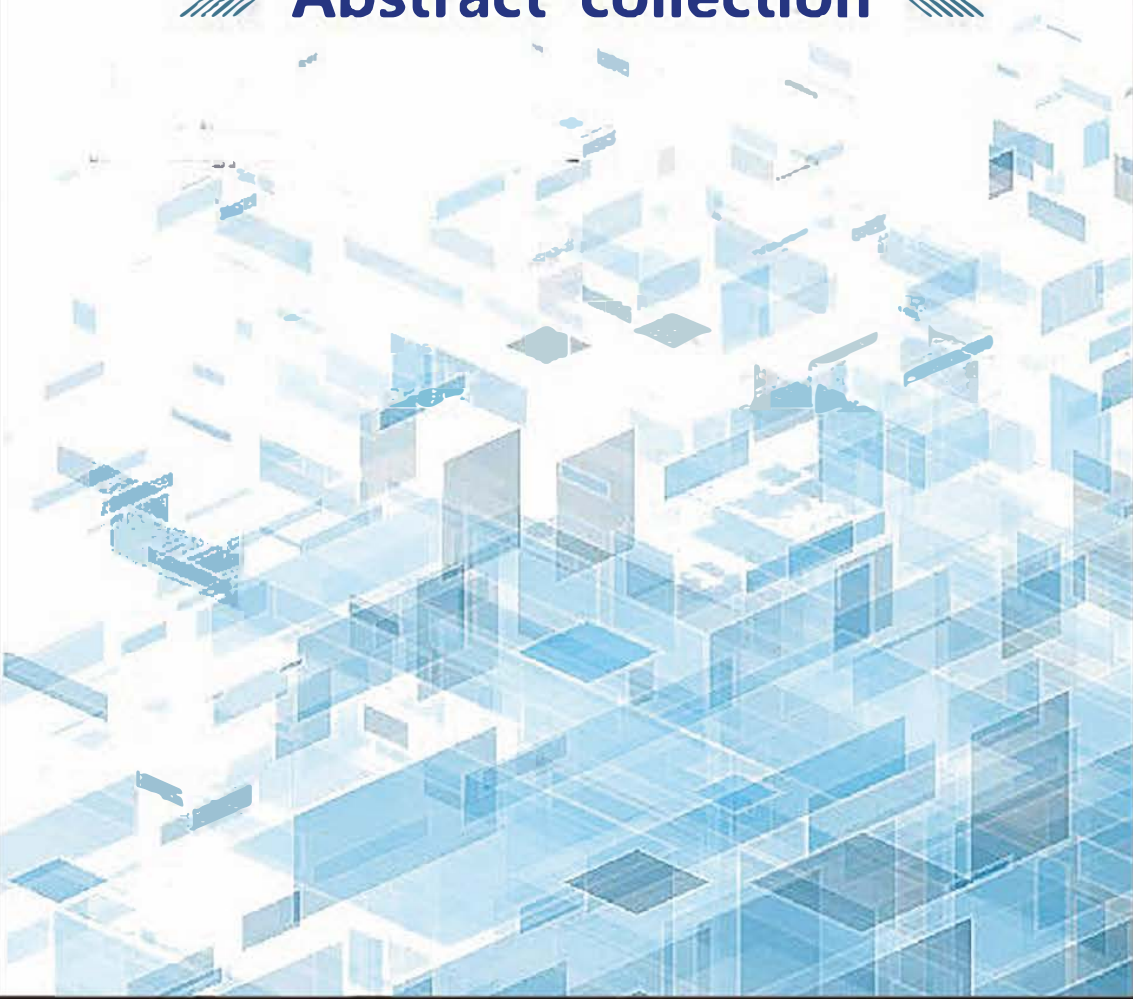


# LSC 2020

## International Conference on Advances in Liquid Scintillation Spectrometry

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»»» **Abstract collection** «««



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I01

## Ultra-trace analysis of $^3\text{H}$ in Helium-3 using Liquid Scintillation Counting

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Helium-3, the lighter isotope of the most inert element, can be widely used in fields such as neutron detection, medical imaging, and cryogenic physics<sup>1</sup>. Helium-3 fills neutron detectors at large neutron-scattering facilities used to probe materials, such as the one at the new Japan Proton Accelerator Research Complex (J-PARC) in Tokai. Rare in nature, the Helium-3 gas comes mainly from the radioactive decay of  $^3\text{H}$  generated in nuclear reactors. To make it meet civilian requirements, the  $^3\text{H}$  content must be reduced to the order of  $10^{-11}\text{Ci/L}$  or less.

It is an extreme challenge to measure the ultra-trace  $^3\text{H}$  in helium-3 gas. Generally, low-concentration  $^3\text{H}$  could be analyzed by ionization chamber, proportional counter and Liquid Scintillation Counting method<sup>2-4</sup>, with the detection limit of about  $10^{-7}\text{Ci/L}$ ,  $10^{-9}\text{Ci/L}$  and  $10^{-12}\text{Ci/L}$ , respectively. The LSC has been applied to analyze  $^3\text{H}$  activity in environmental media (atmosphere, surface water)<sup>5</sup>, adopting the "flow-gas catalytic method" to prepare samples. This sample preparation method requires a large amount of raw gas, which is unprocurable for the scarce resource gas of helium-3.

To resolve this practical problem, it is necessary to design a feasible sample preparation system for the ultra-trace  $^3\text{H}$  measurement in helium-3 by LSC. Besides, the newly designed system (Dilute Catalytic System) could also recover of helium-3 gas during sample preparation, achieving the goal of ultra-trace  $^3\text{H}$  measurement under "zero discharge of helium-3" consideration. Experimental results show that the Dilute Catalytic System can meet the requirement for ultra-trace  $^3\text{H}$  measurement in helium-3 gas.

The gas flow in Dilute Catalytic System for LSC is shown in Figure 1. The schematic diagram of  $^3\text{He}/\text{H}_2$  dilution mixer is shown in Figure 2. The schematic diagram of Catalytic System and Condenser is shown in Figure 3.

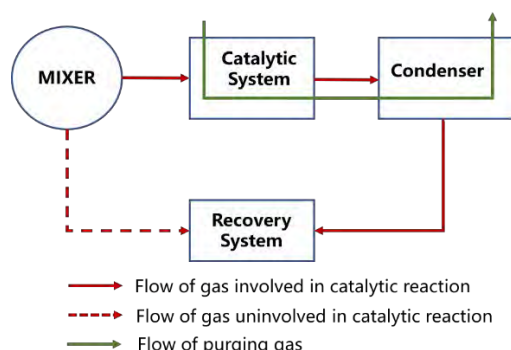


Figure 1 Schematic diagram of gas flow in Dilute Catalytic System for LSC

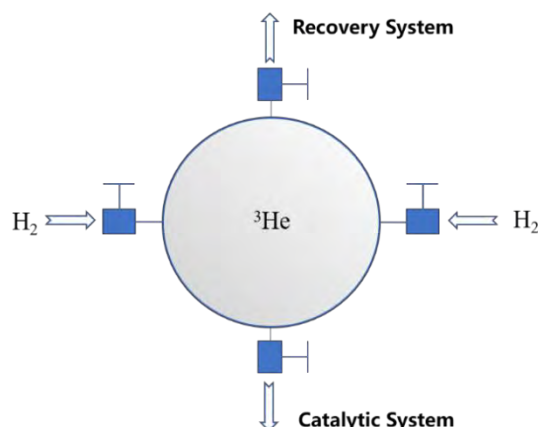


Figure 2 Schematic diagram of helium-3 gas dilution mixer

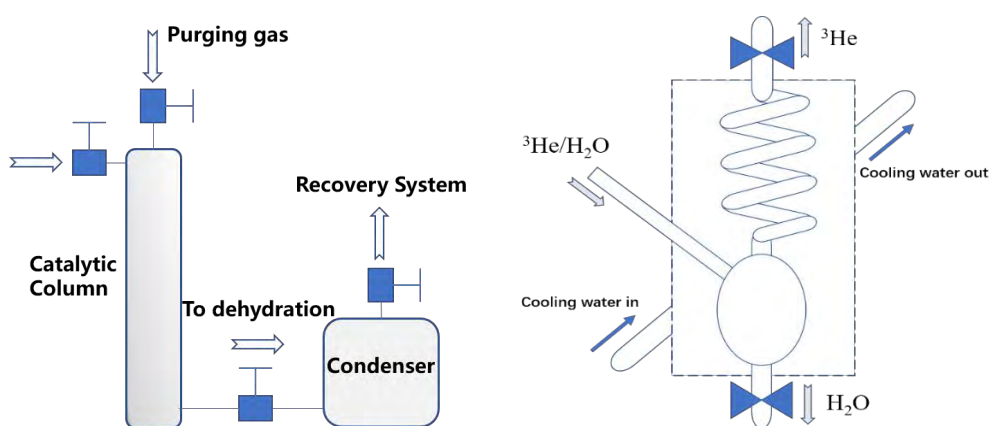


Figure 3 Schematic diagram of Catalytic System (left) and Condenser (right)

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## Rapid and safe method to measure radiostrontium in water samples I - chemical separation method -

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### Introduction

Monitoring of environmental radiostrontium is important because it is known to accumulate in bone and to increase risks of bone cancer and leukemia. Radiostrontium isotopes, <sup>89</sup>Sr and <sup>90</sup>Sr, are fission products. Measurement of those isotopes is important not only for routine monitoring around nuclear facilities but also at nuclear accidents. Those isotopes are pure beta-ray emitters. Because continuous energy distributions of beta-rays Sr should be separated from other elements before the measurement. The conventional methods to measure radiostrontium are time-consuming complicated procedures using lots of deleterious substances. Rapid analysis of radiostrontium in environmental samples is desired especially the case after nuclear accidents.

The purpose of this study is to develop a rapid and safe analysis method of radiostrontium in seawater and terrestrial water. We developed to measure beta-rays in a thin layered solid sample on a filter using a plastic scintillator bottle (PSB)<sup>1,2</sup>. The detail about the PSB is explained by the other study of ours. In this study, three Sr separation methods are described, i.e.; (1) solid phase extraction disk, (2) ion exchange separation, and (3) Sr adsorbent.

### Materials and Methods

#### *Samples*

Samples were artificial seawater (Daigo's Artificial seawater SP, Nihon Pharmaceutical, Co., Ltd.), and natural surface seawater collected at Ise Bay, Mie Prefecture, Japan. Sample water was spiked with radiostrontium, <sup>85</sup>Sr or <sup>90</sup>Sr, or <sup>210</sup>Pb.

#### *Sr separation method*

(1) Solid phase extraction disk<sup>3</sup>: A solid phase extraction disk (Empore™ Sr Rad Disk, 3M Co.) was used. After conditioning the disk with methanol and 2M HNO<sub>3</sub>, 100 mL of sample water adjusted to 3M HNO<sub>3</sub> was applied to the disk. And the disk was rinsed with 2M HNO<sub>3</sub>.

(2) Ion exchange separation: First, 100 mL of the sample water was loaded to a cation ion exchange resin (AG 50W-X8, 200-400 mesh, Bio-Rad) column with 10 mL of column volume to adsorb Sr. Then, Ca, Mg, Pb, etc. were rinsed with the mixture of ammonium acetate (15.4% W/V%) and methanol (1:1). And Sr was eluted with 4M HCl. Finally, Sr was precipitated as strontium carbonate from the eluate, and collected on a membrane filter with 0.45 μm of pore size (Durapore, Millipore Inc.).

(3) Sr adsorbent: A newly produced Sr adsorbent (Pureceram MAq®, Nippon Chemical Industrial, Co. Ltd.) composed of barium silicate was used. The Sr adsorbent of 100 mg was

added to 100 mL of sample water and stirred for 120 min. Finally, Sr adsorbent was collected on the membrane filter same as above.

#### *Sr Measurement by the PSB*

The dried disk or the filter was laminated with a pair of plastic films (100  $\mu\text{m}$ ) to avoid contamination. The laminated sample was in the PSB and measured with a low-background liquid scintillation system (AccuFLEX LSC-LB7, Hitachi, Ltd.).

The chemical yields of Sr and Pb were estimated with  $^{85}\text{Sr}$ ,  $^{90}\text{Sr}$  or  $^{210}\text{Pb}$ , respectively. The chemical yields of the stable elements, Ca, Mg, Na, and K, were measured with an ICP-AES (Thermo Jarrel Ash, IRIS/AP).

#### **Results and Discussion**

The solid phase extraction disk method allows the fastest analysis. The problem is that the disk is rather expensive. Considering the capacity of disk and the concentration of stable Sr in seawater, the maximum sample amount is about 300 mL of seawater per a disk.

The improved ion exchange separation method is safe and economical. Chemical procedure needs about 7 hours (total 2 days) with few deleterious substances. The chemical yields of Sr ranged from 70% to 90%. In this case, a sample with a  $^{90}\text{Sr}$  concentration of up to 0.2 Bq L<sup>-1</sup> can be detected for 60 min counting. By increasing the sample amount up to 1 L, it is possible to measure with the minimum detectable concentration of 0.02 Bq L<sup>-1</sup>.

The method using a strontium adsorbent is quick and simple, and needs no other chemical substances. Under appropriate conditions, the chemical yields of the adsorbent for Sr were >98%. The elements, Na, K, Mg, Ca, and Cs were adsorbed on the adsorbent in <5%. Using the PSB, the capacity of the adsorbent is about 100 mg at the maximum, then the water sample amount is under 100 mL. This method is more improvable in the future.

#### **Conclusion**

A rapid and safe method to measure radiostrontium using the plastic scintillator bottle has been developed. It is preferable to apply the three separation methods properly depending on samples, measurement conditions, and other situations. It was confirmed that this method using the plastic scintillator bottle is effective to monitor radiostrontium in water samples.

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I03

## **Research of H-3 and C-14 Sample Preparation Technology and Equipment in the Environment**

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### **Abstract:**

The H-3 and C-14 in the environment are generally measured by the liquid scintillation counting method, but the H-3 and C-14 samples in the environment have low background content and diversified forms. During the process of analysis and measurement, the sample preparation is a process worth paying attention. This report focuses on the preparation techniques and methods of H-3 and C-14 samples in the environment, including sampling of H-3 and C-14 in the air, concentration and preparation of H-3 and C-14 in environmental waters, and the preparation of H-3 and C-14 samples in organic biological samples. In addition, the application and achievements of Hapstar's OTCS11/3, ECTW-1, WCCS-14, RAH-3, RAC-14 and other series of products in the actual process of analysis and measurement are introduced.

I04

## A study for the selection of NPE-free cocktails for LSC routine measurements

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Liquid scintillation counting (LSC) is a widely used radiometric technique for the measurement of alpha and beta emitters present in environmental samples and materials coming from decommissioning activities (1). This technique makes use of liquid scintillation cocktails and one of the important components of these cocktails is the surfactant (2). Commonly, phenol ethoxylates are used as surfactants in the commercially available cocktails (3). These chemicals are added on the REACH (registration, evaluation, authorization and restriction of chemicals) – list of amendments to Annex XIV, as chemicals which can't be used anymore (4).

At SCK CEN (LRM) an extensive study was performed in order to find/select the best available cocktail(s) that are NPE (nonylphenol ethoxylates) free, to replace NPE based cocktails to be used for routine measurements. A total of 8 cocktails from several producers were selected and investigated in this study. Different parameters were tested, such as homogeneity/compatibility with the media where the radionuclides are present in, inherent background level and the influence on counting efficiency. Besides the specific media obtained after separation of several radionuclides (such as <sup>89/90</sup>Sr, <sup>210</sup>Pb, <sup>226</sup>Ra, <sup>99</sup>Tc, <sup>35</sup>S), also the matrices (such as water, clay water, urine, sea water, Carbosorb E) where direct measurement of several radionuclides (such as <sup>3</sup>H or <sup>14</sup>C), after mixing with the cocktail is performed, were investigated.

This paper will give an overview of obtained results leading to the selection of the best cocktail(s) for our routine applications in order to replace the Optiphase HiSafe 3 cocktail that is currently in use.

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## Application of ALOKA LB7 in atmospheric HTO measurement

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### Abstract:

Atmospheric tritium water (HTO) vapor monitoring can provide an opportunity for understanding its distribution regularities and environmental fate, which is valuable for radioecology and hydrogeological studies. However, the MDA of conventional LSC (e.g. Quantulus 1220) may not be enough sensitive for most atmospheric water samples due to the very low tritium content in the current ( $< 1 \text{ Bq} \cdot \text{L}^{-1}$ )<sup>1</sup>. In response to the limitation of measurement, a new type of LSC (i.e. ALOKA LB7) was considered in environmental tritium determination because it can hold larger counting vial (e.g. 100 ml) and thus can make the MDA decreased<sup>2</sup>. In this study, a series of work were carried on evaluating the performance of LB7. To obtain an optimized counting condition for measurement, the period of dark treatment, the sample/cocktail ratio, and choice of scintillation cocktail were performed under 100 ml counting condition. The appropriated counting window was optimized using self-developed software (The copyright belongs to Dr. Zhao Chao). The MDA of  $0.28 \text{ Bq} \cdot \text{L}^{-1}$  is routinely obtainable for sample sizes of 50 ml water and counting time of 1000 min in our lab, while longer counting time can be used to obtain even lower MDA<sup>3</sup>. To know its radioactivity analysis performance, the long-term stability of spiked samples and background samples, and the interference of radon were tested. In addition, the electrolytic enrichment method was used in co-comparison experiments with a direct measurement method to confirm the reliability of measured data. The results showed that the LB7 can provide a stable and reliable counting environment for the determination of low-level tritium. Furthermore, the total uncertainties of direct measurement method (20 ml condition and 100 ml condition) and the electrolytic enrichment method were evaluated based on the available quality of water sample and given tritium specific activity. Combining the domestically investigated HTO data in 2017-2018<sup>3</sup>, a comprehensive selection strategy of tritium analysis was proposed for large-scale HTO investigation in the future.

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I06

## **Application of scintillation detectors in laser nuclear physics**

Chuangye He, on behalf of the collaboration of laser nuclear physics

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With the development of high intensity laser technologies, lasers became a very useful tool to explore the unknown world of nuclear astrophysics. High intensity lasers can produce some astrophysical environments in stars where nuclear reactions could take place. Nuclear physicist suggested that nuclear reaction rates in high density plasma environments may be different from that obtained in accelerator labs. While the differences could probably solve some puzzles in nuclear astrophysics. Under such circumstance, nuclear diagnostic devices based on scintillation detectors were established. In this talk, I will introduce the recent experiment results that has been done at the Shengguang II laser facility.

I07

## Experimental study of high yield neutron source based on multi reaction channels

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**Abstract:** The short-pulse neutron source based on ultra-short and ultra-intense laser is an ideal neutron source for ultra-fast neutron detection. For many applications of the novel laser neutron source, the neutron yield now becomes a major limitation. It is proposed here that, based on the Target Normal Sheath Acceleration mechanism (TNSA) and the beam-target reaction scheme, the adoption of composite component target LiD as the neutron converter can be an effective path to enhance the neutron yield. Compared with the traditional LiF converter, which has two typical reaction channels p-Li and d-Li, the use of LiD converter has the advantages on introducing two more reactions channels, i.e., p-D and d-D. Therefore, the much more reaction channels are expected to be beneficial for increasing the neutron yield. It was experimentally demonstrated that by using LiD converter, an enhancement of 2~3 folds of neutron yield is achieved compared with the LiF converter. As a result, a neutron beam with a highest yield of  $5.2 \times 10^8$  /sr with a forward beamed distribution is well obtained. The contribution of multi reaction channels is also identified, indicating the enhancement of neutron yield mainly comes from the p-D reaction.

I08

## Simultaneous determination of $^{210}\text{Pb}$ and $^{210}\text{Po}$ in seafood samples using liquid scintillation counting

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Lead-210 ( $T_{1/2} = 22.3$  a) is a member of the natural radionuclides  $^{238}\text{U}$  decay series, it decays to  $^{210}\text{Bi}$  ( $T_{1/2} = 5.013$  d) by emitting  $\beta$  and  $\gamma$  rays, and  $^{210}\text{Bi}$  decays to  $^{210}\text{Po}$  ( $T_{1/2} = 138.4$  d). The consumption of food and drinking water is the main pathway for human exposure to  $^{210}\text{Pb}$  and  $^{210}\text{Po}$ . Owing to their relatively high concentrations compared with those in other food samples,  $^{210}\text{Pb}$  and  $^{210}\text{Po}$  activity concentrations in marine organisms have been well studied. The annual effective dose from ingestion of uranium and thorium series radionuclides was  $120 \mu\text{Sv/a}$ , which was caused by  $^{210}\text{Pb}$  and  $^{210}\text{Po}$  constituting greater than 80% of the total. In this study, a fast and cost-efficient method for the simultaneous determination of  $^{210}\text{Pb}$  and  $^{210}\text{Po}$  in seafood samples by ultralow-level liquid scintillation counting after se

paration on a Sr-spec column was developed. The recoveries of  $^{210}\text{Pb}$  and  $^{210}\text{Po}$  were  $\sim 70\%$  and  $\sim 85\%$ , respectively. The minimum detectable activity of the proposed method for  $^{210}\text{Pb}$  and  $^{210}\text{Po}$  was  $3.85 \text{ Bq/kg}$  and  $1.50 \text{ Bq/kg}$ , respectively, which is suitable for the determination of  $^{210}\text{Pb}$  and  $^{210}\text{Po}$  in seafood samples. The radiochemical procedure was validated by measuring  $^{210}\text{Pb}$  and  $^{210}\text{Po}$  activity concentrations in IAEA-certified reference materials and successfully applied to shrimp and clam samples.

