

CHARACTERIZATION OF LOW AND MEDIUM ACTIVE WASTES

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Introduction

The characterization of a material or an object as waste, a matrix, a coating or a package, consists of determining its specific properties.

In the characterization of nuclear waste, we restrict ourselves to the acquisition of characteristics which have to be known in order to evaluate safety, and temporary and permanent storage conditions.

1. Objectives of Waste Characterization and Characterization Types

Five main objectives are looked for when performing a characterization test. The tests are intended to:

- guarantee the mechanical properties of the coated waste and the matrix,
- test the durability of the physical and mechanical properties of the waste for the storage duration,
- evaluate the behavior of isotopes in the waste and the long-term waste-matrix interaction,
- calculate the loss rate by isotope leaching, and
- compare the results with specifications, and rules specified by their Authorities and Safety notices.

For FRANCE, this involves ANDRA specifications, the Basic Safety Rules (les Règles Fondamentales de Sûreté—RFS) of the Central Service (Ministry of Industry), Safety Instructions from the Nuclear Safety and Protection Institute (l'Institut de Protection et de sûreté Nucléaire—IPSN).

Several types of characterization are necessary to cover all knowledge useful for storage and repository of radioactive waste.

There are three main types of characterization:

- characterization of packages and materials,
- behavioral studies, and
- long-term behavioral studies.

1.1 Characterization of packages and materials

This type of characterization can be defined as the acquisition of the characteristics of a coating, a package or a material in its initial state; it is therefore the creation of a statement. This type of characterisation is carried out in three steps:

- characterization of the waste itself,
- pre-characterization of the material and the coating, and
- characterization of the finished product (the package intended for storage or repository).

1. 1. 1 Characterization of the waste

This is defined as acquisition of data necessary for :

- producing the waste package ,
- classification for its activity and its destination ;
 - Class FA-MA-HA ,
 - Class β , γ or α , and
 - destination ; surface repository , temporary storage before repository at depth ; and
- analysis of basic safety and its entry into the approval file.

1. 1. 2 Pre-characterization (process and coating)

This is the essential stage for the evaluation of the quality of a process and the coating resulting from all the pre-production steps (formulation, reference tests, terminal tests, sensitivity test, etc.).

Tests for this stage are carried out using reference materials.

Two types of objective are involved :

- a comparison of the various performances of the coating (choice of matrices, coating rate, formulation, etc.), and
- a demonstration that the coatings, the packaging, and the finished products satisfy standards and specifications in force.

This step normally results in the production of a packaging specification and the production of nominal specifications for coated waste.

1. 1. 3 Characterization of the finished product

This stage involves three types of investigation :

- examination of the intrinsic characteristics of packagings and coatings ,
- checking the durability of these characteristics (degradability, confinement, etc.), and
- defining the influence of variations of the various process parameters (sensitivity tests) in order to guarantee the quality of the coated material.

Characteristics and acceptance criteria for the finished product are examined within Chapter 2 of this document.

1. 2 Behavioral studies

This second section of the characterization of nuclear waste concerns the *source term* study. It is defined as being the acquisition of basic data for forecasting and modelling isotope emission in storage environments.

Two final results are sought in this type of characterization :

- understanding the phenomena involved in isotope emission , and
- the qualitative and quantitative expression of the basic values and ranges of values necessary for the storage safety analyses.

1. 3 Long-term behavior

This type of characterization is defined as the acquisition of data for mechanisms dealing with *slowly evolving* phenomena. Two approaches can be used :

- the behavior of natural counterparts , and

- the thermodynamic definition of slow phenomena.

The final result that is expected to be obtained from this last characterization stage consists of changing from "high bound" type modelling to a modelling system based on extrapolating variations of characteristic, making use of relevant measurements and bounding values.

2. Package Approval: Specifications, Procedures, and Acceptability Criteria

Inspection of the current situation in countries developing nuclear energy shows that most countries base approval of waste packages on three main data items:

- specifications produced by the authorities,
- procedures produced by approved laboratories and organizations, and
- acceptability criteria or quality criteria which should lead to the respect of specifications.

Although the organization of waste-related responsibilities and therefore the specifications issued by the various authorities vary between countries, all have established some characterization procedures and acceptability criteria for waste packages.

