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Performance comparison of experimental liquid scintillation cocktails

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Abstract

In this article the properties of four new luminophores, from the group of bifluorenes and other substances with multiple aromatic rings, were measured and studied for possible use in liquid scintillation, namely 1-phenyl-3-(2,4,6-trimethyl-phenyl)-2-pyrazoline, 2,2′,7,7′-tetrakis(*N*,*N*-diphenyl-amino)-9,9′-spirobifluorene, 2-(4-biphenylyl)-6-phenylbenzoxazole and 9,9′-bifluorenyl. The measurement was also done for the most commonly used luminophore (2,5-diphenyloxazole (PPO)). To improve counting efficiency the wavelength shifters were added to scintillation cocktail in particular POPOP and bis-MSB. The main effort was focused on the determination of sample counting efficiency of experimental cocktails using radionuclides Am-241, Sr-90 and H-3, especially for acidic, alkaline and neutral water solutions. The results were compared with the commercially used Aqualight scintillation cocktail.

Keywords Experimental LSC cocktails · Performance comparison · Counting efficiency · Sample load capacity · Quench resistance · Liquid scintillation

Introduction

Liquid scintillation technology [1] is a well-established method used to realize a great variety of devices ranging from relatively simple sample detection in the environmental science [2–4], industry [5, 6], biology [7, 8], safety and security [9–11], etc. to huge research experiments [12–15], where massive detectors are employed, in order to search of very rare events. Moreover, the liquid scintillation counting (LSC) has been widely used in the field of radionuclide metrology [16–20], because of its numerous advantages over conventional techniques. These benefits can be seen in high counting efficiency of homogeneous liquid scintillation mixtures for alpha particles, most of which is close to 100%, as well as for beta particles or mono-energy electrons under low quenched conditions. Furthermore, it is characterized by simple and rapid preparation of samples and it is capable to simultaneously determine alpha and beta particles. For the application of LSC, a liquid scintillation cocktail is indispensable for absorbing the energy of particulate radioactive decay and producing scintillation photons, which are detected and converted into electrical pulses by the photomultiplier tube. Commercially available cocktails for LSC consist of complex concoctions of alcohols, phosphates, polymers, and salts in an organic solvent. Three major compounds of scintillation cocktail can be distinguished based on the role they play in LSC. These compounds are solvents, fluors and emulsifiers. Since the beginning of the 1990s several efforts have been undertaken to identify other materials as potential scintillator solvents, able to avoid the drawbacks affecting the traditional choices, especially concerning safety and environmental hazards. In particular phenyl-o-xylyl ethane (PXE), linear alkylbenzene (LAB) and di-isopropylnaphthalene (DIPN) are examples of biodegradable, relatively safe solvents, with a high flash point and low toxicity compared to older types of liquid scintillator solvents. The second compound is the fluor component, or generally primary and secondary scintillators, which are responsible for converting photon energy into visible light. Today's best choice fluor mixtures is 2,5-Diphenyloxazole (PPO) with 1,4-Bis(2-methylstyryl)-benzene (*bis*-MSB) [21–24] or with 1,4-*Bis*(5-phenyl-2-oxazolyl)benzene (POPOP) [25–30] acting as a wavelength shifters. The last component is created by emulsifiers or surfactants. These are employed if, in particular, there is an effort to measure

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aqueous samples and determine their activity. The increasing demand of applications led to the introduction of cocktails based on nonionic (e.g., alkylphenol ethoxylates) and anionic (succinates, sulfonates, sulfates, etc.) surfactants [31–33]. Finally, the ideal LSC cocktail should possess the ability to distinguish between different interacting particles using different types of pulse shape analysis (PSA), e.g., pulse length index (PLI), which is utilized in Hidex devices. This solution uses discrimination between alpha and beta pulses generated in liquid scintillation cocktail is carried on via PSA circuit which compares the area of the pulse tail after 50 ns from the start with its total area, allowing discrimination between long (alpha) pulses which come from de-excitation from triplet states and short (beta) pulses which come from de-excitation from singlet states in scintillator atoms. The type of quenching agent (color or chemical), liquid scintillation cocktail, measured radionuclide, as well as liquid scintillation counter, vial type, chemical sample composition and the type of instrument, all affect alpha/beta resolution during liquid scintillation counting. Spillover (total interference) varies in the presence of quenching agents, incident alpha, and beta particle energy (greater $E_{\max}$ of a beta emitter is known to prolong the length of the signal in photomultiplier). Alpha/beta discrimination is energy dependent, with misclassification increasing with beta event energy, and it is affected by quenching by suppression of the delayed component of the scintillation pulse [34].

To find out an optimal cocktail for liquid scintillation counting (LSC), there are major aspects that need to be taken into consideration. Most important are overall cocktail counting performance and specific laboratory need. Several parameters should be taken into account in order to determine the overall performance of a scintillation. In particular, it is counting efficiency, intrinsic background contribution, quench resistance, sample stability over time, sample load capacity, sample compatibility and α/β discrimination. To fulfill specific laboratory needs for a cocktail, these parameters have to be adjusted for required detection limits, acceptable measurement uncertainty, sample volume customarily used for measurements, chemical and physical properties of routinely measured samples, desired sample throughput, and waste treatment regulations [35, 36].

