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NRD Demonstration Experiments at GELINA

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Abstract

Neutron Resonance Densitometry (NRD), a non-destructive analysis method, is presented. The method has been developed to quantify special nuclear material (SNM) in debris of melted fuel that will be produced during the decommissioning of the Fukushima Daiichi nuclear power plants. The method is based on Neutron Resonance Transmission Analysis (NRTA) and Neutron Resonance Capture Analysis combined with Prompt Gamma–ray analysis (NRCA/PGA). The quantification of SNM relies on the NRTA results. The basic principles of NRD, which are based on well-established methodologies for neutron resonance spectroscopy, are explained.

To develop NRD for the characterization of rock- and particle like heterogeneous samples a JAEA/JRC collaboration has been established. As part of this collaboration a NRD demonstration workshop was organized at the time-of-flight facility GELINA of the JRC-IRMM in Geel (B). The results of this workshop are presented. They illustrate the potential of for measurements of complex mixtures of different elements. It is demonstrated that the elemental composition of an unknown sample predicted by NRTA deviated on average by less than 2% from the declared value. In addition the potential to identify the presence of light elements by NRCA/PGA is shown.

NRD DEMONSTRATION EXPERIMENT

AT GELINA

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Executive summary

Neutron Resonance Densitometry (NRD) is presented as a non-destructive analysis method for the characterisation of special nuclear material (SNM), i.e. ^{235}U and Pu-isotopes. The method has been developed to quantify SNM in debris of melted fuel that will be produced during the decommissioning of the Fukushima Daiichi nuclear power plants. The method is based on Neutron Resonance Transmission Analysis (NRTA) and Neutron Resonance Capture Analysis combined with Prompt Gamma–ray analysis (NRCA/PGA). The quantification of SNM relies on the NRTA results. The basic principles of NRD are explained. They are based on well-established methodologies for neutron resonance spectroscopy, which are applied at the time-of-flight facility GELINA of the JRC-IRMM in Geel (B) for neutron induced reaction cross section measurements

To study and solve these problems the Japan Atomic Energy Agency (JAEA) and the Joint Research Centre of the European Commission (JRC) started a collaboration in 2012 to develop NRD for the characterization of debris samples of the molten cores from the Fukushima-Daiichi nuclear power plants.

To develop NRD for the characterization of rock- and particle like heterogeneous samples the Japan Atomic Energy Agency (JAEA) and the Joint Research Centre of the European Commission (JRC) started a collaboration in 2012. As part of this collaboration a NRD workshop was organized to present the progress made within the collaboration and to demonstrate the performance of NRD at GELINA. The results of this workshop are presented. They illustrate the potential of for measurements of complex mixtures of different elements. It is demonstrated that the elemental composition of an unknown sample predicted by NRTA deviated on average by less than 2% from the declared value. In addition the potential to identify the presence of light elements by NRCA/PGA is shown.

Contents

1. Introduction

Neutron Resonance Densitometry (NRD) is being investigated as a method to quantify special nuclear material (SNM) in rock- and particle-like debris of melted fuel formed during a nuclear accident [1,2]. Such debris samples will consist of a complex mixture of the material present in the reactor core at the time of the accident, i.e. the fuel (U, Pu, minor actinides, fission products), structural materials and neutron absorbing materials from the control and safety rods (e.g. ^{10}B). Due to the presence of SNM, i.e. ^{235}U and Pu isotopes, a careful characterization will be required for safeguards material accountancy at the time of removal of the melted fuel.

NRD is based on Neutron Resonance Transmission Analysis (NRTA) [3,4] and Neutron Resonance Capture Analysis [3,4] combined with Prompt Gamma-ray Analysis [5] (NRCA/PGA). The potential of NRTA as a Non-Destructive Analysis (NDA) method for the characterization of fresh and spent fuel has already been demonstrated by Priesmeyer and Harz [6] and Behrens et al. [7]. Noguere et al. [8] applied NRTA to characterize a PbI_2 sample that was produced from a solution of radioactive waste originating from a reprocessing facility. The samples analysed in Refs. [5,6,7] were homogeneous samples with a regular shape. The analysis of rock- and particle-like debris samples of melted fuel is far more complex and challenging due to their characteristics, in particular [2]:

- the diversity in shape and size;
- the overlapping of resonances dips;
- the presence of neutron absorbing impurities;
- the sample temperature; and
- the radioactivity of the sample.

