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Matthieu Hamel, Anne-Marie Frelin-Labalme, Vesna Simic, Stéphane Normand. N-(2',5'-di-t-butylphenyl)-4-ethoxy-1,8-naphthalimide: A new fluorophore highly efficient for fast neutrons/gamma-rays discrimination in liquid media. Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment, 2009, 602 (2), pp.425-431. <10.1016/j.nima.2009.01.191>. <cea-05493433>

**HAL Id: cea-05493433**

**<https://cea.hal.science/cea-05493433v1>**

Submitted on 4 Feb 2026

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# N-(20,50-di-*t*-butylphenyl)-4-ethoxy-1,8-naphthalimide: A new fluorophore highly efficient for fast neutrons/gamma-rays discrimination in liquid media

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**abstract** Discrimination between fast neutrons and gamma rays has been successfully achieved using a bluefluorescing 4-substituted 1,8-naphthalimide in aromatic solvents. As previously stated in the literature, an important solvent effect was observed, but the substitution groups surrounding the core of the naphthalimide also appeared to be extremely crucial. Our system presents the advantages of being based on the use of a single highly soluble solute, exhibiting a large Stokes shift (to avoid self-absorption process) and presenting an emission spectrum fitting well to the photomultiplier (PM) sensitivity. More importantly, we observed that our scintillator was not affected by the concentration of oxygen dissolved in the medium. This observation has no equivalent in commercial liquid scintillators, which are known to be oxygen-sensitive. The discrimination efficiency of our system is described in terms of scintillation performances, figure of merit and the angle between neutrons and gamma lobes visualized in the bi-parametric discrimination spectrum.

Keywords: Organic scintillator, Fast neutron/gamma discrimination, 1,8-Naphthalimide

## 1. Introduction

NE213 (or BC-501A) and more generally liquid scintillators are by far the most studied detectors for fast neutron/gamma discrimination, due to their low cost and easy availability. However, the use of such liquid scintillators will probably in the future be more and more restricted especially in industrial applications because of their inherent toxicity and flammability. Ideally, plastic scintillators would represent the best alternative to replace them since they can be built at low cost in various geometries and sizes and are safe by nature. Nevertheless they are assumed to be ineffective for fast n/g discrimination. Indeed, to the best of our knowledge, only a single publication refers to an efficient pulse shape discrimination in a plastic mixture of *m*- and *p*-isopropylbiphenyl (already known for its advantageous contributions in liquid scintillation [1]), *p*-terphenyl and POPOP1 dispersed in poly(vinyltoluene) [2].

It is now well-established that pulse shape discrimination between gamma rays and fast neutrons is related with prompt and delayed fluorescence of fluorophores inside the scintillator. Indeed, the incident radiation can induce the excitation of the scintillator molecules from the ground state  $S_0$ , either to singlet states  $S_1$ ,  $S_2$ ,  $S_3$ , etc. or to triplet state  $T_1$ . The decay of the first type of state results in the emission of prompt fluorescence, whereas the second type of state annihilates following the equation  $T_1 + T_1 \rightarrow S_1 + S_0$ , generating delayed fluorescence [3].

The annihilation of the triplet states is directly correlated to the density of the  $T_1$  states, and thus to the ionization density produced by the incident radiation. Consequently, gamma rays (which mainly produce Compton electrons) generate a smaller proportion of delayed fluorescence than neutrons (which mainly produce recoil protons by elastic scattering on hydrogen atoms).

The Neutromania project is a French research collective whose aim is to design new plastic scintillators bearing fast neutrons/ gamma discrimination capabilities. In a preliminary study, we have incorporated different 4-substituted 1,8-naphthalimides (for an explanation about the chemical structure, see Fig. 1) into polymer matrices [4]. We have studied their light response at different concentrations (up to  $40 \text{ g L}^{-1}$ ) and shown that it had no effect on the n/ $\gamma$  discrimination properties, which remained ineffective in this configuration. Besides this result, a possible matrix effect had been pointed out. Therefore, we decided to study the interactions between radiations and the scintillator in liquid systems in order to improve in the future the design of discriminating plastic scintillators.

This paper presents our results concerning the use of fluorescent 1,8-naphthalimides dissolved in liquid scintillators for n/ $\gamma$  discrimination [5]. It shows the complex phenomena related to n/ $\gamma$  discrimination, occurring from the matrix, the design of the fluorophore and the nature of the gas accumulated inside the scintillator.

