

CHARACTERIZATION OF MODERN PLASTIC SCINTILLATORS CONTAINING LARGE STOKES-SHIFT LUMINOPHORES

by

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The development, preparation, and properties of new plastic scintillators, based on polyvinyl toluene matrix EJ-290 and only one luminophore with a large Stokes shift, are addressed. The primary luminophores tested were 2-(1-naphthyl)-5-phenyloxazole, 1-phenyl-3-mesityl-2-pyrazoline, and 2,5-bis(5-tert-butyl-benzoxazol-2-yl)thiophene. These were subsequently compared with the commercially used luminophores 2,5-diphenyloxazole and p-terphenyl in combination with wavelength shifters (secondary luminophores) 1,4-bis(2-methylstyryl)benzene and 1,4-bis(5-phenyl-2-oxazolyl)benzene made from the same polyvinyl toluene matrix in the same dimensions. For plastic scintillators produced in this way, their detection properties, such as Compton edge and detection efficiency, were characterized.

Key words: plastic scintillator, luminophore, wavelength shifter, polyvinyl toluene matrix, Compton edge

INTRODUCTION

Scintillation detectors are among the most widely used detectors for ionizing radiation spectrometry, especially low-resolution gamma spectrometry [1]. The applications of plastic scintillators (PS) in the detection and measurement of radionuclides are countless, because of the different properties of scintillation materials and read-out electronics *e. g.* photomultipliers (PMT), avalanche photodiodes (APVD), and silicon photomultipliers (SiPM). These properties determine their widespread application in nuclear physics for the detection of various types of radiation [2, 3], in radiochemistry [4], radiotherapy [5, 6], metallurgy [7, 8], and in the industry [9, 10], thickness gauges [11], level gauges [12], radiation portal monitors [13] *etc.* Highly sensitive scintillation detectors are commonly used for uranium and thorium detection [14]. The standard composition of PS contains *p*-terphenyl and 1,4-bis(5-phenyloxazol-2-yl)benzene (POPOP) in concentrations between 0.6-3 wt.% and 0.02-0.05 wt.%, respectively, dissolved in polystyrene or polyvinyl toluene matrix. Depending on the application, PS exhibits both advantages and disadvantages. On one hand, they are cheap, available in large sizes and morphologies, doped with various elements, and have a fast response. On the other hand, their light response is

average, the typical light yield is about 10 000 photons per MeV of deposited energy. Furthermore, they have low density and effective atomic number, therefore they are not effective for gamma spectroscopy and show poor resolution unless mathematically treated [2, 15].

The PS can be viewed as a solid solution of one or more organic scintillation luminophores in translucent plastic. The solid plastic medium plays a similar role as the liquid solvent in liquid scintillation [16]. Many types of PS are used today, mainly to measure less penetrating radiation, such as particle radiation, X-rays, and low-energy gamma rays. The PS are also used to measure neutrons through neutron-induced nuclear reactions in a plastic medium [17, 18]. There are two main types of PS constructions. First of all, these are integrated PS, which are constructed from one whole piece of plastic polymer containing the scintillator. They vary in size from microspheres (100 to 1000 μm in diameter) to large detectors (a meter or more) in diameter. In the second case, there are systems of PS optical fibers consisting of several thousand plastic scintillation fibers, each with a diameter of 25 μm to 60 μm , representing individual light guides connected with a diameter of only a few millimeters. Almost any shape of plastic scintillation polymer can be made to suit the requirements of any particular application. This versatility opens the door to a wide range of applications and is therefore one of the main advantages of plastic scintillators [16, 19].