To study and solve these problems the Japan Atomic Energy Agency (JAEA) and the Joint Research Centre of the European Commission (JRC) started a collaboration in 2012 to develop NRD for the characterization of debris samples of the molten cores from the Fukushima-Daiichi nuclear power plants. The project included 2 milestones:

(1) a report about the use of NRTA and NRCA as NDA methods; and
(2) a demonstration experiment at the TOF facility GELINA of the JRC Geel in Belgium.

A report describing NRTA and NRCA and its applications was published in 2014 [4]. It included an extensive overview of the progress made within the JAEA/JRC collaboration. The results of the demonstration experiments together with the basic principles of NRD are summarised in this report.

2. Basic principles of Neutron Resonance Densitometry

NRD is a NDA method that is based on neutron resonance spectroscopy. It uses the neutron Time-Of-Flight (TOF) technique and relies on well-established methodologies for neutron induced reaction cross section measurements [9]. The amount of SNM is determined by NRTA, while NRCA/PGA is applied to identify the presence of matrix materials and strong neutron absorbing materials. The information derived from NRCA/PGA is used to support the NRTA results, i.e. to determine optimum measurement conditions and improve the analysis of the transmission data [1]. Hence, the final accuracy is mainly determined by the NRTA results.

NRTA is based on an analysis of characteristic dips in a transmission spectrum that can be obtained from a measurement of the attenuation of the neutron beam by the sample. In a NRTA experiment the observed quantity is the fraction of the neutron beam traversing the sample without any interaction. For a parallel neutron beam which is perpendicular to a homogeneous slab of material, this fraction or transmission T as a function of neutron energy E is given by

$$T(E) = e^{-\sum_k n_k \bar{\sigma}_{tot,k}(E)}, \qquad (1)$$

where $\bar{\sigma}_{tot,k}$ is the Doppler broadened total cross section and n_k is the number of atoms per unit area (or areal number density) for nuclide k. Hence, knowing the total cross sections for each of the nuclides present in the sample, one can derive the areal number densities from the transmission.

The experimental transmission T_{exp} is derived from the ratio of the counts of a sample-in measurement C_{in} and a sample-out measurement C_{out}, after subtraction of their background contributions B_{in} and B_{out}, respectively:

$$T_{exp} = \frac{C_{in} - B_{in}}{C_{out} - B_{out}}. \quad (2)$$

The spectra in Eq. 2 are corrected for losses due to the dead time of the detector and electronics chain. All spectra are normalized to the same intensity of the neutron beam and TOF bin width. The background is determined by an analytical expression applying the black resonance technique. A detailed discussion on the background determination can be found in Ref. [8].

Eq. 2 shows that the experimental transmission is independent of both the detector efficiency and incoming neutron flux. Therefore, a transmission measurement is an absolute measurement which does not require additional calibration experiments using representative samples or any reference to a standard cross section. In addition, the experimental observable T_{exp} is a direct measure of the theoretical transmission T (Eq. 1) if the measurements are performed in a good transmission geometry. The conditions of an ideal or good transmission geometry can be achieved by a proper collimation of the neutron beam at the sample and detector position [9].

The areal number densities $n_{1,\ldots,p}$ of the nuclides present in the sample can be derived by a least squares fit to the experimental transmission, that is by minimizing the expression [2]

$$\chi^2(n_{1,..,p}) = (T_{exp} - T_M)^T \, V_{T_{exp}}^{-1} \, (T_{exp} - T_M), \quad (3)$$

where T_M is a model describing the experimental observable. The theoretical estimate T_M is the result of a folding to account for the response function of the TOF spectrometer:

$$T_M(t_m) = \frac{\int R(t_m, E)\, e^{-\sum_{k=1}^{p} n_k \bar{\sigma}_{tot,k}(E)}\, dE}{\int R(t_m, E)\, dE}, \quad (4)$$

with t_m the measured time-of-flight. The theoretical model depends on both resonance and experimental parameters. The resonance parameters are used to parameterize the cross sections by the R-matrix theory [10]. The experimental parameters include e.g. the detector and sample characteristics including sample temperature and the areal number densities $n_{1,\ldots,p}$ of the nuclides present in the sample. The least squares fit can be performed by a resonance shape analysis (RSA) code, such as REFIT [11]. This code, has been especially developed to parameterize cross section data in terms of resonance parameters. It accounts for various experimental effects such as Doppler broadening and the response of the TOF spectrometer and detectors.

