
Progress on Standardization of Radioanalytical
Methods for determination of important radionu-
clides for environmental assessment and waste
management in Nordic nuclear industry

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Abstract

The NKS-B STANDMETHOD project was launched in January 2014, aiming to standardize the radioanalytical method for the determination of important radionuclides difficult to measure in Nordic industry. The present status of radioanalysis in Nordic laboratories is reviewed and presented in this report. A review article on this topic was prepared to be published in a peer review journal, and an intercomparison exercise was implemented for determination of ^{63}Ni and ^{55}Fe in three types of water samples: spiked water, reactor coolant water and an acid digested filter from a nuclear reactor. Seven labs participated in the intercomparison and reported their analytical results of ^{63}Ni . Different analytical methods used by the labs, and the results are discussed in this report. The intercomparison results for ^{63}Ni agree relatively well for the spiked water, but a big variation of the results was observed for the real reactor coolant water and for the digested filter sample. This indicates that the methods used in some labs could not remove interfering nuclides to a sufficient extent. An improvement of the analytical method of some labs and a follow-up intercomparison exercise are proposed.

Key words

Radioanalysis, ^{63}Ni , intercomparison, reactor water, radionuclide

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Progress on Standardization of Radioanalytical Methods for determination of important radionuclides for environmental assessment and waste management in Nordic nuclear industry

—NKS-B StandMethod project report 2014

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Abstract

The NKS-B STANDMETHOD project was launched in January 2014, aiming to standardize the radioanalytical method for the determination of important radionuclides difficult to measure in Nordic industry. The present status of radioanalysis in Nordic laboratories is reviewed and presented in this report. A review article on this topic was prepared to be published in a peer review journal, and an intercomparison exercise was implemented for determination of ^{63}Ni and ^{55}Fe in three types of water samples: spiked water, reactor coolant water and an acid digested filter from a nuclear reactor. Seven labs participated in the intercomparison and reported their analytical results of ^{63}Ni . Different analytical methods used by the labs, and the results are discussed in this report. The intercomparison results for ^{63}Ni agree relatively well for the spiked water, but a big variation of the results was observed for the real reactor coolant water and for the digested filter sample. This indicates that the methods used in some labs could not remove interfering nuclides to a sufficient extent. An improvement of the analytical method of some labs and a follow-up intercomparison exercise are proposed.

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1. Introduction

Although the number of nuclear power plants has not increased in the Europe and North America for some years, nuclear power is still one of the major sources of the electricity supply, accounting for 13.5% of the world's electricity production. In the developing countries, like China and India, nuclear power plants are being built, and there are currently a total of 430 commercial nuclear power reactors with more than 370 GWe of total capacity all over the world up to 2013. In addition, there are 240 research reactors and 180 vessels' nuclear power reactors in operation (World Nuclear Association 2013). In the Nordic countries, there are three nuclear power plants in Sweden (Forsmark, Oskarshamn and Ringhals) and two in Finland (Loviisa and Olkiluoto) with a total of 14 power reactors. Additionally, three research reactors (JEEP II and HBWR in Norway and FiR-1 in Finland) are in operation.

The environmental safety of the nuclear installations is a sensitive and critical issue for the public and authorities. In the past years, an increased and more restrictive environmental assessment program has been required by the authorities. Because of this, some radionuclides that are difficult to measure, such as ^{14}C , ^{63}Ni , and ^{55}Fe , have been added to the list of routine measurements in the monitoring programmes for discharges and process waters in the nuclear power plants and research reactors in the Nordic countries as well as in other EU countries. (Commission Recommendation 2003). A further requirement for the measurement of these radionuclides in environmental samples may also be ahead. During the operation of the reactor, various types of radioactive waste are produced, such as ion exchange resins. These waste samples need to be characterised before treatment or final repository. The major challenge is then to determine the pure beta emitters (e.g. ^{14}C , tritium, ^{55}Fe , ^{63}Ni) and the alpha emitting radionuclides (e.g. $^{238, 239, 240}\text{Pu}$). In these cases efficient chemical separation has to be carried out before measurement.

Since a large number of research and power reactors were built several decades ago, these reactors, as well as many other nuclear facilities, are being or will be decommissioned rather soon (IAEA, 2009). In the Nordic countries, some nuclear reactors and nuclear facilities have already been closed. A few of them have been decommissioned, while other are going to be decommissioned (Andersson et al. 2006). In Denmark, two research reactors (DR1 and DR2) have been decommissioned until 2008, and the third reactor is being decommissioned (DD, 2009). In Sweden up to 2013, six nuclear reactors have been shut down. Only one of them (R1 at Royal Institute of Technology) has decommissioned, while the remaining five (two research reactors in Studsvik R2 and R2-0, power reactors in Ågesta, and Barsebäck (1 and 2) are currently being or are going to be decommissioned (SKB 2007, Decommissioning of nuclear facilities, 2014). In Norway, two small research reactors (JEEP I and NORA) were closed in the 1960's and have been decommissioned to stage 2 and 3 (NEA 2009). In Finland, a research reactor FiR-1 at Otaniemi, Espoo is going to be closed, and the decommissioning of

this reactor is planned (Viitanen, 2010). Three NKS seminars on decommissioning have been organized in 2005 and 2010 and 2013 in Halden, Norway, indicating an increased concern and interest in decommissioning in the Nordic communities.

In the decommissioning of nuclear facilities, the radioactivity of various radionuclides has to be determined for characterisation of the produced waste. In this process, besides the nuclear fuel, the decommissioning waste consists of many different types of materials, such as biological shield concrete, ion exchange resins, graphite and metals. Various fission and activation product radionuclides with sufficiently long half-lives will remain in these wastes. Of these radionuclides, gamma emitters such as ^{60}Co , ^{152}Eu , and ^{137}Cs can be instrumentally determined by gamma spectrometry. However, the pure beta and alpha emitters have to be chemically separated from the matrix and other radionuclides before measurement, which is the major challenge in the characterisation of these wastes. ^{14}C , ^3H , ^{55}Fe , and ^{63}Ni are of special concern because of their high concentration.

In the past years, some radiochemical analytical methods have been developed in Nordic laboratories for the determination of some of these radionuclides in some types of samples, such as ion exchange resin, waste water, graphite and concrete (Hou, 2005a, b, c, 2007; Holm, 1993; Sidhu 2006, Skwarzec et al. 2001; Persson, 2005; Persson et al. 2000; Magnusson 2007; Magnusson et al. 2008a, b; Koivula et al. 2003; Lehto & Hou 2009, Eriksson et al. 2013), and a good competence in the Nordic labs has been built in this field. However, since there are many different types of nuclear waste from decommissioning activities, the reported methods do not cover all types of waste. There is always a need to develop new methods for different types of samples. In addition, because no suitable reference material of a similar matrix to the real samples is available with certified values to these radionuclides, it is not easy to validate a new method.

In the recent years, some Nordic laboratories including Technical University of Denmark (DTU), and Studsvik have been worked on radioanalysis for the characterization of decommissioning waste. It was recognized that although the quality assurance is a very important issue in this work, the lack of suitable reference materials makes quality assurance challenging to implement. This can be helped by the organization of intercomparison exercise.

