Milestone 28
Radiochemical procedures for the selected DTM radionuclides
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Abstract

Nuclear waste from dismantling operations consists of a variety of materials (e.g. steel, concrete, resins), forms (liquid, solid), and types of emitters (alpha, beta, gamma). The difficulty lies in the different stages of preparation, from sampling to measurement.

As part of the Predis project in WP4 "Innovations in metallic material treatment and conditioning", we focused on optimizing the separation and purification of four radionuclides (^{59,63}Ni, ^{41}Ca and ^{93}Zr) and their measurement. Our goal was to propose highly selective and efficient separation and purification, and to develop a sensitive to ultra-sensitive measurement method, depending on the radionuclide.

For the measurement of ^{59,63}Ni, two approaches were developed. The first approach targeted to improve evaporation techniques on membrane filters, employed for target preparation and evaluations were performed for ^{55}Fe, ^{60}Co and ^{59,63}Ni. A standard curve for ^{55}Fe showed excellent linearity. The effect of ^{60}Co contamination was also investigated, showing a decrease in signal response at low activities. Results for ^{59}Ni and ^{63}Ni activities also showed good linearity. The second approach was dedicated to a new method involving galvanostatic deposition of nickel. The deposited nickel is measured for ^{59}Ni activity by gamma spectrometry, followed by its immersion in a scintillation cocktail for ^{63}Ni measurement. This approach simplifies the analysis and reduces the need for additional subsamples.

Methods of isobar suppression was also investigated to improve measurement accuracy to distinguish ^{41}Ca from interfering isotopes such as ^{41}K. Various target matrices were tested to identify effective suppression techniques. The goal was to optimize sensitivity and accuracy for ^{41}Ca analysis by AMS. Various conditions were tested to find the best parameters for Zr measurement with a validation of the method showing accurate Zr measurement in the presence of other metals. Purification protocols using chromatographic resins were optimized to remove interfering elements, resulting in Zr good recovery efficiencies. The measurement of ^{93}Zr using liquid scintillation counting was also investigated, providing insight into detection methods.

Keywords

^{59,63}Ni, ^{41}Ca, ^{93}Zr, separation, purification, ultra-sensitive measurement

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1   Milestone Description

Milestone 28, associated with Work package 4, Task 5 has been completed on 30.08.2024.

The justification for the readiness is described below and complies with the Grant Agreement Description of Action noting verification by M4.7 Deliverable 4.7 available.

The readiness of the milestone was reviewed and agreed upon by Abdessalam Abdelous (IMT) as WP4 leader

2   Introduction

Radionuclides of very low energy emission may be present in certain metallic radioactive waste as surface contaminants or neutron activated radionuclides in the base metal. Their potential environmental impact arises if released during decommissioning, dismantling operations, and the assessment of nuclear waste intended for disposal. These radionuclides are categorized as easy-to-measure (ETM) and difficult-to-measure (DTM) radionuclides, typically estimated using scaling factors, which multiply the activity of a key radionuclide (^{60}Co, ^{137}Cs) by a pre-determined factor [1]. Non-destructive methods, such as gamma or neutron measurements, provide assessments of radioactivity within a package without the need for sampling. Destructive measurement methods, which are particularly valuable when limited information (such as chemical composition or irradiation conditions) is available for historical or inherited waste, complement these non-destructive approaches [2]. Accurate quantification of these radionuclides requires a robust and validated radiochemical procedure, considering their characteristic low-energy (E<100keV) and/or low-intensity emissions. This milestone reports the results for four radionuclides selected during the PREDIS project:

- ^{59}Ni: 7.6×10^4 y, β^+, EC, $E_{X-K\alpha1}$ = 6.93 keV (20%), $E_{X-K\alpha2}$ = 6.91 keV (10%), $E_{\gamma\pm}$ = 511 keV,
- ^{63}Ni: 98.7 y, β^-, E_{max} = 66.98 keV (100%),
- ^{41}Ca: 9.94×10^4 y, EC (100%), $E_{Auger\ K}$ = 2.97 keV (76.6 %), $E_{X-K\alpha1}$ = 3.314 keV (7.6%), no γ
- ^{93}Zr: 1.61×10^6 y, β-, E_{max} = 59.5 keV (73%) and 90.3 keV (27%) [3].

