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 ALPS Treated Water

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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 current 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 approximately 30 years.

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 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 other Japanese ministries and organizations 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 source monitoring under the IAEA's ALPS safety review, ALPS treated water samples were taken in October 2022 from two tanks, G4S-B10 and G4S-C8, at FDNPS. Unlike the first ILC to corroborate source monitoring, the samples were taken from standard tanks used for the storage of ALPS treated water and not from a measurement and confirmation unit in the discharge facility at FDNPS. The samples were therefore not representative of a batch of ALPS treated water due to be discharged but nevertheless facilitated comparisons of the measurement results of TEPCO with those of the

IAEA laboratories and with a selected third-party laboratory from the IAEA ALMERA network (Analytical Laboratories for the Measurement of Environmental Radioactivity) [1]. The objective of the ILC is therefore to assess TEPCO's capability to undertake analyses relevant to source monitoring with respect to discharges of ALPS treated water to the required standard and to report high quality and comparable results.

The focus of the analysis efforts for this ILC were on the radionuclides from the source term (Table 1) which are included in the radiological environmental impact assessment conducted by TEPCO. A source term refers to the amount and isotopic composition of radioactive material that is released, in this case as part of a controlled discharge. It is used for modelling releases of radionuclides into the environment and allows for the estimation of doses to be included as part of the radiological environmental impact assessment. The source term also defines the radionuclides that should be included in the respective source monitoring plan and informs the requirements for environmental monitoring.

Analyses were undertaken by TEPCO and by the following three participating IAEA Nuclear Sciences and Applications Laboratories:

- IAEA Marine Environment Laboratories, Radiometrics Laboratory (RML), Monaco;
- Terrestrial Environmental Radiochemistry Laboratory (TERC), Seibersdorf, Austria;
- Isotope Hydrology Laboratory (IHL), Vienna, Austria.

Additionally, under the coordination of the participating IAEA laboratories, a selected third-party laboratory, Korea Institute of Nuclear Safety (KINS), Republic of Korea, a member of the IAEA ALMERA network with demonstrable competence in the methods required, also conducted analyses of samples as a participant in the ILC. ALMERA is a network comprising 195 member laboratories globally that is coordinated jointly by RML and TERC. 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.

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.

For other radionuclides, the detection limits reported by participating laboratories were compared to evaluate whether the analytical methods used by TEPCO were broadly equivalent and thus appropriate and fit for purpose.

The results are presented in tables and charts in this report. Reference is made to the relevant regulatory limit for discharge to sea for each radionuclide as appropriate.

The key findings of this ILC are:

- TEPCO has demonstrated a high level of accuracy in their measurements and technical competence.
- TEPCO's sample collection procedures follow the appropriate methodological standards required to obtain representative samples.

- The selected analytical methods utilized by TEPCO for different radionuclides were appropriate and fit for purpose. The reported detection limits were less than 1% of the respective regulatory limits for all radionuclides included in TEPCO's source term.

The IAEA notes that these findings provide confidence in TEPCO's capability for undertaking accurate and precise measurements related to the discharge of ALPS treated water. Furthermore, based on the observations of the IAEA, TEPCO has demonstrated that they have a sustainable and robust analytical system in place to support the ongoing technical needs at FDNPS during the discharge of ALPS treated water.

Additional ILCs will be conducted in the future for ALPS treated water, as well as environmental samples, and occupational radiation protection[1]. The results from the first ILC for environmental samples, which were taken in November 2022, have been published; these first samples serve as a baseline for future environmental monitoring. Future ILCs, now that discharges of ALPS treated water have started, will allow for an assessment of any changes in the levels of relevant radionuclides in the marine environment, relative to the baseline. 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 Comprehensive Radiation Monitoring Plan 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 fisheries samples. Data from this project are available online[2].

The results from the first ILC for occupational radiation protection will be available later in 2024. This first ILC will focus on external dosimetry, whereas future ILCs for occupational radiation protection will focus on internal dosimetry. ILCs for occupational radiation protection will be conducted between the IAEA's Radiation Safety Technical Services Laboratory and the individual monitoring service used by TEPCO for FDNPS workers.

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[1] The IAEA is currently in discussions with other laboratories to participate in future ILCs.

[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

CONTENTS

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 discharges of ALPS treated water are being evaluated. The scope of the corroboration includes an independent check of the radiological characterization of the discharges. A primary objective is to promote transparency and provide sound information to enable interested parties to evaluate the radiological data used as the basis for planning the discharges of ALPS treated water into the sea.

The IAEA corroboration of source and environmental monitoring related to discharges of ALPS treated water from FDNPS is comprised of three distinct elements (see also Figure 1):

1. Review of sampling and analytical methods for source and environmental monitoring related to ALPS treated water at FDNPS used by TEPCO and relevant Japanese authorities.
2. Corroboration of source monitoring undertaken by TEPCO, including a comprehensive radiological characterization of ALPS treated water samples.
3. Corroboration of environmental monitoring undertaken by TEPCO and relevant Japanese authorities.

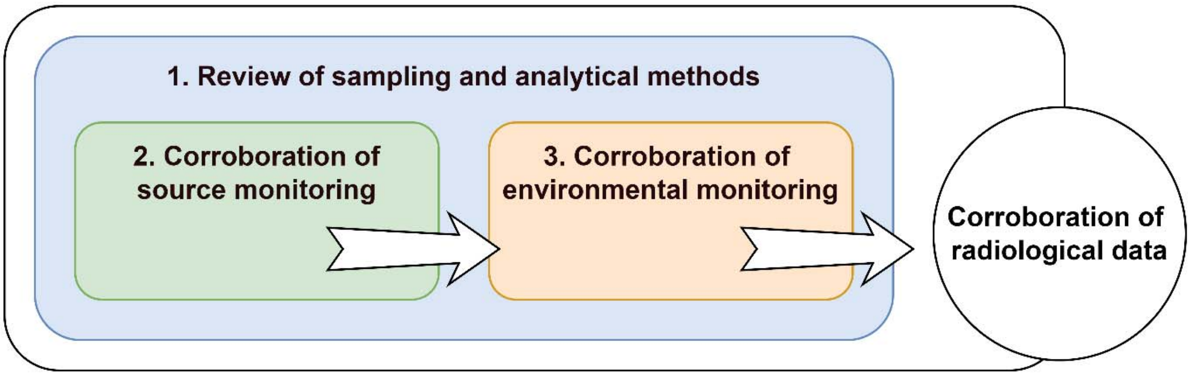

FIG. 1: A schematic overview of the elements of the corroboration being undertaken by the IAEA laboratories and the links between these elements.

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 [2].

This publication reports the results of the second ILC to corroborate source monitoring.

This ILC was based on two samples of ALPS treated water collected from tanks G4S-B10 and G4S-C8 at FDNPS in October 2022 with IAEA observation. Unlike the first ILC to corroborate source monitoring [3], the samples were taken from standard tanks used for the storage of ALPS treated water and not from a measurement and confirmation unit in the discharge facility at FDNPS. The samples were therefore not representative of a batch of ALPS treated water due to be discharged but nevertheless facilitated comparisons of the measurement results of TEPCO with those of the IAEA laboratories and with a selected third-party laboratory from the IAEA ALMERA network (Analytical Laboratories for the Measurement of Environmental Radioactivity) [1]. The objective of the ILC is therefore to assess TEPCO's capability to undertake analyses relevant to its source monitoring to the required standard and to report high quality and comparable results.

The results of the analyses were reported to the IAEA. A data compilation and a statistical evaluation to assess agreement was then carried out by the IAEA.

The IAEA wishes to thank all the 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. The IAEA personnel responsible for this publication was P. McGinnity of the IAEA Nuclear Science and Applications Department.

1.2. OBJECTIVE

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

1.3. SCOPE

This publication reports all aspects of the second ILC to corroborate source 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 descriptions of the ILC design and participating laboratories (Section 2); the methods employed for the sampling of ALPS treated water and for distribution of the samples between participating laboratories (Section 3), the methods used by each participating laboratory to determine activity concentrations of radionuclides in the samples (Section 4) and the methodology employed for the statistical evaluation of the results (Section 5). The results of the ILCs are presented in Section 6 and conclusions in Section 7. Key results are also presented in charts in Appendices II and III.

2. DESIGN AND PARTICIPATING LABORATORIES

2.1. DESIGN

The ILC was comprised of quantitative comparisons of the results of analyses using statistical methods to assess any differences between TEPCO's results and those of the IAEA laboratories and the participating third-party laboratory. Comparisons have been undertaken for all radionuclides that are included in TEPCO's ALPS treated water source term and for which activity concentrations above detection limits were reported by at least two laboratories. The specific radionuclides in the source term – those that have been identified as requiring measurement and assessment within TEPCO's source monitoring plan [4] – are listed in Table 1. TEPCO was required to analyse for all radionuclides in its source terms. The IAEA analysed for as many radionuclides as possible, subject to the analytical capability and resources available and the third-party laboratory was encouraged to do likewise. The results were statistically evaluated according to the methodology described in Section 5.

TABLE 1. RADIONUCLIDES INCLUDED IN TEPCO'S ALPS TREATED WATER SOURCE TERM

^{3}H	^{79}Se	^{125m}Te	^{151}Sm	^{238}Pu
^{14}C	^{90}Sr	^{129}I	^{154}Eu	^{239}Pu
^{54}Mn	^{90}Y	^{134}Cs	^{155}Eu	^{240}Pu
^{55}Fe	^{99}Tc	^{137}Cs	^{234}U	^{241}Pu
^{60}Co	^{106}Ru	^{144}Ce	^{238}U	^{241}Am
^{63}Ni	^{125}Sb	^{147}Pm	^{237}Np	^{244}Cm

Laboratories participating in the ILC were given freedom to determine activity concentrations of radionuclides by any appropriate method of their choice. Approximate activity concentrations of commonly detected radionuclides (^{60}Co, ^{90}Sr, ^{106}Ru, ^{125}Sb, ^{129}I, ^{134}Cs, ^{137}Cs, ^{3}H, ^{14}C and ^{99}Tc) in the G4S tanks from which the samples were collected has been published by TEPCO [5]. This data could be used by the participating laboratories to inform on selection of analytical methods. For other radionuclides, the authorised limits for discharge provided an upper bound when evaluating target detection limits [6].

Detailed instructions for this ILC were sent to all participating laboratories before shipment of the samples. Participating laboratories were requested to submit a single measurement result for each radionuclide analysed decay corrected to the sampling date (19 October 2022). A measurement result was assumed to be comprised of an activity concentration and associated uncertainty, or the detection limit, as appropriate, all expressed in Bq/L. Participating laboratories were asked to report additional information for each radionuclide as described in Appendix I.

2.2. PARTICIPATING LABORATORIES

The laboratories participating in analyses for this ILC are presented in Table 2.

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

- IAEA Marine Environment Laboratories, Radiometrics Laboratory (RML), Monaco;
- Terrestrial Environmental Radiochemistry Laboratory (TERC), Seibersdorf, Austria;
- Isotope Hydrology Laboratory (IHL), Vienna, Austria.

A third-party laboratory from Republic of Korea, the Korean Institute of Nuclear Safety (KINS) a member of the network of Analytical Laboratories for the Measurement of Environmental Radioactivity (ALMERA) [1] with demonstrable competence in the methods required, was selected by the IAEA to participate in the ILC. ALMERA is a network comprising 195 member laboratories globally that is coordinated jointly by RML and TERC. 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.

The participation of each laboratory in specific analyses is presented in Tables 3.

TABLE 2. LABORATORIES PARTICIPATING IN THE ILCS

Identifier	Laboratory
IAEA	IAEA Laboratories, Austria and Monaco
KINS	Korea Institute of Nuclear Safety, Republic of Korea
TEPCO	Tokyo Electric Power Company Holdings, Inc., Japan

TABLE 3. ALPS TREATED WATER SOURCE TERM RADIONUCLIDES ANALYSED BY PARTICIPATING LABORATORIES (G4S-B10 SAMPLES)

Nuclide	IAEA	KINS	TEPCO
^{3}H	🟩	🟩	🟩
^{14}C		🟩	🟩
^{54}Mn	🟨	🟨	🟨
^{55}Fe	🟨	🟨	🟨
^{60}Co	🟩	🟩	🟩
^{63}Ni	🟩	🟩	🟨
^{79}Se			🟨
^{90}Sr[1]	🟩	🟩	🟩
^{99}Tc[2]	🟩	🟨	🟨
^{106}Ru	🟨	🟨	🟨
^{125}Sb[3]	🟩	🟨	🟩
^{129}I[4]	🟩	🟩	🟩
^{134}Cs[2]	🟩	🟨	🟨
^{137}Cs[5]	🟩	🟩	🟩
^{144}Ce	🟨	🟨	🟨
^{147}Pm			🟨
^{151}Sm			🟨
^{154}Eu	🟨	🟨	🟨
^{155}Eu	🟨	🟨	🟨
^{234}U[6]	🟨	🟩	🟨
^{238}U[6]	🟨	🟩	🟨
^{237}Np	🟨	🟨	🟨
^{238}Pu[7]	🟨	🟨	🟨
^{239}Pu[7,8]	🟨	🟨	🟨
^{240}Pu[7,8]		🟨	🟨
^{241}Pu		🟨	🟨
^{241}Am	🟩	🟨	🟨
^{244}Cm			🟨

Notes: A green cell indicates that an activity concentration and associated standard uncertainty was reported, a yellow cell indicates that a detection limit was reported and a blank cell indicates that the analysis was not performed.

