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## NKS-B RadWorkshop 2018 proceedings

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April 2019

## **Abstract**

The NKS-B RadWorkshop 2018 (Workshop on Radioanalytical Chemistry for Nuclear Decommissioning and Waste Management) was held at Risø, Denmark, during 8-12 October 2018. The workshop consisted of 3-day invited lectures and presentations from participants and 2-day lab practice by very experienced professionals. A number of highly reputed experts from authorities, research institutes and nuclear industries were invited to give state-of-the art lectures in different aspects in nuclear decommissioning and waste management with the focus on radioanalytical chemistry. The lab training demonstrated the radiochemical analysis of alpha emitters (Pu-239, 240 and Am-241) and beta emitters (Fe-55, Ni-63) in nuclear decommissioning materials. 90 participants from 41 international organisations attended the workshop.

## **Key words**

Radioanalytical chemistry, workshop, nuclear decommissioning, waste management

Final report for NKS-B Nordic ICP (AFT/B(18)3)

## **NKS-B RadWorkshop 2018 proceedings**

### **Radioanalytical Chemistry for Nuclear E and Waste Management**

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<sup>4</sup> Radiation and Nuclear Safety Authority (STUK), Finland

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## **NKS-B RadWorkshop**

# **Radioanalytical Chemistry for Nuclear Decommissioning and Waste Management**

**8-12 OCTOBER 2018**

**DTU Risø Campus  
Frederiksborgvej 399, Roskilde, Denmark**



### **Workshop Organizers:**

Technical University of Denmark, Center for Nuclear Technologies (DTU Nutech) - Denmark  
Swedish Radiation Safety Authority (SSM) - Sweden  
Norwegian Radiation Protection Agency (NRPA) - Norway  
Radiation and Nuclear Safety Authority (STUK) - Finland



### **Organizing Committee:**

Jixin Qiao, DTU Nutech (Chairman)  
Mats Eriksson, SSM  
Torbjørn Gäfvert, NRPA  
Kaisa Vaaramaa, STUK  
Henrik Efraimsson, SSM  
Xiaolin Hou, DTU Nutech  
Szabolcs Osváth, DTU Nutech  
Marte Varpen Holmstrand, NRPA

### **Workshop Secretary:**

Helle Tofte Holm & Birgitte Sindholt, DTU Nutech  
Tel. +45 4677 5300,  
E-mail: [htho@dtu.dk](mailto:htho@dtu.dk); [bisi@dtu.dk](mailto:bisi@dtu.dk)



## Introduction

A large number of research and power reactors have been built in the 1950-1960s and 1960-1970s, respectively. These reactors, as well as many other nuclear facilities are reaching their designed age and are going to be decommissioned <sup>1</sup>. In recent years, more and more nuclear reactors in Nordic countries are under decommissioning or going to be decommissioned. In Denmark, one research reactor (DR3) is still under decommissioning <sup>2</sup>. In Sweden, 7 nuclear reactors are under decommissioning or preparing for decommissioning and 2 of the 8 operating units will be closed within the next 2 years. In Norway, two small research reactors (JEEP I and NORA) were closed in 1960s and was partly decommissioned <sup>3</sup>. In June 2018 the Institute for Energy Technology (IFE) also decided to permanently close the research reactor in Halden. In Finland, a research reactor FiR-1 at Otaniemi, Espoo has been closed in 2015 and the decommissioning of this reactor is planned <sup>4</sup>. In addition, some other nuclear facilities are also under decommissioning, for example, hot cells at Risø site in Denmark <sup>5</sup>.

The current situation in the Nordic nuclear society sets high demands in boosting capacities of performing radiochemical analysis for nuclear waste characterization or establishing new radiochemical laboratories among Nordic countries with relatively short time frame. To achieve this, there is a need for communication, knowledge/experience sharing and cooperation among different parties in the Nordic nuclear communities. Robust characterization strategies must be clearly aligned with defined characterization objectives and the relevant process control or waste acceptance criteria <sup>6</sup>. This requires collective inputs from waste producers, regulators and the characterizations supply chain. The waste producers need to clarify the decommissioning and waste management strategies and indicate the technical needs in waste characterization. The regulators need to provide adequate and clear requirements for safe decommissioning and management of radioactive wastes, and to have adequate competence for supervision of the operations, including characterization of facilities and waste. The researchers in academic institutes need to better foresee the challenges in radiochemical analysis and develop fit-for-purpose methods, which can be used in real situations. The students and young scientists need to actively gather knowledge and experience from the senior experts in the field and seek for education and training opportunities in order to cope with the demands in the work.



With this aim, we organized a one-week workshop (RadWorkshop 2018) at DTU Risø Campus, Denmark, during 7-17 October 2018, focusing on radiochemical analysis for nuclear decommissioning and waste management. The workshop hosted 90 participants from 41 international organizations were present at the RadWorkshop 2018 (Appendix 1). The audiences were a good mixture of all parties involved in nuclear decommissioning and waste management including regulators, operators, service partners, researchers, young scientists and students. The workshop program is listed in Appendix 2 and it consisted of two major parts:

- 1) 3-day lectures given by 15 invited professionals and 14 seminar participants. 7 posters were also presented at the workshop. The presentations covered different topics including nuclear decommissioning plans and waste management strategies, theoretical principles of radiochemistry and measurement techniques (alpha, beta and gamma), methodology development and application for different analytical purposes in nuclear decommissioning and waste management, recent development of new separation techniques, materials, detection instrument.
- 2) 2-day lab training by experienced professionals. The lab training covered the radiochemical analysis of alpha emitters (Pu-239, 240 and Am-241) and beta emitters (Fe-55, Ni-63) in nuclear decommissioning materials (e.g., waste water).

An abstract book for the seminar was prepared and handed out to each participant at the seminar (Appendix 3). The presentations at the workshop have been made available on the NKS web site (<http://www.nks.org/en/seminars/presentations/nks-b-radworkshop-2018.htm>).

Questionnaires were prepared and filled by participants to evaluate the result of the workshop organization. The overall feedbacks from participants were very positive, see Appendix 4. The participants found the RadWorkshop 2018 to be a useful forum gathering regulators, operators, researchers, young scientists and students to exchange experience, discuss challenges/difficulties encountered, seek for solutions and possibly formulate new collaborative projects to achieve efficient nuclear decommissioning and waste management. The lab practical training was beneficial to the participants, especially for young students and scientists, to gain some hands-on experience in radioanalytical methodology development and application. All participants are looking forward to future activities relevant to the Radioanalytical Chemistry and nuclear decommissioning. Some aspects and findings of the event will be presented in a future conference.<sup>7</sup>

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7. Jixin Qiao, Szabolcs Osváth, Per Roos, Xiaolin Hou, Kasper G. Andersson: Educational activities in radiochemical analysis at DTU Nutech. In: International Conference on Radioanalytical and Nuclear Chemistry (RANC 2019), May 5-10, 2019, Budapest, Hungary

## Appendix 1 - List of Participants

First name	Last name	Organization
Martin	Amft	Swedish Radiation Safety Authority (SSM)
Maria	Anderot	SKB
Kasper	Andersson	DTU Nutech
Eveliina	Arponen	Hidex
Liga	Bech	DTU Nutech
Aude	Bombard	Triskem
Matthias	Bothe	VKTA - Radiation Protection, Analytics & Disposal Rossendorf Inc.
Marie	Bourgeaux-Goget	Institutt for Energiteknikk
Damien	Braekers	Institute for Radioéléments (IRE ELiT)
Filippa	Bruzell	Cyclife Sweden AB
Bernhard	Bugenhagen	TÜV NORD EnSys
Natalia	Chernikova	Chernobyl NPP
Søren	Dalby	Thermo Fisher Scientific
Fredrik	De La Gardie	Swedish Nuclear Fuel and Waste Management Company
Sylvain	Di Pasquale	IRE-Elit
Tony	Dieudonne	IRE-ELIT
Silvia	Dulanská	Comenius University, Department of Nuclear Chemistry
Ronald	Edler	PerkinElmer LAS (Germany) GmbH
Henrik	Efraimsson	Swedish Radiation Safety Authority
Mikkel Spallou	Eriksen	Dansk Dekommissionering
Mats	Eriksson	Swedish Radiation safety Authority (SSM)
Liselotte	Fornander	OKG AB
Pil	Fredericia	DTU Nutech
Torbjörn	Gäfvert	The Norwegian Radiation Protection Authority
Øyvind	Helgerud	Institutt for energiteknikk
Shogo	Higaki	Isotope Science Center, the University of Tokyo
Lise	Holk	DTU Nutech
Helle Tofte	Holm	DTU Nutech
Marte Varpen	Holmstrand	Norwegian Radiation Protection Authority
Xiaolin	Hou	DTU Nutech
Stefan	Isaksson	Gammadata Instrument AB
Ingunn	Isdahl	Institutt for energiteknikk
Taneli	Iso-Markku	Dept. of Chemistry, Radiochemistry, University of Helsinki
Jonatan	Jiselmark	Swedish Radiation safety Authority (SSM)
Soumaya	Khalfallah	Triskem
Jannicke	Kolmskog	PerkinElmer Sverige AB
Trine	Kolstad	Norwegian Radiation Protection Authority
Anumaija	Leskinen	VTT
Isabelle	Levy	International Atomic Energy Agency
Bao	Li	China Institute for Radiation Protection
Xiaoyan	Li	East China University of Technology
Mu	Lin	DTU Nutech
Yunhai	Liu	East China University of Technology
Vladan	Ljubenov	International Atomic Energy Agency
Irene	Loppersum	NRG Petten
Beatriz	Lourino Cabana	EDF
Jens-Peter	Lynov	DTU Nutech
Zsuzsanna	Macsik	International Atomic Energy Agency

First name	Last name	Organization
Rickard	Malmberg	Westinghouse Electric Sweden AB
Gael	Menard	Nuclear Research and Consultancy Group
Laura	Milelli	EDF Lab
Kari Lye	Moum	Institutt for Energiteknikk
Parvine	Naghchbandi	Statens Strålevern (NRPA)
Kirsten Hjerrild	Nielsen	Danish Decommissioning
Grzegorz	Olszewski	Linköping University, Department of Medical and Health Sciences
Radek	Pošvař	ÚJV Řež, a.s.
Josephine Maria	Prendergast	Norwegian Radiation Protection Authority
Jixin	Qiao	DTU Nutech
Emma-Stina	Raitanen	Fennovoima Oy
Per	Roos	DTU Nutech
Daniele	Roudil	CEA DEN/DMRC/CETAMA
Susanna	Salminen-Paatero	Dept. of Chemistry, Radiochemistry, University of Helsinki
Shaun	Smyth	Meridian Biotechnologies Ltd
Torill Kristin	Solheim	Institutt for energiteknikk
Solveig	Stordal	Institute for Energy Technology
Tommy	Suutari	Cyclife Sweden AB
Qin	Svantesson	Westinghouse Electric Sweden
Jacobus	Swartz	DTU Nutech
Osvath	Szabolcs	DTU Nutech
Cato Christian	Szacinski Wendel	Institutt for energiteknikk (IFE)
Jens	Søgaard-Hansen	Danish Decommissioning
Annika	Tovedal	Swedish Defence Research Agency
Gabriele	Trotta	Istituto Zooprofilattico Sperimentale della Puglia e della Basilicata
David	Ulfbeck	Danish Health Authority, Radiation Protection (SIS), Denmark
Nora	Vajda	RADANAL Ltd.
Petra	Valdezova	SURO, v.v.i.
Jacques	van Wonterghem	DTU Nutech
Mirela-Simona	Vasile	SCK-CEN
Kristina	Veirø	DTU Nutech
Kaisa	Vaaramaa	STUK- Radiation and Nuclear Safety Authority
Nanna	Wenzell	Dansk Dekommissionering
Hanne	Wojtaszewski	DTU Nutech
Qifan	Wu	Tsinghua University
Zhao	Xue	DTU Nutech
Mia	Ylä-Mella	Radiation and Nuclear Safety Authority STUK
Liufang (jenny)	Zhou	Canadian Nuclear Laboratories
Liuchao	Zhu	DTU Nutech
Mikkel	Øberg	Danish Decommissioning
Helene	Öhlin	OKG AB
Hanna	Åberg	OKG AB

## Appendix 2 - Seminar Program

### Welcome Message

Dear colleagues,

I am delighted to welcome you to the Workshop on Radioanalytical Chemistry for Nuclear Decommissioning and Waste Management (RadWorkshop 2018). It will take place during October 8-12, 2018 near the historic town of Roskilde, Denmark.

This is the third RadWorkshop supported by NKS. The two previous RadWorkshop with the scope of general aspects in radioanalytical chemistry took places at Risø, Denmark in 2010 and 2013, respectively. Different from the previous RadWorkshops, the upcoming RadWorkshop 2018 will more focus on radioanalytical chemistry for nuclear decommissioning and waste management.

This workshop will aim to create a network for all the partners in connection with nuclear decommissioning and waste management to review the import role of radioanalytical chemistry played in this domain. I hope that RadWorkshop 2018 is successful to share knowledge, exchange experience and further promote international collaborations, while you also enjoy the lovely autumn in Denmark!