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I09

## **Analysis validation of Difficult To Measure Radionuclides in Decommissioning waste**

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**Abstract:** Analysis of difficult to measure radionuclides (DTM) in solid decommissioning waste requires first complete destruction of the solid matrix followed by separation of the analytes of interest from each other and from other interfering radionuclides. As the name suggests, the DTMs are difficult to analyse and validation of the radiochemical analyses is hindered by lack of reference materials. One way to overcome the challenge is participation in an intercomparison exercise. Therefore, a three-year project was proposed for the Nordic Nuclear Safety Research program for yearly intercomparison exercises on DTM analyses in real decommissioning waste. The DTM analyses focused on beta emitters, which are mainly analysed using liquid scintillation counting (LSC), and additionally gamma emitters were analysed. The results were statistically analysed according to ISO 13528 standard [1]. As the original content of the analytes of interest was not known in advance, assigned values were calculated from the participants' results using robust statistical methods [1]. The performance was assessed using z score, which compares the participant results' deviation from the assigned value [1]. The results have been published in the NKS report series [2,3] and further analysed in peer reviewed articles [4,5].

In 2019, 8 laboratories from Finland, Sweden, Norway, Denmark and France carried out DTM analysis of Fe-55 and Ni-63 in activated steel [2,4]. Additionally, some laboratories also carried out analysis of C-14 and Co-60. The results showed that majority (three-fourths) of the Fe-55 and Ni-63 results were satisfactory whereas C-14 results showed variation within same order of magnitude. All Co-60 results were satisfactory.

In 2020, the same 8 laboratories carried out DTM analyses of H-3, C-14, Fe-55, Ni-63 and gamma emitters in activated concrete originating from a biological shield [3,5]. Additionally, some laboratories also carried out analysis of Ca-41 and Cl-36. The results showed difficulties in the solubility of the matrix resulting in varying degrees (estimated) completeness of the acid digestions. Also high original Fe content caused difficulties in the radiochemical procedures and calculation of yield. Therefore, the Fe-55 results varied significantly. The Ni-63 results showed that only 3 out of 13 results were above limit of detection. However, those 3 results were in good agreement. The H-3 results were in good agreement whereas only one C-14 and one Cl-36 result were above limit of detection.

In 2021, in total 9 laboratories from Finland, Norway, Denmark, France and Taiwan are carrying out DTM analysis in spent ion exchange resin. The analyses will focus on analysis of Fe-55, Ni-63, Sr-90 and gamma emitters. Additionally, H-3, C-14, Cl-36, Ni-59 may be analysed by some laboratories. Capabilities for Tc-99, I-129 and Cs-135 were also reported. The results will be published in the NKS report series and in a peer-reviewed article in 2022.

As a conclusion, the intercomparison exercises have emphasised the difficulties encountered with DTM analyses but more importantly, they have strengthened the participating laboratories'

capabilities. Additionally, collaboration has been shown to be highly important when analysing DTMs. The specific challenges encountered with LSC measurements as well as advantages of inter-laboratory discussions during intercomparison data preparation, in respect to method validation and data quality, will be discussed in the presentation.

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I10

## Performance of portable TDCR systems developed at LNE-LNHB

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The Triple to Double Coincidence Ratio (TDCR) liquid scintillation measurement technique is commonly used in National Metrology Institutes (NMIs) to perform standardization of pure beta emitters. The historical device from LNE-LNHB, RCTD1 [1], is a quite large device, which has been developed and commonly used over the past 25 years with its associated electronics [2] on various radionuclides. During the last 2 years, the LNHB has developed a new portable device in order to perform on site calibrations and comparisons.

In this paper, we will present the newly developed portable TDCR liquid scintillation measurement system. This system has been developed and designed at LNE-LNHB and the full system was built using Fused Deposition Modeling (FDM) 3D printer. In total two kinds of devices have been developed: miniTDCR (25 cm diameter 10 cm height) and microTDCR (16 cm diameter 10 cm height) (Fig. 1). With the possibilities offered by the 3D printers, it was possible to design the system in only 5 pieces of plastic with a low density due to a honeycomb structure inside the material. These devices are light and easy to handle and will be commonly used to perform on site calibrations or developments.



Figure 1: Picture of the portable microTDCR developed at LNE-LNHB with the corresponding HV power supply and the nanoTDCR electronics. The device is designed for standard 20ml scintillation vials.

Additionally, to improve the results of the measurement, especially to find the proper optimal kB value, a special care has been taken to design the cap of the device. This new cap possesses a filter holder, which allows to place different levels of grey filters glued with a 3D printed tool (Fig. 2, left). The filters are designed to ensure that they cover the entire surface of the vial and that all the light produced by the liquid is filtered. The reproducibility of the position of the filter is ensured by a 3D printed ring. An example of efficiency variation with 6 filters on a  $^3\text{H}$  in water source is presented in Fig. 2, right.

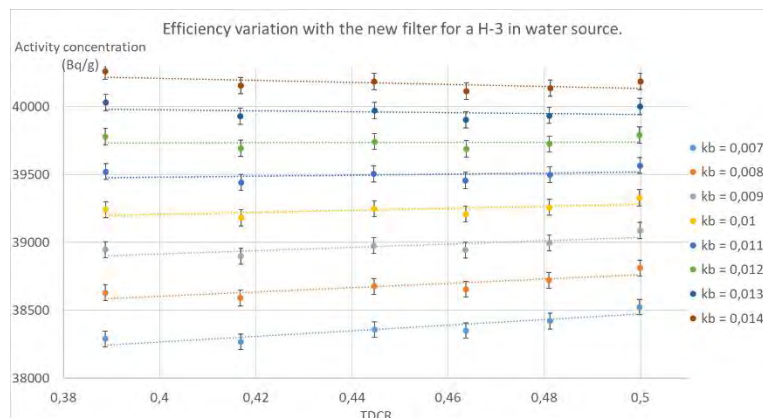


Figure 2: Picture of the older filter and tool at the left, result of efficiency variation and optimal kB value at the right for  $^3\text{H}$  in water measurement.

In this paper we will present the different tests performed in order to qualify the performance of the system. Using the following nuclides:  $^3\text{H}$ ,  $^{63}\text{Ni}$ ,  $^{14}\text{C}$  and  $^{55}\text{Fe}$  it was then possible to compare the portable TDCR results and efficiencies with the RCTD1 system from our laboratory. These new devices present a higher efficiency than RCTD1 and the results of the activity obtained are all in good agreement with less than 0.1% of relative difference for highest energy nuclides up to 0.3% with low energy nuclides.

In addition, the performance of the microTDCR counter was compared to the performance of the miniature TDCR counter developed previously at Sofia University and described in [3]. Both systems give consistent activity results in tests with  $^3\text{H}$  and  $^{14}\text{C}$  unquenched standards. The agreement for the  $^3\text{H}$  sources was within 0.3% and for the  $^{14}\text{C}$  within 0.05%, both within the estimated uncertainties. However, the logical sum of doubles efficiency ( $\epsilon_D$ ) of the microTDCR counter is 11% higher than that of the Sofia University counter in the case of  $^3\text{H}$ . This is attributed to the optimized optical chamber of the microTDCR device.

The new portable systems are operational since the end of 2019 and were used in different calibrations and comparisons in LNE-LNHB but also in other laboratories, as they are easy to transport.

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I11

## Preparation and application of new scintillators

### Abstract

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Scintillator is a type of material that absorbs radiant energy under the action of nuclear radiation ( $\alpha$ ,  $\beta$ ,  $\gamma$  rays, etc.) or high-energy particles, converts partially absorbed radiant energy into light energy, and emits a temporary fluorescence. The report mainly introduces the research on liquid scintillator and plastic scintillator in recent years by our research team. A liquid scintillator is always made up of an aromatic solvent and one or more fluorescent substance. P-Xylene(PX) with stable chemical performance has long decay length and high flash-point. It can be used to prepare low afterglow liquid scintillator. Phenyl-o-xylylethan(PXE) with high refractive index, is used to make high refractive index liquid scintillator with PPO (2,5-Diphenyloxazoze) and bis-MSB (p-bis(o-methylatryl)-benzene) as the primary solute and the second solute. On the basis of previous work, two methods, namely direct dissolution method and solvent extraction method, were used to prepare the Gd doped liquid scintillators, and the performances of the samples by two different methods was compared. As a branch of the organic scintillator, the plastic scintillator attracts extensive attention due to its short decay time, high luminous efficiency, good physical and chemical stability, easy transportation, non-toxicity. A series of non-initiator polystyrene-based plastic scintillators with different emission wavelengths were prepared by thermal polymerization. Especially, the long wavelength plastic scintillators can be used in the field of strong radiation detection. The type and concentration of the first primary additives, the type and concentration of the wavelength shifters, and the fluorescence spectrum of the plastic scintillator and the light yield were studied. In order to improve the detection efficiency of neutrons, lithium-doped plastic scintillators were prepared by lithium methacrylate and methacrylic acid using a thermal polymerization method. At the same time, the fluorescence performance, light yield, decay time and other properties were studied. In order to improve the detection efficiency and image resolution, plastic scintillator fiber array was prepared by two methods, namely a capillary plastic scintillator fiber method and plastic scintillator fiber method.

**Key words:** liquid scintillator; plastic scintillator; lithium-doped plastic scintillator; plastic scintillator fiber array

I12

## Standardization of $^{55}\text{Fe}$ solution using the TDCR method in POLATOM as part of the CCRI (II)-K2.Fe-55.2019 key comparison.

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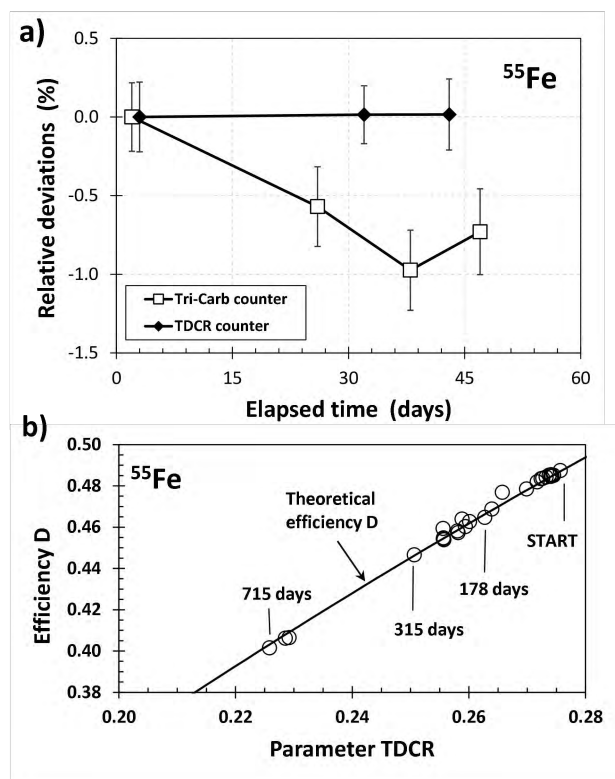
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This paper presents the results of measurements of the  $^{55}\text{Fe}$  solution activity in POLATOM using the TDCR method. Measurements were performed in the TDCR counter containing the MAC3 coincidence module, in which the live-time technique with extendable dead-time was used.

$^{55}\text{Fe}$  decays by electron capture and emits electrons and X photons with low energies of about 5 keV. The emitted X-rays  $K_{\alpha 1}$  and  $K_{\alpha 2}$  have energies close enough that the radiation can be treated as mono-energetic. Therefore,  $^{55}\text{Fe}$  sources are used e.g. in fluorescence methods for calibrating of low-energy photon detectors [1]. The liquid scintillator (LSC) technique is a convenient way to standardize  $^{55}\text{Fe}$ . Due to the simple  $^{55}\text{Fe}$  decay scheme, its measurement is also an interesting test of the application of the LSC technique.

POLATOM and ENEA-INMRI proposed to organize a new key comparison for a  $^{55}\text{Fe}$  solution. Previous international comparisons took place in 1995 [2] and 2006 [3]. In 2019, the proposal was accepted by the CCRI(II) and the comparison was registered as CCRI(II)-K2.Fe-55.2019 [4].

POLATOM, as a pilot laboratory, prepared  $^{55}\text{Fe}$  solution for comparison and the  $^{55}\text{Fe}$  ready-to-measure (R-T-M) sources in the Ultima Gold scintillator in polyethylene vials (diffusive vials). A total of 92 sources were prepared with mass of the active solution ranging from 29.7 mg to 48.5 mg. This paper presents the procedure used to prepare these LS-sources. All R-T-M sources were measured at POLATOM using the triple-double coincidence (TDCR) method over a period of 12 days for a total time of 25.5 hours. Their stability was monitored with a Tri-Carb 2710 TR scintillation spectrometer and the TDCR counter. The stability was monitored for a total of 12 months. Estimated uncertainty of the active solution weighing process in POLATOM was  $\pm 0.1\%$ . The standard



**Fig. 1** Instability of R-T-M sources over time: a) average activity obtained from measurements in two counters, b)

deviation of the R-T-M sources activity measurements in the TDCR counter was  $\pm 0.22\%$ .

The results of measurements of the activity of R-T-M sources in the Tri-Carb 2710 TR spectrometer decreased by about 1% during the first 5 weeks of measurements. The average value of the quenching parameter tSIE within the measurement uncertainty did not change (Fig. 1a). In the TDCR counter, a slow decrease in the measurement efficiency was observed, which amounted to about 4% after 6 months from the first measurement (Fig. 1b). The applied model of calculation the detection efficiency in the TDCR counter [5] corrected very well the occurring changes in the measurement efficiency. The mean value of R-T-M sources activity determined in the TDCR detector did not change (Fig. 1a). The nature of the physico-chemical processes in the R-T-M sources leading to the observed changes in the measurement efficiency has not been clarified. Similar observations were also reported by other authors [2, 6, 7]. The  $^{55}\text{Fe}$  spectrum was calculated with the MICELLE2 program, and the activity of measured sources - with the MetroActivityLSC program developed by POLATOM.  $^{55}\text{Fe}$  atomic data were adopted in accordance with K. Kossert's recommendation [8].

The POLATOM results of measurements of the  $^{55}\text{Fe}$  solution activity were very well consistent within the measurement uncertainty with the key comparison reference value (KCRV). It was observed that the measurement results of the sources prepared in non-diffusive vials were lower than in diffusive vials. The same was observed in the results of the key comparison. It was found that due to the internal light reflection in non-diffusive vials there is a deviation from the Poisson distribution for the emitted light not included in the calculation model. The results of the measurements in non-diffusive vials have been discarded from the computation of the KCRV. The results of participants using diffusive vials were consistent with the values obtained from the measurement of R-T-M sources.

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## **DECLAB: A software for liquid scintillation spectra deconvolution**

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Liquid scintillation spectrometry (LSS) is a commonly used technique for the determination of alpha and beta emitters and during the last decades it has played a leading role in the radiometric characterization of several kind of samples, from surveillance of the environment to managing of nuclear waste produced in industrial facilities. LSS has an appropriate efficiency but poor resolution, which hinders the determination of specific radionuclides in mixtures. For this reason, the sample dissolution is usually preceded from a chemical isolation of the radionuclides of interest before LSS measurement. However, not always is possible to obtain a pure LS spectrum. The main reason is that, in some cases, the sample could contain different radionuclides from the same element, which cannot be separated chemically. Another non-minor issue is the difficulty in removing all the radiochemical interferences in samples with a relative high activity.

For this reason, several authors put their effort to achieve the quantification of several radionuclides from the same LS spectrum using different approaches, from the simplest defining different counting windows [1] to the most complex, as the use of artificial neural networks [2].

The approach followed by our research group is the construction of Partial Least Square regression (PLS) models by means of spectra obtained from standard solutions in a calibration step. In a subsequent step of sample analysis, the quantification of the radionuclides included in the calibration is performed. PLS is a statistical method that reduces the predictors (in the studied case, the counts in each channel of LS spectrum) to a smaller set of uncorrelated principal components and performs least-squares regression on these components, instead of on the original data. The feasibility of multivariate calibration by PLS for LS spectra deconvolution has been previously proved with a method developed using the software Matlab<sup>TM</sup>, a licensed software of MathWorks<sup>®</sup> [3,4]. However, the calculations required for PLS deconvolution are not easy to perform in routine laboratories due to the need of a specialized software.

In this communication we present DECLAB, an user friendly online tool oriented to LS spectra analysis, with classical quantification methods like constant efficiency and quenching curve, and specialized in deconvolution based on PLS regression.

The operation of DECLAB is based on two stages: calibration and analysis.

In the first one, the user provides the tool with standard spectra, as well as their activity data, with which the efficiency calculation will be carried out.

In the analysis stage, we enter the spectra of the samples, as well as the data of their treatment, such as masses before and after the treatment, chemical yield, reference date to which it is required to correct the activity and period of the radionuclide to be analyzed. Then, from the PLS calibration and all this information, DECLAB calculates the activity, uncertainty and detection limit of the target radionuclides in the samples.

DECLAB has three calibration and analysis modes: constant efficiency, quenching curve, and deconvolution by PLS.

In the constant efficiency mode, a single efficiency is calculated from one or more standard spectra, for alpha or beta multichannel or both simultaneously. This efficiency is used in the analysis stage to determine the activity of the samples.

In the quenching curve mode, a series of standards with different levels of extinction are uploaded to DECLAB to obtain a calibration curve that relates the quenching parameter with the efficiency. From this curve and the quenching parameter of the sample, the efficiency value used to calculate the activity of each sample is interpolated.

Finally, the deconvolution mode allows the simultaneous analysis of several radionuclides from a single spectrum of the sample. It is based on the construction of a PLS model fed with standard spectra of the different radionuclides to be analyzed. In the calibration stage, the radionuclides which are expected to be in the sample are selected and a minimum of 3 standard spectra must be uploaded to DECLAB for each of them. With these standard spectra and their activity for each of the selected radionuclides, a PLS model is calculated. In the analysis stage, the PLS model is used to quantify the different radionuclides in spectra of mixtures.

In this first version, DECLAB only allows the analysis of spectra obtained with Quantulus 1220. However, this is an ongoing project, and we are planning to include new input files obtained from other liquid scintillation spectrometers from Perkin Elmer and HIDEX.

DECLAB has been powered by Aktios with the financial support of the Spanish Nuclear Safety Council (CSN).

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I14

## Rapid and safe method to measure radiostrontium in water samples -improvement of measuring method using a plastic scintillator bottle-

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### Introduction

We have studied a simple and rapid method to analyse radiostrontium in seawater. Radiostrontium isotopes in fission products are  $^{89}\text{Sr}$  and  $^{90}\text{Sr}$ . Since those isotopes are pure beta emitters, they must be separated from other nuclides before measurement. We applied three Sr separation methods, i.e.; (1) solid phase extraction disk, (2) ion exchange separation, and (3) Sr adsorbent. Details of the methods are described in the other study of ours. A plastic scintillation bottle (PSB) method was applied to measure activities of  $^{89}\text{Sr}$  and  $^{90}\text{Sr}$  on a disk or a filter. As shown in Fig. 1, the PSB is assembled with a couple of cylindrical plastic scintillators, a spacer contained in a bottle. The sample is sandwiched between the plastic scintillators. To prevent contamination, the sample was laminated with polyethylene terephthalate (PET) type films of 100  $\mu\text{m}$  thickness. Scintillation counting of the PSB was performed with a low background liquid scintillation system (LSC-LB7, Hitachi, Ltd.). The counting efficiency was about 70% on average of  $^{90}\text{Sr}$ - $^{90}\text{Y}$  at the equilibrium stage<sup>1,2</sup>. The counting efficiencies of each nuclide were 40% and 100% for  $^{90}\text{Sr}$  and  $^{90}\text{Y}$ , respectively. The PET film causes a shielding effect for the beta-rays. Thus, the counting efficiency of  $^{90}\text{Sr}$  was lower than that of  $^{90}\text{Y}$  depending on the energy. The purpose of this study is to improve the counting efficiency of  $^{90}\text{Sr}$  by examining the thickness and the material of the laminate films.

### Materials and Methods

Five laminate films were examined i.e.; PET type films with the thickness of 100  $\mu\text{m}$ , 50  $\mu\text{m}$ , 32  $\mu\text{m}$ , and the biaxially oriented polypropylene (OPP) type film of 25  $\mu\text{m}$ , and a plastic scintillator film of 70  $\mu\text{m}$  thickness. As the reference source,  $^{90}\text{Sr}$  solution was deposited and dried on a membrane filter. The filter was laminated with the films, contained in the PSB, and measured with LSC-LB7.

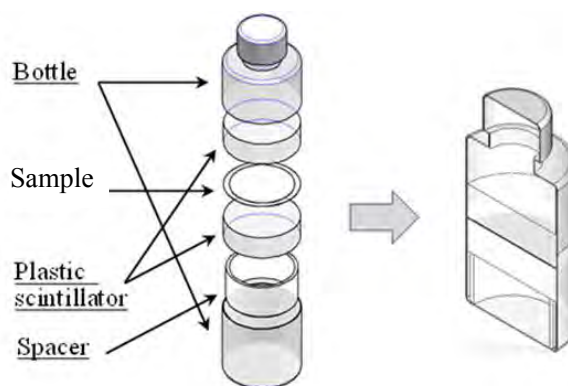


Figure 1 Assembly of the plastic scintillator bottle.