[1] ^{90}Y is included in TEPCO's source but it can be assumed to be in equilibrium with ^{90}Sr and present with an equal activity concentration. TEPCO reported a result for ^{90}Y derived from the measured activity concentration of ^{90}Sr on this basis.

[2] IAEA reported a value less than the detection limit for the sample from G4S-C8.

[3] ^{125m}Te is also included in TEPCO's source term but it can be assumed to be in equilibrium with ^{125}Sb and present with an activity concentration approximately equal to 0.23 times the activity concentration of ^{125}Sb after one year. TEPCO reported a result for ^{125m}Te derived from the measured activity concentration of ^{125}Sb on this basis.

[4] IAEA and KINS reported values less than the detection limits for the sample from G4S-C8.

[5] All laboratories reported values less than the detection limits for the sample from G4S-C8.

[6] KINS reported a value less than the detection limit for the sample from G4S-C8.

[7] IAEA reported a value above the detection limit for the sample from G4S-C8.

[8] A combined value for ^{239,240}Pu was reported by IAEA.

3. COLLECTION AND PREPARATION OF ALPS TREATED WATER SAMPLES

Samples for this ILC were collected from two tanks, G4S-B10 and G4S-C8, on 19 October 2022 from the G4 South tank storage area at FDNPS, with observation by IAEA staff members.

The G4S-B10 and G4S-C8 tanks are standard tanks used for the storage of ALPS treated water at FDNPS. In contrast to the tanks which provided the samples for the first ILC on the determination of radionuclides in ALPS treated water, the contents are not required to be homogenous. To ensure the provision of identical samples to each ILC participant when sampling from the G4S-B10 and G4S-C8 tanks, inter-sample homogeneity was assured by first collecting ALPS treated water from each tank in large plastic containers (500 L for G4S-B10 and 300 L for G4S-C8). In each case, the collected sample volume transferred to a second large plastic container of the same respective size and then back to the first. Sample containers (3 L) were then filled for each participating laboratory. Underwater pumps were used to transfer the water between tanks and into the sample containers. To further promote inter-sample homogeneity, the water was agitated using stirring devices at all times while transferring between large plastic containers and into the sample containers.

The samples were prepared for shipping to each participating laboratory. They were received by the IAEA laboratories in Monaco and Austria, and by the participating ALMERA laboratory in November 2022.

4. ANALYTICAL METHODS

A general description of the analytical methods used for relevant analyses are described in this section. Specific variations are provided for each laboratory.

4.1. GENERAL PRE-TREATMENT OF THE ALPS TREATED WATER SAMPLE

Pre-treatment of the ALPS treated water samples performed by the IAEA laboratories was guided by the results of prior robustness testing with similar samples. This robustness testing was designed to examine the potential sources of variability in analyses of radionuclide activity concentrations of ALPS treated water samples due to sampling, temporary storage and transportation methods and is described in an earlier IAEA report [7]. The robustness testing indicated that while filtration at 0.45µm was a parameter that could potentially affect analyses for activity concentrations of some radionuclides, the results were not conclusive. Therefore, no predictable and, thus, preventable effect was recorded.

Therefore at the IAEA laboratories, the pre-treatment of the samples to be analysed in this ILC followed standard protocol and best practice by separating an aliquot for volatile radionuclides (^{3}H, ^{14}C, ^{129}I), then acidifying the remaining volume to < pH 2 using nitric acid with a delay of 24 hours before analysis. This pre-treatment was designed to ensure that any particle reactive radionuclides (e.g. Pu/Th/U isotopes) present in the samples would be desorbed from the walls of the plastic sampling container and any particulates present to provide the most conservative analysis results.

KINS and TEPCO reported no pre-treatment of the ALPS treated water samples prior to analysis.

4.2. GAMMA-EMITTING RADIONUCLIDE ANALYSES (^{54}Mn, ^{60}Co, ^{106}Ru, ^{125}Sb, ^{129}I, ^{134}Cs, ^{137}Cs, ^{144}Ce, ^{154}Eu, ^{155}Eu, ^{241}Am)

The samples were analysed for the gamma-ray emitting radionuclides ^{54}Mn, ^{60}Co, ^{106}Ru, ^{125}Sb, ^{134}Cs, ^{137}Cs, ^{144}Ce, ^{154}Eu and ^{155}Eu included in TEPCO's ALPS treated water source term by gamma-ray spectrometry using high purity germanium (HPGe) detectors by all participating laboratories. ^{106}Ru is determined by gamma-ray spectrometry via decay of its progeny ^{106}Rh.

At all laboratories, each sample was prepared in Marinelli beakers and counted without further treatment. The volumes of the beakers varied from 1L (IAEA and KINS) to 5L (TEPCO).

Efficiency calibrations were determined from measurements of calibration sources prepared from traceable reference standards. A software-based technique to determine efficiency calibrations, Mirion's LabSOCS (Laboratory Sourceless Calibration Software), was additionally used in IAEA. Correction for true coincidence summing was undertaken, when necessary, also using LabSOCS (IAEA) and EFFTRAN (IAEA, KINS). TEPCO determined correction factors experimentally.

Spectral analysis was undertaken using Mirion's Genie 2000 (IAEA, KINS) and Seiko EG&G's Gamma Station (TEPCO). Nuclide data from DDEP/ Nucléide-Lara was used by IAEA and KINS and Table of Isotopes, 6th Edition by TEPCO.

All participating laboratories reported that regular quality control procedures to control peak centroid, detector resolution, efficiency and background were being undertaken.

4.3. ^{129}I ANALYSIS

The samples were analysed for ^{129}I by IAEA and KINS using the gamma-ray spectrometry method described in the previous section. TEPCO selected a different analytical method using ICP-MS after oxidation state adjustment for the determination of ^{129}I.

4.4. ^{3}H ANALYSIS

IAEA added aliquots of the samples directly into a Teflon-coated LSC vials and mixed them with a scintillation cocktail. These were stored in a dark location for approximately 12 hours prior to measurement by liquid scintillation counting. Recovery was based on a metrologically traceable standard solution, using the same region of interest as the samples [8].

KINS and TEPCO purified the samples by removing organic matter and interfering radionuclides via distillation [8,9] prior to mixing them with a scintillation cocktail and measurement by liquid scintillation counting.

4.5. ^{14}C ANALYSIS

Samples were prepared by creating the evolution of CO_2 followed by collection in an absorbing solution (e.g. Carbo-Sorb E) and mixed with a scintillation cocktail (e.g. PermaFluor E+). TEPCO utilized wet oxidation for the evolution of CO_2; KINS employed a pyrolizer to combust and trap oxidized samples [10,11]. All samples were measured using liquid scintillation counting.

4.6. ^{90}Sr ANALYSIS

All laboratories followed the general procedure to selectively remove impurities and interfering nuclides using extractive chromatographic resin, and gravimetric recovery of Sr ions via strontium carbonate precipitation.

The removal of impurities and interfering radionuclides can be achieved using multiple techniques. IAEA used dicyclohexyl-18-crown-6-ether-based Sr resin column and KINS used cation exchange resin (Dowex 50W-X8) [12,13].

IAEA and KINS measured ^{90}Sr using liquid scintillation counting. TEPCO used a plastic scintillation detector [14].

4.7. ^{55}Fe ANALYSIS

IAEA and KINS used an anion chromatography resin (e.g. 1-X4 Biorad) for chemical separation of ^{55}Fe from the sample matrix followed by liquid scintillation counting [15]. TEPCO selected a different analytical method using an anion chromatography resin for chemical separation, hydroxide precipitation for source preparation and a low-energy photon detector (Ge-LEPS) for analysis [16,17].

IAEA results were provided by subcontractor Laboratorio de Medidas de Baja Actividad de la Universidad del Pais Vasco UPV/EHU, accreditation number 350/LE560 under Entidad Nacional de Accreditacion.

4.8. ^{63}Ni ANALYSIS

All laboratories used a chemical separation with an anion exchange resin (e.g., 1-X4 Biorad) followed by a selective Ni extractive chromatographic resin to extract ^{63}Ni from the sample matrix. Measurements were conducted by liquid scintillation counting [15,18].

IAEA results were provided by subcontractor Laboratorio de Medidas de Baja Actividad de la Universidad del Pais Vasco UPV/EHU, accreditation number 350/LE560 under Entidad Nacional de Accreditacion.

4.9. ^{99}Tc ANALYSIS

IAEA and KINS separated ^{99}Tc from the sample matrix using an extractive chromatographic resin [19]. The IAEA analysis was then performed by liquid scintillation counting and by ICP-MS at KINS.

TEPCO did not separate ^{99}Tc from the sample matrix and used a simple dilution followed by analysis using ICP-MS [18].

IAEA results were provided by subcontractor Laboratorio de Medidas de Baja Actividad de la Universidad del Pais Vasco UPV/EHU, accreditation number 350/LE560 under Entidad Nacional de Accreditacion.

4.10. ^{79}Se ANALYSIS

After selective removal of impurities and interfering nuclides by sedimentation and TEVA resin, TEPCO added hydroxylamine hydrochloride to precipitate Se. After filtering, dissolution and mixing with scintillation cocktails, ^{79}Se was measured using liquid scintillation counting [18].

4.11. ^{241}PU ANALYSIS

KINS performed the analysis of ^{241}Pu by ICP-MS.

4.12. ALPHA-EMITTING RADIONUCLIDE ANALYSIS

Selective removal of impurities and interference radionuclides sample matrix using an extractive chromatographic resin, with recovery measured via addition of suitable tracers. Sources for alpha-particle spectrometry were prepared by electrodeposition on stainless steel discs or via micro co-precipitation.

KINS performed the analysis of ^{237}Np, ^{239}Pu, and ^{240}Pu by ICP-MS. The samples were analysed for ^{241}Am by gamma-ray spectrometry at KINS using the method described above.

TEPCO use gross alpha screening as a conservative estimate of the activity concentrations of individual alpha emitting radionuclides in the samples. Alkali and alkaline earth metal ions were separated via Fe(III) hydroxide co-precipitation. Precipitation was dissolved in HCl and Fe ions were removed by solvent extraction using 2,6-Dimethyl-4-heptanone. The aqueous phase was collected and dried into a solid. Gross alpha activity of the so-prepared sample was conducted on ZnS(Ag) plastic scintillation detector [20].

4.13. ADDITIONAL RADIONUCIDES IN TEPCO'S SOURCE TERM

For three radionuclides, ^{241}Pu, ^{147}Pm and ^{151}Sm, TEPCO reported results that have been calculated from measurements of the activity concentrations of other "reference" radionuclides ($C_{reference}$) in the source term and scaled using the ratio of the FDNPS reactor inventory estimates $I_{measureed}$ and $I_{reference}$ for the two radionuclides:

$$C_{calculated} = C_{reference} \frac{I_{calculated}}{I_{reference}}$$

^{241}Pu was calculated from measurements of ^{238}Pu, and both ^{147}Pm and ^{151}Sm from ^{154}Eu. The inventory activities and reference radionuclides are listed in Table I-7 of TEPCO's REIA [4]. The methodology to estimate the inventory, part of the characterisation of the ALPS treated water source that has been undertaken by TEPCO, is also described in detail the REIA. A summary is provided in an earlier IAEA report [21].

5. STATISTICAL EVALUATION OF THE RESULTS

The IAEA collected and evaluated the results reported by all laboratories participating in the ILC.

If two or three measurement results above the detection limit were received, then one or three zeta tests [2] were performed. The zeta $\zeta_{i,j}$ test is defined as:

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

where:

x_i is the value of laboratory i (Bq/L);
x_j is the value of laboratory j (Bq/L);
u_i is the standard uncertainty for the value of laboratory i (Bq/L); and
u_j is the standard uncertainty for the value of laboratory j (Bq/L).

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 [22], this ζ score is interpreted as follows:

A ζ score between -2 and 2 indicates that the reported results agree 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 results are discrepant 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 results were evaluated as agreeing at a 99.7% confidence level. (Therefore, results corresponding to "warning signals" were considered to be in agreement.) For ζ scores greater than 3 or less than -3, the corresponding results were evaluated as being discrepant at the same confidence level.

For radionuclides for which results could not be compared quantitatively (i.e. those for which activity concentrations above detection limits were reported by just one laboratory), the detection limits reported by participating laboratories were qualitatively compared to evaluate whether the analytical methods used by TEPCO were broadly equivalent and thus appropriate and fit for purpose.

Prior to publication, the compiled results for each laboratory were shared, separately, with all participating laboratories to ensure that their reported results were compiled accurately. Any discrepancies (significantly different results) identified by the statistical evaluation were carefully verified.

6. RESULTS

6.1. GENERAL

The results of the ILC are presented in this section. All reported results are presented in Section 6.2. ζ (Zeta) scores are presented in Section 6.3. Charts showing the activity concentrations and detection limits reported for key radionuclides are presented in Appendices II and III (Figures 2 to 23).

6.1.1. Uncertainties, confidence intervals and rounding

In this report, each measurement result is stated in the format $\mathrm{x} \pm \mathrm{y}$, where x is the activity concentration and y is the numerical value of the combined standard uncertainty, i.e. with a coverage factor of $k = 1$. In cases where an activity was not detected, the detection limit z of the analytical method used is reported in the format $< z$.

