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Overview of the methods used in RATEN ICN for radioactive waste characterisation

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Summary. — The manuscript presents the results of the research and development activities carried out in the Institute for Nuclear Research Pitesti (RATEN ICN) in the field of radioactive waste characterisation. After breafly describing the Romanian context in the field of radioactive waste management, the manuscript gives details on the methods developed/optimized and applied for specific radioactive waste fluxes generated in RATEN ICN but also on RW generated by Cernavoda NPP.

1. – Introduction

In Romania radioactive waste are generated mainly by Cernavoda NPP, having in operation two CANDU 600 units, FCN Pitesti from the fabrication process of CANDU nuclear fuel, and by the two research institutes: Institute for Nuclear Research Pitesti (RATEN ICN) and Horia Hulubei National Institute for R&D in Physics and Nuclear Engineering (IFIN-HH) Bucharest. RATEN ICN operates a TRIGA research reactor and IFIN-HH operated a VVR-S reactor whose decommissioning was finalized in 2020. Small amount of radioactive waste and sealed spent sources are also generated from using the radioactive sources in industry, medicine and academia.

The only final disposal in operation in Romania is Baita Bihor National repository, placed in an old uranium mine and licensed to dispose of the institutional low and intermediate level, short lived radioactive waste (LILW-SL). For the disposal of the LILW-SL generated by operation, refurbishment and decommissioning of Cernavoda NPP, according to the Romanian National Strategy a near surface disposal will be commenced in 2028. For the spent fuel (SF) and long lived waste (LILW-LL) disposal a deep geological repository is foreseen to be in operation in 2055. Until the geological repository will be available the SF and ILW-LL are stored on the generator sites.

The Technologies for Nuclear Energy State Owned Company (RATEN) is a strategic Romanian legal entity that coordinates R&D activities in the field of nuclear energy, while offering and developing scientific and technological support for the National Nuclear Energy Program. RATEN Annual Research Program "Developing National Technical Support and International Cooperation for Nuclear Energy" provides the framework for the development research and development and technological engineering activities at RATEN. The Annual Research Program supported by 18 Research, Development and Technological Engineering Programs (CDIT) responds to the RATEN strategic. Two of them are dedicated to the radioactive waste and spent nuclear fuel management and radioprotection both of them having a research topic dedicated to development and optimization of characterisation methods for measurement the radionuclide inventory in radioactive waste generated by the operation of TRIGA research reactor and by Cernavoda NPP and respectively in environmental samples. These methods address difficult to measure radionuclides (DTM) consisting in both pure beta emitters such as ^{14}C and ^{3}H ^{90}Sr, ^{63}Ni, ^{129}I, ^{99}Tc, but also alpha emitters.

Generally, for the waste matrix dissolution, required for separation and purification of DTM radionuclides for their subsequent measurement by liquid scintillation counting (LSC) or alpha spectrometry, acid mineralization (using simple open vessels or Microwave digestion system) is applied. This method has to be carefully applied when volatile radionuclides (such as ^{14}C or ^{129}I) have to be measured. For ^{14}C and ^{3}H non-catalytic combustion using dedicated equipment may be applied. Additionally, for ^{14}C separation and purification a combination of acid stripping and wet oxidation was optimized for different waste matrices, this method allowing separately absorption of inorganic and organic ^{14}1C. For tritium, simple or under vacuum distillation is applied for majority of waste matrices while for metallic waste a passive sampler was developed in RATEN ICN. For ^{90}Sr, ^{63}Ni, ^{129}I, ^{99}Tc and actinides separation and purification specific Eichrom resins are used after matrix dissolution by acid mineralization.

2. – Methods used for measurement of ^{14}C content in radioactive waste

Due to its long half-life ($T_{1/2}$= 5730 years), high mobility in groundwater system, easy incorporation into man via the food-chain, and high inventory in radioactive waste generated from Cernavoda nuclear power plant (NPP) operation, ^{14}C is a radionuclide of concern for radioactive waste management, both in the predisposal activities as well as for final disposal. Since CANDU reactors use heavy water (D_2O) both as moderator and as coolant, more ^{14}C is generated in this type of reactors due to the large amount of D_2O having higher isotopic abundance in ^{17}O compared to the light water (0.055% in D_2O and 0.037% in H_2O), but also to the higher average thermal neutron flux in CANDU reactors (10^{14} n×cm^{-2} ×s $^{-1}$ compared to 10 ^{13} n×cm^{-2} ×s $^{-1}$ in light water reactors). For this reason, in RATEN R&D programme methods to measure the ^{14}C activity in different waste categories were developed.