In this paper 1-phenyl-3-(2,4,6-trimethyl-phenyl)-2-pyrazoline (PMP), 2,2′,7,7′-tetrakis(*N*,*N*-diphenyl-amino)-9,9′-spirobifluorene (DAS), 2-(4-biphenylyl)-6-phenylbenzoxazole (BPB) and 9,9′-bifluorenyl (BF) were investigated as a potential substituents of today's most popular mixture of PPO and *bis*-MSB used in commercially available LSC cocktails. Each of these four fluors with appropriate wavelength shifters and specific detergents were evaluated as a full-fledged LSC cocktail. The results contained in this paper present overall performance parameters for each cocktail, specifically sample load capacity, sample compatibility, counting efficiency, the figure of merit (FOM), quench resistance, and sample stability. All results were compared to commercially available LSC cocktail AquaLight.

Experimental

Devices and equipment

The measurements were carried out using Liquid Scintillation Counter 300SL (Hidex, Finland) with 20 ml polyethylene vials. The setting of the instrument was as follows: the PLI index was optimized for each fluor with appropriate wavelength shifter, time of the measurement was set to 300 s, alpha and beta window was set to full width (1-1023 channel). AquaLight (Hidex, Finland) was used as an LSC cocktail etalon for standardization and comparative measurement. Proper mixing of cocktail's components was performed by shaker IKA KS 130 basic (IKA, USA).

Chemicals

The investigated primary fluors were PMP, BF, (both Tokyo Chemical Industry, Japan), DAS and BPB, (Sigma-Aldrich, USA). The PPO (Sigma-Aldrich, USA) was utilized as a standard primary fluor used in commercial liquid cocktails. POPOP (Fluka Chemie GmbH, Germany) and MSB (Sigma-Aldrich, USA) were used as traditional wavelength shifters.

DIPN (Fisher Scientific, United Kingdom) was used as a solvent. Triton X-100 (T-100), Triton X-114 (T-144) (both Sigma-Aldrich, USA) and butyldiglycol (Carl Roth GmbH, Germany) were used as surfactants to ensure water miscibility. Scintillation cocktail AquaLight was obtained from Hidex (Finland).

Sample load capacity was determined using reagent-grade NaOH, NaCl, HCl and HNO_3 (Penta, Inc., CZ) diluted by demineralized water with a concentration of $1\ \mathrm{mol\ l^{-1}}$.

Quench resistance was measured by using CCl_4 (Sigma-Aldrich, USA) and phosphate-buffered saline (PBS) consisting of reagent grade NaCl ($137\ \mathrm{mmol\ l^{-1}}$), KCl ($2.7\ \mathrm{mmol\ l^{-1}}$), Na_2HPO_4 ($10\ \mathrm{mmol\ l^{-1}}$) and KH_2PO_4 ($1.8\ \mathrm{mmol\ l^{-1}}$), and pH was adjusted using HCl ($1\ \mathrm{mol\ l^{-1}}$) to a value of 7.4 (all Penta, Inc., CZ).

Water was obtained from a DEMIWA 5 ROSA$^{\mathrm{TM}}$ (Watek) water purification system. All other materials were ACS reagent grade and were used as received.

Water solutions of these radionuclides: Am-241 (1.67 Bq μl^{-1}), Sr-90 (0.55 Bq μl^{-1}) and H-3 (1.04 Bq μl^{-1}) which were diluted from CRM obtained from CMI (the Czech Metrological Institute).

Procedures

Sample load capacity

Every mentioned fluor was separately dissolved in a mixture of 6 ml of DIPN, 3 ml of T-100 or T-114 and 1 ml of butyldiglycol in the concentration of 1 g l^{-1}, to obtain a final volume of 10 ml of a fully-fledged LSC cocktail. To determine better properties of surfactants T-100 and T-114, both were used separately. The wavelength shifters POPOP or MSB were added to the samples containing PPO, BPB, BF and DAS as a primary fluor in the concentration of 0.01 g l^{-1}. Immediately after preparation of a cocktail, they were left in a dark place for 10 min and the background of a cocktail was counted. Additionally, one set of samples was spiked with 30 µl of Am-241 and the second set was spiked with 50 µl of Sr/Y-90. The increment of the sample solutions NaOH, NaCl, HCl and HNO_3 of given concentration in each series of vials increases in steps of 1 ml in the first vial, up to 4 ml in the last one. Each vial was counted for 300 s, Y-Gain set to 20, and PLI limit set to 0. All measurements, including background measurement, were carried out at a temperature controlled environment to attain thermal equilibrium of 22 °C.

