# Characterization, Method Development and Dealing with Old Radioactive Sludge Stored at the Research Station in Studsvik

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# ABSTRACT

During the years 1965-1985 liquid radioactive waste was collected in silos at the research station in Studsvik. Preparations started at the end of the 1980s for dealing with liquids that contained particulate material (ion exchange resin, sludge, sediment). Only parts of the documentation on what had been brought into the silo in question had been kept, which meant that comprehensive characterization work had to be carried out. The characterization included the relationship between bead ion exchange resin, sludge and clear liquid and the distribution of activity between them. The occurrence of chelates was also of interest to investigate. During the characterization phase three forms of treatment (drying, pyrolysis and immobilization in cement) were considered for the radioactive waste. The final choice was solidification, using concrete, in steel moulds with a 1.2 m side.

The characterization of the waste material and the method development resulted in an estimate that 60-70 steel moulds should be produced with an activity content of totally  $1.2 \times 10^{10}$  Bq  $\alpha$  and  $1.86 \times 10^{12}$  Bq  $\beta$ - $\gamma$ . At the beginning of 2005 all waste from the silo was processed. 104 steel moulds were produced, with a total activity of  $1.28 \times 10^{10}$  Bq  $\alpha$  (107 % of estimated activity) and  $6.05 \times 10^{11}$  Bq  $\beta$ - $\gamma$  (33% of estimated activity).

The experiences from the project show that older waste with insufficient documentation requires a great deal of work to characterize the material. The choice of method of dealing with the waste and the form of final-storage pack are largely governed by already existing methods. The waste material accumulated over a 20-year period, and the time spent dealing with this waste was nearly as long.

# **INTRODUCTION**

At the beginning of the 1960s a tank and silo facility for collection, checking and control of liquid-borne waste from radioactive operations, including spent ion exchange resin from R2 (research reactor) and ion exchange resin from Ågesta (heavy-water reactor), was erected in Studsvik.

The facility comprises a tank installation with the relevant pumps, valves and lines, plus two sludge silos (Silo 1 and Silo 2, each 150 m<sup>3</sup>) intended for collection of solids. The entire facility is located below ground level. The facility was modernized in the middle of the 1980s, whereby the silo section was shut down to deal with the waste stored during the period 1965-1985. Liquids that had contained particulate material (ion exchange resin, sludge, sediment) had been stored in Silo 1 whilst clear liquids, in the sense that no particulate material was present, had been stored in Silo 2.

The planning work with regard to dealing with the waste in the silos started in conjunction with HM (treatment facility for low and intermediate level waste) being put into active operation. The liquid-treatment part of HM started 1986, and the contents of Silo 2 (clear liquid) were treated during the years 1990 to 1992.

During trial operation of the solidification section of HM it was observed that the equipment for treatment of ion exchange resin was disturbed if sludge particles (even low contents) were present. This restriction meant that the planned solidification of Silo 1 material in HM could not be implemented, and planning of alternative treatments commenced.

It was now important to identify the most economical method which could at the same time be licensed with regard to working procedure, interim storage, transportation and final storage of the resultant waste packs.

# **PREPARATORY WORK**

Because of the difficulties in transporting and treating ion exchange resin with sludge content in HM, plans were made for solidification of the waste at Silo 1. Initially, before the waste treatment was carried out, a superstructure over the silos was erected. Sampling and analysis of the liquid surface [1] was also initiated during this period. A layer that was removed through absorption on fiber sheeting covered the liquid surface in Silo 1. Removal of the floating layer was a preparation for removal of the clear liquid that had collected above the sedimentary layers as shown in Fig. 1. The clear liquid in the silo was pumped away and treated in HM. This work was completed in 1993, and what then remained in the silo was ion exchange resin, sludge and some clear liquid.



Fig. 1. Outline diagram of Silo 1 with its contents after pumping away clear liquid *1*) Bead ion exchange and sludge. 2) Layer of sludge. 3) Remaining clear liquid. 4) Ground level

# WASTE CHARACTERIZATION

### Documentation

The operating log kept at the silo stated the amount of sludge brought in and the amount of liquid withdrawn, but beyond that only isolated details of the nature of the waste, thus extensive work was necessary regarding the silo's material and activity content.