To separate the ^{14}C from different waste matrices in view of the radioactivity measurements by LSC two main methods are applied: non-catalytic combustion and a combination of acid stripping and wet oxidation, optimized for different radioactive waste categories after the method developed by A. Magnusson for spent ion exchange resins [1].

Applying the non-catalytic combustion by flame oxidation method the radioactive waste samples are combusted in an oxygen-enriched atmosphere with a continuous flow of oxygen using a Sample Oxidizer Model 307 produced by PerkinElmer. In this system any hydrogen and carbon is oxidized to H_2O and D_2Orespectively. As the carbon dioxide readily reacts with compounds containing amines, the Carbo-Sorb® E is used to absorb the CO_2 released during combustion process. The amine contained by the Carbo-Sorb® E solution (RNH2) reacts with the ^{14}C-labeled carbon dioxide to form a zwitterion that further reacts with H_2O to form a stable carbonate compound. This carbonate compound is mixed with an adequate scintillation cocktail (Perma-fluor® E $^{+}$) directly in the counting vial that at the end of the process is ready to be measured by LSC.All process is automated and at the end of the combustion process two separate LSC vials, one for ^{14}C measurement and one for ^{3}H measurement are obtained.

As the mobility of ^{14}C in geosphere depends on its speciation and the speciation under which ^{14}C is released from a disposal facility depends on the speciationof ^{14}C contained in the radioactive waste disposed of, a method for separation of inorganic ^{14}C by the organic ^{14}C was developed for the main radioactive waste matrices. This method consists of sequential extraction of inorganic and organic ^{14}C from the same radioactive waste sample using in the first step the acid stripping and in the second one wet oxidation. The experimental setup used for separation and purification of inorganic and organic ^{14}C is schematically presented in fig.1.

During the acid stripping step, carbonates CO_3^{2-}, bicarbonates HCO_3^{-}, and dissolved CO_2 (aq) equilibrium is shifted towards gaseous carbon dioxide by the weak acids added over the radioactive waste. The ^{14}C released during the acid stripping step is carried by the carrier gas through the alkaline gas washing bottles (B3 and B4 - fig. 1) where the $^{14}CO_2$ is absorbed as sodium carbonate and/or sodium bicarbonate. If any ^{14}C is released as $^{14}CO_2$ or other organic molecules, the gases pass through the scrubbing bottles of the first gas washing line, and further through the catalytic furnace, where $^{14}CO_2$ and other ^{14}C-bearing organic molecules are oxidized to $^{14}CO_2$ ^{14}C, that is subsequently absorbed

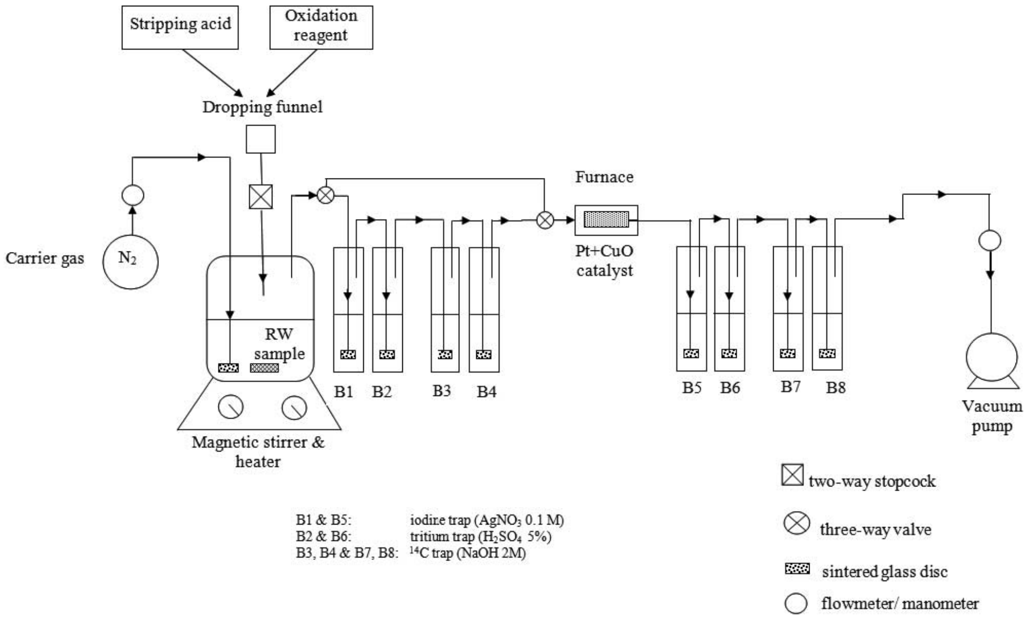

Fig. 1. – Experimental setup for separation of inorganic and organic ^{14}C

in the alkaline scrubbing bottles placed after the furnace (B7 and B8 - fig. 1).

