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

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Abstract
In this paper a study for the selection of an NPE (nonylphenol ethoxylates)—free cocktail is discussed in order to be used for our routine liquid scintillation counting measurements. The NPE are added in the REACH (Registration, Evaluation, Authorization and Restriction of Chemicals)—list of amendments to Annex XIV, as chemicals which can't be used anymore (Commission Regulation (EU) 2017/999). Nine NPE-free cocktails were bought from different producers and investigated with respect to the homogeneity, background level and influence on counting efficiency for different radionuclides. Several sample matrices were considered for direct measurement of ^{3}H and ^{14}C. Based on this study we could select the best cocktail which fulfills our requirements for routine analyses.

Keywords Liquid scintillation cocktail · NPE-free cocktails · REACH

Introduction

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 however recently added in the REACH (Registration, Evaluation, Authorization and Restriction of Chemicals)—list of amendments to Annex XIV, as chemicals which can't be used anymore [4] for routine laboratory work. A first study which reported the performance of an NPE-free cocktail was published by Varlam et al. in 2019 [3]. They report about the performance of Prosafe LT + cocktail for ^{3}H determination in aqueous samples and concluded that this cocktail fulfilled the criteria needed for their routine analyses [3]. Radionuclide metrology is very much relying on liquid scintillation counting where two models, the CIEMAT/NIST approach and the TDCR method are commonly used in radionuclide standardization [5].

Recently, also another approach in scintillation counting is becoming promising which is based on plastic scintillation microspheres/resins (PSm and PSresin respectively). Several publications report the application of PSm/PS-resins for quantification of the different radionuclides in the environmental samples [6–10].The advantages and disadvantages of PSm are described in detail in Handbook of Radioactivity Analyses, 2020 [11]. Reducing the waste costs, high counting efficiencies also for alpha-emitter radionuclides, possibility of reuse them, possibility to impregnate them with different extractants and then combine the separation and the measurement are only a few of the many advantages [11].

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 currently used for routine measurements. A total of nine cocktails from several producers were selected and investigated in this study. Different parameters were tested, such as homogeneity/compatibility with the sample material, inherent background level and the influence on counting efficiency. The specific media obtained after chemical separations of the sample, resulting in a fraction containing a single radionuclide were investigated for several radionuclides (such as ^{90}Sr, ^{210}Pb, ^{63}Ni, ^{99}Tc, ^{14}C in Carbosorb E), but also

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common sample materials (such as water, clay water, urine) where direct measurement of several radionuclides (such as ^{3}H or ^{14}C) after mixing with the cocktail 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 and selected based on a similar study of Verrezen et al. [12].

Experimental

Materials

Cocktails

The overview of the different types of cocktails used in this study is given in Table 1. A total number of 9 cocktails was bought from different producers. All of them are NPE-free cocktails and only one of them is DIN—free, namely Pico Fluor plus. Since one of the most important components of the liquid scintillation cocktail is the surfactant, the mixture of these chemicals present in the studied cocktails are given also in Table 1. The role of the surfactant is to ensure a homogeneous mixture between the organic phase of the cocktail and the aqueous phase where the radionuclide to be determined is present by forming a micro-emulsion [13].

Tracers and matrices

All tracers used in this study were traceable to SI unit and an overview is given in Table 2 together with the type of matrix specific to each investigated radionuclide. Traceability of the radioactivity (Bq) was assured by using working solutions resulting from gravimetrically made dilutions of the certified reference solutions.

Table 1 Overview of the different types of cocktails used in this study

Nr	Cocktail name	Distributing company	Producing company	NPE-free	DIN-free	Surfactant/cosurfactants
1	PICO Fluor plus	Perkin Elmer	Perkin Elmer	yes	yes	Isotrydecylalcohol; phosphoric acid,2-ethylhexyl ester; 2-(2-butoxyethoxy)ethanol; sodium dioctyl sulphosuccinate; 2,2'-iminodiethanol; fattyalcohol polyglycolether
2	Aqualight +	Mirion	Hidex	yes	no	Alcohols, secondary C11–15,ethoxylated; sodium dioctyl sulphosuccinate
3	Proflow G +	Triskem	Meridian	yes	no	Alcohols, secondary C11–15,ethoxylated; 2-(2-butoxyethoxy)ethanol; diethanolamine salt of phosphate ester
4	Prosafe TS +	Triskem	Meridian	yes	no	Alcohols, secondary C11–15,ethoxylated; phosphate ester
5	Prosafe +	Triskem	Meridian	yes	no	Alcohols, secondary C11–15,ethoxylated; 2-(2-butoxyethoxy)ethanol; phosphate ester
6	Prosafe HC +	Triskem	Meridian	yes	no	Alcohols, secondary C11–15,ethoxylated; docusate sodium; phosphate ester
7	Prosafe FC +	Triskem	Meridian	yes	no	Alcohols, secondary C11–15,ethoxylated; phosphate ester; docusate sodium
8	Prosafe LT +	Triskem	Meridian	yes	no	Alcohols, secondary C11–15,ethoxylated; sodium dioctyl sulphosuccinate
9	Quickflow 2 +	Zinser analytic	Zinser analytic	yes	no	Alcohols, secondary C11–15,ethoxylated; 2-(2-butoxyethoxy)ethanol; phosphate ester, diethanolamine salt