## Results and discussion

Figure 2 shows beta-ray energy spectra obtained by the different thickness of the plastic films. It can be seen that the counting rate under 546 keV was increased with decreasing the thickness, where was the region of the beta-rays from  $^{90}\text{Sr}$ . Table 1 shows the counting efficiencies estimated from the experimental results. The counting efficiency of  $^{90}\text{Sr}$  was obtained by subtracting the counting efficiency of  $^{90}\text{Y}$  ( $\cong 100\%$ ) from total counting efficiency. As the thickness of films decreases, the counting efficiency increases. In the cases of the plastic scintillator film and the 25- $\mu\text{m}$  film, 100% counting efficiency was accomplished for  $^{90}\text{Sr}$ . Another study of ours suggests that this method of using plastic scintillator films can be applied to low energy beta and alpha emitters such as  $^3\text{H}$ ,  $^{14}\text{C}$ ,  $^{35}\text{S}$ , and  $^{63}\text{Ni}$ .

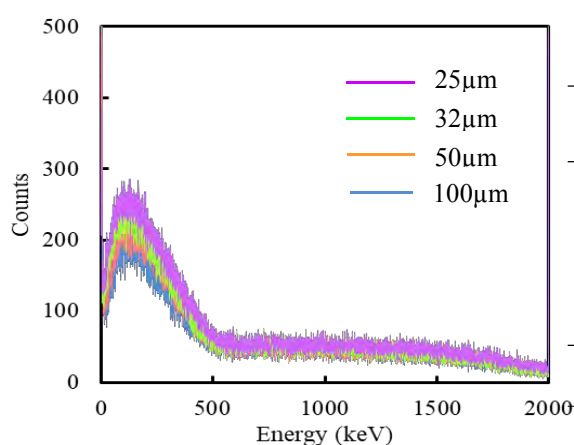


Table 1 Counting efficiencies of  $^{90}\text{Sr} + ^{90}\text{Y}$  and  $^{90}\text{Sr}$

Laminate film type	Thickness ( $\mu\text{m}$ )	$^{90}\text{Sr} + ^{90}\text{Y}$ Eff. (%)	$^{90}\text{Sr}$ Eff. (%)
Plastic (PET)	100	158	58
	50	171	71
	32	173	73
Plastic (OPP)	25	200	100
Plastic scintillator	70	200	100

Figure 2  $^{90}\text{Sr} + ^{90}\text{Y}$  spectra due to difference thickness of laminate films.

The counting efficiency was as high as the conventional liquid scintillation counting method, when the plastic scintillator was applied. Consequently, the method described here is superior to the liquid scintillation counting method at the point of no production of flammable organic liquid wastes. In addition, the plastic scintillator can be used repeatedly because the sample is laminated. So, the method is not only economical but also eco-friendly. Furthermore, this technique has a major feature that quenching peculiar to liquid scintillator does not occur.

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## Liquid Scintillator Neutron Detection System

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Scintillator detectors in inertial confinement fusion experiments are predominantly used to measure neutron yield and ion temperature of the primary fusion reactions. The detection of neutrons in fast-ignition experiments is very challenging since it requires the neutron detection system to recover within 100 ns from a high background orders of magnitude stronger than the signal of interest. Liquid scintillator with different compositions was investigated. We present several designs of liquid scintillator using the Geant4 Code and the X-Lab Code. Our liquid scintillator is based on PPO, dissolved in xylene and enriched with molecular O<sub>2</sub>. The detector consists of a 2-3 liters volume of liquid scintillator coupled to a gated MCP. A small air bubble allows for thermal expansion of the liquid without a significant pressure increase. The gating performance under high-intensity  $\gamma$  rays was experimentally checked. The typical flight time spectrum of the neutrons from (p,n) reaction driven a PW laser is shown in Fig.1. The neutron yield in the fast ignition experiments on Shenguang-II laser facility was successfully measured using this detector. In fast ignition experiments, a PW laser beam was employed to produce high intense electron beams to ignite the compressed fuel. Our neutron detection system could suppress the background signal and eliminate the afterglow present in conventional plastic scintillators.

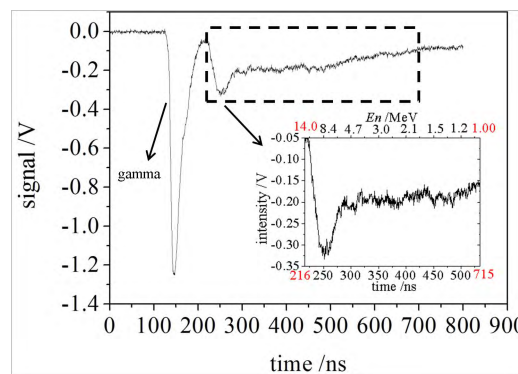


Fig. 1 Neutron flight time spectrum obtained in PW laser

I16

## **The influence of plastic scintillator dimensions, PMT size, and photocathode geometry on the detection efficiency**

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### **Abstract:**

The work deals with the issue of effective miniaturization of the read-out electronics of plastic scintillators while maintaining the current high detection efficiency and sensitivity. The values obtained by measuring the response of model  $\gamma$  emitters (Co-60, Cs-137, Am-241) at different distances by polystyrene plastic scintillators (Nuvia, a.s., CZ) were used to compare detection parameters. Attention was focused on the comparison of classic 2" PMT with circular photocathode with 1" PMT with different geometry of photocathode.

I17

## PSresin for the analysis of alpha-emitting radionuclides: Comparison of diphosphonic acid-based extractants

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The determination of radioactivity in different types of samples and scenarios is a complex process, intensive in human and material resources and that often requires the analysis of a large number of samples. In this context, the development of global screening parameters, such as gross beta and alpha in drinking water, is especially helpful in reducing workload. For this parameter and for the characterization of the activity in other applications, such as those related to the dismantling of nuclear power plants, the contribution of actinides is especially relevant

This work presents a new method to analyse actinides using plastic scintillation resins (PSresins) packed in a solid-phase extraction cartridge. PSresins have already been mainly developed for the measurement of beta-emitting radionuclides such as <sup>90</sup>Sr and <sup>99</sup>Tc<sup>1-2</sup>. In this work, PSresins with different extractants for the selective measurement of alpha-emitting radionuclides have been developed and compared, providing a new methodology for the measurement of alpha emitters that requires less time and fewer reagents. The extractants used in this study are based on diphosphonic acid derivatives (Figure 1), which have attracted a lot of interest due to their effectiveness as a complexing reagent for metal ions, showing high sorption capacities for actinides that make the stripping of the sorbed actinides difficult<sup>3-6</sup>. This property is not an issue in PSresin measurements since the analyte is not eluted from the resin during analysis.

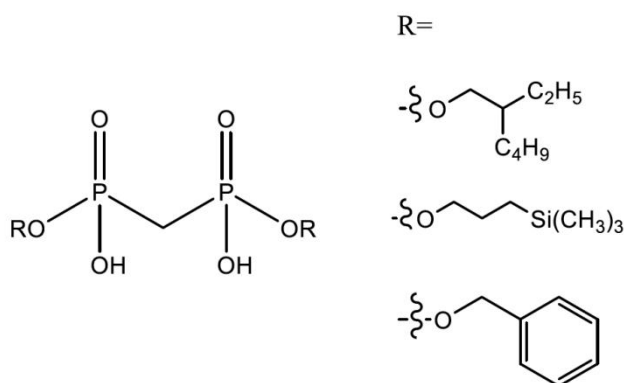


Figure 1. Core structure of the extractant studied and the different radicals.

These extractants were immobilised on plastic scintillation microspheres at a ratio of 1/1:6. From the three PSresin, the EH-PSresin (with 2-ethylhexyl radical group) and Si-PSresin (with 3-(trimethylsilyl)-1-propyl radical group) produce retention and detection efficiency of 100% for

$^{241}\text{Am}$  in HCl 0,5 M, whereas the Bz-PSresin (based on benzylic radical group) presented a lower retention (48%) for  $^{241}\text{Am}$ . EH-PSresin and SI-PSresin were also tested for  $^{230}\text{Th}$ , Uranium and  $^{238}\text{Pu}$  with quantitative retention and values of efficiency of 100% or close. Retention for  $^{210}\text{Po}$  and  $^{226}\text{Ra}$  were lower than 50% in this medium. No significant differences between EH-PSresin and SI-PSresin in terms of retention, detection efficiency, break through volume and capacity were found but it does in terms of alpha and beta discrimination. Based on those results, a batch study for several alpha emitting radionuclides and beta emitting radionuclides in SI-PSresin with different concentrations of hydrochloric acid, nitric acid and phosphoric acid have been performed. Showing high retentions at low acid concentrations.

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## Determination of the $^{213}\text{Bi}$ half-life by Cherenkov counting

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The isotope  $^{213}\text{Bi}$  is part of the  $^{229}\text{Th}$  decay chain, and currently in the focus of several preclinical studies, because  $^{213}\text{Bi}$  could be a suitable candidate for alpha-immunotherapy due to its half-life and decay-scheme [1]. Bismuth-213 is both alpha (2.1%) and beta emitter (97.9%), where the average beta energy lies above 300 keV. This latter property was used in our experiment to determine the half-life of  $^{213}\text{Bi}$  by Cherenkov counting.

In order to measure the half-life of  $^{213}\text{Bi}$ , it is essential to separate it from the preceding part of the decay chain. As it was previously demonstrated [2], this can be achieved without the use of radiochemistry by collection of recoil atoms. Following this approach, recoils from a 750 kBq  $^{225}\text{Ac}$  source were collected on half-cylinders manufactured from clear acrylic glass (polymethyl methacrylate).

The physical properties of acrylic glass make it a suitable material for Cherenkov detectors. The dimensions of the half-cylinders were chosen so, that if two of them are glued together, the obtained cylinder would emulate the outer geometry of a commercial glass vial commonly used in liquid scintillation counting (LSC). This made it possible to measure the samples in existing TDCR (triple-to-double coincidence ratio) setups at PTB.

In total, four Cherenkov samples were produced, and the half-life of  $^{213}\text{Bi}$  was obtained by fitting the observed counting rate of double (D) and triple coincidences (T) as a function of time. The presence of  $^{209}\text{Pb}$  with comparable half-life in the remaining part of the decay chain made the analysis more complicated, but the obtained value of  $T_{1/2}(^{213}\text{Bi}) = (45.64 \pm 0.22)$  minutes is in good agreement with the currently recommended half-life of  $^{213}\text{Bi}$ . The final value will be presented at the conference, with further details of the experiment.

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## **The new International Reference System for pure beta emitters**

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### **Abstract:**

Accurate measurement of the activity content of radiopharmaceuticals is essential to ensure the safe and effective use of pure beta-emitting radionuclides (<sup>90</sup>Y and <sup>89</sup>Sr) for the growing field of radiopharmaceutical therapy amongst other medical applications [1,2]. Pure beta emitters can also be found in the environment and in the food chain, from permitted and accidental discharges from nuclear power plants and fuel cycle facilities and from the historical weapons testing programme; accurate measurement is needed to ensure that risks to health are controlled. However, the measurement of pure beta emitters can be challenging as there are no associated gamma rays.

The reliability of a radioactivity standards must be ensured through an unbroken chain of calibrations from a primary standard held by a metrology institute to the measurement. To claim its capability to produce such primary standards, a National Metrology Institute (NMI) (or an institute to which this work has been designated by the NMI) must participate in international comparison exercises organized through the Consultative Committee for Ionizing Radiation Section II (CCRI(II)) or a Regional Metrology Organization (RMO). Supported by the published results of the international comparison, the NMI can then apply to register a new Calibration and Measurement Capability (CMC) under the CIPM Mutual Recognition Arrangement (MRA) [3].

International comparison exercises are therefore crucial, impacting the quality of all measurements at all levels. However, such exercises, carried out on an international scale, require a significant logistical effort on the part of the lead laboratory which has to manage the despatch and reception of a large number of radioactive solutions. This is one reason why the Bureau International des Poids et Mesures (BIPM) operates an International Reference System (SIR); the SIR [4] is a very stable and reproducible transfer instrument which enables primary standards to be compared on demand, avoiding the need to organize large comparison exercises. The SIR has been in operation since 1976 and covers 52 radionuclides. However, the SIR is suitable only for measuring gamma emitters. The BIPM, the Polish Institute of Atomic Energy (POLATOM), the French Laboratoire National Henri Becquerel (LNHB), the British National Physical Laboratory (NPL), the German Physikalisch-Technische Bundesanstalt (PTB) and the Chinese National Institute of Metrology (NIM) have therefore worked together to construct a new instrument, the Extended SIR (ESIR), to provide an equivalent comparison service for pure beta and alpha emitters.

The new ESIR is based on liquid scintillation counting. The instrument (see Fig 1) uses three photomultiplier tubes (PMT) and implements an approach inspired by the Triple to Double Coincidence Ratio (TDCR) method [5]. Nevertheless, the goal of this system is not to determine the absolute reference activity of a radioactive solution sent by a NMI, but to allow the possibility to compare the activity of the solutions provided by several NMIs. This comparison process must

operate over decades and thus the long-term reproducibility of the system is very challenging. Thus, the robustness of the method must be checked and this is why an experimental protocol was studied to mimic the possible instabilities of the LS counter system under conservative hypothesis. This included the study of a variation of the intrinsic light yield of the LS cocktail, the variation of the optical characteristics of the vial, the change of the quantum efficiencies of the photocathodes of the PMTs and the possible variation of the PMTs gains or of the detector thresholds.

This paper presents the 3-PMTs LS counter developed for this purpose, the specific method for activity comparison and report the results from the validation studies that have been carried out to test the ability to produce long-term stable reference values, in order to compare primary standardized solutions of pure beta emitters.



Figure 1 The TDCR system of the ESIR.

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O01

## Study on the Method of Measuring $^{226}\text{Ra}$ in Water by Liquid Scintillation Counter and its uncertainty evaluation

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Ra is of particular importance owing to the presence of Ra isotopes in all three natural decay series, the relatively long half-lives and short lived progeny of two of the isotopes ( $^{226}\text{Ra}$  and  $^{228}\text{Ra}$ ), the high mobility of Ra in the environment under a number of common environmental conditions and the tendency of Ra to accumulate in bone following uptake into the body. The process of concentration of  $^{226}\text{Ra}$  and its parent nuclides in percolating groundwater is affected by the availability and solubility of the nuclides in the rocks. Thus  $^{226}\text{Ra}$  is one of the important radionuclides in water radioactivity monitoring. At present, the commonly used method in drinking water standard in China is to measure its daughters radon or radium gross  $\alpha$  radioactivity after left the sample for a month by  $\alpha$  counting method.

A rapid method for the determination of  $^{226}\text{Ra}$  in water by liquid scintillation spectrometry has been developed. The radium in the water sample was coprecipitated and purified by adding barium sulfate carrier. The pulse shape discrimination of the instrument (PSD) and the stability of the suspension solution system are the most important factors affecting the measurement results. Standard samples was used to optimize the instrument discriminator Parameters in the Alpha/beta mode to gain better PSD value and the alkalinity of the solution were investigated to improve the stability of the solution. The analysis process is shown in figure 1.

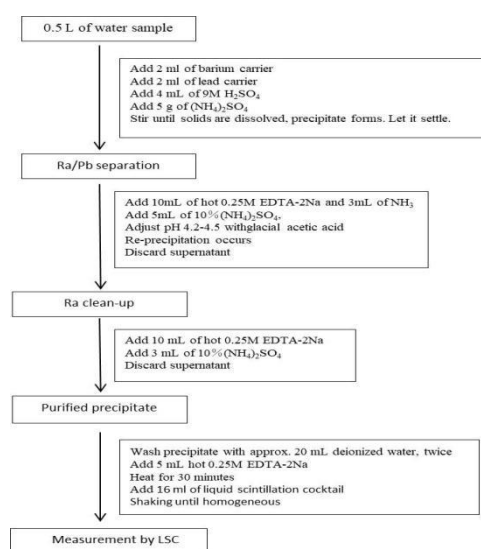


Figure 1 Process of analysis of radium-226 by liquid scintillation counter

The method recovery of  $^{226}\text{Ra}$  in water was 93%~102 %, detection limit was 0.01Bq/L. The relative expanded uncertainty of  $^{226}\text{Ra}$  activity concentration in water which main from counting rate

and whole process recovery rate deviation was 7% ( $k=2$ ). It is potential used for measuring radium in environmental water.

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O02

### **Column Extraction as Rapid Sample Clean-up Method for Tritium (H-3) Analysis**

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Keywords: Tritium H-3, Column Extraction, Rapid Method

Traditionally, water samples are treated by distillation prior to tritium analysis. The distillation setups to efficiently distill small (<100 mL) and large (100 to 500 mL) volume samples are different. Types of reagents recommended for oxidation to reduce the likelihood of collecting other volatile isotopes are also different for different established methods. The entire sample preparation process can be tedious and time consuming.

In our study, test methods covered (i) direct measurement (without clean-up), (ii) distillation and (iii) column extraction (using EICHRON Tritium Column) prior to sample measurement using liquid scintillation counter. The sample was prepared using distilled water spiked with known amount of tritium and other beta-emitting radionuclides. The results showed that clean-up is essential if the sample contained interfering radionuclides. Both distillation and column extraction methods presented similar results and the time taken to perform the steps for treatment of one sample is almost equivalent. However, the design of the column extraction allows multiple samples to be processed at the same time as compared to the distillation setup; permitting higher sample throughput. The drawbacks are the high cost of the column (one-time use) and the lack of comprehensive studies done on the column with regards to the binding properties of resins used in removal of all interfering ions (only tested few in our case) which may affect the tritium analysis. Nevertheless, column extraction method could be more suitable for rapid analysis during emergency response where fast turnaround time is expected.

O03

## New progress in Čerenkov-TDCR method study for pure beta radionuclides

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**Abstract:** Photons of visible radiation are produced when a charged particle travels through a transparent medium when its speed is greater than the speed of light in that medium. This type of radiation is named by the name of Russian physicist P. A. Čerenkov to commemorate his basic research on these phenomena<sup>[1]</sup>. Such visible radiation was first applied to measure the radioactivity in 1953<sup>[2]</sup>. However, the method for standardization of radionuclides which base on Čerenkov radiation was proposed until 1995 and then such approach is extended for a system with three photomultiplier tubes<sup>[3],[4]</sup>.

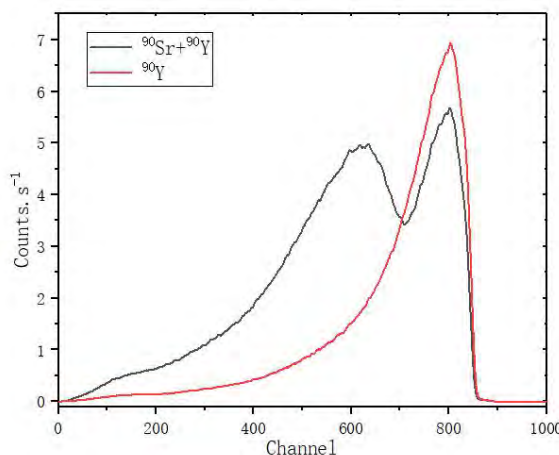
In this paper, the triple to double coincidence ratio (TDCR) counting system in NIM (National Institute of Metrology, China), as shown in Fig. 1, was applied to measure high-energy pure beta radionuclides in aqueous and hydrochloric acid solution. For the analysis of the result, the Čerenkov-TDCR (Č-TDCR) analytical model developed at PTB was adapted<sup>[5]</sup>. The dependence of the refractive index of water on the wavelength and the asymmetry of three PMTs in system are considered.



**Fig. 1.** TDCR counting system in NIM

Before measuring the activity of nuclides, the anisotropy coefficient in the model was derived by comparing the two results of  $^{90}\text{Sr}/^{90}\text{Y}$  obtained from Č-TDCR method and LS-TDCR method. Then the anisotropy coefficient was used to calculate the efficiency of other pure beta radionuclides, such as  $^{32}\text{P}$  and  $^{204}\text{Tl}$ . However, the specific activity of these two nuclides derived from Č-TDCR and LS method were not in agreement. To analyze whether it was the Sr that caused the inconsistency of the results<sup>[5]</sup>, a column packed with SR resin was used to purify Y in effluent from SR column by selective adsorption of Sr. The effluent was concentrated by evaporative solvent method and then diluted with a non-radioactive carrier solution (30  $\mu\text{g/g}$  Sr and 30  $\mu\text{g/g}$  Y in 0.1 M HCl). After shaking at room temperature, the diluted  $^{90}\text{Y}$  solution was prepared into Č-TDCR and LS measurement samples. The energy spectrum (Fig. 2) of LS

samples for the obtained  $^{90}\text{Y}$  product and continuous observation of decay counting indicates that  $^{90}\text{Sr}$  was removed completely.



**Fig. 2.** LS spectrum of  $^{90}\text{Sr}+^{90}\text{Y}$  and  $^{90}\text{Y}$

Then the new anisotropy coefficient in the model was derived by comparing the results of  $^{90}\text{Y}$ . The activities of  $^{32}\text{P}$  and  $^{204}\text{Tl}$  were calculated by introduced the new coefficient, and the results were compared with LS-TDCR and C/N methods. The activity of the two nuclides were in good agreement with LS result within the uncertainty range(the deviation between Č-TDCR and LS method is 0.60% for  $^{32}\text{P}$  and -0.47% for  $^{204}\text{Tl}$ ).

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O04

## **Encountered challenges in determining Difficult-To-Measure radionuclides from nuclear decommissioning waste matrices by LSC**

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**Abstract:** Radioanalytical methods for determining Difficult-To-Measure (DTM) radionuclides from different decommissioning matrices have been developed and refined in two simultaneous and interlinking, domestic and Nordic projects, in Finland during the last three years. In domestic VAMMA and DEMONI projects (funded by national radioactive waste management program), different approaches have been tested for dissolving sample matrices (activated steel, concrete, spent ion exchange resin) and separating radionuclides of interest. In Nordic DTM DECOM project (part of NKS-B research program), the selected radioanalytical methods have been tested in intercomparison exercise for the same sample matrices. Outcome of the projects has been published in [1-5] so far.