The theoretical transmission in Eq. 1 requires that samples are homogeneous. Analytical expressions for homogeneous samples with irregular shapes have been proposed by Harada et al. [12]. A main difficulty for an unbiased analysis of transmission data of rock- and particle-like debris samples is to account for their heterogeneous character. An algorithm that can be applied for the characterization of such heterogeneous samples has been implemented in REFIT by Becker et al. [13]. The algorithm was validated by results of Monte Carlo simulations [14] and experiments [15]. This work was carried out as part of the JAEA/JRC collaboration.

An analysis of the amount of SNM in debris samples originating from melted fuel will be complicated due to the presence of water, concrete, structural materials and in particular boron. Some of them are important

neutron absorbers. Therefore, the transmission will be strongly influenced by the attenuation of the neutron beam due to the presence of these elements. Unfortunately, most of these light elements have resonances only in the high energy region and their presence cannot be identified by transmission measurements at a short flight path. In addition, their content can also not be predicted as in the case of conventional spent fuel from operating reactors. To account for the contribution of strong neutron absorbing matrix materials a method has been proposed and validated in Ref. [2]. The influence of these materials can be taken into account by attributing their contribution to the transmission to a dummy element with a cross section consisting of a constant and a term that is inversely proportional to the velocity of the incoming neutron. This method has been validated by measurements of U_3O_8 samples at GELINA [2]. It was demonstrated that the ^{235}U and ^{238}U content can be determined accurately, i.e. with a bias of less than 2%, even in the presence of a strong absorbing matrix material.

The presence of light elements and ^{10}B can also be identified by NRCA combined with a spectroscopic measurement of prompt γ-rays, i.e. PGA. Most of the light elements emit high energetic prompt γ-rays with a significant intensity after a neutron capture reaction. Such high energetic γ-rays do not suffer from a Compton background due to the detection of γ-rays from the decay of fission products, in particular the 661 keV γ-ray from the decay of ^{137}Cs. On the other hand, the presence of ^{10}B is identified from the 478 keV γ-ray following the $^{10}B(n,\alpha\gamma)$ reaction. The detection of this low energetic γ-ray will be hampered by the presence of the Compton edge at 476 keV resulting from the detection of 661 keV γ-rays. To overcome this difficulty, a LaBr3 γ-ray spectrometer with a favourable full-energy-peak to Compton ratio was designed by JAEA [16,17]. Since a $LaBr_3$ detector has a good time and energy resolution, it can be used for γ-ray spectroscopic TOF measurements. It was shown that with this spectrometer 478 keV γ-rays can be detected even under a high background caused by the presence of ^{137}Cs [17,18].

3. NRD demonstration experiments at GELINA

The performance of NRD was demonstrated by measurements at the 10 m transmission and the 12.5 m capture station of the TOF facility GELINA. Details about this facility, which is installed at the Institute for Reference Materials and Measurements of the JRC (JRC-IRMM) in Geel (B), can be found in Ref. [19]. The demonstration experiment was carried out during a scientific workshop on Neutron Resonance Densitometry held on 4 and 5 March 2015 at JRC-IRMM. It brought together specialists from the International Atomic Energy Agency (IAEA), the US Department of Energy (DOE), the European Commission's Directorate-General for Energy (DG-ENER) and other European and international bodies and institutes. The programme of the workshop and the list of participants are given in Appendix A and B, respectively. Photos taken during the workshop are shown in Figure 1.

Figure 1. NRD workshop photos taken during the presentation by Dr. H. Harada of JAEA (left) and during the opening of the sealed envelope of the NRTA sample (right).

To demonstrate the potential of NRTA and NRCA/PGA two "blind" experiments were organised during the workshop. At the first day representatives of DG-ENER, IAEA and DOE were invited to produce two test

samples: one for NRTA and one for NRCA/PGA. Each test sample consisted of a box which could contain up to 8 different samples chosen from a selection of different materials. After the selection was made the representatives closed and sealed the box and reported the composition of the samples in a sealed envelope. The sealed boxes were placed in the beam at a transmission and capture station of GELINA and measurements were carried out during the night. At the second day of the workshop the data were analysed and the NRTA and NRCA/PGA results were compared with those in the sealed envelopes.