With best regards,



Dr. Jixin Qiao

Technical University of Denmark

Chairman, RadWorkshop 2018 Organizing Committee

## Program Overview

Monday 8 <sup>th</sup> October	Tuesday 9 <sup>th</sup> October	Wednesday 10 <sup>th</sup> October	Thursday 11 <sup>th</sup> October	Friday 12 <sup>th</sup> October
	8:30 Registration and breakfast	8:30 Registration and breakfast		
	9:00 - 10:35 Session 3	9:00 - 10:35 Session 6	9:00 - 12:00 Lab training (department)	9:00 - 12:00 Lab training (department)
10:00 Registration and breakfast	Coffee break	Coffee break		
11:00 - 12:15 Opening session	11:00 - 12:15 Session 4	11:00 - 12:15 Session 7		
Lunch (canteen)	Lunch (canteen)	Lunch (canteen)	Lunch (department)	Lunch (department)
13:30-15:15 Session 1	13:20 - 15:05 Session 5	13:20-15:15 Session 8	13:00 - 16:00 Lab training (department)	13:00 - 16:00 Lab training (department)
Coffee break		Possibility for lab tour		
15:40 - 17:00 Session 2	16:00 - 21:00 Social event			

There is no special poster session.

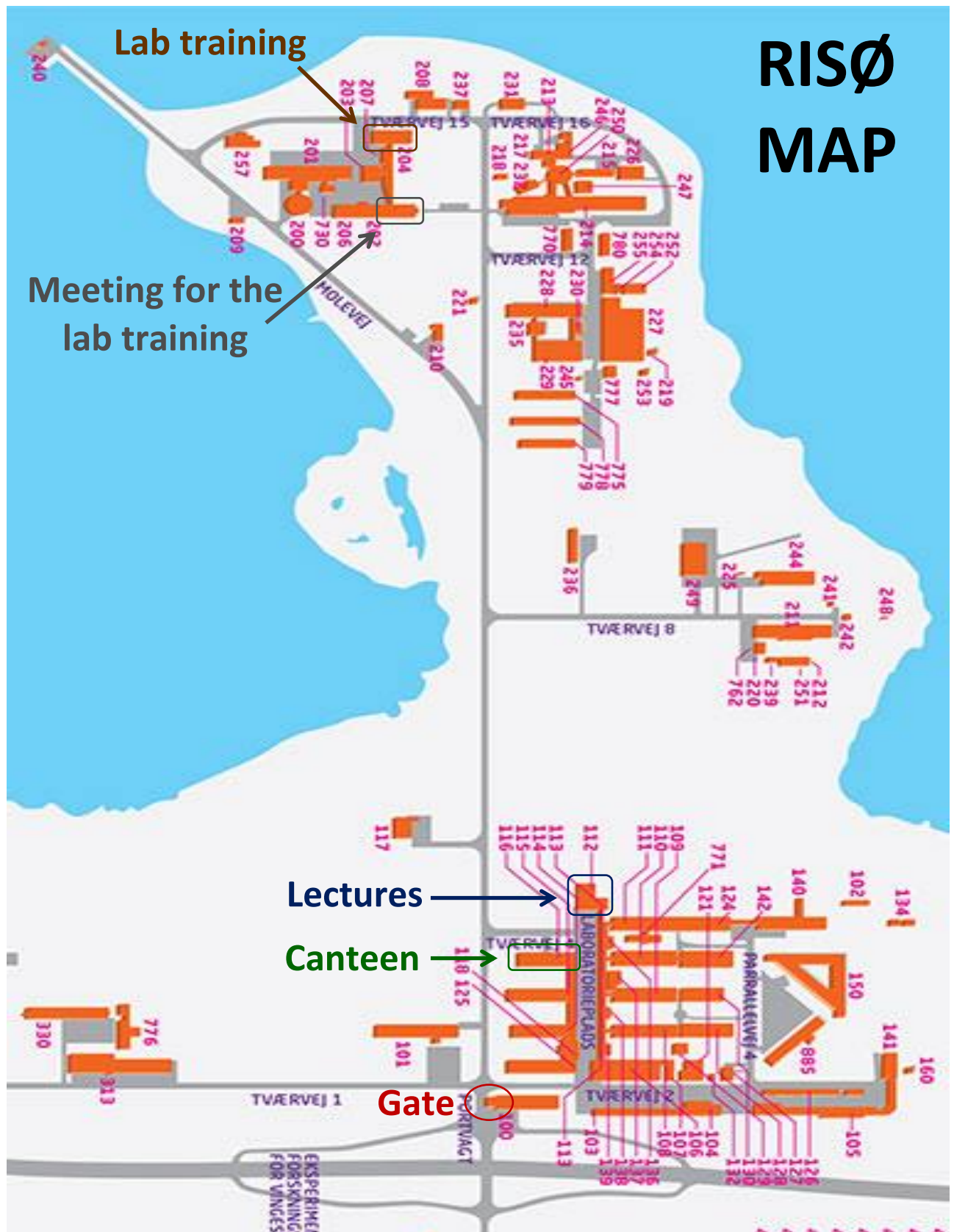
Posters are available from Monday to Wednesday in the entrance hall.

There is also an exhibition of Gammadata (Sweden) in the entrance hall.



Lectures of the workshop will be held in the `Niels Bohr Auditorium` of Building 112.





## Detailed Program

### MONDAY, 8 OCTOBER

10:00 Registration and breakfast

#### Opening session – Chair: Jixin Qiao

11:00 Welcome speech

*(Jens-Peter Lynov, Center for Nuclear Technologies, Technical University of Denmark, Denmark)*

11:15 General introduction of NKS and a view on the use of measurement techniques in emergency management

*(Kasper Grann Andersson, Technical University of Denmark, Denmark / Nordic Nuclear Safety and Research (NKS))*

11:25 General introduction of Radioecology section and the RadWorkshop-2018 program

*(Jixin Qiao, Radioecology, Center for Nuclear Technologies, Technical University of Denmark, Denmark)*

11:40 IAEA Safety Standards on Decommissioning, Clearance of Materials and Release of Sites from Regulatory Control

*(Vladan Ljubenov, International Atomic Energy Agency (IAEA), Division of Radiation, Transport and Waste Safety)*

12:15 Lunch [Canteen]





### Legislation and Status – Chair: Henrik Efraimsson

- 13:30 National legislation on decommissioning, waste management and clearance of materials, buildings and land  
*(David Ulfbeck, Danish Health Authority, Radiation Protection (SIS), Denmark)*
- 14:05 Decommissioning status, challenges and solutions in Sweden  
*(Martin Amft, Swedish Radiation Safety Authority (SSM), Section for operation and decommissioning)*
- 14:40 Decommissioning status, challenges and solutions in Finland  
*(Mia Ylä-Mella, Radiation and Nuclear Safety Authority (STUK), Nuclear Waste and Materials Safeguards Regulation)*
- 15:15 Coffee break

### Methodology – Chair: Torbjørn Gäfvert

- 15:40 The European R&D project INSIDER: Acting on the upstream stages  
*(Daniele Roudil, CEA Nuclear Energy division, Research Department on Mining and fuel Recycling Processes, France)*
- 16:15 The Use of Sampling in Decommissioning Work  
*(Jens Søgaard-Hansen, Mikkel Øberg, Danish Decommissioning)*
- 17:00 End of day

## TUESDAY, 9 OCTOBER

8:30 Registration and breakfast

### Legislation and Status – Chair: Jonatan Jiselmark

- 9:00 Future decommissioning of Norwegian research reactors  
(*Marte Varpen Holmstrand, Norwegian Radiation Protection Authority (NRPA), Norway*)
- 9:35 Overview of decommissioning status, challenges and solutions in Denmark  
(*Kirsten Hjerrild Nielsen, Danish Decommissioning (DD); Waste, Decommissioning and Operation*)
- 10:10 Implementation of the Euratom BSS regulations for clearance in the Swedish legislation  
(*Henrik Efraimsson, Swedish Radiation Safety Authority (SSM)*)
- 10:35 Taking official RadWorkshop photo  
Coffee break

### Methodology – Chair: Kaisa Vaaramaa

- 11:00 Determination of difficult-to-measure nuclides in radioactive wastes of NPP Paks, Hungary  
(*Nóra Vajda, RadAnal Ltd., Hungary*)
- 11:35 Large components, buildings and land – An optimization example on the characterization and clearance process  
(*Jonatan Jiselmark, Swedish Radiation Safety Authority (SSM)*)
- 11:50 Calibration of a  $^{93}\text{Mo}$  standard  
(*Per Roos, Center for Nuclear Technologies (Nutech), Technical University of Denmark (DTU), Denmark*)
- 12:15 Lunch [Canteen]

### Legislation and Status – Chair: Per Roos

- 13:20 Overall planning for decommissioning in Sweden  
(*Fredrik De la Gardie, Swedish Nuclear Fuel and Waste Management Company (SKB)*)
- 13:55 Decommissioning of FiR 1 research reactor: status, challenges and solutions  
(*Anumaija Leskinen, VTT, Finland*)
- 14:30 Decommissioning in Germany: Experience, Current Status and Upcoming Challenges  
(*Bernhard Bugenhagen, TÜV NORD EnSys, Germany*)
- 15:05 End of session

### Social event

- 15:15 Bus transfer to social event.  
Boarding: in front of Building 112 (Niels Bohr Auditorium)
- 16:00 M/S Sagafjord is leaving Roskilde Harbor (Vindeboder 18)
- 18:30 M/S Sagafjord arriving back to Roskilde Harbor
- 21:00 End of social event



## WEDNESDAY, 10 OCTOBER

8:30 Registration and breakfast

### Methodology – Chair: Mats Eriksson

9:00 Radiochemical analysis for characterization of decommissioning waste  
(*Xiaolin Hou, Center for Nuclear Technologies (Nutech), Technical University of Denmark (DTU), Denmark*)

9:35 Analysis of Pu isotopes and Np-237 in seawater by AMS  
(*Isabelle Levy, International Atomic Energy Agency, Marine Environment Laboratory, Monaco*)

10:10 Radiochemical Analysis - experience and challenges  
(*Kaisa Vaaramaa, Radiation and Nuclear Safety Authority (STUK), Finland*)

10:35 Coffee break

### Methodology – Chair: Xiaolin Hou

11:00 Lessons learned – Surprises in Radiological Characterization  
(*Matthias Boethe, VKTA, Germany*)

11:30 Development of rapid and automated radiochemical separation technique  
(*Jixin Qiao, Center for Nuclear Technologies (Nutech), Technical University of Denmark (DTU), Denmark*)

11:45  $^{55}\text{Fe}$ ,  $^{63}\text{Ni}$  and  $^{59}\text{Ni}$  in old nuclear reactor pressure vessel steel samples  
(*Taneli Iso-Markku, Department of Chemistry, Radiochemistry, University of Helsinki, Finland*)

12:00 Sampling and analysis campaign for a rapid assessment of the contamination level in a radiochemistry laboratory  
(*Damien Braekers, Laboratory of Radioactivity Measurement, IRE ELiT, Fleurus, Belgium*)

12:15 Lunch [Canteen]

## Methodology – Chair: Jixin Qiao

- 13:20 Historical overview and current challenges in radionuclide analyses in different waste matrices and materials at SCK•CEN, Belgium  
(*Mirela Vasile, SCK-CEN, Belgium*)
- 13:55 National analytical capabilities to analyze radionuclides in different matrixes occurring in the decommissioning processes  
(*Mats Eriksson, Swedish Radiation Safety Authority (SSM)*)
- 14:20 NRG approach on waste management and radiochemical characterization  
(*Gaël Ménard, Nuclear Research and consultancy Group (NRG), Petten, The Netherlands*)
- 14:45 Measurement of radionuclides and heavy metal elements in maizes for radiological and chemical assessment in a NORM mining site  
(*Wu Qifan, Department of Engineering Physics of Tsinghua University, Beijing, China*)
- 15:00 Determination of  $^{93}\text{Mo}$  (and  $^{94}\text{Nb}$ ) in nuclear decommissioning waste from a nuclear reactor  
(*Szabolcs Osváth, Technical University of Denmark, Center for Nuclear Technologies (DTU Nutech), DTU Risø Campus, Roskilde, Denmark*)
- 15:15 End of day

Those, who do not participate in the laboratory training, are welcome to a 1 hour guided tour in our laboratories. Meeting: in front of Building 112 (Niels Bohr Auditorium) around 15:30.



## THURSDAY AND FRIDAY, 11-12 OCTOBER

### Laboratory training

9:00	Meeting in “Meeting room” of Building 202
9:00	Laboratory exercises in Building 204
12:00	Lunch (sandwich) [“Meeting room” of Building 202]
13:00	Laboratory exercises in Building 204
16:00	End of day

<b>Group 1</b>	<b>Group 2</b>
<b>Alpha emitters</b> ( <sup>239,240</sup> Pu and <sup>241</sup> Am)	<b>Beta emitters</b> ( <sup>55</sup> Fe, <sup>63</sup> Ni)
Nataliya Chernikova	Marie Bourdeaux-Goget
Shogo Higaki	Filippa Bruzell
Taneli Iso-Markku	Sylvain Di Pasquale
Zsuzsanna Mácsik	Silvia Dulanská
Laura Milelli	Ingunn Isdahl
Helene Öhlin	Trine Kolstad
Josephine Maria Prendergast	Beatriz Lourino Cabana
Solveig Stordal	Parvine Naghchbandi
Gabriele Trotta	Radek Pošvař
Petra Valdezova	Tommy Suutari
Qifan Wu	Cato Christian Szacinski Wendel
Eveliina Arponen	Hanna Åberg

If you participate in any of lab trainings, please turn to the registration desk for the handout.