Table 2 Overview of the tracers and type of matrices investigated in this study

Nr	Radionulide	Distributing company	Type of matrix
1	^{3}H	PTB, Germany	RO-water, urine, clay water
2	^{14}C	LEA, France	RO-water, urine, clay water, Carbosorb E
3	^{90}Sr	LEA, France	0.05 M HNO_3 (5 ml)
4	^{210}Pb	PTB, Germany	6 M HNO_3 (1 ml)
5	^{99}Tc	LEA, France	0.5 M HNO_3 (5 ml)
6	^{36}Cl	PTB, Germany	0.1 M NH_4SCN (5 ml)
7	^{63}Ni	LEA, France	3 M HNO_3 evaporated and dissolved in water

All *chemicals* used in this study, such as acids or chemicals used for preparation of final measurement sources (e.g. Sr or Pb—oxalate) are of analytical purity.

All the samples were measured using a low-level liquid scintillation counter, Quantulus 1220™.

Liquid scintillation vials

Low potassium borosilicate vials were used for visual evaluation of the homogeneity and polyethylene vials were used for measurements.

Methods

The approach followed in this study for the final selection of best NPE-free cocktail(s) for our routine analyses is presented in Fig. 1. The first step was identification of the candidate cocktails (see Table 1), then several parameters were considered, such as homogeneous mixture with a direct sample (urine, clay water, RO-water) or with a specific media resulting from a radiochemical separation of the considered radionuclide (see Table 2). Another parameter investigated was FOM (Figure of Merit), defined as E^2/B (E is counting efficiency, B is background level) [2]. The final volume of all the mixtures was 20 ml and the measurement temperature 10 °C. For background level determination the counting time was 100 min and for counting efficiency 30 min.

Homogeneity using direct mixing and specific media with the cocktails

Blank Reverse Osmosis (RO)—water, urine and clay water samples were directly mixed with the cocktails in different sample to cocktail ratios (sample load). The sample load was defined by the ratio: $V_{(sample)}/V_{(total)}$, where $V_{(total)} = V_{(sample)} + V_{(cocktail)}$. The mixing was performed by hand shaking using glass vials. After mixing, the samples were placed in the LS counter for cooling down for at least 1 h. Afterwards the homogeneity of the resulting mixture was visually evaluated (phase separation, turbidity of the mixture, etc.) and the corresponding loading capacity of the cocktail was determined (as the ratio of the volume of the sample in the mixture to the total volume of the mixture). The specific matrices (obtained after radiochemical separation), were also mixed with the cocktails and the homogeneity of the resulting mixtures was evaluated.

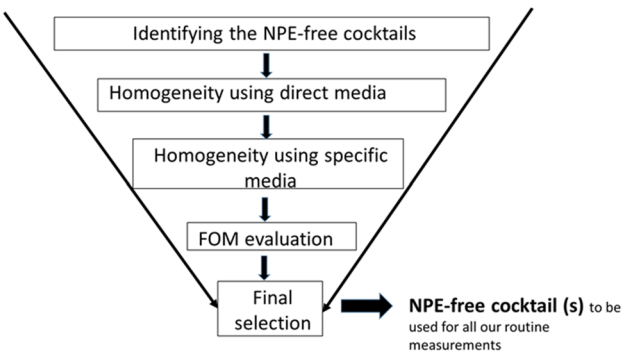

Fig. 1 The decision approach considered in this study for selection of NPE-free cocktail(s)

Figure of merit (FOM) evaluation

The FOM is defined as E^2/B (E is counting efficiency, B is background level) [2]. FOM was chosen as a parameter to compare the performance of the investigated cocktails. The samples which successfully fulfilled the condition of being a homogeneous mixture, were measured using liquid scintillation counting in order to determine the background level. A similar mixture was prepared and spiked with the corresponding tracers in order to determine the counting efficiency.

All the results were compared with the presently used cocktail Optiphase HiSafe 3 (contains NPE). This cocktail was selected in the past based on a similar study described by Verrezen et al. [12].

Results and discussion

Homogeneity using direct mixing and loading capacity

Clay water samples

In Fig. 2, the degree of homogeneity of the investigated cocktails with clay water samples is compared with OptiPhase HiSafe 3. In order to keep the detection limit (DL) for ^{3}H (13.76 Bq l^{-1}) and ^{14}C (3.57 Bq l^{-1}) in clay water samples comparable with the one that is obtained currently using Optisafe HiSafe 3 cocktail, a minimum sample load of 25% is needed. As we can see from Fig. 2, six cocktails fulfill this condition. Two of them, namely Prosafe FC +, Prosafe TS + gave a milky mixture and one of them, namely ProSafe +, gave a turbid but still homogeneous mixture. Our findings are in agreement with the data provided by the manufacturer [14]. Mixtures with a milky appearance or with the occurrence of phase separation are considered to be not acceptable for LSC counting.

