IAEA Review of Safety Related Aspects of Handling ALPS Treated Water at TEPCO's Fukushima Daiichi Nuclear Power Station

Second Interlaboratory Comparison on the Determination of Radionuclides in the Marine Environment

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International Atomic Energy Agency

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EXECUTIVE SUMMARY

In 2021, the IAEA started its review of safety related aspects of handling ALPS (Advanced Liquid Processing System) treated water at TEPCO's Fukushima Daiichi Nuclear Power Station (FDNPS). Consistent with the request from the Government of Japan, the IAEA statutory functions and the mandate of the Task Force, the scope of the IAEA review is tailored to assessing safety related aspects of the implementation of Japan's *Basic Policy on Handling of ALPS Treated Water at the Tokyo Electric Power Company's Holdings' Fukushima Daiichi Nuclear Power Station* against the IAEA's Safety Standards. The approach outlined in the Basic Policy is to conduct a series of controlled discharges of ALPS treated water into the sea ('batch discharges') over a period of decades.

Consistent with the relevant IAEA's Safety Standards, TEPCO is required to determine the characteristics and activity of the ALPS treated water (e.g., through the radiological environmental impact assessment) to be discharged into the sea, and to establish and implement monitoring programmes to ensure that public exposure due to the discharges is adequately assessed and that the assessment is sufficient to verify and demonstrate compliance with the authorization granted by the Nuclear Regulation Authority (NRA).

To conduct its safety review, the IAEA has organized the work of the Task Force into three main components, the assessment of protection and safety; regulatory activities and processes; and sampling, independent analysis and data corroboration. The latter activities include three elements:

- Sampling, analysis and interlaboratory comparison for ALPS treated water from the FDNPS.
- Sampling, analysis and interlaboratory comparison for environmental samples (e.g., seawater, fish) from the surrounding environment of FDNPS.
- Assessment of the capabilities of dosimetry service providers involved in the monitoring of internal and external radiation exposure of workers at FDNPS.

The IAEA's sampling, independent analysis and data corroboration activities also include a review of sampling and analytical methods used by TEPCO and any other relevant technical institutions.

The corroboration of source and environmental monitoring conducted by TEPCO and relevant Japanese authorities is based on interlaboratory comparisons (ILCs). ILCs, along with proficiency tests (PTs), are standard methods for laboratories to assess the quality of their measurement results in comparison with those of other participating laboratories, and to identify any potential improvements. PTs involve the evaluation of performance against pre-established criteria whereas ILCs involve the organization, performance, and evaluation of measurements on the same or similar items by two or more laboratories in accordance with predetermined conditions.

For this second ILC to corroborate the results of environmental monitoring under the IAEA's ALPS safety review, samples of seawater, sediment, fish and seaweed were taken in October 2023 from offshore locations and a fish market close to FDNPS.

Extensive monitoring of the marine environment around the FDNPS is conducted according to the Comprehensive Radiation Monitoring Plan (CRMP). TEPCO and other relevant Japanese authorities including the NRA, the Ministry of the Environment (MOE), and the Japan Fisheries Agency (FAJ), have responsibilities under the CRMP. Enhancements to the CRMP to specifically address the discharge of ALPS treated water were introduced in March 2022. As this ILC is based on samples collected in October 2023; it constitutes the first corroboration by the IAEA of the results of this monitoring since the discharges started in August 2023.

This publication reports the results of the second ILC. It describes the joint sampling mission undertaken to collect seawater, sediment, fish and seaweed samples; the analytical techniques used by participating laboratories – from Japan (participating on behalf of the Japanese authorities); the IAEA and third -party member laboratories of the IAEA ALMERA network (Analytical Laboratories for the Measurement of Environmental Radioactivity)[1]; the measurement results and the statistical evaluation of the results.

In total, ten laboratories undertook analyses and reported results for the ILC. Analyses were undertaken by Japanese laboratories participating in marine monitoring relevant to the ALPS treated water discharges within the CRMP and by the following two IAEA Nuclear Sciences and Applications Laboratories:

- IAEA Marine Environment Laboratories, Radiometrics Laboratory (RML), Monaco;
- Isotope Hydrology Laboratory (IHL), Vienna, Austria.

Additionally, under the coordination of the participating IAEA laboratories, selected third-party laboratories – a consortium of Canadian laboratories, including Canadian Nuclear Laboratories (CNL), Canadian Nuclear Safety Commission (CNSC), and the University of Ottawa (OttawaU), led by the Radiation Protection Bureau (RPB) of Health Canada; Third Institute of Oceanography, Ministry of Natural Resources, China; and the Korea Institute of Nuclear Safety (KINS), Republic of Korea – all members of the network of Analytical Laboratories for the Measurement of Environmental Radioactivity (ALMERA) with demonstrable competence in the methods required, also conducted analyses of samples as ILC participants.

The results of the analyses undertaken at each laboratory were reported to the IAEA. For results that could be intercompared (i.e. for radionuclides for which activity concentrations above detection limits were reported by at least two laboratories) a statistical evaluation to assess agreement was carried out by the IAEA. The results are presented in tables and charts in this report.

Although some discrepancies were found, the low number was impressive since the analytical methods are often complex (e.g. for OBT, TFWT and ^{14}C in fish) and relatively new to some participating laboratories, being implemented specifically to assess the ALPS treated water discharges. It can be noted that higher uncertainties are usually associated with complex analyses of low, and close to detection limit, levels. There were no order of magnitude variations, and no systemic deviations identified between the results reported by Japanese laboratories and those reported by the IAEA and the ALMERA member laboratory. Therefore, despite the discrepancies, the key findings of this ILC are:

- Sample collection procedures follow the appropriate methodological standards required to obtain representative samples.
- Japanese laboratories have reported accurate results that demonstrate a high degree of proficiency.

The IAEA notes that these findings provide confidence in Japan's capability for conducting reliable and high-quality monitoring related to the discharge of ALPS treated water.

Additional ILCs will be conducted in the future for ALPS treated water and environmental samples, as well as occupational radiation protection. Future ILCs will continue to allow for an assessment of Japan's capability to evaluate any changes in the levels of relevant radionuclides in the marine environment, relative to the baseline.

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[1] More information on the ALMERA network is available from the following website: https://nucleus.iaea.org/sites/ReferenceMaterials/Pages/ALMERA.aspx

Furthermore, the corroboration of environmental monitoring complements a separate project – NA3/38 Marine Monitoring: Confidence Building and Data Quality Assurance – addressing the quality of data from marine monitoring undertaken in Japan following the accident at FDNPS. Through project NA3/38, which has been implemented since 2014, the IAEA is assisting the Government of Japan in ensuring that sea area monitoring carried out under the regularly updated CRMP is comprehensive, credible and transparent and is helping to build confidence of the stakeholders in the accuracy and quality of the marine monitoring data. Within project NA3/38, the IAEA has organized a series of ILCs and PTs to test the sampling and analytical performance of the Japanese laboratories for the analysis of radionuclides in seawater, sediment and fish samples. Data from this project are available online[2].

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[2] https://www.iaea.org/about/organizational-structure/department-of-nuclear-sciences-and-applications/division-of-iaea-marine-environment-laboratories/marine-monitoring-confidence-building-and-data-quality-assurance

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1. INTRODUCTION

1.1. BACKGROUND

The corroboration of a representative subset of the radioactivity measurement results reported by TEPCO and relevant Japanese authorities during both the pre-operational and the operational phases of discharge of ALPS treated water to the sea, and a review of the methods for related sampling and analysis used by TEPCO and relevant Japanese authorities is being undertaken by the IAEA. This corroboration provides an independent check of the veracity of the radiological data resulting from source and environmental monitoring programmes related to the ALPS discharges upon which the safety related aspects of the discharge of ALPS treated water are being evaluated. A primary objective is to promote transparency and provide sound information to enable interested parties to evaluate the radiological data used by Japan as the basis for planning and implementing the discharge of ALPS treated water into the sea.

The corroboration of source and environmental monitoring is based on interlaboratory comparisons (ILCs). ILCs, along with proficiency tests (PTs), are standard methods for laboratories to assess the quality of their measurement results in comparison with those of other participating laboratories, and to identify any potential improvements. PTs involve the evaluation of performance against pre-established criteria whereas ILCs involve the organization, performance and evaluation of measurements on the same or similar items by two or more laboratories in accordance with predetermined conditions [1].

This publication reports the results of the second ILC to corroborate environmental monitoring. It describes the joint sampling mission undertaken to collect seawater, sediment, fish and seaweed samples; the analytical techniques used by participating laboratories – from Japan (participating on behalf of the Japanese authorities); the IAEA and third-party member laboratories of the IAEA ALMERA network (Analytical Laboratories for the Measurement of Environmental Radioactivity)[3]; the measurement results and the statistical evaluation of the results.

Extensive monitoring of the marine environment around the FDNPS is conducted according to the Comprehensive Radiation Monitoring Plan (CRMP) [2]. TEPCO and other relevant Japanese authorities have responsibilities under the CRMP. This plan defines sampling locations, frequency of sampling, target detection limits and responsibilities of the organizations involved. The monitoring comprises sampling and analysis of seawater to different depths, sediment, fish and seaweed. The aim of this plan includes ensuring a comprehensive overview of the radiological situation in the marine environment and providing an adequate basis for assessments of radiation exposures from marine pathways. In practice, sampling and analysis are often carried out by contracted laboratories but TEPCO and the other relevant Japanese authorities, as defined in the CMRP, have responsibility for reporting the results of the monitoring with which they have been tasked.

Enhancements to the CRMP to specifically address the discharge of ALPS treated water were introduced in March 2022. These included the monitoring of ^{3}H in seawater at increased frequencies plus monitoring quarterly for ‘seven major radionuclides’, namely ^{60}Co, ^{90}Sr, ^{106}Ru, ^{125}Sb, ^{129}I, ^{134}Cs and ^{137}Cs. Monitoring of organically bound tritium (OBT), tissue free-water tritium (TFWT) and ^{14}C in fish and ^{129}I in seaweed is also being undertaken.

This monitoring started more than one year before the first discharges of ALPS treated water. The results provide a baseline of activity concentrations in the marine environment against which the any

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[3] More information on the ALMERA network is available from the following website: https://nucleus.iaea.org/sites/ReferenceMaterials/Pages/ALMERA.aspx

consequences of the discharges can be assessed. This ILC is based on samples collected in October 2023 and constitutes the first corroboration of the results of this monitoring by the IAEA since the discharges started in August 2023.

The samples were taken at sampling locations defined in the CRMP, with IAEA observation, using the same techniques used for routine monitoring. Similarly, the radionuclides analysed for each sample are defined in the CRMP and the analyses were carried out by the participating Japanese laboratories using the same methods as those used for routine monitoring.

The IAEA wishes to thank all the participating laboratories that took part in this interlaboratory comparison and the Japanese organisations that provided logistical support. The IAEA is also grateful to the Government of Monaco for its support.

1.2. OBJECTIVE

This publication reports the results of the second ILC to corroborate environmental monitoring related to the discharges of ALPS treated water at FDNPS.

1.3. SCOPE

This publication reports all aspects of the second ILC to corroborate environmental monitoring related to the discharge of ALPS treated water at FDNPS including: the ILC design; participating laboratories; the methods employed for sampling and for distribution of the samples between participating laboratories; the analytical methods used by each participating laboratory to determine activity concentrations of radionuclides in the samples; the methodology employed for the statistical evaluation of the results; and the results and conclusions.

1.4. STRUCTURE

This publication contains reports the participating laboratories (Section 2); the methods employed for the sample collection and pre-treatment, and for the distribution of the samples between participating laboratories (Section 3), and the analytical methods used by each participating laboratory to determine activity concentrations of radionuclides in the samples (Section 4). The methodology employed for the statistical evaluation of the results is described in Section 5. The results of the ILCs are presented in Section 6 and conclusions in Section 7. The results of the ILCs are also presented in charts in an Appendix.

2. PARTICIPATING LABORATORIES

In total, ten laboratories participated in the ILC: Six from Japan (participating on behalf of TEPCO and other relevant Japanese authorities having responsibilities under the CRMP); the IAEA; and selected third-party laboratories – a consortium of Canadian laboratories, including Canadian Nuclear Laboratories (CNL), Canadian Nuclear Safety Commission (CNSC), and the University of Ottawa (OttawaU), led by the Radiation Protection Bureau (RPB) of Health Canada; Third Institute of Oceanography (TIO), Ministry of Natural Resources, China; and the Korea Institute of Nuclear Safety (KINS), Republic of Korea – all members of the network of Analytical Laboratories for the Measurement of Environmental Radioactivity (ALMERA).

ALMERA is a network comprising more than 200 member laboratories globally. It provides a platform for maintaining and developing capability on the determination of radionuclides in air, water, soil, sediment and vegetation that can be used for both routine and environmental emergency monitoring in the IAEA Member States.