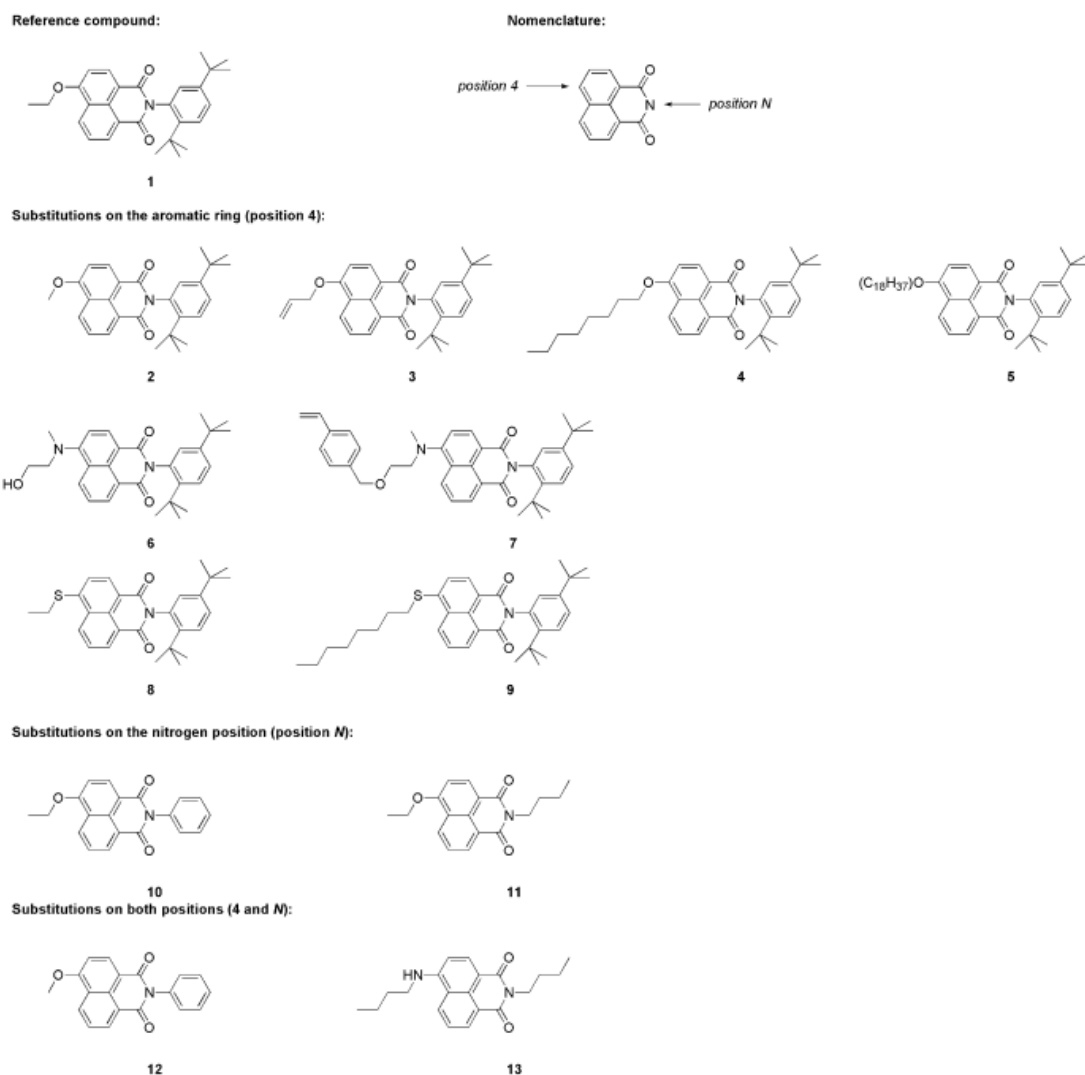


Fig. 1. Different 4- and/or N-substituted 1,8-naphthalimides 1–13 engaged in  $n/\gamma$  discrimination.