# Abstracts of oral presentations

# **IAEA Safety Standards on Decommissioning, Clearance of Materials and Release of Sites from Regulatory Control**

VLADAN LJUBENOV

*International Atomic Energy Agency (IAEA), Division of Radiation, Transport and Waste Safety*

Decommissioning is the last phase in the lifetime of an authorized facility and it comprises administrative and technical actions taken to allow the removal of some or all of the regulatory controls from a facility. It typically involves decontamination and dismantling of systems, structures and components (SSCs) of the facility, but also might involve demolition of building structures and site cleanup. During decommissioning large amount of different types of materials and waste are typically generated, and it is of critical importance to separate the radioactive materials from those that can be managed, recycled or disposed of as non-radioactive. At the end of decommissioning, it has to be demonstrated that the site meets the defined end state criteria for release from regulatory control and for safe reuse for other purposes.

In order to support planning for decommissioning, safe conduct of decommissioning actions, safe and cost-effective management of decommissioning waste, and release of materials, waste and sites from regulatory control, different types of radiological measurements and surveys are performed prior, during and at the end of decommissioning. Determination of concentrations of radionuclides in SSCs, materials, waste, in the soil and groundwater, including “difficult to measure” radionuclides, is one of the key components for proper radiological characterization and for good decision making in different phases of decommissioning. Thus, radioanalytical chemistry plays an important role in supporting decommissioning and waste management activities.

The International Atomic Energy Agency (IAEA) is the focal point for coordination of international cooperation in relation to peaceful use of nuclear technologies and nuclear energy, nuclear safety and security, and protection of people and the environment from harmful effects of ionizing radiation. The IAEA develops and maintains a set of international Safety Standards, applicable to different facilities and activities, that reflect an international consensus on what constitutes a high level of safety for protecting people and the environment. The Safety Standards cover, among other topics, decommissioning and management of radioactive waste. In addition, the IAEA implement numerous activities and project to assist Member States with application of Safety Standards.

This presentation describes the structure and status of the IAEA Safety Standards on decommissioning, and recent developments related to revision of existing Safety Standards, with an emphasis on developments and challenges related to clearance of materials and release of sites from regulatory control. Some complementary IAEA activities that support the revision of the Safety Standards are also described in the presentation. The needs for radiochemical analyses in relation to radiological surveys for decommissioning and waste management, including clearance of materials and release of sites, are addressed.



# **National legislation on decommissioning, waste management and clearance of materials, buildings and land**

DAVID ULFBECK

*Danish Health Authority, Radiation Protection (SIS), Denmark*

Transposition of Council Directive 2013/59/Euratom of 5 December 2013 laying down basic safety standards for protection against the dangers arising from exposure to ionising radiation (the directive) into Danish legislation was completed on 6. February 2018, by the adoption of 1 new law and 3 new orders, replacing 2 laws and in excess of 15 orders. This complete revision of the Danish legislation on radiation protection introduced specific as well as generic exemption and clearance criteria for materials, buildings and land, and represents a change from a previous legal regime, where only generic clearance criteria were specified. Decommissioning and waste management activities are regulated by the same legal provisions as any other activity subject to licensing, and as such is subject to requirements to perform safety assessments, which serve as a foundation for specifying licensing conditions, and sets the level of regulatory oversight.

The bulk of decommissioning and associated waste management activities in Denmark is related to the decommissioning of the (only) Danish nuclear facilities at Risø, where research reactors, hotcell- and fuel fabrication- and waste management facilities are currently being dismantled. The decommissioning activities undertaken by the responsible operator, Danish Decommissioning, are specifically regulated through operational limits and conditions set for these activities, and based on the safety documentation and – assessments related to the operation and decommissioning of the nuclear facilities. The operational limits and conditions make extensive use of references to relevant IAEA guidance on decommissioning and pre-disposal management of radioactive waste, all within the scope of the legal boundary conditions.

Other users of radioactive sources and ionizing radiation in industry, research and in the medical sector are likewise subject to the requirement of performing safety assessments, including decommissioning and clearance, taking into consideration the principles of the graded approach.

The presentation will provide an overview of the legal requirements associated with decommissioning, waste management and clearance of materials, buildings and land, and will provide examples of how these requirements are applied in practice.

# **Decommissioning status, challenges and solutions in Sweden**

MARTIN AMFT

*Swedish Radiation Safety Authority (SSM), Section for operation and decommissioning*

Today, one third of the Swedish NPP fleet is permanently shut down and the first dismantling projects have been conducted or are ongoing, respectively. By 2020 the two oldest NPPs at the Ringhals site will be shut down as well. Furthermore the small reactor at Ågesta is planned to commence decommissioning by 2020 after 45 years in care and maintenance.

Valuable experiences have been gained from the decommissioning of the uranium milling facility at Ranstad and the materials research reactors at Studsvik.

An overview of these decommissioning projects and the key lessons learned will be given. The regulator's perspective on the foreseeable challenges and possible solutions for conducting up to seven large-scale dismantling and demolition projects in parallel will be discussed.

# Decommissioning status, challenges and solutions in Finland

MIA YLÄ-MELLA

*Radiation and Nuclear Safety Authority (STUK), Nuclear Waste and Materials Safeguards Regulation*

The decommissioning of nuclear facilities has been taken into account in Finnish nuclear legislation. Nuclear Energy Act (990/1987) Section 7g sets the basic requirements for the decommissioning. According to Nuclear Energy Act decommissioning shall be taken into account already in the design phase of the new nuclear facilities, the measures taken for the decommissioning may not be postponed without due cause and the decommissioning has to be conducted according to decommissioning plan approved by the Radiation and Nuclear Safety Authority (STUK). The decommissioning plan has to be updated during operation of the nuclear facility at minimum after every six years. The Licensee is responsible on all costs related to decommissioning and nuclear waste management. The funds for decommissioning are collected in to the state owned Nuclear Waste Management Fund. In beginning of 2018 a revised Nuclear Energy Act introduced a license stage for decommissioning (Section 20a). Nuclear Energy Degree (161/1988) and YVL guide D.4 contains more detailed requirements for decommissioning.

The decommissioning of the Finnish operating nuclear power plants (NPP) is not foreseen in the near future. The decommissioning strategy for the Loviisa NPP is based on immediate dismantling, within eleven years from shutdown while for the Olkiluoto NPP the strategy is deferred dismantling containing a safe storage period of about 30 years prior to dismantling is envisaged. The justification for postponed dismantling is based on a decrease in radioactivity and the availability of nuclear site infrastructure, since the Olkiluoto 3 unit will be operational while the Olkiluoto 1 and 2 units are being dismantled. The disposal plans for waste arising from the decommissioning of the NPPs are based on the extension of the existing on-site repositories for LILW.

In 2012 Finland's first nuclear reactor TRIGA Mark II research reactor (250 kW) was decided to shutdown permanently by the operator, Technical Research Centre of Finland Ltd (VTT). The operation of reactor ended in June 2015. The Environmental Impact Assessment (EIA) procedure for the decommissioning was conducted between 2013 – 2015. VTT applied a license for decommissioning in June 2017. The application is under regulatory review and the safety evaluation report of STUK is awaited to be ready by the end of October 2018. VTT's original plan was to start the decommissioning already in 2019, but the project has still uncertainties related to spent fuel and decommissioning waste management, which may cause remarkable delay for the start of the actual dismantling.

The on-going decommissioning project has shown that there is a need for active communication between the Licensee and the Regulator from the beginning of the decommissioning project. Finland has requirements available for the decommissioning phase, but already now it has been noticed that there is need to update the current legislation and guidance for the forthcoming NPP decommissioning projects. Also the way how the regulatory oversight is performed during decommissioning has to be planned and justified based on the nuclear risks during the decommissioning phase.

## **The European R&D project INSIDER: Acting on the upstream stages**

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The main objective of the European project INSIDER (Improved Nuclear Site Characterization for Waste minimization in Decommissioning under Constrained Environment) is to improve the management of waste coming from nuclear sites under D&D operations, in particular to direct D&D operations, so that the production of waste is optimized on the basis of criteria such as minimization of generated volumes, limitation of over-categorized waste, generated waste and effluents as engaged doses on sites during the D&D operations.

INSIDER is a 4 years project launched on June 2017. It is coordinated by CETAMA (CEA Nuclear Energy Division) and includes 18 European partners from 10 member states.

The radiological characterization of a facility prior to performing any D&D and remediation operations is a crucial step in the definition of a viable decommissioning scenario. This scenario must be robust and optimized not only with regards to technical issues, produced waste amount and workers dose, but also with regards to costs, deadlines and safety. INSIDER contributes to building confidence that the nuclear community is fit for taking responsibility for retiring a range of facilities and sites without imposing undue economic, social or radiological burdens.

Today, the reconstitution of a 3D vision of a facility or components is partly based on off-site laboratory analyses, leading to technical and economic difficulties in the realization of representative samples. INSIDER aims at developing and qualify integrated approaches to enhance and support characterization methodologies.

The heart of the technical innovative part of the project is to improve the sampling strategy taking into account different types of measurement data, with different but known levels of accuracy. In-situ analysis techniques are of paramount importance for initial cartography. In constrained environment they must be complemented by more detailed off-site measurements. Realistic knowledge of their performance is essential, without systematic optimization.

The INSIDER project addresses 3 use cases: a fuel cycle facility, a nuclear power plant and a post accidental remediation. Each one is representative for different kind of constraints and analytical challenges. The project-integrated approach will be tested and validated by applying the methodologies on three full-scale D&D sites offered by JRC ISPRA, SCK-CEN and CEA, representative of the use cases.

A benchmarking supplemented by interlaboratory comparisons (ILC) on synthetic reference samples produced within the project will structure the validation and refinement of the INSIDER methodology.

The practical project implementation includes documenting, testing, verifying and assessing characterisation methodologies for the selected test cases. Thereby, a key contributor is effective knowledge sharing within the scientific community.

Final assessment of the outcome will strengthen the recommendations and guidance, and promote and share European expertise through guides and pre-normative texts.

Acknowledgment: The project INSIDER has received funding from the Euratom research and training program 2014-2018, under grant agreement No 755554.

# The Use of Sampling in Decommissioning Work

JENS SØGAARD-HANSEN, MIKKEL ØBERG

*Danish Decommissioning*

The nuclear facilities at the Risø site are undergoing decommissioning. Before, during and after decommissioning sampling is used. Sampling itself comprises the following: the determination of the number of samples, the places where the samples are taken, the size of the samples, the method of obtaining and storing the samples and the method of analysis. Sampling is used for four specific purposes:

- Planning of dismantling
- Monitoring during dismantling
- Characterization of radioactive waste
- Clearance of materials, buildings and land areas

Sampling is normally done for one of the specific purposes because the different purposes have different objectives; however some overlap does exist. In the planning phase results from sampling primarily help guide the health physics measures necessary during the dismantling, but results can also be used to help determine the best way of doing the actual decommissioning. During dismantling sampling is used to further substantiate assumptions made during the planning phase or to explore abnormalities. Characterization of radioactive waste is made to provide data for safe storage (short term and long term) and inventory calculations. In clearance measurements sampling is used when it is impractical or impossible to make a total measurement on the item to be cleared. It is also used when nuclide concentration ratios are to be determined. Examples of the use of sampling including the analyses used will be given.

# Future decommissioning of Norwegian research reactors

MARTE VARPEN HOLMSTRAND

*Norwegian Radiation Protection Authority (NRPA), Norway*

There are currently two research reactors in Norway. In June 2018, one of these reactors, the Halden Boiling Water Reactor (HBWR) is to be decommissioned. Although two previous research reactors were decommissioned in Norway the 1960's, decommissioning of HBWR will in practice be a novel experience for Norwegian Radiation Protection Authority (NRPA) as the regulatory body, the owner of the reactor – the Institute for Energy Technology, and the Norwegian society.

This talk will present some of the challenges with regards to regulatory issues, and also the need to optimize the techniques used in decommissioning in order to achieve the most safe and optimal solution for waste handling.

One challenge with the future decommissioning is the changes in regulations from the 60's and now. In the 60's, the only act that regulated use of radiation, discharges and so on, was the Act on Use of X-Rays from 1938. Today, relevant legislation for decommissioning is the Pollution Control Act of 1982, the Radiation Protection Act of 2000, the Act on Nuclear Energy Activities of 1973 and others. Since these regulations have not yet been used in a decommissioning context, the NRPA are in the process of developing regulatory requirements and expanding our expertise in order to be able to fulfil the expectations to the NRPA as a regulatory body.