An NKS project (Radiowaste) on radiochemical analysis of difficult to measure radionuclides for waste management in decommissioning and repository was carried out in 2010-2011. Through this project, an initial collaboration network among some Nordic labs working in this field was established, and some demands for analytical techniques were identified. To meet this need, a few new analytical methods have been developed (Hou, 2010, 2012). Two NKS workshops on radiochemical analysis have been successfully organized in 2009 and 2013, respectively at Risø, Denmark, with 30-35 young participants of each workshop (Hou, 2009, 2013a). These activities enhanced the competence of the Nordic labs in

radiochemical analysis, especially the education of young scientists. In the recent workshop held 2-6 September 2013, 31 participants from Nordic laboratories, especially from the nuclear industry, took part, showing a strong interest in radioanalysis in the Nordic countries.

During the 2013 workshop, it was recognized that in nuclear power plants in Sweden (Forsmark, OKG and Ringhals NPPs) and Norwegian research reactor (in IFE), ^{63}Ni , ^3H and ^{14}C in the discharge water, coolant water, effluent to air or ion exchange resin of the reactor have been routinely determined, while ^{55}Fe will likely be added to the list of routine analysis in these facilities in the near future. A problem in the determination of ^{63}Ni in reactor coolant water with high radioactive cobalt (^{58}Co and ^{60}Co) has been reported because of insufficient separation of radioactive cobalt from the Ni fraction. To overcome this problem, a long waiting time has sometimes been applied before the measurement to reduce the interference from the relatively short-lived ^{58}Co (half-life 70.9 days). Meanwhile, an interference correction by measuring the amount of ^{60}Co in the separated solution may be applied (Eriksson et al. 2013; Englund & Öhlin, 2013). In addition, because no standard reference material of similar matrix to the real process samples is available for ^{63}Ni analysis, there is a need to find a good approach for quality control of this analysis. Although several Nordic laboratories are working on the measurement of ^{63}Ni in waste and environmental samples, but different analytical procedures are used. During the NKS workshop on Radioanalytical Chemistry in 2013, the participants from Nordic nuclear industries and research institutions show a strong demand in seeking or establishing a standard analytical method for the analysis of these radionuclides.

The NKS STANDMETHOD project aims to establish a close collaboration between research institutions, radiation protection authorities and nuclear industries in Nordic countries; to validate the presently applied radioanalytical methods through intercomparison exercises; to standardize the analytical methods for the important radionuclides for routine analysis in the laboratories of the nuclear industry/research institutes/authorities; and to identify the new needs of the nuclear industry. The latter may require the development of new methods for the radiochemical analysis of nuclear waste.

To fulfill this goal, the project is proposed to be carried out during 2-3 years. This report presents the progress of the project in the first year (2014).

2. Summary of the activities and progress of the project

All planned activities and tasks for 2014 were well fulfilled according to the proposal. They are summarized below.

2.1 Project meetings

A kick-off project meeting was held on 27th March 2014. All seven partners participated in the meeting, where the following issues were discussed: (1) General introduction of the project; (2) Introduction of the status and requests/needs of radioanalysis in their lab by each partner; (3) Discussion on intercomparison exercises: sample types, radionuclides, sample size, time schedule (delivery date, deadline to report), preparation of samples, delivery of samples, participants, etc. (4) Establishment of Nordic network in radioanalysis; (5) A Review article on present status of radioanalysis in Nordic countries, content, organization, time schedule, suggestions. For the inter-comparison exercise, all partners agree that only ^{63}Ni in three water samples (one spiked water and two real water from one of Nordic NPP) will be included, the ^3H and ^{14}C in concrete sample planned in the proposal will not be performed in this project period due to 2 reasons: (1) routine analysis of coolant water and other water samples for ^{63}Ni is requested in the most of Nordic NPPs, while no suitable quality control method is applicable due to lack of relevant standard reference material, an intercomparison of the present methods used in Nordic lab is very useful and urgent; (2) Only 2 labs in the Nordic countries can do analysis of concrete for ^3H and ^{14}C , and the staff in one of these 2 labs who was responsible for this analysis is not available during the project period.

The second project meeting was held on 13rd November at Forsmark NPP. The following issues were addressed: (1) Presentation of all results of the intercomparison; (2) Discussion of the results, remarks from each laboratory; (3) Brief conclusion on the intercomparison (problems and possible resolution); (4) Discussion on further intercomparison exercises in the next term of the project; (5) Discussion on the review article; (6) Discussion on the final report of project 2014. (7) Discussion on the extension of the project for 2015.

2.2 Intercomparison exercise

An intercomparison exercise was organized. Three water samples were prepared:

- artificial water sample spiked with radionuclides,
- fresh reactor coolant water sample from a nuclear power reactor, and
- acid digested filter for integrated sampling of reactor coolant water.

Seven Nordic laboratories participated in the intercomparison exercise and reported analytical results for ^{63}Ni ; three laboratories also analyzed and reported results for ^{55}Fe . The overall results of the intercomparison exercise were discussed during the second project meeting. The detailed discussion on this issue will be given in the next section. It has been agreed that the methods used in the Nordic laboratories are suitable for the determination of ^{63}Ni in water samples containing less of interfering radionuclides such as ^{60}Co and ^{58}Co . Fresh reactor water samples may however contain a high level of

^{58}Co , and an improved method then needs to be applied for some labs. Based on the results of this intercomparison exercise, it is agreed to organize another intercomparison exercise in the second phase of the project in 2015, to verify the improved method in order to establish a Nordic standard/reference method for the determination of ^{63}Ni in water from a nuclear reactor. Only three of the seven laboratories determined ^{55}Fe , but the results from those three laboratories agree well in general. It is agreed by the participating laboratories that ^{55}Fe will be included in the second intercomparison exercise in 2015.

2.3 Evaluation of the present status of radioanalysis in the Nordic laboratories

To evaluate the present status of radioanalysis in Nordic laboratories, the seven project partners have prepared a summary of the routine work and capacities of radioanalysis in their own laboratories, highlighted their requirements and demands for radioanalysis, and problems they meet during their radioanalytical work, as well as their perspectives on radioanalysis. The summaries cover: (1) Requirement for radioanalysis or application of radioanalysis in each Nordic country (institution), (2) Radionuclides to be analyzed in each laboratory; (3) Measurement method used for different radionuclides in each laboratory, and their suitability and limitation against the demand; (4) Methods/procedures used for chemical separation/purification of the analyte (target radionuclide), or group of radionuclides; (5) Problems, difficulties, and requirements of the routine radioanalysis; (6) Facilities/instruments used for radioanalysis or measurement of radionuclides; (7) Needs for new analytical methods or radionuclides to be covered in each Nordic institution/country in near future; (8) New application field of radioanalysis besides monitoring, decommissioning, environmental radioactivity, and waste repository in each Nordic institution/country; (9) Perspectives on radioanalysis.