# Origin

The sediment in Silo 1 accumulated over a period of about 20 years (1965-1985). The main content was bead resin from the R2 and Ågesta reactors plus a sludge-like phase precipitated from various chemicals. Liquid-borne waste had been brought in from a number of facilities with active operations within Studsvik. Furthermore, active solutions had been received from external institutions. In the 1970s the operating log listed individual items from hospitals and industrial companies.

The resin from the R2 and Ågesta reactors comprised mixed ion exchange resins. The most important types from R2 were:

Bead anion resin Duolite A101D and Amberlite IRA-402 Bead cation resin Duolite C20 and Amberlite IR-120

The ion exchange resin from Ågesta comprised: Bead anion resin Dowex 21K Bead cation resin Dowex 50W

Spent Ågesta ion exchange resin was delivered via the Studsvik TJR (heavy water/ion exchange regeneration) facility. This facility was used for concentration of heavy water, reception, dedeuterisation and dumping of spent active ion exchange resin as well as forming and deuterisation of new ion exchange resin. Up until 1974, when Ågesta was shut down, TJR was also used for handling of resin from R2 and TJR's resin cartridges for D<sub>2</sub>O purification. The preparation of new resin included screening out of fines. That material could constitute part of the organic component of the sludge in the Silo 1 sediment.

## Amount of sediment

The volume of sediment in the silo and distribution between resin and sludge/sediment was examined on several occasions during the years 1975-1993 [2,3,4,5]. The results of measurements/calculations varied from 12 m<sup>3</sup> to 55 m<sup>3</sup> of sediment. The variations are principally to be explained by the difficulty of identifying the boundary between the upper liquid surface and the sludge layer in the silo. Methods used were removal of sediment plugs at various positions in the silo and lowering of an aluminum sheet until it made contact with the upper surface of the sediment and reading the level of this in relation to the known bottom level in the silo.

In conjunction with the studies regarding the sediment volume, measurements were also taken of the dry-substance content in the sediment. Values for the dry-substance content in the sediment were given in a study [3] as 50% for drained, 35% for water-saturated resins and about 30% for the sludge. Other studies [5] on average gave 21.5% for mixed water-saturated sediment.

Dry-content determinations in conjunction with trial castings in November 1993 [6] indicated values of at least 30% in a water-saturated state following a long sedimentation period (6 days).

# Activity data

Activity analyses of the waste in Silo 1 were performed on 8 occasions during the period 1979-1993 [3,4,5,7,8,9,10,11], whereby it was established that most of the activity in the sediment was in the sludge fraction (90 % of all  $\alpha$ -emitters, 96-98 % of all  $\beta$ - $\gamma$ -emitters).

The reliability of the estimate of total activity in the silo depended entirely on the reliability of the estimate of the amount of sludge, and varied between  $0.1 \times 10^5$  and  $1.3 \times 10^5$  Bq/kg for  $\alpha$ -emitters and between  $1.4 \times 10^6$  and  $2.30 \times 10^7$  Bq/kg for  $\beta$ - $\gamma$ -emitters (wet weight).

# **Physicochemical nature**

The proportion of the sediment that comprised ion exchange resin was deemed to be fairly well defined, since its origin and type (mixed bead ion exchange resin) were largely known. Ion exchange resin that had been affected by the fuel damage in Ågesta 1968 should have been included in the sediment. This damage was probably a main cause of the content of  $\alpha$ -activity that occurred in Silo 1. The physicochemical specification of the sludge component in the sediment was insufficient. This was because of the long time the silo had been used for collection of liquid-borne waste from a large number of internal and external suppliers with varying operations. The operating logs did not usually specify the nature of waste items received at Silo 1.

On several occasions there were reports of unpleasant odors, indicating degradation of organic material in the sediment. Gas emission was observed in conjunction with lowering of the level of clear liquid in autumn 1993 to just over the sediment's upper surface. Gas formation was also established in the samples taken in conjunction with trial casting (major pressure increase in closed plastic containers). No analyses of the gas were performed.

