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A NEW PLASTIC SCINTILLATOR WITH LARGE STOKES SHIFT

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We have developed a new plastic scintillator with the novel characteristic of highly localized light emission; scintillation and wavelength shifting take place within a few tens of micrometers of the primary ionization. The new scintillator consists of a scintillating polymer base [polyvinyl toluene (PVT) or polystyrene (PS)] doped with a single wavelength shifter, 1-phenyl-3-mesityl-2-pyrazoline (PMP), which has an exceptionally large Stokes shift and therefore a comparatively small self-absorption of its emitted light. In other characteristics (e.g. scintillation efficiency and decay time) the performance of the new scintillator is similar to a good quality commercial plastic scintillator such as NE110.

1. Introduction

We are currently developing * precise compact tracking detectors which are suitable for operation at high luminosity hadron colliders. These detectors involve parallel bundles of narrow-diameter scintillating fibres which are read out via image intensifiers and CCDs. The principal design requirements for the fibres are as follows: narrow diameter (< 50 μm), good light yield for minimum ionizing particles, long attenuation length (> 1 m), fast emission of light (time constant < 10 ns) and radiation-hard.

Plastic scintillating fibres can potentially meet all these requirements and, in addition, possess the attractive property of a relatively long radiation length. At present, there are two major limitations facing plastic scintillating fibres. The first concerns the lack of a plastic scintillator which can provide efficient narrow-diameter fibres; present scintillator/wavelength shifter combinations are unsuitable below fibre diameters of ~ 500 μm. The second concerns reflective losses at the core–cladding interface, which increase with decreasing fibre diameters. It is the first item which is the subject of this paper.

In order to understand this limitation, we must first consider the mechanism of trapping light in scintillating fibres. The "step index" fibre consists of a scintillating core and a nonscintillating cladding of refractive indices n_{co} and n_{cl}, respectively. The fraction of the emitted light which is trapped, in each direction, is $f \sim 0.5(1 - n_{cl}/n_{co})$. This trapping fraction is rather small, e.g. a fibre with a PS-based core ($n_{co} = 1.59$) and a polyvinyl acetate cladding ($n_{cl} = 1.46$) has $f \sim 4\%$.

Since the polymer base, PVT or PS, is opaque to its scintillation light, this must be shifted into a wavelength region where the polymer is transparent (380–600 nm).

If all the scintillation and wavelength shifting takes place within a distance which is small compared with the fibre diameter, the light will be collected with the nominal efficiency, f. However, when the diameter of the fibres is reduced to a size comparable with, or smaller than, the absorption length of the primary, ultraviolet (UV), scintillation light there are two undesirable consequences: (a) light crosses over into adjacent fibres, which may trap light after wavelength shifting (and thereby give rise to fibre crosstalk), and (b) the light output from the original fibre is severely reduced since the primary light must now be trapped before absorption, followed by trapping of the wavelength shifted light, and so the light collection efficiency approaches f^2. As discussed in the next section, this limitation starts in present plastic scintillators at a fibre diameter of ~ 0.5 mm.

A simple solution to this problem would be to increase the concentration of the wavelength shifter and thereby reduce the absorption length of the UV light. However, since all wavelength shifters suffer some self-absorption of their emitted light, this will lead to a

* This work is performed within the framework of the LAA Project [1].

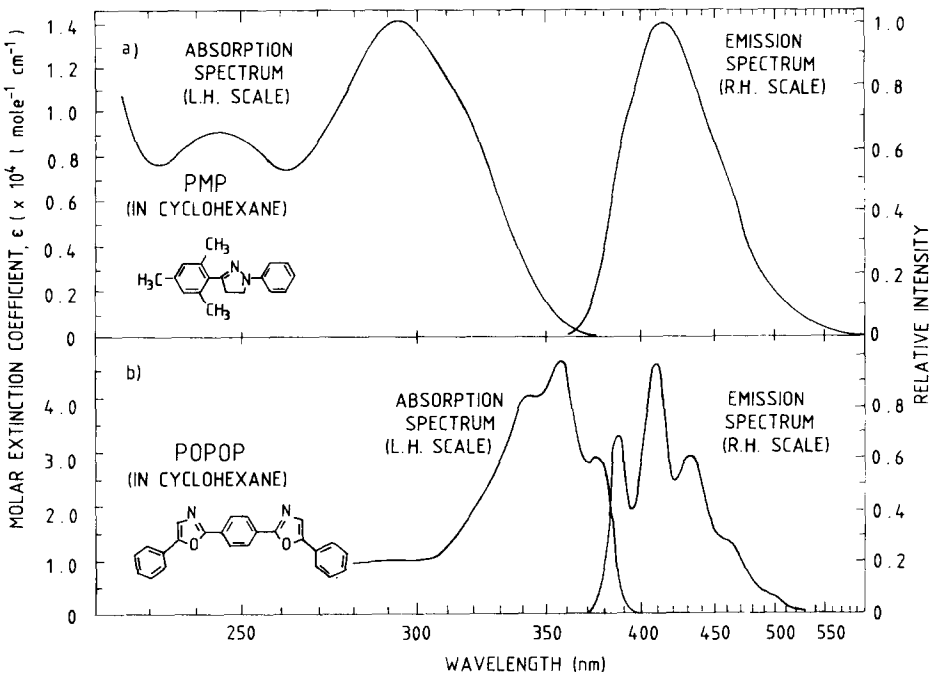

Fig. 1. Absorption and emission spectra of (a) PMP [2] and (b) POPOP [4], dissolved in cyclohexane.

decrease in the "bulk" absorption length of the finally emitted light. Since the wavelength shifters in present use give rise to bulk absorption lengths of ~ 2 m, their concentrations cannot be substantially increased.