2.4 A journal article on present status of radioanalysis in the Nordic countries

Based on the summaries prepared by each partner, the present status of the radioanalysis in the Nordic countries is reviewed, and further demands by the Nordic nuclear industry in the field of radioanalysis are clarified. A review article on the topic is being prepared and reviewed by all partners. This article is going to be submitted in to a peer-reviewed journal (Journal of Radioanalytical and Nuclear Chemistry) for publication in 2015. The content of the article is outlined below: (1) Introduction; (2) Radionuclides measured in Nordic labs and their application; (3) Techniques used for measurement of major radionuclides; (4) Chemical procedures for separation of important radionuclides, (5) Recent progress on radioanalysis of radionuclides and perspectives.

2.5 Nordic network in radioanalysis

A close network in radioanalysis has been established, which is mainly based on e-mail communication. All colleagues from Nordic laboratories working within radiochemical analysis are invited to join. The colleagues from the partner laboratories are the key persons in the network. The major function of this network is: (1) to exchange and share experiences in radioanalysis of radionuclides; (2) to be a source of help to solve problems in radioanalysis; (3) to spread and exchange information and research progress among the Nordic labs; (4) to find suitable partners for possible EU or international projects. The network was very welcome by many colleagues, especially those from Nordic nuclear industry. It was noticed that there is a lack of routine communication among radiochemists / radioanalysts from different organizations. At the same time, experience exchange is a very effective tool to develop the work in any analytical laboratory.

3. Present status of radioanalysis in the Nordic labs

In the past decades, a number of radiochemical analytical methods have been developed in the Nordic laboratories for the determination of various radionuclides difficult to measure in different types of samples. These methods have been used for many years for analysis of various environmental samples such as soil, sediment, aerosol, seawater, fresh water, vegetation, food, and biological samples for environmental radioactivity, radioecology and environmental tracer studies (Chen et al. 2001, Vesterbecka & Ikäheimonen 2005; Salonen & Hukkanen 1997; Salonen 1993a, 1993b). Some methods have also been developed in Nordic laboratories for analysis of samples from nuclear facilities such as ion exchange resins, waste water, filters, graphite, concrete and metals (Hou, 2005a, b, c, 2007; Holm, 1987, 1993, 2002; Sidhu 2006, Skwarzec et al. 2001; Persson, 2005; Persson et al. 2000; Magnusson 2007; Magnusson et al. 2008a, b; Koivula et al. 2003; Lehto & Hou 2009, Eriksson et al. 2013) for monitoring of radioactivity in nuclear power reactors and characterization of nuclear waste for decommissioning. Table 1 lists the major Nordic laboratories where radiochemical analysis of radionuclides difficult to measure nuclides are carried out. In all Nordic laboratories listed in Table 1, high resolution gamma spectrometers are available for the measurement of most gamma emitters. For the determination of pure beta and alpha emitters although some labs also equipped with mass spectrometry instruments (such as ICP-MS) for measurement of long-lived radionuclides, radiometric methods are still the major ones, including liquid scintillation counting and alpha spectrometry.

As shown in Table 1, the most often measured radionuclides difficult to measure are ^3H , ^{14}C , ^{55}Fe , ^{63}Ni , $^{89,90}\text{Sr}$, $^{238,239,240}\text{Pu}$, ^{241}Am , $^{243,244}\text{Cm}$. Table 2 summarizes the most common methods used in the Nordic labs for the determination of ^3H , ^{14}C , ^{55}Fe , ^{63}Ni and ^{90}Sr .

Table 1. Major Nordic laboratories involved in radiochemical analysis of radionuclides difficult to measure

Country	Organization	Purpose of analysis	Main radionuclides
Denmark	Technical University of Denmark	Environmental radioactivity, radioecology, environmental trace, characterization of decommissioning waste, emergency preparedness	^3H , ^{14}C , ^{36}Cl , ^{41}Ca , ^{55}Fe , ^{63}Ni , $^{89,90}\text{Sr}$, ^{99}Tc , ^{129}I , ^{210}Po , ^{210}Pb , ^{222}Rn , $^{226,228}\text{Ra}$, Isotopes of U, Th and Pu, ^{237}Np , ^{241}Am , ^{244}Cm , gross alpha, gross beta
Finland	Radiation and Nuclear Safety Authority (STUK)	Environmental radioactivity, bioassay of radioactivity, emergency preparedness	^3H , ^{14}C , $^{89,90}\text{Sr}$, ^{99}Tc , ^{210}Po , ^{210}Pb , ^{222}Rn , $^{226,228}\text{Ra}$, ^{234}U , ^{235}U , ^{238}U , $^{232,230,228}\text{Th}$, $^{239,240}\text{Pu}$, ^{241}Am , gross alpha, gross beta
	University of Helsinki	Environmental radioactivity and radioecology, analysis of nuclear waste	^3H , ^{14}C , ^{41}Ca , $^{89,90}\text{Sr}$, ^{210}Po , ^{210}Pb , ^{222}Rn , $^{226,228}\text{Ra}$, Isotopes of U, Th and Pu, ^{237}Np , ^{241}Am , gross alpha, gross beta
	Loviisa NPP	Monitoring of radioactivity in the power plant, discharges and surrounding environment	^3H , ^{14}C , ^{63}Ni , ^{55}Fe , gross alpha, gross beta
	Olkiluoto NPP	Monitoring of radioactivity in the power plant, discharges and surrounding environment	^3H , ^{14}C , gross alpha, gross beta
Norway	Institute for Energy Technology (IFE)	Environmental radioactivity, waste management.	^3H , $^{89,90}\text{Sr}$, ^{210}Po , ^{210}Pb , ^{222}Rn , $^{226,228}\text{Ra}$, Isotopes of U, Th and Pu, ^{237}Np , ^{241}Am , gross alpha, gross beta
	Norwegian Norwegian University of Life Sciences	Environmental radioactivity, radioecology, environmental trace,	$^{89,90}\text{Sr}$, ^{99}Tc , ^{210}Po , ^{210}Pb , ^{222}Rn , $^{226,228}\text{Ra}$, isotopes of U, Th and Pu, ^{237}Np , ^{241}Am
	Norwegian Radiation Protection Authority (NRPA)	Environmental radioactivity and radioecology, environmental trace, emergency preparedness	$^{89,90}\text{Sr}$, ^{99}Tc , ^{1210}Po , ^{210}Pb , ^{222}Rn , $^{226,228}\text{Ra}$, Isotopes of U, Th and Pu, ^{237}Np , ^{241}Am , gross alpha, gross beta
Sweden	Studsvik Nuclear AB	Waste management, characterization of decommissioning waste, emergency preparedness	^3H , ^{14}C , ^{36}Cl , ^{55}Fe , ^{63}Ni , $^{89,90}\text{Sr}$, ^{99}Tc , ^{129}I , ^{210}Po , $^{226,228}\text{Ra}$, Isotopes of U, Th and Pu, ^{237}Np , ^{241}Am , ^{242}Cm , ^{244}Cm
	Forsmark NPP	Monitoring of radioactivity in the power plant, discharges and surrounding environment	^3H , ^{14}C , ^{63}Ni , ^{90}Sr , ^{235}U , ^{238}Pu , $^{239+240}\text{Pu}$, $^{242, 243+244}\text{Cm}$, gross alpha, gross beta
	Oskarshamn NPP (OKG AB)	Monitoring of radioactivity in the power plant, discharges and surrounding environment	^3H , ^{14}C , ^{55}Fe , ^{63}Ni , $^{89,90}\text{Sr}$, ^{238}Pu , $^{239,240}\text{Pu}$, ^{241}Am , ^{244}Cm , $^{243,244}\text{Cm}$, gross alpha
	Ringhals NPP	Monitoring of radioactivity in the power plant, discharges and surrounding environment	^3H , ^{14}C , ^{63}Ni , $^{89,90}\text{Sr}$, ^{238}Pu , $^{239,240}\text{Pu}$, ^{241}Am , ^{244}Cm , $^{243,244}\text{Cm}$, gross alpha, gross beta
	Lund University	Radioecology, environmental trace, emergency preparedness	^{14}C , ^{55}Fe , ^{63}Ni , $^{89,90}\text{Sr}$, ^{99}Tc , ^{238}Pu , $^{239,240}\text{Pu}$, ^{241}Am , ^{244}Cm , $^{243,244}\text{Cm}$, gross alpha, gross beta