As usually in any radioactive waste sample beside 14C there are a lot of other radionuclides that by disintegration emit beta radiation (such as ^{3}H, ^{129}I, ^{137}Cs, ^{90}Sr, etc.) and interfere with ^{14}C at LSC measurements, dedicated gas washing bottles are used before the alkaline ones to purify the gases carrying the ^{14}C. For absorption of ^{3}H and other beta-gamma emitters that can be released in the gas phase together with ^{14}C a slightly acidic trap is used (B2 - fig. 1). As iodine could be sorbed together with ^{14}C in the alkaline solution a washing bottle with silver nitrate is used to precipitate any iodine that could be released from the analysed radioactive waste sample and iodine trap is placed before the acidic one (B1 - fig. 1).

After the acid stripping step is accomplished, the first gas washing line is isolated from the system to not allow any ^{14}C released in the wet oxidation step to be absorbed over the inorganic ^{14}C absorbed in the acid stripping step.

Since organic compounds have higher bounding energies between atoms, they are decomposed by strong oxidants such as potassium persulphate ($K_2S_2O_8$). The presence of a catalyst such assilver nitrate ($AgNO_3$) enhances the decomposition of the organic compounds. During the wet oxidation step the temperature of the solution in the reaction vessel is slightly increased to 90 °C.

The ^{14}C released during wet oxidation step of the analytical procedure (both as CO2, but also as CO or CH_4) is carried by the carrier gas through a catalytic furnace where it is oxidized to CO_2, which is afterwards absorbed in the scrubbing bottles of the second gas washing line (fig. 1). To allow absorption of potential organic ^{3}H and iodine that could be released during the wet oxidation step acidic and silver nitrate traps are introduced after the furnace (B5 and respectively B6).

At the end of the oxidation step, aliquots from the alkaline washing bottles are measured by LSC to determine the activity concentration of inorganic and organic ^{14}C in the analysed sample.

This sequential method was adapted and tested for diverse waste matrices such as metallic waste (irradiated Zy-4 and stainless steel), molecular sieves, spent ion exchange resins and irradiated graphite [2-4]. Adequate acid or combination of acids is used in the acid stripping step to release the ^{14}C from a waste matrix. Adequate matrix dissolution is important to ensure that the total amount of the ^{14}C is released. If for easy to dissolve matrix such as spent ion exchange resin sulphuric acid (6M) can be used in the acid stripping step [2] for ceramic matrix such as molecular sieves or for metallic matrices stronger acids have to be used. Following the tests performed in our institute for molecular sieve or ceramic matrices a mixture of three parts concentrated hydrochloric acid and one part concentrated nitric acid (known as aqua regia) proved to be adequate. For irradiated Zy-4, matrix dissolution is achieved using a combination of nitric acid (20%) and diluted fluoric acid (6%) [3], while for harder metals such as stainless steels (SS) aqua regia proved to be suitable.

3. – Methods used for measurement of ^{3}H content in radioactive waste

In CANDU reactor tritium is mainly generated by the neutron activation of deuterium from the heavy water moderator and coolant.

By tritium disintegration low beta rays are emitted with maximum energy of 18.6 keV and average energy of 5.69 keV. As previously mentioned, one of the methods applied in RATEN ICN for tritium separation and purification from a solid or liquid radioactive waste sample is non-catalytic combustion by the flame oxidation. By this method, the radioactive waste samples are combusted in an oxygen-enriched atmosphere with a continuous flow of oxygen using sample oxidizer Model 307 by PerkinElmer. As previously mentioned, in this system any hydrogen and carbon is oxidized to H_2O and CO_2, respectively. The H_2O resulting from the combustion process is condensed in a cooled coil, washed into a counting vial where it is mixed with LSC cocktail (MonophaseS) and the resulting vial is ready for tritium counting by LSC.

This method is suitable for radioactive waste categories that do not contain high amounts of ^{137}Cs or other volatile radionuclides.

