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## Ionic liquids as solvents for liquid scintillation technology. Čerenkov counting with 1-Butyl-3-Methylimidazolium Chloride

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## HIGHLIGHTS

- Čerenkov luminescence was detected when <sup>18</sup>F was dissolved in 1-Butyl-3-Methylimidazolium Chloride (BmimCl) ionic liquid.
- The presence of another type of radiation that could eventually cause coincidences in the photodetectors was safely discarded.
- Čerenkov luminescence serves to determine the activity of a <sup>18</sup>F solution by means of TDCR–Čerenkov technique.
- Some advantages of the use of BmimCl as solvent for Čerenkov counting were listed.

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## ABSTRACT

We report the detection of the Čerenkov luminescence after the incorporation of a few droplets of a physiological solution of 2-deoxy-2-(<sup>18</sup>F)fluorine-D-glucose into the ionic liquid 1-Butyl-3-Methylimidazolium Chloride (BmimCl). The phenomenon is attributed to the  $\beta^+$  particles having energy above the threshold energy value for the Čerenkov radiation in this medium. The presence of another type of radiation that could eventually cause coincidences in the photodetectors was safely discarded. We show that this property serves to determine the activity of a <sup>18</sup>F solution by means of the novel TDCR–Čerenkov technique. The results were compared with those obtained from the classic TDCR scintillation method using a commercial scintillation cocktail. The activity values obtained from both methods were found to be virtually identical within the experimental uncertainties. The fact that high energy  $\beta$  particles in BmimCl generates Čerenkov photons makes this ionic liquid a promising compound for future research in detection and quantification of ionizing radiation, and it provides a potential alternative for applications in nuclear technology.

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## 1. Introduction

The Čerenkov radiation, i.e. the light produced as a charged particle passes through a dielectric medium at a velocity greater than the speed of light in the same medium, has been studied in detail for more than 80 years (Čerenkov, 1937). At the beginning, the analysis of the effects of Čerenkov radiation was restricted to the field of high energetic physics. Only in the 60's, the phenomenon started to be used in liquid scintillation technology, a methodology that involves the detection of the emitted photons coming from a liquid medium, in which a radioactive analyte has been previously dissolved (Rengan, 1983).

Several works in literature have focused attention on how to improve counting efficiency in liquid Čerenkov counters. The use of high refractive index solvents, as a means to decrease the Čerenkov threshold energy (Ross, 1976), and the incorporation of wavelength shifters (Wiebe et al., 1978), are two of the most commonly cited strategies. Although an increment in the refractive index of the solvent does not appear to be significant for activity standardization of high-energy emitters, it becomes relevant for radionuclides emitting  $\beta$  particles with energies comparable to the Čerenkov threshold value. For example, the use of a 95% glycerol/water solution – with a refractive index  $n=1.46$  – promotes a remarkable one hundred-fold increment in the detection efficiency of <sup>99</sup>Tc compared to those reported in water (Ross, 1969). Moreover, the coincidence counting efficiency of <sup>18</sup>F in chlorobutane ( $n=1.52$ ) increases by a factor of two, compared to those reported using dimethyl sulfoxide ( $n=1.47$ ) (Wiebe et al., 1978).

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A good solvent for Čerenkov measurements must fulfill four basic requirements – (a) it should dissolve the analyte of interest; (b) its refractive index should be as high as possible, in order to reduce the threshold energy for Čerenkov photons; (c) it should remain stable against radiation damage; and (d) it should exhibit negligible absorption in the region of emission of Čerenkov photons to prevent a decrease in the counting efficiency as a result of re-absorption phenomena.

In the last 10 years, a new family of compounds, commonly known as ionic liquids, has awakened a great deal of interest in the scientific community. Ionic liquids are room temperature molten salts composed of an organic cation and an inorganic/organic anion (Weingartner, 2008). They have multiple applications in areas such as catalysis (Lombardo et al., 2010), electrochemistry (Silvester et al., 2008), organic synthesis (Castro, 2005), solar cells (Wei et al., 2010), etc. More recently, ionic liquids have also been proposed to be used at different stages of the nuclear fuel cycle (Sun et al., 2011). The large diversity in the choice of the constitutive ions gives rise to the denomination of ILs as “tailored solvents”, capable to equally dissolve polar substances, like water, and natural polymers such as cellulose (Pinkert et al., 2009). Due to their scarce volatility, they have been also proposed as a “green” alternative to the most common volatile organic solvents.