## 2. Experimental

### 2.1. Chemistry

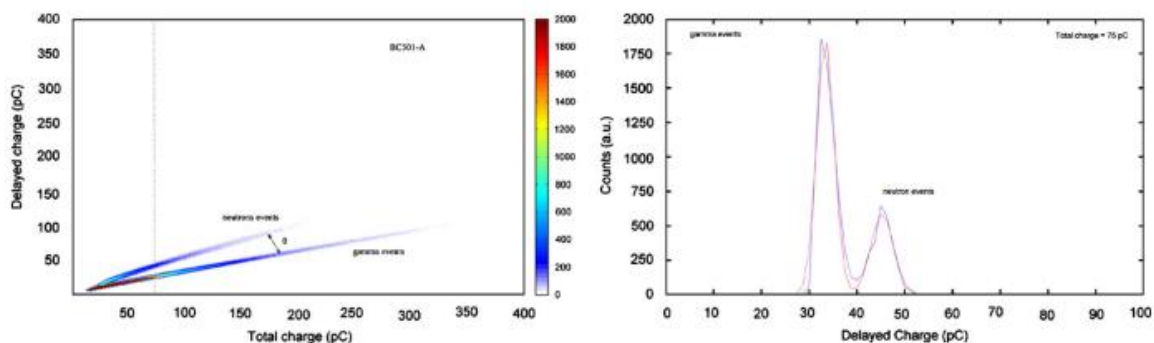
N-(20,50-di-t-butylphenyl)-4-ethoxy-1,8-naphthalimide 1 (molecular weight 429.55 gmol<sup>-1</sup>, see Fig. 1), considered as the fluorophore of reference in our work was prepared according to a published procedure [4]. Compounds 2–13 (also presented in Fig. 1) are 1,8-naphthalimides with similar structures to 1 and were synthesized so as to study the influence of the substituents located on the 1,8-naphthalimide core. 6 and 7 were prepared as described in the publication of Le Barny et al. [6]. The synthesis of 12 has already been described [7] and 10 was prepared accordingly to this method. 13 was isolated in high yield by following the method of Tyman et al. [8]. All other 1,8-naphthalimides (2–5, 8, 9 and 11) have never been described and were obtained according to our synthetic procedure [4] by using the appropriate reagents.

All solvents were of spectroscopic grade or were dried and distilled prior to use. All solutions were fully saturated with argon by performing gas bubbling for several minutes into the scintillator prior to physical experiments.

### 2.2. Physics

NE213 (Nuclear Enterprises) and BC-501A were obtained from Saint-Gobain Crystals. Ultima Gold™ AB was purchased from Perkin-Elmers.

Absorption and emission spectra were, respectively, recorded with a Perkin-Elmer Lambda 19 spectrometer and a Hitachi F-4500 spectrofluorimeter at room temperature. Fluorescence spectra were recorded with an excitation wavelength corresponding to the absorption maximum of the studied species.



**Fig. 2.** Left: Bi-parameter spectrum of delayed charge vs. total charge, characterizing neutron and gamma events. The  $\theta$  angle between neutron and gamma events is represented. Right: Vertical cut of the bi-parameter spectrum on the axis which is displayed on the right side figure and which corresponds to a 75 pC total charge.

The 1,8-naphthalimides solutions have been studied in terms of scintillation yield and time response with a 48 kBq  $^{60}\text{Co}$  source. Thus,  $10\text{ g L}^{-1}$  solutions were enclosed in a  $38 \times 10 \times 10\text{ mm}^3$  tank whose five faces were white painted ( $\text{TiO}_2$ ) to maximize the light collection in the direction of the photomultiplier. The remaining transparent face was optically coupled to a Photonis XP2020 photomultiplier tube with optical grease.

Neutrons/gamma discrimination experiments were performed in the same tank with a 74 MBq  $^{252}\text{Cf}$  source (with the same experimental set up) thanks to the charge comparison method [9]. In this method, the total charge and the tail charge (measured over the half of the signal fall) were simultaneously recorded and computed in a bi-parameter spectrum, which was normalized regarded to the total number of events (see Fig. 2).

The discrimination ability of the different scintillators was quantitatively evaluated by two means: the figure of merit and the angle formed between gamma and neutron events (represented in Fig. 2). It must be noticed that the delayed gate is not optimal for each individual scintillator but the comparison of discrimination properties in a constant experimental configuration allows to distinguish the most promising scintillator (with regards to the discrimination).

The figure of merit (FOM) is calculated for each vertical cut of the spectrum (as the one represented in Fig. 2) thanks to the following relation [10]:

$$\text{FOM} = \frac{m_n - m_\gamma}{\sigma_\gamma + \sigma_n}$$

where  $m_n$  and  $m_\gamma$  are, respectively, the neutron and gamma peaks centroids, and  $\sigma_n$  and  $\sigma_\gamma$  are the corresponding standard deviations.

Thus a FOM value is obtained for each total charge presenting separated neutron and gamma “lobes” and an adequate statistic.

The  $\theta$  angle is defined as the geometrical angle between the central axis of the neutron events lobe and the gamma events lobe.