Table 2 Analytical method used in Nordic labs for determination of ^3H , ^{14}C , ^{55}Fe , ^{63}Ni and ^{90}Sr

Nuclide	Sample type	Separation Method	Laboratory
^{63}Ni	Water	TRU-Ni resin	Forsmark, Oskarshamn and Ringhals NPP, STUK
		Precipitation-ion exchange-Ni resin	DTU Nutech,
	Resin, evaporation waste	Ni resin	Loviisa NPP
	Concrete, graphite, metals	Acid leaching, hydroxides precipitation, anion exchange chromatography and extraction chromatography	DTU Nutech
^{55}Fe	Water	TRU chromatography	Oskarshamn NPP
		Precipitation-anion exchange chromatography	DTU Nutech,
	Resin, evaporation waste		Loviisa NPP
	Concrete, graphite, metals	Acid digestion/leaching, hydroxide precipitation, anion exchange chromatography	DTU Nutech
^3H	Water	Distillation	Most of labs
	Soil sample (concrete, graphite, soil)	Combustion using Packard Oxidizer	DTU Nutech, STUK
		Combustion using tube furnace	Studsvik
	Air	^3H collector (as tritium water and organic form)	Oskarshamn, Ringhals, Forsmark, Olkiluoto NPP
		molecular sieve (water vapor)	Loviisa NPP
^{90}Sr	Water	Carbonate precipitation, $\text{Ca}(\text{OH})_2$ precipitation, Sr (Ra, Ba, Pb) precipitation, $\text{Y}_2(\text{C}_2\text{O}_4)_3$ precipitation for ^{90}Y	DTU Nutech
		Oxalate precipitation, chromate precipitation to remove Pb, carbonate precipitation of Sr, extraction chromatography using Sr resin, LSC measurement	STUK
		Cation exchange chromatography + extraction chromatography using Se resin	Oskarshamn, Ringhals NPP
		Direct solvent extraction of Y	Forsmark NPP

	Environmental and biological samples, Concrete, graphite, metals	Ashing, acid digestion (HCl, or HNO ₃ +HCl), separation using the same procedure as for water sample	DTU Nutech, STUK,
¹⁴ C	Water	Evaporation followed by combustion	DTU Nutech
	Soil sample (concrete, graphite, soil metals)	Combustion using Packard Oxidizer	DTU Nutech, STUK
		Combustion using tube furnace	Studsvik
	Air	¹⁴ C collector (NaOH)	Oskarshamn, Ringhals, Forsmark, Olkiluoto NPP
molecular sieve		Loviisa NPP	

4. Intercomparison exercise on determination of ⁶³Ni and ⁵⁵Fe in water samples

4.1 Intercomparison samples

Three water samples were prepared: (1) DTU-1: Spiked water samples containing ⁶³Ni, ⁵⁵Fe, ⁶⁰Co, and ¹³⁷Cs in 1.0 liter of HNO₃ solution was, prepared by Technical University of Denmark; (2) Forsmark-1 reactor coolant water collected at Forsmark NPP unit 1, 1.0 liter samples were acidified with HNO₃, this sample contains various activation product radionuclides, mainly ³H, ⁵¹Cr, ⁵⁸Co, ⁶⁰Co, ^{110m}Ag, ⁹⁹Mo, ¹²²Sb, ¹⁴⁴Ce; (3) Forsmark-2: A solution of acid digested filter used for sampling of reactor coolant water at unit 1, 5 ml solution of HNO₃ and H₂SO₄ in a 20 ml vial, which also contains various radionuclides, mainly ⁵⁴Mn, ⁵⁸Co, ⁶⁰Co, ⁶⁵Zn. The two real samples were prepared by the partner at Forsmark NPP. Except for the different matrices for the samples Forsmark-1 and -2, these two samples also differ with composition of radionuclides: Forsmark-1 was collected directly from a sampling point, containing short-lived radionuclides produced in the reactor, while the filter used to prepare Forsmark-2 samples was collected from reactor and leaved for several months before treatment. This affects the amount of short-lived radioactivity in the samples. All 3 samples were prepared and delivered to all intercomparison participants in June 2015. Table 3 summarizes the features of the three intercomparison samples.

Table 3 Composition of the intercomparison samples

Code	Sample	Matrix	Major radionuclides contained
DTU-1	Spiked water	1.0 L in HNO ₃	⁶³ Ni, ⁵⁵ Fe, ⁶⁰ Co, ¹³⁷ Cs
Forsmark-1	Reactor coolant water from Forsmark NPP	1.0 L water with HNO ₃	⁶³ Ni, ⁵⁵ Fe, ³ H, ⁵¹ Cr, ⁵⁸ Co, ⁶⁰ Co, ^{110m} Ag, ⁹⁹ Mo, ¹²² Sb, ¹⁴⁴ Ce
Forsmark-2	Acid digested filter	5 mL in HNO ₃ and H ₂ SO ₄	⁶³ Ni, ⁵⁵ Fe, ⁵⁴ Mn, ⁵⁸ Co, ⁶⁰ Co, ⁶⁵ Zn