Urine samples

A loading capacity of at least 20% is needed in order to maintain the same detection limits achieved with OptiPhase HiSafe 3. In Fig. 3, the homogeneity degree of the 9 selected

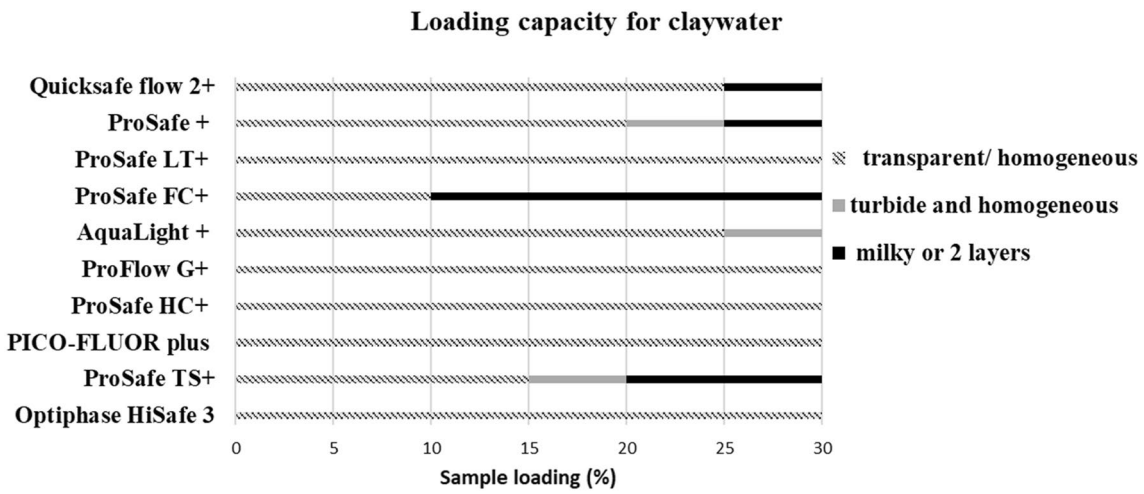

Fig. 2 Overview of the homogeneity and loading capacity of the 9 investigated cocktails using clay water sample

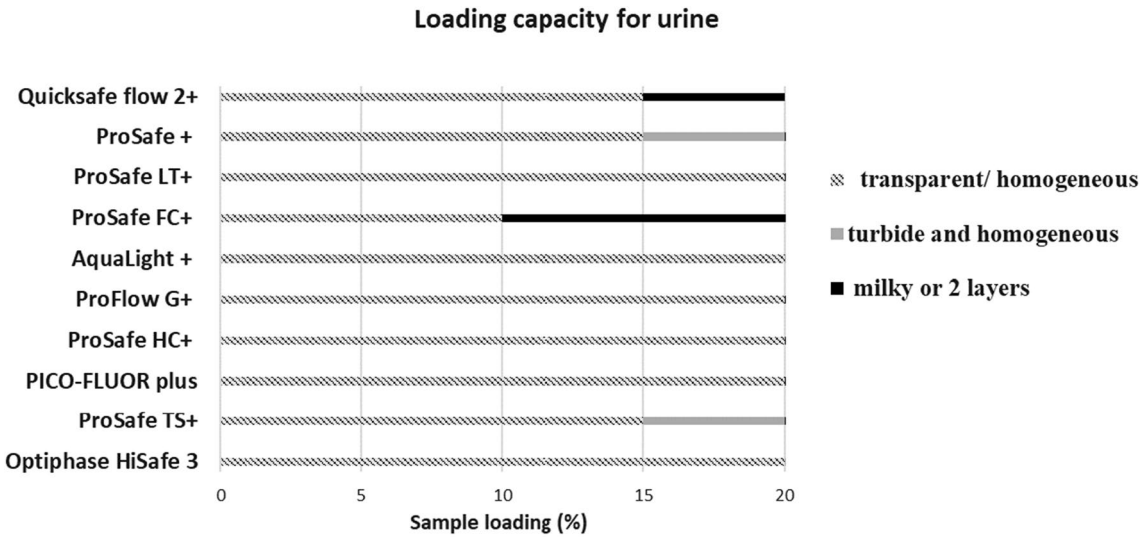

Fig. 3 Overview of the homogeneity and loading capacity of the 9 investigated cocktails using urine sample

cocktails for urine samples is presented. As can be observed five of the nine cocktails meet the condition of 20% loading capacity. Two of them, namely QuickSafe flow2 + and ProSafe FC +, only reach a loading capacity of 15 and 10%, respectively. ProSafe + and ProSafe TS +, can reach the 20% sample load, however at this value the mixture is turbid but still homogeneous. Our findings are in agreement with the data provided by the manufacturer for ProSafe HC +, Prosafe TS + and ProSafe + cocktails [14].