At present, a large number of solutions and emulsions are commonly used as solvents in the Čerenkov detection experiments. The choice of a specific scintillation cocktail depends on the characteristics of the sample to be quantified. Aqueous solutions typically exhibit refractive indices comparable to the one of pure water; as such, their use for quantifying radionuclides emitting beta particles with energies near to the Čerenkov threshold in water is rather limited. On the other hand, the use of organic solvents usually present additional problems associated with their toxicity and flammability. Moreover, many emulsions used to suspend water soluble radionuclides, exhibit stability problems associated with the limiting amount of water that they support and the desired prevailing pH. We remark that the homogeneity of the mixture is a key element that guarantees the best counting results. In this context, ionic liquids emerge as new versatile solvents whose thermodynamic characteristics can normally be easily gaged by the nature of their molecular constitution. The strong ionic nature of their intermolecular interactions guarantees minimum evaporation, whereas flammability problems, normally associated to the more common organic phases, are practically absent.

Motivated by the search of new solvents for scintillation technology, in the present work we focus attention on the interaction between  $\beta$  radiation and a particular ionic liquid, 1-Butyl-3-Methylimidazolium Chloride (BmimCl), a viscous and colorless fluid with a relatively high refractive index  $n=1.54$ . We show that high energy  $\beta$  particles are able to induce the Čerenkov luminescence in this compound and exploit this property to determine the activity of a  $^{18}\text{F}$  solution by means of Čerenkov counting, by using the ionic liquid as solvent in the novel TDCR–Čerenkov method recently developed (Kossert, 2010) which does not require external samples for calibration.

## 2. Experimental

### 2.1. Chemical treatment and sample preparation

BmimCl was obtained by synthesis in our laboratories following standard procedures (Hallett and Welton, 2011). Freshly distilled 1-chlorobutane (Merck-Reagent for synthesis) was added dropwise to distilled 1-methylimidazole (Merck-Reagent for synthesis) (1.3:1 mol proportion) under vigorous stirring. The mixture was kept at 50 °C for 5 days under continuous stirring, in a  $\text{N}_2$  atmosphere, and

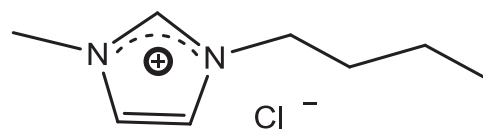


Chart 1. Chemical structure of 1-Butyl-3-Methylimidazolium Chloride ionic liquid.

subsequently purified by recrystallization from acetonitrile–ethyl acetate mixtures. A water content of  $\sim 5000$  ppm was determined by means of a Karl Fischer titration, using a Mettler Toledo DL32 coulometer. The  $^1\text{H-NMR}$  spectrum of the purified compound was (500 MHz,  $\text{D}_2\text{O}$ ,  $\delta$  in ppm) 8.69 (s, 1H), 7.46 (dd, 1H,  $J=2.0$  Hz,  $J=1.5$  Hz), 7.41 (dd, 1H,  $J=2.0$  Hz,  $J=1.5$  Hz), 4.18 (t, 2H,  $J=7.5$  Hz), 3.88 (s, 3H), 1.84 (q, 2H,  $J=7.5$  Hz), 1.32 (m, 2H,  $J=7.5$  Hz), and 0.91 (t, 2H,  $J=7.5$  Hz). The chemical structure of BmimCl is shown in Chart 1. Although, at ambient conditions, BmimCl is solid (melting point 60 °C) (Tang et al., 2008), it may remain in a metastable supercooled liquid state for long periods of time. This supercooled BmimCl phase was used to perform the measurements.

Samples of two different radionuclides  $^{18}\text{F}$  and  $^{14}\text{C}$  were prepared. In both cases, we used glass scintillation vials of 22 ml containing pure BmimCl. For the experiments with  $^{18}\text{F}$ , a few droplets of physiological solution of 2-deoxi-2( $^{18}\text{F}$ )fluorine-D-glucose were added to approximately 12 ml of ionic liquid. For the experiments with  $^{14}\text{C}$ , a few droplets of a glucose water solution (10<sup>-3</sup>% m/m of ( $^{14}\text{C}$ )glucose and 10<sup>-1</sup>% m/m of  $\text{CH}_2\text{O}$ ) were added to approximately the same amount of ionic liquid. The commercial scintillation cocktail UG-AB™ was used for reference measurements.