Reported activity concentrations are stated with a number of decimal places based on the first two significant figures of the associated uncertainty. Detection limits are also reported to two significant places. Both uncertainties and detection limits are also rounded up. Activity concentrations are rounded up or down according to normal rules for rounding [23].

6.1.2. Reference date

All activity concentrations for were reported at a reference date of 19 October 2022.

6.2. RESULTS REPORTED

TABLE 4. ACTIVITY CONCENTRATIONS (Bq/L) IN G4S-B10 ALPS TREATED WATER

Nuclide	IAEA	KINS	TEPCO
^{3}H	476400 ± 2400	504000 ± 17000	447000 ± 20000
^{14}C	-	15.4 ± 1.3	13.4 ± 1.4
^{54}Mn	<0.069	<0.16	<0.028
^{55}Fe	<0.19	<0.64	<15
^{60}Co	0.412 ± 0.013	0.412 ± 0.044	0.408 ± 0.041
^{63}Ni	5.60 ± 0.24	4.64 ± 0.32	<9.1
^{79}Se	-	-	<0.86
^{90}Sr[1]	2.24 ± 0.20	2.49 ± 0.21	2.472 ± 0.055
^{99}Tc	0.0368 ± 0.0087	<0.068	<0.44
^{106}Ru	<0.63	<1.4	<0.29
^{125}Sb	0.120 ± 0.018	<0.44	0.246 ± 0.045
^{129}I	0.219 ± 0.050	0.165 ± 0.016	0.247 ± 0.062
^{134}Cs	0.0292 ± 0.0059	<0.15	<0.035
^{137}Cs	0.918 ± 0.030	0.921 ± 0.052	0.956 ± 0.089
^{144}Ce	<0.60	<1.4	<0.36
^{147}Pm	-	-	<0.33
^{151}Sm	-	-	<0.013
^{154}Eu	<0.11	<0.36	<0.073
^{155}Eu	<0.14	<0.51	<0.13
^{234}U	<0.00037	0.01388 ± 0.00077	<0.026
^{238}U	<0.00047	0.01439 ± 0.00079	<0.026
^{237}Np	<0.000018	<0.0000057	<0.026
^{238}Pu	<0.000050	<0.001	<0.026
^{239}Pu	<0.00040	<0.0000084	<0.026
^{240}Pu		<0.0000028	<0.026
^{241}Pu	-	<0.0000094	<0.72
^{241}Am	0.0039 ± 0.0015	<0.78	<0.026
^{244}Cm	-	-	<0.026

Notes: [1] A value of 2.472 ± 0.055 for ^{90}Y, derived from the measured activity concentration of ^{90}Sr, was reported by TEPCO.

[2] A value of 0.057 ± 0.011 for ^{125m}Te derived from the measured activity concentration of ^{125}Sb, was reported by TEPCO.

[3] A combined value for ^{239,240}Pu was reported by IAEA.

TABLE 5. ACTIVITY CONCENTRATIONS (Bq/L) IN G4S-C8 ALPS TREATED WATER

Nuclide	IAEA	KINS	TEPCO
^{3}H	394400 ± 1100	419000 ± 14000	365000 ± 16000
^{14}C	-	9.7 ± 1.2	9.9 ± 1.1
^{54}Mn	<0.056	<0.15	<0.034
^{55}Fe	<0.019	<0.79	<15
^{60}Co	0.1503 ± 0.0077	0.154 ± 0.018	0.162 ± 0.020
^{63}Ni	4.51 ± 0.19	4.06 ± 0.35	<8.2
^{79}Se	-	-	<0.86
^{90}Sr^{1}	0.67 ± 0.06	0.652 ± 0.070	0.72 ± 0.026
^{99}Tc	<0.016	<0.068	<0.44
^{106}Ru	<0.51	<1.3	<0.26
^{125}Sb	0.198 ± 0.016	<0.45	0.211 ± 0.036
^{129}I	<0.18	<0.049	0.038 ± 0.018
^{134}Cs	<0.049	<0.14	<0.036
^{137}Cs	<0.039	<0.13	<0.035
^{144}Ce	<0.18	<1.4	<0.36
^{147}Pm	-	-	<0.30
^{151}Sm	-	-	<0.012
^{154}Eu	<0.083	<0.32	<0.067
^{155}Eu	<0.12	<0.51	<0.12
^{234}U	<0.00030	<0.0010	<0.026
^{238}U	<0.00040	<0.00075	<0.026
^{237}Np	<0.000056	<0.0000056	<0.026
^{238}Pu	0.00028 ± 0.00016	<0.0025	<0.026
^{239}Pu	0.00140 ± 0.00036	<0.0000087	<0.026
^{240}Pu		<0.0000029	<0.026
^{241}Pu	-	<0.000019	<0.72
^{241}Am	0.00176 ± 0.00051	<0.81	<0.026
^{244}Cm	-	-	<0.026

Notes: [1] A value of 0.72 ± 0.026 for ^{90}Y, derived from the measured activity concentration of ^{90}Sr, was reported by TEPCO.
[2] A value of 0.0487 ± 0.0083 for ^{125m}Te derived from the measured activity concentration of ^{125}Sb, was reported by TEPCO.
[3] A combined value for ^{239,240}Pu was reported by IAEA.

6.3. ζ (ZETA) SCORES

TABLE 6. ZETA SCORES FOR G4S-B10 ALPS TREATED WATER SAMPLES

Nuclide	$\zeta_{IAEA,KINS}$	$\zeta_{IAEA,TEPCO}$	$\zeta_{KINS,TEPCO}$
^{3}H	-1.70	1.54	2.29
^{14}C	-	-	1.11
^{60}Co	0.01	0.09	0.06
^{63}Ni	2.46	-	-
^{90}Sr	-0.88	-1.13	0.09
^{125}Sb	-	-2.65	-
^{129}I	1.04	-0.35	-1.28
^{137}Cs	-0.06	-0.41	-0.34

TABLE 7. ZETA SCORES FOR G4S-C8 ALPS TREATED WATER SAMPLES

Nuclide	$\zeta_{IAEA,KINS}$	$\zeta_{IAEA,TEPCO}$	$\zeta_{KINS,TEPCO}$
^{3}H	-1.86	1.90	2.66
^{14}C	-	-	-0.16
^{60}Co	-0.20	-0.58	-0.31
^{63}Ni	1.14	-	-
^{90}Sr	0.24	-0.71	-0.92
^{125}Sb	-	-0.34	-

Note: Evaluation criteria (99.7% confidence level): ζ score between -3 and 3: corresponding results evaluated as agreeing; ζ score greater than 3 or less than -3: corresponding results evaluated as discrepant.

7. CONCLUSION

This ILC has been implemented to check the capability of TEPCO to undertake analyses relevant to source monitoring with respect to discharges of ALPS treated water to the required standard and to report high quality and comparable results. Samples collected from the G4S-B10 and G4S-C8 tanks at FDNPS have been analysed for radionuclide activity concentrations by TEPCO, the IAEA laboratories and a third-party laboratory, the Korean Institute of Nuclear Safety (KINS), from the IAEA ALMERA network.

7.1. QUANTITATIVE INTERCOMPARISON OF REPORTED ACTIVITY CONCENTRATIONS

The main focus of the ILC was on the radionuclides included in TEPCO's ALPS treated water source term [4] (Table 1). For 24 of these radionuclides (^{3}H, ^{14}C, ^{54}Mn, ^{55}Fe, ^{60}Co, ^{63}Ni, ^{90}Sr, ^{99}Tc, ^{106}Ru, ^{125}Sb, ^{129}I, ^{134}Cs, ^{137}Cs, ^{144}Ce, ^{154}Eu, ^{155}Eu, ^{234}U, ^{238}U, ^{237}Np, ^{238}Pu, ^{239}Pu, ^{240}Pu, ^{241}Pu and ^{241}Am), results were reported by the IAEA laboratories or KINS (or both) in addition to TEPCO. Results for ^{90}Y and ^{125m}Te, which are also included in TEPCO's source term, can be derived from the results for ^{90}Sr and ^{125}Sb, respectively, assuming equilibrium. Results were not reported by either the IAEA laboratories or KINS for four radionuclides: ^{75}Se, ^{147}Pm, ^{151}Sm and ^{244}Cm.

For the samples from the G4S-B10 tank, activity concentrations above detection limits were reported by at least two laboratories for 9 radionuclides: ^{3}H, ^{14}C, ^{60}Co, ^{63}Ni, ^{90}Sr, ^{125}Sb, ^{129}I and ^{137}Cs. For the samples from the G4S-C8 tank, the number of radionuclides was six: ^{3}H, ^{14}C, ^{60}Co, ^{63}Ni, ^{90}Sr and ^{125}Sb. The reported results for these radionuclides have been subjected to the statistical evaluation described in Section 5. Out of a total of the 29 reported results that were assessed using these statistical tests, all (100%), were evaluated as agreeing with a high level of confidence (99.7%). This provides evidence of the high level of competence of all participating laboratories.

A small number of "warning signals"[3] were identified (Section 6.3). This was not unsurprising given the strict statistical tests applied. Even in high performing laboratories, run by knowledgeable and experienced staff, anomalous results may sometimes be obtained. Furthermore, ALPS treated water constitutes a complex, highly processed sample matrix that has been subjected to a series of treatments at FDNPS to lower the radionuclide content, including co-precipitation, physical filtration and adsorption. Despite the measures taken to ensure inter-sample homogeneity (Section 3) some degree of heterogeneity may have persisted. Additionally, radionuclides, other than ^{3}H, were present in the samples at relatively low levels and the participating laboratories were required to use a range of often complex analytical techniques, both resulting in generally higher uncertainties.

7.2. ASSESSMENT OF REPORTED DETECTION LIMITS

For other radionuclides, the detection levels reported by participating laboratories were compared qualitatively. TEPCO's detection limits were in most cases comparable to those reported by the other participating laboratories. Exceptions were ^{55}Fe and ^{63}Ni and the actinides (^{234}U, ^{238}U, ^{237}Np, ^{238}Pu, ^{239}Pu and ^{241}Am) for which the detection limits were generally significantly higher than those reported by the other participating laboratories.

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[3] As defined in section 5.

A comparison of all of the detection limits reported by TEPCO with respective regulatory limits for all radionuclides in its source term showed that for each radionuclide analysed the reported detection limit was less than 1% of the respective regulatory limit in all cases. This demonstrates that the methods selected are appropriate and fit for purpose.

TEPCO assesses actinides included in its source term using a screening approach based on gross alpha counting. The gross alpha result – in this case a detection limit of 0.031 Bq/L – is taken as an upper bound of the activity concentration of each individual actinide included in the source term. Contaminated and ALPS treated water samples collected at FDNPS have been characterised for alpha-emitting radionuclides using radionuclide specific analytical methods in the past. The resulting activity concentrations were <1% of the regulatory limit in all cases. As the screening approach is sufficiently conservative, is implemented to a high quality, and a procedure is in place describing steps to be taken for effective follow up in the event that a pre-stated action limit is exceeded, this approach is considered to be fit for purpose.

It is known, however, that the chemical recovery of the gross alpha method used is determined from a single alpha-emitting radionuclide (^{241}Am). Investigation of the chemical recovery for other targeted radionuclides is recommended to further verify the robustness of this method.

7.3. KEY FINDINGS

The results of this ILC demonstrate a high level of accuracy and competence on the part of TEPCO. This should provide confidence in TEPCO’s capability for undertaking accurate and precise source monitoring related to the discharge of ALPS treated water.

TEPCO's sample collection procedures follow the appropriate methodological standards required to obtain representative samples. This is demonstrated by the generally high degree of comparability in the results reported by all participating laboratories.

The detection limits reported by TEPCO indicate that analytical methods selected were appropriate and fit for purpose. The reported detection limits were less than 1% of the respective regulatory limits for all radionuclides included in TEPCO’s source term.

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APPENDIX I: INSTRUCTIONS SENT TO PARTICIPATING LABORATORIES

Participating laboratories were requested to submit, for each sample, a single measurement result for each radionuclide analysed, decay corrected to the sampling date (19 October 2022). A measurement result was assumed to be comprised of an activity concentration and associated uncertainty, or the detection limit, as appropriate, all expressed in Bq/L.

Participating laboratories were asked to report the following additional information for each radionuclide for which results were to be reported, in addition to the activity concentration and uncertainty:

(a) A short description of the analytical method applied for the sample analysis.
(b) The type of calibration applied.
(c) The software used for the counting/spectrometric systems.
(d) The nuclear data used (e.g., half-life, energy, intensity of gamma emission) in the determination of the results.
(e) The detection limit and decision threshold.
(f) The uncertainty budget with components including the following, as relevant:
    - Statistical counting uncertainty, including any background subtraction.
    - Uncertainty of the detector efficiency, including the uncertainty of the calibration source, as applicable.
    - Uncertainty of chemical yields, as applicable.
    - Uncertainty of mass measurements.
    - Uncertainty of corrections applied, for instance for true coincidence summing or for decay correction.
    - Measurement repeatability uncertainty.

For analyses comprising repetitions, the laboratories were requested to inform IAEA on how the measurement result reported was determined (e.g., an average of the values from each repetition or a single value (from a single selected repetition). For values derived from an average, information was requested on how the uncertainty was estimated (e.g., weighted average, maximum value etc.).

The IAEA recommended samples to be prepared gravimetrically to minimise uncertainties. The value of the density of the sample used to convert from gravimetric to volumetric activity concentrations (i.e., from Bq/kg to Bq/L) was also requested to be reported, as necessary.