Distillation techniques (simple or under vacuum) are applied for a large variety of waste matrices from aqueous and organic liquid radioactive waste to solid radioactive waste (such as textile, cellulose, plastics, molecular sieves, spent ion exchange resins). If for the liquid radioactive waste the volume of the waste used to separate ^{3}H by distillation has to be no more than 2/3 from the distillation flask, for solid radioactive waste the mass is chosen based on the total radioactivity and an adequate volume of distilled water (DW) is added. To be applied on real radioactive waste, tests to determine the recovery yield is performed with matrix similar with the radioactive waste to be analysed marked with known amount of tritiated water.

A passive sampler was patented in RATEN ICN for^{3}H sampling from contaminated metallic waste. The method for tritium measurements from the metallic waste is based on the absorption by the anhydrous calcium chloride powder of tritiated water released under normal conditions (temperature and pressure) from the metallic waste analysed. The sampler mainly consists of a 20 ml LSC vial with a layer of anhydrous calcium chloride powder deposited on the inner surface. An applicator is adapted from a scintillation vial cap with a central hole of 14 mm diameter. The sampling is done iteratively on the same surface until the sampled activity, in a run, is less than A passive sampler was patented in RATEN ICN for ^{3}H sampling from contaminated metallic waste. The method for tritium measurements from the metallic waste is based on the absorption by the anhydrous calcium chloride powder of tritiated water released under normal conditions (temperature and pressure) from the metallic waste analysed. The sampler mainly consists of a 20 ml LSC vial with a layer of anhydrous calcium chloride powder deposited on the inner surface. An applicator is adapted from a scintillation vial cap with a central hole of 14 mm diameter. The sampling is done iteratively on the same surface until the sampled activity, in a run, is less than 10%of the previously sampled one. Counting of samples is performed by LSC, using a compatible scintilation cocktail. 10% of the previously sampled one. Counting of samples is performed by LSC, using a compatible scintilation cocktail.

4. – Method used for measurement of ^{90}Sr content in radioactive waste

The ^{90}Sr radioisotope is a difficult to measure radionuclide because it is a pureβ emitter. Therefore, ^{90}Sr in various types of radioactive waste samples can be measured by liquid scintillation counting (LSC) technique after efficient radiochemical separation of ^{90}Sr from other beta-emitting radionuclides.

The radiochemical separation and purification technique of ^{90}Sr is based on development and testing of the analytical methods applicable to natural isotopes in the form of Sr^{2+} species. There are different methods for strontium separation based on classical and instrumental analytical methods, such as: precipitation/co-precipitation, liquid-liquid extraction, ion-exchange chromatography, extraction chromatography. Extraction chromatographic method presents a series of advantages compared to solvent extraction and ion-exchange: less reagents and chemicals, less hazardous waste, a better effective separation, a good contact of the reagents and fast exchange kinetics [5].

The Eichrom® Sr Resin [6, 7] is mainly used for Sr^{2+} separation and purification on the base of an extraction system with selectivity for strontium. The extractant is a crown-ether [4,4(5′)-di-t-butylcyclohexano-18-crown-6], whose macro-cyclical structure delimits intramolecular cavities in which Sr^{2+} ions are retained, forming complexes with a high stability constant.

The strontium affinity for the resin increases with the nitric acid concentration, reaching a maximum value for a concentration of 8 M HNO_3. Therefore, high decontamination factors of interfering elements are achieved by loading the sample in 8M HNO_3 [8].

The carrier strontium content has a great influence on the chemical yield of strontium. When the strontium amount in the sample is greater than 8 mg Sr/mL resin, the chemical recovery of strontium decreases [8].

The main steps involved in separation and purification of ^{90}Sr are summarized in fig. 2.

5. – Method used for measurement of ^{63}Ni content in radioactive waste

^{63}Ni is mainly produced in the structural steels of nuclear reactor vessel and internal components from neutron activation of ^{62}Ni stable isotope. The measurement of ^{63}Ni radioactivity is based on detection of β particles emitted by ^{63}Ni ^{63}Ni disintegration (with a maximum energy of 67 keV) by liquid scintillation counting (LSC), technique requiring radiochemical separations and purifications of Ni isotopes. The radioactive waste may contain both ^{63}Ni and ^{59}Ni and these radioisotopes will be both present in the final solution resulted by applying a separation and purification method, but it is considered that ^{59}Ni contribution to the ^{63}Ni counting by LSC can be neglected.