IAEA analyses were undertaken at two participating IAEA Nuclear Sciences and Applications Laboratories:

- IAEA Marine Environment Laboratories, Radiometrics Laboratory (RML), Monaco;
- Isotope Hydrology Laboratory (IHL), Vienna, Austria.

The participating laboratories are presented in Table 1, and participation of each in specific analyses in Table 2.

TABLE 1. LABORATORIES PARTICIPATING IN ILC 2023

Identifier	Participant
IAEA	IAEA Laboratories, Austria and Monaco
CAN	A consortium of Canadian laboratories, including Canadian Nuclear Laboratories (CNL), Canadian Nuclear Safety Commission (CNSC), and the University of Ottawa (OttawaU), led by the Radiation Protection Bureau (RPB) of Health Canada.
JCAC	Japan Chemical Analysis Center, Chiba, Japan
KAKEN[1]	KAKEN Co. Ltd., Ibaraki, Japan
KANSO	KANSO TECHNOS Co. Ltd., Osaka, Japan
KEEA	Kyushu Environmental Evaluation Association, Fukuoka, Japan
KINS	Korea Institute of Nuclear Safety, Daejeon, Republic of Korea
MERI	Marine Ecology Research Institute, Chiba, Japan
TIO	Third Institute of Oceanography, Ministry of Natural Resources, Xiamen, China
TPT[1]	Tokyo Power Technology Ltd., Fukushima, Japan

Note:

[1] Laboratories conducting analysis under contract from TEPCO.

TABLE 2. OVERVIEW OF ILC 2023

Sample type	Nuclide	IAEA	CAN	JCAC	KAKEN	KANSO	KEEA	KINS	MERI	TIO	TPT
Seawater	^{3}H	✓	✓	✓	✓	✓	✓	✓	✗	✓	✗
	^{60}Co	✓	✓	✓	✗	✗	✗	✓	✗	✓	✗
	^{90}Sr	✓	✓	✓	✗	✗	✗	✓	✗	✓	✗
	^{106}Ru	✓	✓	✓	✗	✗	✗	✓	✗	✓	✗
	^{125}Sb	✓	✓	✓	✗	✗	✗	✓	✗	✓	✗
	^{129}I	✓	✓	✓	✗	✗	✗	✓	✗	✓	✗
	^{134}Cs	✓	✓	✓	✗	✗	✗	✓	✗	✓	✗
	^{137}Cs	✓	✓	✓	✗	✗	✗	✓	✗	✓	✗
Sediment	^{134}Cs	✓	✓	✓	✗	✗	✗	✓	✗	✓	✓
	^{137}Cs	✓	✓	✓	✗	✗	✗	✓	✗	✓	✓
Fish (market)	^{3}H (OBT)	✓	✓	✗	✗	✓	✓	✓	✗	✗	✗
	^{3}H (TFWT)	✓	✓	✗	✗	✓	✓	✓	✓	✗	✗
	^{14}C	✓	✓	✓	✗	✗	✗	✓	✗	✗	✗
Fish (fixed net)	^{3}H (OBT)	✓	✓	✓	✓	✗	✗	✓	✗	✗	✓
	^{3}H (TFWT)	✓	✓	✓	✓	✗	✗	✓	✗	✗	✓
	^{14}C	✓	✓	✓	✗	✗	✗	✓	✗	✗	✗
Seaweed	^{129}I	✓	✓	✓	✗	✗	✗	✓	✗	✗	✓

Notes:

The symbol ✓ indicates that the laboratory participated in the specific analysis (sample type and radionuclide), the symbol ✗ indicates that it did not participate.

Fish and seaweed samples were collected for TIO but were unable to be imported into China on time and in good condition.

3. SAMPLE COLLECTION, PRETREATMENT AND DISTRIBUTION

Samples of seawater, sediment, fish and seaweed were collected in October 2023 from offshore locations and a fish market close to FDNPS. The sampling locations are shown in Figure 1. The coordinates of these sampling locations are provided in Table 3.

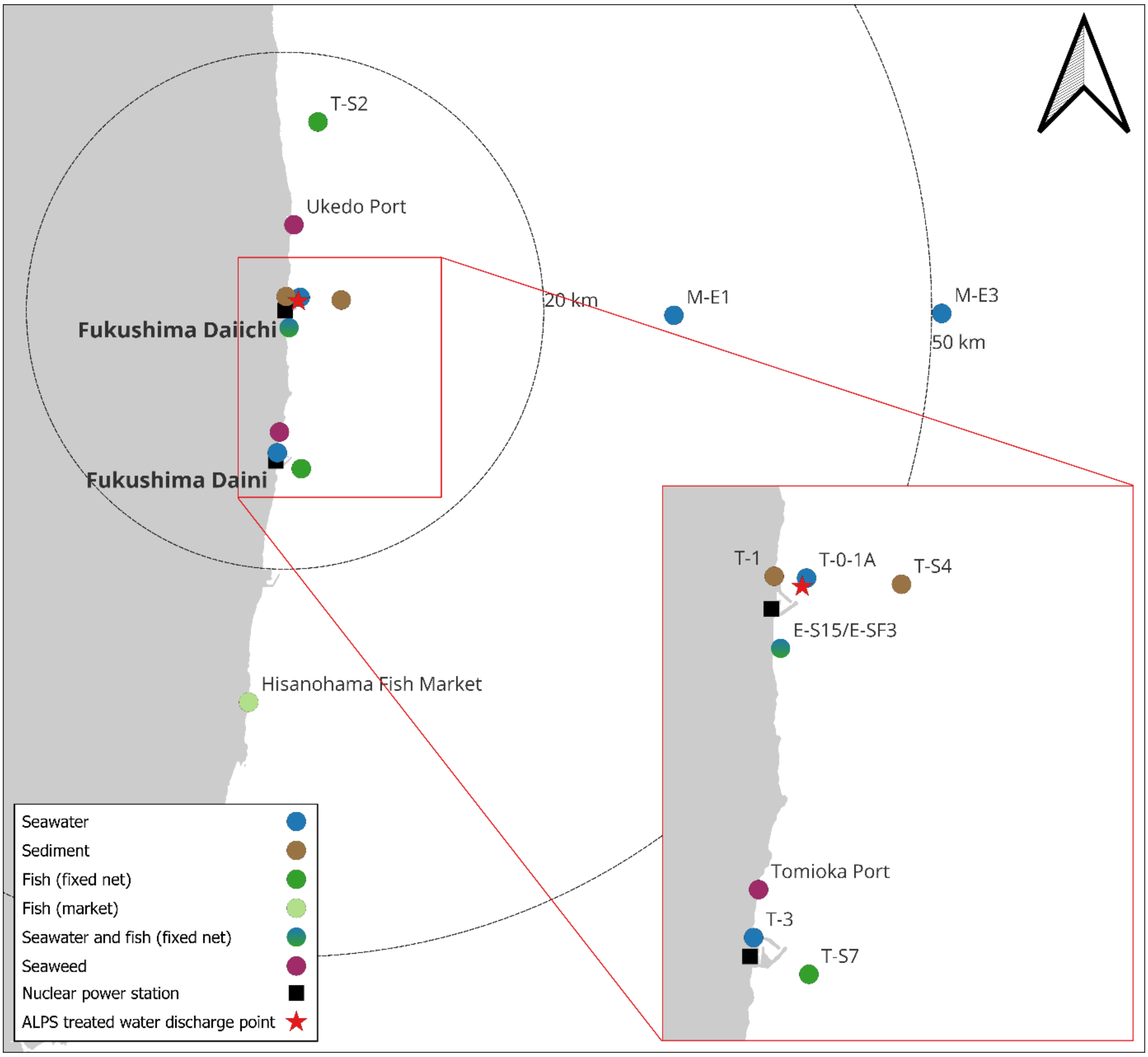

FIG. 1. Sampling locations close to TEPCO's Fukushima Daiichi Nuclear Power Station.

TABLE 3. COORDINATES OF SAMPLING LOCATIONS

Sampling location	Latitude (N)	Longitude (E)	Samples
E-S15/E-SF3[1]	37.4094	141.0368	Seawater and fish (fixed net)
T-0-1A	37.4306	141.0467	Seawater
T-3	37.3222	141.0264	Seawater
M-E1	37.4170	141.3730	Seawater
M-E3	37.4170	141.6070	Seawater
T-1	37.4311	141.0344	Sediment
T-S4	37.4286	141.0825	Sediment
T-S2	37.5528	141.0625	Fish (fixed net)
T-S7	37.3111	141.0472	Fish (fixed net)
Hisanohama Fish Market	37.1484	141.0010	Fish (market)
Ukedo Port	37.4811	141.0413	Seaweed
Tomioka Port	37.3367	141.0283	Seaweed

Note:

[1] The sampling location is the same. However, for seawater (E-S15) it is a point while for fish (E-SF3) the sampling extends along the length of the fixed net.

3.1. SEAWATER

Surface seawater samples were collected at five sampling locations (E-S15, T-0-1A, T-3, M-E1 and M-E3) offshore TEPCO's Fukushima Daiichi Nuclear Power Station (Figure 1 and Table 3). The samples were collected between 16 and 19 October 2023 from each sampling location for subsequent analysis for ^{3}H and, for E-S15, for 'seven major radionuclides', namely ^{60}Co, ^{90}Sr, ^{106}Ru, ^{125}Sb, ^{129}I, ^{134}Cs and ^{137}Cs. Separate samples from each location were provided to participating laboratories for ^{3}H and ^{129}I; for radiocaesium (^{134}Cs and ^{137}Cs); for ^{90}Sr and for other gamma-emitting radionuclides (^{60}Co, ^{106}Ru, ^{125}Sb).

For ^{3}H, six laboratories were planned to participate in the analyses of samples from each sampling location, except for E-S15 for which there were five.

For T-0-1A, T-3, M-E1 and M-E3 the sample collection and distribution methods were:

- Separate 2 L containers were filled, one at a time, from a Niskin sampler, resulting in a total of four 2 L samples from each sampling station.
- One 2 L sample was provided to each laboratory.

For E-S15 the sample collection and distribution methods for ^{3}H were:

- A 400 L plastic container with four valves was first filled with seawater. This container was also used to sample seawater from this location for ^{60}Co, ^{90}Sr, ^{106}Ru, ^{125}Sb, ^{129}I, ^{134}Cs and ^{137}Cs as described layer in this section.
- Separate 2 L containers were filled, two at a time, from the four valves, resulting in a total of five 2 L samples from this sampling location.
- One 2 L sample was provided to each laboratory.

Six laboratories were planned to participate in the analyses for ^{90}Sr; ^{134}Cs and ^{137}Cs; other gamma-emitting radionuclides (^{60}Co, ^{106}Ru, ^{125}Sb); and ^{129}I for sampling location E-S15. The collection and distribution methods were:

- Samples for analysis for ^{129}I were taken using the same method described above for ^{3}H for the E-S15 location. One 2 L sample was provided to each laboratory.
- From the same 400 L plastic container from which the samples to be analysed for ^{3}H were taken, separate 20 L cubitainers (^{90}Sr; ^{134}Cs and ^{137}Cs) and 10 L cubitainers (^{60}Co, ^{106}Ru, ^{125}Sb) and for were filled simultaneously from each of the four valves. A total of eighteen 20 L samples and six 10 L samples were taken. Two separate fills of the 400 L container were required to facilitate provision of the required sample volume to all participants.
- Each sample was acidified to pH 1–2 with concentrated HCl.
- Three 20 L samples and one 10 L sample from E-S15 were provided to each laboratory.

For all seawater samples, the fill sequence, valve numbers and recipient laboratories were recorded, as appropriate, facilitating traceability of each sample container.

3.2. SEDIMENT

Sediment samples were collected using a grab sampler on 16 October 2023 offshore from TEPCO’s FDNPS at sampling location T-S4 and by hand at the shore within the FDNPS site at sampling location T-1 (Figure 1 and Table 3). The samples were subsequently oven-dried at 105°C on large stainless-steel trays, crushed using stainless-steel spatulas, and sieved through a 2-mm mesh sieve at the JCAC laboratory in Chiba. No grinding was required prior to sieving due to the sandy nature of the sediments. The fraction with grain size <2 mm was sieved to ≤250 µm, then placed in a plastic bag and mixed thoroughly to ensure homogeneity. An incremental division method was used for the provision of samples to participating laboratories. Each sample was divided into two aliquots using a splitter; one aliquot was archived and the second one was further divided until the required sample weight for each laboratory was attained. The sequence of division of each sample depended on the total mass of the sieved material. The samples were then bottled in 500 mL plastic bottles and shipped to the IAEA Marine Environment Laboratories in Monaco where their ^{137}Cs homogeneity was checked using gamma-ray spectrometry with High Purity Germanium (HPGe) detectors. Approximately 250 g of homogeneous dried sediment from each station was then shipped to each participating laboratory for analysis for ^{134}Cs and ^{137}Cs.