APPENDIX II: CHARTS OF ACTIVITY CONCENTRATIONS AND DETECTION LIMITS FOR THE SAMPLES FROM TANK G4S-B10

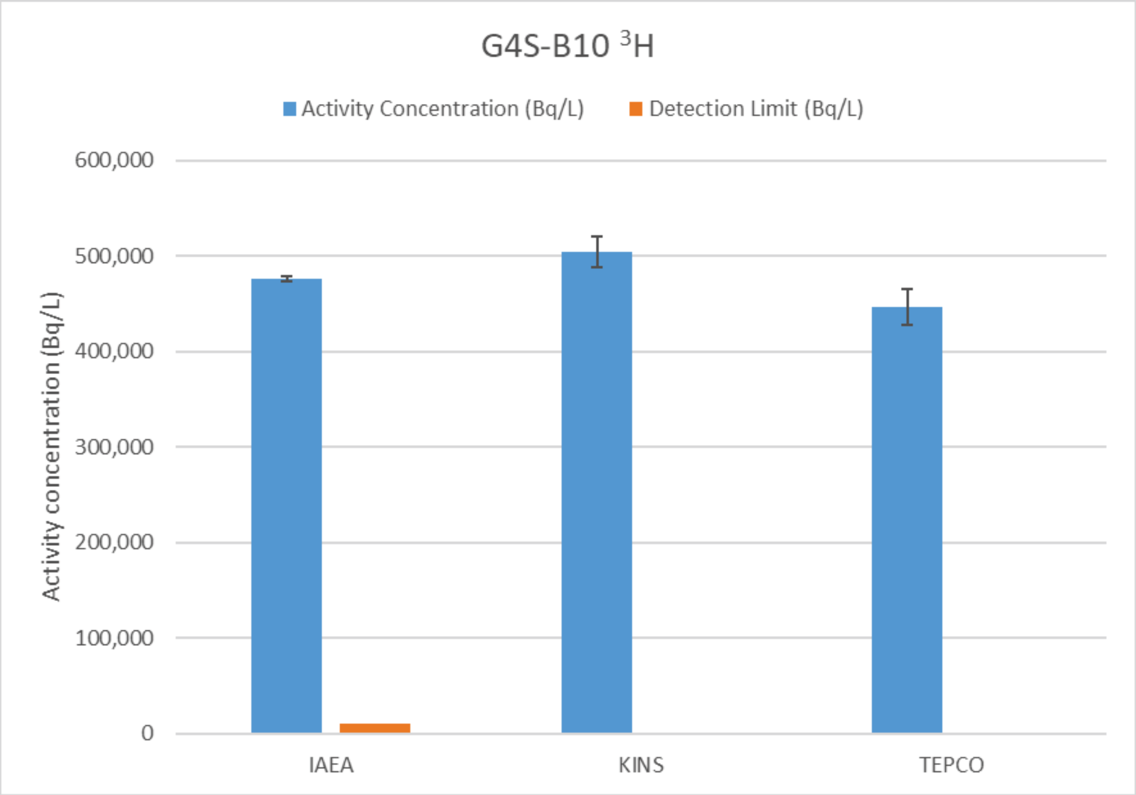

FIG. 2. Activity concentrations and detection limits for ^{3}*H for the samples from tank G4S-B10.*

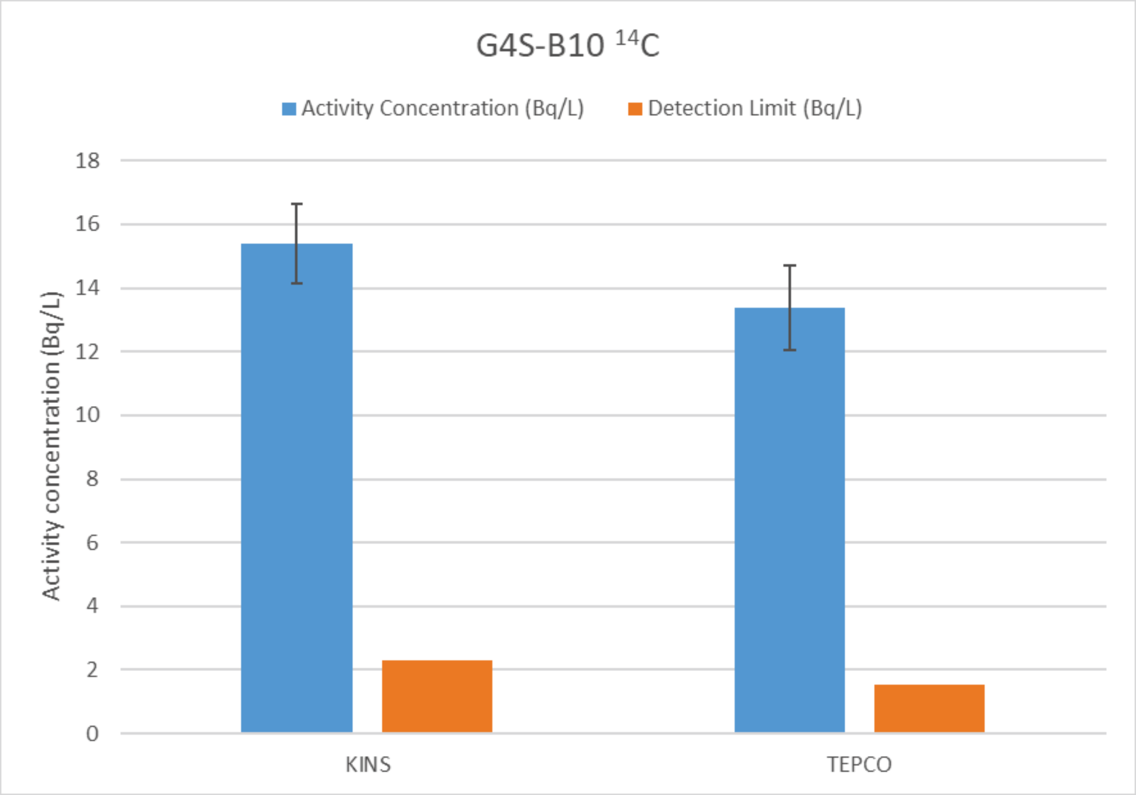

FIG. 3. Activity concentrations and detection limits for ^{14}*C for the samples from tank G4S-B10.*

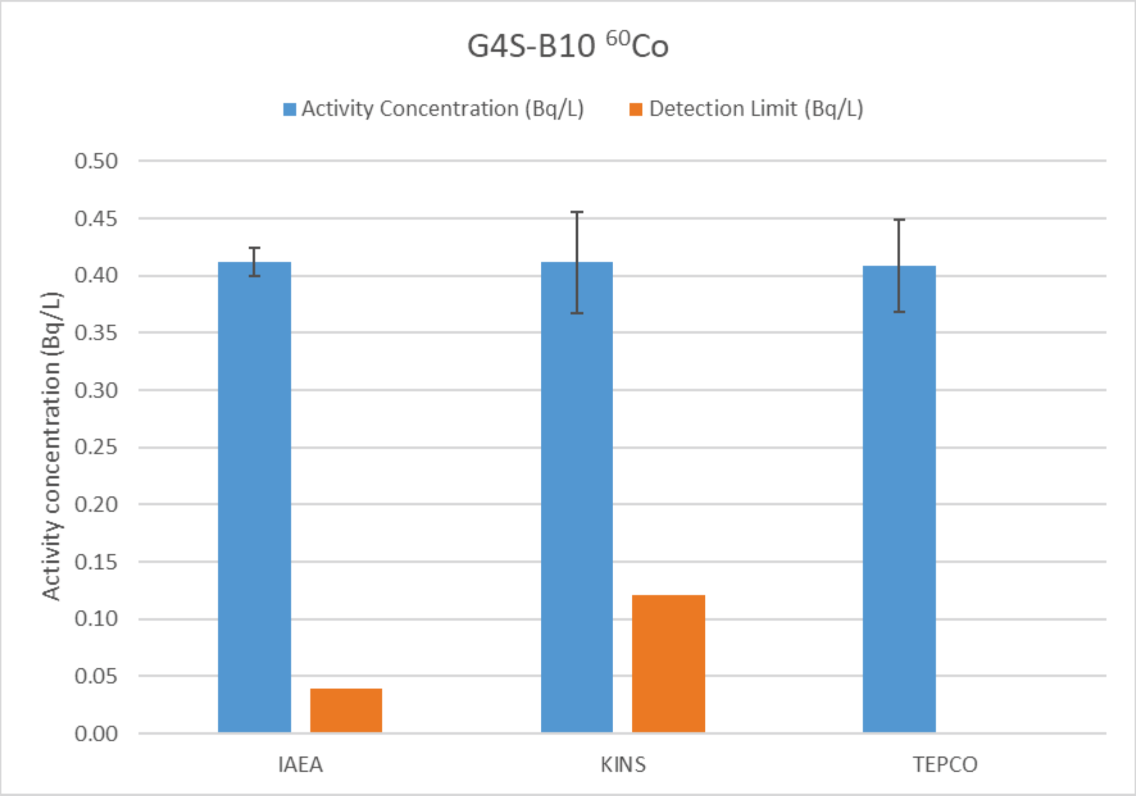

FIG. 4. Activity concentrations and detection limits for ^{60}Co for the samples from tank G4S-B10.

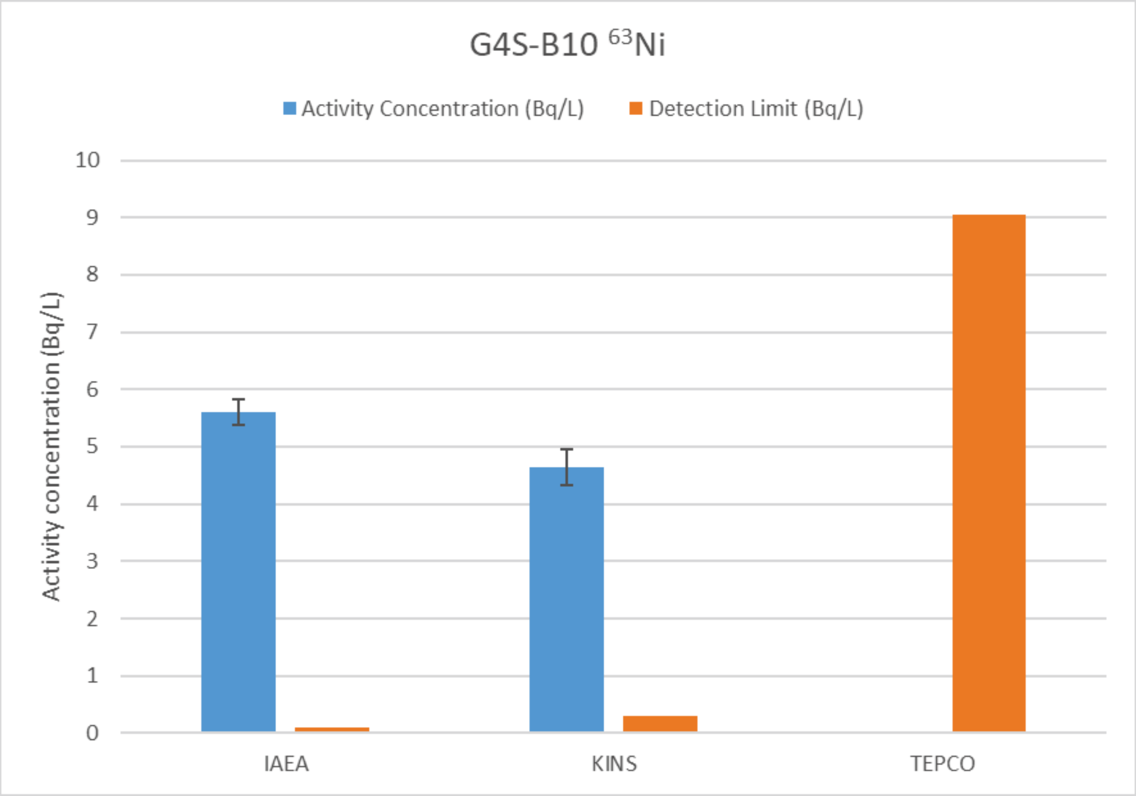

FIG. 5. Activity concentrations and detection limits for ^{63}Ni for the samples from tank G4S-B10.