### 2.2. Scintillation measurements

All scintillation measurements were performed at room temperature ( $20 \pm 1$  °C) using a TDCR system described in detail in a previous paper (Arenillas and Cassette, 2006). The methodology was based on counting double and triple coincidences, using the MAC3 module (Bouchard and Cassette, 2000). At these conditions, the high viscosity of the ionic liquid ( $> 300$  cP, (Liu et al., 2008)) hinders a rapid and effective homogenization of the samples. For this purpose, usually a heating at  $\sim 60$  °C is required. Special care was taken to thermostatize the samples at room temperature after being heated, because samples improperly thermostatized could lead to spurious signals in the phototubes.

## 3. Theoretical approach on TDCR-based methods

### 3.1. TDCR method

The triple-to-double coincidence ratio (TDCR) method is based on the detection of scintillation photons using three photomultipliers in coincidence. The arrangement of detectors makes possible to obtain the experimental relationship between triple coincidences and the logical sum of double coincidences ( $T/D$ ). If one assumes that there are no significant differences between the photomultipliers, the theoretical ratio for the counting efficiencies of the triples ( $e_T$ ) and the logical sum of double coincidences ( $e_D$ ) is given by (Broda et al. 2007)

$$\frac{e_T}{e_D} = \frac{\int_0^E N(E') \left(1 - e^{-\frac{m(E')}{3}}\right)^3 dE'}{\int_0^E N(E') \left[3 \left(1 - e^{-\frac{m(E')}{3}}\right)^2 - 2 \left(1 - e^{-\frac{m(E')}{3}}\right)\right] dE'} \quad (1)$$

For a large number of disintegrations, the  $T/D$  counting ratio converges towards the  $e_T/e_D$  efficiency ratio. In the last expression,  $E$  is the energy,  $N(E')$  is the normalized  $\beta$  spectrum for the

radionuclide of interest, and  $m(E')$  is the mean number of photoelectrons detected. The latter quantity can be computed according to (Broda et al. 2007)

$$m(E) = \frac{\xi \varepsilon_q \mu \lambda}{hc} \int_0^E \frac{A}{1 + k_B \frac{dE'}{dx}} dE' \quad (2)$$

In Eq. 2,  $\xi$  is the detection efficiency of the photomultipliers,  $\varepsilon_q$  is the quantum efficiency of the photocathode,  $\mu$  is the spectral matching factor between the fluorescence spectrum and the spectral response of the photocathode,  $\lambda$  is the wavelength of the emitted photons,  $h$  is the Planck constant,  $c$  is the speed of light,  $A$  is the figure of merit of the number of emitted photons per energy unit,  $dE'/dx$  is the stopping power of the incident particle and  $k_B$  is the Birks parameter (Birks, 1960). For high energies, the term including the Birks parameter in the denominator can be normally neglected leading to the following expression:

$$m(E) = \frac{\xi \varepsilon_q \mu \lambda}{hc} \int_0^E A dE' = \frac{\xi \varepsilon_q \mu \lambda A}{hc} \int_0^E dE' = \frac{\xi \varepsilon_q \mu \lambda A E}{hc} = \nu' E \quad (3)$$

where  $\nu'$  is a free parameter. By settings  $\nu = \nu'/3$  and combining Eqs. (1) and (3) one obtains

$$\frac{T}{D} = \frac{\int_0^E N(E') (1 - e^{-\nu E'})^3 dE'}{\int_0^E N(E') [3(1 - e^{-\nu E'})^2 - 2(1 - e^{-\nu E'})^3] dE'} \quad (4)$$

Where the free parameter  $\nu$  can be varied in order to match the last expression with the experimental  $T/D$  ratio. The value of  $\nu$  that fulfills this requirement can be used to calculate  $e_D$  by simply replacing it in the denominator of Eq. (4). The activity of the sample can be computed dividing the experimental counting of the logical sum of double coincidences by  $e_D$  and by the probability of  $\beta$  decay of the nuclide (0.9686 (16)) in the case of  $^{18}\text{F}$ , (Be et al., 2011).