Quench resistance

The ratio of solvent, primary fluor and emulsifier, background measurement, addition of radionuclides as well as the measurement settings were the same as stated in the first paragraph. An increasing volume of quench resistance agents was added, the steps of CCl_4 addition were 50, 100, 300 and 600 µl and for PBS ranging from 1 to 4 ml in steps of 1 ml.

Long-term stability

The ratio of solvent, primary fluor and emulsifier, background measurement, addition of radionuclides as well as the measurement settings were the same as stated in the first paragraph. The counting efficiencies were determined during periods of time, in total for 45 days.

Diffusion

To determine the matter of diffusion of cocktails components through the scintillation vials, 10 ml of a cocktail containing 6 ml of DIPN, 3 ml of T-100 or T-114 and 1 ml of butyldiglycol and PMP in the concentration of 1 g.l^{-1} was transferred both to a borosilicate vial and to a polyethylene vial. The weight of the vials was measured for 18 days. For comparison 10 ml of the Aqualight cocktail was measured at the same conditions, in borosilicate and polyethylene vials.

Counting efficiency and FOM

The ratio of fluors, solvent and emulsifiers, as well as background measurement were the same as stated in the first paragraph with two exclusions; fluors concentration for the second set varied from 0.01 to 1 g l^{-1} and only T-114 was used. The measurement was carried out for two sets of samples; one set contained the mixed addition of 30 µl of Am-241 and 50 µl of Sr/Y-90 and the second set of samples were spiked with 100 µl of H-3. For this set, the dark temperature adaptation was lengthened to 10 h and template for tritium, recommended by the producer, was used for measurement; tritium template setting was as follows: Y-Gain 35, PLI limit 16, Y offset 5, delay time 35 channels 5–130, counting time 36,000 s. Due to the nature of determination of the detection limit and figure of merit, the appropriate settings were adjusted for each cocktail separately, especially to achieve best possible α/β discrimination.

Results and discussion

Sample load capacity

Choosing the proper LSC fluor cocktail optimizes counting performance. When sample counts are low, it is best to choose a cocktail that can hold as much sample as possible and still provides sufficient counting efficiency and small background count rate. Overall, liquid scintillators should be prepared from reagents with a minimal amount of radionuclides. When dealing with aqueous samples, the limit of detection will also be influenced by the loading capacity.

The selected cocktails were evaluated to determine the sample to cocktail mixing ratio that will give the best performance. The cocktails were tested at various sample loads through its rated capacity, while holding the total geometry constant.

Sample load capacity, meaning a property of a cocktail determining the maximum amount of sample LSC cocktail will accept to form a homogeneous, single phase mixture. As can be seen in Figs. 1 and 2, better results were generally observed when T-114 rather than T-100 was used as surfactant agent. The reason was possibly due to the longer polyethoxyl group of T-100, where a number of repetition

Fig. 1 The dependency of the detection efficiencies of the mixture of DIPN, selected luminophores, butyldiglycol and T-100 on a different sample load; **a** RO water, **b** NaOH, **c** HCl, **d** HNO_3 and **e** NaCl