In addition to the regulatory challenges, the technical issues may be just as challenging, especially when it comes to safe waste handling. Norway has approximately 17 tons of spent fuel accumulated from the early 50's that must be handled properly. This nuclear waste could either be reprocessed or directly disposed. Even if the waste is reprocessed, Norway must handle fission products. This means that Norway needs a deep geological disposal facility. The major waste volume from decommissioning will be low- and intermediate level wastes that arise from decontamination and demolition of the facility. The current repository for low and intermediate level wastes is filling up, and if both reactors with auxiliary facilities were to be decommissioned, this repository does not have enough capacity to accept all the decommissioning waste. Therefore, Norway also needs a new repository for low and intermediate level waste. Work on establishing a new repository is in the very beginning phase. Based on experience from the existing repository for low and intermediate waste, this process will take at least 10 years.

Repositories are expensive and a limited resource. It is important to handle radioactive decommissioning waste safely, but it is also important to not use the repositories for wastes which do not belong. The definition of radioactive waste is based on specific activities. This will most likely be a challenge for large volumes of low level decommissioning waste. Decontamination can also cause liquid radioactive waste, which causes its own challenges, as it either must be discharged or solidified. We need good methods in order to separate radioactive waste from non-radioactive waste, to identify techniques which minimize waste production, and to ensure the quality of both separation and techniques.

# **Overview of decommissioning status, challenges and solutions in Denmark**

KIRSTEN HJERRILD NIELSEN

*Danish Decommissioning (DD); Waste, Decommissioning and Operation*

This presentation will give a status report on decommissioning of the nuclear facilities in Denmark all of them situated at the Risø site. There are six nuclear facilities in all: Three research reactors of which the two have been decommissioned, a Hot Cell, a Fuel Fabrication Plant and a Waste Treatment Plant.

At the moment two large decommissioning projects are ongoing: The DR 3 reactor and the Hot Cells. The focus will be on these two projects. The last decommissioning project, the Waste Treatment Plant, is in the planning phase.

The framework of decommissioning in Denmark will be explained as well as the chosen approach.

The presentation also covers the challenges we have met as well as the solutions that have been chosen. Short videos of the work carried out will be shown.



# **Implementation of the Euratom BSS regulations for clearance in the Swedish legislation**

HENRIK EFRAIMSSON

*Swedish Radiation Safety Authority (SSM)*

The presentation describes the implementation of the Euratom BSS regulations for clearance in the Swedish legislation. The Euratom BSS clearance regulations are partly implemented by the Swedish government in the new radiation protection ordinance and partly by the Swedish Radiation Safety Authority in the authority's regulations. Thereby, the previous regulations concerning clearance of materials, rooms, buildings and land in practices involving ionising radiation (SSMFS 2011:2) have been revised and replaced by the regulations concerning exemptions from the radiation protection act and clearance of materials, building structures and land (SSMFS 2018:3). The revision also included six years of experience from application of the previous regulations SSMFS 2011:2. Key experiences and challenges from the application of the regulations SSMFS 2011:2 during operation and decommissioning will also be described.

# Determination of difficult-to-measure nuclides in radioactive wastes of NPP Paks, Hungary

NÓRA VAJDA

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In Hungary four VVER-440 type nuclear reactors have been operated since the beginning of the 1980-ies. Radioactive wastes have been collected, liquid wastes have been concentrated by evaporation and stored. Recently evaporation concentrates have also been processed to remove  $^{60}\text{Co}$  and radiocesium isotopes and obtain purified water for clearance purposes.

Liquid radioactive wastes contain a great variety of difficult-to-measure (DTM) nuclides that do not emit easy to detect  $\gamma$  radiation. Many of them are long-lived pure  $\alpha$  and  $\beta$  emitting nuclides that affect the long-term behavior of radioactive waste depositories. Development of radiochemical methods to analyze DTM nuclides started parallel to the operation of the NPP. At present activities are focused at two research sites, i.e. the Nuclear Research Institute of the Hungarian Academy of Sciences (ATOMKI) in cooperation with ISOTOPTECH Co (IT) and RADANAL Ltd. (RA). Radiochemical methods have been developed for the determination of actinides (U, Np, Pu, Am, Cm isotopes), fission and activated corrosion products ( $^{90}\text{Sr}$ ,  $^{135}\text{Cs}$ ,  $^{93}\text{Zr}$ ,  $^{93\text{m}}\text{Nb}$ ,  $^{94}\text{Nb}$ ,  $^{55}\text{Fe}$ ,  $^{63}\text{Ni}$ ,  $^{125}\text{Sb}$ ) at RA as well as for the determination of other fission and activation products ( $^3\text{H}$ ,  $^{14}\text{C}$ ,  $^{129}\text{I}$ ,  $^{99}\text{Tc}$ ,  $^{36}\text{Cl}$ ,  $^{108\text{m}}\text{Ag}$ ) at ATOMKI-IT. Other methods ( $^{41}\text{Ca}$ ,  $^{107}\text{Pd}$ ,  $^{79}\text{Se}$ ,  $^{151}\text{Sm}$ ,  $^{147}\text{Pm}$ ) are still under development by the application of selective separation (extraction chromatographic resins) and sensitive measurement (inductively coupled plasma mass spectrometry) techniques.

A historic overview about R&D activities that have been performed in order to characterize radioactive wastes generated by the Nuclear Power Plant Paks will be presented. Various stages of method development will be shown on the example of the determination of actinides.

# **Large components, buildings and land – An optimization example on the characterization and clearance process**

JONATAN JISELMARK

*Swedish Radiation Safety Authority (SSM)*

There are different standards for the characterization and clearance process used globally in the radiological industry. All of them have advantages and disadvantages. This paper describes an example of how to combine two methods in order to use the advantages of both and minimizing the disadvantages.

There have been a standard since several years to use a method based on parametric Bayesian statistics for the characterization and clearance process in Sweden. This method has great advantages close to the clearance levels due to few measurements per m<sup>2</sup>, an ability to add extra measurements if needed and an ability to reshape area units without restarting the clearance process. Since the method is based on units with a normal distribution of the contamination there can be several units far from the clearance levels.

The American MARSSIM method uses non-parametric statistics instead of parametric. In comparison to the Bayesian method, this results in the disadvantage of less accuracy close to the clearance levels but also in the great advantage with the possibility to use few units far from the clearance limits.

By combining the MARSSIM method with the Bayesian statistics method, the amount of measurements needed for clearance may be decreased significantly (compared to using only one of the methods). By using Bayesian statistics close to the clearance levels, the contamination can be estimated in a more accurate way and the risk of having to redo the survey is minimized. By using MARSSIM methods on areas with an assumed contamination below a fraction of the clearance levels, the areas do not need to be divided into units with a normal distribution of the contamination. Thus, larger areas can be analyzed as units, which result in fewer measurements and a faster process with the same confidence level in the results. If the results indicate that an area should be analyzed with Bayesian instead of MARSSIM statistics all performed measurements can be reused since the distribution of measurements has already been randomized.

# Calibration of a $^{93}\text{Mo}$ standard

PER ROOS

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$^{93}\text{Mo}$  is one of the more rare radioisotopes remaining to be analyzed when it comes to decommissioning of nuclear power plants. It decays through electron capture to the long-lived metastable  $^{93\text{m}}\text{Nb}$  (16.1y) in 88% of the decays and in 12% directly to the ground state. The main emission from  $^{93}\text{Mo}$  is thus Auger electrons and characteristic X-rays in the <20 keV region. There is no gamma emission but the daughter  $^{93\text{m}}\text{Nb}$  emits a very weak gamma of 30 keV in 0.0005% of its decays. Apart from a troublesome chemical isolation the quantification of  $^{93}\text{Mo}$  is difficult due lack of calibration standards. The identical emission spectrum of  $^{93}\text{Mo}$  and  $^{93\text{m}}\text{Nb}$  would allow the latter to act as a calibration standard if it was available but this is currently not the case. In this work attempts to calibrate a  $^{93}\text{Mo}$  standard obtained from proton irradiated niobium is presented.

# Overall planning for decommissioning in Sweden

FREDRIK DE LA GARDIE

*Swedish Nuclear Fuel and Waste Management Company (SKB)*

The nuclear industry in Sweden currently makes plans and performs decommissioning of 7 of the 13 Swedish nuclear power reactors and one research reactor. The decommissioning activities in Sweden have increased the last years due to the decisions of shutting down two reactors at the Oskarshamn site and two reactors at the Ringhals site. These decommissioning projects are ongoing along with the dismantling of Barsebäck NPP and the research reactor at Studsvik. The decommissioning of Ågesta NPP has also started and the project is in its planning phase.

The change of the decommissioning scene in Sweden has also showed needs to develop the legal framework within decommissioning. The Swedish Radiation Safety Authority is developing the requirements in a dialogue with the industry and other stakeholders. Within the nuclear industry common guidelines are also been developed with the purpose of applying the requirements in a uniform manner.

The Swedish Nuclear Fuel and Waste Management Company (SKB), is owned by the nuclear power companies and manages and dispose of nuclear waste and spent nuclear fuel from the reactors. In order to manage and dispose the decommissioning waste SKB has applied for an extension of the geological repository for low and intermediate level waste, the SFR facility. When planning decommissioning and designing the waste management system it is important to consider all aspect from 'cradle to grave', such as characterisation, dismantling, sorting, conditioning, storage, transportation, final disposal and the long term safety after closure.

Experiences in decommissioning have been and are gained within the nuclear industry but one main challenge in Sweden is the large scale decommissioning of nuclear power reactors and parallel ongoing projects at different sites where units also are in operation. SKB and the nuclear companies have been collaborating intentionally within decommissioning and waste management and it will continuously be an import forum for exchange of experiences and benchmarking.

# **Decommissioning of FiR 1 research reactor: status, challenges and solutions**

ANUMAIJA LESKINEN, ANTTI RÄTY, MARKUS AIRILA

*VTT, Finland*

FiR1 (Finnish Reactor 1) TRIGA Mark II type research reactor is the first nuclear reactor built in Finland. The over 50 years of operation included intensive neutron beam research, activation analyses, isotope production and a treatment facility for Boron Neutron Capture Therapy (BNCT). Now, after the final shutdown in June 2015, FiR1 is the first reactor under decommissioning in Finland. After intensive dismantling planning and documentation preparation, the license application for decommissioning was submitted to the Government of Finland in June 2017.

While the license application is being reviewed, work is continued for example in document preparation, characterization, planning of the final disposal of the decommissioning waste, and negotiation on the spent nuclear fuel removal. Spent nuclear fuel removal is a prerequisite for dismantling and therefore one of the most critical milestones of the project at this point. Either an international USA solution or a domestic solution needs to be arranged.

Significant effort has already been given for the characterization, since radionuclide specific inventory calculations have been carried out for all reactor materials. In comparison to nuclear power reactors, research reactors have a wider spectrum of material, but with lower activation levels. In the case of FiR1, the BNCT facility also introduces an additional set of materials. Monte Carlo N-particle Transport code and ORIGEN-S were utilized giving a 3D activation model of the reactor materials. Currently, modeling results are being validated and adjusted with experimental data using HR-ICP-MS and ICP-OES for the chemical composition analyses, gamma spectrometry for gamma-emitters, and radiochemical separation methods and liquid scintillation counting for beta-emitters.

# **Decommissioning in Germany: Experience, Current Status and Upcoming Challenges**

BERNHARD BUGENHAGEN  
*TÜV NORD EnSys, Germany*

In the role of TÜV NORD as a technical support organisation (TSO), we support and consult the German regulatory authorities. Among others, this includes the technical assistance in their task of licensing and supervision of the decommissioning of German nuclear installations, mainly power plants. With that, we look back on decades of experience, encompassing the whole process of decommissioning, such as the evaluation of the initial decommissioning license, on-site supervision of dismantling-work as well as the performed clearance measurements for materials, buildings and the final site-release. In addition to that, we assess the equipment used for radiological measurements and conduct our own independent radiological surveys.

In this presentation, we give a short overview of the rather unique German nuclear legislation and regulatory system. In particular, we will shed light on the interplay between the regulatory authorities and the operators and describe our role as a TSO in this landscape.

Furthermore, we will present a selection of the technical milestones reached in German decommissioning projects: Firstly, we will give a TSO's perspective on the full-system-decontamination of a number of reactors and highlight some potential pitfalls. After that, we will show some of the important points that taken into account during the removal of large components on the example of the removal of steam generators. Since the steam-generators were to be transferred abroad for smelting, the compliance of the external smelting and clearance procedure with German requirements for this process are an interesting case study. Finally, we will highlight the challenges during the clearance of the electric generator from a BWR for later re-use in a conventional power plant.

Finally, we will give an outline of the current state of decommissioning in Germany and the connected challenges, including those during the licensing process, the waste management and, of course, the public acceptance.

# Radiochemical analysis for characterization of decommissioning waste

XIAOLIN HOU

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With increasing numbers of nuclear facilities, especially nuclear power reactors, being closed in recent years and from now on, a considerable work is going to be carried out all over the world for decommissioning these nuclear facilities. For this purpose, characterization of various wastes from decommissioning is required for evaluation of the radioactivity inventory in various materials and decision making for management of the produced waste. This is carried out by quantitative determination of various radionuclides present in the materials.

