sampling of unconditioned waste and drilling of cemented waste. The drilling device is air cooled to avoid contact with cooling liquids. A sampling device for high level waste is in the licensing procedure.

The characterisation process carried out by **ENEA-RAD-LAB** (***Italy***) is focused on the checking of the chemical and radiochemical properties of primary wastes (liquid and solid) and mechanical, physical, chemical and radiochemical properties of the waste forms (normally cements) and packages.

The waste streams currently analysed are:
- Liquid solutions coming from reprocessing plants (HLLW, LLW);
- Liquids and solids coming from fuel fabrication plants (including waste containing Pu and U);
- Raw technological wastes (gloves, papers, plastics, small lab equipment, glass, ventilation filters, etc.);
- Cements.

Many determinations are currently in routine, as follows:
- the resistance of waste forms toward: the compressive strength, the thermal cycling, the fire and the biological attacks, the immersion in free water, the high radiation exposure and the water leachability;
- the matrix composition, the radiochemical and isotopic compositions and the amount of the fissile materials;
- the presence of hazardous components and materials that could hinder the correct solidification.

NDA techniques (segmented gamma scanning and passive neutron counting) are widely used for the nuclide determinations in conjunction with sampling and chemical and radiochemical destructive analyses.
Preparation and pre-treatment techniques of the waste stream samples to be investigated by destructive analysis, consist of dissolution methods (e.g., acidic digestion, microwave dissolution, etc.), tracing and dilution until they are easily handled in suitable remote conditions. Nuclide matrix separations, where necessary, are performed by optimised organic extraction (liquid and solid phase), using liquid/liquid extraction systems or columns. HPIC of anions and metals in liquid samples are applied and optimised even in high salt containing solutions.

The instrumentations applied by the Laboratory, in this field, are:
- Inductively Coupled Plasma - Mass Spectrometry
- Inductively Coupled Plasma - Optical Emission Spectrophotometry
- Furnace Atomic Absorption Spectrophotometry
- Dispersive Optical Fluorescence Spectrophotometry
- Thermal Ionization Mass Spectrometry
- UV-VIS Spectrophotometry
- Total alpha, beta, gamma and neutron counting
- Alpha-Spectrometry
- Liquid Scintillation Counting (steady and flow cells)

- Gamma Spectrometry
- X-Ray Low Energy Spectrometry
- High Performance Ionic Chromatography, also equipped with LSC detector
- High Resolution Gas Chromatography - Mass Spectrometry and conventional detectors
- Neutron counting
- Potentiometry.

The general philosophy of the organisation, is to make a good characterisation of primary waste in combination with a strict control of process parameters during the conditioning, in order to avoid sampling of waste packages. This means that the radionuclide content of the waste packages is automatically established and it may be verified only by non-destructive measurements. However, this procedure can't ever be adopted and frequently sampling seems to be necessary.
Retrieved low level solid wastes, stored in 220 litre drums, are periodically characterised in the 018 facility of EUREX plant. Their content is assessed by means of the following equipment:
a glove box for the sampling of the material in the drums [14].
Because of its properties, the glove-box can be used for the sampling and selection of 220 litre drums containing low alpha-contaminated wastes, low exposure rate and with an interior manufacturing of the material such to be extracted manually.
Destructive analysis methods are then applied to the specimens sampled in the above-mentioned glove-box.
For the detection of the radionuclides, γ-emitting nuclides are analysed by global γ-spectrometry.
Then sampling is carried out by drawing out the more representative radioactive specimens from the drum. The content of nuclides of concern is then determined in the solid samples by dissolution, extraction, chemical and radiochemical analyses. Standard addition method is widely used to determine the yield of extractions.
Then the nuclide concentrations are correlated to the γ-emitting nuclide concentrations.
Nuclide correlations are finally used to calculate total inventory of nuclides into the drum.

At **CIEMAT**/DFN (***Spain***), the characterisation of radioactive waste by destructive methods needs to use radiochemical analysis procedures and radiometric techniques for separation, identification and quantification of radionuclides present in radioactive waste.

The technologies applied by the laboratory in this field are:

- Combustion oven (OX-500 Biological Material Oxidiser) used to carry out ^{3}H and ^{14}C analysis before its measured by liquid scintillation counting.
- UV-VIS Spectrophotometry
- ZnS (Ag) scintillation detectors
- NaI detector
- Beta proportional counter
- Alpha spectrometry with semiconductor detector
- Liquid Scintillation Counting
- Ge-HP or Ge-Li detectors associated with 8K channels-spectrometers and a microcomputer. Results are processed using the spectra analysis software "Spectran-AT" from Canberra.
- X-ray spectrometry with a low energy photon detector
- Potentiometry.

First of all, the sampling procedures of the resins, concentrates and sleeve graphite powder are undertaken by the producers (Nuclear Power Plants) who select and send sub-samples to the DFN laboratories [17]. The sampling procedures of the cemented waste packages are performed in "El Cabril". In this case, the cemented waste package, which is going to be analysed by gamma spectrometry, is randomly chosen among all the packages that come from the same batch, that is to say, that are made in the same way (dosage, type of waste, etc). The samples destined to destructive analysis, specimens and powdered samples, are obtained in the following way:

- Specimens: the specimens are extracted by drilling in the longitudinal sense (from the bottom to the lid), so specimens of the high, medium and low zone of the package are obtained.
- Powdered samples: only a sample is obtained from each package since a filter is inserted in the aspiration system of the drill. In order to achieve representative samples, the filter is introduced into different levels of the hole.

These samples are sent to the DFN laboratories for dissolution, homogenisation, chemical preparation and performance of the radiochemical separation processes required for the determination of certain radionuclides (destructive tests).

Then, with respect to the methods used for destructive analysis, it is described, as the most typical example, the procedure carried out in order to analyse an ion-exchange resin. The resin is divided into three aliquots:

- One aliquot is analysed in a combustion oven where the sample combusts in a stream of oxygen gas at 900ºC. In the combustion is formed carbon dioxide and water vapour. Carbon dioxide passes through a carbon trapping solution where it reacts immediately. ^{14}C is analysed by liquid scintillation counting. On the other hand, the water vapour is condensed, trapped and ^{3}H is equally analysed by liquid scintillation counting.
- The second aliquot is dissolved by oxidation with hydrogen peroxide in acid medium and using a reflux cooler, being in this solution analysed ^{99}Tc and ^{129}I [19].
- Finally, the third aliquot is equally dissolved by oxidation. Besides, in this case, as a consequence of the presence of metallic oxides it is necessary to dissolve them by acid treatment. In the solution obtained the rest of nuclides ^{45}Ca, ^{55}Fe, $^{59/63}Ni$, $^{89/90}Sr$, $^{93m/94}Nb$, ^{238}Pu, $^{239/40}Pu$, ^{241}Am, ^{241}Pu, ^{242}Cm and ^{244}Cm are analysed.

Concerning the evaporator concentrate, the same solubilisation procedures are used.

Regarding the general methodology used in each separation process to determine the activity of the long-lived radionuclides the following steps are performed:

- Depending on the radionuclide to analyse, a known amount of carrier is added to an aliquot coming from the dissolution of the original sample.
- In the aliquot is performed a specific procedure of separation which can consist in precipitation, extraction or ion exchange chromatography [8, 18].
- Once the procedure is carried out, the isolated radionuclide is checked by gamma spectrometry, to verify the quality of the separation procedure.
- Finally, the radionuclide is analysed or by liquid scintillation counting, by gamma spectrometry (with a low energy photon detector or Ge-HP or Ge-Li detectors) or by alpha spectrometry.