# CHOOSING THE FORM OF TREATMENT

From the shutdown of Silo 1 (1985) and during the first characterization period (until 1993) three different forms of processing were considered.

# **Pyrolysis**

For the pyrolysis option there were two proposals: one from Studsvik RadWaste and one from NUKEM.

RadWaste's pyrolysis/drying option was based on use of an existing bench-scale facility erected at ACL (Active Central Laboratory, Studsvik) for pyrolysis-powdered resins. This facility had been tested with approx. 100 kg German ion exchange resin as part of a research program.

NUKEM had primarily been consulted regarding cementation, but had also declared its interest in offering a pyrolysis option. NUKEM's offer concerned the requisite equipment.

# Drying and packing in suitable packaging

For resin from nuclear power stations in Sweden there is an established method of performing dewatering/air-drying and packing in concrete tanks. An equivalent method was sought as an alternative for treatment of Silo 1 sediment. Bearing in mind that the sediment from Silo 1

was less homogenous and harder to specify, the condition was that this waste treatment method had to be combined with more suitable packaging.

There were two proposals for the drying option: one from GNS (Gesellschaft für Nuklear Service) and one from Studsvik RadWaste.

GNS had developed a method for direct drying of wet waste in the packaging for final storage. The sediment would be sucked up using a telescopic pipe from the silo and conveyed down to packaging. Drying of the sediment taken up would then take place layer by layer in the packaging by putting it under vacuum and at a temperature slightly above room temperature.

RadWaste proposed a method of drying followed by direct packaging. The proposed pyrolysis equipment/process was to be discontinued before the actual pyrolysis initiates (pyrolysis calls for dried material). The dried material would then be transferred into moulds intended for interim and final storage.

## Immobilization in cement

Solidification in cement was the method originally intended for dealing with the sediment in Silo 1 and was included in the planning of HM. But as mentioned earlier the nature of the sediment was such that the normal HM system for dealing with ion exchange resin was unsuitable for the Silo 1 waste.

For treatment by means of solidification there were two proposals: one from Studsvik RadWaste and one from NUKEM.

Studsvik RadWaste's proposal was to use only the solidification component of the HM system. The end product would then be 200-litre drums of solidified waste.

NUKEM was consulted regarding a proposal for solidification performed in specially erected equipment placed in the silo facility. The method meant that waste and cement were mixed and then weighed out into the waste packaging.

# Choice of final storage

For the waste from Silo 1 there were two options for final storage: SFR (final repository for radioactive operational waste), which is already in operation, and the not-yet constructed facility SFL (deep repository for long lived waste and spent nuclear fuel). While SFR can provide final storage only for limited amounts of long-lived nuclides, SFL is planned for larger amounts of these nuclides.

In order to get a permit for manufacture and final storage of the waste packages from Silo 1, knowledge of the nature of the waste was needed. For SFR information is needed on activity content, amount and type of biologically degradable material, swelling properties and content of gas and chelates, likewise of leaching and degradation products that may form.

There are at present no specified requirements for waste going to SFL, but it can be assumed they will be of a type similar to that for SFR. For the options solidification in cement and RadWaste's pyrolysis method, already certified waste packages could be used.

Based on the information and investigations hitherto and thoughts from SKB (Swedish Nuclear Fuel and Waste Management Co, with operational responsibility for SFR) regarding final storage. It was decided to continue measures aimed at solidifying of the waste in cement.

One investigative measure for improving the possibilities of specifying the sediment's physicochemical properties was deemed necessary; it would primarily be aimed at the organic content. The task was difficult because of the relatively high level of activity, especially the presence of  $\alpha$ -emitters combined with a very complex chemical composition. The inhomogeneous nature of the sediment in situ created difficulties in taking representative samples for quantitative analysis. Furthermore, the technology and location for the solidification process still had to be studied.