### 3.2. TDCR–Čerenkov method

The TDCR–Čerenkov method relies on the detection of Čerenkov photons using three photomultipliers in coincidence. In this case, the expression for  $T/D$  is (Kossert, 2010)

$$\frac{T}{D} = \frac{\int_0^E N(E') (1 - e^{-\nu \alpha_1 k(E')}) (1 - e^{-\nu \alpha_2 k(E')}) (1 - e^{-\nu \alpha_3 k(E')}) dE'}{\int_0^E N(E') \left\{ \frac{1}{2} \sum_{i=1}^3 \sum_{j=1}^3 (1 - \delta_{ij}) (1 - e^{-\nu \alpha_i k(E')}) (1 - e^{-\nu \alpha_j k(E')}) \right.} \\ \left. - 2(1 - e^{-\nu \alpha_1 k(E')}) (1 - e^{-\nu \alpha_2 k(E')}) (1 - e^{-\nu \alpha_3 k(E')}) \right\} dE'} \quad (5)$$

where the anisotropic nature of the Čerenkov radiation is described by a single parameter  $\alpha$ , which in turn, determines the coefficients

$$\alpha_1 = \alpha, \quad \alpha_2 = \frac{3}{2}(1 - \alpha)\alpha, \quad \alpha_3 = 1 - \frac{3}{2}(1 - \alpha)\alpha - \alpha \quad (6)$$

in the previous equation.

The value of  $\alpha$  can be calculated from the following semi-empirical formula:

$$\alpha = \chi \left( \frac{1}{2} + \frac{1}{6n \sqrt{1 - \frac{1}{(E/E_0 + 1)^2}}} \right) \quad (7)$$

where  $E$  is the energy in MeV,  $n$  is the refractive index,  $E_0$  is the electron rest energy (0.511 MeV) and  $\chi$  is an adjustable parameter (Kossert, 2010).

$k(E)$ , i.e. the number of Čerenkov photons as a function of energy, can be computed as (Grau Malonda and Grau Carles, 1998)

$$k(E) = \int_{E_{th}}^E 2\pi \alpha_{FS} \left( \frac{1}{\lambda_1} - \frac{1}{\lambda_2} \right) \left( 1 - \frac{1}{n^2 \left( 1 - \left( \frac{E_0}{E + E_0} \right)^2 \right)} \right) \frac{0.11 \times 22.4 \times E}{\rho \sqrt{1 + 22.4 \times E^2}} dE \quad (8)$$

In the previous expression,  $\lambda_1$  and  $\lambda_2$  indicates the detection wavelength interval in cm,  $\alpha_{FS}$  is the fine-structure constant and  $\rho$  is the density in  $\text{g cm}^{-3}$ . The integration is taken between the maximum energy of the spectrum and the threshold energy for Čerenkov radiation  $E_{th}$ , which can be obtained from

$$E_{th} = E_0 \left( \frac{1}{\sqrt{1 - \frac{1}{n^2}}} - 1 \right) \quad (9)$$

The free parameter  $\nu$  in Eq. (5) can be varied in order to match the expression with the experimental  $T/D$  ratio. The resulting value of  $\nu$  can be used to calculate  $e_D$  and the activity of the sample, in a similar fashion to those previously described for the TDCR method.

## 4. Results and discussion

### 4.1. Photon-discernment test

Fig. 1 shows the rate of the logical sum of double coincidences obtained before (background) and after the addition of few droplets of physiological solution of 2-deoxi-2- $^{18}\text{F}$ fluorine-D-glucose to BmimCl. It is observed that the number of counts with  $^{18}\text{F}$  is significantly higher than the corresponding background. The increment in the coincidence signal can be ascribed to three possible causes (a) the emission of fluorescence photons by BmimCl; (b) the induction of Čerenkov photons by high energy  $\beta^+$  particles as they pass through the ionic medium, and (c) the generation of annihilation photons having high escape probability, which eventually could interact directly with the photodetectors or with the glass of the photomultipliers via Compton scattering, leading coincidence counts.

To determine which one of the three possible effects is the responsible for the observed signal, we first performed experiments interposing a black cardboard between the samples and the detectors. In these experiments, only a background signal was recovered, ruling out the third possibility, since annihilation photons should pass smoothly through the cardboard.