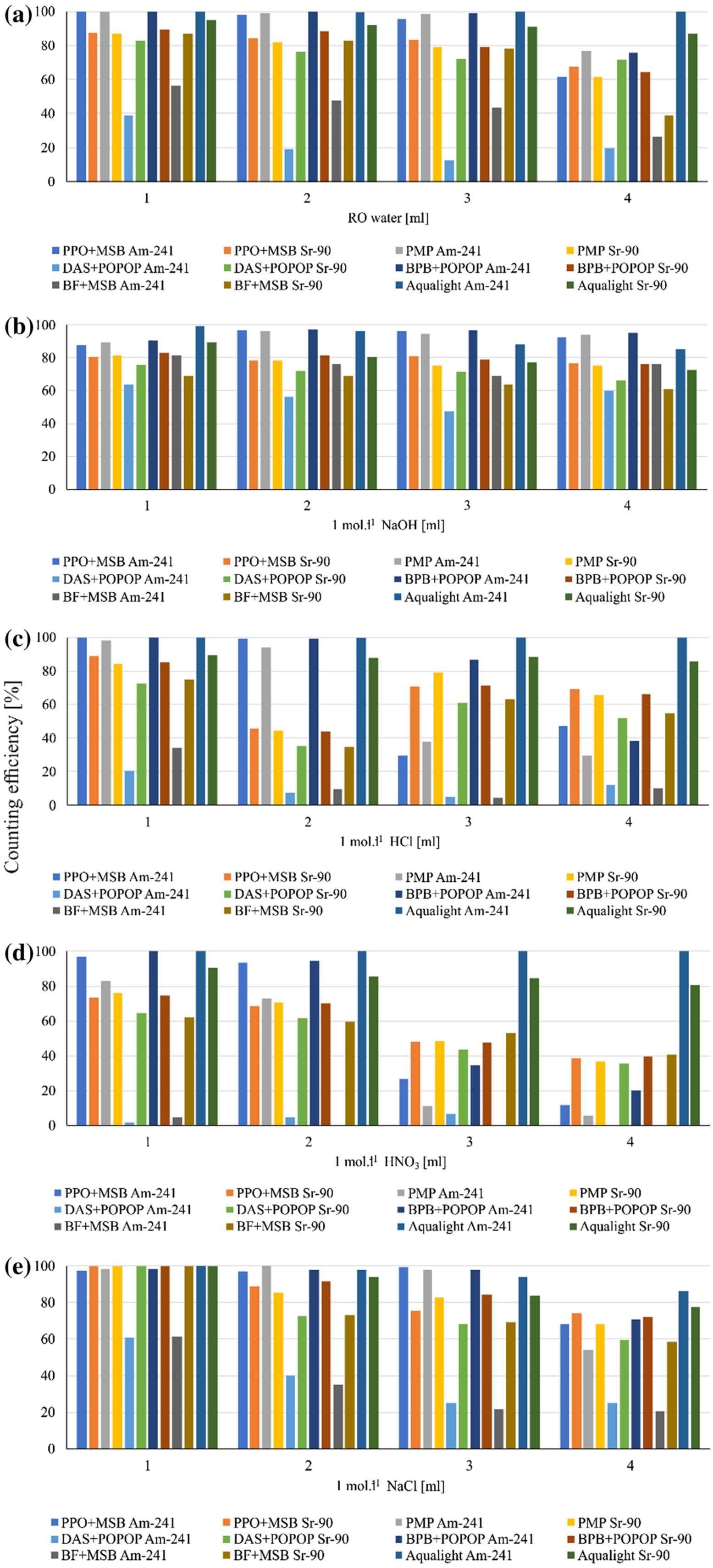
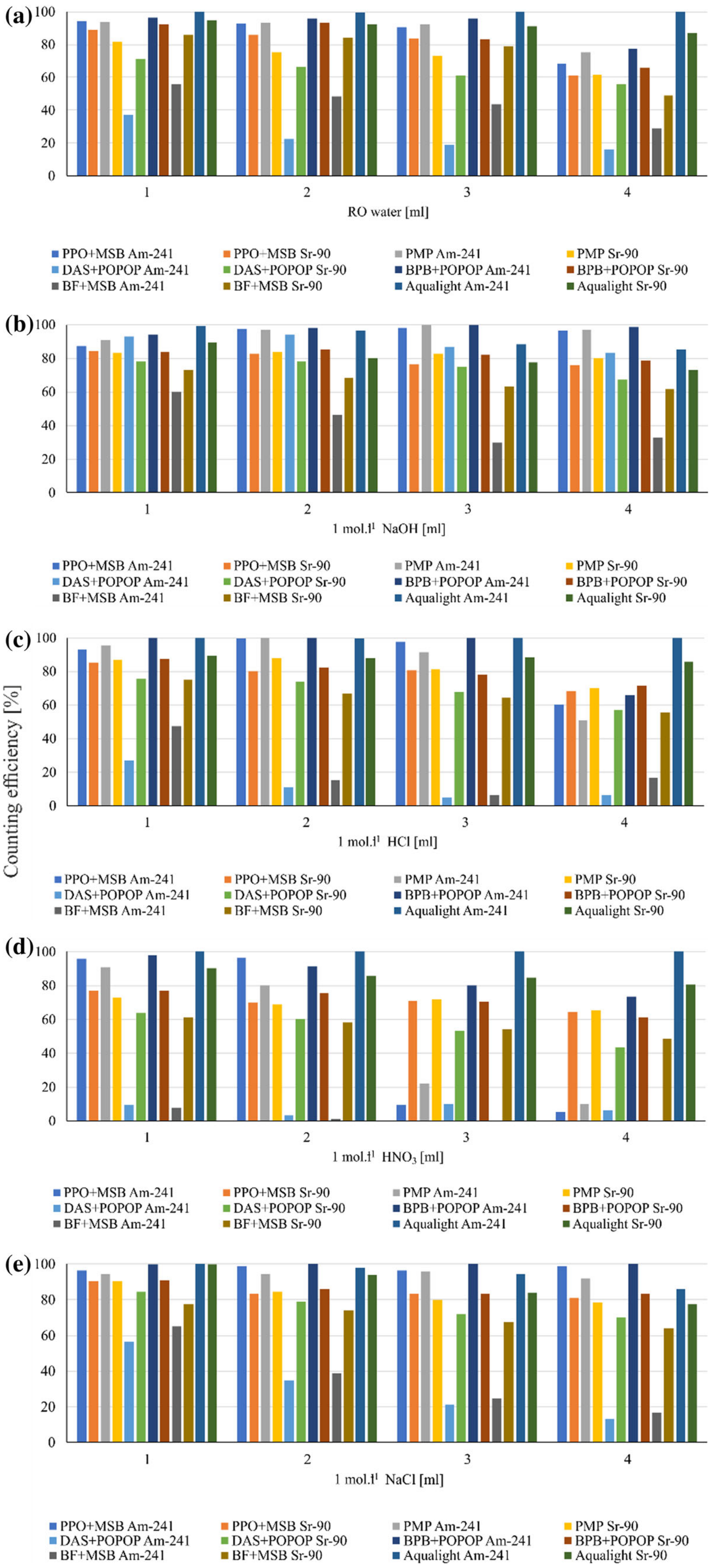