3.3. FISH (MARKET)

Six batches of frozen fish, one each of olive flounder (*Paralichthys olivaceus*), redwing searobin (*Lepidotrigla microptera*), pufferfish (*Takifugu snyderi*), silver croaker (*Pennahia argentata*), crimson sea bream (*Evynnis tumifrons*) and Japanese jack mackerel (*Trachurus japonicus*), were collected from the fish market at Hisanohama Port on 19 October 2023.The fish species were caught by pole and line fishing or bottom trawling on the same date in the vicinity of TEPCO's Fukushima Daiichi Nuclear Power Station at depths between 30 and 143 m. Each batch of fish was divided into two sub-batches: one for provision of samples to be analysed for ^{3}H – organically bound tritium (OBT) and tissue free water tritium (TFWT) – and one for ^{14}C. Special care was taken to ensure that the fish to be analysed for tritium were isolated from ice used for cooling during shipment and, subsequently, from tap water during pre-treatment.

Each sub-batch of fish of each species was prepared separately by fileting, homogenising the muscle tissue and then dividing into separate samples at the MERI laboratory in Chiba on 20 October 2023. These were frozen and shipped to the participating laboratories. Each laboratory received approximately 1 kg of each species of fish for analysis for ^{3}H and 0.5 kg for analysis for ^{14}C.

3.4. FISH (FIXED NET)

Fish samples were collected at three sampling locations (E-SF3, T-S2 and T-S7) offshore TEPCO's Fukushima Daiichi Nuclear Power Station (Figure 1 and Table 3). The fish were caught using fixed gill nets at each location. Nets were installed on 17 October 2023 and hauled in the following morning. One batch of olive flounder (*Paralichthys olivaceus*) was collected at each location. As for the fish collected from market, each batch was divided into two sub-batches: one for provision of samples to be analysed for ^{3}H (OBT and TFWT) and one for ^{14}C.

Each sub-batch of fish of each species was prepared separately by fileting, homogenising the muscle tissue and then dividing into separate samples. The samples from T-S2 and T-S7 were processed at TEPCO's laboratory in Fukushima on 17 October 2023 and the samples from E-SF3 at the JCAC laboratory in Chiba on 20 October 2023. These were frozen and shipped to the participating laboratories. Each laboratory received approximately 1 kg of each species of fish for analysis for ^{3}H and 0.5 kg for analysis for ^{14}C.

3.5. SEAWEED

Seaweed samples were collected at Ukedo Port and Tomioka Port offshore TEPCO's Fukushima Daiichi Nuclear Power Station (Figure 1 and Table 3). At Ukedo Port (sampling location of the Ministry of the Environment), one sample of suringar (*Besa paradoxa*) was collected by a diver. The seaweed was prepared by first removing the leaves and discarding the stems, homogenising and then dividing into separate samples at the JCAC laboratory in Chiba on 20 October 2023. These were frozen and shipped to the participating laboratories. Each laboratory received approximately 1 kg for analysis for ^{129}I.

At Tomioka Port (sampling location of TEPCO) one sample each of Besa suringar (*Besa paradoxa*) and arame (*Eisenia bicyclis*) were collected, also by a diver. The seaweed was prepared as above at the TEPCO laboratory in Fukushima on 17 October 2023. Each laboratory received approximately 1 kg of each species of seaweed, frozen, for analysis for ^{129}I.

4. ANALYTICAL METHODS

4.1. SEAWATER

Radionuclides in seawater were analysed by eight laboratories participating in this ILC: from Japan, JCAC, KAKEN, KANSO and KEEA; IAEA; and CAN, KINS and TIO (see Tables 1 and 2).

4.1.1. ^{3}H analysis

The IAEA used two methods of analysis for ^{3}H in seawater, a low-level method employing mass spectrometry whereby the activity concentration of ^{3}H in a sample is determined through the ingrowth of a decay product ^{3}He under controlled conditions, and liquid scintillation counting (LSC) following purification and electrolytic enrichment of the seawater samples. The ^{3}He ingrowth method was carried out at the IHL. The second method was performed at both IHL and MEL. The results reported were determined using the first method as the detection limit achievable was lower, typically of the order of 10 mBq/L.

For the ^{3}He ingrowth method, a 100 mL aliquot of each seawater sample was added to an all-metal water sample container and de-gassed for one to two hours with a dedicated degassing unit to remove all pre-existing ^{3}He. The degassed samples were stored for a period of five to six weeks to allow sufficient ^{3}He-ingrowth and then counted on a Thermo Fisher Helix SFT split flight tube noble gas mass spectrometer. To ensure accurate measurements, the sensitivity of the mass spectrometer, as well as its temporal drift and linearity, were calibrated by analysing gas samples with known amounts of ^{3}He and water samples with known ^{3}H content [3]. The background of the mass spectrometer system was also determined.

In the second method, the samples were measured by liquid scintillation counting after purification by distillation and electrolytic enrichment. An ultra-low level liquid scintillation counter was used for the counting of an aliquot of the enriched and distilled sample mixed with a scintillation cocktail.

All other participating laboratories analysed the seawater samples in a similar manner: purification by distillation followed by electrolytic enrichment, mixing with scintillation cocktail and measurement by LSC. Enrichment factors were typically 30 to 50 times the initial ^{3}H activity concentration in the seawater. For the Japanese laboratories – JCAC, KAKEN, KANSO and KEEA – the specifics of the methods implemented complied with the “Tritium Analysis Method (Radiation Measurement Method Series 9)” published by the Japanese Ministry of Education, Culture, Sports, Science and Technology [4].

4.1.2. ^{90}Sr analysis

The IAEA and TIO both used liquid-liquid extraction with di-(2-ethylhexyl)phosphoric acid (HDEHP) for the separation of yttrium from the seawater sample, while caesium was precipitated from the same sample by using ammonium molybdophosphate (AMP). The ^{90}Sr activity concentration was determined through the measurement of ^{90}Y (yttrium oxalate source) β activity using a proportional counter

At CAN a stable yttrium carrier (1 mg) was added to 40 kg of seawater. An iron hydroxide/lantthanum hydroxide precipitation, followed by a second lanthanum fluoride precipitation were performed to concentrate yttrium and remove matrix ions. The fluoride precipitate was dissolved and the yttrium fraction was isolated using Eichrom DGA resin. The yttrium fraction was counted for 120 min by LSC by Cerenkov counting. Yttrium carrier recovery was determined by ICP-MS.

JCAC employed a cation-exchange resin column for pre-concentration of strontium from the 40 L seawater sample, followed by precipitation of carbonates and an additional cation exchange resin column for separation of calcium. ^{90}Y was removed by scavenging and, once the sample reached secular equilibrium, ^{90}Y was co-precipitated with iron hydroxide and then was measured using a low background β counter.

At KINS strontium pre-concentration of the 40 L seawater sample was carried out using a cation exchange resin. Eluted strontium was then recovered using strontium carbonate precipitation and then strontium was purified again using fuming nitric acid. ^{90}Y and ^{90}Sr were determined by liquid scintillation counting in Cerenkov mode after allowing two weeks for the sample to reach secular equilibrium. The chemical yield was determined by ICP-OES (Inductively Coupled Plasma Optical Emission Spectroscopy).

4.1.3. ^{134}Cs and ^{137}Cs analysis

At CAN stable caesium (10 mg) was added to the seawater sample as a yield tracer. The seawater was then passed through a 5 g KNiFC-PAN (potassium nickel hexacyanoferrate (II)-polyacrylonitrile) resin. After sample elution, the KNiFC-PAN resin was transferred to a 20 mL glass LSC vial and dried overnight (1 g dry weight) in preparation for analysis by gamma-ray spectrometry using a Canberra Broad Energy HPGe detector. The retention of stable Cs on the KNiFC-PAN resin was determined by ICP-MS. A blank resin sample was prepared using deionized water. An efficiency standard was prepared by spiking resin with ^{134}Cs and ^{137}Cs.

At all other participating laboratories (IAEA, JCAC, KINS and TIO) ammonium molybdophosphate (AMP) was used for the chemical separation of caesium from the seawater sample, followed by gamma-ray spectrometry using a HPGe (High Purity Germanium) detector. The retention of stable Cs in the AMPS was determined by ICP-MS.

4.1.4. Other gamma-emitting radionuclides (^{60}Co, ^{106}Ru and ^{125}Sb) analysis

At TIO, ^{60}Co, ^{106}Ru and ^{125}Sb was precipitated using $Co(OH)_2$, NiS and MnO_2, respectively, followed by counting by gamma-ray spectrometry.

In all other participating laboratories, the sample was prepared in large containers (1 L Marinelli beakers for the IAEA and KINS; 2 L Marinelli beakers for JCAC and 129 ml Parkway jar for CAN) and analysed without further treatment by gamma-ray spectrometry using HPGe detectors.

^{106}Ru was determined by gamma-ray spectrometry via decay of its progeny ^{106}Rh.

4.1.5. ^{129}I analysis

The IAEA analysed for ^{129}I in the seawater sample using gamma-ray spectrometry. The method is as described in the previous section for ^{60}Co, ^{106}Ru and ^{125}Sb.

At JCAC the sea water sampled was filtered, aliquoted and diluted for measurement of ^{129}I by ICP-MS (Inductively Coupled Plasma Mass Spectrometry). Rhenium was employed as an internal standard.

At KINS, after oxidation and reduction processes, the seawater sample was chemically separated using anion exchange resin. The elute was precipitated as PdI_2 using $PdCl_2$ solution, and the PdI_2 precipitate was filtered. Dried PdI_2 mounted on a Teflon planchette was measured by X-ray spectrometry for the determination of ^{129}I.

The CAN sample was analysed by accelerator mass spectrometry (AMS) at the André E. Lalonde AMS Laboratory at the University of Ottawa, using a 3 MV AMS system. Each sample underwent redox separation cycles, after which iodine was precipitated as silver iodide (AgI). Once the AgI precipitate was obtained for each sample, it was mixed with niobium powder and loaded into AMS copper cathodes for measurement. Calibration was carried out using ISO-6II standards and NIST reference materials.

The AMS system ensured rigorous quality control by incorporating process blanks and in-house standards, achieving nominal system blanks ($\leq 5 \times 10^{-14}$ at·at^{-1}).

SEDIMENT

Radionuclides of interest in sediment samples were analysed by six laboratories participating in this ILC: from Japan, JCAC and TPT; IAEA; and CAN, KINS and TIO (see Tables 1 and 2).

4.1.6. ^{134}Cs and ^{137}Cs analysis

All participating laboratories analysed for ^{134}Cs and ^{137}Cs in sediment by gamma-ray spectrometry using HPGe detectors, following preparation of the samples in cylindrical containers.

4.2. FISH

Radionuclides of interest in fish samples collected from the fish market were analysed by seven laboratories participating in this ILC: from Japan, JCAC, KANSO, KEEA and MERI; IAEA; and CAN and KINS (see Tables 1 and 2).

For the fish sampled at sea using fixed nets, radionuclides of interest were also analysed by six laboratories: from Japan, JCAC, KAKEN and TPT; IAEA; and CAN and KINS.

4.2.1. ^{3}H (OBT and TFWT) analysis

The samples were vacuum freeze-dried at all laboratories. The tissue free water and dry materials were recovered, weighed and the drying rate (%) calculated.

At IAEA, the tissue free water was analysed for ^{3}H using the ^{3}He ingrowth method as described in section 4.1.1 for seawater.

At CAN, KINS, KANSO, KEEA and MERI the tissue free water recovered from the samples by vacuum freeze-drying was purified by reflux decomposition and distillation, mixed with scintillation cocktail, and counted by LSC to determine the ^{3}H activity concentration.

JCAC and KAKEN implemented the same method but with additional steps: following distillation the ^{3}H was concentrated by electrolytic enrichment and the samples were distilled again prior to counting by LSC. This method complied with the “Analysis Method of Tritium (Radiation Measurement Method Series 9)” [4].

For OBT, the dried fish material was first ground using a food processor at IAEA. It was then analysed for OBT using two separate analytical methods: the ^{3}He ingrowth method and high-pressure combustion followed by LSC.

For each sample analysed using the ^{3}He ingrowth method, approximately 90g of dry material was placed in an all-metal sample container (diameter 13 cm x 21 cm) and degassed using a Turbo Molecular Pump. It was then stored for ^{3}He ingrowth for a period of 7 to 9 weeks. At the end of this time, the sample container was connected to the ultra-high vacuum helium purification line attached to a Thermo Fisher Helix SFT split flight tube noble gas mass spectrometer to measure the ^{3}He produced from the OBT over the ingrowth period.