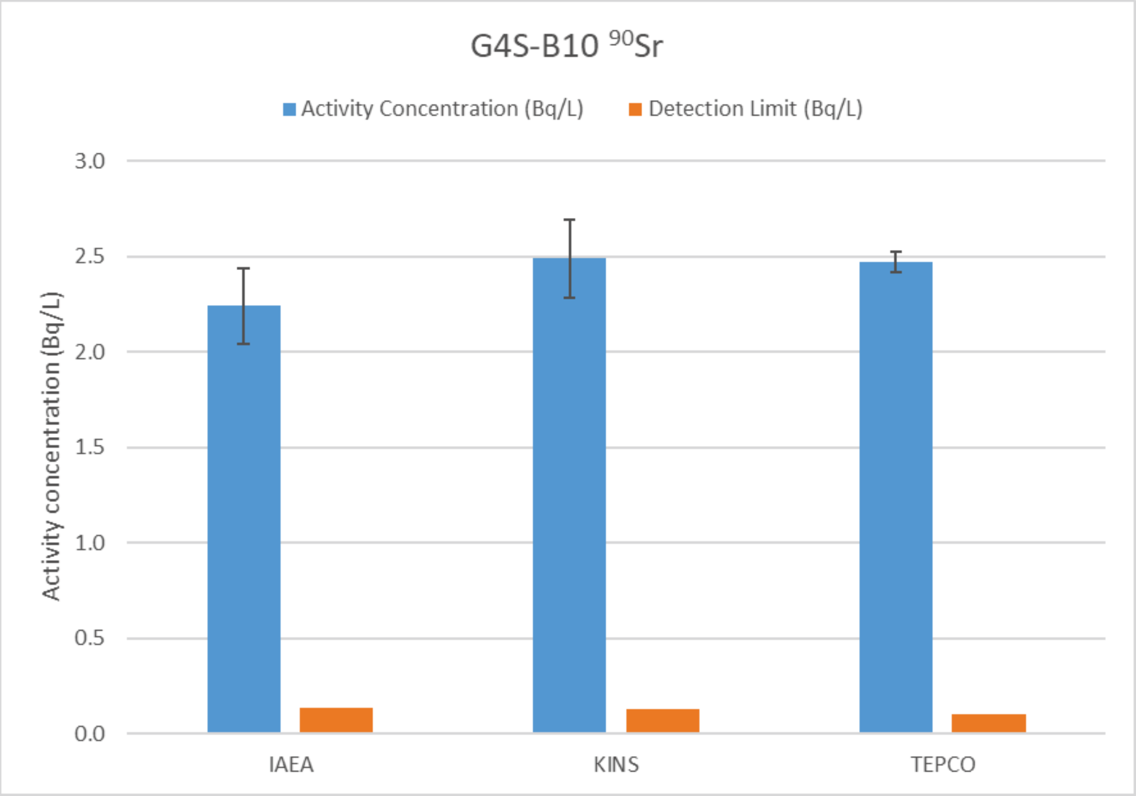

FIG. 6. Activity concentrations and detection limits for ^{90}Sr for the samples from tank G4S-B10.

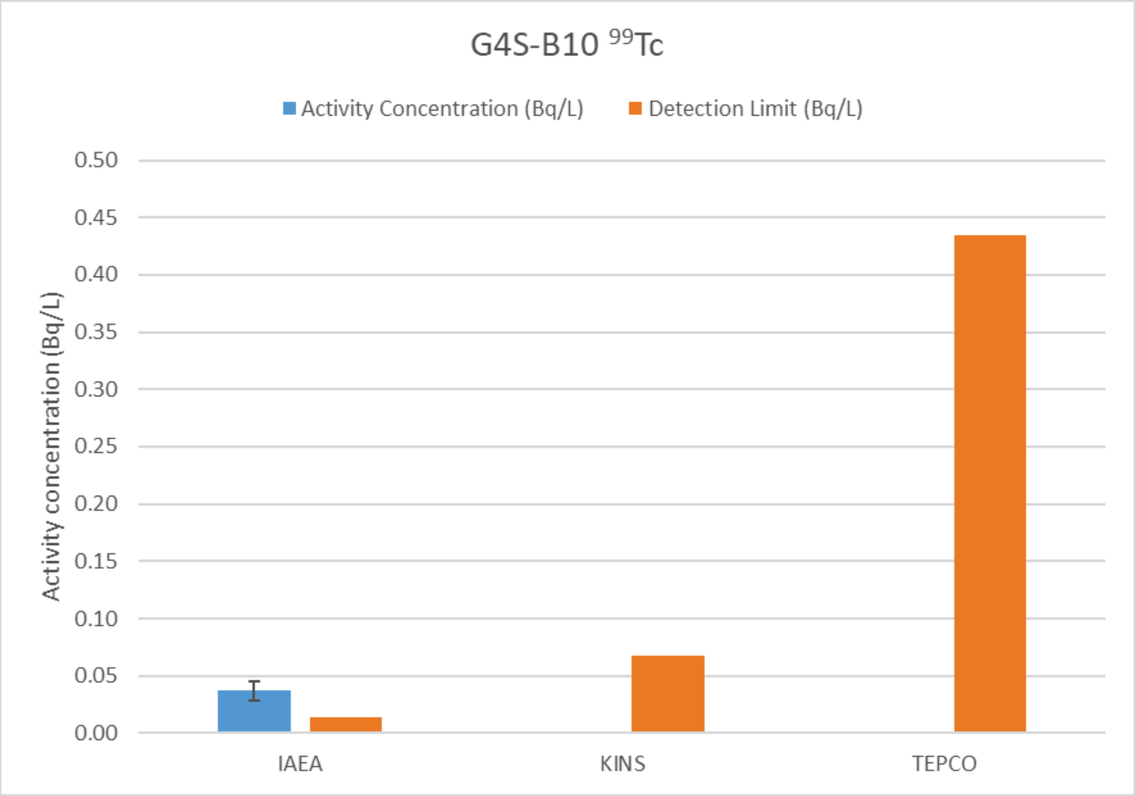

FIG. 7. Activity concentrations and detection limits for ^{99}Tc for the samples from tank G4S-B10.

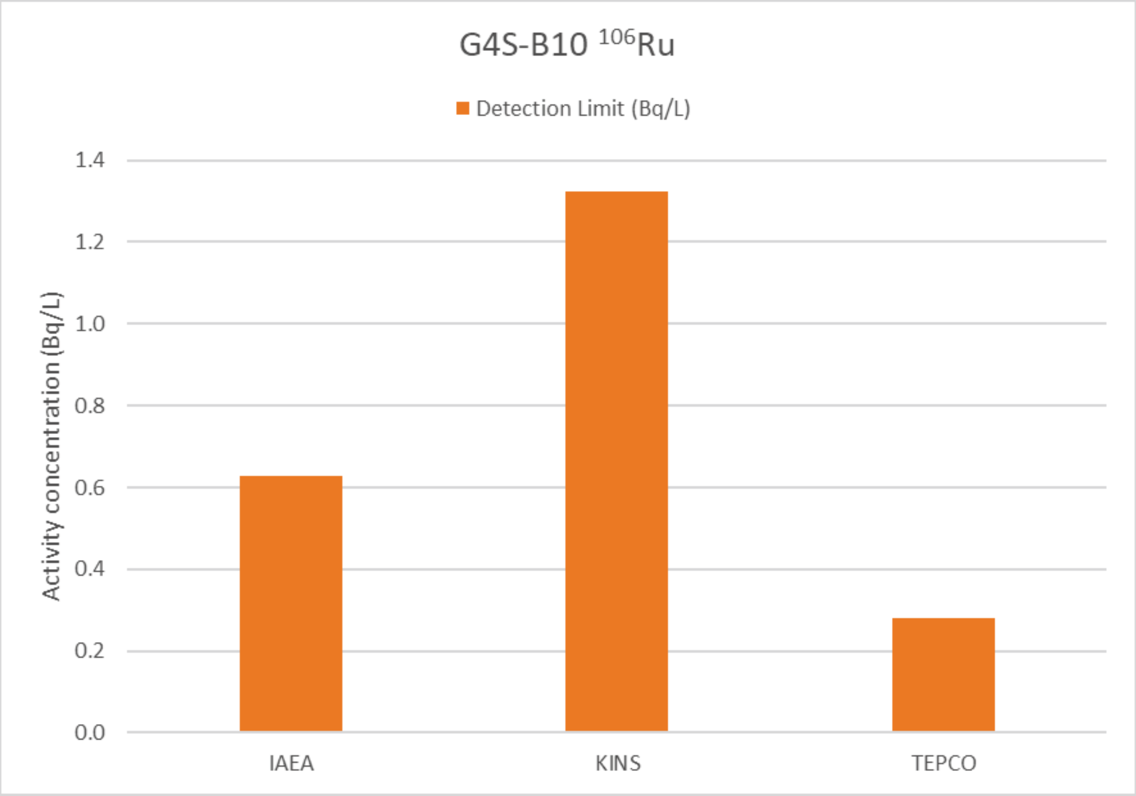

FIG. 8. Detection limits for ^{106}Ru for the samples from tank G4S-B10.

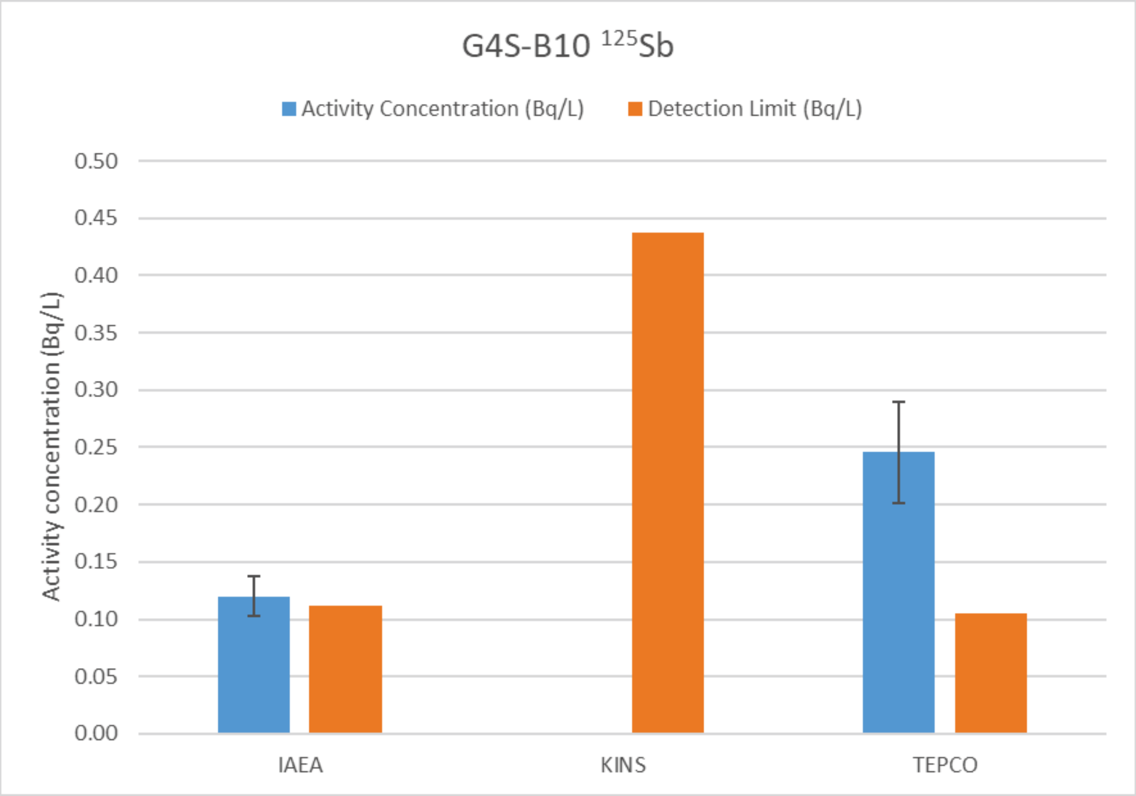

FIG. 9. Activity concentrations and detection limits for ^{125}Sb for the samples from tank G4S-B10.

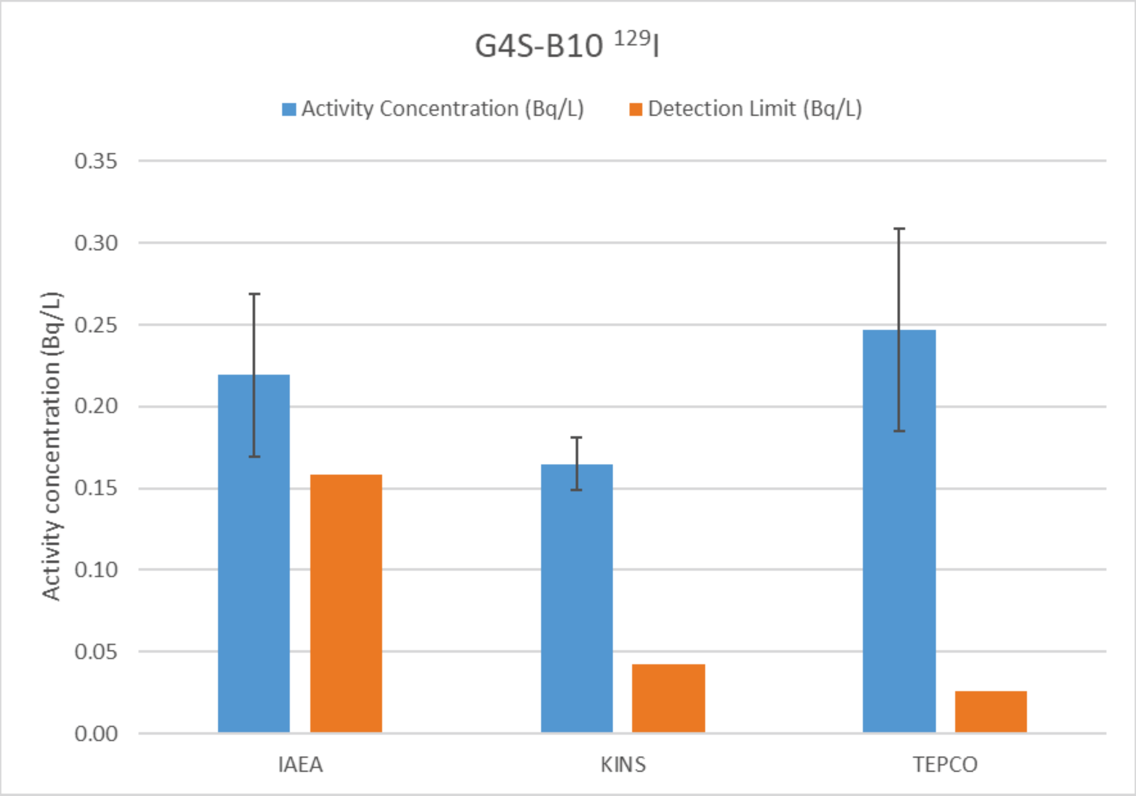

FIG. 10. Activity concentrations and detection limits for ^{129}I for the samples from tank G4S-B10.