2.1 Procedures — Technical sheets

In France, approval of a waste package is announced after inspection of experimental results and tests in accordance with the minimum characterization program defined by ANDRA.

This minimum program affects the three selected activity levels and the two classes of waste packages (homogeneous-heterogeneous). Tests and checks are classified into four categories:

- physical characteristics,
- mechanical characteristics,
- confinement power, and
- stability-degradability (maintenance of confinement power).

In order to reply uniformly to the creation of tests required for the approval file, the Confinement Checking and Evaluation Office (Bureau d'Evaluation et de Contrôle des Confinements—BECC) has published a set of technical forms condensing a general method of executing these tests.

A list of currently available technical forms is given in Table 1.

SHEETS UNDER PREPARATION:

- Behavior on immersion in water,
- Exudation of water under compression,
- Homogeneity test of a coating,
- Non-destructive measurement of γ producers in real waste package, and
- Evaluation of the resistance of bitumen coatings to microorganism attack.

A block diagram has been produced by the Waste and Effluents Management for the approval procedure (Direction des Effluents et Déchets — Dg/ED). Figure 1 summarizes the global routing of an approval circuit and specifies the role of each participant for wastes which can be delivered to ANDRA on a surface site.

ANDRA approval of waste packages requires the production of the following documents:

- Description of the production process for the package containing the waste,

- Quality assurance program for the implementation of the package production processes as described in the process description, and
- Package characterization test report (technical characterization file).

The characterization test report assumes that the producer has previously submitted a draft characterization test program to ANDRA. When this draft has been accepted by ANDRA, it then becomes the *characterization procedure*, containing a description of the checks and tests to be carried out.

Therefore, the creation of a test requires the following definitions (see Figure 2):

- the sample or samples,
- the size and characteristics,
- the test procedure,
- the results to be provided, and
- the test report.

All reports for all tests described in the protocol must then be inspected and used to produce a *test summary report*, or a *characterization technical file* which is forwarded, as already stated, to ANDRA by the producer.

To illustrate the importance of this procedure, we will partially describe the procedure for measuring the initial activity of raw waste and coatings which meets ANDRA's requirement for the monitoring of the activity stored on the site in order to respect site storage capacity for each isotope.

Figure 3 shows ANDRA's mandatory considerations towards the safety authorities, and also the consequences of respecting mandatory quality requirements demanded by ANDRA for producers' packages.

Consequently, waste activity measurement (raw or coating) is one of the most important criteria. Characterization therefore requires the implementation of an adapted measurement scheme for each waste or coating type.

Each type of waste has its own time schedule for taking measurements and for monitoring the procedure so as to satisfy safety authority requirements.

The main procedure for coatings is shown in Figure 4. This Figure clearly shows the two recommended types of measurements:

- non-destructive measurements, and
- destructive measurements with the various related determinations.

Finally, to complete the execution of these measurements, Figure 5 defines the essential elements which must appear in the test report intended for the producer in order to prepare the summary report for all the tests, as required by the final management authority (ANDRA for FRANCE).

2.2 Package acceptability criteria

For FRANCE, characterization deals only with coating and package quality criteria. There are listed in the *minimum characterization program* defined by ANDRA as recalled in Section 2.1.

In view of the available characterization results for various matrices enclosing various types of waste, the BECC established a summary of coating and packaging qualities, as a function of the

main parameters.

Three types of parameters were used for this summary (Table 2):

- quality of the coating-quality of package (RFS+ANDRA specifications),
- implementation and feasibility, and
- control.

An evaluation was made for each type of parameter, matrix, and waste type (homogeneous-heterogeneous) based on the acquired characterization results.

Conclusions

For several years now, research on raw or packaged waste characterization has been carried out in France, in particular those aspects concerning:

- legislation,
- regulations,
- standardization of methods and techniques, and
- laboratory and test hall equipment.

The BECC provides the main communication link between the waste producer, ANDRA, and the CEA which can perform all services related to tests and corresponding measurements, either as part of its own research and development program, or on request by the BECC. The CEA resources, methods, laboratory, and test halls, which are available to the BECC and its customers, now form a characterization system adapted to each waste or packaging type to which a quality assurance program is applied.