For the second method, approximately 10 g of dry material was combusted in a Parr high pressure oxygen combustion vessel and a cold trap in the vacuum line was used to recover the combustion water. The process was repeated three times to collect approximately 15 mL of combustion water in total. The combustion water was distilled, mixed with a scintillation cocktail and counted using by LSC.

CAN and KINS used the same high pressure combustion method as IAEA for OBT.

At JCAC, KANSO, KAKEN, KEEA and TPT combustion water was obtained from the dry material using a quartz tube furnace. Each dried sample was inserted into a quartz tube and combusted at 650°C to 750°C in an electric furnace. The combustion water was collected in a cold trap. Copper oxide was used as a combustion catalyst. Organic matter present in the combustion water was decomposed with refluxing. The sample was then distilled, mixed with scintillator cocktail and counted by LSC to determine the ^{3}H activity concentration.

At all laboratories, conversion from Bq/L to Bq/kg fresh weight for the OBT measurements was based on the dry content calculated in the vacuum freezing process and the hydrogen content obtained by elemental analysis of dried sample.

4.2.2. ^{14}C analysis

The CAN samples were analysed by AMS at the André E. Lalonde AMS Laboratory at the University of Ottawa. Each freeze-dried fish sample was combusted using an elemental analyzer and purified CO_2 was collected in a flame-sealed glass ampoule. The CO_2 was reduced to graphite on a semi-automated graphitization system using a purified Fe catalyst at 550 °C in the presence of H_2 for 3 hours including water removal by cold finger. A graphite sample of approximately 1 mg was pressed into a target and analysed for ^{14}C by AMS. The activity concentration (Bq/KgC) was calculated from the fraction-modern

($F^{14}C$) determination based on the specific activity of the primary standard.At KINS, each freeze-dried sample was combusted to CO_2 which was then collected by bubbling into ammonium water (1:1). Calcium chloride was added to the solution and the precipitate $CaCO_3$ (calcium carbonate) retrieved. $CaCO_3$ (6 g) was reacted with hydrochloric acid water (1:1) on nitrogen purging to generate CO_2. The CO_2 was absorbed using Carbo-Sorb E, mixed with a scintillation cocktail (PermaFluor E+) in a Teflon vial and CO_2 counted by LSC.

The IAEA's samples were analysed using a similar technique by the Low-Level Radioactivity Measurements (LRM) Laboratory of the Belgian Nuclear Research Centre (SCK CEN), under contract. A distinction was that the scintillation cocktail was Pico-Fluor Plus. This method is accredited to ISO/IEC 17025 at SCK-CEN (accreditation number 015-TEST under the Belgian National Accreditation Body, BELAC).

JCAC used different methods for the fish sampled from market and for fish caught by fixed net. For the former, fish muscle was freeze-dried, ground to a powder and homogenised. Then, 5-10 g was thermally decomposed with pure oxygen in a combustion apparatus at 1000°C. CO_2 was collected by bubbling into ammonium water (1:1). Calcium chloride was added to the solution and the precipitate $CaCO_3$ (calcium carbonate) retrieved. $CaCO_3$ (25-27 mg) was reacted with pure phosphoric acid (4 mL) on a vacuum line to generate CO_2. Subsequently, a graphite target (>2 mg) was retrieved by flowing pure hydrogen into the CO_2 and loaded with a pre-purified Fe catalyst at 600°C for 6 hours. After removal of the Fe, pure graphite was measured by AMS for calculation of the ^{14}C specific activity (Bq/g carbon) in the sample.

For fish caught by fixed net, at JCAC a dry matter sample was burned under high oxygen pressure in a combustion device. The generated carbon dioxide was synthesized into benzene using a vacuum line. Then, 2 mL of the synthesized benzene was mixed with 0.5 mL of scintillation cocktail and counted by LSC.

4.3. SEAWEED

Radionuclides of interest in seaweed samples were analysed by four laboratories participating in this ILC: from Japan, JCAC and TPT; IAEA; and KINS (see Tables 1 and 2).

4.3.1. ^{129}I analysis

IAEA and KINS analysed the freeze-dried seaweed samples by gamma spectrometry using HPGe detectors.

The IAEA's samples were analysed using AMS under contract by the Centro Nacional de Aceleradores (CNA) in Seville, Spain, an IAEA Coordinating Centre. The method is based on an acid microwave leaching. The samples were leached together with a stable I carrier and HNO_3. Then, the remaining organic matter was decomposed using NaClO , the iodine was purified through a solvent extraction step and, finally, iodine was precipitated as AgI and pressed into a Cu cathode for AMS measurement.

The CAN samples were also analysed using AMS at the André E. Lalonde AMS Laboratory at the University of Ottawa using the same instrument as for seawater samples. Each sample was prepared by combusting approximately 1 g of the dried seaweed. By placing in a quartz glass tube and heating at 850°C. With oxygen flow, the vapor generated from the seaweed was trapped in a tetramethylammonium hydroxide–sodium bisulfate solution. The sample then underwent redox separation cycles, after which iodine was precipitated as silver iodide (AgI). Once the AgI precipitate was obtained for each sample, it was mixed with niobium powder and loaded into AMS copper cathodes for measurement. Calibration was carried out using ISO-6II standards and NIST reference materials.

The AMS system ensured rigorous quality control by incorporating process blanks and in-house standards, achieving nominal system blanks ($\leq 5 \times 10^{-14}$ at·at^{-1}).

At JCAC, the sample was freeze-dried, ground to a powder and homogenised. Then 0.5-1 g was added to a ceramic boat and mixed with V_2O_5 (Vanadium Oxide). Then the sample was placed in a quartz tube and heated at 1000ºC under a flow of oxygen gas. The iodine released by heating was collected with a trap solution containing TMAH (tetramethyl ammonium hydroxide) and Na_2SO_3 (sodium sulfite). The trap solution was diluted for measurement of ^{129}I by ICP-MS. Rhenium was employed as an internal standard.

TPT freeze-dried each seaweed sample after washing with seawater. It was then crushed and dissolved in 25% TMAH solution. The TMAH solution was centrifuged and the resulting supernatant water filtered. This was weighed and the ^{127}I concentration measured to determine the total iodine content of the wet seaweed sample.

The TMAH solution was exchanged to 1M HNO_3 using an anion-exchanging resin. The ^{129}I/^{127}I isotopic ratio was then measured by triple quadrupole ICP-MS (ICP-QQQ) and multiplied by the total iodine content of the wet seaweed sample.

5. STATISTICAL EVALUATION OF THE RESULTS

The IAEA collected and evaluated the results reported by all ILC participants. The method used for the statistical evaluation depended on the number of results received for each sampling location, sample type and radionuclide.

If two or three measurement results above the detection limit were received, then one or three zeta tests [5] were performed. The zeta $\zeta_{i,j}$ test is defined as:

$$\zeta_{i,j} = \frac{x_i - x_j}{\sqrt{u_i^2 + u_j^2}} \tag{1}$$

where:

x_i is the value of laboratory i (Bq $unit^{-1}$);
x_j is the value of laboratory j (Bq $unit^{-1}$);
u_i is the standard uncertainty for the value of laboratory i (Bq $unit^{-1}$);
u_j is the standard uncertainty for the value of laboratory j (Bq $unit^{-1}$); and
$unit$ is the unit of volume or mass, L or kg, as appropriate for the particular sample type.

If two results were received, $\zeta_{1,2}$ was calculated, while for three received results $\zeta_{1,2}$, $\zeta_{1,3}$ and $\zeta_{2,3}$ were calculated.

Following the current ISO standard for statistical methods for use in proficiency testing [5], ζ scores are interpreted as follows:

A ζ score between -2 and 2 indicates that the reported result is accepted at a 95.4% confidence level;
A ζ score between 2 and 3 or between -2 and -3 is considered to give a “warning signal”; and
A ζ score greater than 3 or less than -3 indicates that the reported result is not accepted at a 99.7% confidence level, an “action signal”.

A standard approach, according to the same ISO standard, is that an “action signal” can be taken as evidence that an anomaly requiring investigation has occurred.

For the purposes of this ILC, for ζ scores between -3 and 3, the corresponding result was evaluated as agreeing with the reference value at a 99.7% confidence level. (Therefore, results corresponding to “warning signals” were considered to agree with the reference value.) For ζ scores greater than 3 or less than -3, the corresponding result was evaluated as being discrepant at the same confidence level.

If the data set contained four or more results, the statistical evaluation consisted of a method for calculating a comparison reference value as a power-moderated mean of the combined results [6], which is currently being used by the Consultative Committee for Ionizing Radiation, Section II: Measurement of radionuclides, CCRI(II). After calculating a reference value, a relative degree of equivalence (DoE) was calculated for each submitted result and if this relative DoE was significantly different from zero, the corresponding result was evaluated as being discrepant. The relative DoE (%) was calculated according to:

$$\mathrm{DoE}\ (\%) = \frac{x_{\mathrm{lab}} - X_{\mathrm{ref}}}{X_{\mathrm{ref}}} . 100 \tag{2}$$

where:

x_{lab} is the individual laboratory result; and
x_{ref} is the reference value calculated as the power-moderated mean of the combined results.

The standard uncertainty of the relative DoE, u_{DoE}, was calculated according to reference [5]. If the absolute value of the relative DoE exceeded 3 times u_{DoE}, the corresponding result was evaluated as being discrepant (at a 99.7% confidence level), as the relative DoE in this case would be significantly different from zero.

6. RESULTS

6.1. GENERAL

The results are presented in Tables 6 – 15 and Figures 2 – 12.

6.1.1. Uncertainties

Uncertainties quoted in this report are combined standard uncertainties, i.e. with a coverage factor of $k = 1$. The numerical result of a measurement is stated in the format xxx ± yyy, where the number following the symbol ± is the numerical value of the combined standard uncertainty and not a confidence interval, unless otherwise indicated (i.e. in Tables 7 and 9).

6.1.2. Reference time

All activity concentrations of radionuclides in seawater, sediment, fish and seaweed samples were reported for a common reference time of 16 October 2023 12:00 UTC.

6.2. SEAWATER

Table 6 contains the results reported by the participating laboratories (IAEA, CAN, JCAC, KAKEN, KANSO, KEEA, KINS and TIO) for activity concentrations of ^{3}H, ^{60}Co, ^{90}Sr, ^{106}Ru, ^{125}Sb, ^{129}I, ^{134}Cs and ^{137}Cs in the seawater samples. Figures 2 – 3 present these results visually.

TABLE 6. ACTIVITY CONCENTRATIONS (mBq L^{-1}) IN SEAWATER SAMPLES

Nuclide	Sample	IAEA	CAN	JCAC	KAKEN	KANSO	KEEA	KINS	TIO	Reference value
^{3}H	E-S15	251 ± 19	244 ± 36	273 ± 25	–	–	–	256 ± 62	500 ± 60	300 ± 48
	T-0-1A	260 ± 21	332 ± 42	302 ± 27	290 ± 36	–	–	290 ± 75	500 ± 50	326 ± 35
	T-3	271 ± 19	246 ± 39	255 ± 25	260 ± 33	–	–	233 ± 66	420 ± 50	278 ± 26
	M-E1	46.0 ± 8.0	<47	83 ± 18	–	55 ± 12	–	<110	220 ± 30	99 ± 40
	M-E3	33.0 ± 6.0	<110	55 ± 10	–	–	44.0 ± 8.0	<110	400 ± 40	131 ± 89
^{60}Co	E-S15	<19	<30	<78	–	–	–	<85	<1.0	–
^{90}Sr		1.06 ± 0.19	0.91 ± 0.084	0.57 ± 0.10	–	–	–	2.39 ± 0.19	0.785 ± 0.046	1.13 ± 0.32
^{106}Ru		<290	<26	<670	–	–	–	<1100	<16	–
^{125}Sb		<77	<22	<170	–	–	–	<290	<10	–
^{129}I		<2.8	0.002616 ± 0.000021	<9.0	–	–	–	<82	–	–
^{134}Cs		0.367 ± 0.061	0.47 ± 0.11	<0.79	–	–	–	<1.0	<1.2	–
^{137}Cs		20.5 ± 1.2	26.1 ± 6.1	23.6 ± 1.3	–	–	–	20.0 ± 1.1	32.7 ± 1.6	24.4 ± 2.5

Table 7 contains the degrees of relative equivalence for the activity concentrations of ^{3}H, ^{60}Co, ^{90}Sr, ^{106}Ru, ^{125}Sb, ^{129}I, ^{134}Cs and ^{137}Cs in the seawater samples.

