Destructive Testing: Dry Drilling Operations with *TruPro*<sup>®</sup> System to Collect Samples in a Powder Form, from a Hull Containing Sludge Immobilised in a Hydraulic Binder

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#### INTRODUCTION

Andra Low Level Waste (LLW) and Intermediate Level Waste (ILW) packages quality-control inspections contribute to the safe storage by following the specification requirements (acceptance of Radwaste packages: embedding threshold, acceptance limits, quantity of fissile materials, etc). One objective of these controls is also to improve continuously the Radwaste quality (e.g. to check the consistency between Andra measurements results and producer declared properties).

Andra has to control the Radwaste packages and to strengthen the acquisition of knowledge on radioactive waste and packages (e.g. long lived radionuclides scale factors used by producers) in order to assess and improve the mastery of the producer radwaste packages quality and to maintain the confidence of the public.

Various quality-control inspection testings on radioactive waste package can be implemented. This includes initial non-destructive testing (visual examinations, radiological measurements), complementary non-destructive testing (gamma spectrometry, neutronic active and passive measurements) and destructive testing (core drillings and cuttings).

The conventional technical assay to characterize the homogeneity of the waste block is a heavy and expensive process, consisting in cuttings and core-drillings of the radioactive waste block. Dose rate and gamma analysis on extracted core drill pieces and radiochemical analysis on samples taken from them need to be done. By this conventional way, radiological and chemical contents are not evaluated.

The purpose of this alternative sampling was for radiological and chemical analyses to thus characterize the radioactive contents of the waste block and check the block's homogeneity in conformance to French waste acceptance criteria and regulations. By sampling the block using  $TruPro^{\circ}$  the number of representative samples collected was maximized whilst minimizing the total quantity of extracted material and also minimized the physical degradation of the package during sampling operations.

These quality-control inspection testing require specialized nuclear facilities and equipment, sensitive and reliable measurement instrumentations and highly technical expertise skills.

### DESCRIPTION OF ACTUAL WORK

The sampling of the waste block consisted of acquiring samples from 12 sampling locations located on the upper surface of the Radwaste hull cap by implementing successful sampling and containment. This allowed to establish its feasibility on an unprotected concrete hull (no biological metallic shields) containing a homogeneous waste matrix.

This destructive testing was planned to confirm the observations highlighted during the nondestructive testing (gamma spectrometry measurements on the concrete hull, to evaluate the activity of the easy to measure radionuclides). The sampling campaign only lasted 4 days due to the simplicity and the efficiency of the sampling process, which is a good performance in comparison with classical assay methodology.

The 144 samples taken were representative of the whole waste block for the purpose of radiological and chemical analyses in order to characterize the radiological content of the waste block (information about its typical radiological spectrum), its chemical content (toxic elements) and its degree of homogeneity. The difficult to measure radionuclides represents approximately 60 % of the total activity of this Radwaste package).

For this radiological and chemical characterization, a (geo-)statistical study allowed to select the samples of interest for analysis and to evaluate the radiological and chemical contents and to check the degree of homogeneity.

# *TruPro<sup>®</sup>* System Characteristics and Specificities

*NMNT's TruPro®* Patented Technology, concrete / metal sampling and profiling tools, provide a superior approach to concrete or bulk material sampling and analysis to the traditional methods of

concrete sampling using a coring machine. The equipment in conjunction with portable radiometric instruments produces a profile of radiological contamination through the material being studied in near real time. The drill head is used under hammer action to penetrate hard surfaces. This causes the bulk material to be pulverized as the drill travels through the efficiently transmitting a radioactive media representative sample of bulk material to the sampling unit.

*TruPro*<sup>®</sup> Accelerates Facility Clean Out and Demolition by:

- Eliminating Historical Unknowns;
- Minimizing Lifecycle Risks, Applying Creative and Innovative Real-Time Deployable Solutions;
- Implementing Effective Sampling and Characterization;
- Identify Program Risks for Problem Areas Enhancing PM Approaches and Principles;
- Ensure Adequate Radiological Controls of Work Areas;
- Improve Site Upfront Understanding and Planning;
- Better Allocate Resources, Raw Materials & Highly Skilled Labor.