BECC manufacturing experience enables it to give correct advice to the waste manufacturer and any nuclear material safety unit, both on the selection of packaging and test procedures, and on equipment and the selection of characterization materials or the organization to be set up. In particular, the BECC can provide the following services:

- qualitative or quantitative analysis of radionuclides present in already packaged waste (including badly packaged waste), or unpackaged waste; and, as far as possible, evaluation of the main physico-mechanical and confinement characteristics,
- technical assistance for the characterization of packages including:
 - developing specific technical test forms according to the customer,
 - establishing BECC type characterization coordination units and training corresponding personnel,
 - transferring basic elements for the implementation of a quality assurance program for package characterization, and
 - establishing basic characterization method and packaging expertise planning, and
- technical assistance with the design and construction of characterization laboratories.

The Figure 6 shows a summary of the main missions provided by the BECC from its experience.

To conclude, we would like to emphasize that the objective of the various units in charge of characterization is to supply technical assistance in view of the harmonious development of waste approval files and the development of test techniques to improve knowledge of the intrinsic characteristics of packaged waste. Above all, the framework in which these characterization activities are carried out must respect the special features of this characterization, which is at the

very interface of the various radioactive waste management partners; safety authorities, waste management (ANDRA), waste producers, and the managers of research and development programs.

Table 1 Technical sheets for FA-MA tests

01. PHYSICAL CHARACTERISTICS

<u>Form number</u>	<u>Title</u>
FT 01. 001	Measurement of the apparent density
FT 01. 002	Determination of the flash point of polymer-based coatings
FT 01. 003	Determination of open vessel fire points on bitumen-based coatings
FT 01. 004	Determination of the spontaneous ignition point of bitumen coatings
FT 01. 005	Exothermicity test on REI coating by a thermosetting resin

02. MECHANICAL CHARACTERISTICS

<u>Form number</u>	<u>Title</u>
FT 02. 003	Determination of the crushing strength
FT 02. 004	Determination of the shock resistance
FT 02. 006	Determination of a coating's fire resistance
FT 02. 010	Determination of the compressive strength (hydraulic binder)
FT 02. 011	Determination of the compressive strength (polymer)
FT 02. 031	Determination of the resistance to thermal effects

03. CHEMICAL CHARACTERISTICS

<u>Form number</u>	<u>Title</u>
FT 03. 001	Determination of the water content of a bitumen coating
FT 03. 002	Determination of the content of salts or REI of bitumen coating

04. CHARACTERISTICS RELATED TO CONFINEMENT POWER

<u>Form number</u>	<u>Title</u>
FT 04. 001	Determination of the porosity of a coating to water
FT 04. 002	Measurement of the permeability to water of material based on hydraulic and/or polymer binders
FT 04. 003	Measurement of the permeation of a gas through a bitumen membrane or a bitumen coating
FT 04. 004	Determination of the porosity to mercury of coatings, coating materials, and waste
FT 04. 011	Measurement of the permeability to gas (nitrogen, helium, etc.) for hydraulic binders
FT 04. 020	Determination of leaching resistance
FT 04. 021	Measurement of radionuclide transfer measurement in hydraulic binder based materials. Application to the confinement of heterogeneous wastes