3.1 Demonstration of NRTA

The main emphasis of the NRD demonstration exercise was on the performance of NRTA to quantify elements from complex transmission spectra obtained from transmission measurements at a relatively short flight path. Therefore measurements were performed at the 10m transmission station of GELINA. Details about this station can be found in [20].

Since the workshop participants were not allowed to enter the controlled area, it was not possible to perform the demonstration experiments with highly-radioactive nuclear material. In total 16 samples of medium-weight elements (Cu, Co, Mn, Nb and Rh) and heavy elements (W and Au) with different thicknesses were made available. These elements were chosen based on their characteristic resonances in the eV range in order to mimic the resonance structure of melted fuel components. In one of the cobalt samples a hole was drilled to artificially reproduce the effect of in-homogeneities present in the melted fuel debris. In addition, two strong neutron absorbing B_4C samples were made available. These samples were made out of $^{nat}B_4C$ powder that was pressed in a thin aluminium container. Figure 2 (left) shows the samples that were made available to produce the unknown sample. The final sealed sample box placed at the transmission station of GELINA is shown in Figure 2 (right).

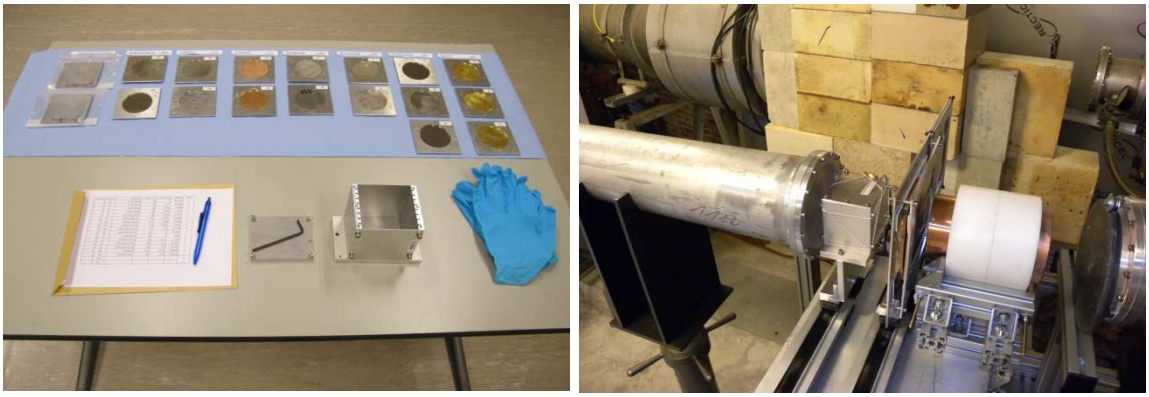

Figure 2. Photo of the samples that were made available for the NRTA demonstration experiments (left) and a photo of the sealed sample box installed at the 10 m transmission station of GELINA (right).

The dead time corrected sample-in and sample-out TOF-spectra together with the corresponding background contributions are shown in Figure 3. The experimental transmission (Eq. 2) was derived from these spectra using the AGS code [21]. This code, which was developed at the EC-JRC-IRMM, has been recommended by the Nuclear Data Section of the IAEA to report TOF cross section data together with the full covariance information in the EXFOR data library [22].

The composition of the sample was obtained from a fit to the experimental transmission. The result of a least squares adjustment with REFIT is shown in Figure 4. The contributions of the different elements and matrix material to the total transmission are also shown. The contribution of the matrix material is based on the introduction of a dummy element as proposed in Ref. [2]. The areal density derived by NRTA and the declared values are listed in Table 1. The declared areal density was derived from a measurement of the total mass and the effective area. The latter was determined by an optical surface inspection with a microscope-based measurement system from Mitutoyo. For homogeneous metal discs the uncertainty on the declared areal density is about 0.1%. The results in Table 1 reveal that the elemental composition derived by NRTA is consistent with the declared composition within 2%.