In short, the long-lived nuclides (alpha, beta and gamma emitters) determined by analytical methods are:

- Alpha emitters: ^{238}Pu, $^{239/240}$Pu, ^{241}Am, ^{242}Cm, ^{244}Cm, ^{234}U and ^{238}U.
- Beta emitters: ^{3}H, ^{14}C, ^{45}Ca, ^{55}Fe, ^{63}Ni, $^{89/90}$Sr, ^{99}Tc, ^{129}I and ^{241}Pu.
- X and gamma emitters: ^{54}Mn, ^{55}Fe, ^{59}Ni, ^{60}Co, ^{65}Zn,$^{93m/94}$Nb, ^{134}Cs, and ^{137}Cs.

The destructive procedures, applied to radioactive waste in Characterisation Laboratory at the El Cabril Disposal Facility **ENRESA** (***Spain***), are:

- Drilling: this is made in the Handling Cell that is equipped with remote manipulators and a leaded window. This zone is used for performance of other operations such as the extraction of samples from packages, the preparation of test pieces from the samples extracted, mechanical testing (i.e.: compression resistance) of these test pieces.
- Leaching: this test can be performed on real, full-scale packages and on samples taken from the packages.

Chemical and Radiochemical Measurements are performed on the solid or liquid samples arising from the operations describes above are transferred to the Preparation Laboratory for dissolution, chemical preparation and performance of the radiochemical separation processes required for the determination of certain radionuclides.

The measurement techniques used are:

- Gamma spectrometry
- Total alpha, beta counting
- Liquid Scintillation Counting
- Inductively Coupled Plasma
- Alpha spectrometry

LVCR EL Cabril for the radionuclides determination are used the following methodologies:

- An aliquot of the original sample and known amounts of carriers are dissolved in acid medium obtaining a solution to analyse.
- ^{3}H and ^{14}C**:** after distillation (^{3}H) and CO_2 collect (^{14}C), these isotopes are measured by LSC. An alternative method is the analysis of an aliquot in a Combustion Oven.
- ^{55}Fe, ^{63}Ni, ^{90}Sr, ^{129}I, ^{94}Nb, ^{238}Pu, $^{239/240}$Pu, ^{241}Am, ^{242}Cm, ^{244}Cm: different procedures of separation are carried out in aliquots coming from the original solution.
- Gamma emitters: the original solution is directly measured by gamma-spectrometry.

The destructive analytical techniques routinely used at **NRG** and **COVRA** (***The Netherlands***) are applied to all aqueous streams, to waste water and reactor water, to different matrices, dust collected on silica or teflon filters.

Before characterising the physical and chemical properties of the waste streams, the samples receive a preparation depending on type. The pre-treatment processes are dissolution and concentration, followed by chemical separation such as distillation, anion-exchange and electro-deposition on stainless steel discs.

After the elimination of the matrix interference, the samples can be analysed by alpha- and beta-spectrometry with LSC, and by gamma-spectrometry with high purity germanium detector.

The alpha and beta measurements of LLW and ILW solid materials are conducted with LSC after extraction in liquid (count-off).

All the work procedures are certified according to ISO 9001.

NNC Destructive testing is carried out at the **WQCL** facility (***United Kingdom***) to provide fingerprint information on the radioisotope content for comparison with the waste producer's declaration. It is also used to identify and quantify the alpha and beta emitting radionuclides since these cannot be measured using NDT techniques.

In general approximately 5% of the packages or drums within a consignment are analysed destructively. The criteria used for the selection of drums for sampling is dependent on Agency requirements and the nature of the waste being assessed. Examination by X-radiography for example, may reveal prohibited materials such as aerosol canisters or free liquids which must be removed. Drums may also be selected from examination of the gamma emitting radioisotope content as found by segmented gamma scanning (SGS) and by specific request from the Agency e.g. based on the origin of the waste within the producer's site.

Once a drum has been selected for destructive testing, it is taken to the radiochemistry laboratory and attached to the waste receipt glove box. The lid is then removed from inside the glove box and the contents of the drum are examined. The glove box is fitted with a fixed video camera and all drum opening operations are recorded. Any prohibited items found in the drum are photographed to provide evidence of the finding and are segregated from the remainder of the waste which is then transferred to a second glove box. Here the waste is packaged if necessary and the contact radiation dose rate and weight are measured and recorded. Representative sub-samples are then taken and transferred to fume hoods for radiochemical analysis.
Destructive testing begins with the preparation of an aqueous solution of the solid sample taken from the waste.
This is accomplished at WQCL using the following methods:

- Acid leaching
- Acid dissolution
- Fusion

Once the primary solution has been prepared, aliquots are first taken for the determination of total alpha, total beta, and individual gamma emitting radioisotopes. These tests are performed using the following techniques:

- Alpha Spectrometry
- Liquid Scintillation Counting
- High resolution Gamma Spectrometry

The results of these tests are used to identify components which may require separation and further analysis. Chemical separations are carried out on the primary solution to isolate radionuclides of interest. Separation techniques commonly used at WQCL are listed below.

- Ion Exchange Chromatography
- Solvent Extraction
- Distillation

WQCL have developed specific methods for the determination of individual radioisotopes. These methods, which are listed below, have all been accredited by the United Kingdom Accreditation Service (UKAS) as reaching the national quality assurance standard for calibration and testing laboratories.

- Radioisotopes of; U, Pu, Am, Th, and Cm
- Beta emitters; ^{3}H, ^{14}C, ^{35}S, ^{36}Cl, $^{90}Sr/^{45}Ca$, ^{99}Tc
- X-ray emitters; ^{55}Fe, $^{125}I/^{129}I$
- Others; $^{210}Pb/^{210}Po$

The content of each package is monitored for radioactive contamination and emitted radiation dose rate. Any waste found with a measurable radiation dose rate or radioactive contamination above background is removed for analysis or further divided into representative sub-samples. All other waste without a measurable level of contamination is representatively sampled. Standard statistical techniques such as coning and quartering are used for most matrices where uniform radiation and contamination is encountered [20].

Radiometric measuring instrumentation routinely calibrated and performance checked using reference sources traceable to National Standards.

Typical waste matrices found in consignments for destructive testing include standard laboratory soft waste such as paper and other cellulosic material, cardboard, tissues, disposable lab coats, plastics and rubbers such as disposable gloves, overshoes, sheeting, sample bags, pipette tips, syringes, and general rubbish such as metals, wood, concrete, cements, dirt, and stones. Organic samples that can have radioisotopes chemically bound to the matrix, such as soil and sediment, where the presence of humic and fulvic acids prevent the simple leaching of actinide species, are first heated in a muffle furnace at increasing temperatures for fixed times to break down the organic species and limit the extent of volatilisation. Matrices such as concrete will be powdered first before monitoring to ensure that no radioactive species remain undetectable prior to sampling. So far, no fully homogeneous drums have been consigned to the department and all samples handled have been readily monitorable.
Representative sampling has been easily achievable.