4.2 Analytical Methods used in Nordic labs for determination of ⁶³Ni and ⁵⁵Fe

Two methods are used in Nordic labs for the separation of ⁵⁵Fe from water samples: extraction chromatography or ion exchange chromatography as shown in Fig. 1 and 2. In the first method (Fig. 1), the solution is evaporated to dryness after the addition of stable iron as carrier and yield tracer, the residue is then dissolved with HNO₃, and the solution is adjusted to a HNO₃ concentration of 8 M. The prepared solution is loaded to an extraction chromatographic column using TRU resin, the column is rinsed with 8M HNO₃ to remove all matrix elements and interfering radionuclides, iron on the column is finally eluted using 2M HNO₃ solution. In the second method (Fig. 2), stable Ni, Eu, Co, and Cr are also added, besides the stable iron carrier and yield tracer, as hold back carriers in order to improve the decontamination of the interfering radionuclides which exist in the samples in a low mass concentration. ⁵⁵Fe in the solution is first separated from the solution by hydroxide precipitation (as Fe(OH)₃). In this case, other transit metals are also precipitated with iron; therefore a further separation using anion exchange chromatography is followed. The hydroxides precipitate is dissolved using concentrated HCl, and the concentration of HCl is adjusted to 9 M, the prepared solution is loaded on a strong base anion exchange column, the column is rinsed with 9M HCl and 4M HCl to remove the most of interfering radionuclides and matrix elements. All element which do not form anions with Cl⁻ such as alkali and earth alkaline metals, rare earth elements, Ni²⁺, Cr³⁺, etc. will pass through the column, metals such as ⁶⁰Co²⁺, Cu²⁺, etc. which form weak anion complex in high concentration of Cl⁻ but not in low concentration of Cl⁻ solution will be removed during rinsing with 4M HCl. Iron on the column is finally eluted using 0.5 M Cl solution, while the strong binding elements such as all anion (I⁻, Br⁻, etc.) and metals forming strong anion complex with Cl⁻ at low HCl concentration solution such as Zn²⁺, will remain on the column. The separated iron in the eluate is finally evaporated to dryness and dissolved in H₃PO₄ solution to obtain a colorless iron solution due to formation of complex of Fe³⁺ with H₃PO₄,

which is used for measurement of ^{55}Fe using liquid scintillation counter by counting the Auger electrons of ^{55}Fe decay.

For the separation of ^{63}Ni from water samples, three methods are used in the Nordic labs. The first and second methods are extraction chromatography using one or two sequential Ni columns (Fig. 1), respectively; while the third method is a combination of ion exchange chromatography (for separation of Fe also) and extraction chromatography using Ni resin (Fig.3). In the first two methods, some labs use the effluent from the separation of ^{55}Fe using TRU column, but others directly use the original water sample. The water sample or eluate is first evaporated to dryness and then dissolved in 1 M HCl solution for separation using Ni column. Therefore, the loading solution contains almost all matrix elements and interfering radionuclides. In the third method, Ni was first separated using hydroxide precipitate and then ion exchange chromatography (Fig. 2), most of matrix elements and interfering radionuclides (including Co, Cr) were removed before extraction chromatography using Ni resin. In the first and third method, one Ni column was applied for separation; while in the second method, two sequential Ni columns were applied for separation of ^{63}Ni to get better purification of ^{63}Ni . For separation of ^{63}Ni using Ni column, ammonium citrate is first added to the prepared solution in 1 M HCl to complex most of transit element to prevent from formation of hydroxides, then ammonium solution is added to adjust pH to about 9, the solution is then loaded to the Ni column, and washing with a ammonium citrate-ammonium solution (pH 9) to remove most of matrix elements and interfering radionuclides. ^{63}Ni remaining on the column as complex of Ni-DMG (dimethylglyoxime) is finally eluted using 3M HNO_3 , which can remove the DMG and the DMG-Ni complex from the resin, the eluate is evaporated to near dryness. In the second method, the evaporated eluate is prepared in ammonium citrate solution again and used for separation using another Ni column as the same procedure as the first Ni column (Fig.1). Finally the evaporated eluate is used for measurement of ^{63}Ni using liquid scintillation counting.

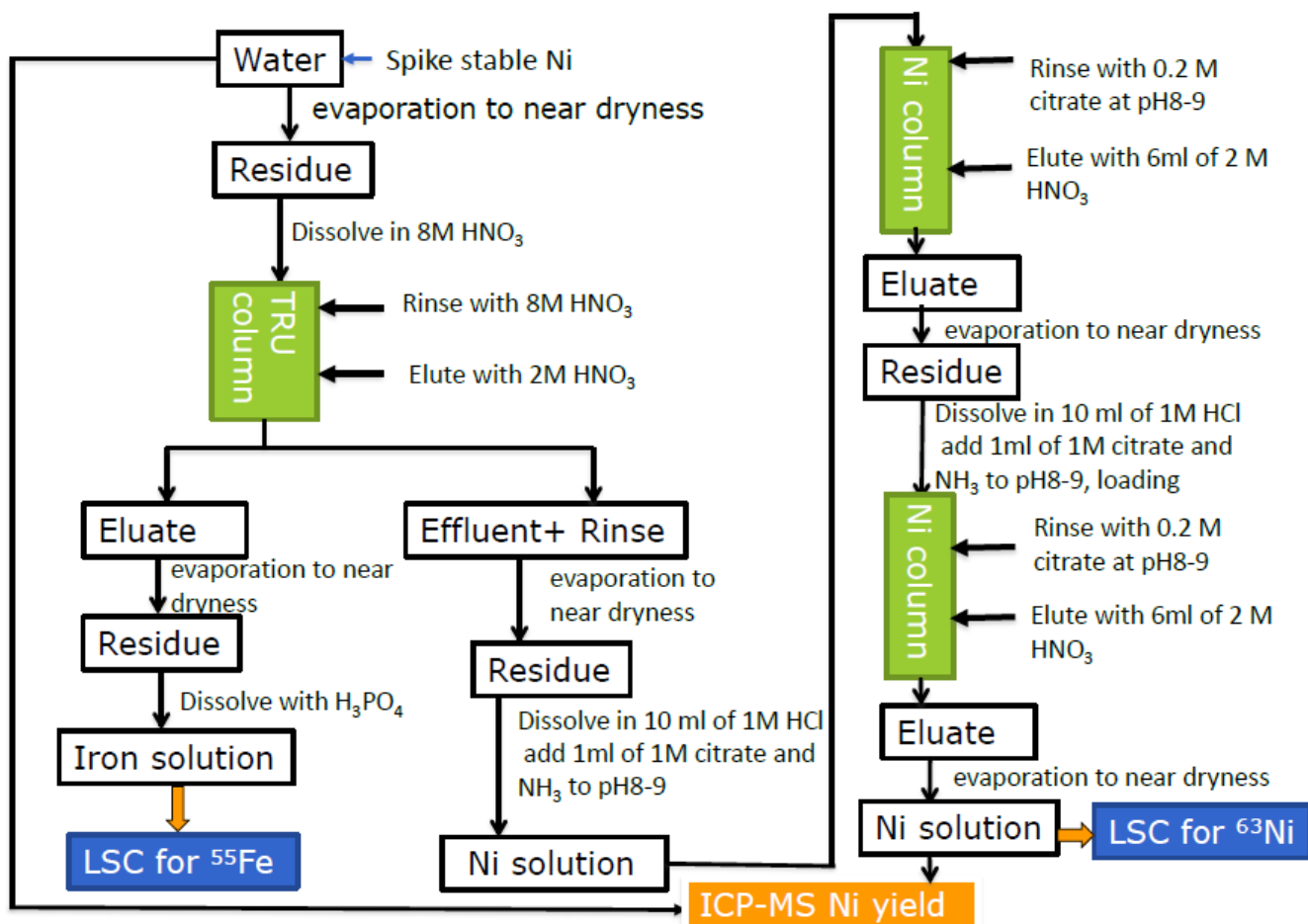


Fig. 1 Analytical procedure for determination of ^{63}Ni and ^{55}Fe in water sample using extraction chromatography (Eriksson, et al. 2013)