The ^{63}Ni activity measurement in a solution resulted by mineralization of a radioactive waste sample, requires developing a three-steps chemical treatment technique [9]:

1) Radioactive waste dissolution by microwave mineralisation (before mineralization stable ^{62}Ni carrier is added to the radioactive waste sample, to be able to determine the recovery yield).
2) Sample matrix conversion to chloride form
3) ^{63}Ni + ^{62}Ni separation from other interfering radionuclides, comprising of the following steps:
    - a. resin conditioning
    - b. sample loading
    - c. resin washing
    - d. ^{63}Ni + ^{62}Ni elution
    - e. effluents preparation
    - f. measurement of ^{63}Ni concentration activity by LSC and ^{62}Ni concentration by ICP-OES

To facilitate the Ni retention on a Nickel selective resin, the liquid sample obtained by the dissolution of radioactive waste sample is evaporated to near dryness and converted to chloride form by repeated evaporations with concentrated HCl. The HCl solution

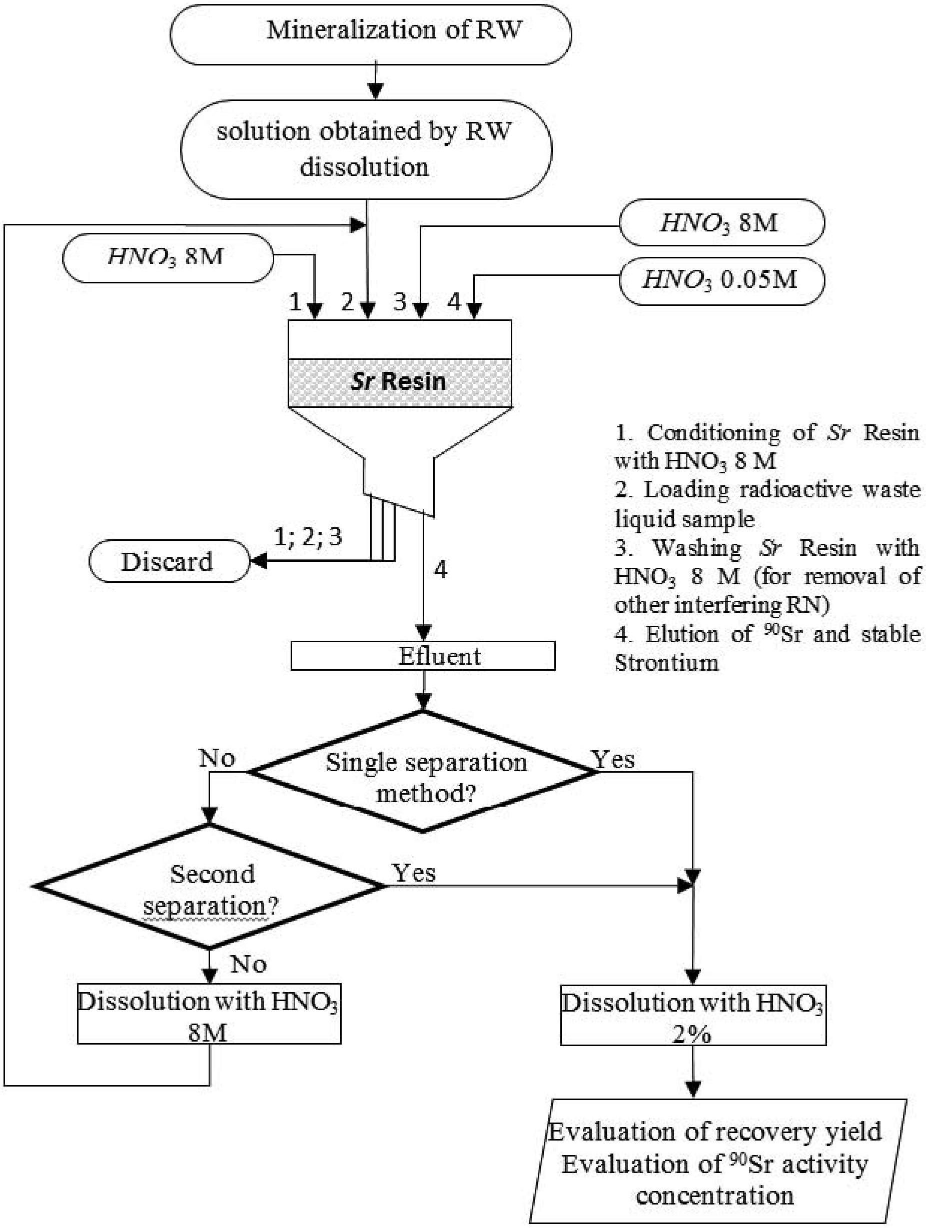

Fig. 2. – Schematic presentation of analytical method for separation and purification of ^{90}S.