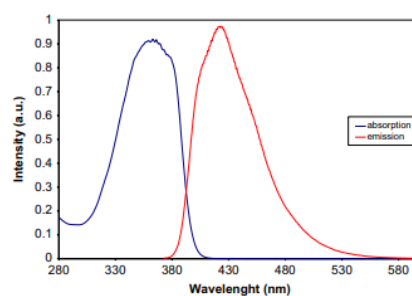
### 3. Results and discussion

The results presented in this section highlight the effects of the main three components of liquid scintillators: the solvent, the fluorophore and the dissolved gas. The influence of each factor will be discussed independently.

#### 3.1. Solvent effect

The absorption spectrum of N-(2',5'-di-*t*-butylphenyl)-4-ethoxy-1,8-naphthalimide 1 presents a maximum at 363 nm corresponding to a S0-S1 ( $\pi$ - $\pi^*$ ) transition. The molar extinction coefficient  $\epsilon$  is equal to  $33\,600\text{ L mol}^{-1}\text{ cm}^{-1}$ . The emission maximum is located around 420 nm (Stokes shift  $\Delta\nu = 3494\text{ cm}^{-1}$ , Fig. 3), which is therefore ideally suited for the Photonis XP2020 PM.

Among the 20 or so organic solvents tested (from non-polar heptane to highly polar DMSO or ionic liquid BmimPF6), good scintillation properties were usually observed, but discrimination was exclusively achieved in aromatic solvents. The most relevant results are resumed in Table 1 and the best result of n/g discrimination can be observed in Fig. 4.



**Fig. 3.** Absorption and emission spectra of compound 1.

The relative light outputs of our scintillators were slightly lower than commercial liquid scintillators (Table 1), with the highest value of 64% (compared to a mixture of *p*-terphenyl  $4\text{ g L}^{-1}$  + POPOP  $0.1\text{ g L}^{-1}$  in toluene) obtained for the naphthalimide/*m*-xylene solution. This value is the same for BC-501A. Table 1 shows also a comparison of rise and fall times for the different scintillators. For both values, the time constants were slightly longer for 1,8-naphthalimide solutions, from 20% up to twice for rise and fall time, respectively. With these results, one should consider that our naphthalimide-containing scintillators are therefore less efficient than commercially available solutions. However, the slowest rise and fall times obtained for our naphthalimide in styrene are very close to those of NE213, but the former does not discriminate fast neutrons from gamma. Hence, a low value of these parameters is not a crucial parameter for determining the discrimination possibilities of a scintillator.

**Table 1**  
Scintillation and discrimination properties of *N*-(2',5'-di-*t*-butylphenyl)-4-ethoxy-1,8-naphthalimide **1** in aromatic solvents and comparison with efficient scintillators.

Liquid scintillator <sup>a</sup>	Light output (%) <sup>b</sup>	Rise time 20/80 (ns)	Fall time 80/20 (ns)	Figure of merit <sup>c</sup>	$\theta$ <sup>d</sup>
<i>p</i> -Terphenyl/POPOP (4/0.1 g L <sup>-1</sup> ) in toluene	100	2.46	7.29	0.6–1.9 (40–280)	5.0
NE213	85	2.77	9.05	0.8–1.8 (35–200)	7.8
Ultima Gold AB	77	2.55	9.90	0.7–1.3 (40–135)	7.0
BC-501A	64	2.73	8.61	1.0–2.4 (35–250)	8.3
1,8-Naphthalimide/ <i>m</i> -xylene	64	2.93	14.86	0.6–1.6 (40–260)	5.0
1,8-Naphthalimide/toluene	59	2.96	14.92	0.6–1.4 (35–250)	5.1
1,8-Naphthalimide/mesitylene	56	3.02	13.82	0.4–1.2 (45–200)	3.5
1,8-Naphthalimide/benzene	51	3.06	15.24	0.3–1.4 (50–200)	7.0
1,8-Naphthalimide/ <i>p</i> -xylene	50	3.01	15.25	0.6–1.4 (40–210)	4.8
1,8-Naphthalimide/ <i>p</i> -isopropylbiphenyl	33	3.45	13.16	0.5–0.9 (45–200)	3.1
1,8-Naphthalimide/styrene	8	2.60	10.07	0	0

<sup>a</sup> 10 g L<sup>-1</sup> of naphthalimide **1** in the corresponding solvent.

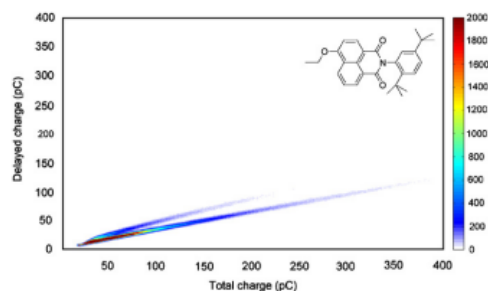
<sup>b</sup> Normalized to 100 for the scintillator reference.