^{63}Ni is a neutron activation product activated by neutron capture of stable ^{62}Ni in for example steel components of primary coolant system of nuclear reactors. ^{63}Ni is a pure beta emitter with a low energy (E_{max} = 66.98 keV) analysed generally using liquid scintillation counting (LSC). The sample preparation for the LSC measurement is quite simple after radiochemical processes using for example ion exchange and chromatographic resins. The other radioactive isotope activated by neutron activation is a very long-lived ^{59}Ni. ^{59}Ni decays by electron capture with X-ray emissions. Both ^{63}Ni and ^{59}Ni are purified in the same fraction as they cannot be chemically separated. However, due to the low sensitivity of ^{59}Ni X-rays in LSC, ^{59}Ni requires another measurement method.

^{41}Ca is also neutron activation product produced by neutron capture of stable ^{40}Ca (97 %) mainly in the concrete shielding of the reactor vessel and related places with high neutron fluxes. ^{41}Ca is a very long-lived (10^5 y), pure EC radionuclide with very low energy emissions of Auger electrons (2.97 keV) and X-rays (3.314 keV as the most intensive). Both these emissions are used for its radiometric detection. For Low Energy Gamma-ray Spectrometry (LEGS) the main drawback is very low measurement efficiency, usually less than 0.1 %, and high (self) absorption of the photons. The more commonly used LSC requires precise separation of interferents and pure sample suppressing quenching. The main problem is interfering ^{45}Ca, that is also produced from stable ^{44}Ca (2 %), and is pure beta emitter (E_{max} = 256 keV) with decent half-life 163 days. ^{45}Ca is not chemically separable from ^{41}Ca and thus only sufficiently old samples can be well measured with LSC. Mass spectrometric

methods are struggling with isobaric interference of [41]K, which can be partially suppressed by chemical and physical separation, but still provides major complication in measurement of low and medium [41]Ca activities. The best option for measurement provides Accelerator Mass Spectrometry (AMS), when using $^{41}CaH_3^-$ beams reaching $^{41}Ca/^{40}Ca$ ratio in order 10^{-16}, but in standard operation with CaF_3^- beam it reaches 10^{-11} ratio.

[93]Zr, a long-lived radionuclide, is generated through the fission reactions involving uranium and plutonium. It is also a neutron activation product originating from stable Zr present in PWR nuclear fuel cladding. The contribution of [93]Zr to the overall waste inventory persists at long term, coexisting with Tc-99. The decay of [93]Zr occurs through beta emission, characterized by its low energy, giving measurement challenges when utilizing liquid scintillation counting.

The objectives were to provide a highly selective and efficient separation and purification, and to develop a sensitive to ultra-sensitive method of measurement, depending on the radionuclide.

3 Sample preparation

3.1 Sample preparation at VTT

A literature surveys showed that analysis of [59]Ni can be carried out with several different ways. The analysis can be divided into three steps; purification of the Ni fraction, preparation of the measurement target and measurement of the measurement target. In VTT's case, purification of the Ni fraction was previously published and tested [4–7]. The analysis of [59]Ni is directly linked with [63]Ni as they are purified in the same fraction. The purification procedure consists of an acid digestion followed by a hydroxide precipitation. [59,63]Ni fraction is separated from [55]Fe using an ion exchange resin. [59,63]Ni fraction is purified from for example ^{60}Co using a chromatographic Nickel resin, in which Ni forms a complex with dimethylglyoxime (DMG). The eluate from the Nickel resin was evaporated to 1-2 ml and an aliquot of the purified fraction was measured using LSC. However, design of the measurement target was required and consequently design of the measurement configuration was required. Taddei et al. [8] reported use of [55]Fe instead of [59]Ni efficiency calibration due to their similar X-ray energies. In general, [59]Ni standard solutions are commercially available but expensive. The measurement target preparation procedure by Taddei et al. [8] included dilution of the purified Ni fraction to 50 ml of deionised water after which the pH is adjusted to 9 with 25% NH_4OH. After pH adjustment, 5 ml of 1 % DMG solution in ethanol is added with stirring. Decanted Ni-DMG precipitate is filtered on a cellulose acetate filter and washed with 3 ml of ethanol three times. The filters are dried at 30 °C for 12 hours. This procedure was tested as other procedures seemed to require extra steps with water baths [12-13]. Taddei et al. [8] utilised a Low Energy Germanium detector in the activity measurements and inductively coupled plasma optical emission spectrometry (ICP-OES) in yield measurements. The measurement of [59]Ni at VTT was selected to be carried out using a Broad Energy Germanium detector (BEGe).