The neutron activation products of components and impurity in the materials used in the nuclear facilities, such as  $^3\text{H}$ ,  $^{14}\text{C}$ ,  $^{36}\text{Cl}$ ,  $^{41}\text{Ca}$ ,  $^{60}\text{Co}$ ,  $^{55}\text{Fe}$ ,  $^{63}\text{Ni}$ ,  $^{133}\text{Ba}$ ,  $^{152}\text{Eu}$ ,  $^{154}\text{Eu}$ , and some transuranics, are the main contributors to the total radioactivity, especially in the construction materials. But some long-lived fission products, such as  $^{90}\text{Sr}$ ,  $^{99}\text{Tc}$ ,  $^{129}\text{I}$ , and  $^{137}\text{Cs}$ , are the major concern for materials contaminated by spent nuclear fuel. Of these radionuclides, the gamma emitting radionuclides, such as  $^{60}\text{Co}$ ,  $^{133}\text{Ba}$ ,  $^{152}\text{Eu}$ ,  $^{154}\text{Eu}$ , and  $^{137}\text{Cs}$ , are easily measured by gamma spectrometry. While the determination of pure beta and alpha emitters including  $^3\text{H}$ ,  $^{14}\text{C}$ ,  $^{36}\text{Cl}$ ,  $^{41}\text{Ca}$ ,  $^{55}\text{Fe}$ ,  $^{63}\text{Ni}$ ,  $^{90}\text{Sr}$ ,  $^{99}\text{Tc}$ ,  $^{129}\text{I}$  and some transuranics is the major challenges, because they could not be measured without separation from the matrix of the samples and from all other radionuclides, this entitles them as the radionuclides of difficult to measure.

Radiochemical analysis is the only way to complete the determination of the radionuclides of difficult to measure by including a completely separation of individual radionuclides from matrix and other radionuclides before measurement by beta counting, alpha spectrometry or mass spectrometry. Although plenty of analytical methods have been reported for the determination of these radionuclides since the discovery of radioactivity, the suitable methods are not always available for the purpose of decommissioning. This is because that a large number of samples are required to be analyzed during the decommissioning, which needs simple and rapid methods to provide a good analytical capacity. In addition, the sample matrix varies very much from concrete, graphite, exchange resin, to various metals, which requires different radiochemical methods for different sample matrix and target radionuclides.

Besides the conventional radiometric methods for the measurement of radionuclides, such as liquid scintillation counting, low level beta counting, and alpha spectrometry for these hard to measure radionuclides, some new methods have been proposed and applied. Among them, mass spectrometry especially ICP-MS and accelerator mass spectrometry (AMS) has been widely used for the measurement of long-lived radionuclides, such as  $^{41}\text{Ca}$ ,  $^{36}\text{Cl}$ ,  $^{99}\text{Tc}$ ,  $^{129}\text{I}$ ,  $^{135}\text{Cs}$  and transuranics. With these techniques, the measurement time can be significantly reduced from few days to some minutes, enabling to rapid measurement of large number of samples. In the past few years, a large numbers of radiochemical analytical methods aiming to characterize various decommissioning waste by determination of various radionuclides of hard to measure have been developed in many laboratories. This work aims to present the state of the art analytical methods for characterization of nuclear waste from the decommissioning of nuclear facilities, not only the updated chemical separation procedure, but also advanced measurement techniques. Meanwhile, some examples of analytical methods will be also presented, including (1) rapid determination of tritium and  $^{14}\text{C}$  in solid materials, such as graphite, concrete, steel, aluminum, paint, silica gel, soil, and dust; (2) determination of  $^{36}\text{Cl}$  and  $^{129}\text{I}$  in graphite, steel,



concrete, waste water, and dust; (3) simultaneously determination of multi-nuclides such as  $^{41}\text{Ca}$ ,  $^{55}\text{Fe}$ ,  $^{63}\text{Ni}$ ,  $^{90}\text{Sr}$  and transuranics; (4) challenge on the determination of low level  $^{93}\text{Mo}$ ,  $^{93}\text{Zr}$ ,  $^{135}\text{Cs}$ ,  $^{79}\text{Se}$  and  $^{126}\text{Sn}$  in decommissioning waste.

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# Analysis of Pu isotopes and Np-237 in seawater by AMS

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Plutonium isotopes and Neptunium-237 have been introduced into the environment mainly from nuclear weapons' testing in the 1950s and early 1960s and releases from nuclear fuel reprocessing facilities. These radionuclides are also of importance in nuclear storage and decommissioning work. The marine environment stores a significant fraction of that anthropogenic radioactivity. For instance, the North Atlantic Ocean has received since 1950-1960's the liquid releases from the European Nuclear Reprocessing plants, introducing a dynamic signal labelling water masses. Therefore, it is important to set up reliable radiochemical and analytical methods to measure Np-237 and Pu isotopes in seawater samples. Until recently, the analysis of these radionuclides in seawater was challenging because of their very low concentration requiring large volumes of seawater to process (up to 150 litres) and long counting times in alpha spectroscopy. Accelerator Mass Spectrometry (AMS), a technique used to analyse long lived isotopes, is suitable for analysing Np-237 and Pu isotopes in the environment. We will present two novel radiochemical procedures that have been developed, optimized for analysis with AMS and applied to seawater samples for the determination of activity concentrations of Pu-239 and Pu-240, and Np-237. The first method was developed for alpha spectrometry and ICP-MS measurements from large volume samples (~100 L) and was adapted for AMS. The most recent one is focused on low volume samples (1-10 L) and incorporates the sequential extraction of U for the final analysis of U-236 by AMS.

Keywords: <sup>237</sup>Np, <sup>239+240</sup>Pu, <sup>239</sup>Pu, <sup>240</sup>Pu, seawater, Accelerator Mass Spectrometry, radiochemistry

# **Radiochemical Analysis - experience and challenges**

KAISA VAARAMAA

*Radiation and Nuclear Safety Authority (STUK), Finland*

Radiological baseline study of the mining and nuclear facility areas are performed before the activity in the area begins. Baseline study measurements give detailed information of the existing levels of radionuclides in the environment. The data is used later to assess the effects of the activity to the environment. Radiochemical analysis is needed also during the activity and in the radioactive waste management.

Even if there are accredited analytical methods in the laboratory, many challenges in the sample pretreatment and analysis may arise. The activity concentrations in the environmental samples are generally low. The sample matrix may cause problems for the analysis, if the sample quantities required for the analysis are large. In case of the industrial waste samples, the radioactivity of the samples might be high, which also cause challenges in the radioactivity analysis.

# **Lessons learned – Surprises in Radiological Characterization**

MATTHIAS BOTHE

*VKTA – Radiation Protection, Analytics & Disposal Rossendorf Inc., Dresden (Germany)*

There are some proved and tested concepts and procedures for radiological characterization. Some of them are described in literature and standards. The application in specific situations requires competent and smart adaption to the circumstances of an installation. In this situation occur surprises because of various reasons. Surprise means deviation from the expected result. Reasons may be:

- insufficient documentation of construction and operation
- mistakes during sampling
- unexpected effects of measurement and analysis
- mistakes in determination of scaling factors and nuclide vectors.

It is important to notice these deviations, to take them seriously, to look for the reasons and to draw conclusions. In the presentation, such surprises will be shown in examples of different decommissioning projects. This may contribute to an exchange of experiences and to direct attention to surprises in radiological characterization.

# **Development of rapid and automated radiochemical separation technique**

JIXIN QIAO

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This presentation overviews the recent development in automation and methodology for radiochemical separation in DTU Nutech. Flow based techniques including flow injections (FI), sequential injection (SI), lab-on-valve bead injection (LOV-BI) were exploited for automation of the analytical processes. Anion exchange and extraction chromatography were used for chemical purification of target radionuclides followed by detection with inductively coupled plasma mass spectrometry (ICP-MS) or accelerates mass spectrometry (AMS). Examples for determination of Pu, Np, U and Tc in environmental and biological samples are given. The analytical results indicate the automated separation techniques provide advantages of short analytical time, low labor intensity and high sample throughput. Faced with the appearing challenges in the heavy work load for nuclear decommission and waste management, application of these automated separation techniques could potentially improve the analytical efficiency and support the individual radiochemistry labs to cope with the heavy demands in the radiochemical analysis of a large number of samples for waste characterization and categorization.

# **$^{55}\text{Fe}$ , $^{63}\text{Ni}$ and $^{59}\text{Ni}$ in old nuclear reactor pressure vessel steel samples**

TANELI ISO-MARKKU<sup>1</sup>, SUSANNA SALMINEN-PAATERO<sup>1</sup>, ANUMAIJA LESKINEN<sup>2</sup> AND ANTTI RÄTY<sup>2</sup>

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Beta-active radionuclides are considered to be “difficult to measure” because they have to be separated radiochemically from the sample matrix and most often from other radionuclides before determining their activity concentration. Nuclear reactor materials contain beta emitting radionuclides whose activity level and radionuclide distribution depend on irradiation history and original composition of the material. Before nuclear reactor decommissioning it is essential to survey the radionuclide distribution and activation level of the reactor materials for planning waste management. Furthermore, the results obtained from radiochemical determinations are utilized in forming of scaling factors and validation of mathematical modelling for evaluating activity distribution and level in a broader scale.

Preliminary tests were performed for two types of steel from nuclear reactor pressure vessels, from BWR and VVER reactors.

The steel samples were dissolved in concentrated acids after which Fe and Ni were separated from each other and sample matrix by anion exchange. Two separation schemes for purifying Ni fraction from anion exchange were tested: DMG (dimethylglyoxime) precipitation, and separation with Ni-resin. Finally the activity concentrations of  $^{55}\text{Fe}$  and  $^{63}\text{Ni}$  were determined by LSC and  $^{59}\text{Ni}$  by x-ray fluorescence spectroscopy. The concentrations of stable Fe and Ni were determined by MP-AES, for evaluating analytical loss of these metals during separation procedure. The results are presented and discussed in the presentation.

# **Sampling and analysis campaign for a rapid assessment of the contamination level in a radiochemistry laboratory**

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Decontamination, decommissioning and site remediation of former contaminated facilities are a major concern since the phasing out of many countries from the production of nuclear electricity or research activities. The starting point of this process is a good knowledge of the state of contamination at the site which is usually assessed by an On-site monitoring campaign. This radiological assessment is very important because it allows the cost reduction of the cleaning up phase by focusing only on the contaminated area(s) within the facilities.

In this study, we present an overview of the sampling and analysis campaign performed in the former radiochemistry laboratories of the University of Liège specialized in the actinide's chemistry for the purpose of its decontamination and its remediation. The sampling strategy is based on the lab's history and on the building infrastructure (e.g. ventilation system, gloves boxes, liquid waste storage tanks, etc.).

Different samples types such as floor and wall samples, swipe tests, waste waters or sediments are collected at different strategic places for the assessment of the contamination. Simple sampling procedures have been adapted to the purpose, to the specific environment and also to the type of measurement during the sampling campaign

The measurement technics used are proportional counting, liquid scintillation, gamma-ray and alpha spectrometry depending on the matrix and on the information required. The analytical procedures are derived from the methodologies used for the radiological monitoring of the environment but they have been adapted to this specific topic in order to increase understanding and knowledge of the state of contamination of the laboratories to be cleaning up.

# **Historical overview and current challenges in radionuclide analyses in different waste matrices and materials at SCK•CEN, Belgium**

MIRELA VASILE  
*SCK-CEN, Belgium*

SCK•CEN has a long history in monitoring the radioactivity in the environment and nuclear waste characterisation and being involved in important projects, like the decommissioning of the BR3 (Belgian Reactor 3). In these studies, the characterisation of waste types and the determination of radionuclides important for the management of nuclear wastes, their long-term disposal and impact on the environment were investigated. Since safety guidelines and legislation evolve with the years, outcome of previous studies needs re-evaluation or needs to be extended with data of additional radionuclides that were considered in more recent safety studies. Main challenges for many laboratories involved in this type of research are the radiochemical separation and quantification of the so called “difficult to measure radionuclides” ( $^{41}\text{Ca}$ ,  $^{79}\text{Se}$ ,  $^{36}\text{Cl}$ ,  $^{94}\text{Nb}$ ,  $^{63}\text{Ni}$ ,  $^{59}\text{Ni}$ ,  $^{151}\text{Sm}$ ,  $^{147}\text{Pm}$ , ...) in different types of waste matrices. At the end of the life of nuclear installations, decommissioning is the most common practice. The characterisation of the materials resulting from the decommissioning of nuclear installations, such as concrete, steel, graphite...and environmental samples, equally requires the quantification of these difficult to measure nuclides accounting for the particular material compositions. The clear need of validated methods for quantifying those radionuclides requires further research in this field.

In a short survey the main characterisation techniques developed and used at SCK•CEN will be discussed and requirements for future characterisation methods for environmental and decommissioning materials will be presented.

Keywords: Low-Level Radioactivity Measurement expert group, SCK•CEN, Belgium



# **National analytical capabilities to analyze radionuclides in different matrixes occurring in the decommissioning processes**

MATS ERIKSSON, PATRIC LINDAHL

*Swedish Radiation Safety Authority (SSM)*

The presentation describes the challenges possessed in the large scale decommissioning of Swedish nuclear power plant from a verification measurement point of view. Focus will be on the national analytical capabilities to analyze so called “difficult to measure” radionuclides in different matrixes occurring in the decommissioning processes. In 2020, Sweden will have 6 reactors in operation and 7 reactors permanently shut down, including Ågesta reactor and Barsebäck 1 and 2. Each reactor will produce between 200 000 – 300 000 ton of decommissioned material where about 92 % are conventional waste, 6 % of the material must be clearance controlled through verifying measurement and about 2 % of the material are nuclear waste. This means that in total for the 7 decommissioned reactors about 100 000 ton must be controlled in terms of its content of radionuclides. Challenges will be to verify so called nuclide vectors of this material, perform measurements on such large quantities in a relatively short period of time as well as to obtain representative sampling for such measurements. In this perspective the national analytical capabilities will be discussed.