<sup>c</sup> In parentheses, range of the total charge in pC considered for the figure of merit calculation.

<sup>d</sup> Angle in degree formed between neutrons and gamma lobes.

Two other parameters are much more important in terms of discrimination: the figure of merit and the angle  $\gamma$  formed between the neutrons and the gamma lobes. It must be noticed that the FOM can be calculated only when the gamma events and the neutron events are correctly separated and when statistics are sufficient. Consequently, the range of total charge where the FOM can be correctly calculated is always specified in parentheses. As shown in Table 1, both these factors for 1,8-naphthalimide **1** solubilized in toluene are quite similar to the NE213 values. In benzene, the angle  $\theta$  reaches the value of the best commercial scintillators.

All the solvents permitting an efficient discrimination present a fluorescence emission at 300–330 nm, which fits well to the absorption maximum of the 1,8-naphthalimide **1**. In the case of styrene, its fluorescence is not only slightly blue-shifted to ca. 285–300 nm, but also known to be a weak fluorophore [3]. This area is not really appropriate for an efficient solvent/solute energy transfer, and as a consequence a weak light output is observed.



**Fig. 4.**  $n/\gamma$  Discrimination of *N*-(2',5'-di-*t*-butylphenyl)-4-ethoxy-1,8-naphthalimide **1** in toluene (10 g L<sup>-1</sup>) saturated in argon.

**Table 2**  
 $n/\gamma$  Discrimination results obtained with differently substituted naphthalimides 1–13.

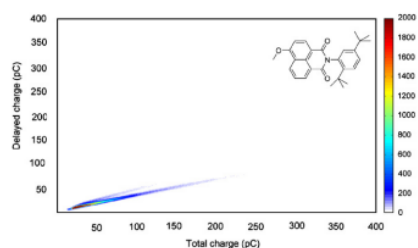
Compound <sup>a</sup>	Position of the variation	$\lambda_{em}$ (nm) <sup>b</sup>	Figure of merit	$\theta$
<b>1</b> <sup>c</sup>	Reference compound	420	0.6–1.4 (35–250)	5.1
<b>2</b>	4	420	0.4–1.1 (35–150)	5.5
<b>3</b> <sup>d</sup>	4	420	Weak	Weak
<b>4</b> <sup>d</sup>	4	420	Weak	Weak
<b>5</b>	4	420	No discrimination	
<b>6</b>	4	500	No discrimination	
<b>7</b>	4	500	No discrimination	
<b>8</b>	4	450	0.3–1.2 (40–180)	4.7
<b>9</b>	4	450	0.4–1.0 (64–150)	6.7
<b>10</b>	N	420	No discrimination	
<b>11</b>	N	420	0.2–0.8 (40–180)	3.6
<b>12</b>	4 and N	420	No discrimination	
<b>13</b>	4 and N	500	No discrimination	

<sup>a</sup> The 1,8-naphthalimide of interest was made soluble at the concentration of 10 g L<sup>-1</sup> in toluene and the solution was saturated with argon during several minutes.

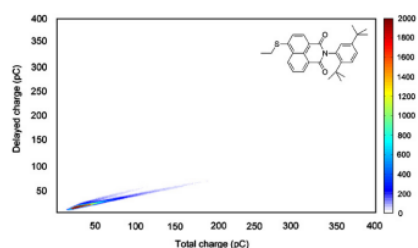
<sup>b</sup> Approximate data, so as to point out the three different families of 1,8-naphthalimides. The maximum of the 1,8-naphthalimide emission is located at  $\pm 5$  nm of this approximate value.

<sup>c</sup> Discrimination results obtained with compound **1** have been repeated in the table for comparison.

<sup>d</sup> Although the figure of merit and the angle  $\theta$  were visible on discrimination figures, they were too low for a precise measurement.



**Fig. 5.**  $n/\gamma$  Discrimination of *N*-(2',5'-di-*t*-butylphenyl)-4-methoxy-1,8-naphthalimide **2**.



**Fig. 6.**  $n/\gamma$  Discrimination of *N*-(2',5'-di-*t*-butylphenyl)-4-ethylsulfanyl-1,8-naphthalimide **8**.

The study of the discrimination performances as a function of the naphthalimide concentration revealed that our system was efficient from 3 g L<sup>-1</sup> until saturation (ca. 40 g L<sup>-1</sup> in xylene at 20 °C). Thus, the figure of merit and the  $\gamma$  angle increase until reaching a plateau at the concentration ca. 20 g L<sup>-1</sup>.