is preferred to the HNO_3 solution because the nickel chloride ($NiCl_2$) is not volatile compared to nickel nitrate, $Ni(NO_3)_2 6H_2O$, that has a boiling point of 1370C. The residue is dissolved in 1M HCl and a solution of 0.2M ammonium citrate is added and the pH of the overall solution is adjusted to 8-9 with ammonium hydroxide [9,10].

The Nickel selective resin produced by Triskem International France (100-150 μm particle size) consists of a dimethylglyoxime extractant coated on an inert support. Ni separation using this resin is based on the on-column precipitation reaction in which two DMG molecules react with Ni^{2+} to form Ni-dimethylglyoxime complex, $Ni(DMG)_2$. The $Ni(DMG)_2$ is insoluble in water, with a high stability constant.

Before using, the Nickel selective resin has to be conditioned with a 0.2M ammonium citrate solution adjusted at pH 8-9 with ammonium hydroxide. The presence of the ammonium citrate prevents any co-precipitation of metal ions that would precipitate as insoluble hydroxides, making the separation process impossible. The pH is adjusted to 8-9 with ammonium hydroxide before loading the resin because the complex of Ni with DMG is stable at pH 7-12. When the samples are loaded on the resin, a red precipitate of Ni with dimethylglyoxime (DMG) is formed. The resin is rinsed with a 0.2M ammonium citrate solution adjusted at pH 8-9.

The Ni(DMG)2 complex is dissolved with a 3M HNO_3 solution, allowing its elution from the resin. After Ni(DMG)2 elution, the resin colour is changed back to white and the eluted solution is colourless [11-12].

The eluate, containing both ^{63}Ni and ^{62}Ni, is evaporated to 0.2-0.5 ml on a hotplate and dissolved in 1% HNO_3 solution to be measured by both by LSC, for ^{63}Ni concentration activity and by ICP-OES to determine the recovery yield [9].

6. – Method used for measurement of ^{99}Tc in radioactive waste

^{99}Tc is produced in nuclear reactors through the three main reactions: by the thermal neutron fission of ^{233}U and ^{239}Pu with a yield of 4.8% and 5.9% respectively, by the fast neutron fission of ^{239}Pu, ^{238}U and ^{232}Th (with 5.9% , 6.3 % and 2.7 % yield) and also through the thermal neutron activation of ^{98}Mo. By ^{99}Tc disintegration a single β particle with maximum energy of 0.292 MeV is emitted.

To measure the ^{99}Tc content in different waste categories it has to be separated and concentrated from the radioactive waste matrices. Among the analytical methods used for the ^{99}Tc separation (anion exchange, solvent extraction, precipitation/co-precipitation, electro-deposition and reductive deposition on active metals) extraction chromatography on TEVA Resin represents the most used method in the ^{99}Tc analysis that was adopted and optimized in RATEN ICN [13]. This method combines the selectivity of liquid-liquid extraction with the easy operation on column chromatography. TEVA Resin is an anion resin with a high affinity for TcO_4 - from low acid and even basic solution. Re carrier is used.

The separation and concentration method is based on optimizing the TCW01 Eichrom Analytical Procedures (Technetium-99 in Water) [14]. This method is applied after waste matrix dissolution using microwave digestion and comprize of the following main steps:

1) TEVA Resin column preparation: after drain the conditioning solution from the column, 5ml of 0.1M HNO3 is added in the column and allow it to drain

2) ^{99}Tc separation on TEVA Resin: the liquid waste sample is loaded on resin; effluent is collected and retained for measurement. The column is washed with 25 ml of 0.01M HNO3 and the rinses are collected for measurement.

3) Stripping of ^{99}Tc from the resin: 99Tc retained on the resin in the previous step is washed with 20 ml of 12M HNO_3 and the effluent is collected in a clean vial for LSC measurement.

4) Evaporation of effluent solution: the effluent solution is slowly evaporated at 80 degrees Celsius to get a volume of around 10 ml and from this apropriate volume is sampled for LSC measurement.

This method was optimized for radioactive waste matrices such as spent ion exchange resins, liquid effluents, solid radioactive waste (textile, cellulose, plastic).