Table 1: **Techniques and intrumentation**

Relevant techniques and Instrumentations currentely used	**States and Laboratories Member**									
	Belgium	**France**	**Germany**		**Italy**	**Spain**		**The Netherlands**		**United Kingdom**
	SCK·CEN	**CEA**	**FZJ**	**TUM/RCM**	**ENEA**	**CIEMAT**	**ENRESA**	**COVRA**	**NRG**	**WQCL**
Atomic Absorption Spectrometry		X		X	X			X		
Alpha Spectrometry	X	X	X	X	X	X	X	X	X	X
Capillary Electrophoresis		X								
Differential Thermal Analysis and/or Thermal gravimetry			X	X						
Dispersive Optical Fluorescence Spectrophotometry					X					
Dispersive X-Ray Fluorescence Spectrometry		X		X						
Electron Microscopy			X	X						
Electro-Thermal Vaporisation and Laser Ablation Systems –ICP-MS		X		X						
Gamma Spectrometry	X	X	X	X	X	X	X	X	X	X
Gas Chromatography equipped with classic detectors			X	X	X					
Gas Chromatography equipped with MS detector			X	X	X					
Ion Chromatography / Ion exchange chromatography	X	X	X		X				X	X
High Performance Ion Chromatography - classic detectors, and HPLC		X	X	X	X					
High Performance Ion Chromatography equipped with LSC detector			X		X					
High Performance Ion Chromatography equipped with ICP-MS detector										
Inductively Coupled Plasma - Atomic Emission Spectrometry	X	X	X	X	X		X			
Inductively Coupled Plasma - Mass Spectrometry (flow injection)	X	X	X	X	X					
Infrared Spectrophotometry		X	X	X						
Liquid Scintillation Counting	X	X	X	X	X	X	X	X	X	X
Neutron Activation Analysis			X	X					X	
Neutron Counting					X					
Potentiometry		X		X	X	X				
Radio Gas Chromatography			X							
Secondary Ion Mass Spectrometry				X						
Scanning Electron Microscopy				X						
Thermal Ionisation Mass Spectrometry	X	X			X					
Total alpha-beta-gamma countings	X		X	X	X	X	X	X	X	
UV-VIS Spectrophotometry		X	X	X	X	X			X	
X-Ray Low Energy Spectrometry	X	X	X		X	X				

Table 2 (part one): Specific isotopes analysed in each laboratory* and techniques currently applied.

Isotope	CEA	CIEMAT	ENEA	ENRESA	FZJ	NRG (COVRA)#	NNC	SCK·CEN	TUM/ RCM
^{3}H	LSC	LSC	LSC	LSC	LSC	LSC#	LSC	LSC	LSC
^{14}C	LSC	LSC	LSC	LSC	LSC	LSC#	LSC	LSC	LSC
^{35}S	LSC					LSC^{c}	LSC		
^{36}Cl	LSC	LSC	LSC HPIC-LSC		LSC	LSC^{c}	LSC	f.d.	LSC
^{41}Ca					XRS				AMS
^{45}Ca		LSC					LSC		
^{54}Mn			HPIC-LSC						
^{55}Fe	LSC	LSC	XRS HPIC-LSC	LSC	XRS	LSC	XRS	XRS	LSC
^{59}Ni	XRS	XRS	XRS HPIC-LSC					XRS	
^{63}Ni	LSC	LSC	LSC HPIC-LSC	LSC	LSC	LSC	u.d.	LSC	LSC
^{65}Zn		γS		γS		γS#	γS		γS
^{79}Se	ICP-MS								AMS
^{89}Sr		LSC				LL βC#	u.d.		LSC
^{90}Sr	LSC	LSC	LSC HPIC-LSC	LSC	LSC	LL βC#	LSC	LL βC	LSC
^{93}Mo	XRS								
^{93}Zr	ICP-MS	u.d.			u.d.			f.d. ICP-MS	
^{93m}Nb	XRS	XRS	u.d.						
^{94}Nb	γS	γS	u.d.	γS			γS	γS	γS
^{99}Tc	ICP-MS	LSC	LSC HPIC-LSC	u.d.	u.d.	LL βC	LSC	LSC ICP-MS	LSC
^{107}Pd	ICP-MS							u.d. ICP-MS	
^{108m}Ag	γS								γS
^{110m}Ag	γS						γS		γS
^{126}Sn	ICP-MS								ICP-MS
^{125}I						γS#	XRS		γS
^{129}I	ICP-MS	XRS	γS	LSC		u.d.	XRS	γS/ICP-MS	NAA
^{131}I						γS#			
$^{134/137}$Cs	γS	γS	γS	γS		γS#	γS	γS	γS
^{135}Cs	ICP-MS		TIMS					TIMS ICP-MS	
^{144}Ce/^{144}Pr									γS
^{151}Sm	LSC		u.d.				γS		
^{210}Po					u.d.	αS	αS		
^{210}Pb						αS LL βC γS	LSC (Cerenkov)		
^{226}Ra	αS				u.d.	γS			
^{228}Ra						γS			

Table 2 (part two)**:** Specific isotopes analysed in each laboratory* and techniques currently applied.

Isotope	CEA	CIEMAT	ENEA	ENRESA	FZJ	NRG (COVRA)#	NNC	SCK·CEN	TUM/ RCM
^{228,230,232}Th						αS	αS		αS
^{228,234}Th						γS	αS		
^{232,233}U			αS				αS		αS
^{234,235,236,238}U	ICP-MS	^{234,238}U αS	O.F. UV-VIS ICP-OES ICP-MS TIMS		αS	αS O. F. γS	αS	TIMS αS ICP-MS	αS
^{237}Np	ICP-MS		αS		αS		γS	αS	αS
$^{238,239+240}$Pu	αS	αS	αS TIMS	αS	αS	αS	αS	TIMS αS	αS
^{241}Pu	LSC	LSC	LSC	u.d.	LSC	LSC	LSC	LSC	LSC
^{241a}m	αS	αS	αS	αS	αS	αS	γS	αS	αS
$^{242,243+244}$Cm	αS	αS	αS	αS	αS	αS	αS	αS	αS

* u.d.: available but not routinely applied
f.d.: planned for future development

\# Both NRG and COVRA apply the technique on the specific isotope. No mark indicates that only NRG analyse the specific isotope

c Only at COVRA

For the other abbreviations and symbols see the glossary section (appendix II)

3.2 Analytical Results: Evaluation and Reporting.

In the face of the differences between the participants in for instance organisation, analytical equipment and analytical skills there is some overlap. In this section a summarisation is given how the participants are dealing with the evaluation and reporting of analytical results without being specific.

All the participants have organised their Quality Assurance (QA) in such a way that instruments that are been used are routinely calibrated and that the performance is checked using reference sources. The (radiochemical) methods used have been evaluated for possible interferences, and the precision, accuracy, and reproducibility has been investigated and estimated. Reference solutions used as tracers in the radiochemical separations or to determine counting efficiencies in LSC are traceable to National Standards. In this respect participation in inter-laboratory comparison exercises is as an example of a powerful tool to ensure QA.

The declared activity content of each consignment is detailed by the waste producer on a standard form or equivalent and contains an itemised listing of the individual radionuclides. There may also be information supplied that details the radioactivity content of each individual drum in the consignment. The results from the non-destructive testing analysis of the drums by gamma-spectroscopy (i.e. SGS) will provide information on the gamma radioactivity content of each drum. These results can be summed to give the total gamma content of the whole consignment along with its gamma specific activity from the weight measurements of the consignment. Furthermore, these results can also be used to determine a gamma fingerprint for each drum and for the consignment as a whole.