Table 1
Comparison of the photophysical properties [a] of PMP [2] with the widely used POPOP [4]

Formula	PMP [b] $C_{18}H_{20}N_2$	POPOP [c] $C_{24}H_{16}N_2O_2$
Molar mass [g mol^{-1}]	264.37	364.40
Wavelength of peak absorption [nm]	295	362
Wave number of peak absorption [cm^{-1}]	33900	27930
Wavelength of peak emission [nm]	425	418
Wave number of peak emission [cm^{-1}]	23530	24390
Stokes shift [cm^{-1}]	10370	3540
Peak molar extinction coefficient ($\times 10^4$ l mol^{-1} cm^{-1})	1.4	4.8
Quantum efficiency	0.88	0.93
Fluorescent decay time constant [ns]	3.01	1.44
Maximum solubility in toluene at 20 °C (mol l^{-1})	1.27	–

[a] Solvent: cyclohexane.
[b] PMP: 1-phenyl-3-mesityl-2-pyrazoline.
[c] POPOP: 1,4-di-2-(5-phenyloxazolyl)benzene.

For these reasons we decided to develop a new plastic scintillator containing a wavelength shifter with a large separation between the absorption and emission spectra (large Stokes shift) matching the emission and the transparent region of the polymer base. It should provide as well the standard properties of large UV absorption cross section, high quantum yield, prompt decay time and good solubility.

After a study of the literature [2], we decided to investigate the wavelength shifter 1-phenyl-3-mesityl-2-pyrazoline (PMP) **. We will now review the properties of this material, followed by the way in which we prepared the samples of plastic scintillator. The final sections concern the measurements performed on the plastic scintillator and the experimental results.

2. Properties of PMP

We summarize in table 1 the photophysical properties [2,4] of PMP and, for comparison, a wavelength shifter (POPOP) commonly used in plastic scintillators. The most striking difference between these two materials is the large Stokes shift of PMP. This is clearly seen in fig. 1, which shows the absorption and emission spectra of PMP [2] and POPOP [4], dissolved in cyclohexane.

** This scintillating material is now being applied in large-volume liquid scintillator detectors [3].

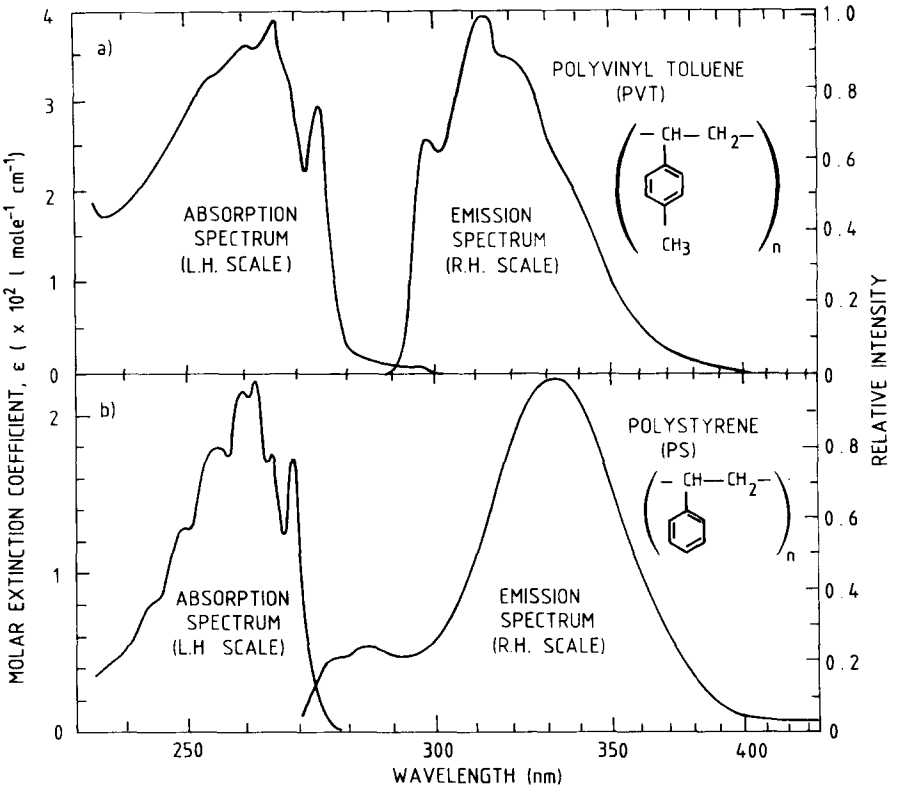

Fig. 2. Absorption and emission spectra of (a) PVT and (b) PS.