05. RADIOACTIVE CHARACTERISTICS

<u>Form number</u>	<u>Title</u>
FT 05. 030	Effects of β , γ radiation

06. GENERAL METHODS

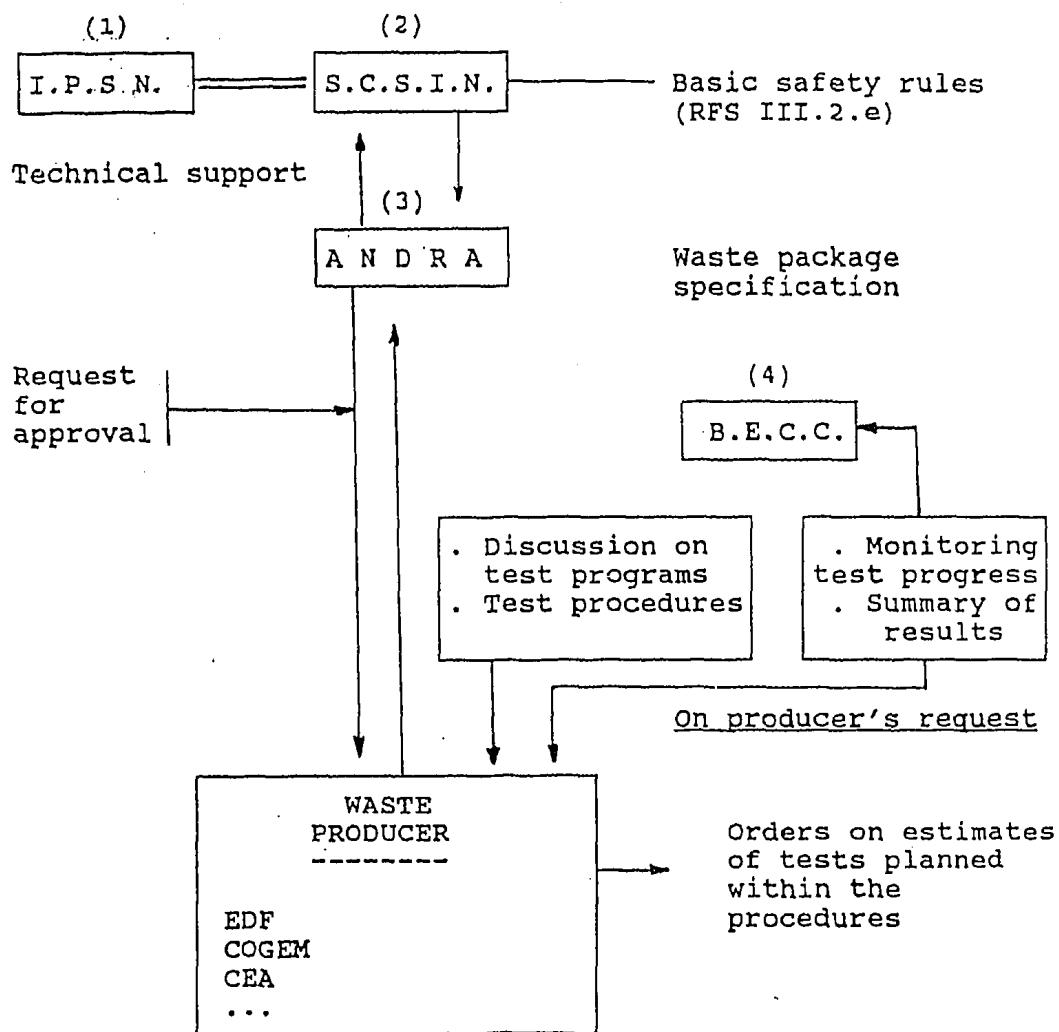
<u>Form number</u>	<u>Title</u>
FT 06. 001	Sample sizing
FT 06. 002	Expression and introduction of leaching test results
FT 06. 003	Determination of report on samples after leaching test
FT 06. 004	As polymers, raw or coated waste
FT 06. 005	Sample identification and monitoring for characterization

Table 2 List of parameters and criteria

1. QUALITY OF COATING - QUALITY OF PACKAGING (RFS-ANDRA SPECIFICATIONS)
 - 1.1 Physical characteristics
 - porosity,
 - permeability to water,
 - permeability to gas,
 - coating density,
 - shrinkage,
 - state change temperature,
 - homogeneity of coated or blocked waste,
 - homogeneity of coating,
 - percentage of contained water,
 - exudation of water in the waste under compression force,
 - solubility in water of the coating-overcoating material, and
 - filling rate.
 - 1.2 Mechanical characteristics
 - compression strength,
 - behavior of the packages under load, and
 - package shock resistance.
 - 1.3 Confinement power
 - 1.4 Stability - Degradability - Maintenance of confinement power
 - temperature cycle,
 - behavior under radiation,
 - sensitivity to contact with water,
 - packaging resistance to corrosion in a humid environment, and
 - package behavior under fire.
2. IMPLEMENTATION AND FEASIBILITY
 - 2.1 Industrial process
 - Type of industrial installation; fixed or mobile installation,
 - coating or blocking methods; continuous-discontinuous process, and

- installation capacity: high capacity $>50 \text{ m}^3/\text{year}$, low capacity or pilot $<50 \text{ m}^3/\text{year}$.
- 2. 2 Process capacity for improvement
 - Related to the process itself: operating parameter,
 - related to formulation: addition, and
 - related to package produced: package dimension.
- 2. 3 Additional packaging and safety
 - Inflammability of basic materials,
 - need for overcoating, and
 - special packaging.
- 3. CONTROL
 - 3. 1 Procurement of basic materials
 - 3. 2 Storage conditions of basic materials
 - 3. 3 Cost price of basic materials
 - 3. 4 Constraints related to use of the process
 - Energy consumption (8°), and
 - Constraints related to the number of parameters to be considered.
 - 3. 5 Storage - Handling
 - Core-polymerization time before delivery, and
 - Storage conditions on the facility.