The drums that are selected for destructive testing are sampled. Any individual samples that are removed for analysis are weighed prior to radiochemical analysis on the prepared solution or solid sample. The radiochemical content of each leach is determined and the specific activity of the original solid sample can be calculated. The radioactivity content of each package in the drum can then be calculated from the sub-samples. All the results are checked and finally a vertical audit will be carried out on all the destructive and non-destructive results. Routine audits are performed on each stage of the operations to ensure the integrity of the data. Once all the results have been verified the full discussion report is produced.

4.0 Conclusion

Within the European Network, Working Group B has been set up to promote harmonisation of destructive radiochemical analyses between the partners.

All participants contributed to a combined manual, referred to as a "cookbook" of destructive radiochemical techniques, outlining types of radionuclide, methods, limits of detection and instrumentation.

The compilation of various methods has pointed out a lot of similarities and some useful differences in the destructive analyses. These differences help to highlight possible areas of improvement between partners.

The varying views of the partners have helped to forge more understanding and exchange of information has undoubtedly benefited the members of Working Group B.

In the different laboratories, a common strategy is carried out, including sampling, dissolution, separation and measurement steps.

Sampling procedure is a key step of destructive methodology, and each partner has explained it's sampling techniques. The common problem of taking a representative subsample was addressed in this project. By evaluation of statistical sampling techniques, each partner was able to obtain improved homogenous sub-samples from the heterogeneous radioactive waste matrix.

Dissolution techniques were investigated, and it was found that leaching, fusion and microwave dissolution were the most widely used methods. The choice of the method depends on many parameters, i.e. matrix, sample size, detection limit and the physical/chemical properties of the radionuclide.

From the work carried out, it was concluded that it is of great importance to develop methods to isolate radionuclides from the bulk matrix. These techniques are based on precipitation, liquid-liquid extraction and chromatography, especially HPLC.

Finally, the measurement and identification of radionuclides can be done by common radiometric counting techniques, and also by mass spectrometry. The common counting techniques are:

Alpha spectroscopy
Liquid scintillation counting
Gas proportional counters
Gamma spectroscopy
Low energy x-ray spectroscopy.

The mass spectroscopic techniques used include ICPMS, TIMS, SIMS, and AMS.

The coupling of separation techniques is sometimes used to enhance detection limits and resolution, e.g. HPLC-LSC and ETV-ICPMS.

This review has highlighted a need for further research and co-operation in development of existing methods in order to improve some separation techniques, analytical procedures and determination of uncertainties, on radionuclides already considered or on radionuclides not considered by all members and that can be of its interest. That was the general feeling of all members of WGB and it was also supported by the panel of experts selected by the Commission for the evaluation of proposals presented for financial support to the Fifth Framework Programme of the European Commission in Nuclear Energy. As result of this, two multipartner projects (DACAPO and Interlab-analysis) [21-22] are being developed. Results obtained from these two projects could be the point for a better starting and a more harmonised treatment of some aspects such as sampling, detection limits and the separations techniques, analytical procedures and determination of uncertainties previously mentioned.

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Belgium: *SCK•CEN*

Nuclides	Chemical preparation	Measurement	Application
^{3}H	Acid digestion - Vacuum Distillation	LSC	Concentrates, resins, filters
^{14}C	Acid oxidant digestion - Off-gas trapping - Acidolyse - Trapping in Carbosorb®	LSC	Concentrates, resins, filters
^{55}Fe	Acid digestion - chromatography on anionic resins - evaporation on disk	X-ray	Reactor steel
^{59}Ni	Acid digestion - Extraction with D.M.G.	X-ray	Concentrates, resins, filters
^{63}Ni	Acid digestion - Extraction with D.M.G.	LSC	Concentrates, resins, filters
^{90}Sr	Acid digestion - solid phase liquid extraction on Sr•Spec® Columns - ^{90}Yttrium precipitation - counting planchet	ß-counting Low Level Proportional Counter	Concentrates, resins, filters
^{94}Nb	Acid digestion - Extraction with TBP - Backextraction with dil. HCl	γ-spectrometry	Concentrates, resins, filters
^{99}Tc	Acid digestion - Extraction with TnOA/Xylene	LSC/ ICP-MS	*under development*
^{129}I	Acid digestion - Ion exchange on solid AgI	γ-spectro / ICP-MS	MOX fuels
^{129}I	Acid digestion - Ion exchange on solid AgI	NA	*under development*
^{135}Cs	Acid digestion - Extraction with Na-TPB/Amylacetate - Backextraction with HNO_3	TIMS /ICP-MS	Concentrates, resins, filters
^{234,235}U ^{236,238}U	Acid digestion - Anion exchange	TIMS	Concentrates, resins, filters
^{239,240}Pu	Acid digestion - Anion exchange	TIMS	Concentrates, resins, filters
^{237}Np	Acid digestion - solid phase liquid extraction on Spec® Columns	α-spectrometry	Concentrates, resins, filters
^{241}Am	Acid digestion - solid phase liquid extraction on Spec® Columns	α-spectrometry	Concentrates, resins, filters
^{242,243}Cm	Acid digestion - solid phase liquid extraction on Spec® Columns	α-spectrometry	Concentrates, resins, filters

France: *CEA/DCC/DESD/SCCD/LARC*

Nuclides	Chemical preparation	Measurement	Application
^{3}**H**	Acid digestion - Distillation	LSC	Bitumen waste, graphite
^{14}**C**	Acid-oxydant digestion - Distillation	LSC	Cemented waste, resins, sludge
^{36}**Cl**	Acid digestion or combustion or pyrohydrolysis – Precipitation – ion exchange	LSC	Resins, graphite
^{59}**Ni**	Acid digestion - Extraction with DMG/$CHCl_3$	LSC	Cemented waste, resins, sludge
^{63}**Ni**	Acid digestion - Extraction with DMG/$CHCl_3$	LSC	Cemented waste, graphite, resins, sludge, hulls
^{90}**Sr**	Acid digestion - Extraction with crown ether	LSC	Cemented waste, graphite, resins, sludge, hulls
^{93}**Zr**	Acid digestion - Chromatography on TBP column	ICP-MS	Resins, water filters
^{93m}**Nb**	Acid digestion - Extraction with TBP	LSC	Cemented waste, graphite, resins, sludge, hulls
^{93}**Mo**	Acid digestion – Extraction with ABO - Precipitation	XRS	Resins
^{94}**Nb**	Acid digestion - Extraction with TBP	γ spectro	Cemented waste, graphite, resins, sludge, hulls
^{99}**Tc**	Acid digestion - Chromatography with TEVA-Spec	ICP-MS	Resins
^{99}**Tc**	Pyrohydrolysis	ICP-MS	Water filters
^{107}**Pd**	Acid digestion - Extraction with triphenyl phosphine	ETV/ICP-MS	Resins, water filters, graphite
^{129}**I**	Pyrohydrolysis	ICP-MS	Water filters
^{135}**Cs**	Soxhlet extraction or acid digestion - Chromatography with anionic resin	ETV/ICP-MS	Resins, water filters
^{151}**Sm**	Acid digestion - Precipitation with Fe^{3+} - Chromatography with RE-Spec and then with αHIBA	LSC	Graphite
^{226}**Ra**	Acid digestion – Co-precipitation with $BaSO_4$ - Dissolution with EDTA-NH_4OH	LSC (^{222}Rn)	Cemented waste, graphite, liquid waste, resins, sludge, hulls
^{237}**Np**	Acid digestion - Extraction with TTA/xylene	ICP-MS	Uranium containing waste
$^{238,\ 239+240}$**Pu**	Acid digestion - Chromatography on anionic resins - Electrodeposition	α spectro	Cemented waste, graphite, liquid waste, resins, sludge, hulls
^{241}**Pu**	Acid digestion - Chromatography on anionic resins - Electrodeposition - dissolution of the deposit	LSC	Cemented waste, graphite, liquid waste, resins, sludge, hulls
^{241}**Am**	Acid digestion - Chromatography on anionic resins - Electrodeposition	α spectro	Cemented waste, graphite, liquid waste, resins, sludge, hulls
$^{242,\ 243+244}$**Cm**	Acid digestion - Chromatography on anionic resins - Electrodeposition	α spectro	Cemented waste, graphite, liquid waste, resins, sludge, hulls