Reversed osmosis water samples

For reversed osmosis water a maximum loading capacity is needed for our laboratory in order to obtain a comparable detection limit with the samples mixed with OptiPhase HiSafe 3. In Fig. 4 the homogeneity degree of different sample load is shown for each of the 9 selected cocktails. As can be observed, only one cocktail, namely QuickSafe flow2 +, meets the condition of 45% loading capacity. ProSafe LT + and PicoFluor Plus, achieve a 40% loading capacity with a homogeneous mixture but a turbid appearance. Aqualight + cocktail achieved a 35% loading capacity with a homogeneous but turbid sample. The remaining five cocktails: ProSafe +, ProSafe FC +, Proflow G +, ProSafe HC +, ProSafe TS +, do not produce a homogeneous sample when loaded with at least 35% sample load. Our findings are in agreement with the data provided by the manufacturer, exept for ProSafe HC + cocktail, where we obtained a loading capacity using RO-water of 25% [14].

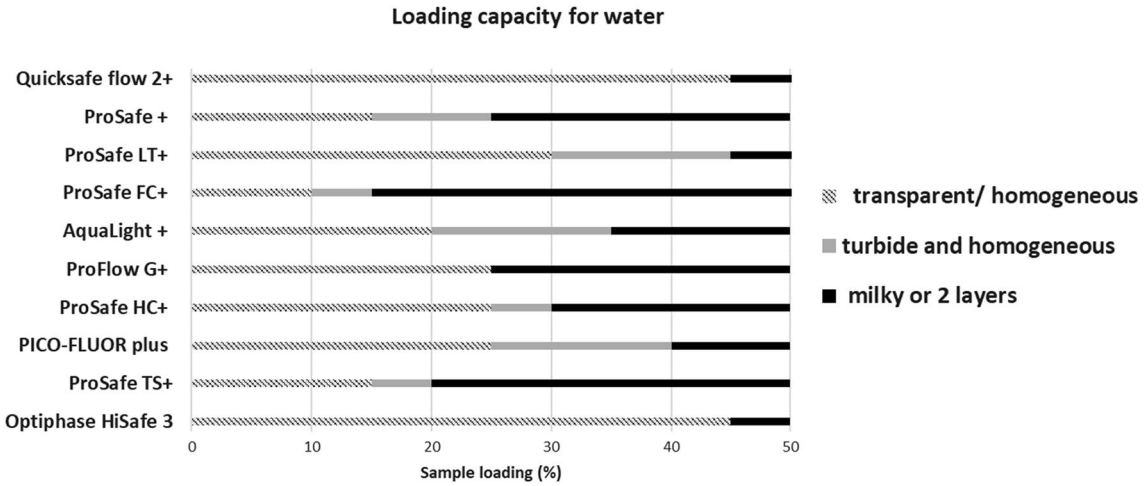

Fig. 4 Overview of the homogeneity and loading capacity of the 9 investigated cocktails using RO water sample

Based on the evaluations described above, it was decided that three cocktails, namely ProSafe +, ProSafeFC +, ProSafe TS + were not considered for further testing using specific media. These cocktails didn't fulfill the requirements for the minimum loading capacity. The remaining six cocktails were further tested for homogeneity using specific media resulting from a radiochemical separation. However, three of the cocktails, namely ProSafe LT +, Pico-Fluor plus and Aqualight +, even if they gave a turbid, but homogeneous, mixture were considered for further testing since obtaining a slightly lower counting efficiency did not compromise the final DL. Due to the turbidity, the counting efficiency was mainly affecting ^{3}H determinations.

As can be observed in Table 1, the main surfactant used in all the LS cocktails is based on ethoxylates. As co-surfactants, different chemicals were used, such as sodium dioctyl sulphosuccinate (in 5 of the studied cocktails), phosphate esters (in 7 of the studied cocktails) and diethanolamine salt (in 3 of the studied cocktails). The sulphosuccinates are known to improve the micro-emulsion formation, however the performance of the phosphate esters is better especially for difficult matrices [12]. When the two best performing cocktails from Fig. 4 are compared, the highest sample load is obtained with Quicksafe Flow 2 + followed by, Prosafe LT +. The first cocktail contains phosphate ester, while the second one contains dioctyl sulphosuccinate. Pico-Fluor plus cocktail contains both chemicals, phosphate ester and dioctyl sulphosuccinate, but the sample load is lower than Quickflow 2 +.