3.1.1 Measurement target preparation

Different types of measurement targets were studied. Initially, Ni-DMG precipitations were filtered on membrane filters following the procedure by Taddei et al. [8]. As the results were inconsistent, direct evaporation was studied.

3.1.2 ^{55}Fe standard curve

A ^{55}Fe standard curve was established by preparation of several ^{55}Fe standard measurement targets between activities of 20 Bq and 1000 Bq. Each standard was measured at least 3 times to study repeatability.

3.1.3 ^{60}Co contamination

Ni and Co behave chemically similarly and consequently ^{60}Co contamination can be detected in the purified Ni fraction. Even though the ^{60}Co can be removed by repetition of the Nickel resin purification, a study was carried out to establish the possible effect of ^{60}Co contamination in ^{59}Ni measurement.

3.1.4 Study of ^{59}Ni and ^{63}Ni in high activity steel sample

Radiochemical analysis of ^{59}Ni and ^{63}Ni were carried out for high activity stainless steel samples with 3 activity concentrations (i.e. different irradiation profiles). The ^{63}Ni analyses were carried out according to the published method [5] and ^{59}Ni with the new analysis method. In total, 8 analyses of sub-samples were carried out.

3.2 Sample preparation at CTU

3.2.1 Sample preparation for mass spectrometric analysis of target matrices

All samples for mass analysis at Tandetron MC-4310 were prepared by transferring approximately 200 mg of the compound to be analysed with the selected additive in a mass ratio of 1:1 (or 1:1:1 if a third compound was used) as a well-mixed dispersed powder into a silver or copper cathode. The powder was then suitably pressed in the cathode and placed into the caesium ion source.

For Ca analysis, precipitated CaF_2 in the mixture of PbF_2 in the 1:1 mass ratio was pressed into silver cathode. CaF_2 was prepared by precipitation of $CaCl_2$ solution with NH_4F, product was carefully washed with deionized water and ethanol and dried in vacuum drier.

Potassium samples were prepared by reaction of $AlCl_3$ with KOH, when 40% HF was added to the final solution. Precipitate K_3AlF_6 was again washed and dried in vacuum drier. KF was not selected due to its hygroscopicity, solubility in water and possible content of HF.

Samples for Ni and Co, were prepared from the respective metal powders mixed with PbF_2 in the mass 1:1 ratio. All chemicals were of analytical purity.

3.2.2 Sample preparation for electrodeposition of Ni

For all the electrodeposition experiments, 30 mL of the electrolyte was used per experiment as the optimum volume of the cell. Parameters of the process were optimized, so concentrations, time regime, electric potential and current were varied. During optimization, concentration of nickel was varied in the range 10^{-4} up to 10^{-2} mol/L.

3.2.3 Sample preparation for AMS

CaF_2 samples were prepared by precipitation from distilled water (1,2), sample (3) by precipitation from ultrapure water and finally sample (4) by precipitation from tap water. This should mimic variation in potassium content. After precipitation and centrifugation, all samples were washed three times with ethanol to purify the sample from unreacted reactants - mainly NH_4F. In the case of sample (2), three washes with ultrapure water were performed in sequence. Finally, the samples were dried

in a vacuum oven for 24 hours and pressed into Cu or Ti cathodes with selected additives (Nb powder, Ag powder, PbF_2).

3.3 Sample preparation at IMT

3.3.1 Preparation of stock solutions

An approach in two steps was developed to optimize the measurement of Zr from metallic samples: 1- synthetic solution, 2- non-oxidized metal sample, 3- oxidized metal sample.

a. Synthetic solution

Utilizing the known metal composition of Inconel 600, employed in a PWR steam generator, a laboratory-synthesized solution of Fe, Ni, and Cr was prepared using salts with a purity level of 99% from Sigma Aldrich (for iron sulfate heptahydrate), Acros Organics (for chromium nitrate nonahydrate) and Merck (for nickel nitrate hexahydrate). The targeted concentrations for this solution were set at [Ni] 0.50 mol/L, [Cr] 0.12 mol/L, and [Fe] 0.07 mol/L. Additionally, a stable Zr was added to the solution at a concentration of 1×10^{-8} mol/L to simulate its presence in a real sample.