Activated pressure vessel steel was the sample matrix, containing <sup>55</sup>Fe, <sup>63</sup>Ni, <sup>14</sup>C, and <sup>60</sup>Co as the main radionuclides of interest in the first project year. It was concluded, that several phenomena challenged the LSC measurements of <sup>55</sup>Fe, including luminescence, quenching of the K-shell Auger and X-ray emissions, and acid tolerance of the liquid scintillation cocktail. Analysis of <sup>63</sup>Ni concluded that interference from <sup>60</sup>Co in the LSC spectrum can be significant, if <sup>63</sup>Ni sample is not properly purified. The LSC measurements of <sup>14</sup>C may contain spectral interference from other volatile radionuclides and persistent chemiluminescence in NaOH trapping solution.

Activated concrete was the sample matrix in the second project year, including non-volatile <sup>55</sup>Fe, <sup>63</sup>Ni, <sup>41</sup>Ca, and volatile <sup>3</sup>H, <sup>14</sup>C, and <sup>36</sup>Cl beta emitting radionuclides of interest. Major challenge in DTM determination was the low radioactivity level of the concrete. As volatile DTMs, <sup>3</sup>H and <sup>36</sup>Cl have similar challenges in the sample preparation as <sup>14</sup>C. Several <sup>41</sup>Ca results were submitted in the intercomparison, but all laboratories reported difficulties in the LSC measurement, i.e. spectral interference and significant quenching of weak X-rays of <sup>41</sup>Ca. Quenching occurred also with determination of <sup>55</sup>Fe by LSC, due to high concentration of Fe in the concrete and high sample mass (requirement from low activity concentration level).

It has been found out during the intercomparison project that besides radioanalytical problems originating from sample composition, radiochemical impurities and quenching in LSC, other concerns exist in inter-laboratory comparison. Various and sometimes divergent methods for calculating detection limits and activity uncertainties are in use in the laboratories, preventing true harmonisation of the intercomparison results.

Third, on-going project year focuses on analysing spent ion exchange resin, expected beta emitters of interest being mainly <sup>55</sup>Fe, <sup>63</sup>Ni and <sup>90</sup>Sr. The results for DTM radionuclides in the resin from both domestic and Nordic projects will be published in 2022.

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O05

## Selectivity analysis of $^{99}\text{Tc}$ determination by Liquid Scintillation Counting (LSC) in the field of nuclear decommissioning

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### 1. Introduction

$^{99}\text{Tc}$  is one of the most significant fission product of  $^{235}\text{U}$ , with a half-life of  $211.5 \cdot 10^3$  y. It is one of the usual isotopes in the radionuclide inventory of nuclear power plants (NPPs) [1 – 4], and therefore, it has to be considered in decommissioning.

$^{99}\text{Tc}$  is a high-energy (293.8 keV) beta emitter, so its measurement is commonly carried out by liquid scintillation spectrometry (LSS), after sample treatment and Tc radiochemical isolation.

In routine environmental monitoring, radiochemical isolation by ion exchange on a TEVA column provides pure  $^{99}\text{Tc}$  spectra and high chemical yields. However, in the field of decommissioning other problems arise, not only because some of the samples are chemically very complex, leading to low chemical yields, but also because some of the samples may contain high activities of other radionuclides potentially capable of interfering with  $^{99}\text{Tc}$  during its measurement.

For these reasons, in this work an alternative Tc radiochemical isolation by using Empore™ Tc Rad Disk is proposed and compared with the conventional one by using TEVA resin, in terms of selectivity and time and financial costs.

### 2. Material and methods

In this work, radionuclide inventories of LWR (light water reactor) and PHWR (pressurized heavy water reactor) plants [1 – 4] have been considered. In them, possible interferers in  $^{99}\text{Tc}$  spectrum have been identified according to these criteria: their expected activity is almost  $> 0.1$   $^{99}\text{Tc}$  activity; their decay mode is beta or electron capture; and their half-life is greater than 2.7 y, as decommissioning of NPPs takes, on average, 15 y to be completed [1]. Short-lived radionuclides have only been considered when they are in secular equilibrium with their parent.

After that, for each chosen interferer, 100 mL of 0.2 M HCl have been spiked with around 30 Bq of it, activity 1000 times greater than  $^{99}\text{Tc}$  detection limit.

Half of each dissolution has been passed through a TEVA column; and the resin has been added into a 20 mL polyethylene vial, together with 10 mL of Ultima Gold LLT scintillation cocktail.

The other half of each dissolution has been filtered using an Empore™ Tc Rad Disk, at a flow rate of  $5 - 10 \text{ mL min}^{-1}$ . Then, the disk has been placed into a 20 mL polyethylene vial, according to Fons-Castells et al. [5], and mixed with 20 mL of UG LLT.

Blanks have been prepared in the same way as samples; and calibration sources for  $^{99}\text{Tc}$  and interferers, when necessary, have been prepared by spiking the resin and disks with a known amount

of activity.

Finally, vials have been stored for 6 hours in the dark inside an ultra-low background liquid scintillation spectrometer 1220 QUANTULUS™, from Perkin Elmer, and measured by using  $^{14}\text{C}$  protocol.

### 3. Results and discussion

According to the criteria described in the previous section, the main possible interferers in  $^{99}\text{Tc}$  spectrum are  $^3\text{H}$ ,  $^{36}\text{Cl}$ ,  $^{55}\text{Fe}$ ,  $^{60}\text{Co}$ ,  $^{59}\text{Ni}$ ,  $^{63}\text{Ni}$ ,  $^{90}\text{Sr}/^{90}\text{Y}$ ,  $^{93}\text{Zr}$ ,  $^{94}\text{Nb}$ ,  $^{137}\text{Cs}$ ,  $^{154}\text{Eu}$  and  $^{241}\text{Pu}$ .

Among those radionuclides,  $^{60}\text{Co}$ ,  $^{63}\text{Ni}$ ,  $^{90}\text{Sr}/^{90}\text{Y}$  and  $^{137}\text{Cs}$  have been chosen as potential interferers to cover a wide energy range, from 66.980 keV ( $^{63}\text{Ni}$ ) to 2823.1 keV ( $^{60}\text{Co}$ ). Using  $^{14}\text{C}$  protocol for measurement, low-energy signals are negligible in the spectrum; so, radionuclides with emissions of less energy than 66.980 keV ( $^3\text{H}$ ,  $^{55}\text{Fe}$  and  $^{241}\text{Pu}$ ) have not been considered in this work.

Results of the tests carried out are summarised in the following table:

	$^{60}\text{Co}$	$^{63}\text{Ni}$	$^{90}\text{Sr}$	$^{90}\text{Y}$	$^{137}\text{Cs}$
Decay mode	$\beta^-$	$\beta^-$	$\beta^-$	$\beta^-$	$\beta^-$
Energy (keV)	2823.1	66.980	545.90	2278.7	1175.6
Half-life (y)	5.27	98.7	28.8	2.67 d	30.1
Interference using TEVA resin	No	No	No	63.42 %	No
Interference using Tc Rad Disk	0.42 %	No	0.14 %	0.14 %	0.16 %

As we can see in the table, only  $^{90}\text{Y}$  is retained in the resin; while  $^{60}\text{Co}$ ,  $^{90}\text{Sr}/^{90}\text{Y}$  and  $^{137}\text{Cs}$  are retained in the disk, but in much less amount. However, the shorter half-life of  $^{90}\text{Y}$  allows interference to reduce over time.

In this work, it has also been observed that doubling the activity of  $^{60}\text{Co}$ ,  $^{90}\text{Sr}/^{90}\text{Y}$  and  $^{137}\text{Cs}$ , their retention on disk increases around 10 times; so, depending on their activity, interferences may be reduced. However, in view of the spectra in Figure 1, the best option to solve this problem may be spectrum deconvolution.

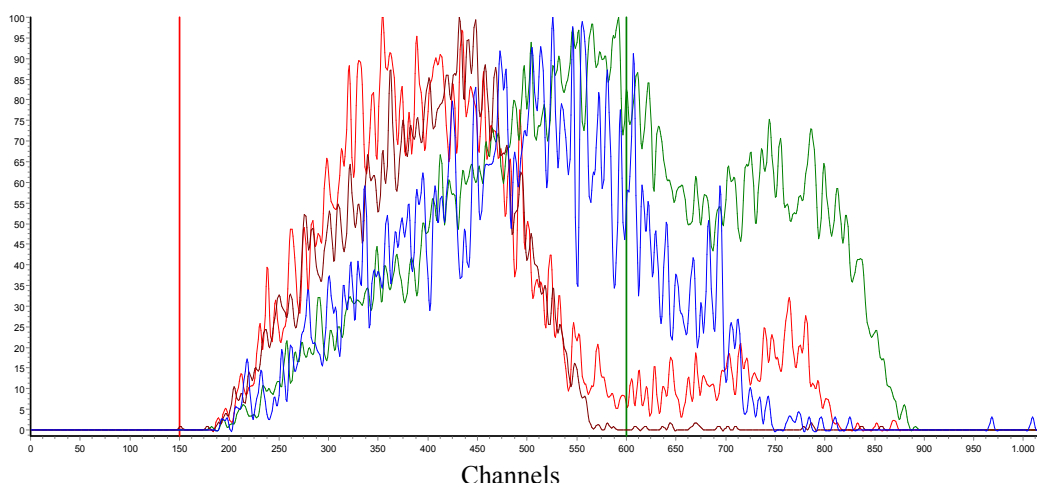


Figure 1 Spectra of  $^{60}\text{Co}$  (red),  $^{90}\text{Sr}/^{90}\text{Y}$  (green),  $^{99}\text{Tc}$  (brown) and  $^{137}\text{Cs}$  (blue). Window for  $^{99}\text{Tc}$  is between channels 150 and 600.

Finally,  $^{99}\text{Tc}$  yield using TEVA resin is around 80 %, while using Tc Rad Disk is 97 %. Conventional Tc radiochemical isolation takes, on average, 3 days; but using disks, isolation reduces to 30 minutes. Regarding financial costs, both methods are similar.

#### 4. Conclusions

After comparing two methods with different Tc radiochemical isolation, it can be concluded that TEVA resin is more selective than Tc Rad Disk, but slower; and financial costs are similar.

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O06

## The effect of $\gamma$ rays on the determination of $\alpha$ or $\beta$ emitters by LSC

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Since  $\alpha$  or  $\beta$  decay may leave the daughter nucleus in an excited state,  $\alpha$  or  $\beta$  radiations are usually accompanied by emitting  $\gamma$  rays. The effect of  $\gamma$  rays on the counting efficiency of  $\alpha$  or  $\beta$  particles should be taken into consideration for the absolute measurement of radioactivity through counting  $\alpha$  or  $\beta$  particles. This kind of effect varies not only with the energy of the  $\gamma$  ray itself but also with the following aspects: (1) is the  $\gamma$  ray coming from an  $\alpha$  emitter or a  $\beta$  emitter? (2) is the  $\gamma$  ray coincident with the  $\alpha$  or  $\beta$  particle? (3) is the function of anticoincidence shield (using guard detectors) set as active or inactive? (4) is the function of  $\alpha/\beta$  discrimination applied? From the investigations of low quenched samples containing  $^{241}\text{Am}$ ,  $^{137}\text{Cs}$ , or  $^{60}\text{Co}$  by liquid scintillation counting (LSC) using two-vial configuration method or different scintillation volume method, we draw some conclusions about the effect of  $\gamma$  rays on the determination of  $\alpha$  or  $\beta$  emitters. For  $\beta$  determination, the effect of  $\gamma$  rays mainly depends on whether they are in coincidence with the  $\beta$  particles. While for  $\alpha$  determination, the counting efficiency of  $\alpha$  particles can hardly be affected by the  $\gamma$  rays if a proper  $\alpha/\beta$  discrimination parameter is applied. When coincidence exists, summation effects can offset the effect of the  $\gamma$  rays.

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O07

## **Comparison of mathematical methods to improve LSC background – Hidex Digital Pb Shield (DigPb) and Guard Compensation Technology (GCT)**

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The background evaluation is a key point in the analysis of environmental samples due to extremely low amount of radioactivity usually present. Manufacturers of the counters are often comparing the sensitivity of the counters using Figure of Merit (FOM) by assuming the background counts rate (B) follows Poisson distribution ( $FOM = E^2/B$ ). This is, however, misleading if B is not following Poisson distribution caused e.g. by system malfunctioning.

We have compared two mathematical methods for improving the background and sensitivity of Liquid Scintillation Counting (LSC) measurements: Hidex Digital Lead Shield (DigPb)<sup>2</sup> and Guard Compensation Technology (GCT)<sup>3</sup> patented by Perkin Elmer.

DigPb method is deterministic double label counting method where one component (CPM of background) is known. Calibration of the assay is performed by measuring spectral distribution of active sample (A), spectral distribution of background (B) and total background CPM. Poisson-weighted least squares fitting is employed for unknown samples to find the most probable isotope CPM (A). If there is difference between spectral distribution of A and spectral distribution of B, the uncertainty (fluctuation) of A CPM improves. The improvement is similar to “normal” background subtraction in instrument with lower B CPM. As consequence, the assay sensitivity is improved.

GCT is patented by Harazin and implemented in PerkinElmer TriCarb BGO instrument model with new model name Quantulus GCT 6220. Evaluation of the patent yields in a finding that the use of GCT results in lower background CPM, but uncertainty does not decrease- GCT data does not follow Poisson statistics. As consequence, the FOM nor detection limit of the assay is not improving.

In the presentation we'll describe in detail the principle of both methods, evaluate the statistics and how they affect the uncertainty of the results – directly correlating to the assay sensitivity.

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2. Hidex Technical Note 513-012 *Digital Pb Shield*

O08

## Optimization of the direct LSC method for determination of biogenic component in liquids by applying $^{14}\text{C}$

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The  $^{14}\text{C}$  method can be successfully applied for determination of biogenic component in any type of samples and different measurement techniques can be applied. For a special case of liquid fuels, direct method by measurement  $^{14}\text{C}$  activity of liquid fuel samples in liquid scintillation counter (the „direct LSC method“) has been recognized as a powerful and reliable method of determination of biogenic component ( $f_{\text{bio}}$ ). In the Ruđer Bošković Institute (RBI) Radiocarbon Laboratory we have been implemented our own data evaluation method based on the quench properties of fuels (SQP(E) – Standard Quench Parameter in LSC Quantulus 1220) [1]. We participated in the international intercomparison study ILC/2018 *Content of biocomponent in liquid fuel samples*, which was organized in 2018 by the Institute of Ceramics and Building Materials (Opole, Poland). Here we compare our  $f_{\text{bio}}$  results with the  $f_{\text{bio}}$ -ILC official results and report optimization of the direct LSC method.

Laboratories obtained seven samples of diesel type of fuel having different colors. Consequently, the samples resulted in different SQP(E) values when the direct LSC methods was applied. Three samples were analyzed by the AMS (Accelerator Mass Spectrometry) method. The LSC results were quantitatively acceptable for four samples with SQP(E) values above 700 (Figure 1), which was taken as the lowest limit of quantitative  $f_{\text{bio}}$  determination. Qualitatively acceptable, but quantitatively unacceptable results, were obtained for two samples with the SQP(E) values between 600 and 700, that was defined as the region of limited applicability. For one of the samples the quenching was high, SQP(E) < 600, and the direct LSC method did not give any result. However, all AMS results were acceptable (Figure 1) since AMS does not depend on the sample color.

The obtained results have been used for further validation and optimization of the direct LSC method. We defined the limits of applicability of the direct-LSC method for both quantitative (SQP(E) > 700) and qualitative results (600 < SQP(E) < 700). A sample of used edible oil (UEO, laboratory code Z-7226) was used to test these limits. The UEO was a dark sample giving the SQP(E) value of 546 (below the limit of qualitative applicability), while the AMS revealed  $f_{\text{bio}} = 97.9 \pm 0.3 \%$  (and  $\delta^{13}\text{C} = -29.6 \text{‰}$ ). We mixed the UEO with the (fossil) petrol (benzene) sample (laboratory code Z-6266), which we used as the background sample ( $f_{\text{bio}} = 0 \%$ ) with good quenching properties (SQP(E) = 864). We monitored changes in the SQP(E) and  $f_{\text{bio}}$  values in UEO-petrol mixtures in the concentration range 0 – 100 %. The total mixture volume was 10 mL and 10 mL of Ultima Gold F scintillation cocktail was added, i.e., the measurement was performed in the same manner as all other organic liquid samples [1]. Mixtures of 10 % and 20 % of UEO gave SQP(E) values of 773 and 759, and the  $f_{\text{bio}}$  was calculated to be  $104.0 \pm 1.2 \%$  and  $101.1 \pm 1.3 \%$ , respectively, confirming the quantitative region of SQP(E) values above 700. Mixtures containing 30 – 50 % of UEO resulted in SQP(E) values between 671 and 609, while the  $f_{\text{bio}}$  values were about 150 %, confirming qualitatively

acceptable results. Mixtures containing more than 60 % of UEO had SQP values below 600 and  $f_{\text{bio}}$  values had a large spread.

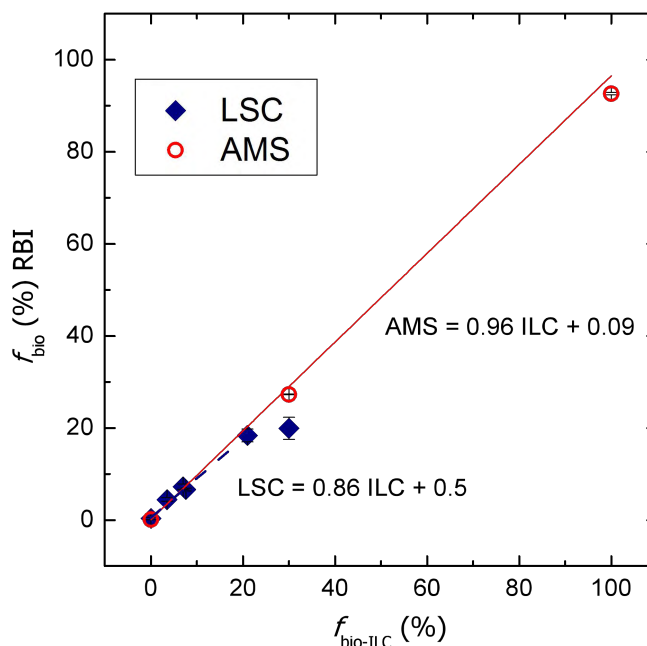


Figure 1 Comparison of the percentage of the biogenic component ( $f_{\text{bio}}$ ) in ILC intercomparison diesel-type samples determined at the RBI by the two measurement methods, LSC (direct method by liquid scintillation counting) and AMS (accelerator mass spectrometry) with the official results  $f_{\text{bio-ILC}}$ .

The presented results showed that the direct LSC method with an evaluation method developed in our laboratory was suitable for determination of the  $f_{\text{bio}}$  in liquid fuels, providing the correctly defined limits of applicability for highly quenched samples. The RBI Laboratory has a possibility of applying also AMS  $^{14}\text{C}$  measurement technique that can satisfactorily determine  $f_{\text{bio}}$  also in highly quenched samples, but the AMS technique is more complicated and more expensive.

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O09

## A summary on the monitoring method verification for tritium analysis in water

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**Abstract:** The state analysis method for tritium in water of China, with an official number of GB 12375, was first published in 1990 and haven't been revised for almost 3 decades. Compared to the ISO<sup>1,2</sup> and ASTM<sup>3</sup> technical standards, which was always revised in a 3-5 years interval, the currently version of GB 12375 is definitely out of time. Therefore, the Regulatory agency of China urged a revision program.

Since the suspension of aboveground nuclear tests, tritium level in the atmosphere and other environmental medias have been decreased year by year, which requires liquid scintillation counters with better performance for low background counting. As a matter of fact, for surface water and biota mediums in many districts, tritium activity concentration are far below the lower limit of detection of most LSCs<sup>4,5,6,7</sup>, which means that an electrolysis enrichment process is in need, thus aiming to acquire an accurate monitoring result. Currently, basic electrolysis and solid polymer electrolysis (SPE) are two normally used enrichment methods, which use alkaline and solid polymer as electrolyte<sup>1,8</sup>, respectively.

In this study, a revision draft of GB 12375 was written initially, SPE process was introduced for the first time. Analysis procedure for tritium in water was composed of the following steps. Potassium permanganate was added into the collected water sample, impurities in the sample was removed via distillation under normal pressure, for water samples which contains a tritium activity concentration below instrument LLD level, tritium in water was enriched through basic or SPE electrolysis, for the other samples, no enrichment was conducted. For purified water sample, as well as the enriched sample, water was mixed with liquid scintillation cocktail with a certain ratio, so as to form a homogeneous counting mixture, the mixture was hence to be counted in a low background LSC.

To verify the accuracy and precision of the procedure described in the draft, six samples, including three environmental samples and three reference sample, were chosen to conduct a series of inter comparison experiments with exactly the same procedure as the draft. Each sample was pretreated and analyzed in six individual radiochemical analysis labs, either.

Data were collected and processed by using the preset statistical models. LLD value of the revised draft turned to be lower than 0.14 Bq L<sup>-1</sup> under typical counting conditions, while the repeatability limit (r) and reproducibility limit (R) turned to be below 0.85 Bq L<sup>-1</sup> and 0.89 Bq

L<sup>-1</sup>, respectively. The relative error between reference sample and the total average measured data of six labs turned to be below 9.48%, either.

