



Non-destructive analysis results of the second fuel debris sample (Prompt report)

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This report is related to the FY2025 Subsidized Project of Decommissioning, Contaminated Water and Treated Water Management (Development of Analysis and Estimation Technology for Characterization of Fuel Debris).



- During the second trial retrieval, a fuel debris sample was taken from the floor inside the Unit 2 pedestal.
- The fuel debris sample was received by the JAEA Oarai Nuclear Engineering Institute Irradiated Fuel Assembly Test Facility (Fuels Monitoring Facility; FMF) on April 25 and non-destructive analysis commenced on April 28







Fuel debris sample inside a specimen container

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 These are the latest results following the conclusion of non-destructive analyses of external, mass, dose rate and γ-ray spectrometry.



*1 Overview and purpose of each analysis are described in the reference documents



Results from the external appearance observation of the second fuel debris sample



External appearance of fuel debris sample (color samples (color separation guide) with scale)



Enlarged photos of external appearance of fuel debris sample (taken from directly above) 4

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External appearance observations, mass, and dose rate measurements for the second fuel debris sample





(Front side: Photo of the same surface as the previous page photographed from approx. 45 degrees diagonally)



(Back side: Photographed from approx. 45 degrees diagonally)

Enlarged photos of external appearance of fuel debris sample

<External appearance>

- The received fuel debris sample is heterogeneous.
- Overall, the sample is brownish bronze (lighter in color than the first sample*) with black areas and holes found on the surface.*As seen with the naked eye
- The largest sample measured using a scale was approx. 5mm x approx. 4mm

<Mass> 0.187g (Total mass of the sample)

<Dose rate> Approx. 0.3mSv/h

(γ -rays: An ionization chamber was used to measure the sample while it was still inside a polypropylene container (at a distance of 1~2 cm from the sample)) * IP imagery (dose distribution) could not show an accurate distribution due to the high dose rates and small size of the sample.



Results of γ-ray spectrometry measurement of the second fuel debris sample





- Since Am-241, which is produced by neutron capture reaction of U-238 in the nuclear fuel, etc., is detected in addition to Eu-154, the sample is considered to contain nuclear fuel components.
- The detected nuclides are the same as those detected from the first fuel debris sample.





[Summary]

- The received sample is heterogeneous and brownish bronze overall (lighter in color than the first sample) with black areas and holes found on the surface.
- The largest sample is approx. 5mm x approx. 4mm, the mass is 0.187g and dose rate (γ-rays) is approx. 0.3mSv/h.
- γ-Ray spectrometry detected Am-241, thereby indicating the presence of fuel components.

[Future analysis plans for the second sample]

- Non-destructive analyses will continue and the results compiled during the summer. Updates will be provided.
- Detailed analysis (solid analysis and liquid analysis) will take one year to one and a half years after receipt at the analysis institutes after which the results will be compiled.

) Reference Analysis purpose of the fuel debris sample **TEPCO**

- By analyzing the obtained sample, grasp the condition of the sampled area to estimate the formation process of the fuel debris.
- ⇒ More precise estimation of the condition inside the core will become the basis for review of full-scale fuel debris retrieval to safely retrieve fuel debris and realize thoroughly managed stable storage.

<Example of incorporating "estimation of the condition inside the core" into "review of fuel debris retrieval methods">

- ➢ Estimate hardness of fuel debris → select retrieval methods and tools
- ➢ Possibility of criticality of fuel debris → review safety measures and storage methods



Grasping the condition of the sampled area

Acquisition of information tailored to decommissioning needs

 ✓ Grasp the type and concentration of major components (nuclide/element) in the sample and review the <u>origin of each component</u>

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✓ Grasp the <u>content and distribution of fuel components</u> in the sample

Estimation of formation process of fuel debris

- Estimation of fuel debris properties through review of in-core environment during the accident
 - Estimate the <u>formation conditions of the sample</u> based on microstructure, composition of constituent phases and crystal structure of phases including U in the sample.
 - ✓ Evaluate the <u>surrounding of the sampled area</u> based on the comparison of existing accident scenarios with the internal investigation results (evaluate based on the results of multiple future sample analyses)
- [1] JAEA, FY2022 Report of the FY2022 Subsidized Project of Decommissioning, Contaminated Water and Treated Water Management (Development of Analysis and Estimation Technology for Grasping Fuel Debris Properties (Development of Technology for Estimating Damage Conditions of the Reactor Pressure Vessel)).