# **NRG approach on waste management and radiochemical characterization**

GAËL MÉNARD, ARJEN POLEY, JAAP HART

*Nuclear Research and consultancy Group (NRG), Petten, The Netherlands*

The Nuclear Research and consultancy Group is located at Petten, the Netherlands, and operates the High Flux Reactor (HFR). The HFR is used for isotope production for medical purposes as well as for material research. During its exploitation several types of radioactive waste are produced, which partly have accumulated at the reactor site.

In addition, NRG operated the Low Flux Reactor (LFR), a small 30 kW<sub>max</sub> Argonaut type reactor which was used for research and education purposes.

Over the past decade several projects on waste management and decommissioning were started and carried out requiring various approaches of waste management, including the radiochemical characterisation of the different emerging waste streams as required by the Dutch regulator:

## **Dutch historical waste management program:**

During the last 5 decades, over 1700 drums of historical waste have accumulated and stored at the Petten site. As required by law, this historical waste must be transferred to the central storage facility near Vlissingen in the South-West part of the Netherlands, which is operated by COVRA, the Dutch waste management organization. NRG is in charge to transfer the waste from the Petten site to the COVRA facility. Since COVRA does not accept insufficiently characterized radioactive wastes, the contents of each of the 1700 drums must be established. NRG took this challenge by setting up a program for the destructive and non-destructive characterization of the historic waste. Determination of nuclides scaling vectors via calculation and rapid gamma detection are used to segregate the historical waste into three categories, i.e. low-level, intermediate-level and high level radioactive waste.

A destructive characterization, performed on selected samples, provided both a complement and a verification to the nuclide scaling vectors.

## **Ion exchange resins contingent characterisation:**

Over the years IER have been accumulated and this created a stream of legacy waste that should be pragmatically treated as space to on-site storage at Petten becomes scarce. We identified a number of processes from cementation to steam reformation for reducing the volume this type of waste. It appeared that incineration of the IER is the most efficient and at the same time the most economically advantageous process for volume reduction. Characterizing the IER started with the anionic ion exchange resins for this is less radioactive and therefore easier to handle. We developed also a set of non-destructive (gamma spectrometry) and destructive analyses (beta-nuclide, total alpha, total beta) for characterization of the IERs.

## **Decommission of the Low-Flux Reactor:**

The low-flux reactor (LFR) was an Argonaut type, small sized reactor with maximum power of 30 kW, which was used during approximately 50 years for research and education purposes. The LFR was shut down in 2010. NRG successfully carried the dismantling and decommissioning of the LFR, and completed it in the beginning of 2018. A significant part of the radioactive waste comprised activated concrete which formed the biological shield of the

LFR. The decommissioning team took samples from the activated concrete for further radiological analysis at NRGs laboratories. Furthermore, NRG defined a project in which we will crush the concrete into small pieces or even powder to examine the specific radiological content of each of the component of the concrete (cement, sand, rocks). The aim of this project is to determine whether any activated material is concentrated in one of these components. If that would be the case, that particular component could be isolated from the other components, thereby reducing the total amount of radioactive waste.

# Measurement of radionuclides and heavy metal elements in maizes for radiological and chemical assessment in a NORM mining site

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Radiological and ecological impact of NORM activities on the environment has been concerned lately. In addition, heavy metal chemical risks are often associated with such activities due to the similar geochemical properties. A project has been completed in the area of NORM activity, which is rich in uranium and other metal elements. The results of our research presented that radioactivity concentrations of naturally occurring radionuclides in some crops are obviously higher, and some heavy metal elements are also high, which exceeds the national standard for food (GB2762-2012). Crop samples in the study area were collected and analyzed.  $^{210}\text{Po}$  and  $^{210}\text{Pb}$  contribute the bulk of the dose in the diet. The samples after radiochemical process, concentrations of  $^{210}\text{Po}$  and  $^{210}\text{Pb}$  in crops have been completed by alpha spectrum analysis and low level beta analysis respectively, while heavy metal elements, or trace elements have been analyzed by ICP-MS. The results showed that the concentrations of  $^{210}\text{Po}$  and  $^{210}\text{Pb}$  nuclides in 12 maize samples were from 160 mBq/kg to 810 mBq/kg, average value 450 mBq/kg and 160 mBq/kg to 1130 mBq/kg, average value 350 mBq/kg respectively. The concentrations of  $^{210}\text{Po}$  and  $^{210}\text{Pb}$  in crops were much higher than the reference values recommended by IAEA, and also higher than the typical values in China. The average content of heavy metal, such as Cr, Cu, Zn, Pb was 3 to 10 times higher than the national standard respectively. The concentrations of  $^{210}\text{Po}$  and  $^{210}\text{Pb}$ , and contents of heavy metal elements in other five crops are the similar situation as the result mentioned above.

# Determination of $^{93}\text{Mo}$ (and $^{94}\text{Nb}$ ) in nuclear decommissioning waste from a nuclear reactor

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Molybdenum is an alloying component of structural materials used in nuclear reactors. The activation product of stable  $^{92}\text{Mo}$  (14.65% abundance) is  $^{93}\text{Mo}$ , which has a long half-life ( $(4.0 \pm 0.8) \times 10^3$  years); so it is an important contributor of nuclear power plant wastes (especially dismantling wastes). As it decays by electron capture; its proper detection is very difficult. X-ray spectrometry, LSC and mass spectrometry (ICP-MS) can be used, but chemical separation is needed to separate  $^{93}\text{Mo}$  from the matrix and interferences before detection. Although some methods have been reported for its determination, a more thorough isolation is required in case of nuclear decommissioning waste, where activities of  $^{60}\text{Co}$ ,  $^{125}\text{Sb}$ ,  $^{65}\text{Zn}$  and many other radionuclides are 3-8 orders of magnitude higher, than that of  $^{93}\text{Mo}$ . So a newly developed method for this purpose will be presented.

Several metallic samples from a nuclear power plant were analyzed. Each sample was dissolved using aqua regia and HF, and prepared in diluted HF acid for separation.

A combined chromatographic separation procedure was applied to separate Mo from the matrix and interfering radionuclides. The majority of activity was removed using cation exchange chromatography, as the activation products of most metallic components ( $^{54}\text{Mn}$ ,  $^{55}\text{Fe}$ ,  $^{60}\text{Co}$ ,  $^{59}\text{Ni}$ ,  $^{63}\text{Ni}$ ,  $^{65}\text{Zn}$ ) were retained on a cation exchange resin as cations. However, Mo as anion (e. g.  $\text{MoF}_7^-$ ,  $\text{MoOF}_5^-$ ,  $\text{MoO}_2\text{F}_3^-$  or  $[\text{MoO}_2\text{F}_4]^{2-}$ ) passes through the cation exchange column and remains in effluent; just like anion complexes of Zr ( $^{93}\text{Zr}$ ) and Nb ( $^{93\text{m}}\text{Nb}$ ,  $^{94}\text{Nb}$ ) ( $\text{ZrF}_6^{2-}$ ,  $\text{NbF}_6^-$ ,  $\text{NbOF}_5^{2-}$ ), as well as  $^{99}\text{Tc}$  ( $\text{TcO}_4^-$ ).

Due to the removal of the main gamma emitters at this stage,  $^{94}\text{Nb}$  in the effluent can directly be measured using gamma spectrometry.

Before measurement of  $^{93}\text{Mo}$  using LSC, further separation of Mo is required from all other radionuclides, especially from the anions of Zr and Nb. The different affinities of these anions to TEVA resin were utilized to perform a nearly clean isolation of Mo from Zr and Nb; and by this procedure most of interferences can be removed.

For further purification, the separated  $^{93}\text{Mo}$  was prepared in a diluted  $\text{HNO}_3$  solution, and loaded to an alumina ( $\text{Al}_2\text{O}_3$ ) column. After rinsing with diluted  $\text{HNO}_3$ , the adsorbed  $^{93}\text{Mo}$  was finally eluted with ammonia solution. This solution was concentrated and prepared in water for LSC measurement after adding scintillation cocktail.

The recovery of Mo in the whole procedure was determined by measuring stable Mo in the separated solution and the initial solution using ICP-OES, and found to be more than 70 %. The decontamination factors for the key interfering radionuclides are higher than  $10^5$ .

An internal solution of  $^{93}\text{Mo}$  was prepared from a proton irradiated niobium target, and standardized using X-ray spectrometry using a HPGe detector (to determine the activity of  $^{93}\text{Mo}$  due to its X-ray radiation). This solution was used to prepare quench curve of  $^{93}\text{Mo}$  for the calibration of the LSC instrument, and quantitative measurement of  $^{93}\text{Mo}$  in the separated sample solutions.

# Abstracts of poster presentations

# **A Study of Po-210 Content in the Urine of People Living in Homes with High Radon Concentrations**

PETRA VALDEZOVA

*SURO, Czechia*

The objective of the presented research is to find a possible effect of living and/or working in places where the radon concentration is more than 1000 Bq/m<sup>3</sup>, exceeding the Czech guideline (reference) level of 300 Bq/m<sup>3</sup>. The first step was to determine <sup>210</sup>Po concentration in urine samples from such people. Twenty-nine non-smokers volunteered to provide a urine sample excreted within 24 hours. They were asked to not consume fish, seafood, liver and kidney one week before and during the urine collection. These factors influence the intake of <sup>210</sup>Po, and therefore its excretion in urine, as it was shown in the review paper by Hölgýe and Straková (2009).

The values of <sup>210</sup>Po concentration found in urine samples were in the range of 3.1 - 28.8 mBq/day. The arithmetic mean (AM) was calculated at  $7.64 \pm 5.78$  mBq/day and a geometric mean (GM) was 6.4 mBq. On the other hand, people who have not been exposed to high radon concentrations had a range of 1.4 - 10.8 mBq/day of daily excretion of <sup>210</sup>Po, AM  $4.1 \pm 2.0$  mBq/day and GM 3.7 mBq/day, as was found by Hölgýe (2013) in a group of 40 Prague citizens. Based on these results, it can be suggested that the individuals staying in an environment with increased radon activity concentration excrete more <sup>210</sup>Po in the urine.

Knowledge of <sup>222</sup>Rn activity concentrations in homes of the volunteers alone is not adequate to determine the possible impact of radon content in houses on <sup>210</sup>Po excretion. To evaluate this effect, additional data about the environment in which the people under investigation are living and/or working and the habits of these people (ventilation, outdoor stay, etc.) will be obtained and used in this study.

# **Radiostrontium Levels in Foodstuffs: 4-years Control Activity by Italian Reference Centre, as a Contribution to Risk Assessment**

GABRIELE TROTTA

*Istituto Zooprofilattico Sperimentale della Puglia e della Basilicata, Italy*

$^{90}\text{Sr}$  is considered an important contaminant relating to food supply chains. In this study, 176 liquid and 260 solid foods, were analysed in order to quantify  $^{90}\text{Sr}$ .

Through ruggedness tests, the application field of radiochemical methods used was extended successfully to all most important types of foodstuffs.

Regarding liquid matrices, milk samples resulted the most important indicator about  $^{90}\text{Sr}$  contamination, with mean  $^{90}\text{Sr}$  activity concentration equal to  $0.058 \text{ Bq L}^{-1}$ . Among other liquid foods, wine/spirits and livestock watering resulted the most contaminated, with mean contamination levels equal to  $0.022$  and  $0.035 \text{ Bq L}^{-1}$ , respectively.

Concerning solid matrices, cheeses produced from sheep's milk and animal feeds resulted the most contaminated (mean levels:  $1.237$  and  $1.557 \text{ Bq kg}^{-1}$ , respectively). Meat products and seafood showed contamination levels not significant within this survey; while, among vegetables, cacao/chocolate and spices resulted in contamination levels comparable with those of cheese obtained from milk of cows origin.

**Keywords:** radiostrontium, milk, dairy products, animal feed, validation, liquid scintillation, radioactivity, beta emitters.



# **Optimisation and Validation of a Multi-matrix Ultrasensible Radiochemical Method for the Determination of Radiostrontium in Solid Foodstuffs by Liquid Scintillation Counting**

GABRIELE TROTTA

*Istituto Zooprofilattico Sperimentale della Puglia e della Basilicata, Italy*

Radiostrontium is a radiotoxic isotope, chemically analogue to calcium. For these reason it may follow similar pathways to this essential nutrient and it is considered an important contaminant of several food supply chains.

Different analytical methods are currently available for the determination of radiostrontium; however, they were optimised especially for the determinations in liquid (milk, water, etc.) and environmental matrices. Moreover, the validation procedures, necessary to assure methods reliability, are still lacking.

In this work, a radiochemical analytical method for the determination of radiostrontium in several solid foodstuffs (meat, seafood, dairy products, wheat and derived products) was optimised and validated, following an in-house validation model, according to reference legislation.