As a result, the revised draft turned to be an appropriate monitoring procedure for analyzing tritium in water.

**Key words: tritium; radiation monitoring; radiochemical analysis.**

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O10

## **Validation of radon-222 measurement in drinking water using LSC under the ISO/IEC 17025 criteria**

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A method for the measurement of radon-222 in drinking water using LSC has been validated under the ISO/IEC 17025 criteria. Initially, several methods were tested: degassing method (emanometry), gamma spectrometry and homogeneous counting using LSC (Pujol and Perez-Zabaleta, 2017). Finally, two-phase counting method (selective extraction of radon) using LSC was selected. This method has several advantages: sample manipulation is easy, detection system is provided with automatic sample changer, the volume used is low (that is, 10 ml), radon leak from vials is negligible, the method is selective, short counting time is required (10 minutes) and minimum detectable activity is very low, that is, 0.5 Bq/l.

Two types of cocktails were used from PerkinElmer for two-phase counting method: High Efficiency Mineral Oil Scintillator (product number 6NE9571) and Opti-Fluor O (product number 6013331). One type of 20 ml polyethylene low diffusion vials were used (product number 6000477 from PerkinElmer). 10 ml of liquid scintillation cocktail were used.

Samples were measured on two different Ultra Low Level LSC Quantulus 1220<sup>TM</sup> (PerkinElmer) detector system. Counting parameters setting were studied for both cocktails: the alpha/beta discrimination parameter (PSA) and counting window. In the case of Opti-Fluor O cocktail, the optimum PSA was set up in 60 and the counting window for <sup>222</sup>Rn measurement from channel 740 to channel 910 and, in the case of Mineral Oil cocktail, the optimum PSA was set up in 70 and the counting window for <sup>222</sup>Rn measurement from channel 700 to channel 860. The counting window depends on the detector system used. Besides background were determined for both cocktails.

Although several validation parameters are applicable for all types of test (ISO/IEC 17025, 2017), the validation parameters considered in our quality procedure were: precision, accuracy, detection limit and uncertainty estimation. Counting statistics, efficiency, sample manipulation and repeatability were considered for uncertainty estimation. In this work, it was observed that sample preparation can lead to significant bias in the <sup>222</sup>Rn activity due to radon-loss so it was considered in the uncertainty estimation. Also the influence of sample handling on radon-loss was tested and quantified by other authors (Jobbagy et al., 2018).

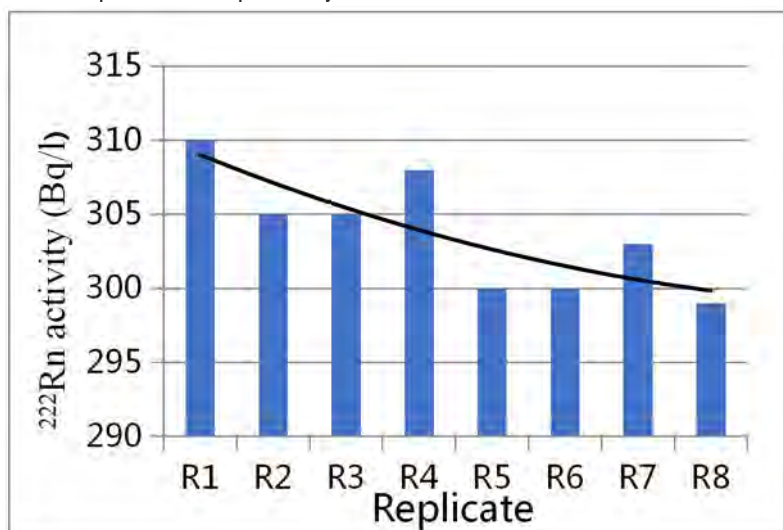


Figure 1  $^{222}\text{Rn}$  activity (Bq/l) in the eight replicates prepared when sample is transferred to the vial.

Additionally, the validation of the method included the comparison of results achieved with other methods described previously (degassing method, gamma spectrometry and homogeneous counting using LSC) and two interlaboratory comparisons.

In order to assuring the validity of the tests undertaken by the laboratory some internal quality controls should be done and trends should be analyzed. This monitoring shall be planned and reviewed. It includes: background and efficiency determination, precision and accuracy. Furthermore regular participation in interlaboratory comparisons or proficiency-testing programmes are compulsory.

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O11

## Determination of PDD in $^{222}\text{Rn}$ Calibration for Water Analyses by Liquid Scintillation Counting

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### Abstract:

In order to perform the calibration of the liquid scintillation counter Tri-Carb 3170 TR / SL (Perkin-Elmer) for Rn-222 determination in waters, 10 standard samples of Ra-226 were prepared. To measure Rn-222, the secular equilibrium  $\text{Ra-226} \rightleftharpoons \text{Rn-222}$  must be achieved and then the PDD<sup>1</sup> value is adjusted until the percentage of alpha events reach a plateau. When using this counter, it is not possible to make the automatic determination of the PDD value. For that reason, one of the standards was used for PDD evaluation. Before the adjustment start, the flask was vigorously agitated to extract Rn-222 to the scintillation cocktail. After 3 h stabilization, the tuning was initiated at PDD 80 with 15 min counting time. With an increment of 10, measures were taken until PDD 200 one by one. A graphic of the event fraction versus PDD was created and after analyzing the existing plateau, it is possible to observe the variation where the fraction of events was at a minimum. The PDD value was established in the middle of that plateau. 10 standard samples and 10 blank samples were measured in an assay type Alpha/Beta at the determined PDD. After the calibration process was finished, some control samples were measured to evaluate the data confidence and the values were accepted.

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<sup>1</sup> The optimal value of the Pulse Decay Discriminator (PDD) minimizes the probability of alpha events being counted as beta events and vice versa. The value of the ideal discriminator is where the misclassification of alpha and beta are minimal.

## O12

### Determination of $^{228}\text{Ra}$ in natural water by liquid scintillation counting and its application in environmental monitoring

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**Abstract:** The results of the First National Pollution Source Census in 2007 and the Second National Pollution Source Census in 2017, organized by the Ministry of Ecology and Environment of China show that the radiological impact of thorium and its decay series daughters, especially  $^{228}\text{Ra}$ , is significant in the NORM industrial activities, such as rare earths and zircon minerals mining and extraction. However, the measurement standard of  $^{228}\text{Ra}$  in the environment has not been issued in China. Determination of  $^{228}\text{Ra}$  in the environment has not been used for routine radiation environmental monitoring purposes. The analysis of  $^{228}\text{Ra}$  in China now is mainly based on the radiochemical separation of  $^{228}\text{Ac}$ , a daughter of  $^{228}\text{Ra}$ , and then the activity concentration of  $^{228}\text{Ac}$  is determined by  $\beta$  counter measurement or gamma spectrometry. So as to determinate the activity concentration of  $^{228}\text{Ra}$ . The analysis method is quite practical. However, the methods are non radionuclide-specific by  $\beta$  counter measurement and poor efficiency by gamma spectrometry. Recently, the IAEA recommended a method of determination of  $^{228}\text{Ra}$  in environmental samples, especially in liquids by liquid scintillation counting (LSC). The activity concentration of  $^{228}\text{Ra}$  in environmental samples can be quickly determined. Based on the method recommended by IAEA, this paper established a technical method to analyze the activity concentration of  $^{228}\text{Ra}$  in environmental water by LSC. This method has been applied to monitoring of environmental water in Baotou, Inner Mongolia, where it is the well-known rare earths production. The activity concentrations of  $^{228}\text{Ra}$  in three monitoring locations along the Yellow River within of Baotou district were reported for the first time. The results showed that the activity concentrations of  $^{228}\text{Ra}$  in water from the locations of Dengkou, Zhaojunfen and Huajiangyingzi were  $42 \pm 3.6$  mBq / L,  $63 \pm 4.7$  mBq / L,  $21 \pm 2.3$  mBq / L, respectively. The activity concentrations of  $^{238}\text{U}$ ,  $^{226}\text{Ra}$  and  $^{232}\text{Th}$  were 61.9 mBq / L, 7.45 mBq / L, 1.45 mBq / L in Dengkou, 50.8 mBq / L, 4.29 mBq / L, 1.86 mBq / L in Zhaojunfen, 44.4 mBq / L, 11.8 mBq / L, 1.66 mBq / L in Huajiangyingzi, respectively. The activity concentration of  $^{228}\text{Ra}$  in water from the Yellow River in Baotou is higher than that of thorium, and also higher than that of  $^{226}\text{Ra}$ .

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O13

## **Evaluating the robustness of analytical method for the determination of Tritium (H-3) in seawater using Plackett-Burman designs**

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**Keywords:** Tritium H-3, Robustness, Plackett-Burman

The procedure for tritium analysis employs distillation as the sample clean up step followed by liquid scintillation counting with the addition of scintillant. There are numerous test parameters from sample preparation for distillation to sample conditioning prior to actual measurement. In our study, seven test parameters for the determination of tritium (without enrichment) were investigated using Plackett-Burman designs. The results highlighted the critical test parameters and showed that the method was robust for analysing tritium in seawater varying from pH 2-12, distilled at the rate of 0.75-2 mL/min and dark-adapted between 0 to 24 hours. The measurements were performed using Perkin Elmer Quantulus 1220 and the detection limit for the method was 4 Bq/L.

O14

## **The applications of compact liquid scintillation detectors in fusion neutron diagnostics at EAST**

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Liquid scintillation detectors (LSD) have high detection efficiency and good time resolution. Because of their good neutron/gamma-ray discrimination capability and compact size, the liquid scintillators have been used to measure neutron yield and neutron energy spectrum in D-D or D-T fusion experiments in magnetic confinement devices, like HL-2A, EAST, JET, and ITER tokamaks. The neutron emission spectroscopy (NES) measurements using a compact LSD for neutral beam injection (NBI) heated plasma have demonstrated their capability in fast-ion physics studies at Experimental Advanced Superconducting Tokamak (EAST) [1,2]. Under NBI heating scenarios, the time trace of the neutron yield monitored by LSD is similar with the one monitored by a standard <sup>235</sup>U fission chamber. The experimental pulse height spectra (PHS) are also similar with the simulated ones generated by folding simulated neutron energy spectra with detector response functions. In addition, based on recent experimental PHS measurements in EAST, the synergy effect between ion cyclotron resonance heating (ICRH) and NBI was observed by LSD which provides the experimental evidences with a clear shape modification in energetic tail of the PHS. The applications of the compact LSD at EAST demonstrate its capability in fusion neutron diagnostics and fast-ion physics studies.

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O15

## **A new method for measuring uranium and plutonium aerosols in the atmosphere by using liquid scintillation spectrometer**

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A new method for measuring uranium and plutonium aerosols in atmospheric environment based on a liquid scintillation spectrometer with  $\alpha/\beta$  discrimination function is put forward. Firstly, obtain the glass fiber filter paper or polypropylene filter membrane sample in the atmosphere by filtering and dust collection method, after pulverization, microwave digestion, hydroxide co-precipitation, acid concentration system conversion, and valence state adjustment, the upper column solution was obtained. Secondly, the upper column solution is sequentially passed through a series chromatographic column composed of TEVA resin and DGA resin to realize the separation and purification of uranium and plutonium. Thirdly, the TEVA resin is eluted with dilute hydrochloric acid to obtain the plutonium eluent, and the DGA resin is eluted with dilute nitric acid to obtain the uranium eluent. Finally, a certain amount of eluent is mixed with a certain amount of scintillation liquid, and the  $\alpha$  activity of the sample is measured with a liquid scintillation spectrometer with  $\alpha/\beta$  discrimination function. As a conclusion, the activity concentration of uranium and plutonium nuclides in the radioactive aerosol are calculated.

Experiments show that the overall recovery rate of this method was 84.06%, and the detection limit was  $1.4862 \times 10^{-6}$  Bq/m<sup>3</sup>. Using this method to measure atmospheric samples under certain conventional conditions, the results show that the activity concentration of uranium in the atmosphere was  $1.1439 \times 10^{-4}$  Bq/m<sup>3</sup>, and the presence of plutonium was basically not detected.

O16

## **Study on field effect in high energy density physics using liquid scintillator**

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In the research of high energy density physics, with the deepening of study, field effect has attracted more and more attention. This work introduces the field effect research in two typical cases. 1) In the implosion process of laser driven inertial confinement fusion (ICF), when a strong shock propagates through the material interface in the gas, due to the huge density and temperature gradient in the front of the shock wave, the mass and charge state differences between different particles will drive the charge separation, and then produce an electric field. Since the electric field evolves spontaneously with plasma density and pressure, diagnosing the electric field will help to understand the shock wave structure and its evolution in the kinetic region, and then understand the deviation between the kinetic effect and radiation hydrodynamics. Based on Shenguang II upgraded laser facility, this study explores the self-generated field distribution and evolution caused by the strong shock generated by indirectly driving CH material in low-density gas. The deflection images of protons by the self-generated field at different times were successfully obtained by RCFs, and the self-generated field intensity and distribution are analyzed. Further, the neutrons produced by the interaction between D ions reflected by the self-generated field and LiF are measured by liquid scintillator detector. The results show that the peak intensity of self-generated electric field is about  $10^7$  V/m. 2) Study on nuclear reaction in colliding plasma. In the process of CD plasma collision, neutrons will be produced in the collision region. The neutron generation mechanism may include beam target reaction and field effect, which is still controversial. Based on the Shenguang II upgraded laser facility, the experimental research is carried out, and the neutron yield is measured by liquid scintillator. The experimental results show that the neutrons produced by CDCD collision are much more than those produced by single CD and CDCH; It shows that neutrons are mainly produced by D ion collision. Because in the collision region, the collision mean free path of D ions is much larger than the target size, it is speculated that in the collision region, plasma convection filamentation will restrict D ions and greatly enhance the collision cross section in a small range. However, more detailed conclusions need to be further studied.

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O17

## Optical Improvements of Plastic Scintillators by Nanophotonics

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**Abstract:** We discuss the optical improvements of plastic scintillators by nanophotonics, which is mainly focused on the methods of enhancing the light extraction efficiency rather than increasing the internal quantum efficiency of plastic scintillators. This is a typical application of the knowledge of nanophotonics, such as photonic crystals, on plastic scintillators. Total internal reflection strongly limits light extraction. An important breakthrough can be achieved thanks to nanostructures with wavelength scales on the surface of plastic scintillators, giving rise to directional and wavelength-dependent emission enhancement and thus enhancing the performance of scintillator-based detectors. The physical mechanisms are discussed in details. Suitable patterning techniques for the surfaces of plastic scintillators, including nanoimprint lithography and self-assembly lithography, are introduced. As an application, we present a typical example of the enhanced ability of neutron detection by photonic crystals on plastic scintillators. It should be emphasized that the application of nanophotonics principles on light output of plastic scintillators should be combined with traditional coupling methods with the aim at optimization of detection performance.

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O18

## Automated Oxidizer for LSC sample preparation

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Sample combustion is a rapid and effective method for C-14 and H-3 sample preparation to LSC. Organic sample is oxidized to carbon dioxide and water vapor followed by collection of the CO<sub>2</sub> and H<sub>2</sub>O into separated vials. If the sample contains carbon-14 and tritium, the oxidation products will have the isotopes incorporated as <sup>14</sup>CO<sub>2</sub> and <sup>3</sup>H<sub>2</sub>O. The oxidized sample is free from chemiluminescence and color quenching because all organic material is oxidized and, inorganic portion either remains in the sample loading cup or flows through the system in a gas format. The Hidex 600 OX Oxidizer is a modern combustion instrument for automated sample processing. The system is based on high temperature (900 °C) heater, active oxygen flow and catalysts for optimal sample oxidation. Variety of different sample types such as, tissue, plant, soil and concrete, can be combusted from extremely small size up to 2 grams. Automated gas tightness test before every sample oxidation ensures safe and high-performance sample preparation without the fear of gas leakage.

High performance has been measured in verification testing. The C-14 recovery and memory was 99% and 0.1%, respectively, and H-3 recovery of 94% and memory of 1% was measured. In addition, combustion and collection of Cl-36 has been successful in preliminary testing and, will be developed further for the analysis of nuclear power plant decommissioning waste.



Figure 1. Hidex 600 OX Oxidizer instrument

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O19

### **Analysis of plutonium using PSresin**

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Plutonium radionuclides are found in the environment due to nuclear activities, including weapon testing, satellites and reactor accidents and discharges from nuclear power plants and reprocessing facilities. The measurement of plutonium in the environment is important to control the correct behaviour of all these activities but, also, could be an indicative of accidents or terrorism actions. Therefore, it is necessary to have a technique for rapid measurement of plutonium to provide response in these emergency situations where a large amount of samples need to be measured in a short time. The PSresins raises as an adequate tool to achieve this purpose. The PSresins consist in a plastic scintillation microspheres support coated with a selective extractant. In this system, the radionuclide is selectively retained in the extractant, preconcentrating and purifying it and the measurement is performed in the same resin by the scintillating support, unifying the separation and measure preparation in one step and therefore reducing time, reagents and manpower needed.

The objective of this study is to develop a methodology to determine plutonium using a PSresin based on Aliquat·336.

Separation conditions were established according to the literature using HNO<sub>3</sub> 3 M for the loading step and two rinsing steps with HCl 9 M and 0.5 M nitric acid respectively. Next, the oxidation state adjustment was tested establishing that the best procedure includes iron sulphamate (II), ascorbic acid and sodium nitrite. In these conditions, quantitative retention and 100% detection efficiency was achieved. Next, the interference of several radionuclides in the measurement of plutonium was tested (<sup>238</sup>U, <sup>232</sup>Th, <sup>241</sup>Am, <sup>210</sup>Pb and <sup>99</sup>Tc). The retention of the different interferences was low except for <sup>99</sup>Tc, but taking into account the low occurrence in environmental samples and the position in the spectrum, it would not be a problem in the measurement of plutonium. Final, the use of a stable tracer for the calculation of the PSresin separation yield was studied concluding that gold is suitable for this purpose.

This procedure was applied to the analysis of spiked sea and river water samples, obtaining errors lower than 10% in its quantification.

O20

## **Novel $^{14}\text{C}$ LSC method for biofuels with variable color**

- Direct measurement without a background sample -

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Fossil based fuels are increasingly replaced by fuels of biologic origin, such as hydrotreated vegetable oil (HVO) and bioethanol. Percentage of biogenic component in fuel can be determined with accelerator mass spectrometry (AMS) and LSC with benzene synthesis<sup>1</sup>, both based on  $^{14}\text{C}$  dating principle. These methods provide accurate results but require expensive and time-consuming sample preparation. Direct detection by LSC is an attractive alternative as liquid fuel samples can be measured directly after mixing with scintillation cocktail<sup>2,3</sup>. However, fuel samples are often highly variable and intense in color, which is affecting counting efficiency and background CPM. This is a challenge especially with blends of fuels, as a fossil-based background sample with similar quenching properties is rarely available. We have found a solution to this challenge – a biofuel algorithm, which is using a combination of different types of quench parameters to find background count rate without a fuel specific background sample. The same algorithm finds also counting efficiency for each sample.

The method employs a 3-PMT LS counter enabling triple-double coincidence ratio (TDCR) output and an external standard. The biofuel algorithm is derived from background and efficiency quench curves with chemical and color quenchers. TDCR and external standard quench parameters are used for the calculation of background and counting efficiency of unknown samples.

The algorithm has been tested with various types of fuels and their blends. Here we present results from HVO-diesel samples. The samples were prepared mixing 10 ml of fuel with 10 ml of MaxiLight+ cocktail. CPMs were determined from optimized luminescence free triple coincidence ROI, as fuel samples often emit luminescence. Bio-% was calculated according to DIN 51637 assuming that the specific  $^{14}\text{C}$  activity of carbon is 13.56 DPM/g.

$$\text{Bio-\%} = \text{DPM measured} / (\text{grams of biofuel} * \text{carbon content} * 13.56) * 100$$

Results are presented in the Table 1. The samples were measured with Hidex 300SL super low level TDCR liquid scintillation counter equipped with cooling and external standard source for 420 min. Shorter measurement times 60-120 min can be used for scanning purposes.

Expected	Measured
100	99.0 ± 0.7
78.8	76.8 ± 0.6
57.7	57.7 ± 0.6
38.5	37.7 ± 0.5
19.2	18.9 ± 0.4
13.9	13.8 ± 0.3
9.4	9.8 ± 0.3
4.6	4.3 ± 0.3
2.7	2.7 ± 0.3
0.9	1.2 ± 0.3
0	-0.2 ± 0.2



Table 1. On the left: comparison of HVO-diesel blends: expected bio-% versus bio-% with  $\pm 1\sigma$  uncertainty determined with Hidex TDCR counter using biofuel algorithm.

Figure 1. On the right: compilation of various colored real life biofuel-cocktail mixtures applicable for the biofuel algorithm. The bio percentages determined with the algorithm were in line with AMS results.

## SUMMARY

Biofuel algorithm expands the applicability of direct LSC.

- Direct in-house measurement of bio-%
- No need for a fuel specific background sample
- Minimal sample preparation
- Suitable for wide variety of fuel types, and their blends with variable color
- No luminescence interference
- Detection limit down to 1 wt-% biogenic content
- Limitation: Not applicable for samples with highest color intensity

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O21

## **Radon concentration measurement & radon mitigation in large-scale liquid nitrogen cryogenic system**

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### **Abstract:**

China Dark Matter Experiment(CDEX) pursues the direct detection of dark matter searches using an array of germanium detector at China Jinping underground laboratory(CJPL) for more than a decade. The new projects, CDEX-50dm experiment for dark matter searches and CDEX-300v experiment for neutrinoless double beta decay searches will be employed a couple of strings of bare germanium detectors bathed in an around 1700 m<sup>3</sup> liquid nitrogen tank performed at the new site of CJPL. The cryogenic tank is utilized not only for cooling the germanium detectors but for being shielded from the radioactivity sources. Similar strategy will be the pilot phase of the projected program of CDEX-1T. Radon 222 and its progeny in liquid nitrogen would be one of the significant radioactivity sources according to the noble liquid detector experiments.