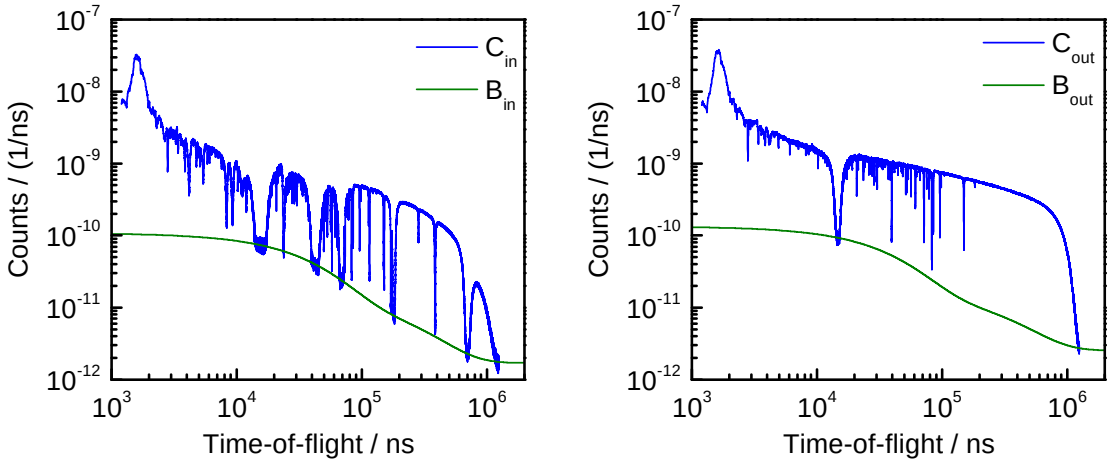

Figure 3. Time-of-flight spectra of the transmission measurements with (left) and without (right) the sealed sample box in the beam. The spectra together with their background contributions are shown. The measurements were taken with a fixed Na background filter and a 4 mm thick Cd overlap filter in the beam.

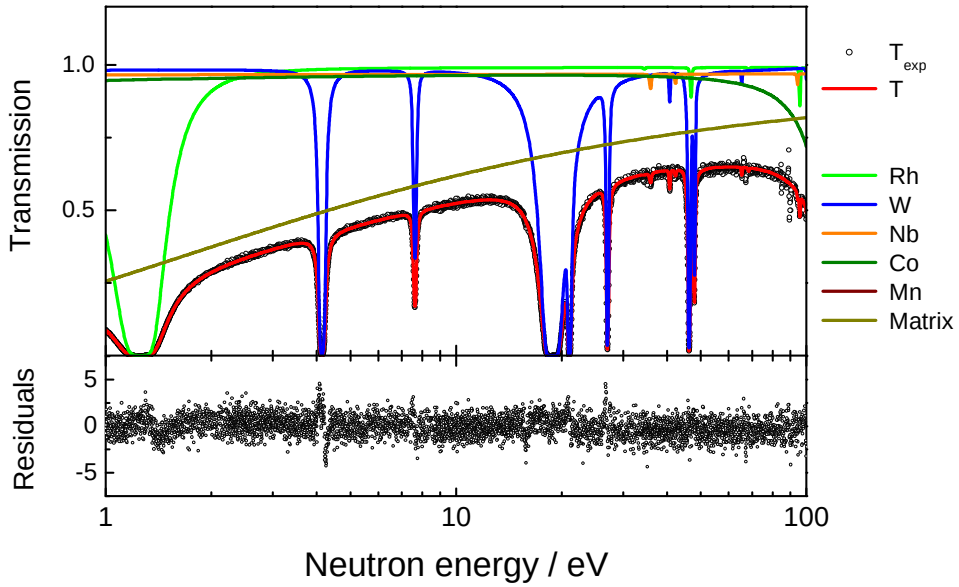

Figure 4. Experimental transmission (T_{exp}) together with the calculated transmission T as a function of neutron energy. The theoretical transmission resulted from an analysis with REFIT. The areal densities were determined from a fit to the experimental data. The contribution of the different elements present in the sample to the total transmission is also shown.

Table 1. Comparison of the declared sample composition (n_D) and the composition derived from NRTA (n_{NRTA}). The ratio between the two values is given in the last column. The NRTA data result from the demonstration experiment carried out during the NRD workshop. The uncertainties on the NRTA data are only due to counting statistics. All uncertainties are quoted at 1 standard deviation.