Good analytical performances were obtained. Method specificity and linearity were ascertained together with measurement uncertainty (equal to 16.0%). The minimal detectable activity was equal to 8.0 mBq kg<sup>-1</sup>, while the mean repeatability (CV%) and recovery values were equal to 14.5% and 90.5% respectively. A test on a reference material was also effected, confirming method reliability for <sup>90</sup>Sr quantifications in solid foodstuffs.

Keywords: radiostrontium, meat, seafood, dairy products, wheat, validation, liquid scintillation, radioactivity, beta emitters.

# Basic study on the use cyclic oligosaccharides for the mutual separation of strontium, yttrium and zirconium to facilitate quantitative ICPMS measurements of strontium-90

SHOGO HIGAKI

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## 1. Purpose

The quantification of various radionuclides generated by nuclear fission occurring in fuel debris or contaminated water is important for the advancement of efficient waste furnaces to clean accident sites such as the Fukushima Dai-ichi Nuclear Power Plant. If a  $\gamma$ -emitting nuclide has an appropriate half-life, it can be quantified relatively easily because a multi-elemental analysis can be performed using a germanium semiconductor detector. However, it is difficult to quantitatively measure several nuclides; therefore, new measurement techniques must be developed. One measurement method involves the use of  $^{90}\text{Sr}$ , which has a relatively high nuclear fission yield of  $^{235}\text{U}$  and does not disperse into the atmosphere because of its high melting point. It behaves similarly to calcium (as a congener), accumulating in the bone when taken into the human body, and has biological half-life of  $\sim 50$  years. It is also a pure  $\beta$ -emitter, which allows  $^{90}\text{Sr}$  measurements to be conducted using conventional methods with beta rays based on the radioactive equilibrium between  $^{90}\text{Sr}$  and  $^{90}\text{Y}$  (half-life: 64 hours). However, this approach often requires two or three weeks and the need for chemical manipulation gives rise to fluctuations in the quantitative measurements depending on the proficiency of the operator.

Recently, rapid measurement methods for  $^{90}\text{Sr}$  that do not involve measuring radiation have been developed. In one such approach, an inductively coupled plasma mass spectrometer (ICPMS) has been used to measure the levels of contaminants in the Fukushima Dai-ichi Nuclear Power Station. However, applying the ICPMS method to measure  $^{90}\text{Sr}$  levels requires pretreatment by chemical separation to remove  $^{90}\text{Zr}$ , which is a stable isobar. In particular, since zirconium is the main component of the zirconia contained in the structure of the reactor, it is expected to be present in large amounts in the debris. This pretreatment can be performed using the strontium resin method. However, to reduce the workers' exposure to radiation and to quickly conduct the pretreatment on a large scale, a simpler pretreatment method is required.

We previously conducted a study to improve the frequency of radiotherapy by scavenging the radioactive waste liquid to reduce the concentration of  $^{131}\text{I}$ . One of the scavengers that can be used is cyclodextrin (CD), a circular truncated oligosaccharide that is formed by combining a plurality of D-glucose molecules. An  $\alpha$ -CD molecule comprises six glucose molecules, a  $\beta$ -CD molecule comprises seven glucose molecules, and a  $\gamma$ -CD molecule comprises eight glucose molecules (Figure 1). Therefore, CD selectively incorporates hydrophobic substances (0.5–1 nm) into its structure and sequesters it. We previously reported the radiation tolerance of CD and established that it has no influence on the  $^{131}\text{I}$ -scavenging efficiency up to an absorption dose of 30,000 Gy. In this study, we investigated the separation of strontium and yttrium from zirconium using a  $\gamma$ -CD insoluble polymer ( $\gamma$ -CDP) as a potential pretreatment for ICPMS measurements of  $^{90}\text{Sr}$ .

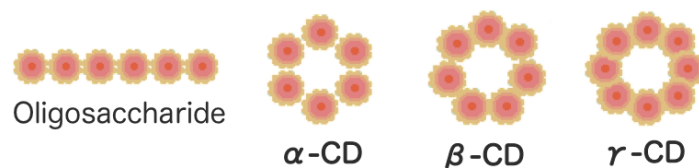


Figure 1 Images of oligosaccharide and cyclodextrin (CD) molecules.

## 2. Experiment

12 mL of an aqueous solution containing 0.1 mg/L of Zr was sealed in a centrifuge tube and 0.36 g of  $\gamma$ -CDP was added. Then, aqueous solutions containing stable Sr and Y were combined such that the resulting Sr/Y-to-Zr ratio would be 1:1, 1:100, or 1:10,000. The samples were shaken with a shaker at 90 rotations per minute for 24 h (first trial). After shaking, the samples were filtered through a 0.45- $\mu$ m filter and subsequently analyzed using an ICPMS (Agilent 8800) in the O<sub>2</sub> reaction mode. The stable Sr was counted by 88 m/z, Y was counted by 105 m/z (= <sup>89</sup>Y+<sup>16</sup>O), and Zr was counted by 106 m/z (= <sup>90</sup>Zr+<sup>16</sup>O). Then, additional 0.36 g of  $\gamma$ -CDP was added to the remaining 12-mL aqueous samples and again shaken at 90 rotations per minute for 24 h (second trial).

## 3. Result and discussion

The residual ratio of each element was calculated using the following formula.

$$\text{Residual ratio (\%)} = \frac{\text{Counts of each elements in sample with } \gamma\text{-CDP}}{\text{Counts of each elements in sample without } \gamma\text{-CDP}} \times 100$$

Figure 2 shows the residual rates of Sr, Y, and Zr after treatment with  $\gamma$ -CDP. In the first trial, the concentration of Zr in the aqueous solution was reduced by 30% from 0.1 mg/L with the addition of 3%  $\gamma$ -CDP by mass. In contrast, the amounts of Sr and Y were not significantly reduced. The concentration of Zr was also reduced when the amount of Zr was 100 or 10,000 greater than that of Sr. In the second trial, the concentration of Zr was decreased from 60% to 65%. The same amount of Zr was adsorbed by the  $\gamma$ -CDP as in the first trial and mainly Zr was collected with good specificity.

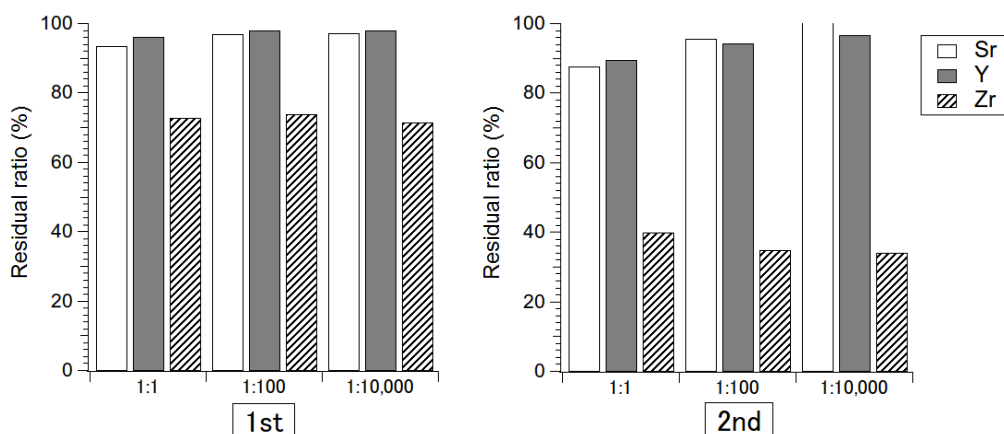


Figure 2 Residual rates of Sr, Y, and Zr after treatment with 3%  $\gamma$ -CDP by mass for different starting concentration ratios of Sr/Y:Zr.

## **Technetium-99: New Resins Developments For Separation And Isolation From Various Matrices**

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Technetium-99 is of interest in decommissioning and radioactive waste management as it is part of the so called difficult-to-measure (DTM) long-lived fission products. As it is a beta emitter potentially interfering radionuclides need to be removed very thoroughly before its radiometric quantification e.g. by LSC. The development of analysis and determination of radionuclides by mass spectrometry urges the need to reduce and/or eliminate isobars prior to analysis. Further for both detection techniques it is beneficial to avoid eluting Tc using highly concentrated acid.

Tc-99m on the other hand remains the ‘work-horse’ in medical imaging via SPECT, accordingly rapid, easy and highly selective separation of Tc from different matrices, arising from different production process such as fission or irradiation of Mo targets, remains of very high interest.

In this frame, Triskem has developed two resins designed for the extraction of Technetium from acidic and alkaline media with special focus on its separation from molybdenum. The TK201 resin is based on a tertiary amine which allows for the load in acidic to moderate acidic conditions, and specific elution of Tc in slightly acidic to slightly alkaline conditions. The PEG resin is based on a Polyethylene glycol group grafted on a polymer support. This resin allows for the extraction of Tc-99 from strongly alkaline media such as 5 M NaOH and subsequent and quantitative elution of Tc in water. In different experiments, rhenium was used as stable homologue of technetium. In both resins, separation of rhenium is clean and quantitative from different chosen elements and more specifically from molybdenum.

## **The Laboratory for Environmental Radioactivity at the Norwegian Radiation Protection Authority at Østerås, Norway**

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The Norwegian Radiation Protection Authority (NRPA) is located at three sites in Norway, all with laboratory facilities, with its main office in Østerås, a few kilometers outside of Oslo. The Laboratory for Environmental Radioactivity at the NRPA (Østerås) is a non-commercial laboratory, with a staff of 7 people, and has been located here since the late 1970s when the building was new. The Environmental Laboratory has facilities suitable for radiochemical work with low activity samples and a gamma laboratory built with low activity materials, selected in order to reduce background radiation.

In the 1970s, the main focus of the lab was on the analysis of samples containing Sr-90 and Cs-137, which originated from atmospheric bomb tests. However from 1986 onwards, the focus changed to gamma spectrometric analysis of samples from areas in Norway affected by the Chernobyl accident. In the late 1980s and early 1990s environmental monitoring in the arctic and Norwegian marine environment led to the implementation of actinide analysis by alpha spectrometry and analysis of Tc-99 in marine biota and seawater. From early 2000, the capacity of the lab has increased and radiochemical analysis for naturally occurring radionuclides, such as U, Th, Ra and Po-210, have been implemented. Implementation of new analytical methods at the lab over the years have to a large extent been facilitated by Nordic cooperation by, for example, participation in NKS-projects. Development and research in recent years have primarily focused on in situ gamma spectrometry for application in emergency preparedness and nuclear forensics. Future development of the lab aims to also include analytical methods for radionuclides relevant to nuclear decommissioning.

Since 2000 sample measurements with gamma spectrometry has been accredited according to the standard EN ISO 17025 and the lab is also active in the IAEA ALMERA and RANET networks. As a means of quality assurance, the lab participates annually in a number of intercomparison exercises arranged by IAEA, NPL; NKS or other similar bodies. At present, most of the samples come from the national environmental monitoring programs for the terrestrial and marine environment in addition to air filters from monitoring stations around Norway. In total approximately 1000 samples are analyzed annually by gamma spectrometry while a few hundred are analyzed by alpha spectrometry or liquid scintillation counting following radiochemical separation.

# **The Laboratory of Radioecological Monitoring of the ChNPP**

CHERNIKOVA NATALIA

*Chernobyl NPP, Ukraine*

The Laboratory of Radioecological Monitoring (LREM) is a subdivision of the Radiation Safety Department (RSD), which performs work on measuring radiation-hazardous factors, and on assessing their impact on personnel and the environment. LREM also performs work on reporting on the direction and progress of decommissioning activities at the Chernobyl NPP (ChNPP) units, and on activities to support maintaining a safe state of the Shelter Object. The goal of the LREM is to demonstrate that the personnel, the public and the environment are protected from the impact of radioactive materials located at the Chernobyl NPP site, including the power units 1-3, the new Shelter Object, the spent nuclear fuel storage facilities ISF-1 and ISF-2, the processing plants for management of solid and liquid radioactive waste, as well as to build and maintain complete confidence, both in society and within the organization.

The LREM is performing the monitoring and analyses of:

1. Gaseous and aerosol emissions into the environment;
2. Aerosols in the air of the production premises of the ChNPP units, on the industrial site of the ChNPP and the Shelter Object;
3. Discharge of technical water from ISF-1 in the circulating water supply system;
4. Discharge of radioactive substances into the cooling pond;
5. Atmospheric fallout;
6. Content of radioactive substances in the ground layer of air;
7. Soil;
8. Groundwater from the network of observation wells;
9. Solid radioactive waste (SRW) - construction debris, metal, rags, etc;
10. Liquid radioactive waste (LRW) - drain water, concentrated solutions from evaporation, radioactive contaminated water, etc;
11. Containers with high-level waste (HLW).

The following methods are used:

1. Spectrometric analysis of selected samples;
2. Radiometric analysis of selected samples;
3. Radiochemical analysis of selected samples.

The parameters measured include:

1. Specific and volumetric activity of gamma-emitting radionuclides;
2. Specific and volume activity of Sr-90;
3. Specific and volumetric activity of alpha-emitting radionuclides;
4. Isotopic composition of gamma-emitting radionuclides in air and in soil samples;
5. Volumetric activity of mixtures of long-lived alpha-emitting and long-lived beta-emitting radionuclides;
6. Density of deposition of radioactive substances.