The liquid nitrogen materials acts as a shield to reduce the external background level. In the meantime, it does contribute the background. The physics goal for the leading light dark matter searches requires the background level smaller than 0.01 count/keV/kg/day(cpkkd), giving rise to the constraint on the radon sensitivity of 10μBq/kg in liquid nitrogen in order to reach the background level of 0.001 cpkkd. Therefore, high sensitivity radon measurements and radon mitigation in liquid nitrogen becomes a vital challenge.

Before Rn222 decays to the relatively stable Pb210, there will be 3 alpha decays and 2 beta decays. The main method of measuring the radioactivity of radon is to detect the coincident signals of a β from 214Bi decay and an α from 214Po decay. This study focuses on the use of liquid scintillation to measure the radon concentration in liquid nitrogen. The β-α cascade decay from Bi214 decay to Po214 to Pb210 was screened out, combined with the law of radioactive decay balance, and then the radon concentration was determined.

The cascade liquid scintillation radon measurement system independently developed by this subject has been experimentally measured to have a maximum background of about 60μBq, which has reached an internationally good level, and its detector detection limit can be less than 10 mBq/m<sup>3</sup>. In the future, it is expected that the lower detection limit can be reduced by at least one order of magnitude by upgrading the detector structure materials; at the same time, the use of low-temperature activated carbon to achieve radon enrichment is expected to further reduce the detection limit by about 2 orders of magnitude, and ultimately achieve a low radon of <10 μBq/m<sup>3</sup> measurement.

022

## Development of a portable TDCR system at NIM

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### Abstract:

The triple-to-double coincidence ratio (TDCR) method is a very powerful technique and has been successfully applied to the activity standardization of a number of radionuclides [1, 2]. In order to meet the demand of on-site measurement for radionuclides, a portable liquid scintillation TDCR system was developed at National Institute of Metrology (NIM), China. Figure 1 shows the simplified block diagram of the newly developed portable liquid scintillation TDCR system. The system consists small TDCR counter for the measurement of liquid scintillation sources, and digital electronics for coincident pulses processing. The TDCR counter consists of an optical chamber, a light-avoidance housing, and is equipped with three miniature photomultiplier tubes (PMTs). The optical chamber adopts Teflon material with high diffuse reflection efficiency.

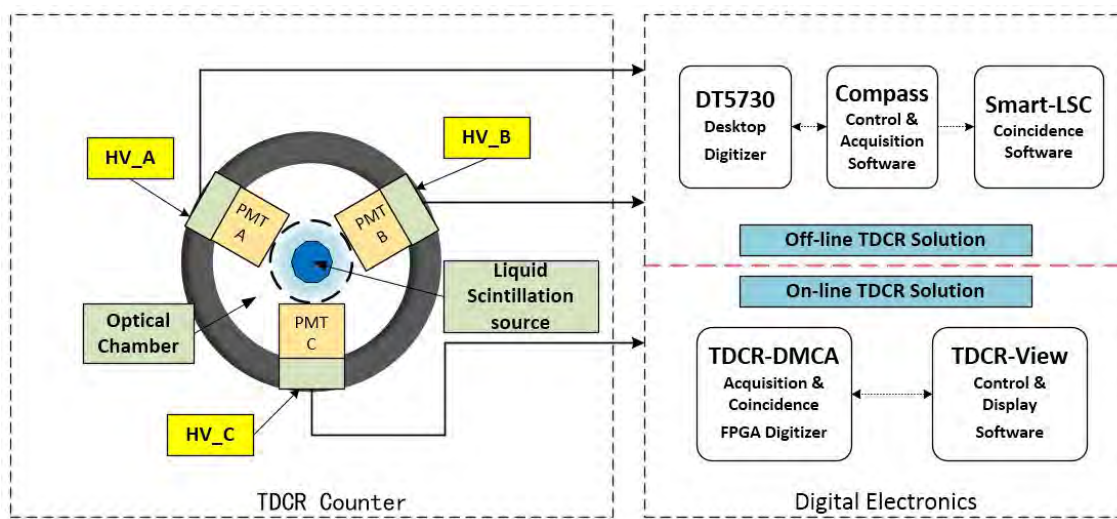


Figure 1 Block diagram of portable liquid scintillation TDCR system at NIM

Two parallel signal processing solutions were used here for TDCR counting. One adopted the off-line TDCR solution based on a stand-alone desktop digitizer of CAEN, and the other employed the on-line TDCR solution based on FPGA counting module named TDCR-DMCA. Two solutions are applied to perform coincidence, dead-time and counting operations follow by MAC3 logic [3].

For matching assumption of TDCR model, the thresholds of amplitude discriminators of each PMT channel was properly adjusted by single electron peak (SEP) measurement. The performance of the TDCR counting system was tested in benchmark comparison with the custom-build TDCR counting system at NIM through activity measurements of  $^3\text{H}$ ,  $^{14}\text{C}$  and  $^{210}\text{Po}$ .

Good agreement between these two systems was observed.

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O23

## **Coprecipitation technique as a new approach of using solid-state scintillation**

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### Abstract:

In this paper, the coprecipitation methods of selected radionuclides in connection with solid-state scintillation counting were explored. The calcium phosphate co-precipitation method exhibited best results from all investigated methods reaching almost absolute detection efficiencies for selected actinides and more than 60% for Sr/Y-90. The linear response of the measurement was in the range of 0.4 to 411.5 Bq for alpha/beta separation mode, respective 0.4 to 2000 Bq nuclear counting (high activity mode). Computed and optimized MDA values exhibited better results in comparison with previous works.

O24

## **Optimization of composition of water accepting scintillation cocktail**

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**Abstract:** This work was focused on the optimization of the composition of scintillation cocktails, mainly the part, which is responsible for accepting water. The properties of individual components were discussed, from primary and secondary luminophores, through various types of organic solvents, to mixtures of commonly used anionic and non-ionic detergents. It included suggestions for new scintillation cocktails which, in comparison with commonly available scintillation cocktails, achieved better properties in the field of detection efficiency and stability and at the same time achieving better results in terms of sample load capacity, especially sample load capacity with aqueous samples. Since a small volume of aqueous sample inserted into a scintillation cocktail may not be sufficient to perform the analysis, so the ability to hold a larger volume of aqueous sample is crucial. This work also dealt with new luminophores, showing better scintillation properties than commonly used luminophores in commercial cocktails. The requirements for individual properties of scintillation cocktails in laboratory use were characterized, from luminescence properties, through the influence of primary and secondary luminophore concentration, alpha/beta discrimination ability, afterglow properties of individual components, sample load capacity, quenching resistance, long-term stability, solvent diffusion, detection limit, and FOM, up to detection efficiency. Finally, the work presents the composition of an ideal scintillation cocktail designed, among other things, for efficient alpha/beta separation, which can hold a large amount of sample while maintaining high detection efficiency.

O25

## **Effects of the photocathode non-uniformity on the radon measurements by scintillation counting.**

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This work studies the effects of the photocathode non-uniformity on the radon measurements by scintillation counting. Plastic scintillators exposed to radon are used to study the effect of the photocathode non-uniformity on the measured alpha spectra for two photomultiplier tube types – Hamamatsu R7600U-200 and Hamamatsu R9779. The photocathode non-uniformity is found to be the source of a significant tailing of the alpha- peaks in the  $^{222}\text{Rn}$  spectrum. This effect is discussed from the points of view of alpha-spectrometry by scintillation counting. Possible effect of the photocathode non-uniformity on the applications of the TDCR counting method will also be discussed.

O26

## Aryl group Grafting Polysilane: Novel Scintillation Solute for High Efficient and Environment-friendly Scintillator

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**Abstract:** Liquid scintillator is a core material for future high-precision and large-scale radiation detection due to its flexible design, unlimited shape and size. However, at present, it has some disadvantages such as low photonic quantum yield, high packaging requirements and high environmental toxicity. Polysilanes containing Si-Si long chain have flexibility and electron conjugated structure, which provide a unique fluorescence performance. By using the scintillation solute and the wavelength shifter as the grafting group, the electron delocalization transfer is realized by relying on the conjugated structure between the Si-Si chain and the aryl scintillating group, which leads to the improvement on the fluorescence emission spectra and scintillation sensitivity. At the same time, the flexibility of Si-Si bond is used to retain the good solubility, which could select non-toxic solvent to form the environment-friendly scintillator. This study has confirmed that when dissolved in n-hexane at 1% concentration, the fluorescence spectrum of 1,4-bis(2-methylstyryl) benzene grafting polysilane is similar to that of commercial liquid scintillation, and its scintillation counting rate can reach 43% of the latter. It can be concluded that the fluorescence efficiency of the grafting polysilane is significantly higher than that of the commercial liquid scintillator, and the use of non-toxic solvent can better take into account the requirements of environmental friendliness.

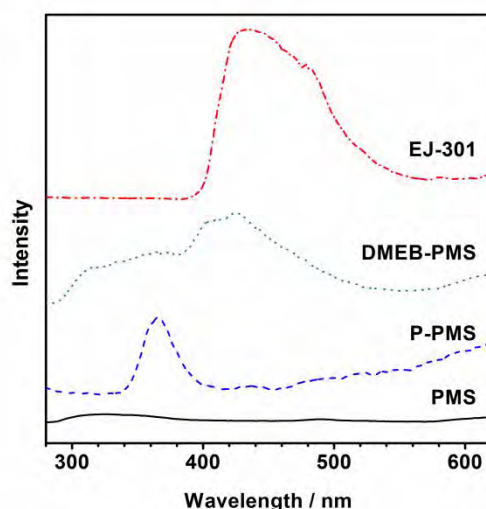


Figure 1 fluorescence emission spectra of several grafting polysilanes and EJ301 liquid scintillator

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O27

### **Development of a Detector to Detect 9.17MeV Gamma Ray**

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The ability to detect explosive material has become a problem of contemporary importance globally. Because of the complexity environments, the detection and identification problem can be quite difficult. One of the most interesting detection methods, and yet least investigated, involves characteristic  $\gamma$  ray resonant absorption.

Since most explosives contain a higher concentration of nitrogen than other common materials, it is possible to use  $^{14}\text{N}(\gamma, p)^{13}\text{C}$  resonant reaction induced by 9.17 MeV  $\gamma$  ray to detect explosives. In this method, an inexpensive liquid scintillation detector to detect 9.17 MeV gamma ray is developed in our lab. We add some high-nitrogen-containing organic matter to the traditional liquid scintillator and make it possible to detect 9.17 MeV gamma rays efficiently. The detecting principle is similar to the way of neutron detection. It has to distinguish the detector signals from gamma ray or resonant reactions. A preliminary work has been done for the further explosive detector array.

O28

## Liquid scintillator for Neutrino experiments such as Daya Bay and JUNO

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Abstract: Neutrino is one of the leading and hot issues in high energy physics. Lots of progresses had been made recently by large experimental projects such as SuperK<sup>[1]</sup>, SNO<sup>[2]</sup>, KamLAND<sup>[3]</sup>, T2K<sup>[4]</sup>, and Daya Bay experiment<sup>[5]</sup>. Liquid scintillator plays an important role in neutrino physics because of several advantages. It's relatively cheap, easy to realize large-scale production and on-line purification, and can be doped with other element for different purpose. The optical property and radioactive background of liquid scintillator can be very satisfying when suitable recipe and purification methods were chosen. There are several neutrino experiments in the world using liquid scintillator for catching neutrinos, such as Kamland<sup>[3]</sup>, SNO+<sup>[6]</sup>, Daya Bay experiment<sup>[7]</sup> and JUNO<sup>[8]</sup>. Gadolinium-loaded liquid scintillator (Gd-LS) was utilized in Daya Bay experiment. Recipe of Daya Bay Gd-LS was developed by us at IHEP since 2005. 185 tons of Gd-LS was produced in 2011 and then filled into Daya Bay Anti-neutrino Detectors. Optical properties of Gd-LS remain stable till now based on the detector data. Now we are working for JUNO to provide 20,000 tons of liquid scintillator with higher transparency and lower radio-active background.

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O29

## **The effect of scintillator materials on the fast neutron multiplicity measurement**

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**Abstract:** The fast neutron multiplicity measurement method adopts a scintillator detector to take measurements without going through the neutron slowing process. However, there are problems such as low detection efficiency and large measurement errors, which affect the accuracy of measurement results.

To analyze the effect of scintillator detectors on the measurement error of fast neutron multiplicity, this paper uses Geant4 to simulate and study three types of scintillator detectors: BC501A, EJ-309 and Stilbene, which are commonly used in fast neutron multiplicity measurements. According to the comparison of the detection efficiency and measurement deviation, EJ-309 with the lowest hydrocarbon ratio has the highest detection efficiency and the smallest measurement deviation. According to the response law of the scintillator detector, the energy spectrum information of the recoil particles is collected to analyze the reasons for the difference in measurement results of the three scintillator materials. The results show that Stilbene has the lowest proportion of hydrogen nuclei in recoil particles, which causes poor measurement results. On this basis, by changing the hydrocarbon ratio, nuclide type and density of materials, we found that a lower hydrocarbon ratio and a greater density correspond to a better measurement effect, and nuclide deuterium has better measurement effect than hydrogen nuclide. This study provides a theoretical reference for the subsequent development of high-performance fast neutron multiplicity detectors and is of great significance for improving the measurement effect of fast neutron multiplicity.

**Key words:** fast neutron multiplicity measurement hydrocarbon ratio recoil nuclide

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O30

## Trial-production of liquid scintillation fiber array

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**Abstract :** Considering today's severe energy situation and prominent environmental problems, controlled nuclear fusion has been a frontier of international efforts to explore. In this study, liquid scintillation fiber array is trial-produced for neutron penumbra imaging<sup>[1,2]</sup>, which is of great significance for neutron detection.

As shown in Fig.1. An optical fiber array consists of many identical arrays of fiber tubes. Each optical fiber can be divided into three layers. After passing through the surface of the fiber array, the neutron rays enter into the fiber core layer. The n-p reaction occurs at different positions to generate recoil protons, after which the recoil protons excited fluorescent substances to generate fluorescence.

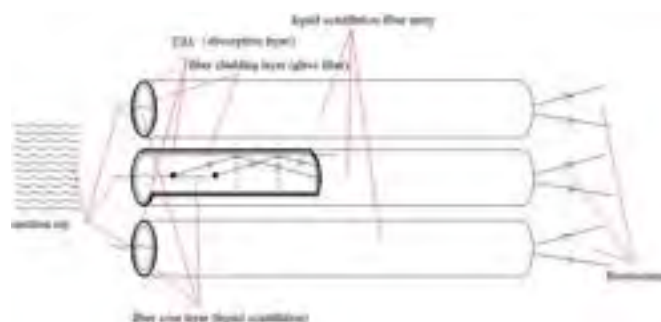


Fig.1 Diagram of liquid scintillation fiber array

The work of this study mainly includes five parts.

### 1. Manufacture of fiber tubes

The refractive index of the fiber tube must be less than that of liquid scintillation. The fiber tube selected is a glass capillary tube with an inner diameter of 0.9-1.1mm, wall thickness of 0.10-0.15mm and tube length of about 100mm.

The fiber tube is coated with an EMA absorption layer. The aluminum foil cladding is used as the EMA layer, and the thickness of the aluminum foil is 0.125mm. The processed fiber tube is shown in Fig.2.



## 2. Production of shells and end caps

Fig.3show the design of the fiber array shell. Fig.4 shows the design of the end cap.

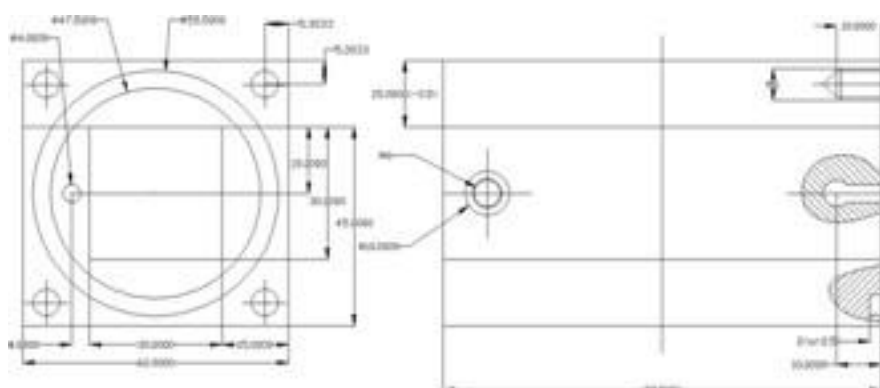


Fig.3 Main view and left view of the shell

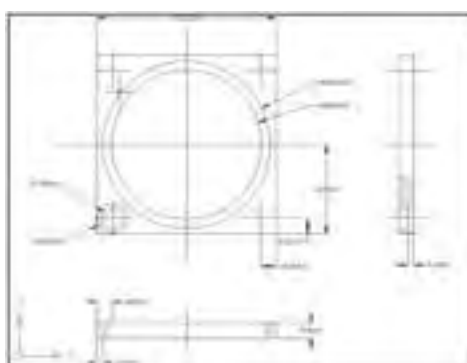


Fig.4 Design of the end cap

A hollow area of 30mm x 30mm in the center of the shell is used to place the liquid scintillation fiber array. The whole shell is divided into two parts: the upper shell and the lower shell. Grooves 4mm wide and 2.3mm deep are respectively arranged at both ends of the shell.

Besides, two filling channels are introduced in order to fill the liquid scintillation smoothly under the condition that the whole has been sealed.

Fig.5-1,5-2,5-3 show the pictures of the lower shell. Fig.6-1,6-2 show the pictures of the upper shell. Fig.7 shows the picture of the end cap.



Fig.5-1 Main view of lower shell



Fig.5-2 Right view of lower shell



Fig.5-3 Vertical view of lower shell



Fig.6-1 Main view of upper shell



Fig.6-2 Vertical view of upper shell



Fig.7 End cap

### 3. Selection and processing of optical glasses

Two flat optical glasses are used as the windows of the array section. Fig.9 is the picture of the optical glasses.



Fig.9 Optical glasses

### 4. Selection of liquid scintillation

The liquid scintillation used in this study is the BTD-4. Its main performance: 1) Light output (anthracene): 78%; 2) Strongest emission wavelength: 425nm; 3) Attenuation time: 3ns; 4) H/C ratio: 1.3.

### 5. Selection of "O" ring

A "fluorine rubber" material with good corrosion resistance is selected as the "O" ring material, with an outer diameter of 55.00mm and a cross-sectional diameter of 3.50mm.

After the processing of each component is completed, it will be assembled into a complete liquid scintillation fiber array:

- 1) Arrange the glass fiber tubes in the center area of the shell.
- 2) Cover the upper and lower shells tightly.
- 3) Glue and seal the upper and lower shells.
- 4) Put the "O" rings into the "O" ring grooves. Tighten the optical glasses and the end caps on the shell.
- 5) At last, a liquid scintillation fiber array can be completed by deoxygenating the liquid scintillation with certain equipment and system and then filling the fiber array.

This article has completed the fabrication of the main part of the liquid scintillation fiber array. Next, we will continue to improve the deoxygenation and canning of the liquid scintillation.

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#### Acknowledgements:

Thanks to Master Wang Tiechuang, who is responsible for the selection of the fiber tube and the processing of the EMA layer. Thanks to Shou Hongtao, who is responsible for the production of the shell and end caps. Also thanks to Master Zhao for his help on optical glasses.

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O31

## Characterization of the neutron/ $\gamma$ -ray discrimination performance of an EJ-301 liquid scintillator for application to prompt fission neutron spectrum measurements at CSNS

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**Abstract:** Prompt fission neutron spectrum (PFNS) is one of the most important nuclear data and is of significant importance on fundamental nuclear physics. With the high intensity and wide energy range neutrons provided by the back stream neutron beam line (Back-n) at the China Spallation Neutron Source (CSNS), a Fission Neutron spectrum Detector Array (FINDA), which consists of 64 liquid scintillators (EJ301) to measure prompt fast neutrons, has been designed and is under construction for high quality PFNS measurements using time-of-flight (ToF) technique. In order to test the neutron- $\gamma$  discrimination performance of the EJ301 scintillator that will be used as a detector unit in the FINDA, a comparative study using three conventional digital pulse shape discrimination (PSD) methods has been carried out. An Am-Be source has been used to generate the mixed neutron- $\gamma$  radiation and the waveform data has been obtained with a fast digitizer. Pulse interpolation, baseline restoration and pulses pile-up rejection have been applied for digital pulse processing. Charge comparison (CC) method and simplified charge comparison (SCC) method show a better capability of neutron- $\gamma$  discrimination than rise time comparison (RTC) method over the entire energy range. Rise time comparison method needs a very fast digitizer or pulse interpolation to improve the PSD ability. The analysis of neutron- $\gamma$  discrimination performance of the EJ301 scintillator provides a strong technical support for fast neutron measurement using the FINDA at CSNS.