Homogeneity using specific media (after radiochemical separation) with the cocktails

In Table 2 (see "Materials" and "Methods" sections) the specific media used for this study are given for each radionuclide investigated. The results of these tests are given in Table 3. The cocktails are selected based on visual evaluation of the homogeneity of the mixture. As can be seen, Carbosorb E, Quicksafe flow2 + and AquaLight + cocktails can't be used since a turbid mixture is obtained. Picofluor plus cocktails gave turbid mixtures with the final medium for ^{99}Tc (5 ml of 0.5 M HNO_3) and ^{210}Pb (1 ml 6 M HNO_3). ProSafe HC + was also not compatible with the media of ^{90}Sr (0.05 M HNO_3). All the other cocktails meet the homogeneity requirement. All six cocktails were considered further

Table 3 Overview of the selected cocktails which fulfill the homogeneity requirement

Matrix	^{14}C Carbosorb E	^{99}Tc	^{90}Sr	^{210}Pb	^{36}Cl	^{63}Ni
Selected cocktails	1.ProSafe LT +	1.Quicksafe flow2 +	1.Quicksafe flow2 +	1.Quicksafe flow2 +	1.Quicksafe flow2 +	1.Quicksafe flow2 +
	2.ProFlow G +	2.ProSafe LT +	2.ProSafe LT +	2.ProSafe LT +	2.ProSafe LT +	2.ProSafe LT +
	3.ProSafe HC +	3.AquaLight +	3.AquaLight +	3.AquaLight +	3.AquaLight +	3.AquaLight +
	4.Pico-Fluor Plus	4.ProFlow G +	4.ProFlow G +	4.ProFlow G +	4.ProFlow G +	4.ProFlow G +
		5.ProSafe HC +	5.Pico-Fluor Plus	5.ProSafe HC +	5.ProSafe HC +	5.ProSafe HC +
					6.Pico-flour plus	6.Pico-flour plus

for determination of the background level and counting efficiency.

Figure of merit (FOM) evaluation

Based on measurements of a blank sample and the counting efficiency, the FOM parameter was determined for different matrices and radionuclides. The uncertainty components which are taken into account for the final expanded uncertainty calculation are: weighing of the tracer, activity of the tracer (from the certificate), dilution factor, counting statistics of the blanc and the sample. The obtained values are compared with the values that we presently use in our routine procedure. In order to take a decision, we selected the three best performing cocktails.

Clay water samples

In Figs. 5 and 6, FOM for clay water samples and ^{3}H and ^{14}C (sample load 25%), respectively, are presented. We decided to select the AquaLight +, ProSafe LT +, ProSafe HC + (see Table 4), since the FOM values are close to the one obtained with OptiPhase Hisafe 3.

Urine samples

In Figs. 7 and 8, FOM for urine samples containing ^{3}H and ^{14}C, respectively, at 20% sample load are presented. We decided to select the Pico-fluor plus, ProSafe LT +, AquaLight + (see Table 4), since the FOM values are close to the one obtained with OptiPhase Hisafe 3.

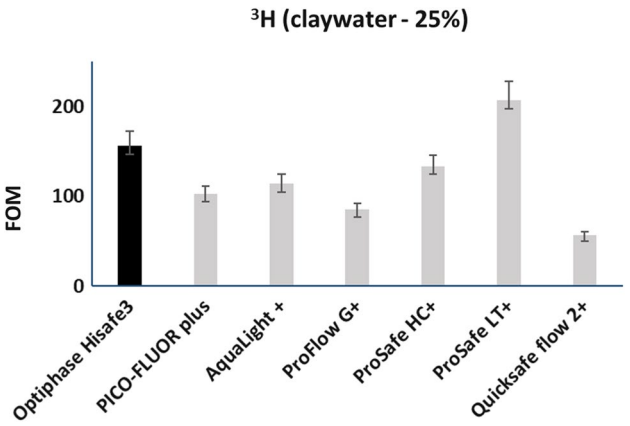

Fig. 5 Overview of FOM performance for ^{3}H in clay water samples (sample load 25%) using six cocktails (grey bars) and compared with routinely used OptiPhase Hisafe3 (black bar). Uncertainty is given as expanded uncertainty ($k = 2$)

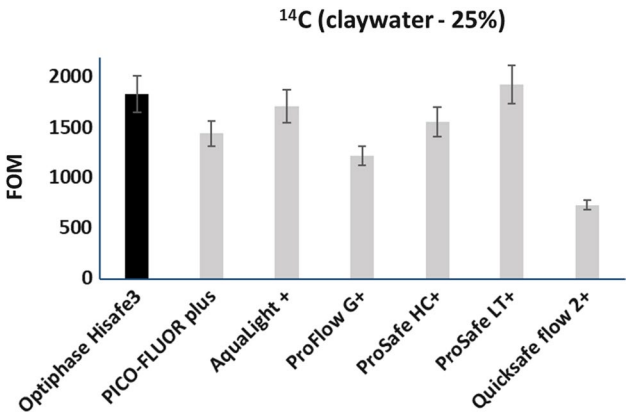

Fig. 6 Overview of FOM performance for ^{14}C in clay water samples (sample load 25%) using six cocktails (grey bars) and compared with routinely used OptiPhase Hisafe3 (black bar). Uncertainty is given as expanded uncertainty ($k = 2$)

Reversed osmosis water

In Figs. 9 and 10, FOM for RO water samples, ^{3}H and ^{14}C, and the selected cocktails based on homogeneity study are presented. We decided to select the Picofluor plus, ProSafe LT + and Aqualight + cocktails(see Table 4), since the FOM values are close to the one obtained with OptiPhase Hisafe 3.