For the analyses the LREM uses the following equipment:

- Multi-channel system PIC MDS-32 model based on alpha, beta counters (Protean Instrument Corporation);
- Gamma spectrometer - "Gamma plus";
- Gamma spectrometers - "DSA-1000" and "DSA-2000" (Canberra);
- Beta-spectrometer SEB-01;
- Air samplers T-8400E, Air samplers LV-22E, Air samplers H-810, H-810DC;
- Radiometer KPK-1-01.

Radiochemical analysis includes determination of volumetric activity of strontium-90 in samples of:

- natural and technological water media;
- aerosols taken from ambient air, industrial premises, as well as from gaseous discharges into the atmosphere;
- atmospheric precipitation;
- soil;
- solid and liquid radioactive waste.

Separation of Sr-90 from the sample is based on separation (purification) from interfering radionuclides by coprecipitation in the form of strontium sulphate with a stable carrier, and post-purification from interfering radionuclides by reprecipitation of strontium carbonate from solution in the presence of Trilon B.

The chemical yield of Sr-90 is controlled by introducing a known mass of stable strontium as an indicator (weight method).

Radiometric determination of Sr-90 activity is performed on the basis of its beta-radiation, using the radiometers of MPC9604 and KPK-1 types.

About 15,000 aerosol samples, 1780 samples of aqueous media, 144 samples of atmospheric deposition, 120 samples of liquid radioactive waste and 245 samples of solid radioactive waste are sampled and analysed annually by the laboratory.

In the next period, radiochemical analyses of Am-241, Pu-238 and Pu-239+240 in groundwaters from observation wells are to be established.

## List of posters

A Study of Po-210 Content in the Urine of People Living in Homes with High Radon Concentrations (Petra Valdezova, SURO, Czechia)

Radiostrontium Levels in Foodstuffs: 4-years Control Activity by Italian Reference Centre, as a Contribution to Risk Assessment (Gabriele Trotta, Istituto Zooprofilattico Sperimentale della Puglia e della Basilicata, Italy)

Optimisation and Validation of a Multi-matrix Ultrasensible Radiochemical Method for the Determination of Radiostrontium in Solid Foodstuffs by Liquid Scintillation Counting (Gabriele Trotta, Istituto Zooprofilattico Sperimentale della Puglia e della Basilicata, Italy)

Basic study on the use cyclic oligosaccharides for the mutual separation of strontium, yttrium and zirconium to facilitate quantitative ICPMS measurements of strontium-90 (Shogo Higaki, Isotope Science Center, The University of Tokyo, Japan)

Technetium-99: New Resins Developments For Separation And Isolation From Various Matrices (Maudoux N., Phelippeau L., Bas M., Vajda N., Bombard A., Happel S., C. Dirks, TrisKem International, France; Radanal, Hungary; Munich Technical University, Germany)

The Laboratory for Environmental Radioactivity at the Norwegian Radiation Protection Authority at Østerås, Norway (T. Gäfvert, A. K. Kolstad, P. Naghchbandi, J. M. Prendergast, The Norwegian Radiation Protection Authority, Norway)

The Laboratory of Radioecological Monitoring of the ChNPP (Chernikova Natalia, Chernobyl NPP, Ukraine)



# Introduction of Radioecology Section of DTU Nutech

## *Characterization of decommissioning materials — All in one place*

DTU Nutech is capable of conducting all steps of sample processing, data analysis and customer support. Our state-of-the-art methods have been applied successfully for the determination of difficult-to-measure radionuclides including  $^3\text{H}$ ,  $^{14}\text{C}$ ,  $^{36}\text{Cl}$ ,  $^{45}\text{Ca}$ ,  $^{55}\text{Fe}$ ,  $^{59}\text{Ni}$ ,  $^{63}\text{Ni}$ ,  $^{90}\text{Sr}$ ,  $^{93}\text{Mo}$ ,  $^{93}\text{Zr}$ ,  $^{94}\text{Nb}$ ,  $^{99}\text{Tc}$ ,  $^{129}\text{I}$ ,  $^{210}\text{Po}$ ,  $^{210}\text{Pb}$ ,  $^{226}\text{Ra}$ ,  $^{237}\text{Np}$ ,  $^{234}\text{U}$ ,  $^{235}\text{U}$ ,  $^{238}\text{U}$ ,  $^{238}\text{U}$ ,  $^{238}\text{Pu}$ ,  $^{239}\text{Pu}$ ,  $^{240}\text{Pu}$ ,  $^{241}\text{Pu}$ ,  $^{241}\text{Am}$  and  $^{244}\text{Cm}$  as well as gamma emitters in various samples from nuclear decommissioning.

## *Outstanding sample capacity*

The laboratories include facilities suited for chemical and radiochemical analytical purposes. The facilities include the following equipment:

- 32 x low-level alpha spectrometers
- 8 x low-level gamma spectrometers
- 35 x low-level gas flow beta counters
- 1 x TriCarb and 1 x Quantulus, low-level LSCs
- 1 x ICP-OES and 1 x ICP-MS



## *Analysis under Accreditation — Clear and Transparent*

Since 2012 the radioanalytical laboratory at DTU Nutech carries out radiochemistry and chemical testing for selected elements and radionuclides under accreditation standard DS/EN ISO/IEC 17025:2005.

## *Training on radiochemistry and radioecology*

DTU Nutech offers intensive courses to train students and laboratory staff with technical knowledge and skills to develop sampling, sample preparation and analytical protocols for analysis of radioisotopes of interest. Moreover, the courses are individually tailored to meet the participants' needs.



## **DTU Nutech:**

DTU Nutech is the national competence center for nuclear technologies conducting research in the following scientific areas:

- Radioecology (environmental radioactivity and associated assessments)
- Radiochemistry and radiopharmacy
- Emergency preparedness
- Luminescence physics
- Ionizing radiation dosimetry
- Neutronics (neutron transport through materials)
- Isotope production and accelerator technology

DTU Nutech has a long tradition of transferring research results into numerous services to authorities, hospitals, industry and media. The Center has a solid, international position in environmental research based on nuclear technologies. It has vast experience in characterization and radiochemical analyses of various waste materials and environmental samples from nuclear decommissioning activities.

## **Analyses we offer in the Radioecology Section:**

- 1) Expertise since 1956 on characterization and radiochemical analyses of materials from nuclear facilities
- 2) Complete analytical services including sample preparation, analyses, reporting and advisory support
- 3) Customized analyses to meet individual requirements
- 4) Analytical work performed under accreditation (ISO/IEC 17025:2005)
- 5) Comprehensive training programmes in radiochemistry and radioecology

## **CENTER FOR NUCLEAR TECHNOLOGIES**

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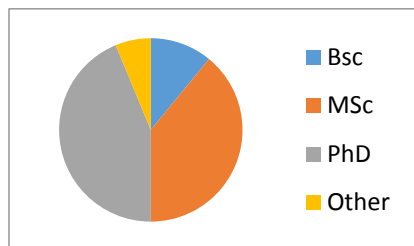
## Appendix 4 - Questionnaire Summary

After the NKS RadWorkshop 2018 event, the participants were asked a number of questions that were thought to be helpful to know the answers to, in evaluating the workshop, and when planning the way forward in addressing Nordic measurement challenges in connection with decommissioning and waste management.

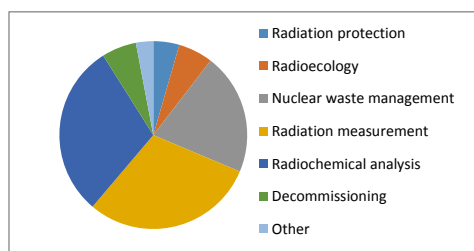
### *Personal and scientific background:*

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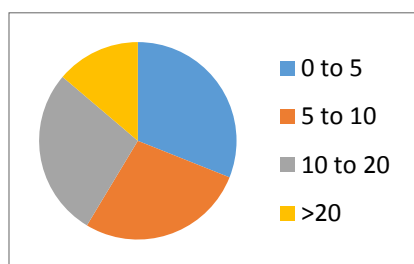
Question: What is the level of your highest education?



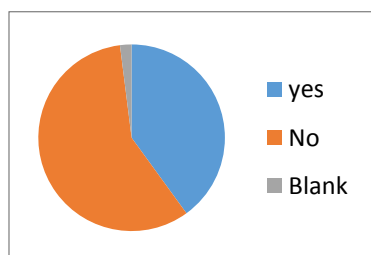
Question: Which work area do you specialise in?



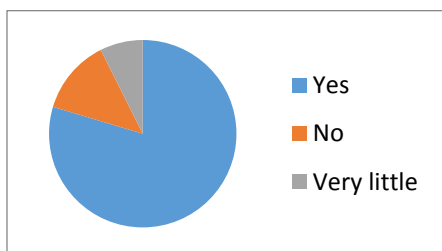
Question: How many years have you worked in this field?



Question: Are you currently working on a decommissioning project?



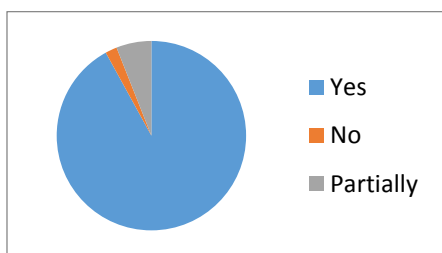
Question: Do you have experience in measuring 'difficult to determine' radionuclides (especially Fe-55, Ni-63, Pu239/240 and Am-241)?



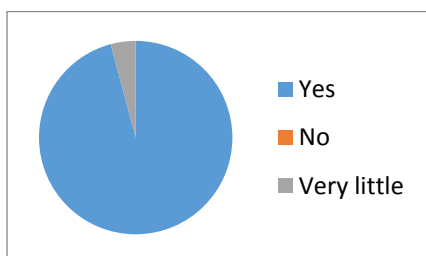
*Present workshop:*

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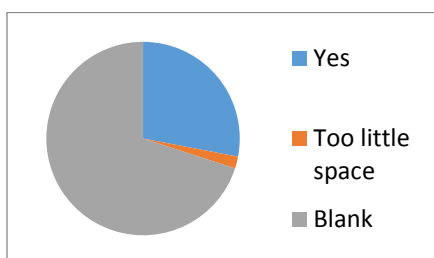
Question: Did the workshop meet your expectations?



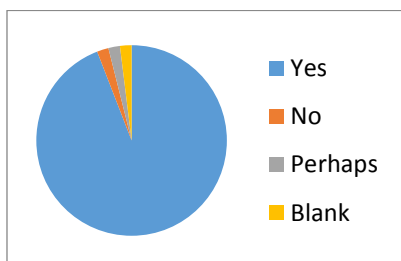
Question: Did the lectures give you new knowledge?



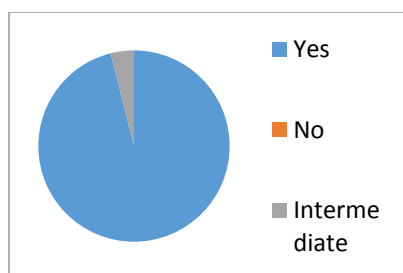
Question: Were the amounts and outcomes of the workshop's lab training OK? (if you participated)



Question: Did you make new useful contacts?



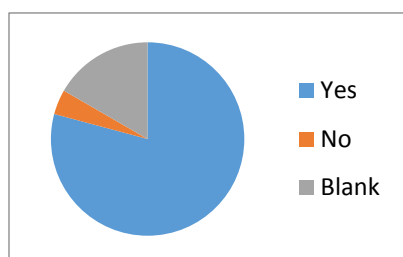
Question: Did you find the workshop well arranged?



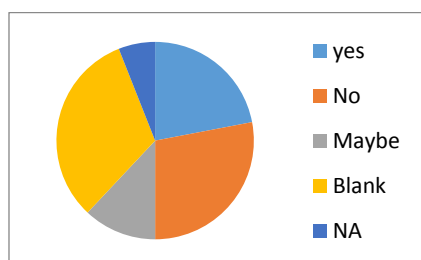
Ideas about the future:

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Question: Should we follow up with more NKS activities on decommissioning/waste measurements?



Question: Would you consider writing an NKS funding application? (only possible for Nordic participants)



Title	NKS-B RadWorkshop 2018 proceedings
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Project	NKS-B / RADWORKSHOP 2018
No. of pages	70
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No. of illustrations	12
No. of references	5
Abstract max. 2000 characters	The NKS-B RadWorkshop 2018 (Workshop on Radioanalytical Chemistry for Nuclear Decommissioning and Waste Management) was held at Risø, Denmark, during 8-12 October 2018. The workshop consisted of 3-day invited lectures and presentations from participants and 2-day lab practice by very experienced professionals. A number of highly reputed experts from authorities, research institutes and nuclear industries were invited to give state-of-the art lectures in different aspects in nuclear decommissioning and waste management with the focus on radioanalytical chemistry. The lab training demonstrated the radiochemical analysis of alpha emitters (Pu-239, 240 and Am-241) and beta emitters (Fe-55, Ni-63)

in nuclear decommissioning materials. 90 participants from 41 international organisations attended the workshop.

**Key words**

Radioanalytical chemistry, workshop, nuclear decommissioning, waste management