Fig. 2 The dependency of the detection efficiencies of the mixture of DIPN, selected luminophores, butyldiglycol and T-114 on a different sample load; **a** RO water, **b** NaOH, **c** HCl, **d** HNO_3 and **e** NaCl



of the functional group *n* is 8–9 instead of 7–8 of T-114. With no additions of investigated solutions all counting efficiencies were 99 ± 4%. As can be seen in Fig. 1a and 2a, additions of pure RO water caused a significant decrease only for DAS + POPOP and BF + POPOP, where the counting efficiency dropped to 20% or 30% respectively when Am-241 was measured. In the case of Sr/Y-90 the decrease was smaller, only 30% or 50% respectively. Other cocktails maintained high counting efficiency usually about 70%. AquaLight exhibited more stable behavior in counting efficiency across the investigated region of the addition of RO resulting in absolute detection efficiency for Am-241 and an only slight decrease of about 14% when Sr/Y-90 was measured. The reason was probably due to the composition of additional emulsifiers.

In the alkaline environment (Figs. 1b and 2b), with additions of NaOH, every cocktail showed high counting efficiency except for DAS + POPOP in combination with T-100, where the detection efficiency was approx. 60% for Am-241 and BF + MSB in combination with T-114, where the detection efficiency was approx. 30%. PMP and BPB + POPOP achieved better results than AquaLight, regardless of whether T-100 or T-114 was used. Addition of even 1 ml of NaOH caused in Aqualight very high spectrum deterioration mainly of two-dimensional spectra, where proper adjusting of alpha/beta separation was very demanding due to spectra overlapping while in other cocktails quality of spectra remained acceptable.

In an acid environment, with additions of HCl, higher efficiencies remained for PPO + MSB, PMP and BPB + POPOP, where the average detection efficiency was 58 ± 7% for Am-241 measurement and 53 ± 4% for Sr/Y-90 respectively. DAS + POPOP and BF + MSB dropped rapidly below 20% for Am-241 measurement and 30% for Sr/Y-90 respectively, as can be seen in Figs. 1c and 2c. Addition of 3 ml of HNO_3 caused a huge decrease in detection efficiencies below 10% for Am-241 measurement. The only one exception was observed when BPB + POPOP together with T-114 was measured. The detection efficiency was more than 70%. The average detection efficiency of Sr/Y-90 when 4 ml of HNO_3 were added was 56 ± 10%. Contrary to that, the AquaLight reached absolute detection efficiency for Am-241and 80% for Sr/Y-90 (Fig. 1d, 2d).

Addition of NaCl (Figs. 1e, 2e) significantly influenced counting efficiency only for DAS + POPOP and BF + MSB, where the detection efficiency for Am-241 dropped to 13% or 16% respectively. As in the case of NaOH, PPO, PMP and BPB exhibited higher detection efficiency for both Am-241 (97 ± 6%) and Sr/Y-90 (81 ± 3%) contrary to AquaLight, where the detection efficiency for Am-241 was 86 ± 2% and for Sr/Y-90 was 78 ± 2%. In conclusion, emulsifier T-114 showed better results than T-100, therefore in following measurements T-114 was used primarily.

Quench resistance

One of the most important factors causing a reduction in counting efficiency in LSC cocktails is quenching. The main influence is based on reducing reliability (uncertainty) of results and the susceptibility (detection limit) of gained calculated results of the method. Thus, it is important for a cocktail to have high resistance to quenching. As Fig. 3 shows, PPO + MSB, PMP and BPB + POPOP kept relatively high counting efficiency, especially with T-114 (Fig. 3b), where the counting efficiency decreased by 10% when Am-241 was measured. However counting efficiency of DAS + POPOP and BF + MSB dropped rapidly to 15% or 30% respectively, when Am-241 was measured, achieving better results with T-100. In case of Sr/Y-90 the decrease for cocktails with T-100 was approx. 20% instead of 10% for T-114.

On the contrary, all investigated cocktails suffered from high chemical quench when CCl_4 was applied. Therefore, the addition of CCl_4 ranged only from 0 to 600 µl. As Fig. 4 shows, even a small addition of 50 µl caused a significant decrease in counting efficiency for all cocktails. Only PPO + MSB and BPB + POPOP kept at least some counting efficiency after addition of 50 µl. The detection efficiency for Am-241 was approx. 15% and 35% for Sr/Y-90 respectively. The Aqualight remained relatively stable with the addition of 300 µl and dropped afterward with higher additions.