Fig. 1 Characterization of Waste Packages for Delivery to ANDRA



- (1) IPSN ; Institut de Protection et de sûreté Nucléaire (CEA).
 (2) SCSIN ; Service Central de sûreté des Installations Nucléaires (Ministry of Industry).
 (3) ANDRA ; Agence nationale pour la Gestion des Déchets Radioactifs (National Agency for Radioactive Waste Management).
 (4) BECC ; Confinement checking and Evaluation Office (attached to the Service dealing with the characterization, confinement evaluation, and analysis at CEA).

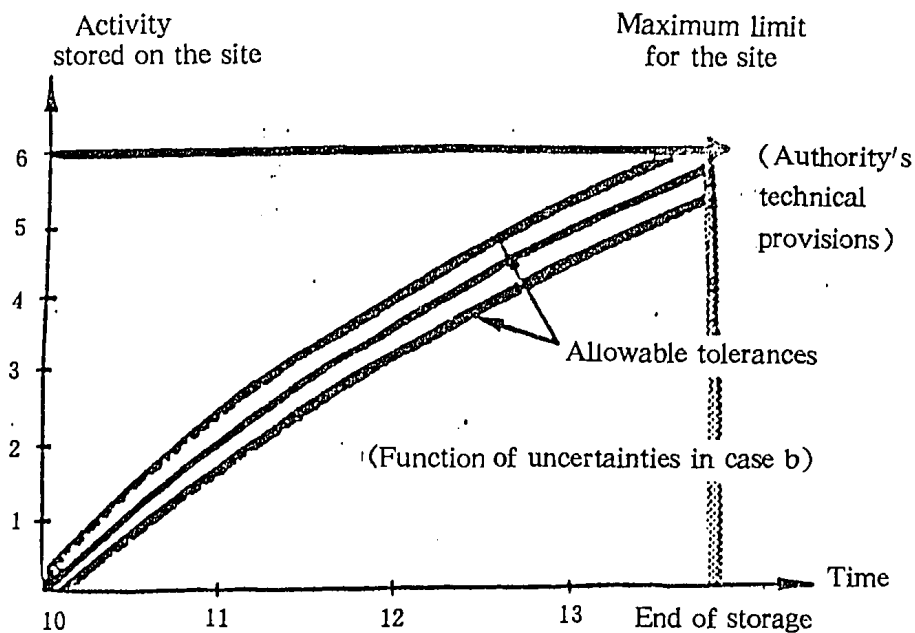
Fig. 2 The HE Characterization Procedure

CONTENTS

- MAIN PROCESS DATA (COATING - WASTE COMPOSITION - GENERAL PERFORMANCES),
- DESCRIPTION OF CHARACTERIZATION TESTS:
 - FOR THE COATING:
 - INACTIVE TESTS
 - LIST OF TESTS; NUMBER OF SAMPLES
 - DIMENSION
 - PREPARATION
 - TECHNICAL FORM REFERENCE
 - ACTIVE TESTS (DITTO)
 - * FOR THE MATRIX AND THE WASTE (POSSIBLY)
 - * FOR THE PACKAGE:
 - FILLING RATE
 - OTHER TESTS; FIRE - DROP - LEACHING - THERMAL CYCLE, etc.
- PRESENTATION OF RESULTS - TEST REPORTS
- EXECUTION SCHEDULE
- REFERENCES; PROCESS BOOKS
- ANDRA SPECIFICATIONS
- RFS