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The PS are divided into binary, tertiary, or even higher-order scintillators. A binary scintillator consists of two components, namely a solvent (plastic or organic liquid) and a solute (organic luminophore or dopant). The solvent absorbs most of the energy scattered by the incident radiation. This absorbed energy is, among other things, converted into excitation energy of the solvent molecules and is then passed to the solute molecules mostly via non-radiative energy transfer. The transition from the excited state to the ground state of the solute molecules results in fluorescence or the emission of visible light. The addition of a wavelength shifter, or secondary solute, forms a tertiary system. In such a system, the energy transfer process is extended by a third component, called a secondary luminophore, *i. e.* the excited primary luminophore transfers its excitation energy both radiatively and non-radiatively to the secondary luminophore. The whole energy transfer theory is rather too complicated to be fully described here and can be found in [19]. Secondary luminophores are used primarily to provide fluorescence with a maximum emission wavelength that can be better matched to the sensitivity of the PMT. High concentrations of naphthalene (10-15%) are used in some plastic scintillators, which serves as a secondary solvent [16, 20, 21]. The role of the secondary solvent is to facilitate the transfer of energy from the primary solvent (plastic) to the primary luminophore. The secondary solvent effect is particularly important for energy transfer between the π -electron triplet energy state of solvent and solute and for applications such as pulse height discrimination that rely on the associated delayed scintillation component. However, in general, secondary solvents and secondary solutes degrade the time resolution of the scintillation system [16, 19, 22].

EXPERIMENTAL

Devices and equipment

To determine the positions of Compton edges of the new plastic scintillators, a light-tight chamber was used, which housed a 2" PMT type 9266 KB (ET Enterprises, Great Britain), MCA InSpektor 2000 with Genie 2000 Gamma Acquisition and Analyses version 3.4 evaluation software (both Mirion, USA).

A 2" PMT model digiBASE (Ortec, USA) controlled by the spectrometry software GammaVision for Windows Model A66-B32 version 6.07 (Ortec, USA) was used for the detection efficiency measurements.

Calculations for Compton edge determination were performed in MATLAB software version R2022b (MathWorks, USA).

Chemicals

The primary luminophores used were 2,5-diphenyloxazole (hereafter PPO, 99%, Sigma

Aldrich, USA), 1-phenyl-3-mesityl-2-pyrazoline (hereafter PYR, 98%, TCI Chemicals, USA), 2-(1-naphthyl)-5-phenyloxazole (hereinafter NPO, 99%, abcr GmbH, Germany), 2,5-bis(5-tert-butylbenzoxazol-2-yl)thiophene (hereinafter THIO, 99%, Alchimica, Czech Republic) and p-terphenyl (hereinafter referred to as pTP, 99%, Sigma Aldrich, USA).

The secondary luminophores selected were 1,4-bis(2-methylstyryl)benzene (hereafter MSB, 98%, Sigma Aldrich, USA) and 1,4-bis(5-phenyl-2-oxazolyl)benzene (hereafter POPOP, 99%, Sigma Aldrich, USA), acting as a wavelength shifter.

For the production of the plastic matrix, a modified kit EJ-290 (Eljen Technology, USA) without luminophore was used, containing partially polymerized vinyltoluene (hereafter Resin), lauroyl peroxide (97%) acting as a polymerization catalyst and vinyltoluene monomer (hereafter VT) for catalyst dissolution. Demineralized (DEMI) water was obtained using the DEMIWA 5 ROSA™ water purification system (Watek).

The PPO+POPOP plastic scintillator with a PPO concentration of 30 wt.% in styrene matrix (Tesla, Czechoslovakia) was used. Such high percentage plastic scintillators are mainly used in gamma/neutron spectrometry. Usually, the high luminophore concentration causes self-quenching resulting in lower light yield, thus are not suitable for standard measurement. To clarify this issue, it was measured together with other prepared scintillators.

The used radionuclides are listed in tab. 1.

Procedures

Production of scintillators

The following procedure was carried out to produce 50 g of the new plastic scintillator. The 75 ml of Resin was measured into a suitable glass beaker. Next, 0.375 g of lauroyl peroxide was mixed with 3.75 ml of VT to form a 10% solution of lauroyl peroxide in VT. This was poured into 75 ml of Resin with constant stirring until homogeneity was achieved. At this stage

Table 1. Used radionuclides

Radionuclide	Type	Supplier	Activity
²⁴¹ Am	ER X K – open source	Czech Metrology Institute	50.98 MBq
⁶⁰ Co	CO1HK – sealed emitter	Eckert and Ziegler	12 MBq
¹³⁷ Cs	Cs7.P03 – sealed emitter	Eckert and Ziegler	9.62 MBq
²⁴¹ Am	EG-3	Czech Metrology Institute	470.2 kBq
⁶⁰ Co	EG-3	Czech Metrology Institute	222.4 kBq
¹³⁷ Cs	EG-3	Czech Metrology Institute	316.0 kBq