Element	Areal number density (at/b)		n_{NRTA}/n_D
	n_D	n_{NRTA}	
Mn	1.901 (0.002) x 10^{-2}	1.928 (0.003) x 10^{-2}	1.014 (0.002)
Co	4.583 (0.005) x 10^{-3}	4.509 (0.015) x 10^{-3}	0.984 (0.003)
Cu	0		
Nb	5.485 (0.006) x 10^{-3}	5.382 (0.010) x 10^{-3}	0.981 (0.002)
Rh	1.856 (0.002) x 10^{-3}	1.891 (0.003) x 10^{-3}	1.019 (0.002)
W	2.269 (0.001) x 10^{-3}	2.250 (0.002) x 10^{-3}	0.992 (0.001)
Au	0		

Prior to the NRD workshop a similar exercise was performed to anticipate a possible failure of the GELINA accelerator during the workshop. A nuclear inspector of DG-ENER and a staff member of Oak Ridge National Laboratory ORNL (US) were asked to assemble a test sample. The box was closed and sealed by the DG-ENER inspector before the sample was placed in the beam. The results of this exercise, which were also presented at the workshop, are given in Table 2. Also in this case the composition of the unknown sample derived from NRTA deviated by less than 2% from the declared values. In addition, from an analysis of the profile of the resonance dip close to the 132 eV resonance of ^{59}Co it was concluded that the Co sample was not homogeneous and contained a hole.

Table 2. Comparison of the declared sample composition (n_D) and the composition derived from NRTA (n_{NRTA}). The ratio between the two values is given in the last column. The data result from the demonstration experiment carried out before the organisation of the NRD workshop. The uncertainties on the NRTA data are only due to counting statistics. All uncertainties are quoted at 1 standard deviation.

Element	Areal number dentsity (at/b)		n_{NRTA}/n_D
	n_D	n_{NRTA}	
Mn	1.901 (0.002) x 10^{-2}	1.886 (0.002) x 10^{-2}	0.992 (0.002)
Co	4.585 (0.005) x 10^{-3}	4.550 (0.066) x 10^{-3}	0.992 (0.015)
Cu	0		
Nb	0		
Rh	0		
W	1.337 (0.001) x 10^{-3}	1.334 (0.002) x 10^{-3}	0.998 (0.002)
Au	6.844 (0.007) x 10^{-3}	6.862 (0.005) x 10^{-3}	1.003 (0.001)

3.2 Demonstration of NRCA/PGA

The NRCA/PGA demonstration experiment was carried out at the 12.5 m capture station of GELINA. Details about this measurement station can be found in [23]. An additional large volume $LaBr_3$ detector was placed close to the sample. This detector was used to determine the presence of ^{10}B and structural materials from γ-ray spectroscopic measurements. Details about this detector are given in Refs. [16,17]. For this exercise the DG-ENER, IAEA and DOE representatives could choose out of a Fe, Ni, Cr, Gd and HF metal disc and 2 B_4C samples.

The γ-ray spectrum in Figure 5 (left) was used to verify the presence of ^{10}B and light elements. The TOF spectrum in Figure 5 (right) was used to identify the presence of the medium and heavy elements with resonances in the low energy region below 100 eV. From these spectra it was deduced that Ni, Hf and Gd samples were present in the box. This observation was confirmed by the the DG-ENER, IAEA and DOE representatives producing the unknown sample.

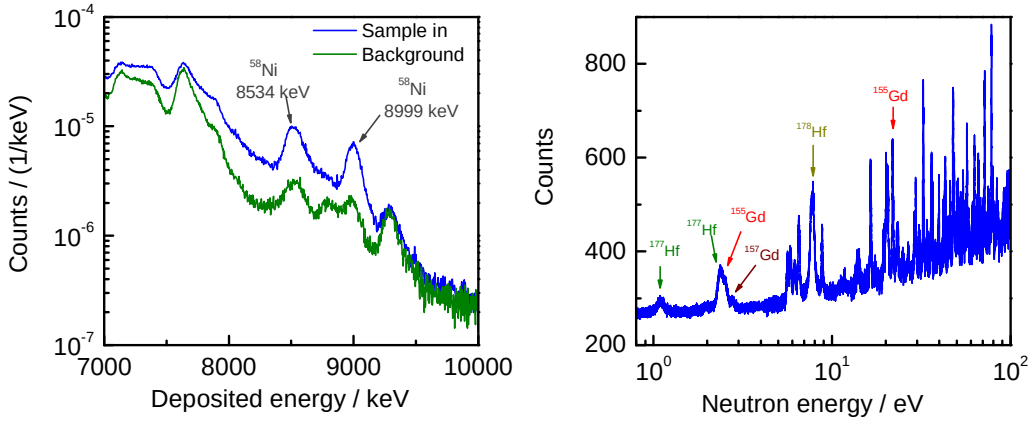

Figure 5. Gamma-ray spectra obtained with the $LaBr_3$ detector (left) and TOF spectrum (right) taken with the same detector. The spectra result from the demonstration experiment carried out during the NRD workshop. The prompt γ-rays indicating the presence of Ni and the resonances indicating the presence Hf and Gd are specified.