Long-term stability

LSC cocktails in some cases have to be stored for longer periods of time, after their preparation. The reasons of low stability of samples include degradation of the components, adsorption of the nuclide on the walls of the counting vial and formation of quenching in the cocktail owing to a chemical change in its composition. Thus, it is important for a cocktail to be highly stable. As Fig. 5 shows, all cocktails (containing DIPN as a solvent) shows high stability and minimum change in counting efficiencies. During a period of 45 days, measuring every 15th day, counting efficiencies were calculated for selected cocktails. For all samples change in counting efficiency did not exceed 7%. Cocktails were measured at same conditions for Am-241 and Sr/Y-90 in polyethylene vials. For comparison 10 ml of the Aqualight cocktail was measured.

Fig. 3 The dependency of the detection efficiencies of the mixture of DIPN, selected luminophores, butyldiglycol and T-100 (**a**) and T-114 (**b**) on a PBS quenching agent

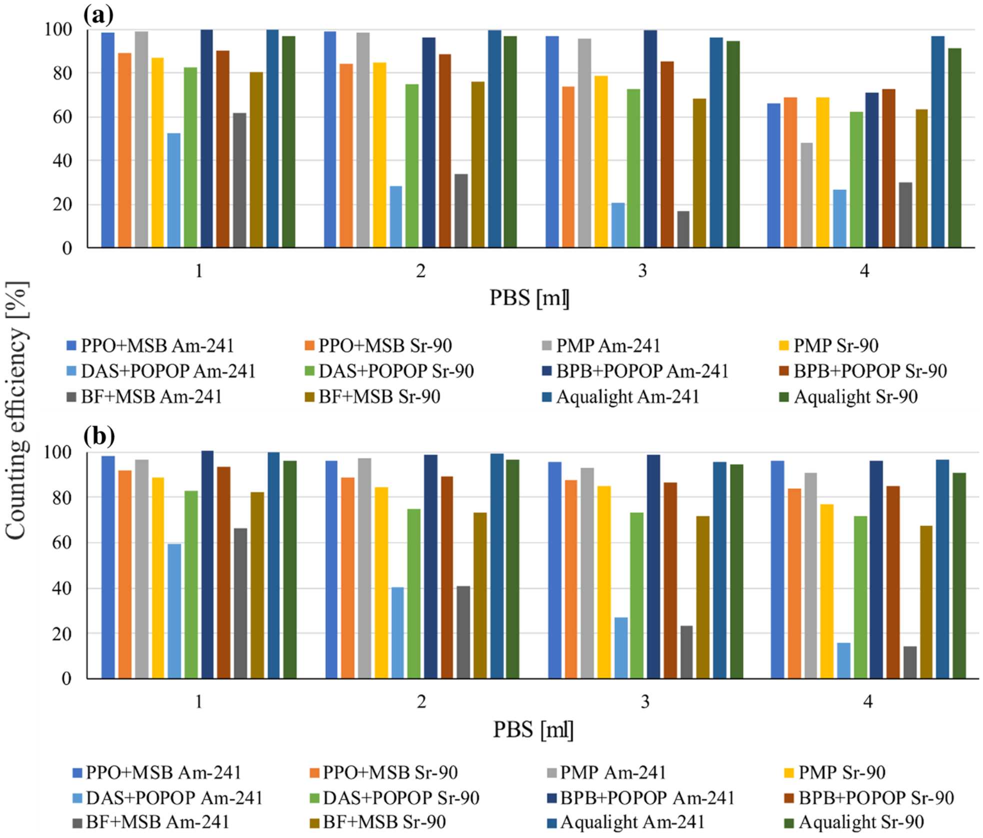
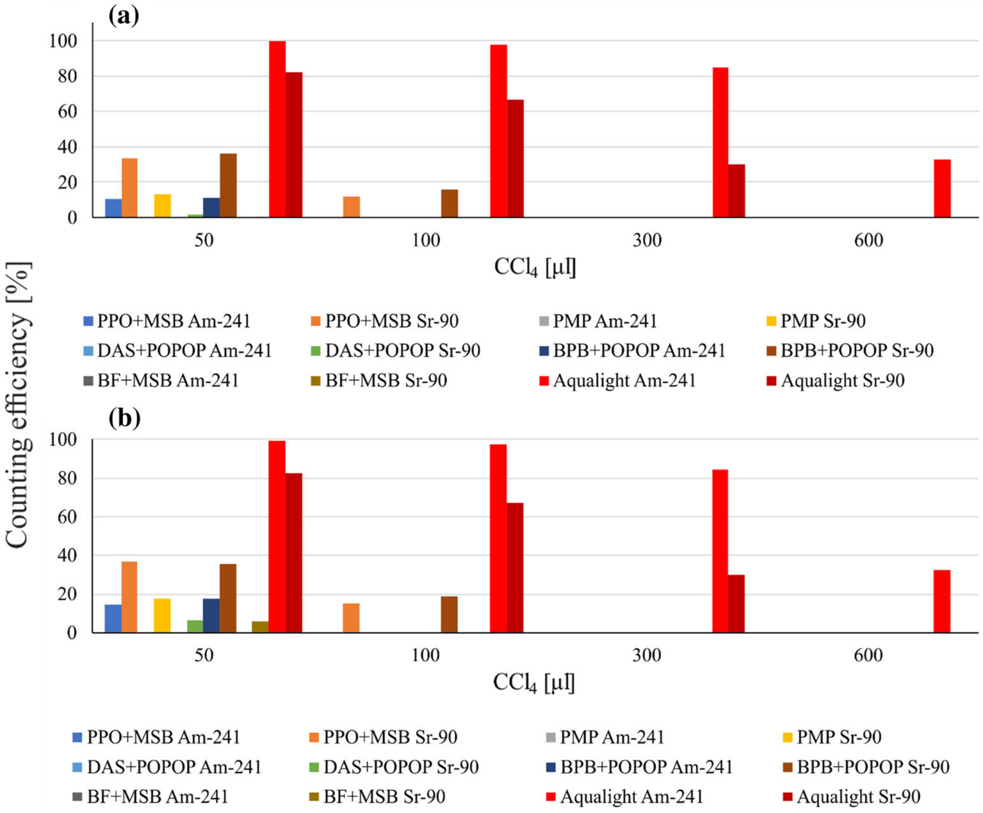