In our application, the most critical feature of these curves concerns the overlap of the absorption spectra into the region of the emission spectra. However, as we shall now see, the sensitivity of these data in this region is insufficient for our purpose. Therefore, the large Stokes shift observed in fig. 1a provides only qualitative support for the suitability of PMP to our application.

For the absorption curves of fig. 1, the molar extinction coefficient, ϵ (l mol^{-1} cm^{-1}), is defined by the expression

$$I = I_0 \times 10^{-d\epsilon c} = I_0 \, e^{-d\epsilon c(\ln 10)},$$

where I_0 and I are the incident and transmitted light intensities, respectively, d (cm) is the distance of light propagation and c (mol l^{-1}) is the concentration. The typical concentration of POPOP in plastic scintillators is ~ 0.5 g l^{-1} (1.4×10^{-3} mol l^{-1}). Therefore, in order to have a bulk absorption length $\Lambda > 1$ m, the mean molar extinction coefficient in the region of its emission spectrum is given by $100\epsilon \times 1.4 \times 10^{-3} \ln 10 \leqq 1$, i.e. $\epsilon \leqq 3$. Such values are clearly below the sensitivity of the measurements shown in fig. 1.

A further advantage of its large Stokes shift and broad absorption spectrum (fig. 1a) is that PMP can be used as a single scintillator in a PVT or PS solute. The emission spectra of PVT and PS (fig. 2) are suitably matched to the absorption spectrum of PMP. This is in contrast with presently used wavelength shifters, such as POPOP, which require an additional scintillator, e.g. p-terphenyl (pTP), to convert the UV scintillation light in an intermediate step.

We can estimate the absorption length of the UV light by POPOP in present plastic scintillators. The UV absorption length $\Lambda_{UV} = 1/(\epsilon_{UV} c \ln 10)$, where ϵ_{UV} is the mean value of the molar extinction coefficient, averaged over the incident spectrum. If we take into account the pTP emission spectrum then, for POPOP, $\epsilon_{UV} \sim 2 \times 10^4$ l mol^{-1} cm^{-1} and so $\Lambda_{UV} = 1/(2 \times 10^4 \times 1.4 \times 10^{-3} \ln 10)$ cm $= 150$ μm. The unsuitability of POPOP for our purposes is clear; we would have to raise the POPOP concentration by a factor of 15 to achieve the desired value, $\Lambda_{UV} \sim 10$ μm, and this would result in a bulk absorption length of the output light, $\Lambda \sim 200/15 = 13$ cm.

In the case of PMP in PVT, $\epsilon_{UV} \sim 10^4$ l mol^{-1} cm^{-1} and so the PMP concentration which is necessary for $\Lambda_{UV} = 10$ μm is $c \sim 1/(10^4 \times 10^{-3} \ln 10) = 0.043$ mol l^{-1}. This concentration is equivalent to 12 g l^{-1} or $\sim 1.2\%$ by weight. This estimate represents an upper limit for the required concentration since part of the energy transfer from PVT is radiationless.

3. Preparation of the scintillating plastics

Measurements [5] on PS scintillators have shown that the scintillation efficiency is highly dependent upon the purity of the initial monomers. To eliminate, as far as possible, the presence of contaminants during polymerization, monomers and chain transfer agents (CTA) were distilled twice under vacuum. A special apparatus allowed us to handle all the products without their contact with the atmosphere. To prepare this apparatus for use, it was initially cleaned with sulfochromic acid, rinsed with de-ionized water and then carefully dried with a stream of hot argon gas. In order to prepare the glass polymerization tube (of 40 cm length and 3 cm diameter) it was cleaned with nitric and hydrochloric acids, rinsed with distilled water and treated for about ten minutes in a 3% solution of dimethyldichlorosilane in chloroform, and finally rinsed in sequence with chloroform, methanol and de-ionized water. This treatment built a hydrophobic Langmuir layer on the walls of the tube which helped the removal of the sample after polymerization.

A specified amount (between 0.025 and 0.05 mol l^{-1}) of PMP * was placed in the tube, which was located with the purification apparatus. Monomers with CTA and initiators (when needed) were injected under vacuum. The mixture was then degased with repeated freezing–thawing cycles (usually 6 cycles) until no bubbles formed in the liquid. Finally, the tube was sealed off under vacuum.

Monomers thus prepared were immersed in an oil bath equipped with a stirrer. The oil bath allowed us to maintain the sample within ± 0.1° C of the desired temperature. The thermal cycle of the bath (which was controlled with a microcomputer) started by raising up to a constant temperature (95–120 ° C depending on the monomer) for a period of time which was long enough to reach the solid state. The temperature was then gradually increased to 160 ° C and kept constant for 24 h. Finally, the tube was cooled to room temperature at a very slow rate, especially near the transition temperature. After cooling, the tube was sawed across its upper part and the polymer was extracted very easily (it did not stick to the glass). The final sample was a cylinder, 25 cm in length and 28 mm in diameter.

Several samples were prepared in this way, using PVT and PS plastics. Each was subsequently cut and polished into several smaller samples of thicknesses ranging between 0.1 and 130 mm.