Specific media and radionuclides

When evaluating the FOM for specific matrix and radionuclides we observed two distinct groups of radionuclides. Based on this, we decided to perform the evaluation of FOM in two groups: (1) ^{14}C in Carbosorb E, ^{63}Ni and ^{36}Cl and (2) ^{99}Tc, ^{90}Sr and ^{210}Pb.

Evaluation of FOM for ^{14}C in Carbosorb E, ^{63}Ni and ^{36}Cl The results of the FOM for ^{14}C, ^{63}Ni and ^{36}Cl are presented in Figs. 11, 12, and 13. For ^{14}C in Carbosorb, the only cocktail which gave comparable results with the currently used OptiPhase Hisafe 3 is the Picofluor plus cocktail. The rest of the cocktails gave much lower results. In case of ^{63}Ni and ^{36}Cl comparable results with OptiPhase Hisafe 3 were obtained for Pico-fluor plus, ProSafe LT +, AquaLight +.

Evaluation of FOM for ^{99}Tc, ^{90}Sr and ^{210}Pb The results of the FOM for ^{99}Tc, ^{90}Sr and ^{210}Pb are presented in Figs. 14, 15, and 16. For ^{99}Tc comparable results with OptiPhase Hisafe 3 were obtained for AquaLight +, ProSafe LT +, Quicksafe flow 2 +. For ^{90}Sr, AquaLight +, Pico-fluor plus and Prosafe HC + cocktails were the three giving results close to the OptiPhase Hisafe 3, while for ^{210}Pb, Proflow G +, Prosafe HC + and Quicksafe flow 2 + gave results much better than the one currently used.

Table 4 Overview of the selected cocktails based on FOM evaluation for different matrices

Matrix	Clay water (^{3}H and ^{14}C)	Urine (^{3}H and ^{14}C)	RO water (^{3}H and ^{14}C)	Carbosorb E, ^{63}Ni, ^{36}Cl	^{99}Tc	^{210}Pb, ^{90}Sr
Selected cocktail based on FOM	AquaLight + ProSafe LT + ProSafe HC +	Pico-fluor plus ProSafe LT + AquaLight +	Pico-fluor plus AquaLight + ProSafe LT +	Pico-fluor plus ProSafe LT + AquaLight +	AquaLight + ProSafe LT + Quicksafe flow2 +	AquaLight + ProSafe HC + Pico-fluor plus

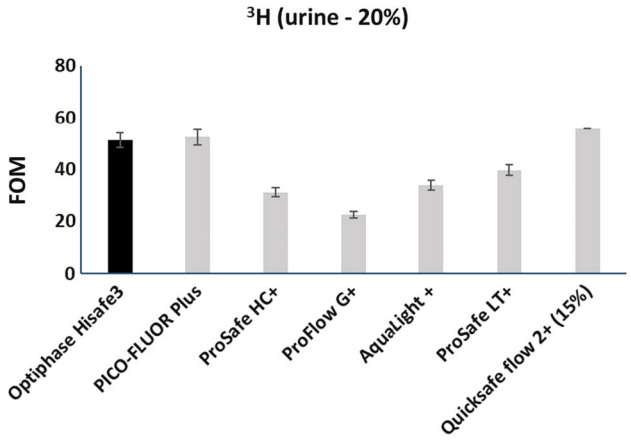

Fig. 7 Overview of FOM performance for ^{3}H in urine samples using six cocktails (grey bars) and compared with routinely used OptiPhase Hisafe3 (black bar). Uncertainty is given as expanded uncertainty ($k=2$)

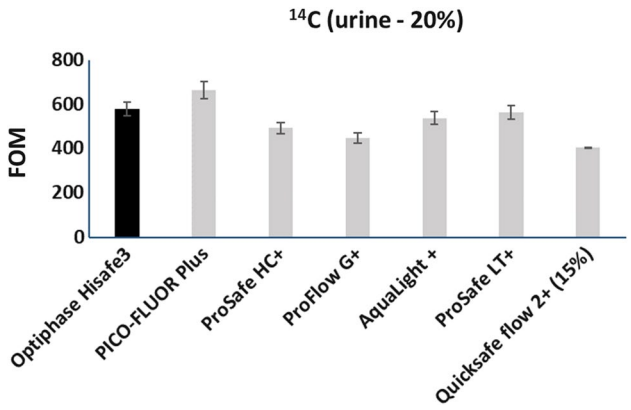

Fig. 8 Overview of FOM performance for ^{14}C in urine samples using six cocktails (grey bars) and compared with routinely used OptiPhase Hisafe3 (black bar). Uncertainty is given as expanded uncertainty ($k=2$)