It is noteworthy that doping a scintillator composed of **1** in toluene at the concentration 10 g L<sup>-1</sup> with *p*-terphenyl as primary fluorophore did not afford a better discrimination, in terms of figure of merit or  $\gamma$  angle. This indicates that the simple combination of **1** in the appropriate solvent is able to discriminate, contrary to commercial liquid scintillators. Indeed, they are usually made of complex blends of fluorophores, which are all necessary for the discrimination efficiency of the scintillator.

This first part allowed us to determine the most favourable solvent conditions for the  $n/\gamma$  discrimination with **1** as fluorophore. These newly created scintillators exhibit nearly the same properties as commercial products in terms of light output and decay times. The following section is a discussion about the fluorophore itself, and how slight structural modifications on the core of the 1,8-naphthalimide lead to huge variations in discrimination.

### 3.2. Substitution effects inside the fluorophore

These first results seem to hint that the major effect on  $n/\gamma$  discrimination efficiency comes primarily from the solvent. With regard to the structure of the parent molecule *N*-(20,50-di-*t*-butylphenyl)-4-ethoxy-1,8-naphthalimide **1**, we investigated the electrodonating substituent effect located on the position 4 of the 1,8-naphthalimide (i.e. on the aromatic cycle) and the substituent effect carried by the nitrogen atom of the naphthalimide moiety (located at the N position).

All the  $n/\gamma$  discrimination results of liquid scintillators prepared from 10 g L<sup>-1</sup> toluenic solutions of 1,8-naphthalimides 1–13 (represented in Fig. 1) are resumed in Table 2. Some examples of discrimination results for molecules whose structure are close to that of **1** (differences located on the substitution of the naphthalimide core) are represented in Figs. 5–8.

As expected, the fluorescence can be modulated by the electrodonating substituent located on the position 4 of the 1,8-naphthalimide, whereas the moiety located on

the amine does not have any spectroscopic effect [11]. Thus, oxygen-derived naphthalimides fluoresce in the blue region (close to 420 nm), whereas sulphur-derived naphthalimides fluoresce around 450 nm and nitrogen-derived compounds fluoresce around 500 nm. This property can be very useful since 1,8-naphthalimides-based scintillators can provide different emission spectra without any wavelength shifters, especially for small scintillators where this shift is often incomplete. Regarding the substituent located at the N position, bulky groups such as di-*t*-butylphenyl units are preferred since they are expected to reduce self-quenching by preventing adjacent molecules from *p*-stacking. Moreover, this substituent induces also a huge diminution of the polarity of the molecule, thus allowing a better solubility in non-polar solvents such as toluene.

The results compiled in Table 2 and the bi-parametric spectra (Figs. 5–8) clearly show that the effects of the substituents are as significant as the solvent effect on discrimination. Nearly all the 1,8-naphthalimides bearing this di-*t*-butylphenyl bulky substituent are able to discriminate in solution fast neutrons from gamma rays.

On the one hand, an electrostatic potential (ESP) calculation of naphthalimide 1 performed with Arguslab software [12] showed that the di-*t*-butylphenyl moiety is extremely electronenriched, due to the strong field effect from both tertio-butyl groups (this figure is presented in the Supplementary data, available at [www.sciencedirect.com](http://www.sciencedirect.com)). The high local electron density might facilitate interactions between neutrons-generated protons recoil and fluorophores. This calculation confirms also the position of the di-*t*-butylphenyl moiety, which in the most energetic favourable state is perpendicular to the naphthalimide core (with the same calculations, the phenyl moiety for compound 10 is deviated of ca. 301 from the naphthalimide). On the other hand, when we try to figure out the differences between 1 and 2, only a single CH<sub>2</sub> element is absent for the latter. These compounds have the same spectroscopic characteristics, the same solubility in organic solvents and so on. The main difference may probably arise from the energy of the triplet states. As the wavelength of the singlet state does not have to be confined close to 420 nm for an efficient discrimination (since naphthalimide fluorescing from ca. 400 to 450 nm are able to discriminate), we can assume that the level of the triplet state is changed by varying the substituents. Finally, for compounds fluorescing around 500 nm, discrimination was never observable.