# ADDITIONAL INVESTIGATIONS 1993-1995

The main starting point for the additional physicochemical determinations was investigation of parameters specified by SKB that were of interest regarding final storage in SFR. The content of chelates, oil, solvents, cellulose and cyanide was to be studied, as well as studies concerning feasibility of using HM for treatment of the silo content.

To sum up, the additional investigative measures [12] showed that:

- Resins and sludge are unevenly layered in the silo.
- The total volume and volume distribution between resin and sludge is hard to determine by studies of sediment plugs.
- The physicochemical properties of the waste should be compatible with solidification in cement. This was confirmed by casting of sample bodies.
- The concentrations of certain waste components that SKB deemed to be of interest for final storage in SFR were low, and the level of activity in the silo was close to that of previous measurements/assessments.
- Treatment of the waste in HM was deemed less attractive, both technically and financially, than options based on solidification in the silo building.

Three suppliers submitted offers for an embedment facility for the silo building.

On the basis of the results of the additional investigations it was decided that solidification of the waste in cement was to be performed in the silo building. Pumps were to be installed for homogenization of the silo content. Trial castings were to be performed on a laboratory scale using the homogenized material.

# METHOD DEVELOPMENT AND TRIAL CASTINGS

Trial castings on a laboratory scale had previously been performed with heterogeneous silo mass [6], but since the solidification was to take place using homogenized silo mass, further trial castings was scheduled. In conjunction with the trial castings the investigations with regard to organic material (especially cellulose), chelates and gas producing substances performed on previous samples were to be verified on the homogenized material.

To sum up, the results [13] from these trial castings showed no noteworthy difference compared with previous analysis of the silo content.

The laboratory trial casting included verification of the embedment formula (admixtures SIKA 1 and SIKAMENT 10) and tests of its tensile properties.

The description of the method of solidification in accordance with [14] was revised due to formation of colloidal suspensions in the solution, due to homogenization of the silo content. The excess water used during pumping was now to be used as casting water. Dry cement was to be used instead of cement paste.

The embedment method that had now been proposed was in brief as [15] follows.

## PROCESS DESCRIPTION 1 (1996-1998)

#### **Steel moulds**

The steel moulds are welded cubic sheet-metal containers with a 1.2 m side. Their upper section is provided with lifting eye bolts adapted for lifting with the aid of special lifting yokes.

The outside of the steel mould is treated with rust proofing paint.

The steel moulds are equipped with an agitator and a splashing plate as shown in Fig. 2. During manufacture of the steel moulds a serial number is entered on one side.



Fig. 2. Waste container with filling and agitator equipment

## Solidification and activity measurements

The casting process commences when the silo 1 content is homogenized. Homogenization is achieved through vigorous pumping inside the silo.

After homogenization of the silo contents a predetermined volume of waste slurry is pumped from the silo to the container. The output dose rate from the container is measured continuously and pumping is interrupted if the 1 m dose rate exceeds 2 mSv/h.

After a predetermined sedimentation period the container's volume of the settled mixture of resin and sludge is measured. The sediment volume is determined through density measurements. The sediment volume measured in the mould determines the amount of additive (cement, Hydrofix, Sikament 10) and free water for the container in question. Normally the requirement is that about a third of the container's free water could be pumped back to the silo. Final filling of the container to the specified filling level takes place upon lid casting with concrete.

If the measured amount of settled resin and sludge is less than the expected minimum value, more of waste slurry is pumped from the silo to the container after the free water has been pumped back to the silo. Renewed measurement of the sediment level is then performed.

After the waste matrix has hardened a concrete lid is cast before transportation to interim storage.

The following measurements were planned during the process casting:

- Measurement of gamma spectrum for every single container.
- Analysis of dry-substance content,  $\alpha$ -activity and  $\beta$ - $\gamma$ -activity for every tenth container. On the condition that the relevant analysis for total specific activity does not deviate by more than ±10% from average, the sample volumes from every tenth container are mixed to form a sample that is analyzed regarding the  $\alpha$ -activity. If the total specific  $\alpha$ -activity deviates by more than ±10% from average, deviating sample volumes are also analyzed.

The  $\alpha$ -content of individual steel moulds is estimated on the basis of the above measurements and the amount of waste measured per container.