Fig. 4 The dependency of the detection efficiencies of the mixture of DIPN, selected luminophores, butyldiglycol and T-100 (**a**) and T-114 (**b**) on a CCl_4 quenching agent



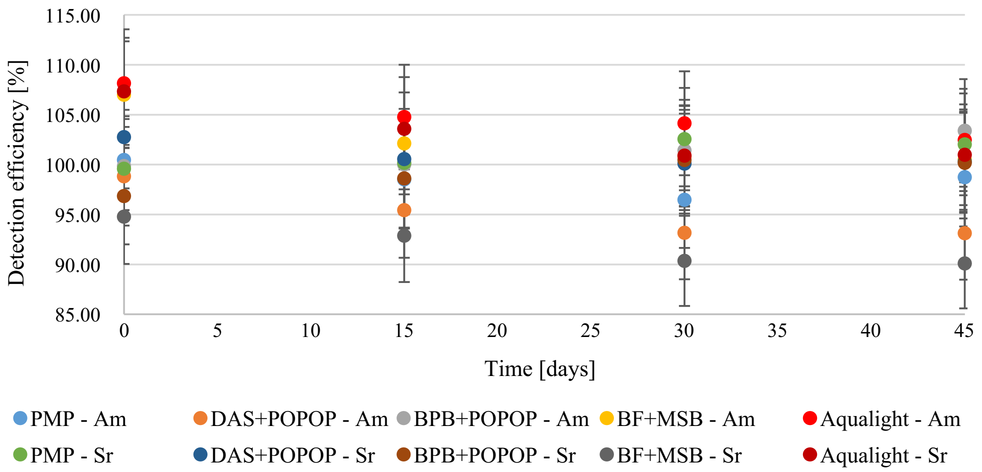


Fig. 5 Dependence of the detection efficiency on the elapsed time

Diffusion

Another reason that should be taken into consideration, that can influence the stability of cocktail, is the diffusion of a solvent through a vial. Both plastic and glass LSC vials have certain disadvantages in terms of background, solvent permeability, fragility and transparency. Polyethylene plastic vials are permeable when stored containing the traditional fluor solvent such as benzene, toluene etc. Diffusion through vials was determined by weighing both borosilicate and polyethylene vials four times during the period of 18 days. The measurement results did not show any measurable changes in weight, indicating extraordinary stability of DIPN contrary to volatile solvents such as toluene.

Counting efficiency and FOM

The signal-to-noise ratio is an important instrument to determine the detection limit of the measurement device. That applies to LSC and is expressed as a function of counting efficiency (*E*) and background contribution (*B*) and is represented as the FOM ($= E^2/B$). Original concentration of primary luminophores tested for tritium was 1 g l^{-1} and the results did not achieve very high detection efficiency. Therefore, the lower concentrations were tested, but it didn't exhibit any improvement, rather the opposite, as can be seen in Table 1.

The oxazole molecules (PPO and BPB) exhibited the highest detection efficiency of about 86% of detection efficiency achieved by AquaLight. The detection efficiency of PMP and BF was ten percent less than the previous luminophores. The worst detection efficiency of about 60% of AquaLight was observed when DAS + POPOP was measured. Table 2 represents results achieved by measuring the mixture of Am-241 and Sr/Y-90 radionuclides. In order to properly determine the detection efficiency and FOM, the PLI optimization throughout spill over correction was performed. The highest detection efficiencies, which were comparable with those observed for AquaLight, were achieved for PMP and DAS luminophores, while the lowest detection efficiencies were observed for DAS and BF luminophores. The reason for the low efficiency and therefore a small number of FOM was possibly due to the ''micelle effect'' and is in accordance with [20, 37, 38]. As can be seen in the Tables 1 and 2, this problem can be particularly acute for low-energy Auger-electron-emitting radionuclides. When an electron is emitted from radionuclide, it loses energy while traversing the aqueous material within a micelle. This energy is not deposited in the scintillation material, and so does not result in scintillation light. The distance that an electron must travel through an aqueous medium prior to interacting with the organic scintillator is therefore of interest when calculating theoretical scintillation efficiencies for a particular radionuclide in a particular scintillation cocktail.