7. – Method used for measurement of ^{129}I in radioactive waste

^{129}I is a long-lived radionuclide (T1/2=1.5x10^7 years), with a lower specific activity (1.73x10^{-4} Ci/g) that requires an adequate analytical method for its separation and purification. Extraction chromatography method based on Cl-resin was adapted to be used for iodide separation and purification. The selectively retained capacity of silver loaded Cl-resin is relying on the strength of precipitation/-complexation reactions between the silver cations and anions (Cl- and I-) present in the radioactive waste samples and also on the competition amongst these anions [15]. The chloride and iodide anions from the radioactive waste sample are retained on the resin from slightly acidic or neutral media, forming sparely or insoluble Ag complexes. The elution of these anions from the Cl-resin is performed using different solubility of AgI and AgCl in eluent solutions: at the beginning, chloride is easily eluted from the resin using SCN^- solutions whereas iodide remains fixed, followed by the iodide mobilizing using an elevated concentration solution of S^{-2}.

The separation process of ^{129}I - from a radioactive waste comprises of the following main steps [16]:

- I. Matrix dissolution
- II. Column preparation:
  - a. Placing the column in a proper rack and discarding the conditioned solution (HNO_3 1M)
  - b. Loading the column with 5 mL of 1M H_2SO_4 and soaking for at least 45 minutes, followed by the discarding of acid solution
  - c. Adding 2 mL of Ag, loading solution (50mg $AgNO_3$ in 5ml 1M H2SO_4) onto the column, allowing to stand for several min (45 min) for Ag uptake, and discarding the effluent
  - d. Rinsing the column with 5 ml 1M H_2SO_4 and discarding the effluent
- III. Loading of liquid radioactive waste sample obtained in step I to remove the interfering radionuclides
  - a. Loading column with 10 ml sample and allowing to drain
  - b. Rinsing the column with 5 ml 1M H2SO4 and collecting de effluent for measure
  - c. Rinsing the column with 2x5ml distillate water
  - d. Rinsing the column with 10 ml 1
- IV. Separation of 129I- fraction

- a. Adding 5 ml 0.35M K_2S and collecting the I- fraction in a clean volumetric flask

Iodine stable or radioactive carrier can be added to determine in the same test the recovery yield, or separate tests can be performed.

As for other radionuclides, this method was optimized for different radioactive waste matrices.

8. – Method used for measurement of actinide content in radioactive waste

Actinides can occur in radioactive waste as a result of contamination from nuclear fuel failure, in the reactor, or from testing activities of the failed fuel in the post-irradiation examination laboratory.

To be able to measure the actinide content by alpha spectrometry, the radioactive waste sample has first to be mineralized, process carried out to eliminate the organic matrix and to solubilize the cations present in the radioactive waste samples. Through different physico-chemical processes, the radionuclides in the sample are brought into aqueous solutions compatible with the radiochemical separation method.

The procedure implemented in RATEN ICN for the sequential separation of actinides is optimized following the Eichrom ACW03 procedure whose main steps are described below and schematically represented in fig. 3.

The separation of U and Th from other actinides is achieved using the UTEVA resin. For this, the Pu and Am are reduced to the valence state 3 by the addition of ferrous sulphamate to the liquid sample. When liquid radioactive waste sample passes through the UTEVA column, tetravalent U, Th and Np will be retained, while Am and Pu remain in the aqueous phase. Solutions of 3M HCl and 5M HCl - $C_2H_2O_4$ 0.05M are used to elute the Th and Np from the resin and 1M HCl solution for uranium elution.

The separation of americium from plutonium is carried out by using TRU resin, which shows a specific affinity for tetravalent actinides and for Am(III) in HNO_3 environments with a concentration exceeding 2 mol/l. For the most efficient retention of Pu on the resin, it is necessary to oxidize it by using sodium nitrite. The selective separation of Pu and Am is carried out in HCl medium, where the affinity of the resin for americium decreases a lot, but the tetravalent actinides remain strongly bound to the organic extractant. The concentrated hydrochloric acid solution used to strip the americium is evaporated to dryness, and the residue is re-dissolved with HCl solution with a concentration lower than 1M. The stripping of plutonium from the TRU resin is carried out by complexing it with ammonium oxalate.

9. – Conclusions

Radioactive waste characterisation is an important activity in the radioactive waste management and developing an adequate characterisation programme allow selecting the most appropriate processing technologies and disposal routes and also avoid accumulating waste forms that may not accomplish the waste acceptance criteria for a specific storage or disposal facility.