* The PMP was purchased from Merck, P.O. Box 4119, D-6100 Darmstadt 1, FRG.

4. Test apparatus and procedure

In order to evaluate the new scintillator, we performed a parallel set of measurements on the PMP samples and on "standard" samples. The standard samples were prepared with identical physical dimensions and surface polish as the PMP samples, in order to remove differences in the geometrical collection efficiencies of the light. For the standard samples we chose a high-quality PVT plastic scintillator, NE110 **. Tests were performed – using a spectrophotometer and using radioactive sources, as described below – to investigate the scintillation efficiencies, UV absorption characteristics, transmission properties and response time of the plastic scintillators.

4.1. Spectrophotometer measurements

The initial evaluation of the extinction coefficient of PMP in the wavelength region of its emission spectrum was performed by spectrophotometer measurements of the transmission of PMP dissolved in cyclohexane (C_6H_{12}). This solvent was chosen since it has excellent transparency in the wavelength range of interest (> 370 nm). Pure cyclohexane, followed by two PMP solutions (of 0.026 mol l^{-1} and 0.15 mol l^{-1}), were measured in a quartz cuvette with an optical path of 5 cm.

Subsequently samples of undoped plastic and of plastic scintillator were measured. A thickness of 6 cm was generally used, corresponding to the maximum size which is accepted by the spectrophotometer.

These data were used both directly to measure the transmission vs wavelength (λ) and also to calculate the molar extinction coefficient and the bulk absorption length. The molar extinction coefficient $\epsilon(\lambda)$ was calculated from the transmitted intensity fraction I/I_0, the sample thickness d and the concentration c as follows:

$$\epsilon = [\log_{10}(I_0/I)]/(dc),$$

and the absorption length $\Lambda(\lambda)$ was calculated from the expression

$$I = I_0\, e^{-d/\Lambda}, \text{ i.e. } \Lambda = d/\ln(I_0/I).$$

In calculating Λ, we assumed perfect transparency at $\lambda = 600$ nm and set I_0 equal to the measured transmission at this wavelength. This value was typically (86 ± 2)% and provides a measurement of the losses due to reflection and scattering at the entrance and exit faces of the particular sample under test. The bulk absorption length was calculated by computing the mean value of

** NE110 is manufactured by Nuclear Enterprises Ltd., Sighthill, Edingburgh EH11 4BY, Scotland. This product is equivalent to BC412, manufactured by Bicron Corp., 12345 Kinsman Road, Newbury, OH 44065, USA.

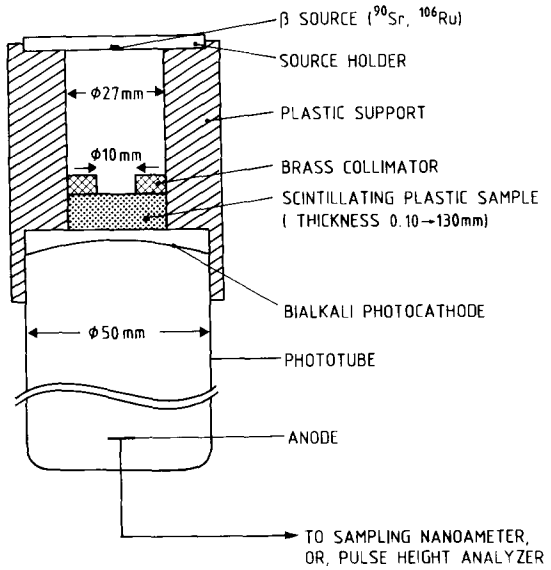

Fig. 3. Test apparatus for measurement of the light yields of the plastic scintillators.

$\Lambda(\lambda)$, weighted by the appropriate emission spectrum. In calculating ϵ, I_0 was determined from the measured transmission of a sample of pure solute (i.e. pure cyclohexane, PVT or PS, as appropriate) with identical physical dimensions as the doped sample, after normalizing the transmissions to the same value at $\lambda = 600$ nm.

4.2. Measurements using radioactive sources

The responses to ionizing particles were evaluated using β sources: ^{90}Sr (β_{max}: 0.55 MeV) ^{90}Y (β_{max}: 2.3 MeV), and ^{106}Ru (β_{max}: 0.04 MeV) + ^{106}Rh (β_{max}: 3.4 MeV and γ transitions of 0.51 and 0.62 MeV). The apparatus is shown in fig. 3. The sample under test was placed directly on the entrance face of the phototube and irradiated from above by means of a collimated β source. For samples exceeding a length of 60 mm, the collimator was removed and the source holder was placed directly on the top face of the sample. In the case of thin (≤ 1 mm) samples, measurements were also taken with a 60 mm pure PVT filter inserted between the sample and the phototube in order to eliminate UV light (from pTP emission) which had not been previously absorbed and wavelength shifted.

The output of the phototube was recorded in two ways: (a) a measurement of the direct current (dc) using a 1 s sampling nanoameter and (b) a measurement of the pulse height distribution of individual β particles.