Average compositions and possible variations of the waste product are shown in Table I.

	Average composition	Possible variations
Resin/sludge/water even level	Approx. 640 liters 15-25% dry-substance content	550-700 liters 10-45% dry-substance content
Free casting water in container	Approx. 400 kg	200-450 kg
Standard cement	Approx. 1300 kg	1,100-1,500 kg
Sikament 10	1% of cement weight	0.8-1.2%
Hydrofix	0.8% of cement weight	0.6-2%

Table I. Average Composition and Possible Variations of the Final Waste Product

#### **Final storage**

Final storage of the waste packs produced was to be in SFR and in its silo storage facility (the silo storage facility is licensed for the highest activity content of long-lived nuclides).

## **CHANGES MADE TO PROCESS DESCRIPTION 1**

Process Description 1 stated that admixtures (water reducer, Sikament 10 and water permeability reducer, Hydrofix) should be used for the solidification process and that part of the free water should be pumped back to the silo at the time of each sedimentation period.

Since the admixtures could act as chelates in the final storage (SFR) and thus facilitate the migration of radionuclides, it was decided that these admixers should not be used for solidification of the silo waste, and renewed laboratory castings for determination of tensile strength and leaching [16] were performed.

Further investigations were performed [17] to clarify whether strong chelates could be demonstrated in the waste or whether activity bound to fixed aggregates could be released in the cement, e.g. through chelates that are active at a high pH. The results showed that:

The activity is mainly bound to the suspended solid particles (sludge).

 $\alpha$ -emitting nuclides are released in an acid, oxidizing or strong acid environment, but not in an alkaline environment.

No significantly increased release was observed in the cement. Presence of strong chelates of acid nature is improbable.

High contents of ISA (isosaccharinic acid from cellulose) led to release of  $\alpha$ -activity (not Am, possibly Pu); possible occurrence of ISA in the original waste slurry (or formed at high pH) was not indicated.

Nothing arose that indicated the occurrence of  $\alpha$ -emitting nuclides in such a form that they would be mobilized in cement.

The solidification method in accordance with Process Description 1 included a step whereby the homogenized waste slurry was pumped up to the container, after which the slurry was to sediment in the container and the 'clear liquid' be pumped back into the silo. This was to be repeated a number of times until a specific predetermined amount of solids had been transferred to the container. The aim of this method was to minimize the number of produced steel moulds. The remaining clear liquid in Silo 1 would then be processed at the HM facility. Since there would probably be particulate material remaining there was some concerns as to whether the liquid could be treated in HM. Thus, the solidification method was modified in such a way that a constant weight of the homogenized waste slurry was pumped up to the mould, to which cement was added whilst agitating. No sedimentation and drainage of 'clear liquid' were performed. The method meant an increase in the number of steel moulds were to be produced, but this was compensated for by the easier handling and the fact that no liquid would be left remaining in the silo for further waste treatment.

The solidification method now proposed was in brief as follows [18].

## PROCESS DESCRIPTION 2 (1999-2001)

## Steel moulds

No change from Process Description 1

## Embedment and measurement of activity

Process casting starts with homogenization of the content of Silo 1. This is performed by the waste being pumped around down in the silo.

After homogenization of the silo content a predetermined weight (1,000 kg) of waste slurry is pumped from silo to container. The dose rate at the container is measured continuously and pumping is interrupted if the dose rate at 1 m exceeds 2 mSv/h.

A predetermined quantity (1,160 kg) of cement is added to the slurry.

After the waste matrix has hardened a concrete lid is cast prior to transportation to interim storage.

During the casting process the following activity measurements are planned:

During casting samples are taken of the waste material. The samplings are performed at the end of the homogenization period that precedes dosage to the container. As ten steel moulds have been cast samples are taken from each one. These samples are put together and analyzed regarding its dry-substance content,  $\alpha$ -,  $\beta$ - and  $\gamma$ -activity.

The activity content in individual packs is estimated based on the above measurements and the measured amount of waste per container.