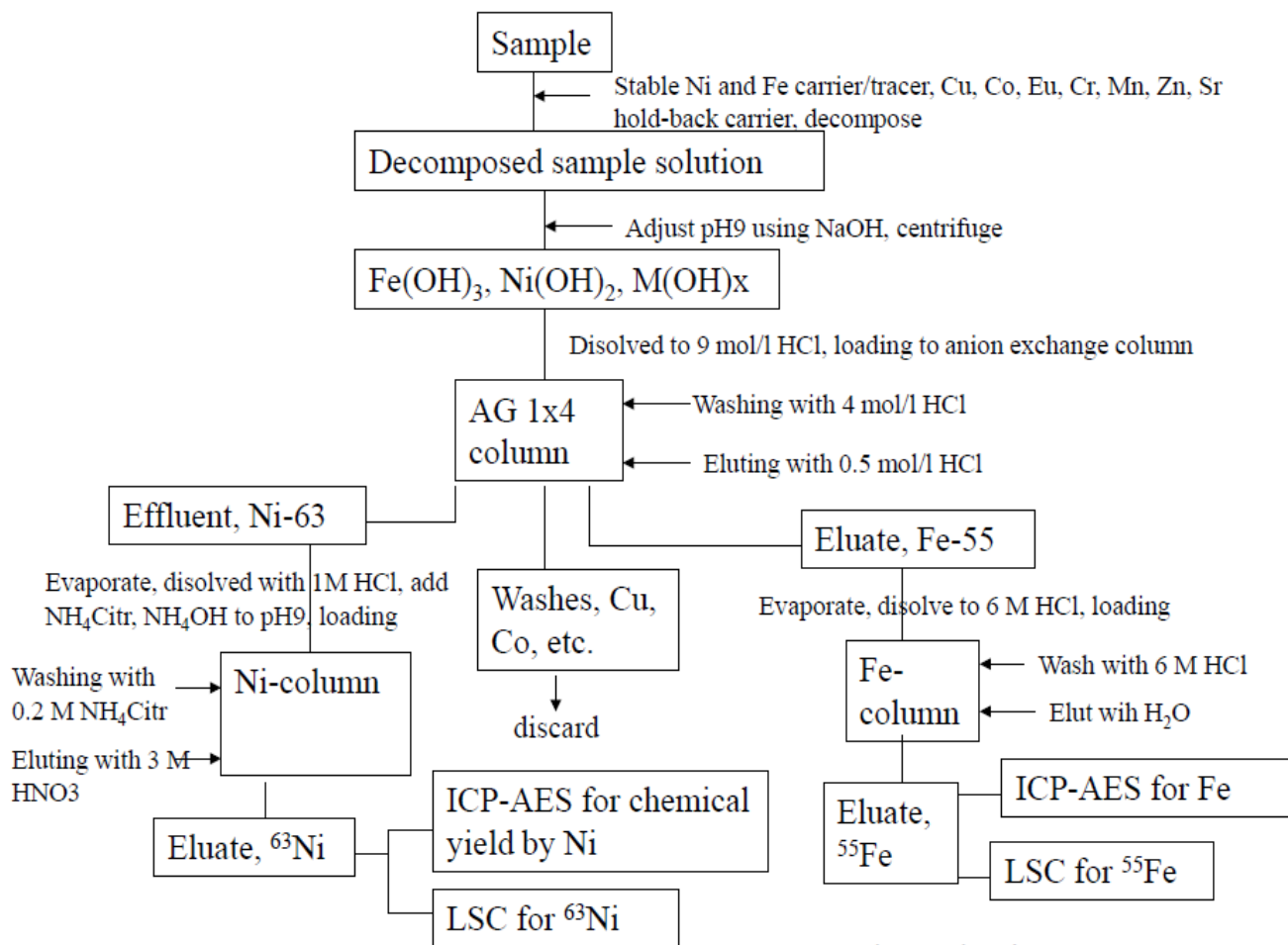


Fig. 2 Analytical procedure for determination of ^{63}Ni and ^{55}Fe using combined techniques of precipitation, ion exchange chromatography and extraction chromatography (Hou, et al. 2005)

4.3 Results of the intercomparison

Six partner labs and an invited lab (Loviisa NPP) participated in the analysis of the three intercomparison water samples. Seven laboratories reported their analytical results for ^{63}Ni , and three labs reported ^{55}Fe results. Fig. 3-5 show the analytical results of ^{63}Ni and Table 4 presents the analytical results of ^{55}Fe . It should be noted that the determination of ^{55}Fe in the intercomparison samples was not requested, therefore only three labs reported this.

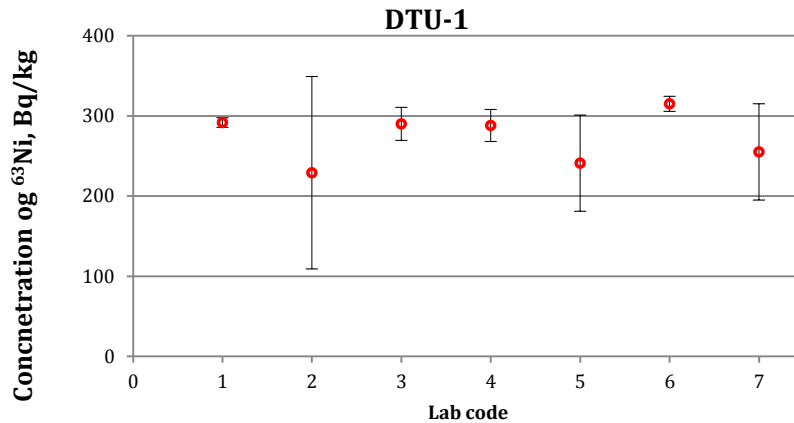


Fig.3 Intercomparison results of ⁶³Ni in DTU-1 sample (Spiked water)

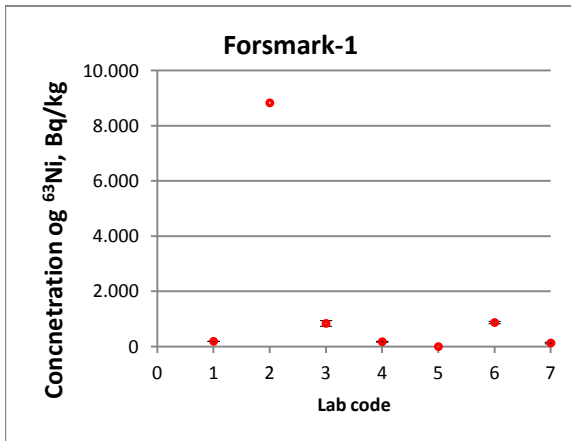


Fig.4 Intercomparison results of ⁶³Ni in Forsmark-1 samples (Reactor coolant)