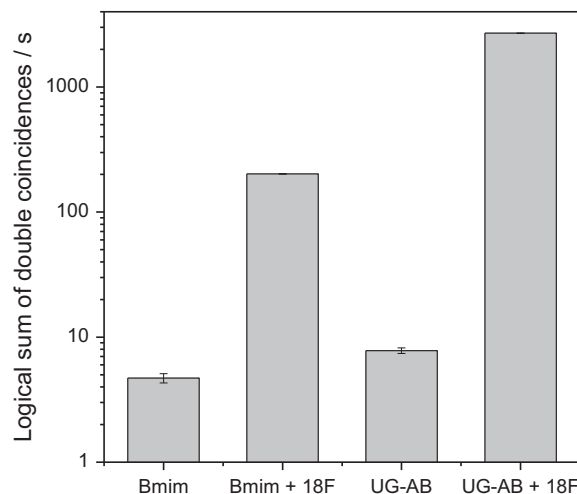


Fig. 1. Logical sum of double coincidences per second, for UB-AB™ and pure BmimCl with and without the addition of  $^{18}\text{F}$ .

In Fig. 1 it is also observed that, for the same dose of radiation, the number of coincidence events obtained with the commercial scintillation cocktail ultima gold AB™ is approximately 10 times higher than those obtained with the BmimCl. This result would suggest that the coincidence counts observed in the case of the ionic liquid were generated by photons of different natures.

In the case of the commercial cocktail, it is clear that luminescence photons are created within the solution by scintillation events. However, in the case of BmimCl, the origin of the photons needs to be clarified.

Usually, liquid scintillation cocktails comprise aromatic solvents and primary and secondary organic scintillators, whose function is to maximize the emitted light. These substances normally exhibit high luminescence quantum yields, in order to ensure a good conversion of the excitation energy into emitted light. Note that this does not seem to be the case of BmimCl, for which a very low luminescence quantum yield of about 0.02 has been reported by Samanta et al. (Mandal et al., 2005). This feature makes the emission of large amounts of light by the ionic liquid – as a result of their excitation with the ionizing radiation – unlikely. On the other hand, the nuclide  $^{18}\text{F}$  radiates  $\beta^+$  particles with maximum energy of 630 keV. These positrons have enough energy to generate Čerenkov radiation, since the  $E_{th}$  in BmimCl – calculated from Eq. (9) for  $n=1.54$  – is about 164 keV.

In order to confirm the hypothesis of Čerenkov photons detection from the ionic liquid, we performed measurements using  $^{14}\text{C}$ , a pure  $\beta^-$  emitter whose energy is not enough to produce Čerenkov radiation in this medium. Fig. 2 shows the results obtained after the addition of  $^{14}\text{C}$  to BmimCl and Ultima Gold AB™. No differences are observed between the sample and the background in the case of BmimCl whereas, of course, a large number of double coincidences above the background are detected for the commercial cocktail.

The previous analysis leads us to conclude that BmimCl does not generate fluorescence photons under the influence of  $\beta$  particles, and would confirm that those generated by the positrons from  $^{18}\text{F}$  are Čerenkov photons exclusively.

#### 4.2. Activity determination

We exploited the property of BmimCl to generate Čerenkov photons to measure the activity of a solution of  $^{18}\text{F}$  by means of the TDCR–Čerenkov technique, using the ionic liquid as solvent. The standardization measurements were made on  $^{18}\text{F}$  solutions with the purpose to show that activities of analyte solutions with low

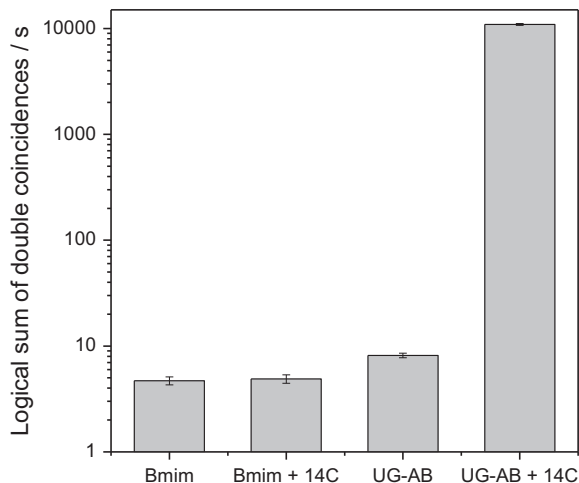


Fig. 2. Logical sum of double coincidences per second, for UB-AB™ and pure BmimCl with and without the addition of  $^{14}\text{C}$ .

Table 1

Comparison between the activities of  $^{18}\text{F}$  solution calculated using TDCR and TDCR–Čerenkov methods.