4. Summary and conclusions

Neutron Resonance Densitometry (NRD), a non-destructive analysis method, was presented. The method has been developed to quantify special nuclear material (SNM) in debris of melted fuel that will be produced during the decommissioning of the Fukushima Daiichi nuclear power plants. The method is based on Neutron Resonance Transmission Analysis (NRTA) and Neutron Resonance Capture Analysis combined with Prompt Gamma–ray analysis (NRCA/PGA). The quantification of SNM relies on the NRTA results. The basic principles of NRD, which are based on well-established methodologies for neutron resonance spectroscopy, have been explained.

To develop NRD for the characterization of rock- and particle like heterogeneous samples a JAEA/JRC collaboration has been established. As part of this collaboration a NRD demonstration workshop was organized at the time-of-flight facility GELINA of the JRC-IRMM in Geel (B). The potential of NRD was demonstrated by measurements on a complex mixture of different elements. It was demonstrated that the elemental composition of an unknown sample predicted by NRTA deviated on average by less than 2% from the declared value. In addition the potential to identify the presence of light elements by NRCA/PGA was shown.

Acknowledgments

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Appendix A

Workshop on Neutron Resonance Densitometry (NRD)

04 –05 March, 2015

IRMM | Geel, Belgium

Programme

Day 1 | Wednesday, 4 March, 2015

13h45 – 13h55	Opening of the workshop	E. Anklam JRC-IRMM
Introduction of Neutron Resonance Densitometry (NRD)		
13h55 – 14h25	Principles of NRTA and NRCA	P. Schillebeeckx JRC-IRMM
14h25 – 14h50	Challenge towards quantification of SNM from Fukushima Daiichi's by NRD	H. Harada JAEA-NSEC
Demonstration (1)		
14h50 – 15h15	Explanation of demonstration	J. Heyse JRC-IRMM
15h15 – 16h15	Visit TOF-facility GELINA	
16h15 – 16h35	Coffee break	
Progress in the development of NRD		
16h35 – 17h00	Analysis of heterogeneous samples	B. Becker
17h00 – 17h25	How to measure prompt γ-rays under strong background conditions	M. Koizumi JAEA -NSEC
17h25 – 17h50	Results of NRTA on nuclear materials	S. Kopecky JRC-IRMM
17h50 – 18h15	A conceptual NRD system and its performance evaluation	H. Tsuchiya JAEA - NSEC

Day 2 | Thursday, 5 March 2015

Demonstration (2)		
09h00 – 09h15	Qualitative discussion of NRTA data	C. Paradela JRC-IRMM
Other active neutron NDA technologies		
09h15 – 09h40	NDA methods developed at KURRI	K. Nakajima Kyoto Univ.
09h40 – 10h05	NDA methods developed at JRC-ITU	B. Pedersen JRC-ITU
10h05 – 10h30	NDA R&D activities at JAEA	M. Kureta JAEA – NSEC
10h30 – 11h55	Differential die-away techniques	V. Henzl LANL
11h55 – 11h20	Coffee Break	
Demonstration (3)		
11h20 – 11h50	Presentation of quantitative NRTA results	S. Kopecky JRC-IRMM
11h50 – 12h15	Presentation of quantitative NRCA results	M. Koizumi JAEA-NSEC
12h15 – 13h45	Lunch	
Importance of NDA and future prospects		
13h45 – 14h10	Detection of delayed γ-rays	V. Mozin, LLNL
14h10 – 14h35	Overview of R&D Activities Targeting IAEA Needs in Non-destructive Assay of Nuclear Materials	S. Zykov, IAEA
14h35 – 15h00	Non-destructive analysis research overview	A. Bakel, US - DOE
15h00 – 15h25	Development activities on NDA in Japan	M. Seya JAEA-ISCN
15h25 – 15h50	NDA for decommissioning and spent fuel	K. van der Meer, SCK•CEN
15h50 – 16h00	Closing remarks	Y. Naoi IAEA – ISCN S. Abousahl JRC-HQ

Appendix B

Workshop on Neutron Resonance Densitometry (NRD)

04 –05 March, 2015
IRMM | Geel, Belgium

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