Except the methods for ^{14}C and ^{3}H measurement in radioactive waste, the methods optimized in RATEN ICN for other DTM radionuclides are based on extraction chromatography combined with ion exchange processes using selective resins. To apply these

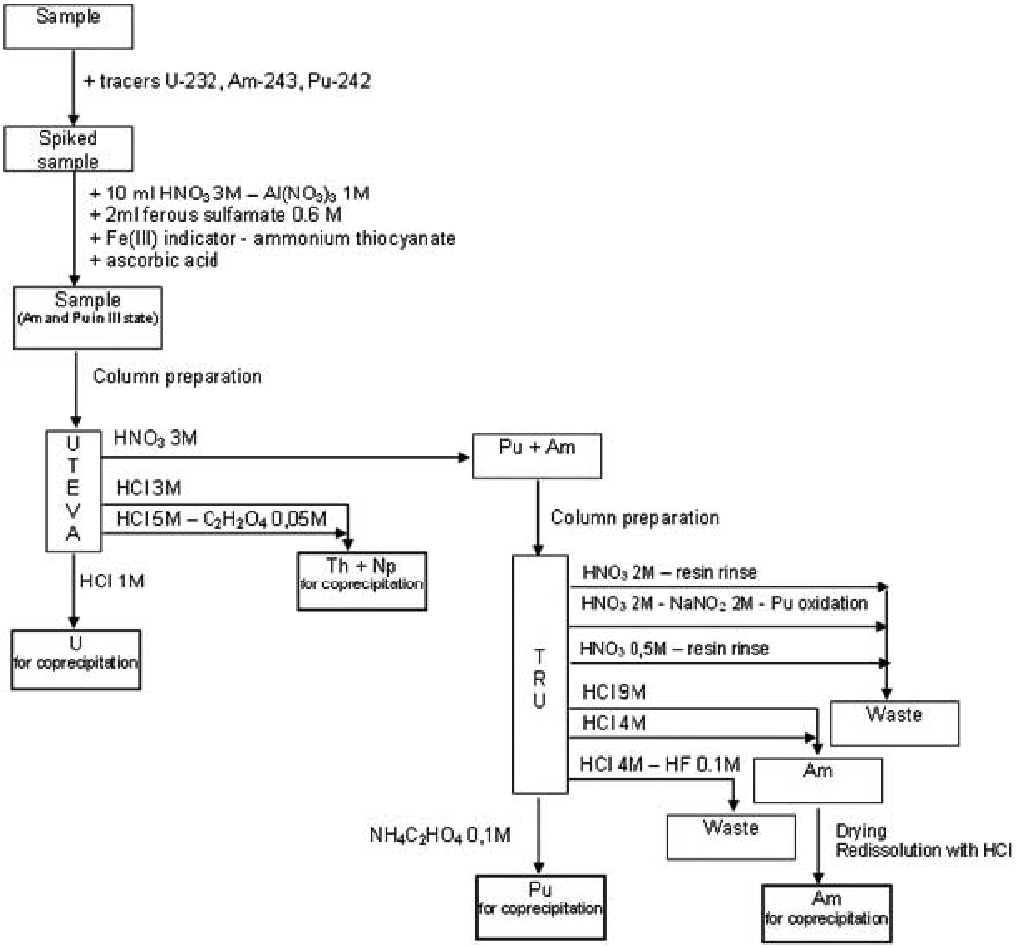

Fig. 3. – Schematically representation of the sequential method for actinide separation and purification

methods, the radioactive waste matrix is mineralized by acid digestion using a microwave digestion system.

The methods base on extraction chromatography combined with ion exchange processes are cost-effective and efficient and are not time consuming, while keeping the secondary waste generated quite low.

The combination of acid stripping wet oxidation for ^{14}C separation and purification from different categories of waste matrix is an efficient method allowing separately determination of inorganic and organic ^{14}C.

As tritium is present in radioactive waste mainly in the form of tritiated water, the techniques adopted for its separation and purification is simple or under vacuum distillation. An in-house passive sampler was designed and proved to be very efficient in determination of tritium activity concentration on metallic radioactive waste.

Depending on the complexity of the waste matrix and the radionuclide of interest, the recovery yield for the methods described above ranges between 70 and 99% but are very reproducible and can be used to correct the concentration activity of the interest radionuclide in a given waste matrix.

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