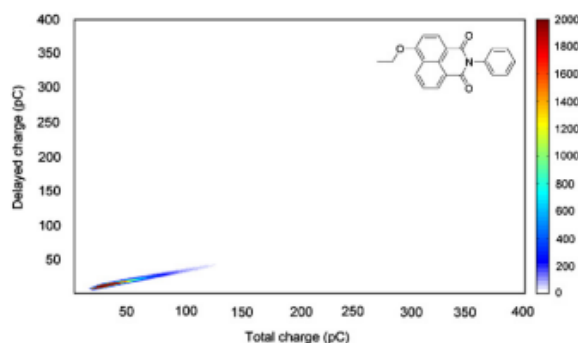


Fig. 7.  $n/\gamma$  Discrimination of *N*-phenyl-4-ethoxy-1,8-naphthalimide 10.

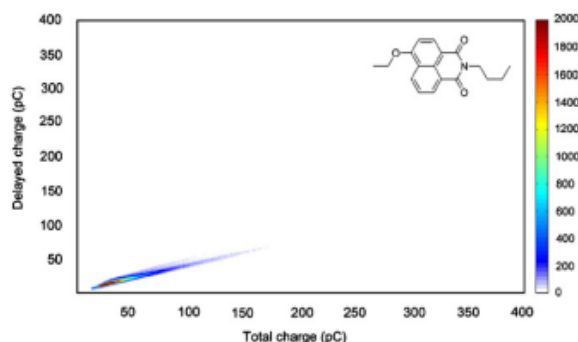


Fig. 8.  $n/\gamma$  Discrimination of *N*-butyl-4-ethoxy-1,8-naphthalimide 11.

**Table 3**  
Influence of dissolved gases in  $n/\gamma$  discrimination performances in liquid scintillators.

Liquid scintillators and gas concerned <sup>a</sup>		Figure number	FOM <sup>b</sup>	$\langle \text{FOM} \rangle$ <sup>c</sup>	$\theta$
Ar	O <sub>2</sub>				
1,8-Naphthalimide/toluene		4	0.6–1.6 (35–270)	0.93	5.1
BC-501A		10	1.0–2.4 (35–250)	1.66	8.3
NE213		12	0.8–1.8 (35–200)	1.39	7.8
Ultima Gold AB		–	0.7–1.3 (40–135)	0.91	6.3
	1,8-Naphthalimide/toluene	9	0.5–1.8 (35–250)	1.00	5.5
	BC-501A	11	0.3–1.8 (35–240)	1.12	4.1
	NE213	13	0.3–1.5 (35–220)	0.97	4.1
	Ultima Gold AB	–	0.3–1.2 (40–200)	0.73	4.4

<sup>a</sup> The naphthalimide involved in this study is *N*-(2',5'-di-*t*-butylphenyl)-4-ethoxy-1,8-naphthalimide 1, at the concentration 10 g L<sup>-1</sup> in spectroscopic grade toluene.

<sup>b</sup> Figure of merit.

<sup>c</sup> Average of the figure of merit in the range 50–100 pC.

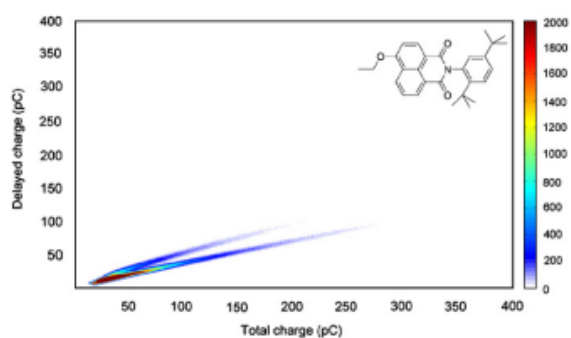


Fig. 9.  $n/\gamma$  Discrimination of *N*-(2',5'-di-*t*-butylphenyl)-4-ethoxy-1,8-naphthalimide 1 in toluene saturated with oxygen.

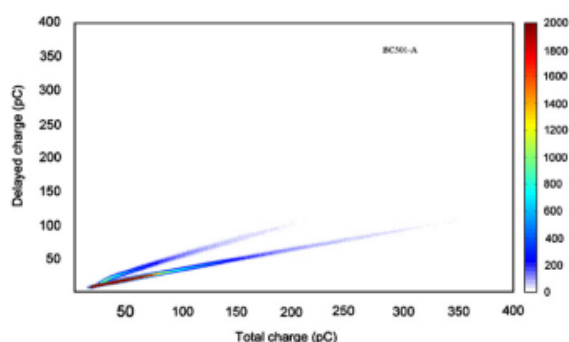


Fig. 10.  $n/\gamma$  Discrimination of BC-501A saturated with argon.

### 3.3. Dissolved gas effect

Dissolved oxygen is known to drastically affect the scintillation of liquid scintillators [13]. It has been demonstrated that this gas is able to quench triplet states and therefore inhibit discrimination. The behaviour of a scintillator composed of the naphthalimide 1 in gas-saturated toluene, either with argon or oxygen, was compared with commercial liquid scintillators in the same conditions. The discrimination results are presented in Table 3 and in the following figures.