Reference Analysis items and evaluation details of the fuel debris sample

1. Grasping the condition of the sampled area

Blue: Additional analysis

Analysis items	Analysis methods	Evaluation details	Examples of major applications for decommissioning
Basic information •External appearance, weight •Dose rate •Density distribution	 Exterior, weight, dose rate measurement Imaging plate (IP) X-ray CT 	Organization of basic information	Basic information to review retrieval (existence and mount of pores, etc.)
Element content (elemental composition)	• ICP-MS, ICP-AES	Content of fuel components Origin of major components	Basic information to review safety measures at retrieval, such as criticality evaluation, and storage methods
lsotope ratio	•TIMS •SIMS	U isotope ratio	
Element and compound distribution	•SEM-EDX、SEM-WDX •TEM-EDX •XRD	Evaluation of distribution of elements and compounds (including pores)	Basic information to review retrieval methods and tools (estimation of hardness, toughness, etc.)
Radioactive concentration •γ-ray spectrometry •α-ray spectrometry •β-ray spectrometry •Liquid scintillation counter, etc.		Accompanied condition of U with focal nuclides Amount of radioactivity of nuclides targeted for analysis	Information to review technology development for non-destructive measurement at fuel debris retrieval Information needed to deliberate treatment/disposal

2. Estimation of formation process of fuel debris

Analysis items	Analysis methods	Evaluation details	Examples of major applications for decommissioning
Crystal structure and composition of phases including U	 SEM-EDX, SEM-WDX TEM-EDX Raman spectroscopy XRD µ-XAFS µ-XRF µ-XRD 	Estimation of temperature and atmosphere when U particles, etc. are formed Oxidation state of U, etc.	Precise estimated drawing of the condition inside the core to review retrieval methods and internal investigation

See the list of abbreviations at the end of the document for abbreviations of analysis methods

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Reference Overview and purpose of each item in debris analysis



The following 3 types of analysis are used to analyze the fuel debris sample and identify its characteristics and how it was formed.

• Non-destructive analysis

- [Overview] Roughly grasp information, such as distribution of pores and high-density materials and contained components, without changing the state of the received sample as much as possible.
- [Purpose] Obtain basic information of the sample, and confirm the presence or absence of components derived from nuclear fuel (uranium, radioactive nuclides, etc.) early on. Additionally, review how to specifically proceed with the analysis, such as which area to focus on in the solid analysis and liquid analysis to be conducted later on and whether the location of the sample after fluctuation can be obtained.

[Analysis methods] External appearance, weight, dose rate, IP, X-ray CT, γ-ray spectrometry, SEM-WDX (surface)

• Solid analysis

- [Overview] Confirm what kind of state uranium, zirconium and other components from the reactor are in (what the coexisting elements are, whether it retains its pre-accident state, whether it is oxidized, etc.), by fractionating parts of the sample and observing its cross section in detail.
- [Purpose] Obtain information on "how the sample was formed", such as which materials reacted under what temperature or atmosphere* to form the sample.

*The synchrotron analysis of SPring-8, which was newly added after the previous report, is considered to enable more accurate estimation of the temperature and atmosphere at the time of the accident, since more detailed data than the conventional observation method based on electron microscopes can be obtained such as three-dimensional distribution of elements in the sample and valence of uranium.

[Analysis methods] SEM-EDX, SEM-WDX, TEM-EDX, SIMS, Raman spectroscopy, µ-XAFS, µ-XRF, µ-XRD

Liquid analysis

- [Overview] Fractionate part of the sample and dissolve it in acid to measure the elements and nuclide content in the resulting dissolving solution.
- [Purpose] Obtain necessary information to review the process to safely retrieve and stably store fuel debris, such as uranium isotope ratio and radioactive nuclide concentration.

[Analysis methods] ICP-MS, ICP-AES, TIMS, γ -ray spectrometry, α -ray spectrometry

Continuing the series of analyses will gradually identify the characteristics of fuel debris deposited in the core and contribute to safety evaluation and rationalization for fuel debris retrieval and storage.