Conclusions

Based on the Table 4 where all the "best performing" cocktails for the different matrices and radionuclides are summarized, we conclude that ProSafe LT + and Aqualight + cocktails fulfill best our criteria (minimum sample load, type of matrix, needed detection limit). The composition of these two cocktails is similar, both contain dioctyl sulphosuccinate as co-surfactant. Picofluor plus was selected as the best performing cocktail to be used with Carbosorb E. However, for ^{90}Sr and ^{210}Pb we observe that the candidate cocktail ProSafe LT + performance is inferior than the one presently used in routine measurements. Even so, we decided to use Prosafe LT + for all our routine analyses. Picofluor plus cocktail also can be considered as one of the best performing scintillation cocktails. Since the

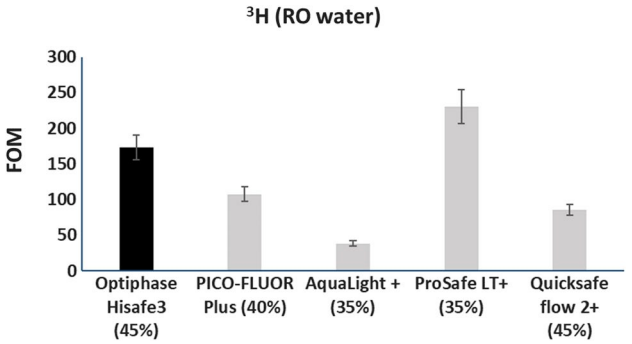

Fig. 9 Overview of FOM performance for ^{3}H in RO water using four cocktails (grey bars) and compared with routinely used OptiPhase Hisafe3 (black bar). Uncertainty is given as expanded uncertainty ($k=2$). For every cocktail the loading capacity is mentioned in the figure

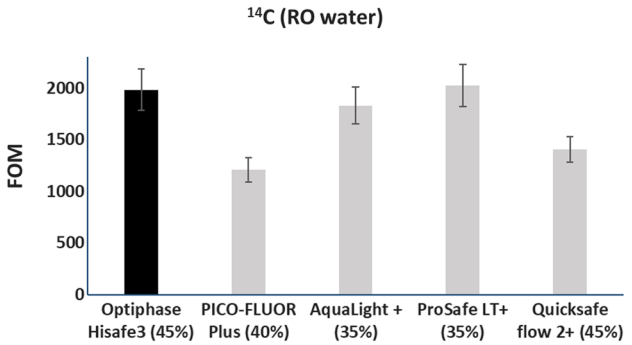

Fig. 10 Overview of FOM performance for ^{14}C in RO water using four cocktails (grey bars) and compared with routinely used OptiPhase Hisafe3 (black bar). Uncertainty is given as expanded uncertainty ($k=2$). For every cocktail the loading capacity is mentioned in the figure

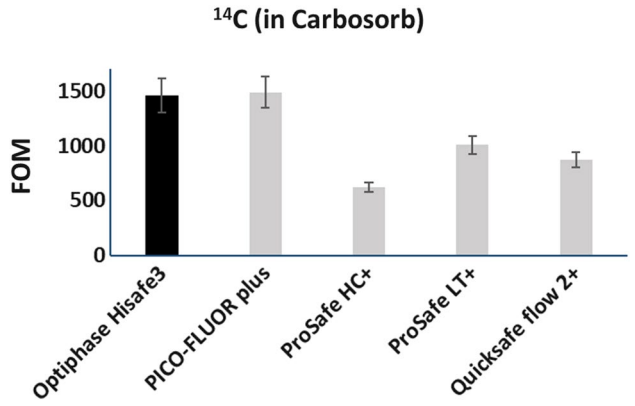

Fig. 11 Overview of FOM performance for ^{14}C trapped in Carbosorb E using four cocktails (grey bars) and compared with routinely used OptiPhase Hisafe3 (black bar). Uncertainty is given as expanded uncertainty ($k=2$)

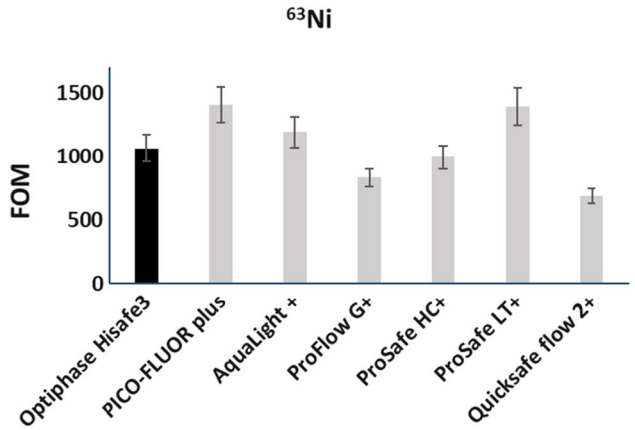

Fig. 12 Overview of FOM performance for ^{63}Ni using six cocktails (grey bars) and compared with routinely used OptiPhase Hisafe3 (black bar). Uncertainty is given as expanded uncertainty ($k=2$)