The motivation behind using this simplified synthetic solution was to enhance the efficiency of a separation-purification method. This method relies on a series of chromatographic resins, specifically UTEVA and TK400. The use of a synthetic solution was aimed at mitigating potential impurities that could interfere with optimization processes. Moreover, it served to assess the detection and quantification limits of Inductively Coupled Plasma Mass Spectrometry (ICP-MS).

b. Non-radioactive metallic sample

Non-radioactive Inconel 600 and stainless steel samples were digested to prepare a stock solution for subsequent use, following a similar procedure as employed for the synthetic solution. Both non-oxidized and oxidized metal samples were digested at 60°C using a mixture of nitric HNO_3 and HCl between 4 to 10 mol/L, known as aqua regia. This digestion aimed to validate a procedure to solubilize the metal sample into a liquid form. This step serves the dual purpose of confirming the optimization achieved in the preceding phase and closely simulating a complex real-world system.

In this context, additional chemical elements were introduced to the solution to mimic the potential presence of contaminants such as Mn, Sb, and Ce. The incorporation of these elements accounted for potential interference during the subsequent separation-purification step or analyses, particularly if present at trace or ultra-trace levels.

3.3.2 Standard solutions

Purchasing ^{93}Zr as a standard solution directly proved challenging due to its limited production availability. Similarly, obtaining a non-certified ^{93}Zr faced difficulties for the same reason. As an alternative, consideration was given to substituting it with ^{95}Zr (E_{max} = 368.1 keV (54.46%) and 400.6 keV (44.34%) [3]). However, it was noted that the energy range emission of ^{95}Zr differs from that of ^{93}Zr (E_{max} = 59.5 keV (73%) and 90.3 keV (27%) [3]).

Consequently, ^{63}Ni emerged as a suitable choice for a standard radioactive source. ^{63}Ni possesses a comparable β^- energy range of emission, with E_{max} = 66.98 keV (100%). This substitution

overcomes the difficulties posed by the limited availability of ^{93}Zr, providing a more practical and feasible solution for standardization within the experimental setup.

4 Characterisation

4.1 Germanium detector

The quantification of ^{59}Ni at VTT was carried out using Mirion Broad Energy Gamma Detector (BEGe). The efficiency calibrations are carried out using In-situ Object Counting System (ISOCS) in which the lowest point of efficiency curve is at 10 keV. Efficiency calibration for the low energy ^{59}Ni X-rays were prepared experimentally using ^{55}Fe.

4.2 Multifunction system Tandetron MC 4310

For the measurement of mass spectra of the selected ions and target matrix compounds, a Tandetron linear accelerator located in the Laboratory of Tandetron of the Nuclear Physics Institute (NPI) of the Czech Academy of Sciences (CAS) was used. It belongs among facilities of the Centre of Accelerators and Nuclear Analytical Methods Research Infrastructure (CANAM RI) (http://canam.ujf.cas.cz). The medium-current version Tandetron (MC-4310, High Voltage Engineering Europe, Netherlands) with a maximum terminal voltage of 3 MV is used for the production of ion beams with energies in the range from 600 keV to about 30 MeV. For the purpose of testing matrices for potential use in the AMS, a Cs sputtering source for the production of ions from the solid-state target was used, which should provide similar sputtering and ion production as the AMS one. The simplified scheme of the system is shown on Figure 1 and description can be found in [9].
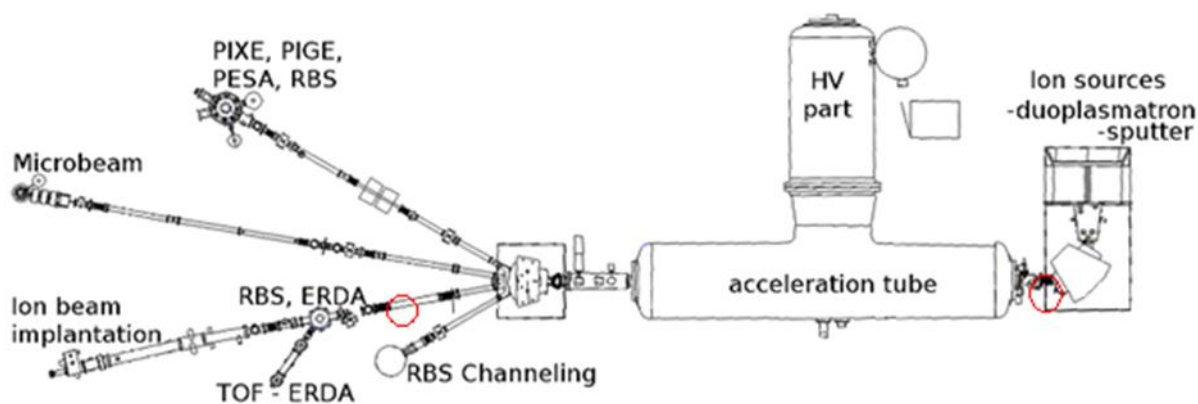
Figure 1. Overview of Tandetron 4130 MC with marked Faraday cups used for measurement at the Laboratory of Nuclear analytical methods, CAS, Řež