TABLE 7. DEGREES OF EQUIVALENCE (%) OF ACTIVITY CONCENTRATION OF RADIONUCLIDES IN SEAWATER SAMPLES

Nuclide	Sample	IAEA	CAN	JCAC	KAKEN	KANSO	KEEA	KINS	TIO
^{3}H	E-S15	-16 ± 50	-19 ± 55	-9 ± 51	-	-	-	-15 ± 69	67 ± 68
	T-0-1A	-20 ± 36	2 ± 45	-7 ± 38	-11 ± 42	-	-	-11 ± 69	**53 ± 50**
	T-3	-3 ± 32	-12 ± 45	-8 ± 35	-7 ± 40	-	-	-16 ± 69	51 ± 54
	M-E1	-53 ± 123	-	-16 ± 127	-	-44 ± 124	-	-	123 ± 138
	M-E3	-75 ± 203	-	-58 ± 204	-	-	-66 ± 204	-	206 ± 213
^{60}Co	E-S15	DL	DL	DL	–	–	–	DL	DL
^{90}Sr		-7 ± 94	-20 ± 87	-50 ± 88	–	–	–	**111 ± 94**	-31 ± 86
^{106}Ru		DL	DL	DL	–	–	–	DL	DL
^{125}Sb		DL	DL	DL	–	–	–	DL	DL
^{129}I		DL	Note 1	DL	–	–	–	DL	DL
^{134}Cs		Note 2	Note 2	DL	–	–	–	DL	DL
^{137}Cs		-16 ± 32	7 ± 73	-3 ± 33	–	–	–	-18 ± 32	**34 ± 34**

Notes:

The numerical results in this table are stated in the format xx ± yy, where the number following the symbol ± is the 99% confidence interval.

Note 1: No evaluation was possible as only one value above the detection limit was submitted.

Note 2: Value of 0.82 for $\zeta_{1,2}$.

DL: As a value less than the detection limit was submitted, no evaluation was performed.

$\zeta_{i,j}$ indexes: number 1 refers to IAEA, number 2 to CAN, number 3 to JCAC, number 4 to KAKEN, number 5 to KANSO, number 6 to KEEA, number 7 to KINS and number 8 to TIO.

6.3. SEDIMENT

Table 8 contains the results reported by the participating laboratories (IAEA, CAN, JCAC, KINS, TPT and TIO) for activity concentrations of ^{134}Cs and ^{137}Cs in the sediment samples. Figures 4 and 5 present these results visually.

TABLE 8. ACTIVITY CONCENTRATIONS OF ^{134}Cs AND ^{137}Cs (Bq kg^{-1} dry weight) IN SEDIMENT SAMPLES

Nuclide	Sample	IAEA	CAN	JCAC	KINS	TIO	TPT	Reference value
^{134}Cs	T-1	2.1 ± 0.14	1.65 ± 0.19	1.79 ± 0.24	1.67 ± 0.18	1.14 ± 0.14	1.61 ± 0.29	1.66 ± 0.14
	T-S4	0.201 ± 0.037	0.165 ± 0.024	<0.66	<0.45	<0.32	<0.85	–
^{137}Cs	T-1	91.7 ± 3.6	102 ± 11	105 ± 5.3	89 ± 2.9	97.3 ± 5	91.7 ± 2.7	94.4 ± 2.5
	T-S4	8.98 ± 0.37	9.12 ± 0.59	7.50 ± 0.43	8.81 ± 0.43	10.98 ± 0.6	11.4 ± 0.46	9.4 ± 0.6

Table 9 contains the degrees of relative equivalence for the activity concentrations of ^{134}Cs and ^{137}Cs in the sediment samples.

TABLE 9. DEGREES OF EQUIVALENCE (%) OF ACTIVITY CONCENTRATIONS OF ^{134}Cs AND ^{137}Cs IN SEDIMENT SAMPLES

Nuclide	Sample	IAEA	CAN	JCAC	KINS	TIO	TPT
^{134}Cs	T-1	27 ± 32	-1 ± 38	8 ± 45	1 ± 37	-3 ± 52	-31 ± 32
	T-S4	Note 1	Note 1	DL	DL	DL	DL
^{137}Cs	T-1	-3 ± 12	8 ± 34	11 ± 17	-5.8 ± 10.4	-2.9 ± 10.1	3 ± 16
	T-S4	-5 ± 22	-3 ± 25	-21 ± 22	-7 ± 22	21 ± 23	16 ± 25

Notes:

The numerical results in this table are stated in the format xx ± yy, where the number following the symbol ± is the 99% confidence interval.

Note 1: Value of 0.82 for $\zeta_{1,2}$.

DL: As a value less than the detection limit was submitted, no evaluation was performed.

$\zeta_{i,j}$ indexes: number 1 refers to IAEA, number 2 to CAN, number 3 to JCAC, number 4 to KINS, number 5 to TPT and number 6 to TIO.

6.4. FISH

Table 10 contains the results reported by the participating laboratories (IAEA, CAN, KANSO, KEEA, KINS and MERI) for activity concentrations of ^{3}H – organically bound tritium (OBT) and tissue free water tritium (TFWT) – in the fish samples collected from the fish market. Figures 6 and 7 present these results visually.

TABLE 10. ACTIVITY CONCENTRATIONS OF ^{3}H (OBT, TFWT) IN FISH SAMPLES

Nuclide	Sample number: Species	IAEA	CAN	KANSO	KEEA	KINS	MERI
^{3}H (OBT, (Bq kg^{-1} f.w.)	T23FA0001: Olive flounder	0.0155 ± 0.0021	<0.064	<0.043	<0.028	<0.32	–
	T23FA0002: Redwing searobin	0.0175 ± 0.0023	<0.064	<0.043	<0.030	<0.32	–
	T23FA0003: Pufferfish	0.0119 ± 0.0019	<0.15	<0.062	<0.030	<0.33	–
	T23FA0004: Silver croaker	0.0145 ± 0.0026	<0.16	<0.049	<0.033	<0.4	–
	T23FA0005: Crimson sea bream	0.0090 ± 0.0020	<0.15	<0.044	<0.029	<0.35	–
	T23FA0006: Japanese jack mackerel	0.0163 ± 0.0028	<0.18	<0.054	<0.035	<0.43	–
^{3}H (TFWT, Bq L^{-1})	T23FA0001: Olive flounder	0.067 ± 0.011	<0.12	<0.30	<0.20	<2.8	<0.32
	T23FA0002: Redwing searobin	0.076 ± 0.013	<0.11	<0.30	<0.21	<2.8	<0.33
	T23FA0003: Pufferfish	0.077 ± 0.013	<0.096	<0.33	<0.21	<2.8	<0.33
	T23FA0004: Silver croaker	0.072 ± 0.011	<0.055	<0.33	<0.21	<2.8	<0.31
	T23FA0005: Crimson sea bream	0.0690 ± 0.0090	<0.11	<0.31	<0.21	<2.7	<0.31
	T23FA0006: Japanese jack mackerel	0.055 ± 0.011	<0.053	<0.31	<0.21	<2.8	<0.31

Table 11 contains the results reported by the participating laboratories (IAEA, CAN, JCAC and KINS) for activity concentrations of ^{14}C in the fish samples collected from the fish market. Figure 8 presents these results visually.

TABLE 11. ACTIVITY CONCENTRATIONS OF ^{14}C (Bq kg^{-1} fresh weight) IN FISH SAMPLES[1]

Nuclide	Sample number: Species	IAEA	CAN	JCAC	KINS	Reference value
^{14}C	23FA0001: Olive flounder	22.2 ± 2	25.03 ± 0.51	22.3 ± 0.12	19.75 ± 0.66	22.3 ± 1.2
	23FA0002: Redwing searobin	24.02 ± 0.97	26.17 ± 0.54	22.6 ± 0.12	20.63 ± 0.82	23.4 ± 1.2
	23FA0003: Pufferfish	19.4 ± 4	24.95 ± 0.51	21.6 ± 0.11	20.61 ± 0.72	22.1 ± 1.2
	23FA0004: Silver croaker	20.6 ± 2.2	27.64 ± 0.57	26.5 ± 0.14	18.55 ± 0.85	23.4 ± 2.3
	23FA0005: Crimson sea bream	23.3 ± 2.1	27.81 ± 0.57	24.4 ± 0.13	23.5 ± 1.4	25.0 ± 1.1
	23FA0006: Japanese jack mackerel	22.4 ± 1.0	32.77 ± 0.67	29.9 ± 0.16	25.45 ± 0.93	27.7 ± 2.4

Table 12 contains the degrees of relative equivalence for the activity concentrations of ^{14}C in the fish samples collected from the fish market.

TABLE 12. DEGREES OF EQUIVALENCE (%) OF ACTIVITY CONCENTRATIONS OF ^{14}C IN FISH SAMPLES

Nuclide	Sample number: Species	IAEA	CAN	JCAC	KINS
^{14}C	23FA0001: Olive flounder	-1 ± 26	12 ± 16	0 ± 16	-12 ± 17
	23FA0002: Redwing searobin	3 ± 18	12 ± 16	-3 ± 16	-12 ± 17
	23FA0003: Pufferfish	-12 ± 51	13 ± 17	-2 ± 16	-7 ± 18
	23FA0004: Silver croaker	-7 ± 24	11 ± 14	-2 ± 13	-6 ± 18
	23FA0005: Crimson sea bream	-19 ± 27	19 ± 26	8 ± 26	-8 ± 27
	23FA0006: Japanese jack mackerel	-21 ± 28	19 ± 27	9 ± 27	-8 ± 28

Notes:

The numerical results in this table are stated in the format xx ± yy, where the number following the symbol ± is the 99% confidence interval.

Table 13 contains the results reported by the participating laboratories (IAEA, JCAC, KAKEN and KINS) for activity concentrations of ^{3}H – OBT and TFWT – and ^{14}C in the fish samples in fish samples caught by fixed gill nets. Figures 9 – 11 present these results visually.

TABLE 13. ACTIVITY CONCENTRATIONS OF ^{3}H (OBT, TFWT) AND ^{14}C IN FISH SAMPLES

Nuclide	Sample	IAEA	CAN	JCAC	KAKEN	KINS	TPT	Reference value
^{3}H (OBT, (Bq kg^{-1} f.w.)	E-SF3: Olive flounder	0.0462 ± 0.0044	<0.067	0.066 ± 0.016	–	<0.37	–	–
	T-S2: Olive flounder	0.0251 ± 0.0031	<0.066	–	<0.035	<0.37	–	–
	T-S7: Olive flounder	0.0188 ± 0.0025	<0.073	–	–	<0.37	<0.032	–
^{3}H (TFWT, Bq L^{-1})	E-SF3: Olive flounder	1.218 ± 0.041	1.24 ± 0.08	1.17 ± 0.059	–	<1.8	–	–
	T-S2: Olive flounder	0.086 ± 0.016	<0.11	–	0.092 ± 0.026	<1.8	–	–
	T-S7: Olive flounder	0.14 ± 0.015	0.14 ± 0.04	–	–	<1.8	0.12 ± 0.021	–
^{14}C (Bq kg^{-1} f.w)	E-SF3: Olive flounder	22.7 ± 4.6	25.69 ± 0.53	22.7 ± 0.35	–	21.64 ± 0.71	–	23.31 ± 0.99

Table 14 contains the degrees of relative equivalence for the activity concentrations of ^{14}C in the fish samples in fish samples caught by fixed gill net.

TABLE 14. DEGREES OF EQUIVALENCE (%) OF ACTIVITY CONCENTRATIONS OF ^{3}H (OBT, TFWT) AND ^{14}C IN FISH SAMPLES

Nuclide	Sample	IAEA	CAN	JCAC	KAKEN	KINS	TPT
^{3}H (OBT, (Bq kg^{-1} f.w.)	E-SF3: Olive flounder	Note 1	DL	Note 1	–	DL	–
	T-S2: Olive flounder	Note 2	DL		DL	DL	–
	T-S7: Olive flounder	Note 2	DL	–	–	DL	DL
^{3}H (TFWT, Bq L^{-1})	E-SF3: Olive flounder	Note 3	Note 3	Note 3	–	DL	–
	T-S2: Olive flounder	Note 4	DL	Note 4	DL	DL	–
	T-S7: Olive flounder	Note 5	Note 5	–	–	DL	Note 5
^{14}C (Bq kg^{-1} f.w)	E-SF3: Olive flounder	-2 ± 56	10 ± 14	-3 ± 14	–	-7 ± 15	–

Notes:

The numerical results in this table are stated in the format xx ± yy, where the number following the symbol ± is the 99% confidence interval.

Note 1: Value -1.18 for $\zeta_{1,3}$.

Note 2: No evaluation was possible as only one value above the detection limit was submitted.

Note 3: Values of -0.24, 0.67 and 0.70 for $\zeta_{1,2}$, $\zeta_{1,3}$ and $\zeta_{2,3}$, respectively.

Note 4: Value of -0.20 for $\zeta_{1,4}$.

Note 5: Values of 0.00, 0.77 and 0.44 for $\zeta_{1,2}$, $\zeta_{1,6}$ and $\zeta_{2,6}$, respectively.

DL: As a value less than the detection limit was submitted, no evaluation was performed.