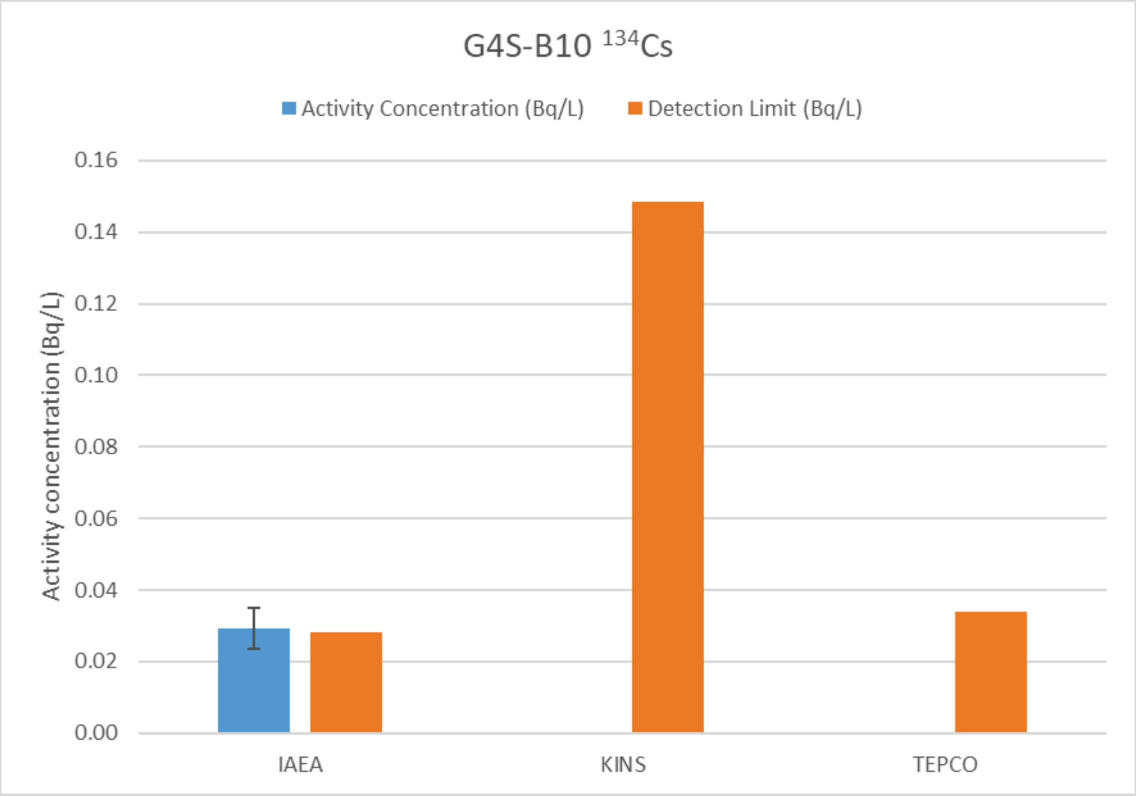

FIG. 11. Activity concentrations and detection limits for ^{134}Cs for the samples from tank G4S-B10.

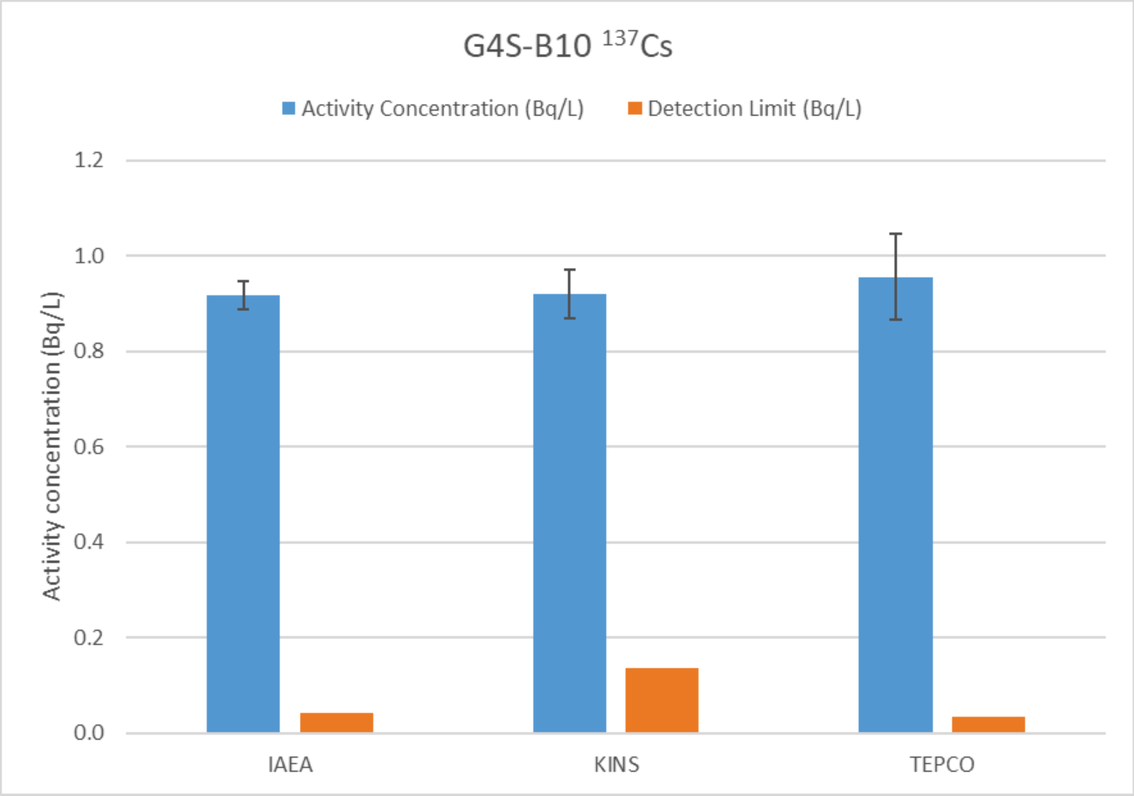

FIG. 12. Activity concentrations and detection limits for ^{137}*Cs for the samples from tank G4S-B10.*

APPENDIX III: CHARTS OF ACTIVITY CONCENTRATIONS AND DETECTION LIMITS FOR THE SAMPLES FROM TANK G4S-C8

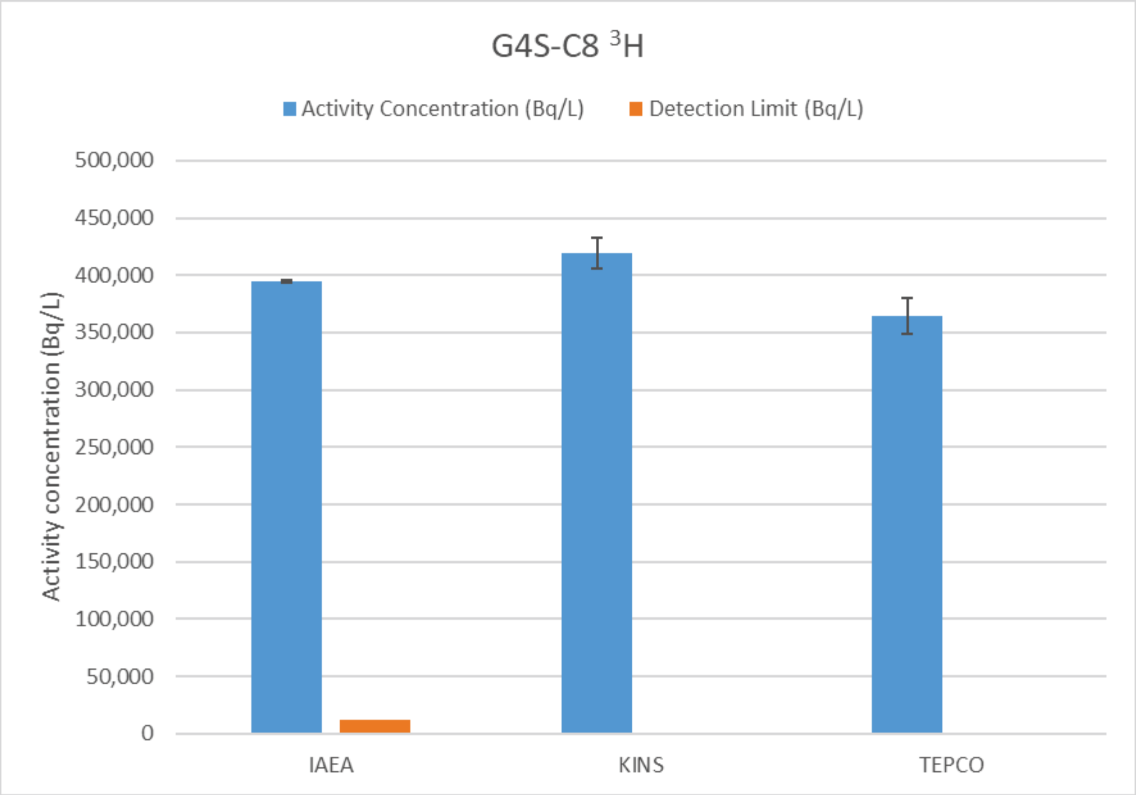

FIG. 13. Activity concentrations and detection limits for ^{3}H for the samples from tank G4S-C8.

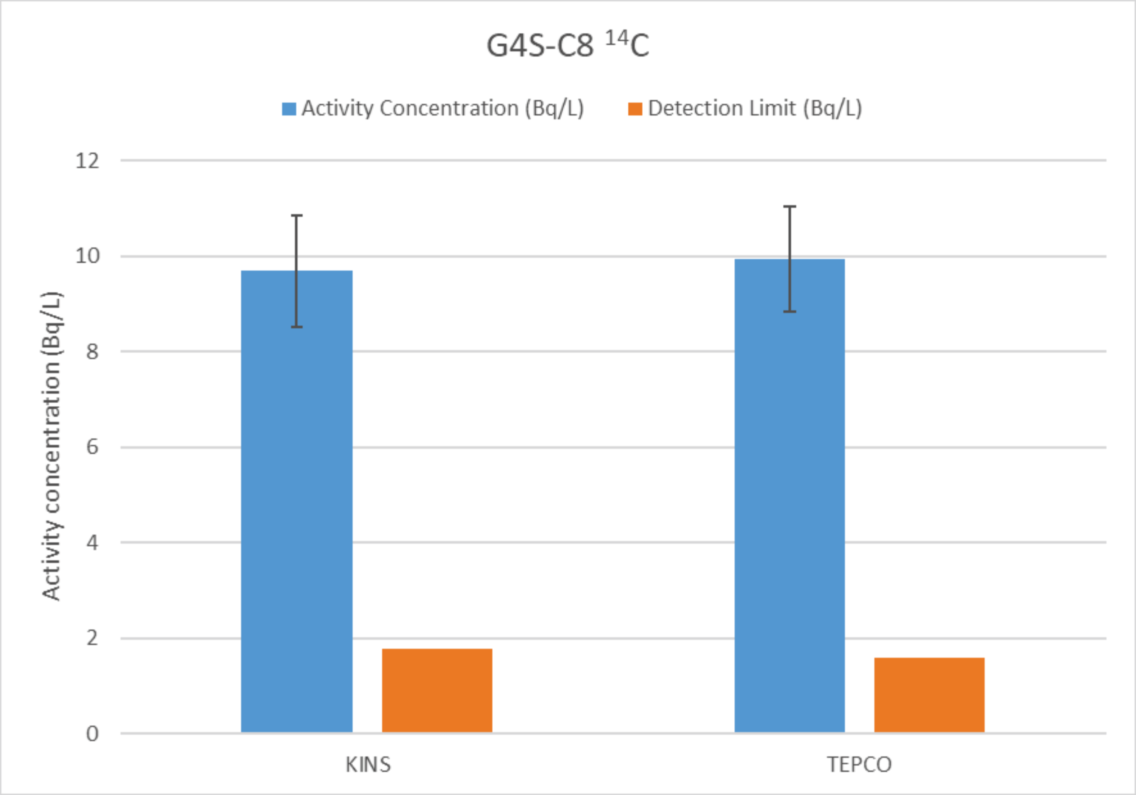

FIG. 14. Activity concentrations and detection limits for ^{14}C for the samples from tank G4S-C8.