For the matrix analysis, only the low energy part of the system was used, a Faraday detector directly behind the magnet was used for the mass analysis of the beam. Ion sources setting used during mass spectra analysis are in the Table 1.

Table 1: AMS ion sources setting

Parameter	Value	Parameter	Value
Caesium temperature	29–30 °C	Extraction potential	30 kV
Cathode potential	2.5 – 5 kV	Lens 1 potential	10.9 kV
Total ion source current	max 2.5 mA	Lens 2 potential	14.3 kV

4.3 Accelerator mass spectrometry

A Multi-Isotope Low-Energy Accelerator mass spectrometer (MILEA, Ionplus AG, Switzerland) is based on vacuum-insulated design. The inbuilt accelerator operates at low terminal voltages of up to 300 kV. It is designed to determine ^{10}Be, ^{14}C, ^{26}Al, ^{41}Ca, ^{129}I, ^{236}U, ^{239}Pu and other actinides with the limits of detection several orders of magnitude lower in comparison with conventional radiometric and mass spectrometric techniques. The low-energy part of the instrument comprises a Cs-sputtering negative ion source, an achromatic combination of 90° electrostatic and magnetic deflectors. The negative ions pass through a 300 kV tandem accelerator on a vacuum-insulated high-voltage platform with a He-stripping chamber. A quadrupole triplet after the accelerator unit provides similar ion optical conditions for all measured isotopes and facilitates tuning. The following high-energy side consists of 90° magnetic, 120° electrostatic and 110° magnetic deflectors. A low-noise ΔE-E_{res} gas ionisation chamber at the end of the beam line provides final separation and identification of interfering particles [10].

Tuning for ^{41}Ca was derived from parameters of ^{129}I and ^{26}Al, with the accelerator voltage of 260 kV, where ^{40}Ca was measured in the HE-4 Faraday cup.

4.4 Liquid scintillation counting

The quantification of ^{63}Ni at VTT was carried out using Hidex 300 SL liquid scintillation counter. An 100-1000 µl aliquot of the purified Ni fraction was mixed with 10 ml HiSafe 3 scintillation cocktail. Triple-to-Double-Count-Rate (TDCR) with CoreF correction was used in the efficiency calibrations. The ^{55}Fe activities in the measurement targets were analysed similarly using TDCR with CoreF correction.

At CTU, the LSC measurement was carried out in the Triahler (Multilabel tester type 425-004, Hidex, portable scintillation counter, built in Pb shielding, alpha – beta separation, S/N 2010301) using modified H-3 settings, and in the laboratory experimental setup using naked photomultiplier in the light-tight shielding (details of the measurement windows, sample geometry and composition in chapter below).

The quantification of ^{63}Ni by IMT, serving as a standard to simulate ^{93}Zr measurement, was conducted through Liquid Scintillation Counting (LSC, Tri-Carb 3170 TR/SL, Perkin Elmer) employing a conventional Tri-Carb counter. Various scintillation cocktails, Ultima Gold LLT or Ultima Gold AB, were combined with the standard to assess the optimal detection efficiency and determine the associated merit factors.