Since the background of the cocktails was many times higher than the background of AquaLight, the origin of this phenomenon was investigated. It turned out that the major contributor to the afterglow is a connection of DIPN with nonionic surfactants (Fig. 6), therefore relatively high values of background caused low values of FOM.

Conclusion

New experimental cocktails investigated in this paper reported moderate to high sample load capacity for most of the selected samples, especially for those exhibiting neutral or alkaline properties, such as NaCl and NaOH. On the other hand, acidic samples caused lower counting efficiency even for small volume. Regarding a property of quench resistance, cocktails proved to have relatively high resistance using PBS, however, examined cocktails did not

Table 1 Detection efficiency and FOM of selected cocktails of H-3 for different concentrations of primary fluor

Fluor + wavelength shifter	Counting efficiency of H-3 [100 µl]based on the concentration of primary fluor [g l^{-1}]			FOM		
	1	0.1	0.01	1	0.1	0.01
PPO + MSB	37 ± 2	31 ± 1	18 ± 1	15	7	0.3
PMP	33 ± 1	7 ± 1	N/A	17	0.3	N/A
DAS + POPOP	25 ± 1	19 ± 2	N/A	10	1.5	N/A
BPB + POPOP	37 ± 1	25 ± 2	12 ± 2	18	3	0.2
BF + MSB	34 ± 2	26 ± 1	15 ± 2	14	4	0.2
AquaLight	43 ± 2	29				

Table 2 Detection efficiency and FOM of selected cocktails for a mixture of Am-241 and Sr/Y-90

Fluor + wavelength shifter	Y-gain	PLI limit	Am-241		Sr/Y-90	
			Detection efficiency [%]	FOM	Detection efficiency [%]	FOM
PPO + MSB	20	7	88 ± 3	1042	96 ± 1	38
PMP	20	8	100 ± 1	1324	64 ± 1	20
DAS + POPOP	20	6	82 ± 1	513	90 ± 2	46
BPB + POPOP	20	7	105 ± 5	1159	94 ± 3	41
BF + MSB	20	9	93 ± 4	784	82 ± 4	28
AquaLight	30	11	101 ± 1	1905	99 ± 2	171

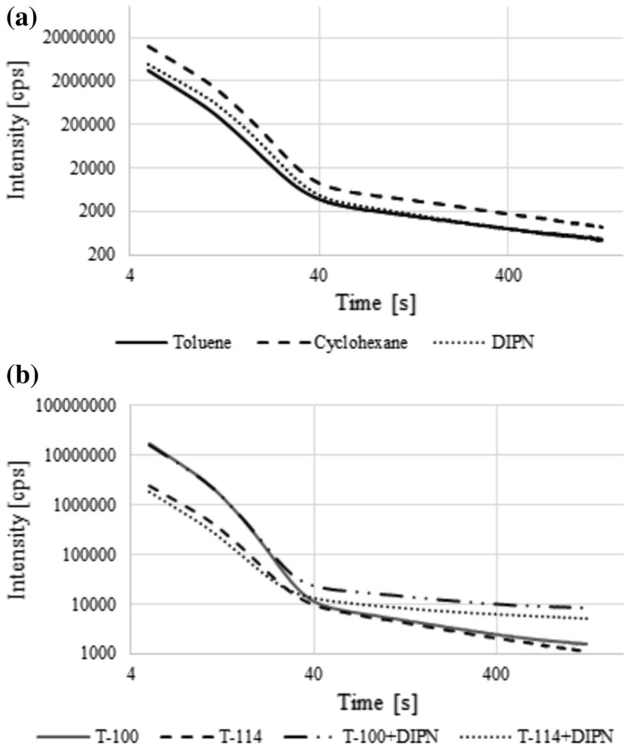


Fig. 6 Phosphorescence relaxation of different solvents (**a**) and surfactants (**b**)

achieve as good results for CCl_4 as Aqualight, cocktail used as a standard. The possible reason for this is because of its composition, containing additional emulsifiers. In a manner of long-term stability, all cocktails were to be considered as highly stable, reporting only a minimum change in counting efficiency during the investigated time. Diffusion of a cocktail's components, especially solvent (DIPN), was monitored and was resolved as to have very small to negligible effect regarding properties of cocktails. Lastly, counting efficiency and figure of merit were calculated for a tritium and for the mixture of Am-241 and Sr/Y-90. Cocktails exhibited the high capability of α/β discrimination for a mixture of radionuclides and achieved satisfactory results of FOM. The low counting efficiency and FOM for H-3 were possibly due to the "micelle effect". Overall, these experimental cocktails show a promising possible substitute for commercially used scintillation cocktails when utilized with proper emulsifiers that would increase especially quench resistance of the cocktails and lower the background of the cocktail at the same time.

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