Fig. 3 Producer's and ANDRA's Objectives

Case a) Manager-Authority



Case b) Producer

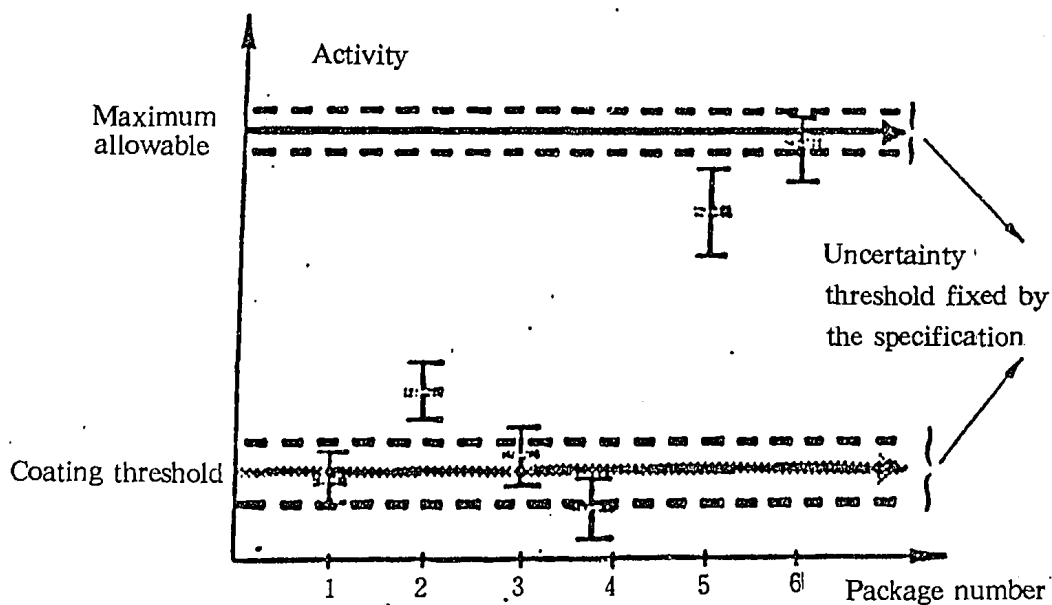


Fig. 4 Measurements of Coated Homogeneous Wastes

1. BITUMENS

OUTLINE

1) NON-DESTRUCTIVE MEASUREMENTS 

2) DESTRUCTIVE MEASUREMENTS

SALT EXTRACTION

BITUMEN PHASE ACTIVITY CHECK

$\beta\gamma$, α , β MEASUREMENTS ON SALTS

CHEMICAL MEASUREMENTS

2. POLYMERS

OUTLINE

1) NON-DESTRUCTIVE MEASUREMENTS 

2) DESTRUCTIVE MEASUREMENTS

→ MINERALIZATION - DISSOLUTION (HNO_3 - H_2O_2)

→ $\beta\gamma$, α , β MEASUREMENTS

→ SPECIAL CHEMICAL MEASUREMENTS (BORON, ETC.)

3. CEMENTS

OUTLINE

1) NON-DESTRUCTIVE MEASUREMENTS 

2) DESTRUCTIVE MEASUREMENTS

MINERALIZATION - DISSOLUTION (HNO_3 - HF)

$\beta\gamma$, α , β MEASUREMENTS

SPECIAL CHEMICAL MEASUREMENTS (BORON, Cl, ETC.)

Fig. 5 Report

THE LABORATORY WILL ESTABLISH AND PROVIDE THE FOLLOWING MINIMUM INFORMATION:

- SAMPLE IDENTIFICATION,
- MEASUREMENT DATE,
- DATE OF THE MOST RECENT INSTRUMENT CALIBRATION,
- RESULTS WITH UNCERTAINTIES,
- FOR DESTRUCTIVE MEASUREMENTS, THE PRINCIPLE OF THE METHOD AND THE RESULTS OF ALIQUOT CHECKS,
- NAME AND SIGNATURE OF THE PERSON RESPONSIBLE, AND
- MODIFICATIONS OF OPERATING METHODS IF A STANDARD EXISTS.

Fig. 6 Main Missions Provided by Contract as a Technical Assistance by the CEA-BECC

MISSION 1:

Procurements and supplies of technical information and sheets about typical wastes and coatings

MISSION 2:

Structure organizing for characterization and expertise planning. BECC equivalent creation.

MISSION 3:

Tests and measures by CEA laboratories of real embedded wastes

MISSION 4:

Technical training of scientific supervisory personnel for structure organizing and tests of characterizations carrying out

MISSION 5:

Setting a quality assurance programme of raw and embedded waste in conformity with each country