Germany: *FZ-Jülich*

Nuclides	Chemical preparation[1]	Measurement	Application
^{3}**H**	Combustion, off-gas trapping, distillation	LSC	Graphite, metalls, resins
^{14}**C**	Combustion, off-gas trapping, precipitation as $BaCO_3$	LSC	Graphite, metalls, resins
^{36}**Cl**	Combustion, off-gas trapping, ion exchange	LSC	Graphite, metalls, resins
^{41}**Ca**	Combustion, acid dissolution of combustion residues, ion exchange	X-ray	Graphite
^{55}**Fe**	Combustion, acid dissolution of combustion residues, ion exchange	X-ray	Graphite
^{63}**Ni**	Combustion, acid dissolution of combustion residues, HPLC, Liquid-Liquid extraction with Cyanex 301	LSC	Graphite, resins
^{63}**Ni**	Combustion, acid dissolution of combustion residues, HPLC, Liquid-Liquid extraction with Cyanex 301	LSC	Metalls
^{90}**Sr**	Combustion, acid dissolution of combustion residues, HPLC	LSC	Graphite, resins
^{90}**Sr**	Microwave digestion, solid phase extraction with crown ether	LSC	Slag, ash, cemented waste
^{93}**Zr**	Under development	ICP-MS	
^{99}**Tc**	Solid phase extraction with TEVA-Resin®	LSC	Liquid samples, cemented waste, sediments
^{236}**Ra**	Under development	LSC	Liquid samples, cemented waste, sediments
^{210}**Pb**	Under development	LSC	Liquid samples, cemented waste, sediments
^{234,235,236,238}**U**	Combustion, acid dissolution of combustion residues, ion exchange	α-spectro	Graphite
^{234,235,236,238}**U**	Microwave acid digestion, liquid-liquid extraction with TOPO [2]	α-spectro	Cemented waste, slag, ash, resins, crud, metalls
^{237}**Np**	Microwave acid digestion, liquid-liquid extraction with TTA [2]	α-spectro	Cemented waste, slag, ash, resins, crud, metalls
$^{238,239+240}$**Pu**	Combustion, acid dissolution of combustion residues, ion exchange	α-spectro	Graphite
$^{238,239+240}$**Pu**	Microwave acid digestion, liquid-liquid extraction with TOPO [2]	α-spectro	Cemented waste, slag, ash, resins, crud, metalls
^{241}**Pu**	Microwave acid digestion, liquid-liquid extraction with TOPO [2]	LSC	Cemented waste, slag, ash, resins, crud, metalls
^{241}**Am**	Microwave acid digestion, liquid-liquid extraction with CMPO [2]	α-spectro	Cemented waste, slag, ash, resins, crud, metalls
^{242,244}**Cm**	Microwave acid digestion, liquid-liquid extraction with CMPO [2]	α-spectro	Cemented waste, slag, ash, resins, crud, metalls

[1] Standard methods, to be modified due to sample properties

[2] Automated separation methods based on solid phase extraction under development

Germany: *TUM/RCM*

Nuclides	Chemical preparation	Measurement	Application
^{3}H	Combustion and oxidation in an oven; distillation	LSC	Resins, alloys, evaporator concentrates, cement samples
^{14}C	Combustion and oxidation in an oven; treatment with acids. CO_2 absorption in NaOH, precipitation as $BaCO_3$.	LSC	Resins, alloys, evaporator concentrates, cement samples
^{129}I	Combustion and oxidation in an oven, absorption in NaOH (solid samples, metals, alloys). Extraction, precipitation.	NAA	Resins, alloys, evaporator concentrates, cement samples
^{55}Fe	Thermal decomposition, liquid decomposition, oxidation. Addition of Fe carrier. In HCl medium, ketone extraction.	LSC	Resins, alloys, evaporator concentrates, cement samples
^{63}Ni	Thermal decomposition, liquid decomposition, oxidation. Addition of Ni carrier. Precipitation as Ni dimethyl glyoxime.	LSC	Resins, alloys, evaporator concentrates, cement samples
^{90}Sr	Thermal decomposition, liquid decomposition, oxidation. Addition of Sr carrier. Precipitation as $SrSO_4$.	LSC	Resins, alloys, evaporator concentrates, cement samples
$^{108m/110m}$Ag	Thermal decomposition, liquid decomposition, oxidation. Addition of Ag carrier. Precipitation as AgCl. Redox purification	γ spectro	Resins, alloys, evaporator concentrates, cement samples
^{144}Ce	Thermal decomposition, liquid decomposition, oxidation. Addition of Ce carrier and ^{141}Ce as tracer. Separation from ^{60}Co by ionic chromatography. Precipitation as Ce-fluoride.	γ spectro	Resins, alloys, evaporator concentrates, cement samples
^{36}Cl	Acid treatment. Addition of Cl^- as carrier. Ion chromatographic separation from Purification by precipitation as AgCl.	LSC	Resins, alloys, evaporator concentrates, cement samples
^{79}Se	Thermal, acid decomposition. Addition of Se(IV) as carrier. Reduction, cleaning procedures as dissolution, distillation.	AMS	Resins, alloys, evaporator concentrates, cement samples
^{94}Nb	Decomposition by thermal treatment, acid treatment. Addition of Nb as carrier. Dissolution in HF, extraction procedures.	γS	Resins, alloys, evaporator concentrates, cement samples
^{99}Tc	Thermal decomposition, liquid decomposition, oxidation. Addition of ^{99m}Tc as tracer. Ion chromatographic separation. Purification by precipitation. Isolation by selective resins.	LSC	Resins, alloys, evaporator concentrates, cement samples
^{125}Sb	Decomposition by thermal treatment, acid treatment. Addition of Sb as carrier. Dissolution in HF, extraction procedures.	γ spectro	Resins, alloys, evaporator concentrates
Actinides	Thermal decomposition, liquid decomposition, oxidation. Addition of tracer nuclides (^{232}U, ^{236}Pu, ^{243}Am). Separation of U, Pu by TBP extraction. Re-extraction, ion chromatographic cleaning procedures, preparation of the measurement sample by electrodeposition. Isolation of Am, Cm with CMPO resin (Eichrom) and electrodeposition.	^{232}Th by NAA α spectro	Resins, alloys, evaporator concentrates, cement samples