Method	TDCR	TDCR–Čerenkov
Medium	UG-AB™	BmimCl
Mass of analyte solution/mg	40.88 ± 0.04	42.94 ± 0.04
Logical sum of double coincidences/s	2686.4 ± 2.0	196.6 ± 1.4
TDCR	0.9959 ± 0.0015	0.1253 ± 0.0019
$e_D$	0.9992 ± 0.0003	0.0669 ± 0.0014
$\beta^+$ transition probability of $^{18}\text{F}$	0.9686 ± 0.0016	0.9686 ± 0.0016
Decay uncertainty (% error)	0.64	0.65
Calculated activity/Bq mg	67.70 ± 0.45	68.89 ± 1.62

intrinsic  $e_D$  can be assessed with reasonable uncertainty using the ionic liquid. Of course, standardization of solutions of higher energy emitters can also be achieved, resulting in greater efficiency counting and, consequently, in a reduction in the activity uncertainties.

We used the commercial scintillation cocktail UG-AB™ to obtain the activity of the solution via the TDCR scintillation method and compared it with those obtained from the TDCR–Čerenkov method. The entries of Table 1 summarize the experimental values and their uncertainties. The activities are presented with their corresponding combined uncertainties. The computation of  $k(E)$  for the Čerenkov measurements was performed by settings  $\lambda_1=330$  nm,  $\lambda_2=450$  nm,  $\rho=1.07$  g cm $^{-3}$  and  $n=1.54$ . Accurate values for the lower and upper limits for the wavelength interval and density are not required, since they appear as a factor of  $v$ . The  $N(E)$  spectrum of  $^{18}\text{F}$  used for the calculations was obtained from literature (Jan et al., 2005).

Assuming  $\chi=0.655$  for the parameter of the anisotropic coefficient  $\alpha$  (Kossert, 2010), one can observe that both activity values are virtually identical within the experimental uncertainty. As such, despite the relatively high combined uncertainty of the TDCR–Čerenkov result compared with those obtained for the TDCR scintillation method, BmimCl looks suitable to determinate activities of higher  $\beta$  emitters via Čerenkov counting.

Note that the relatively high refractive index of the ionic liquid represents an advantageous property for Čerenkov measurements. For example, in the case of  $^{18}\text{F}$ , the high refractive index of BmimCl makes it possible to obtain  $e_D$  values of the order of  $\sim 7\%$ , which represents a two-fold increment compared to the  $\sim 2\%$  obtained in 1 M aqueous solution of HCl ( $n=1.34$ ) (data not shown).

During and after the experiments, neither chemical degradation nor changes in the optical properties of the ionic liquid was observed. This feature makes the eventual reuse of the ionic liquid possible for new counting experiments in the case of short-lived nucleotides, provided all the remaining water is carefully removed to avoid changes in the refractive index. The removal of water from the ionic liquid can be achieved by simply using a vacuum oven, and represents a clear advantage over the volatile organic solvents.

## 5. Conclusions

The results presented in this paper reveal that high energy  $\beta$  radiation is able to induce Čerenkov luminescence in BmimCl. The Compton scattering of annihilation photons with the glass of the photomultiplier was ruled out by undertaking experiments in which the scintillation vial was screened with a black surrounding. Moreover, the absence of fluorescence photons was confirmed based on experimental results obtained with  $^{14}\text{C}$ .

We determined the activity of a diluted solution of  $^{18}\text{F}$  in BmimCl by means of the TDCR–Čerenkov technique. The results are in good agreement with the determination performed by using the classical TDCR technique, with a commercial scintillation cocktail.

Compared with other compounds, the use of BmimCl as a solvent for Čerenkov counting presents several advantages. For example, their high refractive index allows measuring  $e_D$  values of the order of 7% for  $^{18}\text{F}$ , a clear improvement compared to the use of aqueous solutions, in which this nuclide is barely detected.

Another clear attractive feature – this time against the use of volatile organic solvents – is the possibility of reusing the ionic liquid in new counting experiments in the case of short-lived nucleotides, by simply removing all water content in a vacuum oven.

The fact that BmimCl can generate Čerenkov photons combined with (a) its full miscibility with water, (b) its good chemical stability against ionizing radiation, and (c) its relatively high refractive index, opens interesting possibilities for the use of this compound to detect and quantify ionizing radiation.

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