The dc measurement involved the ^{90}Sr source and an EMI 9813KB phototube. The tube HV was varied over a range of values between 800 and 1500 V, corresponding to dc of 1 nA to 2 μA. In this way, spurious measurements could be identified and a single response figure determined with the best accuracy. We estimate a relative uncertainty between two measurements of ±4%.

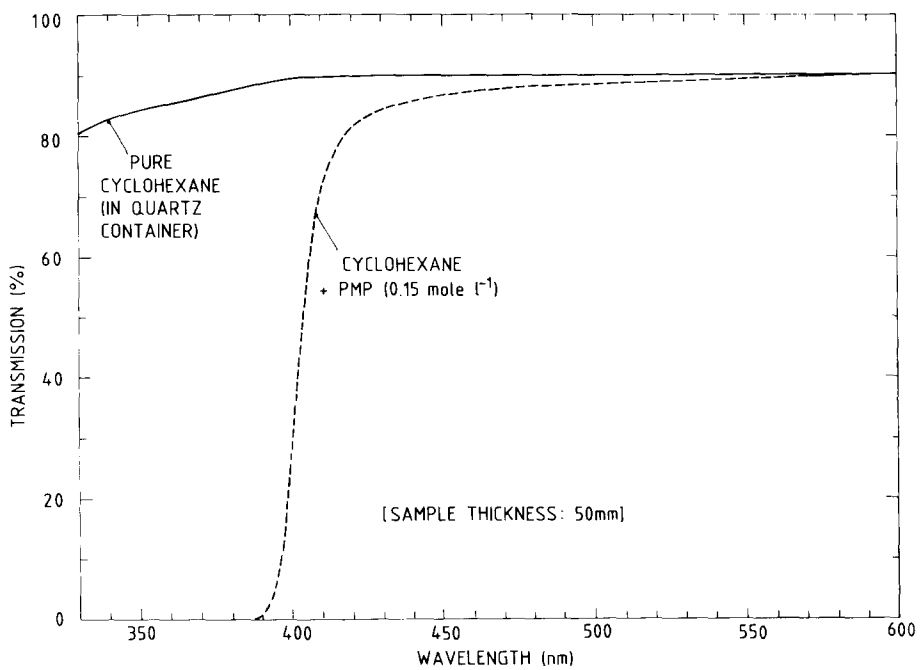

Fig. 4. Transmission vs wavelength for a quartz cuvette, of 5 cm internal dimension, containing (a) cyclohexane and (b) 0.15 mol l^{-1} PMP dissolved in cyclohexane.

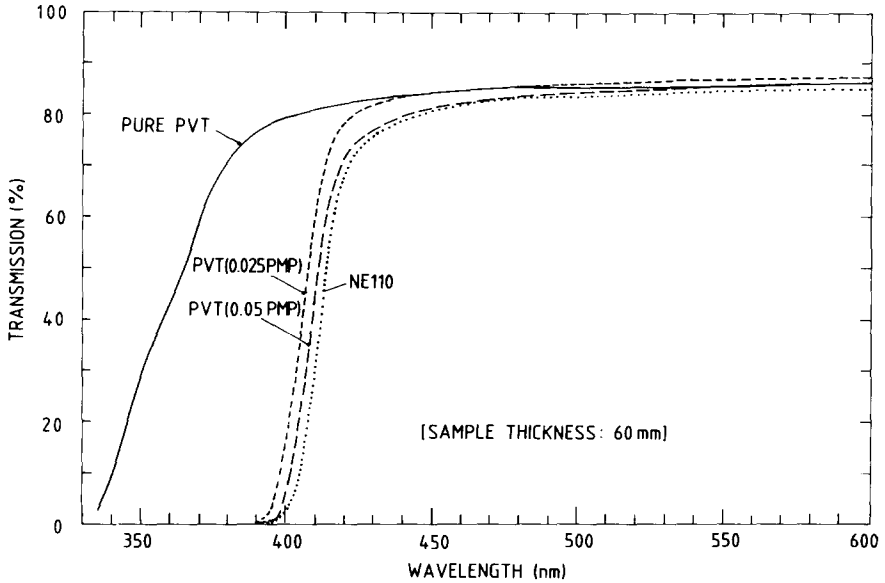

Fig. 5. Transmission vs wavelength for PVT plastic samples and NE110, of thickness 60 mm. The PMP concentrations (mol l^{-1}) are indicated in parentheses.

For a few of the samples, the pulse height distribution was measured using fast electronics. This was done both as an independent check of the accuracy of the dc measurements and, more significantly, in order to confirm the absence of any component of light with a long decay time constant. The pulse height was measured in a time interval of 100 ns from the start of the pulse and so, in contrast with the dc measurement, was insensitive to light emitted with a long decay time constant. For these tests we used the ^{106}Ru source, RCA 8850 (quantacon) phototube and a LeCroy 3001 qVt multichannel analyzer.