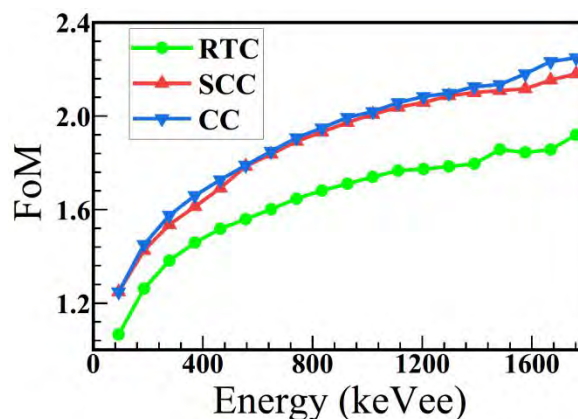


Figure 1 Comparison of the FoM results as a function of energy threshold with the three digital PSD methods

O32

## **Definition and optimization of a procedure for obtaining crosslinked plastic scintillation microspheres.**

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Measurement of radioactivity in the environment is a necessity that is derived from the potential biological danger that it can cause in living organisms. This radioactivity could be originated by natural sources, as is the case of  $^{222}\text{Rn}$ , which is originated from soils rich in  $^{226}\text{Ra}$  ores; or by artificial sources, either in radioactive repositories, hospitals or unlawful acts. This is the reason for the development of effective detection control systems are necessary, in order to avoid risk situations for living organisms. Following the objective of obtaining efficient and respectful detection systems, with a low generation of residues, plastic scintillators in form of microspheres (PSm) has been proposed in the last years as an alternative to liquid scintillation. The use of plastic scintillators avoid the generation of mixed wastes when measuring radionuclides, allow high selectivity when these are combined with extracting agents (PSresins) as well as provide high viability for applications in continuous detection systems.

Previous experience of the authors on PSm based on polystyrene and prepared by evaporation/extraction method has shown that PSm or PSresins provide a high detection efficiency in the determination of alpha and medium or high energetic beta emitting radionuclides as long as PSm present a diameter between 10 and 100 micrometres. However, these can be used in a framework of limited conditions since the polymer is soluble in organic solvents. For this reason, the research group is developing microspheres including a crosslinking monomer, divinylbenzene, which is copolymerized with styrene creating a crosslinked scintillating microsphere (CPSm). These CPSm allow, according to their structure chains of polymer linked one to another in different points, to be used in aggressive conditions, such as organic matrices opening the possibility to measure organic samples or treat it with organic solvents to modify its structure (addition of pores) or its surface (adding functional groups on its surface)

The objective of the present study was to define and optimize a methodology to obtain CPSm by polymerisation of styrene with the presence of DVB, and the evaluation of the morphological and scintillation properties of CPSm. The method used is based on a free radical polymerisation in a dispersant medium with the presence of a surfactant (PVA). Initially, the effect of the styrene-DVB ratio was verified, synthesizing CPSm in different ratios. It was proved that a higher proportion of DVB produces a reduction in the medium size of the CPSm (ranging from 40 to 100 micrometers), without significantly altering the radiometric properties, as it is shown in Figure 1. Values of detection efficiency are comparable to those PSm prepared by the evaporation/extraction method and containing only polystyrene which demonstrate the low effect of crosslinking with DVB in the scintillation mechanism. To optimize the synthesis yield, the dispersing medium was modified by adding sodium chloride, as well as a modification in the concentration of activator in the monomeric mixture. It was found that the presence of sodium chloride produces an increase in yield

regardless of its concentration. On the other hand, the variation on activator proportion does not produce appreciable effects. As a conclusion the methodology developed allows the preparation of CPSm with appropriate diameter and good radiometric capacities that could lead to the development of new and advanced scintillation materials.

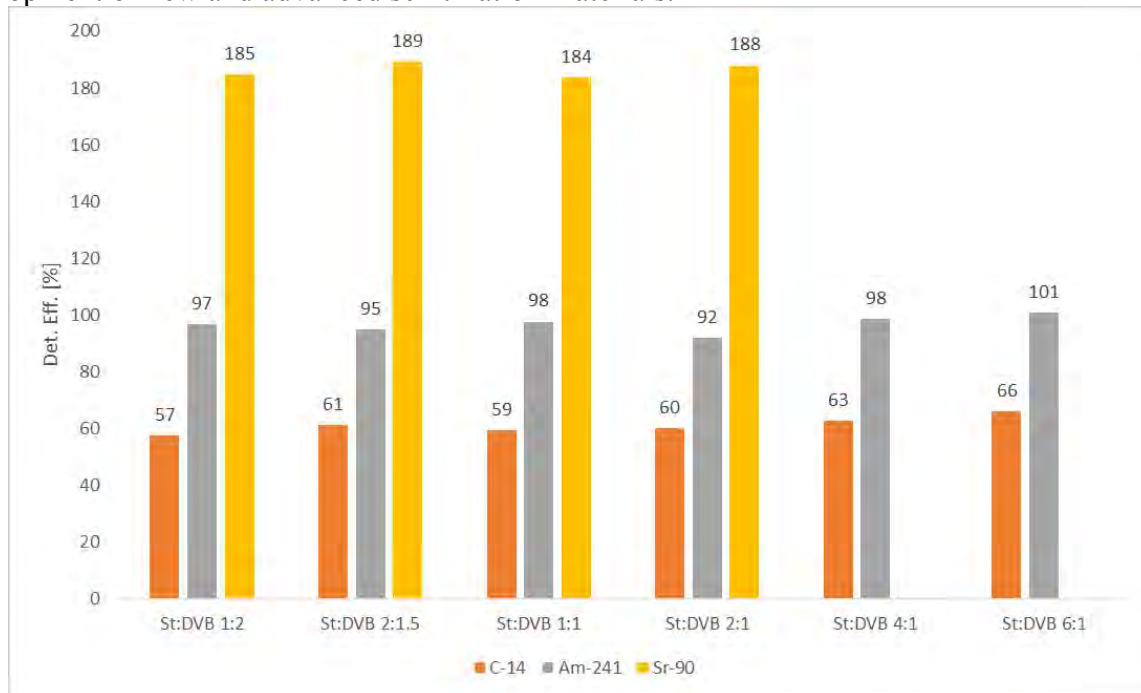


Figure 1. Detection efficiency determined for C-14, Am-241 and Sr-90; in CPSm with different St:DVB ratios. For CPSm in proportions 4:1 and 6:1, Sr-90 could not be measured due to the low synthesis yield obtained.

O33

## **A Plastic Scintillator with enhanced Alpha-/Beta- PSD. Part I: Rationale and preparation**

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Recent developments in the field of plastic scintillator modifications are mainly focused on the Pulse Shape Discrimination (PSD) of fast neutrons from gamma rays. However, this PSD feature should also be effective for other particles showing different  $dE/dx$  values. In this context, we have designed and prepared a plastic scintillator that was originally purposed for  $n/\gamma$  discrimination, but was also used for alpha/beta PSD as well. This scintillator displayed tremendous capabilities.

This first part of this presentation will show the rationale underneath the PSD in plastics and how to achieve it from material design. The synthesis will be explained, followed by photophysical and nuclear characterization. Then the second part will present the characterization for radon measurement.

O34

## **A Plastic Scintillator with enhanced Alpha-/Beta- PSD. Part II: Characterization for $^{222}\text{Rn}$ measurement**

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In this work a plastic scintillator with enhanced pulse shape discrimination (PSD) capabilities has been characterized for  $^{222}\text{Rn}$  measurements. The characterization includes: evaluation of the alpha-/beta- PSD figure of merit, experimental determination of the radon solubility and diffusion length in the scintillator, characterization of the alpha spectra of radon absorbed in the scintillator after PSD and evaluation of the temporal stability of the scintillator. The alpha-/beta- PSD properties of the scintillator are evaluated with three experimental systems: a PERALS spectrometer; an inhouse made detector with optimized optical chamber for optimal light collection equipped with a nanoPSD Real-Time Digital Pulse-Shape Discriminator and Spectrometer with Simultaneous Amplitude and Pulse-Shape Discrimination; an inhouse made detector with optimized optical chamber for optimal light collection equipped with a CAEN DT5751 digitizer for pulse-shape analysis. It is demonstrated that the developed plastic scintillator has exceptionally good PSD performance for alpha-/beta- pulse shape discrimination and that it provides interesting opportunities for radon detection and measurement.

O35

## Standardization of $^{90}\text{Y}$ solution by mean of a Cerenkov Counting in comparison with a Liquid Scintillation Counting technique.

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This paper presents the standardization of  $^{90}\text{Y}$  solution using the Cerenkov method measuring in the TDCR counter [1]. The Triple-to-Double coincidence counter commonly used in the liquid scintillator technique [2] are also feasible for the detection of the Cerenkov light emitted by high-energy  $\beta$ -rays in transparent liquids.  $^{90}\text{Y}$  decays by beta minus emission with the maximum-point energy of 2278.7 keV which is greater than the Cerenkov threshold energy [3].

The counting efficiencies for the production of Cerenkov light were applying the Frank and Tamm theory [4]. The anisotropy effect for the Cerenkov light emission and an asymmetry of the measurement system was taken into account by fitting the free parameter. The detection efficiency for the TDCR counter was calculated in a home-made code.

A set of ten sources in 20 mL high-performance PerkinElmer glass vials filled-in with 10 mL of Ultima Gold liquid scintillator was prepared for the liquid scintillation counting measurement method. Additionally, the series was chemically quenched by nitromethane in order to change the efficiency of the TDCR detector. A set of three sources in 20 mL polyethylene PerkinElmer vials filled-in with 10 mL of 1 M HCl was prepared for measurements using the Cerenkov counting method.

The prepared measurement samples were measured in two different TDCR detectors. The first system was equipped with ET Enterprises 9214B photomultipliers and the second with BURLE 8850 photomultipliers. Measurement of chemically quenched samples in Ultima Gold showed the goodness of the ET Enterprises 9214B photomultipliers, the obtained activity was constant for the entire series. On the other hand, their excitation was observed with BURLE 8850 photomultipliers. The highest efficiency samples were rejected in this case.

For both measurement techniques, the activity was determined and compared. The good agreement of the results obtained between the methods was below 0.1%. The efficiency of the TDCR detector for the Cerenkov technique was calculated correctly. The results of this paper enable to continue measurements of radionuclides with a high beta-emission by mean of the Cerenkov Counting technique.

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O36

## **Significance of the correction for accidental coincidences in liquid scintillation counting measurements**

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Methods for the evaluation of the accidental coincidences counting rates in Triple-to-Double Coincidences Ratio (TDCR) Liquid Scintillation (LS) measurements have been proposed recently [1]. The methods were used in this study where the corrections for accidental coincidences are applied to TDCR measurements of different radionuclides with a wide range of activities and in different measurement conditions. The contribution of the accidental coincidences to the true coincidences counting rates is evaluated.

The corrections for accidental coincidences are also applied to TDCR measurements of  $^{18}\text{F}$  with counting rates from  $430000\text{ s}^{-1}$  to  $150\text{ s}^{-1}$ . The contribution of the accidental coincidences for each order of magnitude of the counting rate is presented. The effect of the accidental coincidences to the evaluated half-life of  $^{18}\text{F}$  is presented and discussed.

This study also shows how the corrections can be extended to a four-detector system as used in the Compton Source Efficiency Tracing (CSET) method [2] or the Zombies method [3]. For such detector systems, employing an external gamma-ray source and a Compton spectrometer, the counting rate in the LS must be very high to observe a reasonable counting rate in the gamma-ray detector. In this case the correction for accidental coincidences is far from negligible.

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O37

### Standardization of $^{177}\text{Lu}$ by means of $4\pi(\text{LS})\beta\text{-}\gamma$ coincidence counting.

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The Lutetium-177 ground state disintegrates by beta minus emission to the ground state and to three excited levels of  $^{177}\text{Hf}$ . Due to a short half-life of this rare earth isotope, it is a useful nuclide for treatment of various tumors.  $^{177}\text{Lu}$  has proven efficacy to treat gastroenteropancreatic neuroendocrine tumors in candidates with advanced disease. This therapy decreases tumor size, improves symptoms, and improves quality of life[1]. Due to this, the precise determination of  $^{177}\text{Lu}$ -solution activity is extremely important.

The  $^{177}\text{Lu}$ -solution was measured by means of  $4\pi\beta\text{-}\gamma$ -coincidence and anticoincidence counting techniques. The  $4\pi\beta(\text{LS})\text{-}\gamma$  system, constructed at POLATOM, contains two PMTs working in coincidence and the liquid scintillator in the  $\beta$ -channel and two PMTs with NaI(Tl) crystals working in the sum mode in the  $\gamma$ -channel. The  $\beta$  and  $\gamma$  channels are working in coincidence or anticoincidence[2].

To determine the activity of a radionuclide, a series of 6 vials was investigated. Counting rates of  $\gamma$  channel events ( $N_\gamma$ ),  $\beta$  channel coincidence events ( $N_\beta$ ), coincidence between  $\beta$  and  $\gamma$  channels events ( $N_C$ ) and anticoincidence between  $\beta$  and  $\gamma$  channels events ( $N_{AC}$ ) were registered. The counting efficiency was changed by high voltage applied to PMTs in  $\beta$  channel and for each source a set of 7 counting points was obtained. Counting rates were registered and corrected for a dead-time and the background rate was subtracted. The data collecting and analysis was made by the custom-built data-taking and data-analyzing software. A linear function  $f(x)$  was fitted to the data by means of the least squares method and the function was extrapolated to  $\varepsilon=1$  giving the disintegration rate.

Applying the two primary methods ( $4\pi\beta\text{-}\gamma$  coincidence and anticoincidence) the activity per unit mass of  $^{177}\text{Lu}$ -solution,  $A$ , was obtained. The results from coincidence and anticoincidence are in good agreement. The relative uncertainty of the weighed mean of activity value is 0.26%. The current uncertainty is smaller than the uncertainty obtained in the previous  $^{177}\text{Lu}$ -solution activity measurement (0.54%). Previous measurement was made for an international comparison in 2009. In this comparison, the POLATOM result was in good agreement with Comparison Reference Value [3].

The combined result was used to establish a new, more precise, calibration factor for a  $4\pi$  ionization chamber for  $^{177}\text{Lu}$  isotope. This measurement allowed to reduce the uncertainty of activity determination, using the ionization chamber, by almost 30%.

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## P01

### 20 Years DGFS Society for Liquid Scintillation – New Developments

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The Society for Liquid Scintillation DGFS (<https://DGFS-ev.eu>) as non-profit society was founded along the LSC2001 Conference in Karlsruhe with the aim of development and transfer of modern Liquid Scintillation (LS) to a broader community. Selected analytical procedures have been compiled recently in the 2nd extended edition of the Handbook on New LS Measuring Procedures 2018 (1).

Besides the academic education and training in LS as key object, DGFS heads to develop new procedures and optimize existing ones in liquid scintillation spectrometry. Besides for artificial radionuclides e.g. in the nuclear fuel cycle LS enables a comprehensive survey of natural key nuclides in water processing (see (1) pp55). In combination with RAD Disks for Ra and Pb enrichment a simple and fast method in drinking water has been developed recently (see (1) pp46). Liquid Scintillation presents a key analytical skill for Tritium in the present KATRIN neutrino mass detection experiment at KIT, where its energy spectrum is examined. Various procedures were adapted for its detection in pipes after oxidation and for radiation protection purposes. For the detection of <sup>210</sup>Pb Black Powder deposits in the newly built Northstream Pipeline as suggested recently see procedure pp119 in (1). The ability of luminescence counting favor LS as tool for analysis of photon induced DNA/RNA labelling in future cancer and serum research. A summary of new analytical developments as described in (1) is provided in this presentation.

In order to further spread this modern, universal and future prospective methodology, DGFS heads to transfer LS into the academical education of developing countries by applying simple LS arrangements (see (1) pp22). For successful developing future programs worldwide, you may help by active participation in our Society.

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Liquid Scintillation – Measuring Procedures, New Developments - 2<sup>nd</sup> extended edition  
2018, DGFS e.V. German Society for Liquid Scintillation Spectrometry and KIT  
Karlsruhe Institute of Technology, 2018 Karlsruhe

P02

## Automated radionuclide extraction chromatography instrument

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Extraction chromatography (EXC) is an efficient technique for separation and concentration of radionuclides from a variety of sample matrices. The EXC utilizes selectivity of liquid-liquid extraction in an easy-to-use column chromatography format. Typical applications include: 1) Environmental control measurements of natural and man-made radionuclides from diverse sample types such as water, various food products and soil/sediment. 2) Radiopharmacy products separation, purification, and quality control. 3) Decommissioning sample analysis from nuclear installations. The EXC is time-consuming to perform and requires handling of hazardous strong acids and, therefore, it is not a safe user-friendly laboratory method.

The Hidex Q-ARE is an automated extraction chromatography instrument that processes the samples and provides a total walk-away freedom for the user. The instrument can process up to eight samples simultaneously utilizing six different mobile phase reagents including strong acids. The system is able to perform single and tandem EXC and, up to five elution fractions can be collected from one sample. Various size of prepacked and self-packed columns from 1 ml to 20 ml are compatible with the Q-ARE instrument. The instrument performance has been verified in multiple laboratories analyzing water, soil and food samples.



Figure 1. Hidex Q-ARE 100plus instrument

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P03

**A method for detection for all three naturally occurring radon**

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**Keywords:** Radiation; Radon; Radon Isotopes; Radon detection,, Detector, Liquid Scintillator

**Abstract**

Radon is the most important source of ionizing radiation which poses human health risks especial lung cancer after smoking. There are three naturally occurring radon isotopes Rn-222, Rn-220 and Rn-219 which are members of natural decay series of U-238, Th-232 and U-235, respectively. We are developing a method which can be used to detect all three naturally occurring radon isotopes. Our technique is based on liquid scintillator and photomultiplier tube using delayed coincidence technique and pulse shape discrimination method implemented by digital charge comparison. In these ways, isotopes having a relatively short half-life can be selected out by their characteristic energy and decay time distributions. This method can be used to detect all three naturally occurring Rn-222, Rn-220 and Rn-219 rather than other developed methods which are based only on the detection of Rn-222. The gamma response functions of ultima gold AB cocktail detector standard gamma sources are under study with Monte Carlo simulation. We will report on development of this method in this meeting.

P04

## High-energy X-ray detection characteristics of $\text{Hf}_x\text{Si}_{1-x}\text{O}_2$ particle-loaded PVK-based plastic scintillators synthesized by sol-gel method

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Fast detection of high-energy X-rays is essential in synchrotron radiation facilities. Heavy metal-loaded plastic scintillators are potential candidates in this regard. We applied the sol-gel method for the loading of heavy metals. In this study, we used poly(9-vinylcarbazole) (PVK), which is the hole transport material in organic electroluminescence devices, as the host polymer. We fabricated  $\text{Hf}_x\text{Si}_{1-x}\text{O}_2$  particle-loaded PVK-based plastic scintillators by the sol-gel method and characterized their X-ray detection characteristics.

PVK,  $\text{HfOCl}_2 \cdot 8\text{H}_2\text{O}$ , phenyltrimethoxysilane (PhTMOS), and 1,4-bis(2-methylstyryl)benzene (bis-MSB) were used as the host polymer, heavy metal compound, silica source, and organic phosphor, respectively. PVK was dissolved in a mixed solvent composed of tetrahydrofuran, methanol, 0.1 M HCl, and *N,N*-dimethylformamide (DMF). Subsequently,  $\text{HfOCl}_2 \cdot 8\text{H}_2\text{O}$ , PhTMOS, and bis-MSB were added to the solution and allowed to dissolve. Samples were obtained by drying the solvent at 58°C for 2 days. Bis-MSB at 1.0 mol% was added to 9-vinylcarbazole monomer units. The samples were loaded with  $\text{HfOCl}_2 \cdot 8\text{H}_2\text{O}$  and PhTMOS at weight percentages of Hf relative to the total amount of the samples at 0–20 wt%. The molar ratio of Hf to Si was fixed at 1:1. The pulse-height spectra were measured using a synchrotron X-ray beam, whose energy was set to 67.41 keV.

Fig. 1 shows the pulse-height spectra of scintillation detectors equipped with the unloaded and Hf-loaded samples, and EJ-256 for 67.41 keV X-rays. Table 1 summarizes the detection efficiency and light yield. The detection efficiency was successfully enhanced as the concentration of Hf was increased. Additionally, the reduction in light yield was successfully suppressed by using sol-gel method. In particular, the detection efficiency and light yield of the Hf 12.5 wt%-loaded sample were 2.0 and 1.1 times higher, respectively, than those of EJ-256, which is a commercial plastic scintillator loaded with Pb 5 wt%. Thus, we successfully improved the detection efficiencies of PVK-based plastic scintillators by using the sol-gel method while suppressing the reduction in light yield by Hf loading.

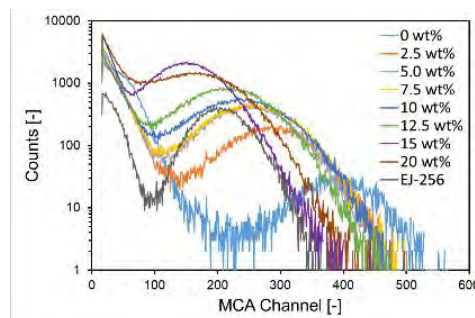


Fig. 1 Pulse-height spectra.

Table 1 Detection efficiency and light yield.