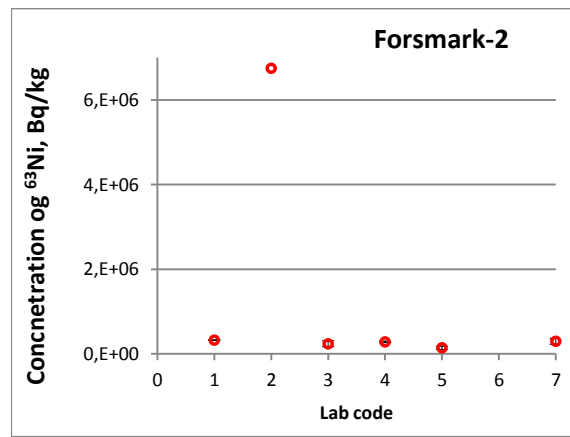


Fig. 5 Intercomparison results of ⁶³Ni in Forsmark-2 sample (digested filter)

Table 4 Analytical results of ⁵⁵Fe concentration (Bq/kg) in three intercomparison water samples

Lab code	DTU-1		Forsmark-1		Forsmark-2	
	⁵⁵ Fe conc.	Uncertainty	⁵⁵ Fe conc.	Uncertainty	⁵⁵ Fe conc.	Uncertainty
1	311.23	10.54	7.33	1.40	1.26×10 ⁵	0.04×10 ⁵
4 *	292.00		9.86		1.15×10 ⁵	
6	326.80	45.72				
Spiked value	312.68	6.26				

* No analytical uncertainty was reported.

For the spiked sample (DTU-1), the analytical results of ^{63}Ni from all laboratories vary from 229 Bq/kg to 315 Bq/kg, and the spiked concentration of ^{63}Ni in this sample is 290.2 ± 3.2 Bq/kg. All results are acceptable within relative bias of 25%, and the statistic test also shows that no difference between the reported results and the spiked ^{63}Ni concentration (Fig. 3). For the results obtained using the second and third method, the reported uncertainties are relatively small (<10%), while the results reported with a larger uncertainty (25-52%) were obtained by the first method (One Ni column separation).

For the two real reactor water samples (Forsmark-1 reactor coolant water and Forsmark-2 acid digested filter), the ^{63}Ni analytical results show large variation (Fig. 4 and 5). Among all reported results, the data in two samples reported by lab 2 are more than 20 times higher than others; this might be attributed to an unsuitable method to remove the interfering radionuclides. The two reactor water samples were measured by gamma spectrometry, which show that ^{60}Co concentrations are 9700 Bq/kg for Forsmark-1 and 2.2×10^6 Bq/kg for Forsmark-2. Forsmarks-1 (coolant water) also contains a high concentration of ^{58}Co (2.0×10^4 Bq/kg) and other activated corrosion products such as ^{51}Cr , ^{54}Mn and ^{144}Ce . It was confirmed by gamma measurement that the separated Ni fraction using the first method contains large amount of ^{58}Co and ^{60}Co . Therefore the application of the first method for ^{63}Ni separation without correction for ^{60}Co and ^{58}Co in the Ni fraction is likely the main reason for the extremely high value of ^{63}Ni reported by lab 2.

Fig. 6 and 7 show the ^{63}Ni results in the two Forsmark samples excluding the lab 2. It can be seen that the variation of the ^{63}Ni concentration in Forsmark-1 is still very large. The data reported by lab 3 and 6 are about 4 times higher than those by lab 1, 4 and 7, while the value reported by lab 5 is two orders of magnitude lower than the value reported by other labs. The method 1 was used by lab 3, 5 and 6, where the interference from ^{60}Co was corrected by measurement of ^{60}Co content in the separated ^{63}Ni fraction, while no correction of interference from ^{58}Co was done. Because high ^{58}Co concentration was measured in the fresh coolant water sample (Forsmark-1), the interference of ^{58}Co is significant. This is likely the reason for the high ^{63}Ni value reported by lab 3 and 5 in this case. The very low value reported by lab 5 might be attributed to the high ^{60}Co content in the separated ^{63}Ni fraction and a large uncertainty in the correction, which causes an over-subtraction of the contribution of ^{60}Co . The ^{63}Ni results for the two Forsmark samples reported by lab 1, 4 and 7 agree relatively well. The second method (two Ni columns) was used by lab 4 for Ni separation, and the third method (precipitation plus anion exchange plus extraction chromatography) was applied by lab 1 and 6 for separation of ^{63}Ni . Fig. 8 and 9 show the gamma spectra of the separated ^{63}Ni fractions using the third method. It can be seen that no ^{60}Co , ^{58}Co as well as other gamma emitters were present in the separated ^{63}Ni fraction. It has also been reported that the decontamination factors for most of interfering radionuclides including ^{60}Co in the third method is higher than 10^5 (Hou et al. 2005). The good agreement for ^{63}Ni between the

second and the third methods indicate that separation using two sequential Ni columns, combined with interference correction, can give a reliable analytical result of ^{63}Ni in reactor water samples.

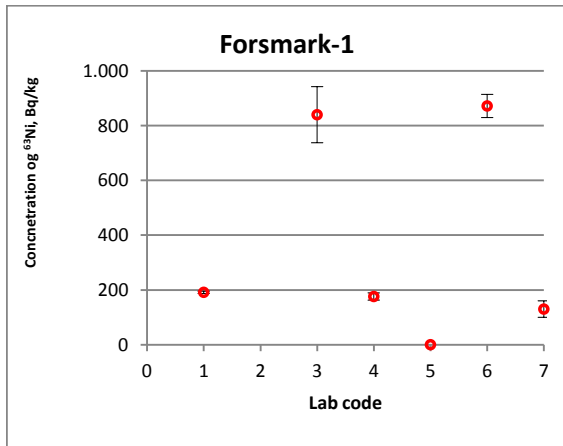


Fig. 6 Intercomparison results of ^{63}Ni in Forsmark-1 excluding the data of the lab 2

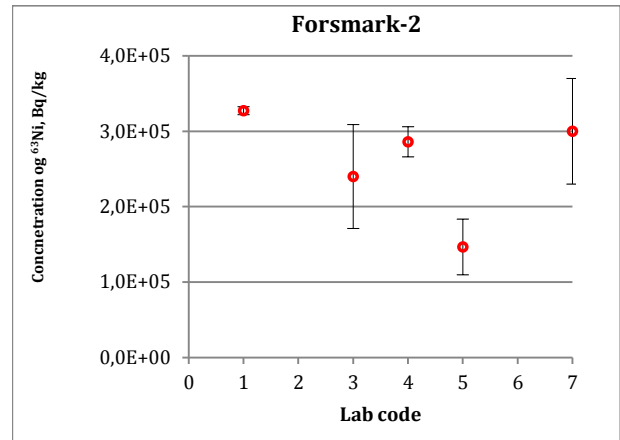


Fig. 7 Intercomparison results of ^{63}Ni in Forsmark-2 excluding the data of the lab 2