Table 3 points out that our naphthalimide-based scintillator remains efficient even when the solvent is saturated with oxygen, whereas the discrimination properties of commercial scintillators are partially affected. Indeed, the neutron and gamma lobes are less defined, the figure of merit and the  $\theta$  angle decrease as well. The average of the figure of merit calculated in the range 50–100 pC loose 20–30%, depending on the commercial scintillator. To the best of our knowledge, it is the first example in the literature for this kind of statement. Unlike commercial products, these new scintillators do not therefore need a special gaseous treatment before use.

It is also noteworthy that unlike commercial scintillators, our products do not deteriorate with light or time. Indeed, n/Y discrimination with toluenic solutions of 1 prepared several months ago and conserved in the fridge provided similar results to freshly prepared liquid scintillators (Figs. 9–13).

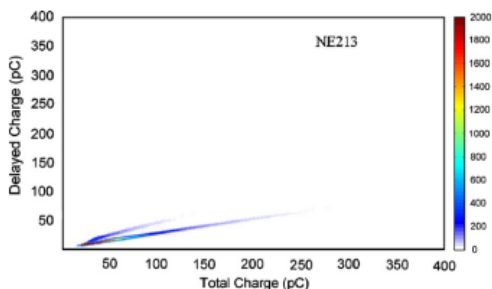


Fig. 12. n/Y Discrimination of NE213 saturated with argon.

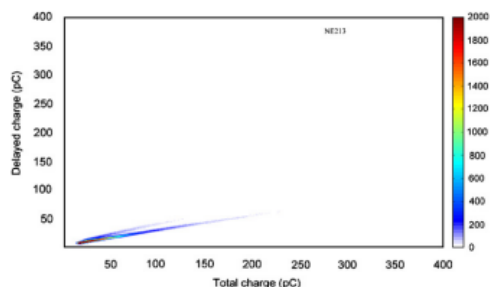


Fig. 13. n/Y Discrimination of NE213 saturated with oxygen.

### Acknowledgements

The authors are indebted to Dr. Nicolas Saettel for his computing calculations on the 1,8-naphthalimide core. This work was carried out with the support of the French governmental agency “Agence Nationale de la Recherche” and NEUTROMANIA program for the grant to M.H.

### Appendix A. Supplementary materials

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.nima.2009.01.191.

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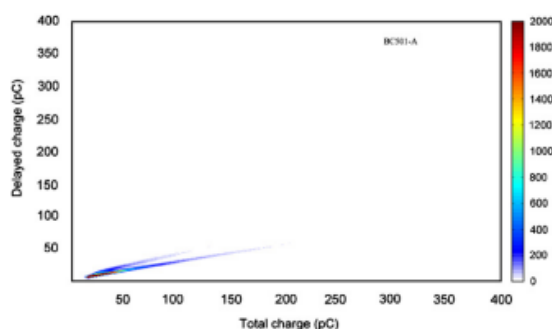


Fig. 11. n/Y Discrimination of BC-501A saturated with oxygen.

### 4. Conclusion

We propose herein a new 1,8-naphthalimide fluorophore specially designed for n/Y discrimination. A study of the structure/property relationships showed that both substituents located on 4- and N- positions were of importance. The reasons for these effects are still under study, especially for the energetic modelling of the triplet states.

A preliminary study concerning the solvent influence on both scintillation and discrimination properties pointed out that our best scintillator presents relatively similar light output and decay time, comparable with commercial scintillators. Three main advantages can be retained.

First is the simplicity of the liquid scintillator composition, based on the use of a single naphthalimide dissolved in the appropriate solvent. Indeed, there is no need of primary fluorophore for a good discrimination.

Second is the broad range of concentrations that can be envisaged. Thus, discrimination is visible from only 3 g L<sup>-1</sup> until saturation of the naphthalimide in the solvent. The discrimination efficiency normalized to the number of counts of a 1,8-naphthalimide/toluene 10 g L<sup>-1</sup> system (Fig. 4) is much better than NE213 (Fig. 12), since it displays a better energy sensitivity for both neutrons and gamma.

Last, it is now not necessary to remove oxygen dissolved in the medium to observe an efficient discrimination. The study of different analogs of 1,8-naphthalimides will permit us to understand all the mechanisms involved in radiation/scintillator relationships. We have developed maybe the simplest liquid scintillator, available from commercial toluene with a good fluorophore suitable for n/Y discrimination.

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