desired luminophore(s) were dissolved in the mixture under vigorous stirring at a concentration corresponding to 5 gL⁻¹ and 0.05 gL⁻¹, respectively, which is the luminophore concentration commonly used in plastic scintillators. The solution was then transferred to a desiccator where the air pressure was gradually reduced up to 10 kPa to remove the bubbles. Subsequently, the solution was placed in a glycerine bath tempered to 47 °C, which was placed in a nitrogen atmosphere and left for 14 days. The plastic scintillator was then transferred to a hot air oven tempered to 80 °C for 8 hours. The glass mold was removed and the plastic scintillator was machined and polished into a cylinder shape with dimensions of 45 mm in diameter and 45 mm in height.

Light yield measurement

The light yield (LY) was determined based on the position of the Compton edges for the individual scintillators. The LY determination was performed in a light-tight chamber equipped with a 2" PMT optically coupled with the plastic scintillator. The measurement setup was: number of channels 1024, gain 1x, HV = 648 V, and the measurement time was set to 7200 seconds to collect a sufficient amount of counts. The LY was investigated for ²⁴¹Am, ⁶⁰Co, and ¹³⁷Cs (all EG-3), placed at a distance of 25 cm from the plastic scintillator. The Compton edge was then determined using MATLAB software for all individual measurements by using non-parametric estimation (local regression). The estimation was calculated only at one point, *x*, and only from the part of the data that lie in the interval [*x* - *h*, *x* + *h*], where *h* is called the smoothing parameter or the width of the smoothing window (bandwidth). The point *x* was moved along the area of interest. Furthermore, a regression line was used, specifically its value at the point *x*, which is then an estimate of the unknown regression function *f* at the point *x*. The first derivative can also be estimated from the regression line – the coefficient of *x* in the expression of the line [23].

Detection efficiency

A light-encapsulated detection system, consisting of the plastic scintillator and a 2" PMT with a digiBASE analyzer attached, was placed at a height of 1.2 m. The radionuclides ²⁴¹Am (ER X K), ⁶⁰Co (CO1HK), and ¹³⁷Cs (Cs7.P03) were placed at distances of 0.5, 1.0, 2.0, and 3.0 m in a horizontal line with the detection system at the same height (1.2 m). The measurement setup was: number of channels 1024, gain 1x, HV = 915 V, and measurement time 3600 s. The background of the scintillator was subtracted from the total sum of pulses and then the detection efficiency was calculated according to eq. (1)

$$\epsilon = \frac{I_{\text{smp}} - I_{\text{bcg}}}{A} \cdot 100 \text{ [%]} \quad (1)$$

where ϵ is the detection efficiency, I_{smp} – the sum of pulses with radionuclide, I_{bcg} – the sum of background pulses, and A – the activity [Bq] calculated to the date of measurement according to the certificate.

RESULTS AND DISCUSSION

Production of scintillators

Several factors had to be taken into account in the production of plastic scintillators. The formation of bubbles during the polymerization process itself proved to be a major problem, especially for the THIO scintillator. Since this phenomenon was observed only at this luminophore, it is believed that it may be due to the effect of the luminophore itself, since it was optical grade purity. The final curing of the plastic scintillator was changed from 80 °C to 70 °C after several experiments in which the plastic matrix was cracked or *fogged* and the tempering time was extended to 24 hours. These problematic factors were avoided in the resulting plastic scintillators, with only a slight fogging occurring in the case of the NPO scintillator.

Light yield

The position of the Compton edge of a given radionuclide was used to measure the LY of individual scintillators. The commercially used plastic scintillator pTP+POPOP was chosen as the standard. The LY of the other scintillators was normalized to its light yield values. It can be seen in tab. 2 that the lowest light yields were achieved for the plastic scintillator containing the PYR luminophore, which is less than half of the standard. The exception is ²⁴¹Am, but this may be due to an incorrectly chosen position of the Compton edge. The other possibility could be *noise* due to imperfect polymerization resulting in pulses generation originating from chemiluminescence. The second and third plastic scintillators showing the lowest light yield were NPO and THIO with light yields of 80 ± 4 % and 85 ± 2 % of the standard, respectively. For the NPO and PYR scintillators, both Compton edges could not be reliably determined for ⁶⁰Co. For this reason, only one average was determined for both energies of ⁶⁰Co. This may indicate their inferior