Italy: *ENEA-RAD-LAB*

Nuclides	Chemical preparation	Measurement	Application
^{3}H	Acid digestion / Bi-distillation	LSC	Cements, RTW, Liquids
^{14}C	Acid oxidant digestion / Alkaline off-gas trapping	LSC	Cements, RTW, Liquids
^{36}Cl	Acid oxidant digestion / Off-gas reduction trapping /LSC *(u. q.)* Acid digestion / HPIC-LSC	LSC HPIC-LSC	RTW Cements, liquids
^{55}Fe	Acid digestion	XRS / HPIC-LSC	Cements, RTW, Liquids
^{59}Ni	Acid digestion / Extraction with D.M.G. Acid digestion	XRS HPIC-LSC	Cements, RTW, Liquids
^{54}Mn	Acid digestion	HPIC-LSC	Cements
^{63}Ni	Acid digestion / Extraction with D.M.G. Acid digestion	LSC HPIC-LSC	Cements, RTW, Liquids
^{90}Sr	Acid digestion / Extraction on Sr-Spec columns / stripping Acid digestion	LSC HPIC-LSC	Cements, RTW, Liquids Cements, Liquids
^{99}Tc	Acid digestion / Extraction on TEVA Spec columns / Stripping Acid digestion	LSC HPIC-LSC	Cements, RTW, Liquids
^{129}I	Acid digestion / Absorbtion on solid Ag-zeolites	γ-spectro	Ventilation filters
$^{134/137}$Cs	Acidic digestion / Extraction with NaTPB	γ-spectro HPIC-LSC	Cements, RTW, Liquids
^{135}Cs	Acid digestion - Extraction with Na-TPB / Stripping HNO_3 *(under qualification)*	TIMS	Cements, RTW, Liquids
U **Isot. com.**	Acid digestion / Extraction TBP/ Stripping / NaF Acid digestion / Extraction hexone / Stripping Isotopic dilution / Anion extraction / Stripping Anion extraction Stripping Davies & Gray	Optical Fluor. UV-VIS/ICP- OES /MS TIMS TIMS Potentiometry	Cements, RTW, Liquids RTW fuel fabrication Fuel, MOX Solutions Solutions
^{232,233}U	Acid digestion / Extraction hexone / Cupel	α-spectro	Cements, RTW, Liquids
Pu α and 241	Acid digestion / Extraction TTA / Cupel	α-spectro/LSC	Cements, RTW, Liquids
Pu isot. com.	Acid digestion - Anion exchange	T.I.M.S.	Cements, RTW, Liquids
^{237}Np	Acid digestion / Extraction	α-spectro /LSC	Cements, RTW, Liquids
^{241}Am	Acid digestion - pH controlled extraction TTA / Cupel	α-spectro	Cements, RTW, Liquids
^{242,244}Cm	Acid digestion / pH controlled extraction TTA / Cupel	α-spectro	Cements, RTW, Liquids
all γ - emitting	Depending of sample: dissolution	γ-spectro	
all α - emitting	Depending of sample: dissolution and cupel deposition	α-spectro	

u. q. = under qualification: method not routinely applied.
Nuclides under development: ^{93m}Nb, ^{94}Nb, ^{151}Sm and Lanthanides.

Spain: *CIEMAT*

Nuclides	Chemical preparation	Measurement	Application
^{3}H	Separation in a combustion oven	LSC	Evaporator concentrates, resins, graphite, deposit of corrosion products on nuclear steam-generator tubing, metals, resins or concentrates immobilised in cement
^{14}C	Separation in a combustion oven	LSC	
^{45}Ca	Separation by selective precipitations	LSC	Evaporator concentrates, resins, graphite, resins or concentrates immobilised in cement
^{55}Fe	Precipitation of ferric ions as hydroxides by ammonia and dissolution in acid medium	LSC	Evaporator concentrates, resins, graphite, deposit of corrosion products on nuclear steam-generator tubing, metals, resins or concentrates immobilised in cement
^{59}Ni **^{63}Ni**	Precipitation with dimethylglyoxime - Liquid-liquid extraction and re-extraction in acid medium	X-ray spectro LSC	
^{89}Sr **^{90}Sr**	Selective extraction on a Sr-Spec (Eichrom) chromatographic column	LSC	
^{93m}Nb **^{94}Nb**	Selective extraction by TBP in sulphuric medium	X-ray spectro γ-spectro	
^{99}Tc	Selective liquid-liquid extraction by a crown-ether in a toluene-acetone mixture	LSC	Evaporator concentrates, resins
^{129}I	Co-precipitation of interferences with a mixture of oxides and chloroplatinic acid	X-ray spectro	
^{238}Pu **$^{239/40}$Pu**	Separation using anion exchange chromatography. Electrodeposition	α-spectro	Evaporator concentrates, resins, graphite, deposit of corrosion products on nuclear steam-generator tubing, metals, resins or concentrates immobilised in cement
^{241}Pu	Separation using anion exchange chromatography	LSC	
^{241}Am **^{242}Cm, ^{244}Cm**	Separation using anion exchange chromatography, liquid-liquid extraction and electrodeposition	α-spectro	
Gamma Emitters	The gamma emitters with high energy are directly measured by gamma spectrometry.	γ-spectro	

Spain: *ENRESA*

Nuclides	**Chemical preparation**	**Measurement**	**Application**
^{3}H	Acid digestion – Distillation / Catalytical Combustion	LSC	Concrete, resins, liquids, concentrates
^{14}C	Acid digestion – CO_2 collect / Catalytical Combustion	LSC	Concrete, resins, liquids, concentrates
^{54}Mn	Acid digestion	γ -spectro	Concrete, resins, liquids, concentrates
^{55}Fe	Acid digestion – Chromatography on anionic resin	LSC	Concrete, resins, liquids, concentrates
^{60}Co	Acid digestion	γ -spectro	Concrete, resins, liquids, concentrates
^{63}Ni	Acid digestion – Precipitation with DMG	LSC	Concrete, resins, liquids, concentrates
^{90}Sr	Acid digestion – Extraction with TBP/HNO_3	LSC	Concrete, resins, liquids, concentrates
^{99}Tc	Acid digestion – Chromatography with TEVA.Spec	LSC	*Under development*
^{129}I	Acid digestion – Extraction with Toluene	LSC	Concrete, resins, liquids, concentrates
^{137}Cs	Acid digestion	γ -spectro	Concrete, resins, liquids, concentrates
^{238}Pu	Acid digestion – Chromatography on anionic resin - Electrodeposition	α-spectro	Concrete, resins, liquids, concentrates
$^{239/240}$Pu	Acid digestion – Chromatography on anionic resin - Electrodeposition	α-spectro	Concrete, resins, liquids, concentrates
^{241}Am	Acid digestion – Chromatography on anionic resin / TRU.Spec- Electrodeposition	α-spectro	Concrete, resins, liquids, concentrates
^{242}Cm	Acid digestion – Chromatography on anionic resin / TRU.Spec- Electrodeposition	α-spectro	Concrete, resins, liquids, concentrates
^{244}Cm	Acid digestion – Chromatography on anionic resin / TRU.Spec- Electrodeposition	α-spectro	Concrete, resins, liquids, concentrates
^{241}Pu	Acid digestion – Chromatography on anionic resin	LSC	Under development
^{94}Nb	Acid digestion - Extraction with TBP/H_2SO_4	γ -spectro	Concrete, resins, liquids, concentrates
Other gamma emmitters	Acid digestion	γ -spectro	Concrete, resins, liquids, concentrates