These data were used to evaluate the scintillation efficiencies of the PMP scintillators relative to NE110, as well as the absolute photon yields for minimum ionizing particles. They also measure the relative performance of thin scintillator films, where the advantage of the localized emission in the new scintillator can be seen

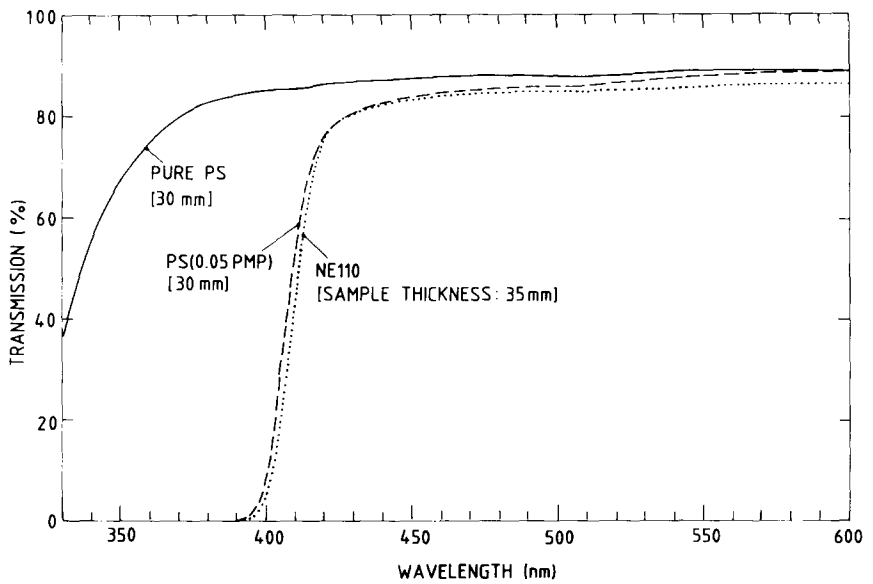

Fig. 6. Transmission vs wavelength for PS plastic samples of thickness 30 mm. For comparison, the transmission of a 35 mm thick sample of NE110 is included.

directly. Finally, these data show the transparency of each plastic scintillator to its own output light over distances ≤ 13 cm.

5. Results

The spectrophotometer measurements of PMP dissolved in cyclohexane (fig. 4) indicate excellent transmission above ~ 415 nm. This concentration of PMP (0.15 mol l^{-1}) corresponds to $\Lambda_{UV} = 1/(\epsilon_{UV} c \ln 10) \sim 1/(10^4 \times 0.15 \ln 10)$ cm = 3 μm. It is apparent that, even at such high concentrations, PMP is transparent to a large fraction of its emission spectrum (fig. 1a).

The spectrophotometer measurements of plastic scintillators doped with PMP are shown in fig. 5 (PVT) and fig. 6 (PS). The same qualitative feature of excellent transmission above a certain wavelength is preserved between the liquid and plastic measurements. This is a nontrivial result since the plastic involves a solid matrix and heat processing. However, some differences are observed; in particular the absorption edge occurs at a slightly (15 nm) longer wavelength in the plastic medium. In order to investigate whether a similar shift was also present in the emission spectrum, we performed preliminary spectrofluorometer measurements on PMP in cyclohexane and on PMP in PVT. We observed an identical shift of about +15 nm between these two emission spectra and therefore conclude that the transparency of the PMP to its emitted light is unaffected. With regard to heat processing, we performed a set of spectrofluorometer measurements on samples of PMP which had been heated up to temperatures of 180 °C. Providing care was taken in eliminating oxygen contamination during these tests, we found no change in the photophysical properties of PMP after this heat cycling.

The curves in fig. 5 show a decrease in the transmission efficiency with increasing concentration of PMP. At a PMP concentration of 0.05 mol l^{-1}, the transmission is equivalent to NE110 which is known to have an excellent bulk absorption length. After integrating these transmission data over the appropriate emission spectra, we calculate bulk absorption lengths of ~ 2 m for both PVT (0.05 mol l^{-1} PMP) and NE110 (however, as we have already discussed, the concentration of the final wavelength shifter in NE110 is much too low for a narrow-diameter scintillating fibre). Of course, in other applications which do not require such a low value of Λ_{UV}, the PMP concentration can be reduced – for example a concentration of 5×10^{-3} mol l^{-1}, having $\Lambda_{UV} \sim 100$ μm – to result in a bulk absorption length significantly longer than 2 m.

The data in figs. 5 and 6 were used to calculate the molar extinction coefficients vs wavelength in the region of the emission spectrum of PMP, as described in section 4.1. The results are shown in fig. 7. We have also included the calculation for NE110, which has 5×10^{-4} mol l^{-1} concentration of the final wavelength shifter, of which the identity is proprietary.