# Advantages of TruPro®

- Rate of sampling in conjunction with cheap and timely quantitative analysis of radionuclides of interest using portable infield instrumentation and radioanalysis;
- Minimal sample turnaround time, approx 2 hrs;
- Highly strategic, directional and focused sampling regime to areas of concern maximizing manpower and financial resources;
- No secondary wastes just sample using environmentally friendly extractants
- Containment of drill cuttings;
- The minimization of secondary drilling waste;
- Representative sample and quality from bulk materials where the baseline method cannot retrieve high quality representative samples;
- Increased safety due to less hands on exposure to physical hazards and waste contaminants and;
- Sampling incrementally to depths of up to 5 meters.

# **Profiling Objectives**

The sampling was done by carrying out "dry" drilling operations with TruPro® process (mechanical means) to collect samples in a powder form, so as to collect a maximum number of samples representative of the waste block, while minimizing the total quantity of extracted material

(less than 0,1% or radioactive material is taken) and the degradation of the package.

Drilling locations were chosen so as to examine the radial distribution (from the center of the waste block to its periphery) and the volumetric distribution (in the four corners of the hull).

This testing also focused on the drilling process, positioning of the hollow drill bit every 80 mm of each vertical drilling hole for obtaining samples of the different materials at the interface "concrete cap and waste block". For each sampling location, the first material to be penetrated was the concrete type hydraulic binder, a plug of homogeneous density 2.3 g/cm<sup>3</sup>. It was imperative to determine the thickness of the cap layer and be able to differentiate the plug material from the top of the radioactive waste block during sampling operations.

An important requirement of this destructive testing was also to test a new process of filling the 12 drilling holes with an expansive foam containing mortar, completed with a concrete mortar at the top of the drilling hole.



Fig 1. Sampling Locations.

144 samples of the waste block (mass about 15 grams), 18 samples of the concrete cap (mass about 40 grams) and 3 samples of silica and charcoal cartridges (trapping system for volatile radionuclides) were collected and then packaged and referenced. A part of these samples (about 10 to 20) was intended for radiological and chemical analysis in laboratory, after selection on the basis of gamma spectrometry measurements results with HPGe detector - and (geo-)statistical study of all the data (also for measured dose rates,  $\alpha\beta$ surface contamination values, first gamma measurements results with NaI(Tl) detector and density values).

#### DATA ANALYSIS AND RESULTS

Data analysis is mainly focused on the representation of the spatial distribution of activity levels within the cemented matrix and therefore on the analysis of the heterogeneity of the packaged waste (expected to be relatively homogeneous).

The vertical regular pattern gives enough data to analyze the spatial continuity along this axis. Moreover, to address several issues regarding spatial variability in the horizontal plane, the drill hole locations have been thoroughly designed: regular distribution over quadrants, along different sampling axes from the center to the edge.

#### **Correlation Clouds and Classical Statistics**

The first step of the data analysis consists in studying the correlation between the data. Dose rate measurements were collected during the drilling work and results from laboratory were available a few weeks later on. <sup>108m</sup>Ag, <sup>110m</sup>Ag and <sup>137</sup>Cs show good consistency

<sup>108m</sup>Ag, <sup>110m</sup>Ag and <sup>137</sup>Cs show good consistency on correlation clouds. Fig 2 illustrates a nice linear regression in addition with a slope close to 1, with a limited number of outliers. The regression is slightly deteriorated with <sup>60</sup>Co due to one sample with the highest value in <sup>60</sup>Co. This point is significantly outside the rest of the correlation cloud. Other measured nuclides such as <sup>54</sup>Mn and <sup>94</sup>Nb only have a couple of measures that exceed the detection limit.



In addition, measured dose rate at contact of the samples during the drilling works (and in particular regions of interest of the gamma spectrum) are correctly correlated with laboratory analysis once corrected by the sample mass. The point with high <sup>60</sup>Co levels was already identified during these real-time measurements.

To complete this first statistical analysis, histograms of <sup>137</sup>Cs and <sup>60</sup>Co (Fig 3) show a regular and symmetric distribution for <sup>137</sup>Cs while the highest value of <sup>60</sup>Co introduces a strong dissymmetry for this nuclide. Nevertheless, a dozen of <sup>60</sup>Co values (ranging between 5,000 and 15,000 Bq/g) are significantly above the rest of the distribution (see also discussion about heterogeneity criterion hereinafter).