The Netherlands: *NRG*

Nuclides	Chemical preparation	Measurement	Application
^{3}H	Distillation and/or enrichment	LSC	Liquid waste, resins, water
^{14}C	Acid oxidation/digestion - Amine absorption	LSC	Under development
^{89}Sr/^{90}Sr	Acid digestion/^{90}Y precipitation	LL β-spectro	Liquid waste, solid waste, filters, resins, crud
^{55}Fe	Precipitation of ferric ions	LSC	Liquid waste, resins, water
^{63}Ni	Nickel-dimethylglycomine complexation	LSC	Liquid waste, resins, water
^{99}Tc	Acid digestion/TIO-xyleen	LL β-spectro	Liquid waste, resins
^{129}I	Non-destructive	γ-spectro	Under development
^{131}I	Non-destructive	γ-spectro	Active carbon
$^{238,239+240}$Pu	Acid digestion/Chromatography by TRU-resin/ Electrodeposition	α-spectro	Biological samples, liquid waste, water, filters, soil, sediment, solid waste, resins, crud
^{241}Pu	Acid digestion/Chromatography by TRU-resin	LSC	Biological samples, liquid waste, water, filters, soil, sediment, solid waste, resins, crud
^{241}Am	Acid digestion/Chromatography by TRU-resin/ Electrodeposition	α-spectro	Biological samples, liquid waste, water, filters, soil, sediment, solid waste, resins, crud
$^{242,243+244}$Cm	Acid digestion/Chromatography by TRU-resin/ Electrodeposition	α-spectro	Biological samples, liquid waste, water, filters, soil, sediment, solid waste, resins, crud
U	Evaporation/NaF-melt	Fluorimetry	Liquid waste, water
^{234,235,238}U	Acid digestion/Chromatography by TRU-resin/ Electrodeposition	α-spectro	Biological samples, liquid waste, water, filters, soil, sediment, solid waste, resins, crud
^{238}U	Non-destructive/By ^{234}Th	α-spectro	NORM waste, LLW, sediments, soil, liquids
^{228,230,232}Th	Acid digestion/Chromatography by TRU-resin/ Electrodeposition	α-spectro	Biological samples, liquid waste, water, filters, soil, sediment, solid waste, resins, crud
228Th	Non-destructive/By ^{208}Tl	γ-spectro	NORM waste, LLW, sediments, soil, liquids
234Th	Non-destructive/Transmission correction	γ-spectro	NORM waste, LLW, sediments, soil, liquids
^{210}Pb	1. Acid digestion/Renewed ingrowth of ^{210}Po after total depletion 2. Acid digestion/Ion exchange chromato-graphy /Leadchromate precipitation 3. Non-destructive/Transmission correction	α-spectro LL β-spectro γ-spectro	NORM waste, LLW, sediments, soil, liquids
^{210}Po	Acid digestion/Electrochemical deposition	α-spectro	NORM waste, LLW, sediments, soil, liquids
^{228}Ra	Non-destructive/By ^{228}Ac	γ-spectro	NORM waste, LLW, sediments, soil, liquids
^{226}Ra	Non-destructive/By ^{214}Pb and ^{214}Bi	γ-spectro	NORM waste, LLW, sediments, soil, liquids
Other γ-emitters	Non-destructive	γ-spectro	Liquid waste, solid waste, NORM waste, sediments, soil, liquids, filters

The Netherlands: *COVRA*

Nuclides	Chemical preparation	Measurement	Application
^{3}H	Distillation of the water sample and enrichment of tritium	LSC	Waste water and reactor water
^{14}C	Acid oxidation/digestion – trapping Carbosorb	LSC	Concentrates, filters
^{35}S	Non-destructive	LSC	Waste water
^{36}Cl	Non-destructive	LSC	Waste water
^{65}Zn	Non-destructive	γ-spectro	Waste water
$^{89}Sr/^{90}Sr$	Acid digestion/^{90}Y precipitation	Low level beta counters	Different matrices, dust collected on silicium or teflon filters
^{125}I	Non-destructive	γ-spectro	Active carbon
$^{134+137}Cs$	Non-destructive	γ-spectro	Different matrices

United Kingdom: *NNC Ltd*

Nuclide	Chemical Preparation	Measurement	Application
^{3}H	Acid digestion - Alkaline distillation	LSC	LLW, resin
^{14}C	Acid oxidation/digestion - Amine absorption	LSC	LLW, resin, graphite, cement
^{35}S	Acid digestion - Cation exchange chromatography - Precipitation as $Ba^{35}SO_4$	LSC	LLW, resin
^{45}Ca	Acid digestion - Oxalate precipitation - Fuming nitric acid separation	LSC	LLW, resin, sediment, soil
^{55}Fe	Acid digestion - Extraction with Di-isopropyl ether	γ- Spectro	LLW, resin
^{90}Sr	Acid digestion - Oxalate precipitation - Fuming nitric acid separation - ^{90}Y grow-in	LSC	LLW, resin, sediment, soil
^{99}Tc	Acid digestion - Extraction with TnOA/xylene	LSC	LLW, resin, sediment, soil
$^{125, 129}$I	Alkaline digestion - Redox adjustment - Anion exchange chromatography	γ- Spectro	LLW
$^{228, 230, 232}$Th	Acid digestion - Anion exchange chromatography - Electrodeposition	α- Spectro	LLW, resin, sediment, soil
$^{234, 235, 238}$U	Acid digestion - Anion exchange chromatography - Electrodeposition	α- Spectro	LLW, resin, sediment, soil
$^{238, 239 + 240}$Pu	Acid digestion - Anion exchange chromatography - Electrodeposition	α- Spectro	LLW, resin, sediment, soil
^{241}Pu	Acid digestion - Anion exchange chromatography - Electrodeposition - Dissolution of deposit	α- Spectro LSC	LLW, resin, sediment, soil
^{241}Am	Acid digestion - Anion exchange chromatography - Electrodeposition	α- Spectro	LLW, resin, sediment, soil
$^{242, 243 + 244}$Cm	Acid digestion - Anion exchange chromatography - Electrodeposition	α- Spectro	LLW, resin, sediment, soil

GLOSSARY

AAS: Atomic Absorbance Spectrometry

αS: Alpha spectrometry

AhibA: α-hydroxyisobutyric acid

ANDRA: Agence Nationale pour la Gestion des Déchets Radioactifs :
French National Agency for the Management of Radioactive Wastes

AMS: Accelerator Mass Spectrometry

BfS: Bundesamt für Strahlenschutz
Federal Office for Radiation Protection (Germany)

BNFL: British Nuclear Fuels plc

CEA: Commissariat à l'Energie Atomique
French Atomic Commission

CIEMAT: Centro de Investigaciones Energéticas, Medioambientales y Tecnológicas.
Spanish Energy, Environmental and Technological Research Centre

COGEMA: Compagnie Genérale des Matières nucléaires
French General Company for nuclear materials

Conditioning of waste: those operations that transform waste into a form suitable for transport and/or storage and/or disposal. The operations may include converting the waste to another form, enclosing the waste in containers, and providing additional packaging.

COVRA: Centrale Organisatie Voor Radiactief Afval
Central Organisation for Radioactive Waste Management (The Netherlands)

Criteria: principles of standards on which a decision or judgement can be based. They may be qualitative or quantitative. Acceptance criteria are set by a regulatory authority.

CSN: Consejo de Seguridad Nuclear
Nuclear Security Authority

DA: Destructive Analysis.

DT: Destructive Technique

DESD: Département d'Entreposage et de Stockage des Déchets
Department of Radioactive Waste Storage and Disposal

Detection of the nuclides present in the item assayed:
after a sample preparation procedure that modifies the physical and chemical properties of the material

DFN: Departamento de Fisión Nuclear
Nuclear Fission Department

DMG: Dimethylglyoxime complex

DOFS: Dispersive Optical Fluorescence Spectrophotometry

DTA: Differential Thermal Analysis

EA: Environment Agency

EC: European Commission

EDF: Electricité De France
French Nuclear Power Producer

EDTA: ethylenediaminetetraacetic acid

ENEA: Ente per le Nuove Tecnologie l' Energia e l' Ambiente
Italian Organisation for Energy, Environment and New Technologies

ENRESA: Empresa Nacional de Residuos, S.A.
Radioactive Wastes National Company, S.A.