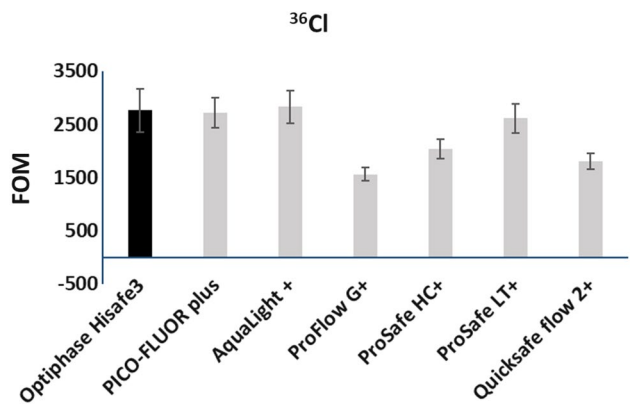

Fig. 13 Overview of FOM performance for ^{36}Cl using six cocktails (grey bars) and compared with routinely used OptiPhase Hisafe3 (black bar). Uncertainty is given as expanded uncertainty ($k=2$)

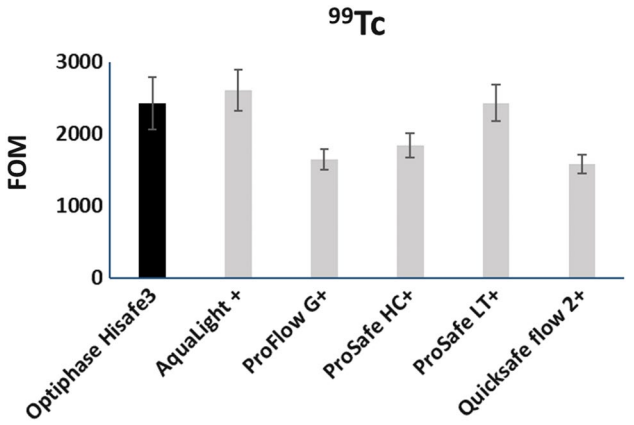

Fig. 14 Overview of FOM performance for ^{99}Tc using five cocktails (grey bars) and compared with routinely used OptiPhase HiSafe3 (black bar). Uncertainty is given as expanded unceratinty ($k=2$)

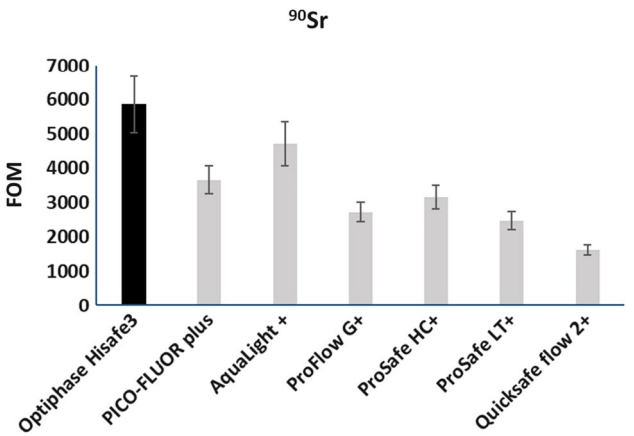

Fig. 15 Overview of FOM performance for ^{90}Sr using five cocktails (grey bars) and compared with routinely used OptiPhase HiSafe3 (black bar). Uncertainty is given as expanded unceratinty ($k=2$)

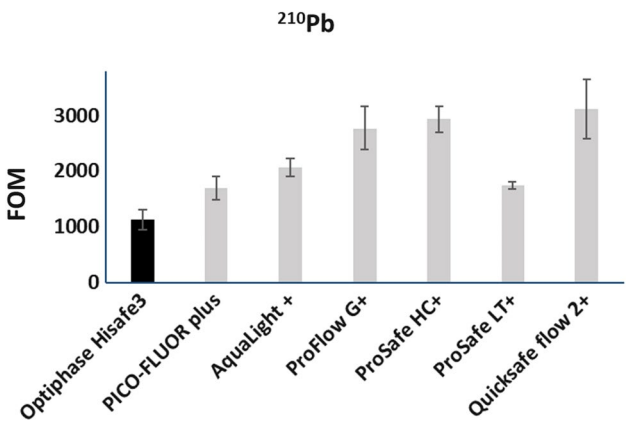

Fig. 16 Overview of FOM performance for ^{210}Pb using six cocktails (grey bars) and compared with routinely used OptiPhase HiSafe3 (black bar). Uncertainty is given as expanded unceratinty ($k=2$)

legislation is becoming more and more restrictive regarding the chemicals to be allowed for use in the production of the liquid scintillation cocktails, maybe another alternative can be considered for this type of measurements, such as plastic scintillation microspheres (PSm) or plastic scintillations resins (PS-resin).

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