Table 2. Light yields of prepared plastic scintillators normalized to pTP+POPOP values

Luminophore	Normalised LY to pTP+POPOP [%]				Average LY [%]
	²⁴¹ Am	¹³⁷ Cs	⁶⁰ Co	⁶⁰ Co	
NPO	85	74		80	80 ± 4
PPO+POPOP	104	109	103	102	105 ± 3
PPO+MSB	111	108	110	112	110 ± 1
PYR	111	42		44	66 ± 32
THIO	89	83	84	84	85 ± 2

scintillation properties (resolution) in this application. The plastic scintillator containing the PPO+MSB luminophores achieved the best results with an average light yield of $110 \pm 1\%$ of the standard. Despite the very high PPO dopant (30%), no significant reduction in light yield due to self-absorption as with liquid scintillators was observed, as demonstrated by the average light yield PPO+POPOP of $105 \pm 3\%$ of the standard value.

Detection efficiency

Measurement of detection efficiency was performed for low-, mid-, and high-energy radionuclides. In the case of low energy ^{241}Am radionuclide was used. The detection efficiency of all measured PS achieved values that rather correspond to the detection efficiency of the standard except for PYR. It can be said that in terms of detection efficiency, the pTP+POPOP standard achieved the best results followed closely by PPO+MSB, THIO, and PPO+POPOP, as can be seen in fig. 1.

Middle and high energies represented by ^{137}Cs and ^{60}Co did not exhibit such large differences in detection efficiencies, figs. 2 and 3. As in the case of ^{241}Am the pTP+POPOP standard outperformed all other luminophores. For the ^{137}Cs the order in detection efficiency was as follows – PPO+MSB, PPO+POPOP, and THIO. Slightly worse results were then achieved by NPO and the last one was again PYR.

When dealing with ^{60}Co the differences in detection efficiencies of all prepared PS from standard pTP+POPOP were even smaller than in the case of ^{137}Cs . The results obtained when the pTP+POPOP standard was measured were closely followed by THIO and PPO+MSB. Not very significant differences were then achieved by PPO+POPOP and NPO and lastly by PYR.

The PYR performed the worst, which can be explained by the fact that the absorption spectrum of PYR does not fully match the emission spectrum of polystyrene, and energy transfer is worse. Moreover, the self-absorption effect of emitted radiation by the matrix is possible due to a smaller shift between absorption and emission spectra. The addition of a spectrum shifter could improve the LY, but this was not the goal of the paper, which dealt with the use and compar-

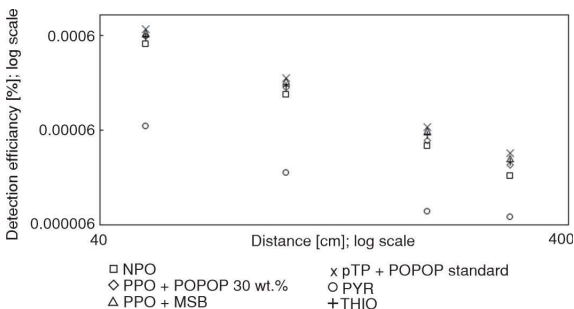


Figure 1. Dependence of detection efficiency on distance of radionuclide from PMT+plastic scintillator for ^{241}Am

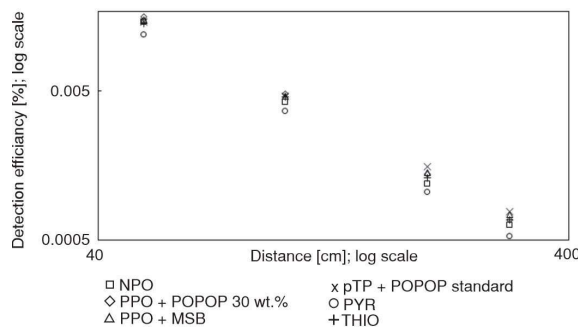


Figure 2. Dependence of detection efficiency on distance of radionuclide from PMT+plastic scintillator for ^{137}Cs