ENSA: Equipos Nucleares, S.A.
Nuclear Equipments, S.A.

ENUSA: Empresa Nacional del Uranio, S.A.
Uranium National Company, S.A.

EUREX (plant): Enriched URanium EXtraction
Italian reprocessing plant operated by ENEA

FAAS: Furnace Atomic Absorption Spectrophotometry

Final disposal: ultimate repository of wastes for long term isolation from man’s environment.

FRM: Forschungsreaktur München, Garching

FZJ: Forschungszentrum Jülich
Research Centre Jülich, Germany

γS: Gamma spectrometry

Ge-HP: High Pure Germanium detector

Ge-Li: Germanium - Lithium detector

Handling Cell: Air - tight, shielded enclosure made of concrete and lined inside with stainless steel.

HLW: High Level Waste. **HLLW**: High Level Liquid Waste.

I. The highly radioactive liquid, containing mainly fission products, as well as some actinides, which is separated during chemical reprocessing of irradiated fuel (aqueous waste from the first solvent extraction cycle and those waste streams combined with it)

II. Spent reactor fuel, if it is declared waste

III. Any other waste with a radioactivity level comparable to i. or ii.

HPIC: High Performance Ion Chromatography

HPLC: High Performance Liquid Chromatography

HRGC-MS: High Resolution Gas Chromatography-Mass Spectrometry

IC-LSC: Ion Chromatography coupled to Liquid Scintillation Counting

ICP-AES: Inductively Coupled Plasma-Atomic Emission Spectrometry

ICP-MS: Inductively Coupled Plasma-Mass Spectrometry

ICP-OES: Inductively Coupled Plasma-Optical Emission Spectrophotometry

ILW: Intermediate Level Waste

ISR: Institut für Sicherheitsforschung und Reaktortechnik
Institute for Safety Research and Reactor Technology in Germany

LAINSA: Limpiezas y Acondicionamientos Industriales S.A.
Industrials Conditioning and Cleaning, S.A.

LARC: Laboratoire d'Analyses Radiochimiques et Chimiques
Laboratory of Radiochemical and Chemical Analyses (CEA/DESD)

LL βC: low level beta counting

LLW: Low Level Waste

Long term: in waste management, refers to periods of time which exceed the time during which administrative controls can be expected to last.

LSC: Liquid Scintillation Counting

LVCR (El Cabril): Laboratorio de Verificación de la Calidad del Residuo
Waste Verification Laboratory

MLW: Medium Level Waste

NAA: Neutron Activation Analysis

NAGRA: Nationale Genossenschaft fuer die Lagerung Radioaktiver Abfaelle
National companionship for storage of radioactive wastes in Switzerland

Na-TPB: Sodium Tetraphenylborate

NDA: Non Destructive Analysis. **NDT**: Non Destructive Technique.
Observation of spontaneous or stimulated nuclear radiations, interpreted to estimate the content of one or more nuclides of interest in the item assayed, without affecting the physical or chemical form of the material

NIRAS/ONDRAF: Nationale Instelling voor Radioactief Afval en verrijkte Splijtstoffen/Organisme National des Déchets RAdioactifs et des matières Fissiles enrichies.
Belgian National Institute for Radioactive Waste and enriched Fissiles

NNC: English National Nuclear Corporation

NPI: non-permitted item

NRG: Nuclear **R**esearch and consultancy **G**roup in The Netherlands

PKS: Produktkontrollstelle des Bundesamt für Strahlenschutz
German Quality Control Group for Radioactive Waste

PWR: Pressure Water Reactor

QA: Quality Assurance. Planned and systematic actions aimed at providing adequate confidence that an item facility or person will perform satisfactorily in service

Quality control: actions which provide a means to control and measure the characteristics of an item, process, facility or person in accordance with quality assurance requirements

Radioactive Waste Characterisation:
determination of physical, chemical, biological, mechanical and radiological properties of the radioactive waste, in order to stabilise its successive treatment and conditioning. Characterisation is performed in every step of radioactive waste management

Radioactive waste management: all activities, administrative and operational, that are involved in the handling, treatment, conditioning, transportation, storage and disposal of waste

Radioactive waste: any material that contains or is contaminated with radionuclides at concentrations or radioactivity levels greater than the "exempt quantities" established by the competent authorities and for which no use is foreseen

RAD-LAB: Unità Rifiuti Radioattivi - Laboratorio Nazionale per la Caratterizzazione dei Rifiuti Radioattivi
Radioactive waste unit - National Laboratory for Radioactive Waste Characterisation

R&D: Research and Development

Regulatory authority:
an authority or systems of authorities designated by the Government of a Member State as having the legal authority for conducting the licensing process, for issuing of licenses and thereby for regulating the siting, design, construction, commissioning, operation, shutdown, decommissioning and subsequent control of nuclear facilities (e.g. waste repositories) or specific aspects thereof. This authority could be a body (existing or to be established) in the field of nuclear-related health and safety or mining safety or environmental protection, vested with such legal or environmental protection, vested with such legal authority, or it could be the Government or a department of the Government, or it could be an international agency

Requirement:
a condition defined as necessary to meet product, material, or process criteria

RE-Spec: commercial extraction chromatographic material for the analysis of Rare Earth Metals

RTW: Raw Technological Waste

SCK•CEN: StudieCentrum voor Kernenergie•Centre d'Étude de l'Énergie Nucléaire
Belgian Nuclear Chemistry and Services. (Nuclear Research Centre)

SEM-EDXA: Scanning Electron Microscope-Energy Dispersive X-ray Analysis

SGS: Segmented Gamma Scanner

SIMS: Secondary Ion Mass Spectrometry

Sr-Spec: commercial extraction chromatographic material for the analysis of Strontium

Storage: the placement of waste in a facility with the intent that it will be retrieved at a later time

TBP: Tributylphosphate

TECNATOM: Engineering Company

TEVA-Spec: commercial extraction chromatographic material for the analysis of Technetium, Uranium and tetravalent Actinides

TG: Thermal Gravimetry

TIMS: Thermal Ionisation Mass Spectrometry

TnOA: Tri-n-Octylamine

TTA: 2-thenoyltrifluoroacetone

TUM/RCM: Technischen Universität München/Institut für Radiochemie
Technical University of Munich

TÜV: Technischer Überwachungsverein (TÜV Süddeutschland)
Bavarian technical inspection company (Germany)

Waste acceptance criteria: those criteria relevant to the acceptance of waste for storage, transport and disposal

Waste form: the physical and chemical form of the waste (e.g. liquid, in concrete, in glass, etc.) without its packaging

Waste generator: the responsible organisation for the facility where the waste is generated

Waste package: the waste form and any container as prepared for handling, transport, treatment, conditioning, transportation, storage and disposal of radioactive waste. A cask or overpack may be a permanent part of the waste package or it may be re-usable for any waste management step. The waste package may vary for the different steps in waste management

Waste stream: quasi continuous flow of wastes contained in a drum, package, container, box, etc

WQCL: British Waste Quality Checking Laboratory
XRS: X-ray spectrometry

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