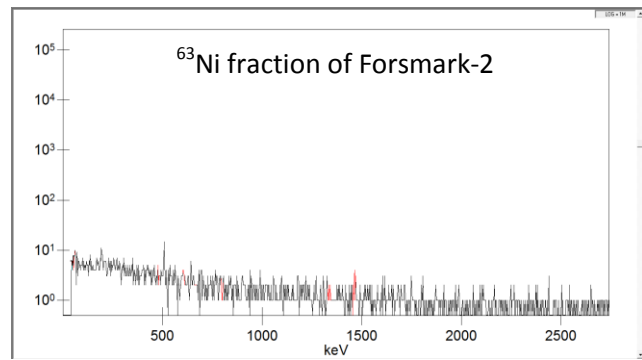
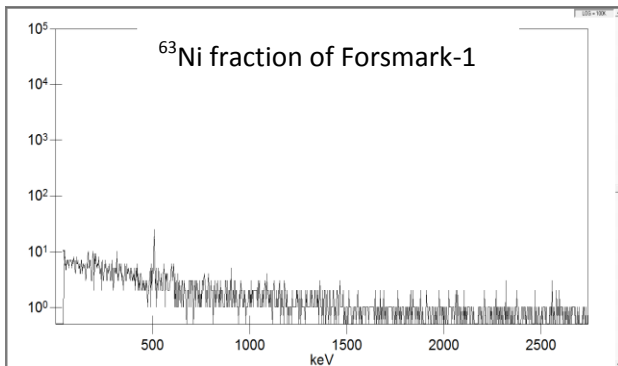
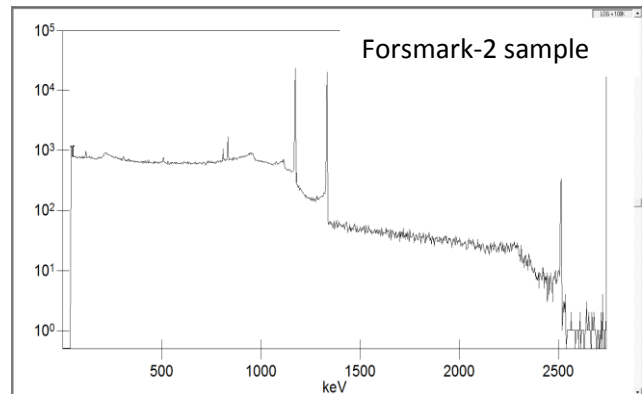
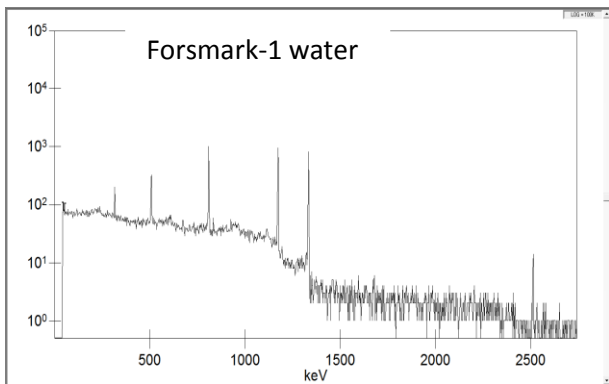


Fig. 8 γ spectra of Forsmark-1 water sample (upper) and separated ^{63}Ni fraction

Fig. 9 γ spectra of Forsmark-2 sample (upper) and separated ^{63}Ni fraction

The analytical results of ^{55}Fe reported by the three labs agree well in general. Two methods were used for separation of ^{55}Fe from the matrix and interfering nuclides. It was observed that ^{51}Cr was present in the separated ^{55}Fe fraction, indicating unsatisfactory separation of ^{51}Cr from the iron fraction for the first method. However, with correction of the ^{51}Cr interference, the final result of ^{55}Fe by this method (reported by lab 4) is comparable with the results from the second method, although the estimation of the analytical uncertainty by this method was not given. Because the intercomparison exercise in this project focuses on the determination of ^{63}Ni , not many labs reported the analytical results of ^{55}Fe . Therefore a comprehensive evaluation of the analytical methods for ^{55}Fe may become a future task.

4. Conclusion and perspective

Throughout this project, the collaboration in radioanalysis among Nordic labs, especially laboratories from Nordic nuclear power plants and other nuclear industry, was strengthened. The present status of the radioanalysis in the Nordic laboratories was reviewed, and a draft for a review article was prepared for publication in a peer reviewed journal. It was recognized that different methods were used in the Nordic labs for the determination of important radionuclides. In order to evaluate these methods, a Nordic intercomparison exercise has been organized with three water samples (one spiked sample and two real samples from Forsmark nuclear power plant) for ^{63}Ni . The results showed that the measured ^{63}Ni values in the spiked sample agree relatively well for all laboratories. However, the measured values in the two real samples are significantly scattered by a factor of more than 10. This variation is mainly attributed to the analytical methods used in some laboratories, which could not effectively remove interfering radionuclides, and/or had no suitable correction for these interferences. These results also highlight that it is important and necessary to improve the present method used in the Nordic industry for enhancing analytical quality to get a reliable estimation of the impact of the radioactive discharges from the nuclear industry. A modification and improvement of the present method used in the Nordic industry may be needed, and a follow-up intercomparison to evaluate the improved method for determination of ^{63}Ni is proposed. In addition, it is proposed to fully include the determination of ^{55}Fe in the next intercomparison exercise, to be able to evaluate the present methods used in the Nordic industry and to possibly provide improvements in reliability for the method for ^{55}Fe determination in reactor coolant water in the future.

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Title	Progress on Standardization of Radioanalytical Methods for determination of important radionuclides for environmental assessment and waste management in Nordic nuclear industry
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Abstract max. 2000 characters	The NKS-B STANDMETHOD project was launched in January 2014, aiming to standardize the radioanalytical method for the determination of important radionuclides difficult to measure in Nordic industry. The present status of radioanalysis in Nordic laboratories is reviewed and presented in this report. A review article on this topic was prepared to be published in a peer review journal, and an intercomparison exercise was implemented for determination of ⁶³ Ni and ⁵⁵ Fe in three types of water samples: spiked water, reactor coolant water and an acid digested filter from a nuclear reactor. Seven labs participated in the intercomparison and reported their analytical results of ⁶³ Ni. Different analytical methods used by the labs, and the results are discussed in this report. The intercomparison results for ⁶³ Ni agree relatively well for the spiked water, but a big variation of the results was observed for the real reactor coolant water and for the digested filter sample. This indicates that the methods used in some labs could not remove interfering nuclides to a sufficient extent. An improvement of the analytical method of some labs and a follow-up intercomparison exercise are proposed.
Key words	Radioanalysis, ⁶³ Ni, intercomparison, reactor water, radionuclide