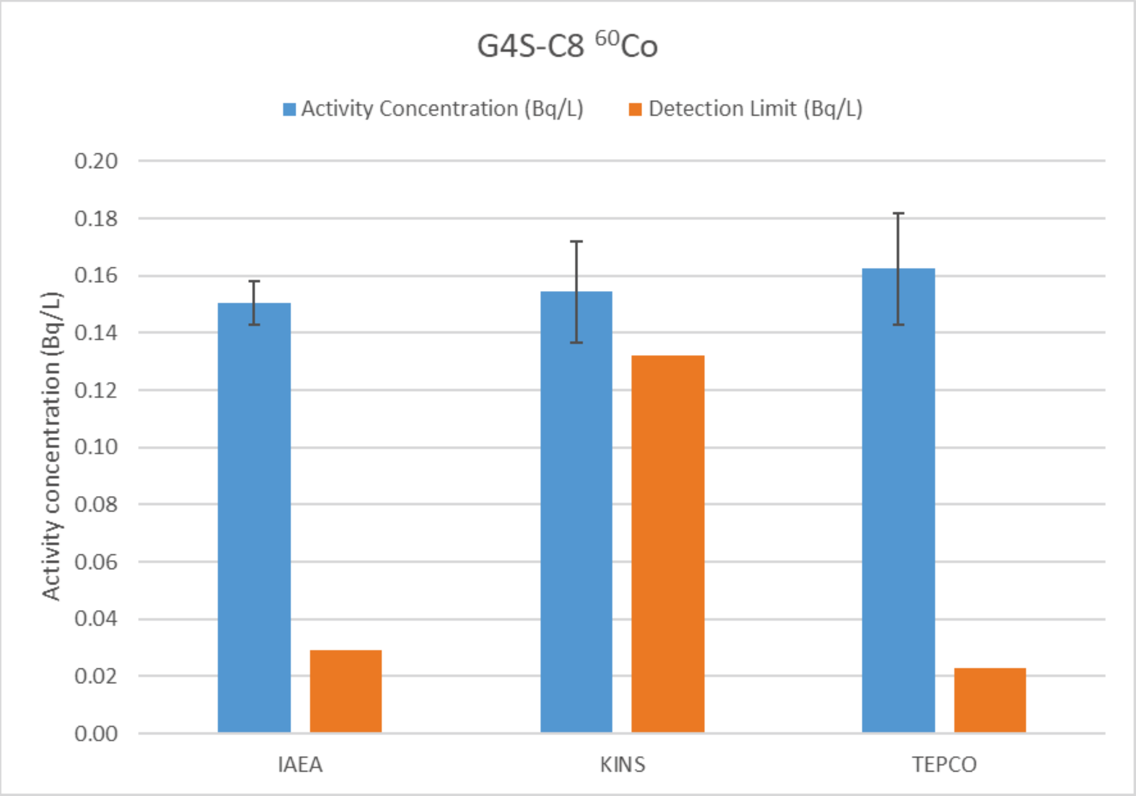

FIG. 15. Activity concentrations and detection limits for ^{60}Co for the samples from tank G4S-C8.

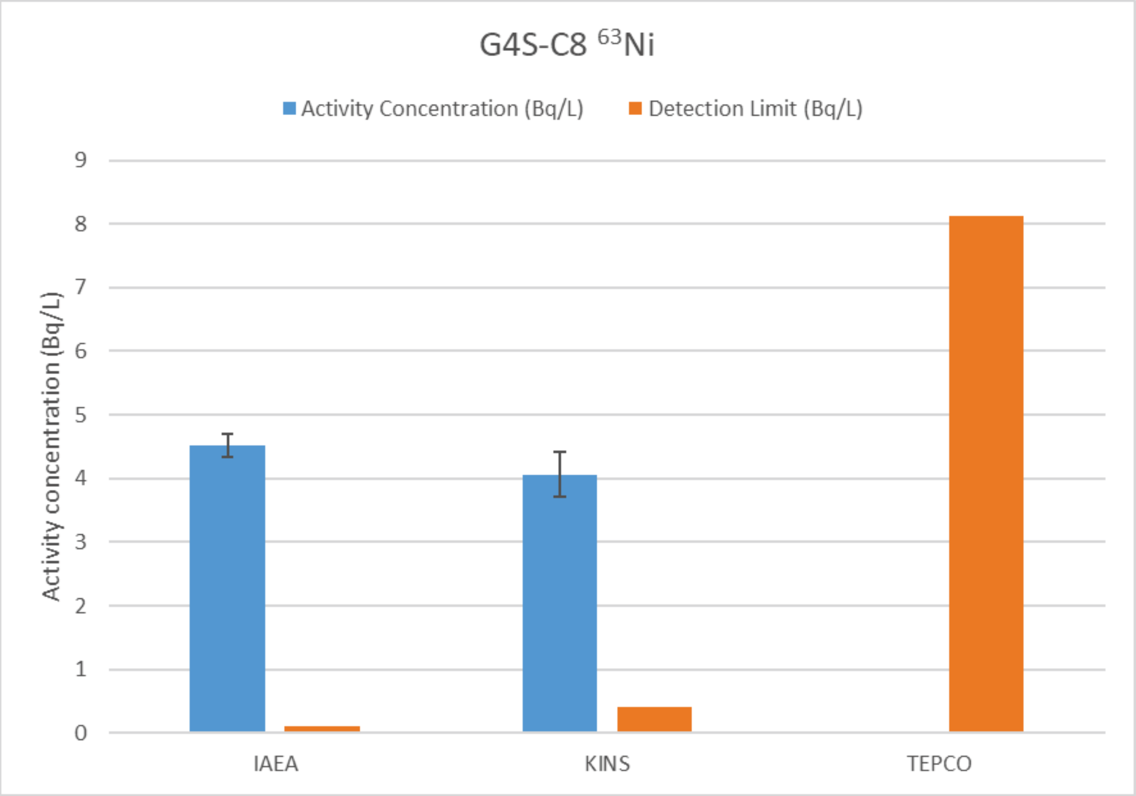

FIG. 16. Activity concentrations and detection limits for ^{63}Ni for the samples from tank G4S-C8.

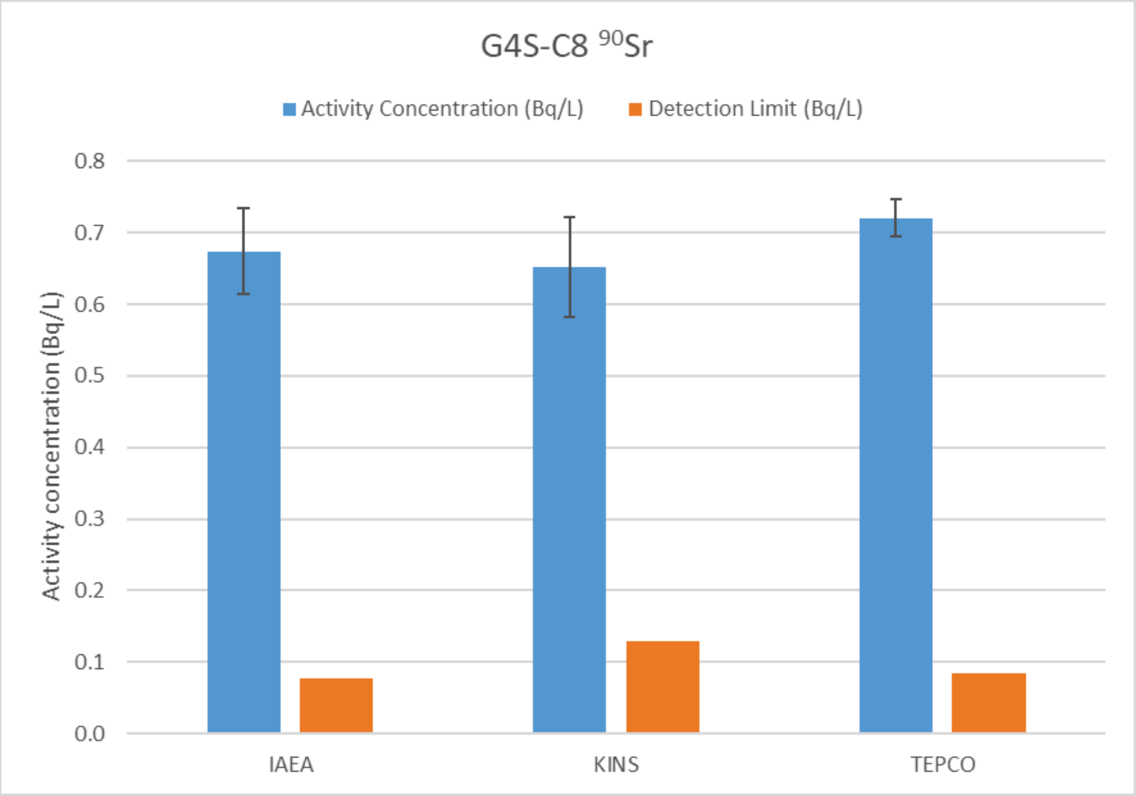

FIG. 17. *Activity concentrations and detection limits for* ^{90}*Sr for the samples from tank G4S-C8.*