Reference Abbreviation and overview of analysis methods **TEPCO**¹¹

Analysis method abbreviation	Analysis method name	Analysis method overview
ICP-AES	Inductively coupled plasma atomic emission spectroscopy	Qualitative and quantitative analysis method of elements by introducing atomized samples into high- temperature plasma and obtaining element-specific spectra by spectroscopy of the issued light.
ICP-MS	Inductively coupled plasma mass spectrometry	Method of measuring the concentration of elements and its isotopes by introducing atomized samples into high-temperature plasma, ionizing elements in the sample and measuring the number of ions in ion mass-to-charge ratio (m/z) by mass spectrometry.
TIMS	Thermal ionization mass spectrometry	Method of measuring the concentration of elements and its isotopes by applying samples onto metal filament, ionizing the atoms by heating under vacuum and measuring the number of ions in ion mass-to-charge ratio (m/z) by mass spectrometry.
SEM	Scanning electron microscope	Device that can observe the sample surface by irradiating the surface with electron beams, and can also analyze elements by attaching an X-ray analyzer.
EDX	Energy dispersive X-ray spectroscopy	Method of elemental analysis and compositional analysis by detecting characteristic X-rays generated by electron irradiation and categorizing them by the energy of characteristic X-rays.
WDX	Wavelength dispersive X- ray spectroscopy	Method of elemental analysis and compositional analysis by detecting characteristic X-rays generated by electron irradiation and performing spectroscopy at the wavelength of characteristic X-rays.
TEM	Transmission electron microscope	Method of imaging electrons transmitted through the sample and scattered electrons for observation under high magnification by irradiating thinned samples with electron beams, and also conducting elemental analysis by attaching an X-ray analyzer. Crystal structure can also be obtained from the diffraction image.
SIMS	Secondary ion mass spectrometry	Method of measuring the concentration of elements and its isotopes by measuring the secondary ions generated by irradiating the sample surface with a beam of ions with a mass spectrometer and measuring the number of ions in ion mass-to-charge ratio (m/z) by mass spectrometry.
Raman spectroscopy	Micro Raman spectroscopy	Method of obtaining properties such as molecular structure, temperature, stress, electrical properties, orientation and crystallinity by irradiating the sample surface with light and dispersing Raman scattering light. Information on chemical form of micro-regions on μ m order can be obtained by combining Raman spectroscopy with conventional optical microscopes.
X-ray CT	X-ray computed tomography	Method of obtaining density distribution of the sample interior by irradiating the sample with X-rays, capturing the transmitted X-ray intensity by a computer and scanning it three-dimensionally. Distribution of phases of different density can be obtained.

Reference Abbreviation and overview of analysis methods **TEPCO**¹²

Analysis method abbreviation	Analysis method name	Analysis method overview
XAFS	X-ray absorption fine structure spectroscopy	Method of analyzing the internal structure of materials at the molecular and atomic level by irradiating the sample with X-rays and precisely observing the absorbed X-ray energy
XRF	X-ray fluorescence spectroscopy	Method of qualitative analysis of content of constituent elements by measuring the wavelength and energy of X-rays (X-ray fluorescence) generated according to the substance by irradiating the sample with X-rays
XRD	X-ray diffraction analysis	Method of analyzing the crystal structure, crystal orientation, crystal lattice size, etc. of the object by irradiating the sample with X-rays and measuring the resulting X-rays (diffracted X-ray)
IP	Imaging plate	Radiation image measuring instrument that detects radiation energy as stimulable luminescence. Dose distribution of the sample can be obtained.

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Reference Results from the external observation of the first fuel debris sample



Enlarged photo of External appearance of fuel debris sample (photographed from approx. 45 degrees diagonally)

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TEPC



Reference External appearance observations, mass, and dose rate measurements for the first fuel debris sample





<External appearance>

- The received fuel debris sample was heterogeneous.
- The sample was reddish brown overall with black and glossy spots on the surface.
- The size of the sample was approx. 9mm × approx. 7mm.

<Mass> 0.693g

< Dose rate > Approx. 8mSv/h

(γ -rays: An ionization chamber was used to measure the sample while it was still inside a polypropylene container (at a distance of 1~2 cm from the sample))

* High dose rates prevented IP imagery (dose distribution)



Reference γ-ray spectrometry measurement result (Comparison of first and second samples)

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