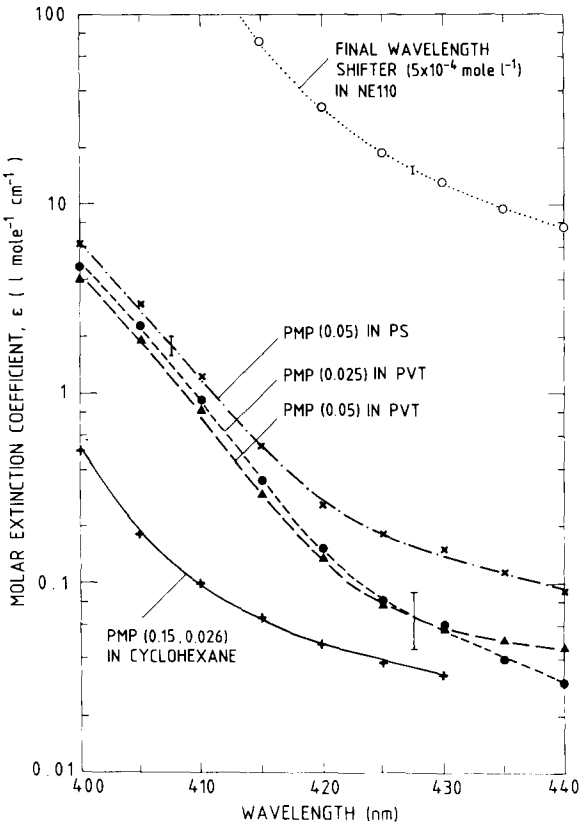

Fig. 7. The molar extinction coefficient vs λ for PMP in liquid (cyclohexane) and plastic (PVT and PS) solutes, in the region of PMP fluorescence. For comparison, the molar extinction coefficient is also shown for the final (proprietary) wavelength shifter in NE110. The estimated errors on these measurements are indicated in several regions by representative error bars.

In the absence of chemical interaction between PMP and its host material, the value of ε should be independent of concentration or solute. As indicated earlier, we observe a shift in ε of +15 nm between the liquid and plastic solutes. Within experimental errors, we observe no dependence of ε on the type of plastic (PVT or PS) or on the concentration. These measurements show that the molar extinction coefficient of PMP is sufficiently small in the emission region to be used in high concentrations in liquid and plastic solutes. For comparison we see that the final wavelength shifter in NE110 has ε values which are approximately two orders of magnitude larger and is consequently limited to low concentrations.

The measurements of the light yields taken with β sources are displayed in fig. 8. The relative yields (PMP

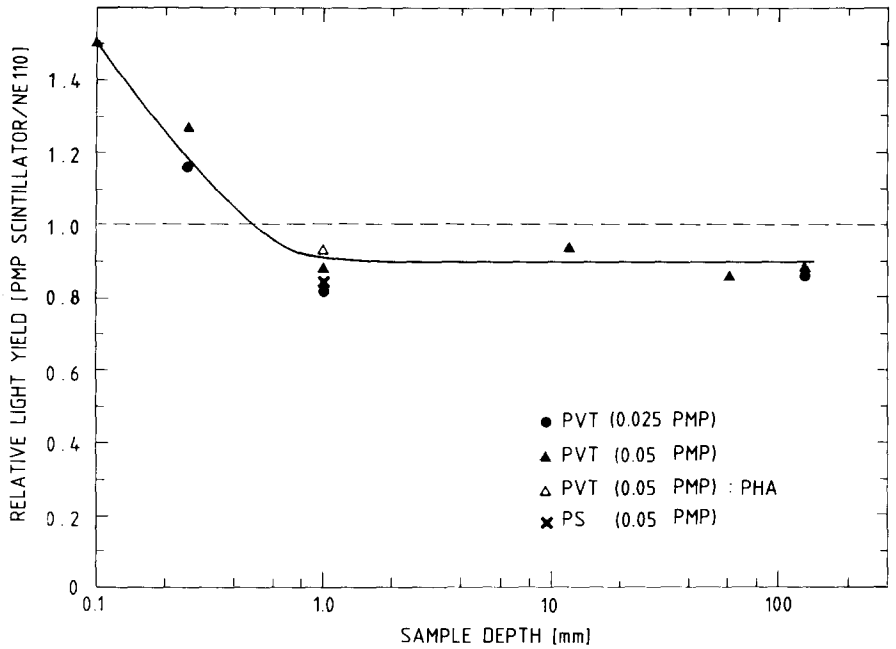

Fig. 8. The relative light yields (PMP plastic scintillator/NE110) observed in samples of several thicknesses. The measurements were taken with the apparatus of fig. 3, recording the anode dc except where PHA (pulse height analysis) is indicated. The estimated error on each point is ±4%. At thicknesses < 1 mm the light yield of NE decreases because its UV absorption length becomes comparable to the sample thickness. In thin fibres, however, this tendency should be much more pronounced, since the collection of scintillating light is different from that in the presented disc geometry.

plastic scintillator/NE110) are shown at several different thicknesses of samples. For thin (~ 1 mm) samples, these data directly measure the overall scintillation efficiency, i.e. the relative output light after scintillation and wavelength shifting. The longer samples include also the effects of reflection at the sides of the sample and of absorption within the sample.

We observe that the PMP plastic scintillator and NE110 are quite similar (within ±20%) over most of this range. The variations in the relative light yield above 1 mm relate to differences in the rate of absorption of the shortest wavelengths of the emission spectra. From these data we measure the relative scintillation efficiency (relative light yield) PMP plastic scintillator/NE110 = 0.95 ± 0.05. This efficiency is found to be insensitive to PMP concentration within the measured range (0.025–0.05 mol l^{-1}); we expect the efficiency will remain the same down to very low concentrations of PMP.