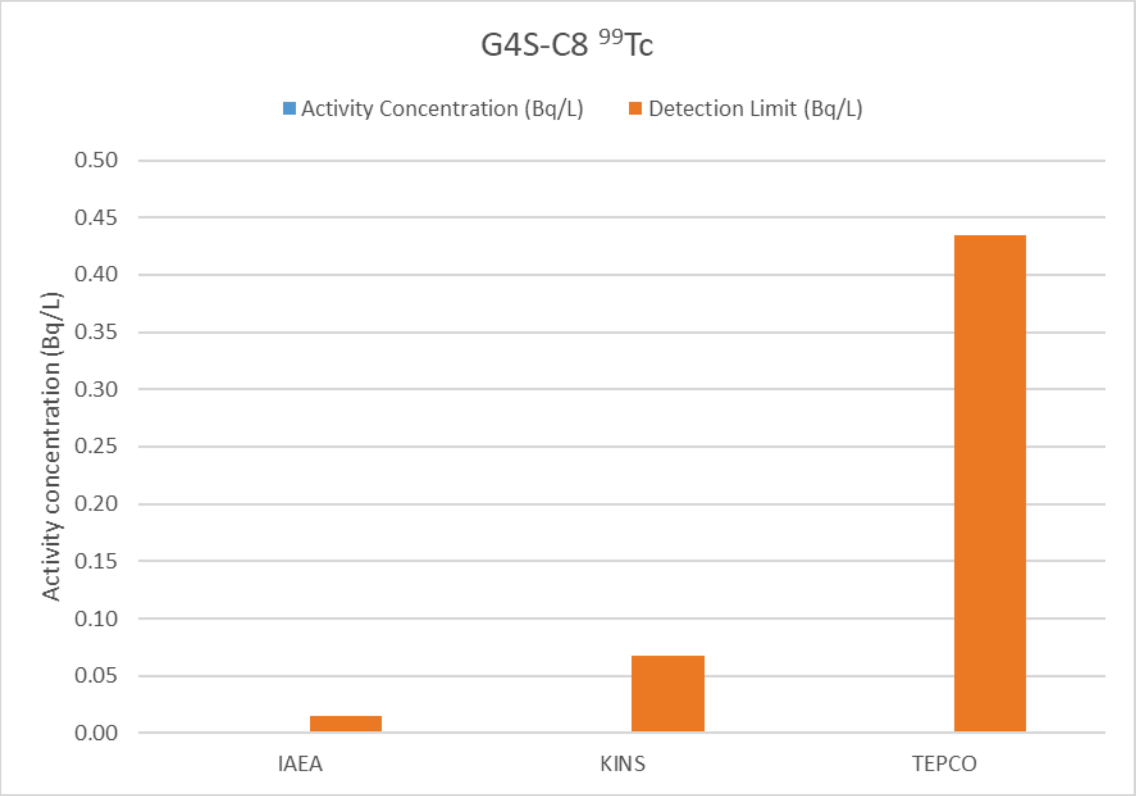

FIG. 18. *Detection limits for* ^{99}*Tc for the samples from tank G4S-C8.*

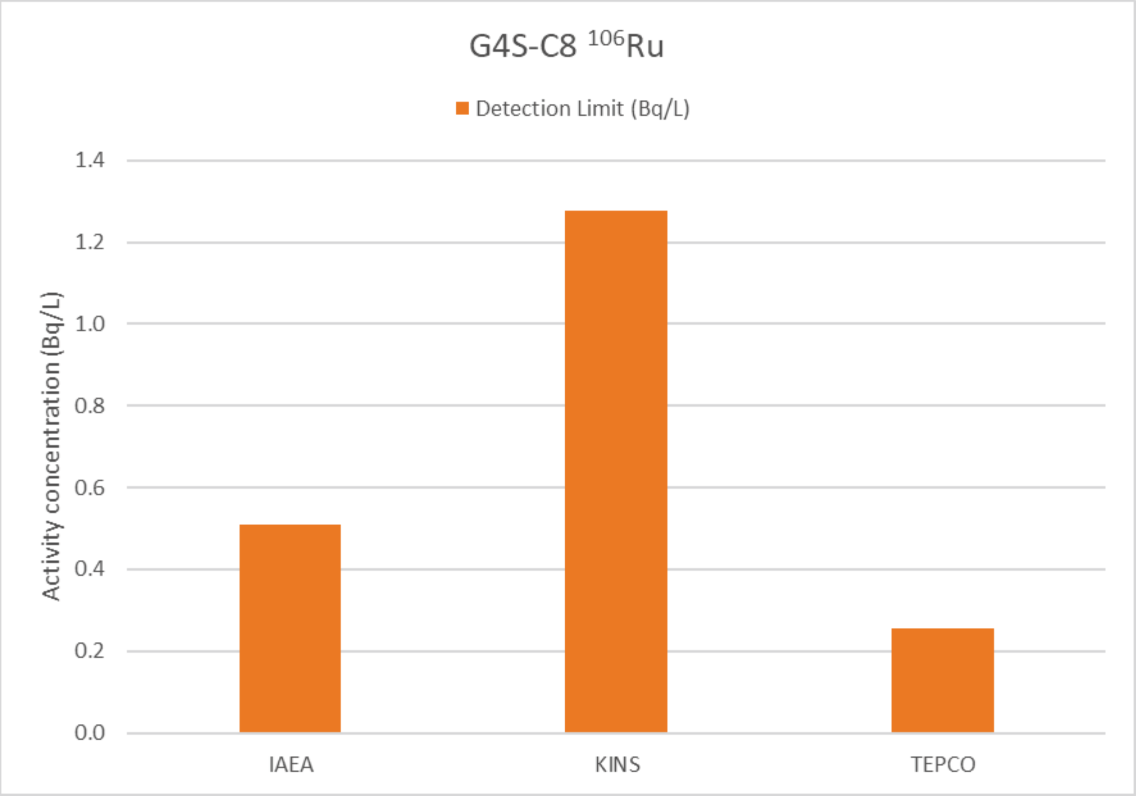

FIG. 19. Detection limits for ^{106}*Ru for the samples from tank G4S-C8.*

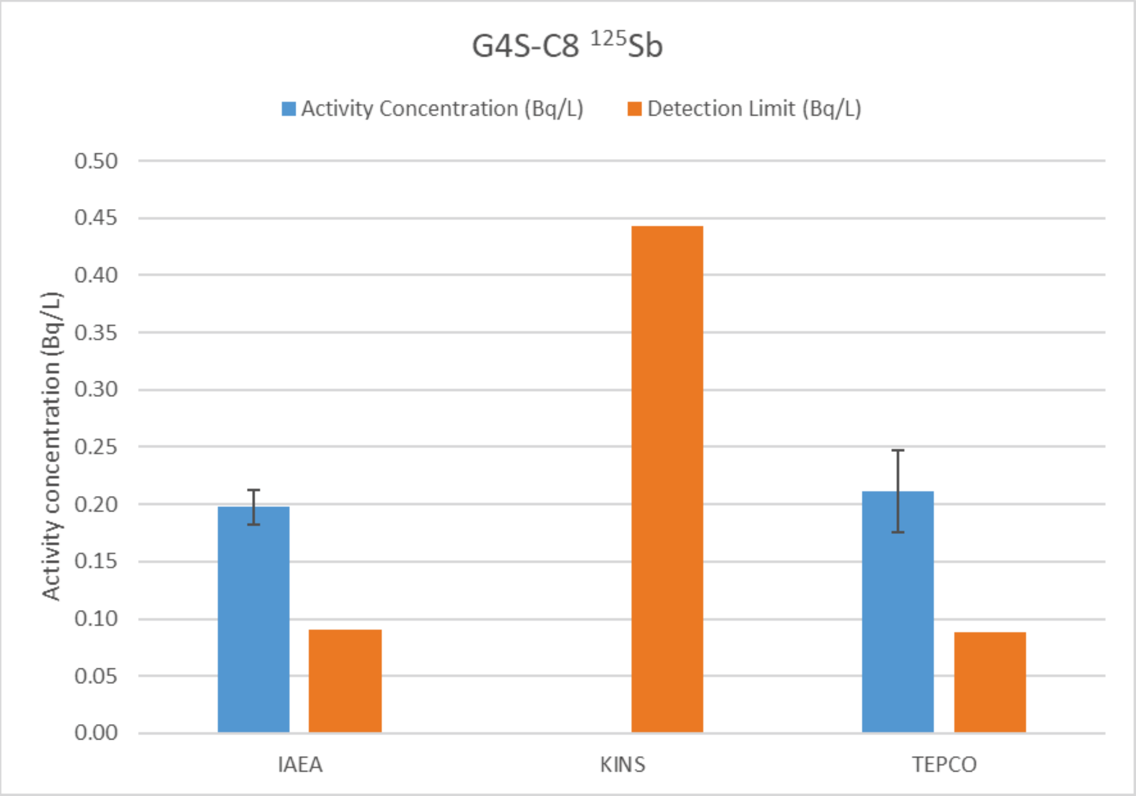

FIG. 20. Activity concentrations and detection limits for ^{125}*Sb for the samples from tank G4S-C8.*

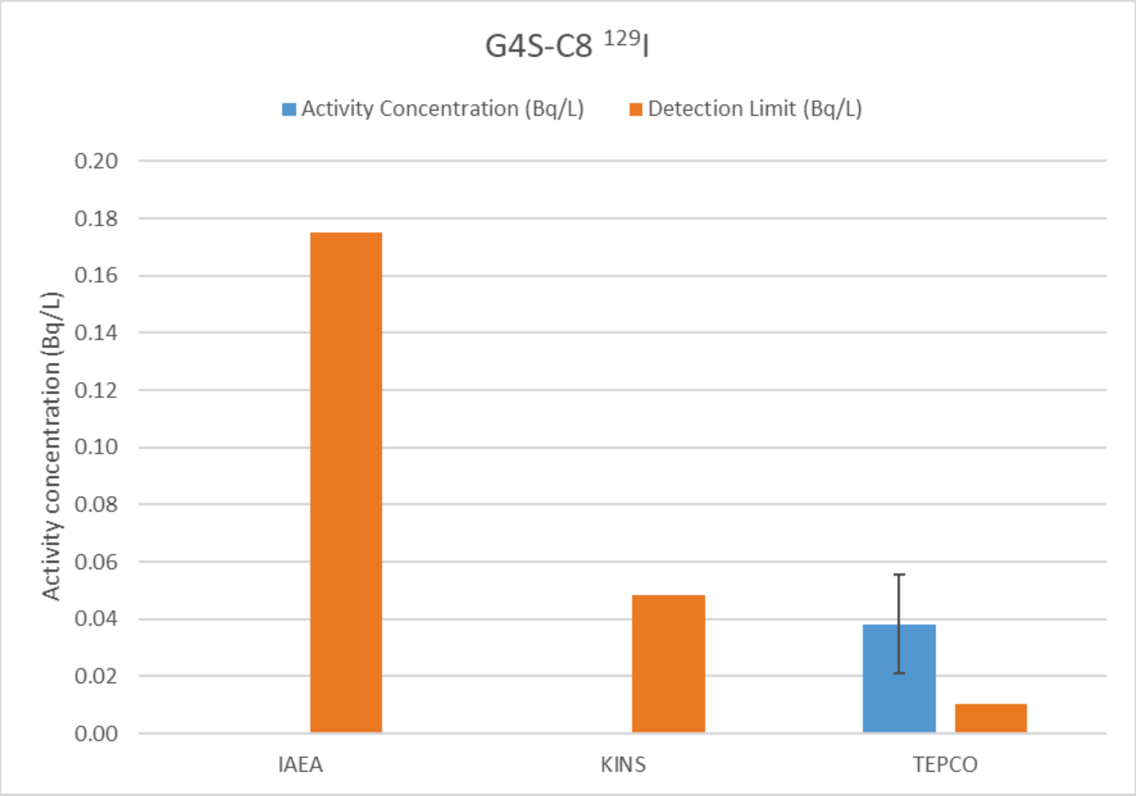

FIG. 21. Activity concentrations and detection limits for ^{129}I for the samples from tank G4S-C8.

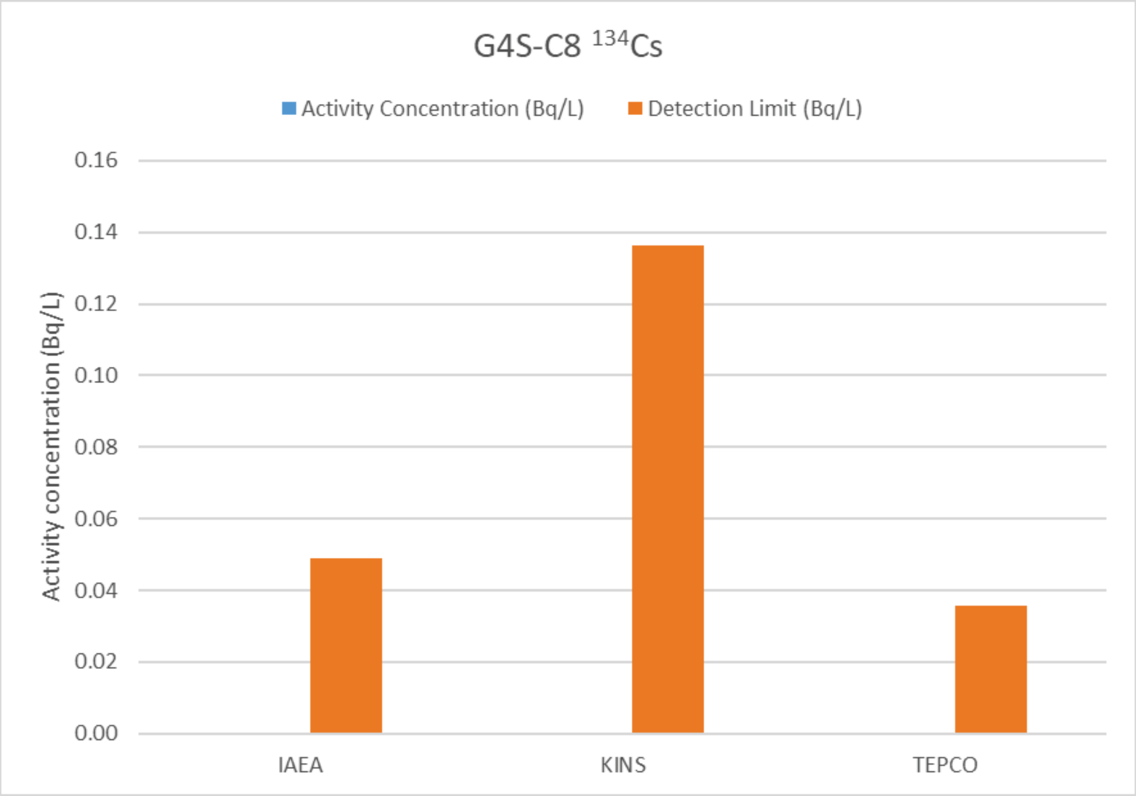

FIG. 22. Detection limits for ^{134}Cs for the samples from tank G4S-C8.

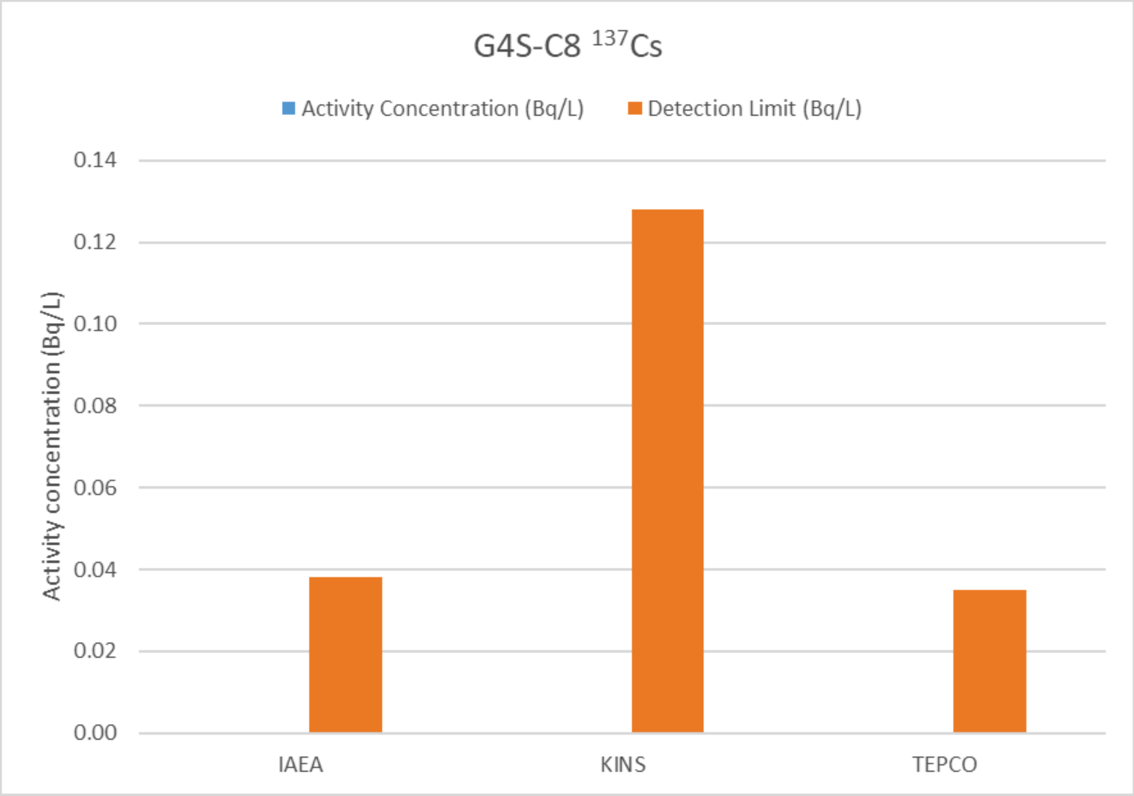

FIG. 23. Detection limits for ^{137}*Cs for the samples from tank G4S-C8.*

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