# **Final storage**

No change from Process Description 1

# **CHANGES MADE TO PROCESS DESCRIPTION 2**

Before Process Description 2 could be reviewed and approved by the Swedish authorities SKI (Swedish Nuclear Power Inspectorate) and SSI (Swedish Radiation Protection Authority) and by SKB (Swedish Nuclear Fuel and Waste Management Co Ltd), there were full-scale castings [19] with inactive resin (mixture of bead and powder ion exchange resin) and with homogenized silo waste. The casting with the inactive resin would clarify the issue of the homogeneity of the finished pack, and the casting of the active waste would verify the function of the solidification and the increase in external dose rate on the surface of the container, and would provide sample material for laboratory experiments for verification of the concrete's tensile strength.

The homogeneity of the immobilization of the inactive resin was verified by splitting the container diagonally, as shown in Fig. 3. No cavities or heterogeneous zones were detected in the waste matrix [19]. Furthermore, it was established that the amount of cement was too high. This was corrected during preparation of the forthcoming active trial solidification process.



Fig. 3. Method of cutting through the sheet-metal container diagonally

The trial solidification of homogenized Silo 1 mass was performed without any problems, and laboratory samples of the removed cement/silo mixture showed that the pack was of a sufficient strength [16] for final storage in SFR's silo storage facility.

# PERMIT FOR MANUFACTURE, TRANSPORT AND FINAL STORAGE

Before waste packs can be manufactured, transported and put into final storage in Sweden a permit must be issued by the Swedish authorities SSI and SKI.

The documentation for this permit is a type classification [20] stipulating:

- The origin of the waste
- Treatment
- Interim storage
- Handling and transportation
- Deposition in final storage facility

According to the preliminary investigations the silo contained about 25 m<sup>3</sup> of resin/water and about 25 m<sup>3</sup> of sludge/water. The intended solidification process formula for the resin/sludge/water phase would result in a maximum of 90 steel moulds (earlier estimated to 60-70 steel moulds).

The total activity in the steel moulds produced was estimated to  $1.2 \times 10^{10}$  Bq  $\alpha$  and  $1.86 \times 10^{12}$  Bq  $\beta$ - $\gamma$ .

The permit for manufacture, transportation and final storage was received on 19.12.2001. The solidification process began on 05.02.2002 and was completed practically on 03.05.2005.

# RESULTS

#### Number of steel moulds

Cement solidification was initially an uninterrupted process. As about a quarter of the waste in the silo remained further pumping became impossible due to the highly compacted waste found in the lower part of the silo. The only way of resolving the problem was to bring the compacted sediment into suspension by means of high-pressure rinsing with water, thus increasing the volume of waste. Furthermore, the amount of waste was larger than estimated in the earlier studies. These circumstances led to an increase in the number of steel moulds finally produced.

At the beginning of 2005 all waste from Silo 1 had been dealt with and the project was concluded. 104 steel moulds were produced.

## Activity

The total activity content in the 104 steel moulds was  $1.28 \times 10^{10}$  Bq  $\alpha$  (33% of estimated activity) and  $6.05 \times 10^{11}$  Bq  $\beta$ - $\gamma$  (107% of estimated activity), distributed amongst nuclides in accordance with Tables II and III.

Nuclide	Bq
Am-241	2.29E+09
Pu-239+240	4.30E+09
Pu-238	5.50E+09
Cm-244	7.48E+08
Total	1.28E+10

Table II. α-Emitting Nuclides

Table III.	$\beta$ - $\gamma$ -Emitting Nuclides
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Nuclide	Bq
Cr-51	1.95E+07
Co-60	2.35E+10
Ag-108m	4.52E+07
Cs-134	7.98E+07
Cs-137	5.81E+11
Eu-154	4.22E+08
Eu-155	6.01E+07
Total	6.05E+11

## Experiences

The experiences from the project show that older waste for which there is insufficient documentation requires more work in terms of material characterization. The choice of handling method and the form of final-storage packs are largely governed by the methods already existing. The waste material accumulated over a 20-year period, and the time for dealing with this waste was nearly as long.

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All reference material is in Swedish.

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