$\zeta_{i,j}$ indexes: number 1 refers to IAEA, number 2 to CAN, number 3 to JCAC, number 4 to KAKEN, number 5 to KINS and number 6 to TPT.

6.5. SEAWEED

Table 15 contains the results reported by the participating laboratories (IAEA, JCAC, KINS and TPT) for activity concentrations of ^{129}I in the seaweed samples. Figure 12 presents these results visually.

TABLE 15. ACTIVITY CONCENTRATIONS OF ^{129}I (Bq kg^{-1} f.w.) IN SEAWEED SAMPLES

Nuclide	Sample	IAEA	CAN	JCAC	KINS	TPT
^{129}I	Tomioka Port (*Besa paradoxa*)	0.0095 ± 0.0010	0.01553 ± 0.00015	<0.070	<0.085	–
	Tomioka Port (*Eisenia bicyclis*)	0.0132 ± 0.00035	0.011372 ± 0.000091	–	<0.076	<0.10
	Ukedo Port (*Besa paradoxa*)	0.00583 ± 0.00031	0.003944 ± 0.000038	–	<0.090	<0.10

Evaluation of the results submitted by IAEA and CAN for the three samples has not been performed as the values are extremely low (between 7 and 25 times lower than the target detection limit of 0.1 Bq/kg f.w. defined in Japan’s Comprehensive Radiation Monitoring Plan (CRMP) [2]) and, although both based on measurement of ^{127}I/^{129}I ratios by AMS, the analytical methods used are quite different, particularly the specifications of the AMS instrumentation used, the sample preparation procedures and the estimation of measurement uncertainties. In addition, the small mass of sample material used for each AMS (approximately 1 g) could result in small scale inhomogeneities in the analyses due to variable uptake of ^{129}I by different parts of the seaweed. Therefore, it was deemed that a valid intercomparison was not possible.

7. CONCLUSION

A detailed data analysis was performed on the activity concentrations reported for ^{3}H, ^{60}Co, ^{90}Sr, ^{106}Ru, ^{125}Sb, ^{129}I, ^{134}Cs and ^{137}Cs in seawater samples, for ^{134}Cs and ^{137}Cs in sediment samples, for organically bound tritium (OBT), tissue free water tritium (TFWT) and ^{14}C in fish samples, and ^{129}I seaweed samples. All samples were collected offshore TEPCO's FDNPS in October 2023. The samples were shared between ten laboratories: six from Japan (JCAC, KAKEN, KANSO, KEEA, MERI and TPT); IAEA; three ALMERA network member laboratories (CAN, KINS and TIO).

IAEA analyses were undertaken at two participating IAEA Nuclear Sciences and Applications Laboratories:

- IAEA Marine Environment Laboratories, Radiometrics Laboratory (RML), Monaco; and
- Isotope Hydrology Laboratory (IHL), Vienna, Austria.

From this analysis it can be concluded that the majority of results are not significantly different from each other. A global analysis of the whole data set demonstrated three discrepant[4] values from the 87 statistical tests applied to the data, i.e. more than 96 % were passed with a high level of confidence (99.7%). The exceptions were the following cases where the relative DoE was significantly different from zero:

- DoE (%) = 53 ± 50 for the ^{3}H activity concentration in the seawater sample from T-0-1A submitted by TIO.
- DoE (%) = 111 ± 94 for the ^{90}Sr activity concentration in the seawater sample from E-S15 submitted by KINS.
- DoE (%) = 34 ± 34 for the ^{137}Cs activity concentration in the seawater sample from E-S15 submitted by TIO (after rounding).

The low number of discrepant results in this ILC is impressive, especially since the analytical methods are often complex (e.g. for OBT, TFWT and ^{14}C in fish) and relatively new to some participating laboratories, being implemented specifically to assess the ALPS treated water discharges. It is noted that higher uncertainties are usually associated with complex analyses of low, and close to detection limit, levels. There were no order of magnitude variations, and no systemic deviations identified between the results reported by Japanese laboratories and those reported by the IAEA and the ALMERA member laboratory. Therefore, despite the three discrepancies, it can be said with confidence that the laboratories are reporting fit-for-purpose reliable and comparable results for the tested radionuclides in seawater, sediment, fish and seaweed samples prepared and analysed according to each laboratory's regularly used methods. It can also be noted that many results reported were below detection limits and therefore could not be quantitatively intercompared. However, the values reported adhered to the target detection limits for reach sample and radionuclide as defined in the CRMP. The IAEA recommends the organisation of such ILCs annually to maintain and improve the quality of the data reported by the participating laboratories.

Therefore, based on the results of its evaluation, the IAEA can report that Japan's sample collection procedures follow the appropriate methodological standards required to obtain representative samples. The accurate results obtained in this ILC demonstrate a high degree of proficiency on the part of the

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[4] As defined in section 5.

Japanese laboratories involved in the analyses of radionuclides in marine samples for environmental monitoring related to the discharges of ALPS treated water as part of the Government of Japan’s CRMP.

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[5] Note: 3rd revision published in October 2023: https://www.kankyo-hoshano.go.jp/wp-content/uploads/2023/10/No09_R510.pdf

APPENDIX: FIGURES

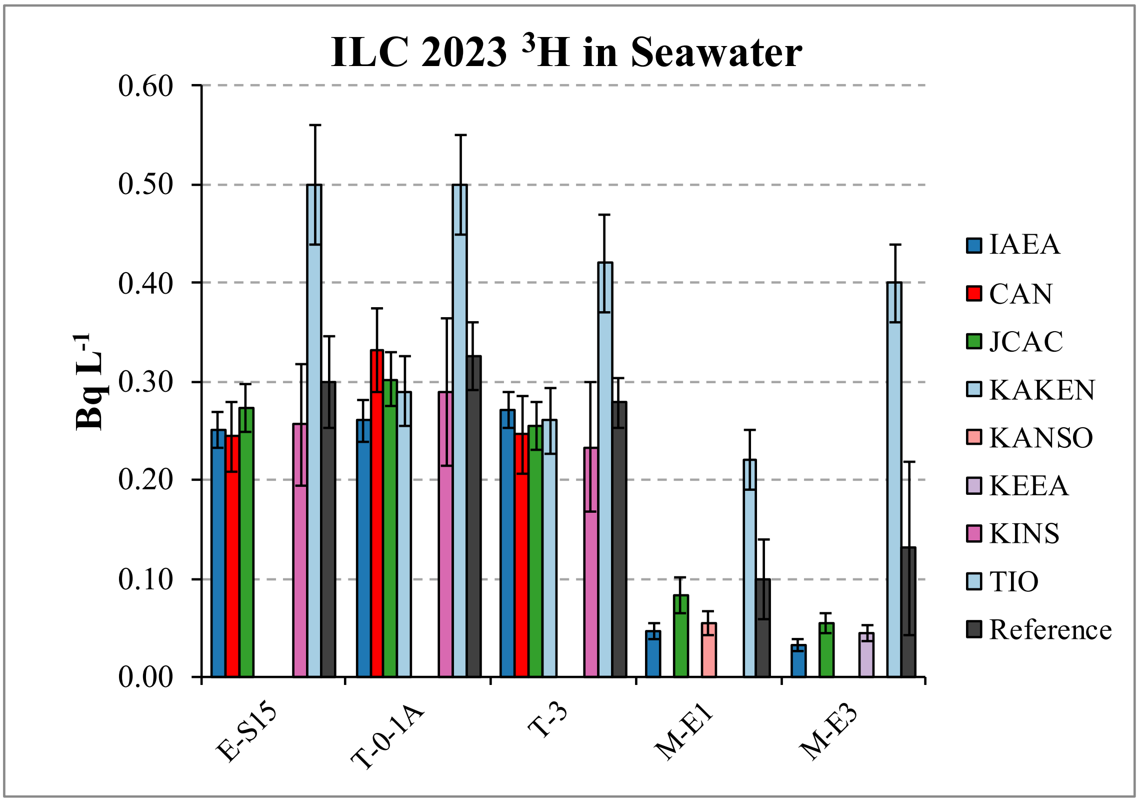

FIG. 2. Activity concentrations of ^{3}H in seawater samples.

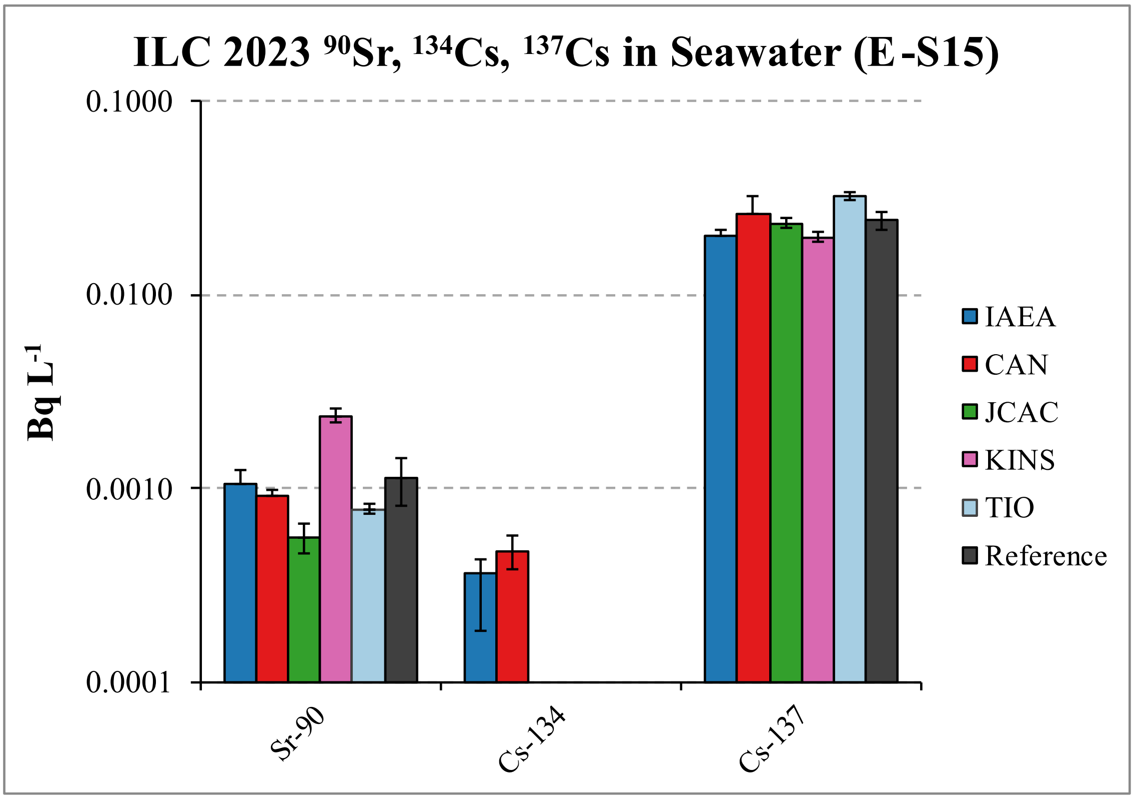

FIG. 3. Activity concentrations of ^{90}*Sr,* ^{134}*Cs and* ^{137}*Cs in the seawater sample from E-S15 (note logarithmic scale).*