4.5 ICP-MS

The analysis of Zr, Fe, Ni, Cr, Mn, Sb, Ce, Y, Nb and Mo are performed by an inductively coupled plasma mass spectrometry (Quadrupole ICP-MS Xseries 2, Thermoelectron). Internal standards are also used with In-115, Sc-45 and Bi-209 to correct the final concentrations. A combination of HNO_3 5% and HCl 1 mol/L solution is used for the dilutions, and the rinsing of the instrument is performed with a solution of HNO_3 2% and $H_2C_2O_4$ 10^{-4} mol/L. The quantification limits are: 1.5×10^{-10} mol/L for Zr, 1.2×10^{-8} mol/L for Fe, 6.5×10^{-10} mol/L for Ni, 7.5×10^{-10} mol/L for Mn, 5.6×10^{-10} mol/L for Sb, 2.6×10^{-10} mol/L for Ce, 1.5×10^{-9} mol/L for Y, 4.4×10^{-10} mol/L for Nb and 4.7×10^{-10} mol/L for Mo.

4.6 ICP-OES

The Ni yield analyses were carried out using Agilent SVDV 5100 ICP-OES at VTT. Multielement standard solutions were used in the concentration calibrations.

5 Conclusion and Perspectives

The protocol of separation and purification for the measurement of Zr from a complex metallic solution is reached with a good recovery (~90%) and low detection limit by LSC of 0.04 Bq/g based on ^{63}Ni calibration curve used as a surrogate, and by Q-ICP-MS of 1.5×10^{-10} mol/L. This protocol needs a validation with a standard solution of ^{93}Zr, currently in discussion with potential suppliers.

The ^{59}Ni method development by VTT resulted in an established protocol for the radiochemical analysis and detection of X-rays. The established protocol was also implemented in analysis of real stainless steel samples with consistent results. A peer reviewed article on the ^{59}Ni analysis in high activity steel is under preparation.

The established protocol will be assessed in the MoNi intercomparison exercise 2024 organised within Nordic nuclear research program. The results will be available as an NKS report early 2025 and a peer-reviewed publication in late 2025. Additionally, during the final months of PREDIS, VTT has been carrying out method development for radiochemical analysis and ICP-MS detection of ^{93}Mo. Interest has been given additionally to ^{93}Zr and ^{94}Nb as their purification is included in the ^{93}Mo procedure. The ^{93}Mo analysis performance will be also assessed in the MoNi intercomparison exercise. The concept of ICP-MS in DTM detection will be continued in EURAD 2 ICARUS project.

The Ni preparation with the electroplating method provides high yield (>99 %) and repeatability for Ni recovery from the solution was achieved, with the overall measurement efficiency of ^{63}Ni on the planchette of (ca 56 %). On the other hand, plating from solution with Ni carrier of 1 mmol/L resulted on 93% self-absorption. Further testing also with ^{59}Ni will be necessary to get full picture of the procedure for its comparison with standard methods using Ni-DMG precipitation and finalize it in publication, mainly in testing of carrier-free solutions. In addition, in the mass spectrometric screening, it was proven that using fluoride matrices cannot suppress isobaric ^{59}Co and so AMS machine with low terminal voltage is not suitable for ^{59}Ni measurement without any additional improvements.

In the case of ^{41}Ca, Tandetron measurement suggested option to use [CaF$_4$]- molecular ion to suppress the isobaric ^{41}K. During AMS measurement campaign, tuning the machine showed that in the sample composition tested – mixture of CaF$_2$ and PbF$_2$ in the 1:1 ratio, it is possible to use standard [CaF$_3$]$^-$ molecular ion, but current from the expected [CaF$_4$]$^-$ was too low. This will require more ^{41}Ca standard material to be tested with increased amount of PbF$_2$ to find optimum ions source chemical conditions. The research in this direction will continue to get validated information about [CaF$_4$]$^-$ availability from the AMS MILEA sputter source and related ^{41}K-suppressing sample preparation. For this purposes ^{41}Ca standards with precise and levelled ^{41}Ca/^{40}Ca isotopic ratio will be used. In addition, CTU continues in testing of selenium and palladium to get better knowledge about their behaviour in the ion sources of AMS machines.

All the mass spectrometry screening experiments are summarized in master thesis of F. Babčický and are planned to be published in prepared summary publication.

6 Acknowledgements

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