Samples	Thickness [mm]	Detection efficiency [%] (1.0 mm conversion)	Light yield [photons/MeV]
0 wt%	0.54	1.2	9900
2.5 wt%	0.28	2.1	7300
5.0 wt%	0.33	2.8	6400
7.5 wt%	0.28	3.4	6500
10 wt%	0.31	3.7	6000
12.5 wt%	0.32	4.7	5500
15 wt%	0.40	6.6	3800
20 wt%	0.30	8.8	4200
EJ-256	2.00	2.3	5200

P05

## Radiostrontium determination in aerosol filters and vegetation in emergency situations using PS resin

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### Abstract:

In emergency situations, a rapid determination of radiostrontium in environmental matrices is necessary to evaluate the contamination and give a fast response. Pure beta emitters, such as <sup>89</sup>Sr and <sup>90</sup>Sr, require a radiochemical separation prior to measurement. The use of plastic scintillation resins (PS resin), which combine the separation and measurement preparation into a single step, allow a simplification of the radiochemistry and a reduction in the time of analysis [1,2].

In this work, rapid and novelty methods for radiostrontium determination in aerosol filters and vegetation based on the optimization of the pre-treatment steps and the use of PS resin are proposed [3]. The time of analysis was reduced to 8 h and 12 h for aerosol filters and vegetation, respectively, and the limits of detection were on average 0.04 Bq (filter)<sup>-1</sup> and 3 Bq (kg-fresh)<sup>-1</sup>. The method obtained high recoveries (82% on average) and relative bias for total radiostrontium were below 30% for different types of aerosol filters (glass microfiber and cellulose) and vegetation samples (grass, rosemary and pine needles) spiked in the laboratory with different ratios of <sup>89</sup>Sr/<sup>90</sup>Sr (1:1, 2:1, 4:1 and 8:1), and an intercomparison sample (spruce needles). Individual activities of <sup>89</sup>Sr and <sup>90</sup>Sr were obtained by deconvolution methods. Relative bias for individual activities <sup>89</sup>Sr and <sup>90</sup>Sr were below 30% in almost all the samples, but <sup>90</sup>Sr activity is overestimated when ratios <sup>89</sup>Sr/<sup>90</sup>Sr are high.

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## P06

### Nationwide survey of Tritium concentration in environmental water in Thailand

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**Abstract:** To estimate the baseline level of tritium concentration of environmental waters before the operation of the first Thailand nuclear power plant/fusion reactor/TRIGA 10000 kW research reactor at Ongkharak, etc, nationwide survey of tritium concentration in air vapor water, precipitation, river water, lake water, tap water, well water, spring water, and sea water will be planned by sampling and measuring with the bilateral joint research cooperation between Thailand and Japan. A pilot study was already carried out in November 2018 by sampling various regions in Thailand including Phayao, Srisaket, Bangkok, Chanthaburi, and Trang [1]. Tap water from four water treatment plants and ten pump stations in Bangkok metropolitan region was also checked in July 2020 and rain water at Sriracha campus of Kasetsart University was checked from May to November 2020. The latitude dependency of tritium concentration was suggested and the nationwide survey in the region of the earth equator will be an important/variable data for “Hadley circulation” and seasonal variation, too. This study is based on our leading cutting-edge technology with the sensitivity of no limit less than 1 Bq/L by introducing electrolytic enrichment via solid polymer electrolyte and low background liquid scintillator system (AccuFLEX LSC-LB7).

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P07

## **Analysis of a home-made code used in calculation of the efficiency TDCR system as part of the CCRI(II)-K2.Fe55.2019 key comparison.**

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In 2019, a key comparison for a  $^{55}\text{Fe}$  solution took place [1]. The pilot laboratory of the comparison was POLATOM. Twelve laboratories participated in this comparison. Most of the participants performed the efficiency calculations with similar codes (Fe55\_6 and Fe55\_fom). The home-made code developed by POLATOM clearly showed the differences in the calculation of the activity compared with the others results. This paper presents the applied new method and an analysis of the differences in efficiency calculations. The impact of selecting assumptions in the efficiency counting model is also presented.

The home-made code allows to determine the efficiency of the TDCR detector taking into account the asymmetry of the photomultipliers. For radionuclides with decay by electron capture or more complex decay schemes, it uses simulated energies interacting with a liquid scintillator from the MICELLE2 code. The program allows, among others, to adjust the calculated Birks function by selecting various assumptions for the stopping power function.

A set of six sources in 20 mL polyethylene PerkinElmer vials filled-in with 10 mL Ultima Gold liquid scintillator was used for analysis. The series of sources were chemically quenched by nitromethane and measured according to the standard procedure used in POLATOM. The measurement file (raw data) was repeatedly recalculated with the various calculation models. The influence of changing the given physical parameters of the liquid scintillator, assumptions of the stopping power function, the integration range and the kB parameter were checked.

The obtained results showed a significant influence of the efficiency TDCR detector calculation model. The differences in the results were at the level of 1%. There was also good agreement between the various assumptions of the Birks function when the appropriate kB parameter was selected. The differences obtained by POLATOM in the key Fe-55 comparison were also influenced by the using of different atomic rearrangement models in applied codes. This paper shows the need to perform measurements with another independent and absolute method in order to confirm the correct result of  $^{55}\text{Fe}$  solution activity.

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## P08

**Method for the analysis of  $^{14}\text{C}$  in urine**

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**Abstract** In order to improve the analysis method of radionuclide  $^{14}\text{C}$ , estimate the internal dose caused by  $^{14}\text{C}$  to human body, and protect the health of workers and the public in  $^{14}\text{C}$  exposed workplaces. Urine samples were pretreated by wet oxidation. In the analysis, potassium persulfate was used as an oxidant to decompose urea into carbon dioxide, which was absorbed by 1mol/L sodium hydroxide, and the absorption solution was converted into calcium carbonate precipitation. After calcium carbonate powder was prepared into sample source by suspension method, the low background liquid scintillation counter was applied to measure the radioactivity, which can be used for calculating the results. The reaction time optimized by using carbamide as carrier was 1 h. For 80 mL urine, the usage amount of potassium persulfate was 10 g. The method recovery rate reached about 100%. Four real urine samples were tested using the optimized method, and the results of  $^{14}\text{C}$  activity concentration was 0.32 Bq/L, 0.60 Bq/L, 0.86 Bq/L, 0.74 Bq/L, respectively. The optimized method had good stability, high accuracy and stable experimental results, which could meet the needs of routine radiological detection. The establishment of a quantitative method for  $^{14}\text{C}$  in urine sample has further improved the method system for carbon-14 monitoring.

**Key words**  $^{14}\text{C}$ ;  $\beta$ -emitters; Urine sample; Internal irradiation

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P09

**Determination of  $^{32}\text{P}$  by Liquid Scintillation counting for RI wastes**

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Most unsealed sources in RI waste drums(200 liter) at a storage facility are low-level beta-emitters with short half-lives, which is impossible to measure their inventories by a nondestructive analysis. Therefore, the concentration of a certain radionuclide has to be assayed by chemical analysis. A variety of radionuclides such as  $^3\text{H}$ ,  $^{14}\text{C}$ ,  $^{32}\text{P}$ ,  $^{35}\text{S}$ ,  $^{51}\text{Cr}$ ,  $^{99\text{m}}\text{Tc}$ ,  $^{125}\text{I}$ ,  $^{131}\text{I}$ ,  $^{147}\text{Pm}$  and  $\gamma$ -emitter ( $^{60}\text{Co}$ ,  $^{137}\text{Cs}$ ) are generated from RI utilizing institutes. Among them, we focused on determination of  $^{32}\text{P}$  nuclide that are pure beta emitter. According to the use of phosphorous-32( $^{32}\text{P}$ ) has greatly increased in the field of medicine, biochemistry and molecular biology, the generation of  $^{32}\text{P}$  radioactive wastes have steadily increased.  $^{32}\text{P}$  is a commonly used radionuclide with a half-life of 14.3 days, emitting beta particles with a maximum energy of 1710.7 keV. Because of its radiochemical properties, it is essential to keep systematic management for ensuring safety.

In this study, we describes methods as follows; 1) acid leaching, 2) water organic-solvent extraction, 3) separation, 4) measurement of  $^{32}\text{P}$ . Due to its high counting efficiency and average chemical yield( $93.91 \pm 0.03\%$ ), liquid scintillation counter(LSC) was used for detection of radioactivity. Verification of validity about separation procedure and calibration of LSC are also covered.

P10

## Direct sample preparation method of carbon -14

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### Abstract:

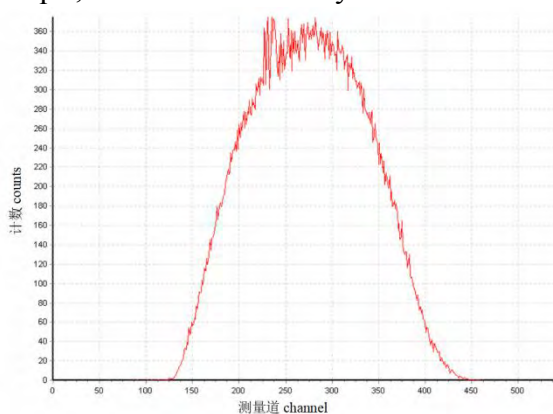
**Background:** Carbon-14, as a radioisotope of carbon with a long half-life (5730 years), participates in the global carbon cycle, and contributes a lot to the collective dose caused by long-term.

**Purpose:** According to the monitoring of carbon-14 in environmental radiation, a preliminary study on the direct sample preparation method of carbon-14 was carried out from the perspectives of sample preparation method, experimental study of direct sample preparation and application analysis of the method.

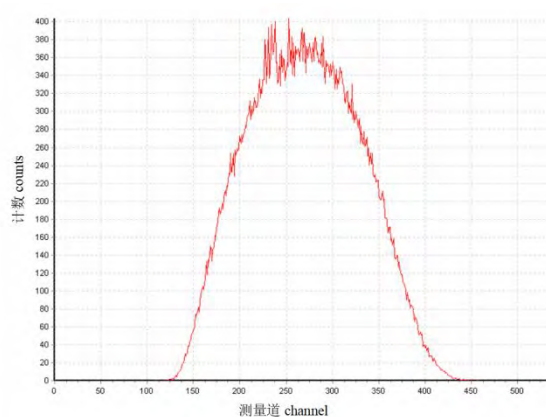
**Methods:** A direct sample preparation method of carbon-14 was established. The carbon in the sample was converted into carbon dioxide and then absorbed by NaOH solution. A proper amount of lye was taken and mixed with scintillation cocktail, and then measured by liquid scintillation spectrometer.

**Results:** The preliminary experimental results show that the counting efficiency of this method for carbon -14 is 64%, and the detection limit for carbon-14 in 2 mL 1mol/L NaOH is 16 mBq (measuring time is 300 min).

**Conclusions:** It is suitable for the analysis of samples with carbon-14 activity of above 100 Bq/L, but not for the analysis of carbon-14 in environmental samples



a) 1mLNaOH: 10mL cocktail



b) 2mLNaOH: 15mL cocktail

Fig.1 Spectra with Different Ratios of NaOH and Scintillation Cocktail

P11

**Study on Determination of  $^{90}\text{Sr}$  in Different Matrix via LSC**XU Ping<sup>1</sup>, PAN Linjing<sup>2</sup>, YU Guobing<sup>3</sup>, DING Chenlu<sup>3</sup>, CHEN Zhi<sup>1</sup>

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**Abstract:** 2-windows method for determination of  $^{90}\text{Sr}$  activity concentration using a liquid scintillation counter (LSC) in environmental samples is presented. This method was verified with spiked tests. The relative biases are less than 5% between the analytical activity of  $^{90}\text{Sr}$  and the expected activity. Recommended calculation method, the criteria of ISO 18589-5:2009 and the 2-window method were utilized to analyze environmental samples. The results obtained from the two methods are in good agreement and the average relative biases of the two methods are about 5%.

**Key word:** 2-windows method,  $^{90}\text{Sr}$ , LSC

P12

## **Fabrication of liquid scintillators loaded with 6-phenylhexanoic acid-modified ZrO<sub>2</sub> nanoparticles for observing neutrinoless double beta decay**

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Neutrinoless double beta decay ( $0\nu\beta\beta$ ) events are important in nuclear and particle physics, because they help in verifying the Majorana nature and finite mass of neutrinos. However, the rare occurrence of  $0\nu\beta\beta$  events limits their observation. Thus, a high-energy resolution detector containing a large amount of candidate isotopes is needed. Candidate isotope-loaded liquid scintillators are useful for observing  $0\nu\beta\beta$  events. Most candidate isotopes dissolve in the liquid scintillators as metal complexes. However, operational changes occur owing to the complexes' low solubility or scintillation quenching by a coordination molecule of the complex.<sup>1, 2</sup> Thus, we propose the synthesis of nanoparticles containing candidate isotopes, for dispersion in liquid scintillators. We synthesized organically modified zirconium dioxide (ZrO<sub>2</sub>) nanoparticles (containing the candidate isotope <sup>96</sup>Zr), using sub/supercritical hydrothermal synthesis methods.<sup>3</sup> Subsequently, we prepared liquid scintillators loaded with the ZrO<sub>2</sub> nanoparticles and evaluated their scintillation characteristics.

ZrO<sub>2</sub> nanoparticles were synthesized using sub/supercritical hydrothermal synthesis methods in batch reactors (inner volume: 5 cc). We prepared a precursor solution containing 0.1-M ZrOCl<sub>2</sub>·H<sub>2</sub>O dissolved in distilled water and adjusted the pH to 5.8, by adding KOH. Further, 6- phenylhexanoic acid (PHA) was selected as the organic modifier. The precursor solution and the modifier (molar ratio of Zr/PHA = 1/6) were added to the batch reactors. Next, we observed the effect of synthesis temperature through preparing the nanoparticles at 250°C, 300°C, 350°C, and 400°C. All synthesis reactions were performed under the constant pressure of 30 MPa for 10 min. The nanoparticle dispersions were prepared by recovering the product with toluene after the reactions. In addition, we washed the nanoparticle dispersion with ethanol several times and dried it to obtain a powder sample of nanoparticles. Subsequently, we characterized the nanoparticles through transmission electron microscopy (TEM) and thermogravimetric analysis (TGA). The Zr concentration of the nanoparticle dispersions was measured using inductively

coupled plasma atomic emission spectroscopy (ICP-AES). We fabricated nanoparticle-loaded liquid scintillators, by dissolving 2,5-diphenyloxazole (DPO) and 1,4-bis(5-phenyl-2-oxazolyl)benzene (POPOP) as phosphors in nanoparticle dispersions. Finally, we recorded the X-ray- induced radioluminescence (XRL) spectra for the prepared scintillators.

Figure 1 shows the TEM images of the synthesized nanoparticles. The respective particle sizes of nanoparticles synthesized at 250°C, 300°C, 350°C, and 400°C were  $4.0 \pm 1.3$ ,  $3.7 \pm 1.2$ ,  $6.7 \pm 1.8$ , and  $5.6 \pm 1.4$  nm. Thus, the TEM results show the formation of single nanometer-sized particles for all synthesis temperatures. TGA measurement showed a weight loss that corresponded to the desorption of organic modifiers from the surface of nanoparticles from 250°C to 500°C in all samples. The respective weight loss values were 23, 24, 8.6, and 8.4 wt%. Further, using the particle sizes and weight loss values, we calculated the respective PHA modification densities on the nanoparticle surface to be 2.8, 3.1, 0.9, 0.9 molecules/nm<sup>2</sup>. In addition, the respective Zr concentrations in the nanoparticle dispersions obtained from ICP analysis were  $1.1 \times 10^{-1}$ ,  $3.3 \times 10^{-1}$ ,  $5.7 \times 10^{-2}$ , and  $9.2 \times 10^{-3}$  wt%. At low temperatures (250°C and 300°C) with high modification densities, the dispersion concentration of Zr was also high. In addition, The modification density and the Zr dispersion concentration were both maxima at 300°C. The results suggest a correlation between modification density and dispersion concentration. Figure 2 shows the XRL spectra of the liquid scintillators. All the samples exhibit a clear emission peak at 425 nm. Thus, we achieved the successful fabrication of ZrO<sub>2</sub> nanoparticle-loaded liquid scintillators.

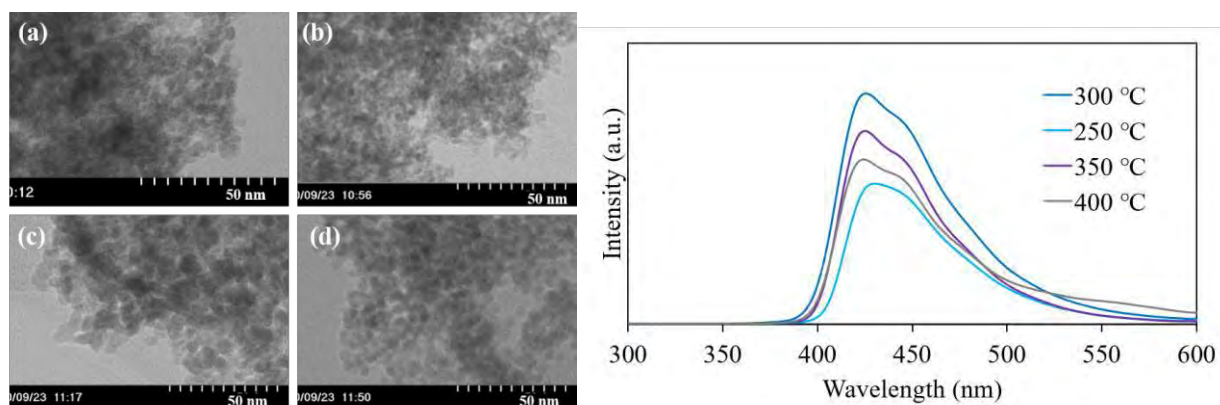


Figure 1. TEM images of nanoparticles synthesized at different temperatures: (a) 250°C, (b) 300°C, (c) 350°C, and (d) 400°C.

Figure 2. X-ray induced radioluminescence spectra of the fabricated liquid scintillators.

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P13

**undetermined**

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P14

## **The fast plastic scintillator used for neutron spectrum diagnostic in inertial confinement fusion experiments**

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**Abstract:** In inertial confinement fusion (ICF) implosion experiments, the fusion neutrons leaving the capsule carry significant information about fuel ions. The fuel ions acquire a thermal velocity distribution, which causes the neutron energy spectrum to be Doppler broad. The fuel ion temperature can be obtained by measuring the spectral broadened width. When fusion neutrons traverse the capsule, a percentage of primary neutrons will collide with cold ions in the dense fuel surrounding the hot spot and downscatter to lower energies. The fuel areal density can be deduced by measuring the spectra of downscattered neutrons. Due to drive asymmetry, the implosion fuel may undergo bulk motion in a specific direction. The velocity along the line of sight of detector will shift the peak of neutron spectrum. The condition of fuel ions can be identified by measuring the changes of neutron spectra at many viewing angles of the capsule, which is helpful for precise control of laser-driven target to obtain high implosion quality. The neutron time-of-flight (nTOF) detector is used to diagnose the neutron spectrum by measuring the arrival time distribution of neutrons. The nTOF detector consists of fast plastic scintillator coupled to a microchannel plate photomultiplier tube. The time responses of this detector system are about 0.88 ns for DT neutrons and 2.56 ns for DD neutrons. The key information of implosion experiments, such as fuel ion temperature, the fuel areal density and the bulk velocity can be obtained by using nTOF detectors.

P15

## Ultrafast plastic scintillation detector for neutron bang time detection in inertial confinement fusion

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**Abstract:** In inertial confinement fusion implosions, fuel atoms are heated and compressed directly or indirectly by laser pulse. Once the condition of thermonuclear fusion is achieved, energetic particles, such as charged ions, photons, and neutrons, are released. The time interval between the beginning of the laser pulse and the peak of neutron emission, i.e., neutron bang time, can provide important information about target performance. It offers details of the hydrodynamic response and the energy absorption of the target capsules, and contributes to the development of numerical models.

Since neutrons are produced in a very short instant, a system with a timing accuracy of less than 100-ps is needed to learn the slight variation in the physical process. The neutron bang time detection system, which has been implemented at the largest laser facility of China, is consists of a fiber bundle, a fast plastic scintillator, a microchannel-plate photomultiplier tube, and a digital oscilloscope. The system possesses a timing precision of about 30 ps for the ultrafast scintillator, and the high timing precisions of the MCP-PMT and the oscilloscope.

P16

## Preparation and performance of a new type liquid scintillator

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### Abstract

In this group, the mixed solvent of PC-LAB was used as the matrix of liquid scintillator for the first time, and the advantages and disadvantages of the two solvents were combined to optimize the overall performance of liquid scintillator. The optimum proportion of mixed solvent was studied. Based on the light yield, attenuation length and chemical activity, it was found that the mixed solvent of 20% PC-80% LAB achieved better results. The PPO and bis-MSB were successively chosen as the primary fluor and wavelength shifter. The optimal formula of liquid scintillator was explored, and the properties such as the light yield, attenuation length, decay time and compatibility with acrylic materials were characterized. The new liquid scintillator prepared by direct dissolution method and solvent extraction method were doped with Gd, and the performance of the Gd-doped liquid scintillator prepared by the two methods was compared. Recently, the group has also prepared long-wavelength liquid scintillator and studied the optimal concentration of its solute.

**Key words:** liquid scintillator; PC-LAB; long-wavelength liquid scintillator;

**P17**

## **Photoneutron generation and detection induced by laser-plasma interaction**

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### **Abstract:**

Neutrons can be emitted when laser induced electrons transport in Tantalum convertor by  $(\gamma, n)$  reaction. This new kind of neutron source has the advantages of small spot size and short pulse duration. Energy spectrum, space distribution and time evolution of photoneutrons are simulated using Monte Carlo code FLUKA. Direct and indirect photoneutron generation have been performed on the XGIII laser facility at the Laser Fusion Research Center (LFRC). A 200 times enhancement of neutron yield was obtained in indirect way comparing with the direct way.