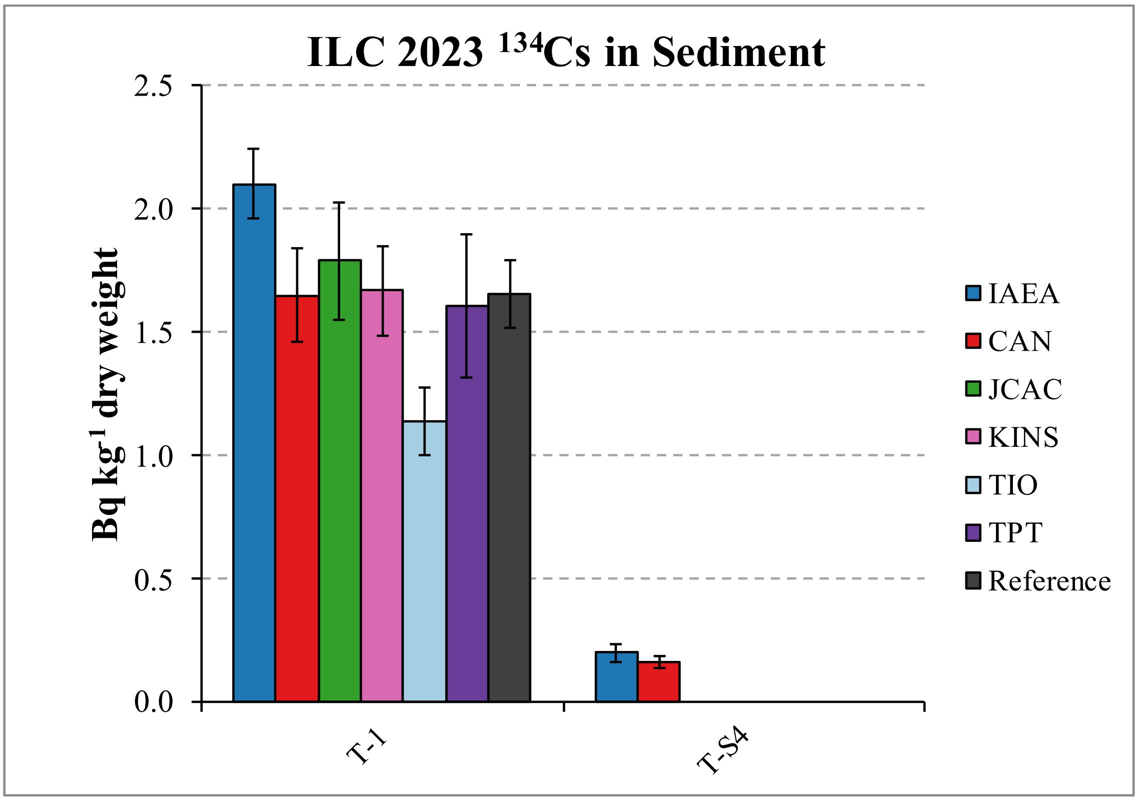

FIG. 4. Activity concentrations of ^{134}*Cs in sediment samples.*

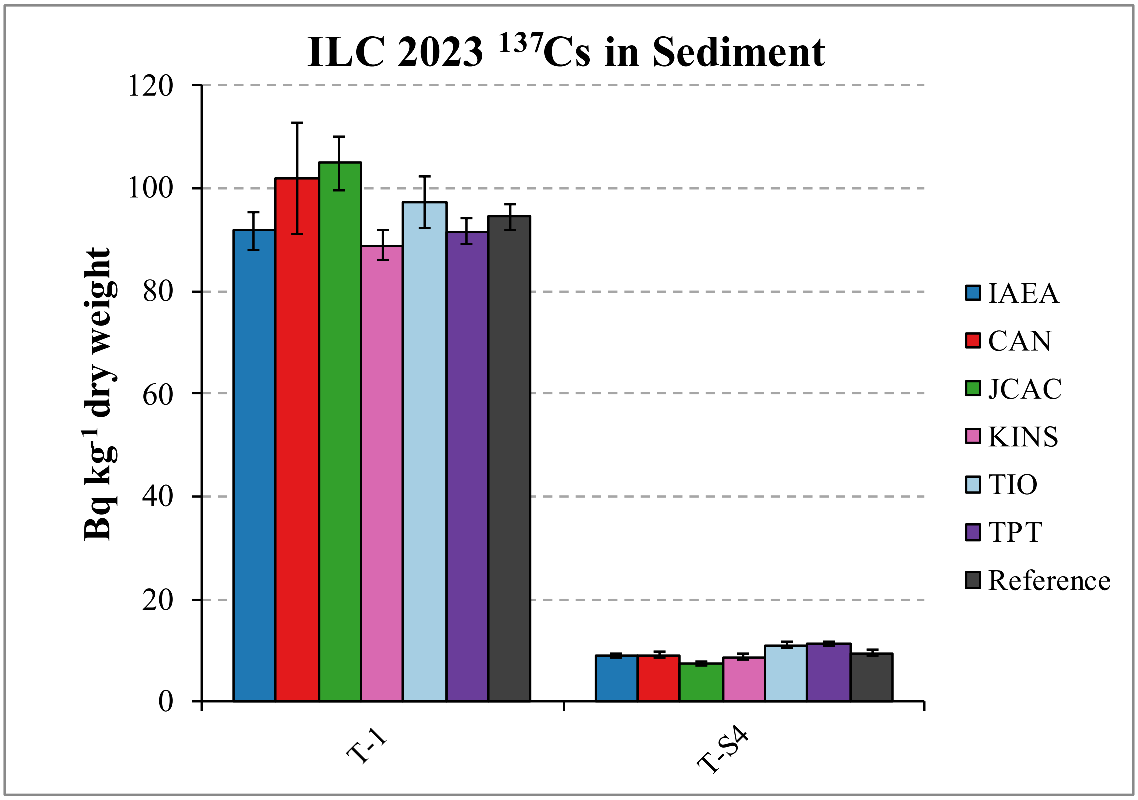

FIG. 5. Activity concentrations of ^{137}Cs in sediment samples.

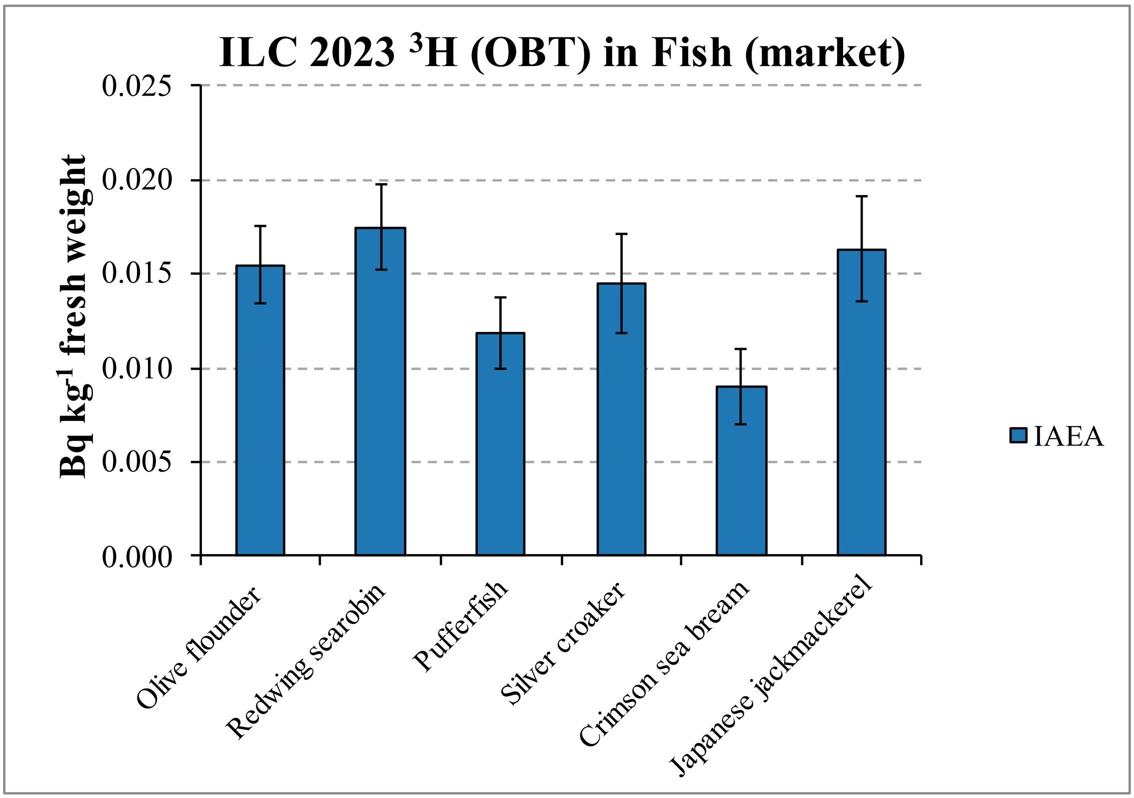

FIG. 6. Activity concentrations of ^{3}H (OBT) in fish samples collected from a fish market.

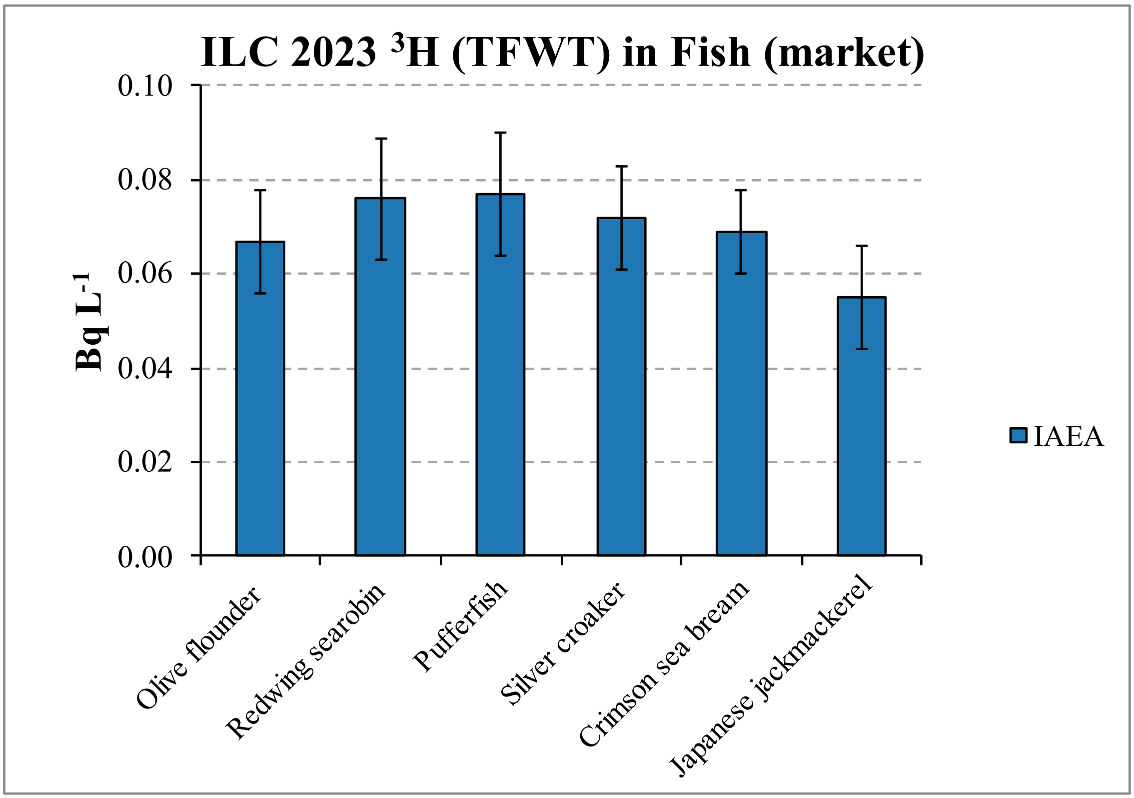

FIG. 7. Activity concentrations of ^{3}H (FWT) in fish samples collected from a fish market.

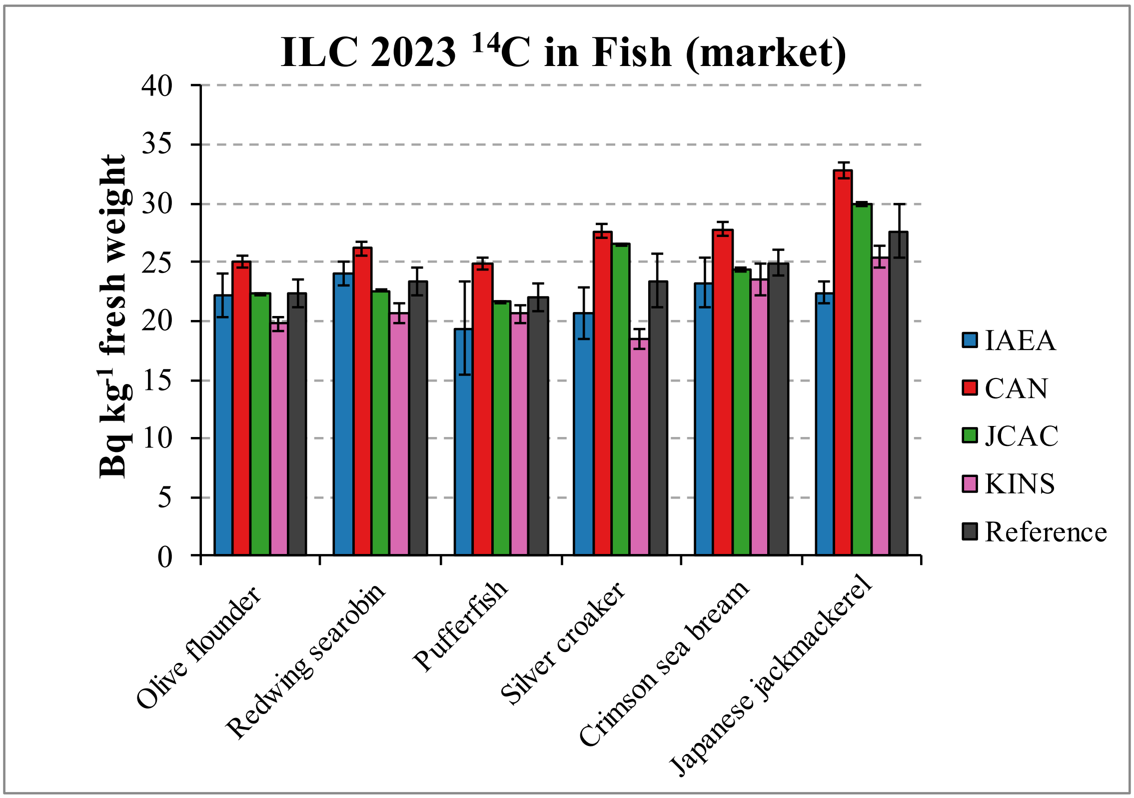

FIG. 8. Activity concentrations of ^{14}C in fish samples collected from a fish market.