Fig 3. Statistical distributions of <sup>137</sup>Cs (top) and <sup>60</sup>Co (bottom). Activity levels in Bq/g.

#### **Vertical Profiles**

The analysis of vertical profiles put the emphasis on lower activity levels for first and last sampling levels within the cemented matrix as

shown in Fig 4. Moreover the highest <sup>60</sup>Co value bellows to a sample collected in the top of the cemented matrix as well.



Fig 4. Vertical profiles of the 12 drillings.

These particular observations may be connected with, on the top of the cemented waste, a partial sampling of the first sample of each drill borehole with the non-contaminated concrete cap material and, on the bottom of the cemented waste, with a differential sedimentation due to the mixing process of the sludge with the hydraulic binder.

# Analysis of the Spatial Continuity

As for spatial continuity of the contamination, the variographic analysis shows a pure nugget effect both in vertical and horizontal directions. Whatever the distance, the activity variability between two samples is equal to the dataset variance. This is partly visible in Fig 4., where maximum values for each drilling appear isolated (vertically and laterally).

This absence of spatial structure is a good point for the waste homogeneity as it implies the randomness distribution of activity levels.

No advanced geostatistical processing's (Kriging, simulations...) are therefore implemented.

# Checking the Waste Homogeneity

As seen on vertical profiles, very first and very last samples are probably not representative of the activity inside the cemented waste because they are located at the interface with surrounding materials (see above remarks of fig. 4). They are excluded from the homogeneity analysis.

As mentioned before, the packaged waste is expected to be relatively homogeneous. In Andra's specifications, hot spots should not exceed a given percentage of the mean value. We are going to consider 50% and 100% as criteria to detect heterogeneity (Table 1). As  $^{108m}\mathrm{Ag}$  and  $^{110m}\mathrm{Ag}$  are strongly correlated with  $^{137}\mathrm{Cs},$  proportions are very comparable.

Table 1. Proportion of 137Cs and 60Co hot spots in comparison to mean activity.

	Mean (Bq/g)	> 50% exceeding	> 100% exceeding
137Cs	122	10%	3%
60Co	3,341	13%	<b>9</b> %

<sup>60</sup>Co looks like the most penalizing nuclide as regards homogeneity of the waste with almost 10% of the samples that exceed twice the mean concentration.

In addition, with slightly dissymmetric distribution (which is the case for <sup>60</sup>Co), median is a more robust measure of central tendency. In comparison to mean values, median for <sup>137</sup>Cs and <sup>60</sup>Co are respectively 116 and 2,725 Bq/g. For <sup>60</sup>Co in particular, the proportion of hot spots in comparison to median concentration increase even more (18% for 50% of exceeding).

### CONCLUSIONS/LESSONS LEARNED

This innovative characterization combines efficiently advanced sampling collection by TruPro® system and appropriate data analysis. In parallel with the actual standardized methodology to control the compliance of radiological and physicochemical specifications of Andra's SL-ILW cemented package, it strongly improves ANDRA's knowledge concerning nuclide heterogeneity in these packages in relation to its production and its matrix structure (sludge mixing, waste homogeneity in relation with specific drainage and nuclide migration during matrix drying...), radiological content and chemical species quantities (heavy metals notably). Experience feedback is also gained for a better and quicker characterization of next packages as regards sampling optimization (number and location) and rationalization of laboratory analyses.

This kind of destructive testing minimizes the degradation of the waste block envelope (concrete container and cap) and the quantity of radioactive material, hence less secondary waste. The implementation of the filling the drilled holes process also proved the integrity of the block. For the laboratory, solid samples in a powder form are easier to analyze and there is no subsampling requirement.

Moreover taking of samples, this destructive testing showed a uniform thickness of the cap (measured for each of 12 drillings) superior to the one of the container.

The dose rate and spectrometry gamma results highlighted that no significant value was measured

on the 18 samples of the cap, confirming that the *TruPro®* process can provide samples without any dispersion of radioactive material.

Another destructive testing using *TruPro*<sup>®</sup> process could be soon realized on a heterogeneous radwaste (filter of water immobilized in a hydraulic binder).