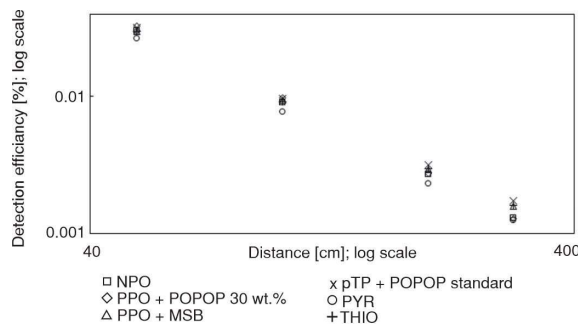


Figure 3. Dependence of detection efficiency on distance of radionuclide from PMT+plastic scintillator for ^{60}Co

ison of only one luminophore with a large Stokes shift. An interesting fact is that PYR excels as a primary luminophore in liquid scintillation cocktails [24, 25], but does not confirm its unique properties in plastic scintillators. This problem may be due to the interaction or the presence of a different solvent, which is the main component that differs when using PYR in plastic and liquid scintillators. It also shows very good results in terms of neutron/gamma discrimination properties in liquid scintillation cocktails, presenting even better properties than PPO [26, 27].

CONCLUSIONS

Any chemical compound containing π -electrons could be a potential scintillator or part of a scintillation system. Its usefulness as a scintillator then depends on many properties based on the intended use. These could be fluorescence decay mode and time, solubility, toxicity, temperature and chemical stability, ability to withstand the polymerization process, etc. Binary scintillation systems will probably never outperform multicomponent scintillation systems in plastics, as the shared radiation energy is self-absorbed during the scintillation process, thus reducing the scintillation efficiency.

Concentration quenching when comparing PPO+POPOP and PPO+MSB is not as significant as it was believed to be based on the literature and measurements of liquid scintillation cocktails. That is likely

due to the matrix effect, as both PVT and styrene behave differently from other aromatic solvents in terms of concentration quenching, *i. e.* the light yield increases with increasing concentration until the formation of a plateau which is consistent with the results obtained for these two plastic scintillators [27].

In terms of detection efficiency, it could be expected that for the low energies, represented by ^{241}Am , the NPO and PYR, scintillators would perform worst due to the absence of a wavelength shifter, but this was not confirmed for THIO, which performed comparable to the standard. The standard pTP+POPOP, PPO+MSB, and THIO performed the best overall. In particular, it should be noted that THIO achieved comparable results to the standard in all cases, even without the wavelength shifter.

According to the tested properties, overall PPO+MSB and PPO+POPOP can be evaluated as suitable theoretical substitutes for the pTP+POPOP system. As a subject of further research, it might be possible to verify the properties of a THIO luminophore with a wavelength shifter (MSB or POPOP), whereby theoretically good light yield results could also be achieved.

AUTHORS' CONTRIBUTIONS

The idea for this study was put forward by J. Janda. Manufacturing of plastic scintillators and data evaluation were carried out by J. Janda and E. Rajchl. The measurements were performed by E. Rajchl. Theoretical calculations were performed by J. Zelinka.

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**КАРАКТЕРИЗАЦИЈА МОДЕРНИХ ПЛАСТИЧНИХ СЦИНТИЛАТОРА
КОЈИ САДРЖЕ ЛУМИНОФОРЕ СА ВЕЛИКИМ СТОКСОВИМ ПОМАКОМ**

Обрађени су развој, припрема и својства нових пластичних сцинтилатора, заснованих на поливинил толуенској матрици EJ-290 и само једним луминофором са великим Стоксовим помаком. Примарни тестирани луминофори су 2-(1-нафтил)-5-фенилоказол, 1-фенил-3-мезитил-2-пиразолин и 2,5-бис(5-терц-бутил-бензоказол-2-ил)тиофен. Они су затим упоређени са комерцијално коришћеним луминофорима 2,5-дифенилоказолом и п-терфенилом у комбинацији са померањима таласне дужине (секундарним луминофорима) 1,4-бис(2-метилстирил)бензеном и 1,4-бис(5-фенил-2-оксазолил)бензеном направљеним од исте поливинил толуенске матрице у истим димензијама. За пластичне сцинтилаторе произведене на овај начин, окарактерисана су њихова својства детекције, као што су Комптонова ивица и ефикасност детекције.

Кључне речи: пластични сцинтилатор, луминофора, померањ таласне дужине, поливинил толуенска матрица, Комптонова ивица