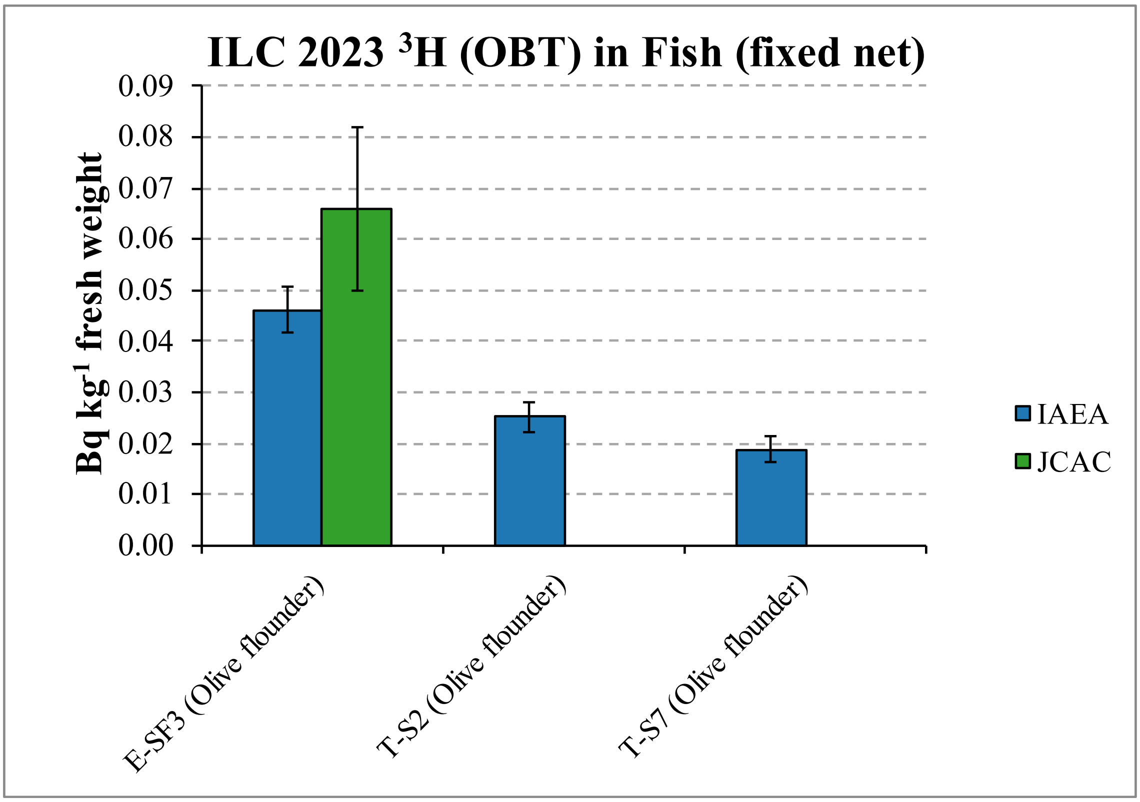

FIG. 9. Activity concentrations of ^{3}H (OBT) in fish samples caught by fixed gill net.

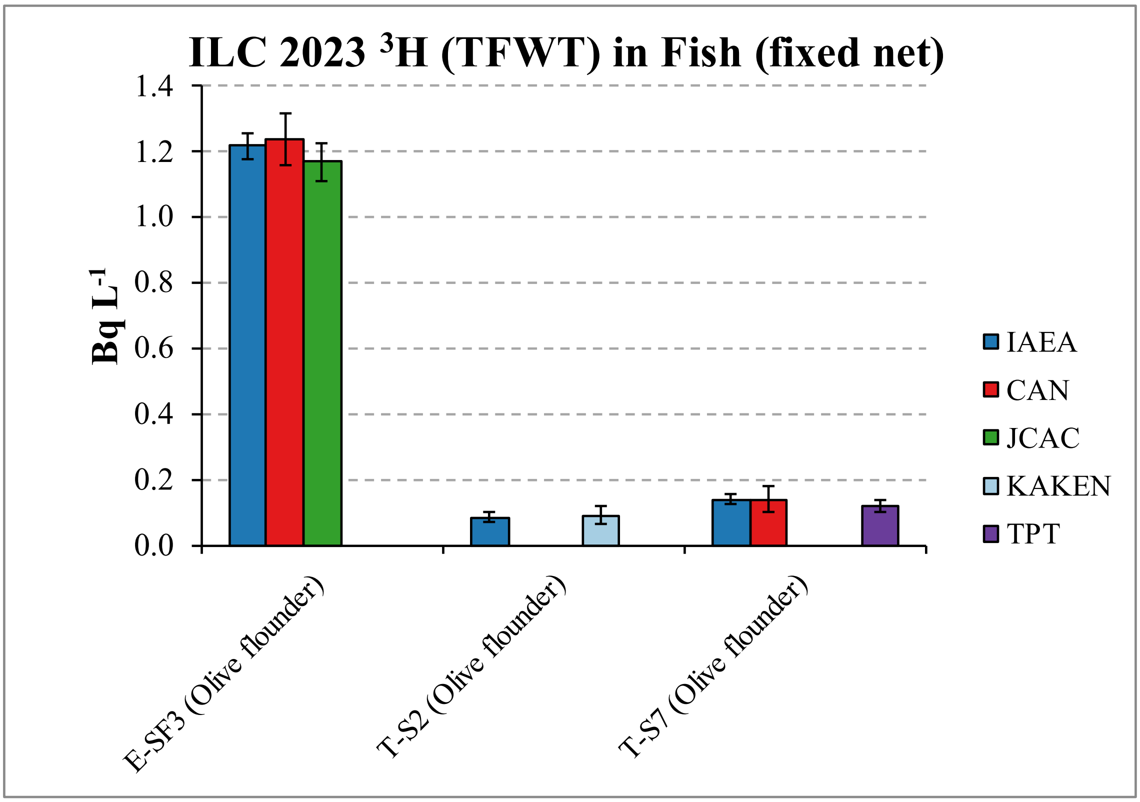

FIG. 10. Activity concentrations of ^{3}H (TFWT) in fish samples caught by fixed gill net.

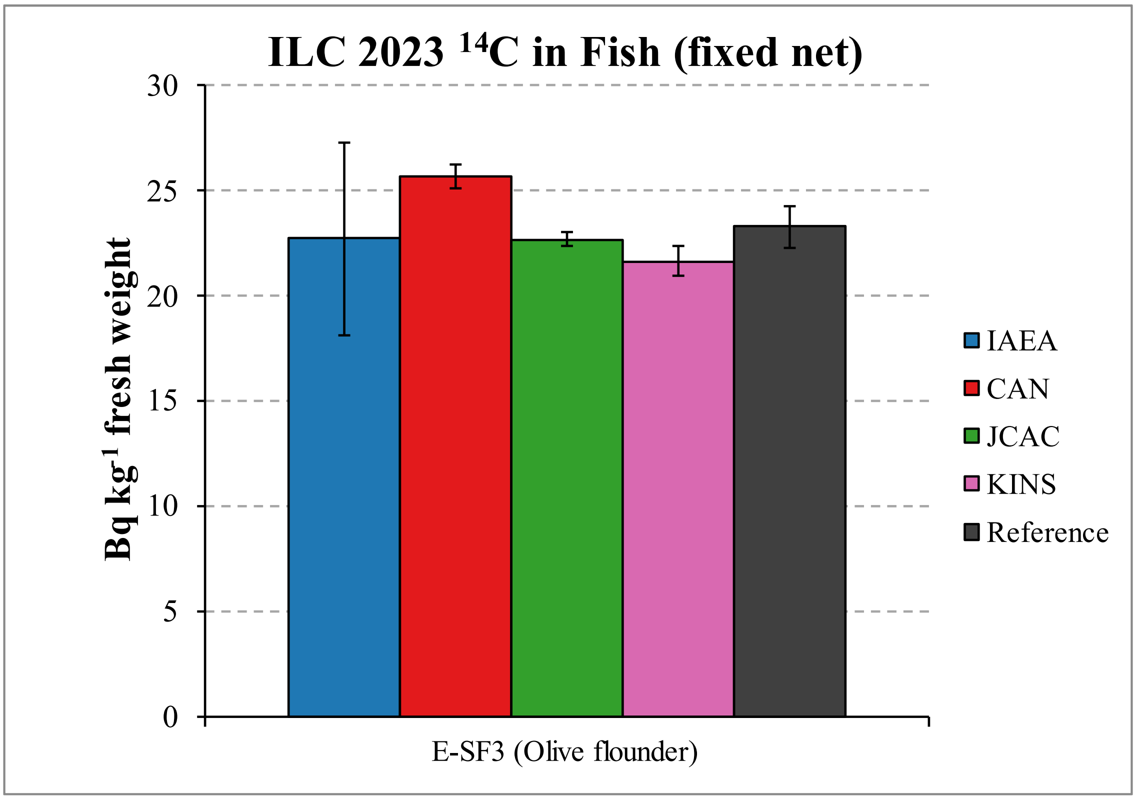

FIG. 11. Activity concentrations of ^{14}C in fish samples caught by fixed gill net.

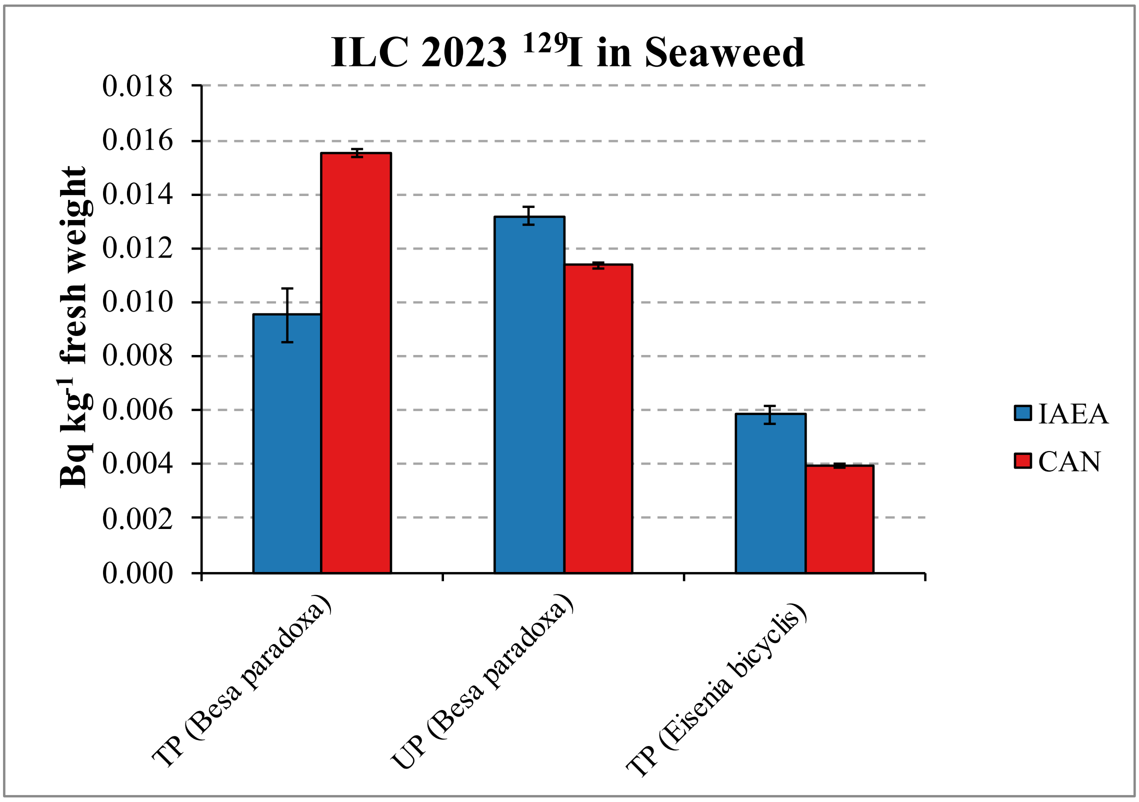

FIG. 12. Activity concentrations of ^{129}I in seaweed samples. (TP = Tomioka Port; UP = Ukedo Port)

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