For thin (< 1 mm) samples, a significant fraction of the light emerging from the NE110 has not yet been absorbed and shifted by the second wavelength shifter, i.e. it is UV light emitted either directly from the scintillation of the PVT or from the first wavelength shifter. This UV light is mainly in the wavelength range 320–380 nm, for which the bialkali photocathode is sensitive, and so is included in the measurements. However, since this UV light is absorbed in a few centimetres of PVT or PS, our measurement overestimates the light yield of thin samples of NE110 which would be useful for scintillating fibres. We have corrected for this effect with a filter of 6 cm of pure PVT, as described in section 4.2. The improvement in performance of thin samples of PMP plastic scintillators relative to NE110 is clearly seen in fig. 8, which shows the development of the light yield for samples of 26 mm diameter. Since the light collection in fibres is different, a much stronger decrease of the NE light yield for fibre diameters < 1 mm must be expected.

Most of the data in fig. 8 involve measurements of the phototube dc. However, we also made several fast-pulse measurements using a pulse-height analyzer in order to check for the presence of any unforeseen light emitted with a long time constant. Such light would, of course, present a serious drawback to our application at high-energy hadron colliders, where the event rate could reach 10^8 Hz. As seen in fig. 8, the two measurements – dc and fast-pulse (100 ns integration time) – were found to agree with each other within experimental errors. We therefore find no evidence for light emitted over a long decay time. As seen in fig. 9, the phototube anode pulse shapes of PMP plastic scintillator and NE110 are indistinguishable (in fact, since both plastic scintillators have an expected emission time $\tau < 3$ ns, the observed rise

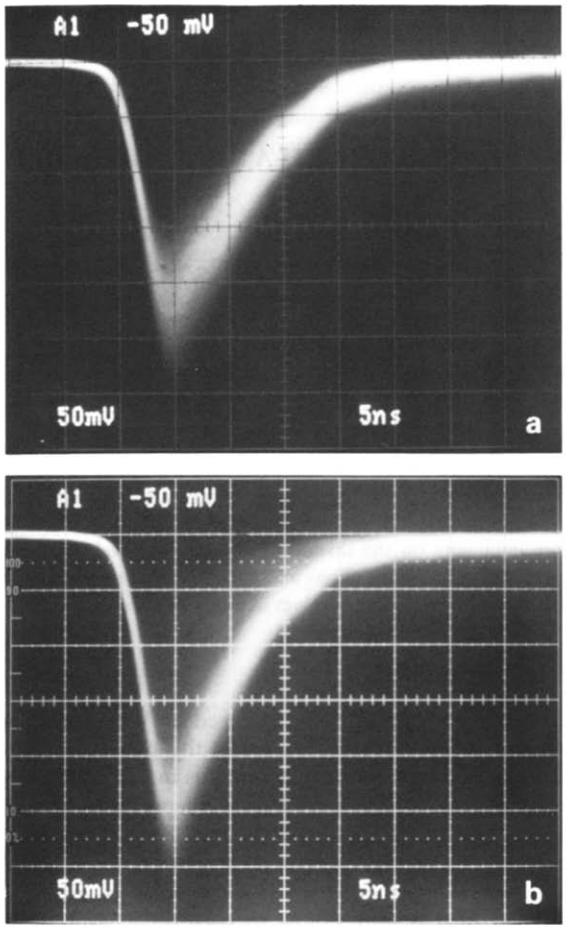

Fig. 9. (a) Phototube pulse shape from PVT+PMP (0.05 mol l^{-1}) plastic scintillator excited by a 5.5 MeV α source. (b) Phototube pulse shape for NE110. The horizontal scale is 5 ns per division.

and decay times of the anode pulses are dominated by the characteristics of the electronics and not of the plastic scintillator).

The pulse height measurement also provides a direct measurement of the photoelectron yield of the plastic scintillators. In the case of a 1 mm thick sample of PVT + PMP (0.05 mol l^{-1}), we observe (180 ± 15) photoelectrons per millimeter. From this measurement, we estimate the absolute photon yield over 4π sr, for minimum ionizing particles, to be (9.1 ± 2) keV^{-1}.

6. Conclusions

We summarize the key properties of PMP plastic scintillators and NE110 in table 2. It is apparent that plastic scintillators containing PMP match the performance of good quality commercial plastic scintillators in all respects, while introducing the novel characteristic of highly localized light emission. We have determined that PMP is compatible with the scintillating polymers PVT and PS. This new class of plastic scintillators which contain PMP opens the way for the production of efficient narrow-diameter plastic scintillating fibres.

Table 2
Comparison of PMP plastic scintillator with NE110

Feature	Quantity	PVT + PMP (0.05 mol l^{-1})	NE110
(1) Locality of light emission	UV absorption length, Λ_{UV} [μm]	10	300
(2) Transparency	Bulk absorption length, Λ [m]	~ 2	~ 2
(3) Scintillation efficiency (minimum ionizing particles)	(a) Relative light output	0.90 ± 0.05	1.0
	(b) Absolute photon yield over 4π sr [keV^{-1}]	9 ± 2	10 ± 2
(4) Rate capability	Decay time constant [ns]	< 5	< 5

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