Stoichiometric Cerium Compounds as Scintillators, II. CeP$_5$O$_{14}$

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Abstract—Results concerning the relative importance of several processes which are responsible for the efficiency and speed of a scintillator are presented here. Arguments for concentrated (stoichiometric) materials are presented and illustrated in the case of Ce-pentaphosphate, (CeP$_5$O$_{14}$). The low density of this material does not make it a viable scintillator, but its remarkable simplicity and relatively easy technology make it perfect to study basic physical mechanisms of scintillation in concentrated materials. Based on studies of a series of Ce$_x$La$_{1-x}$P$_5$O$_{14}$ crystals, we formulate general conclusions concerning the relative importance of the process starting with direct ionization of the emitting centers (Ce^{3+}) versus long range gathering of electron-hole pairs. These conclusions provide guidance form optimizing speed and efficiency of future concentrated scintillators. In addition some peculiarities of the Ce-pentaphosphate luminescence are reported and interpreted.

I. INTRODUCTION

IN LIGHTLY doped materials direct ionization of activator ions constitutes only a small fraction of the total energy deposited in the material by a high energy particle. Therefore, it is clear that some efficient mechanisms of energy transfer and/or charge transport and diffusion are needed to deliver the excitation to the activator ion and produce scintillation light, [1], [2]. By contrast, the expected advantage of the concentrated materials, in which the activator (in the form of an ion or molecule) is present in every unit cell, is that the "prompt" scintillation processes will essentially drain all of the excitations produced in the material, thus eliminating the need and importance of longer range transfer processes. The degree, to which this expectation is fulfilled determines the approach to an "ultimate" scintillator combining maximum speed and efficiency, [3].

CeF$_3$, cerium trifluoride, is a new and promising concentrated scintillator, [4]–[6]. However it appears to be rather far from the "ultimate scintillator". As we have shown previously, it has specific structural problems which tend to reduce its efficiency, [7].

Looking for an alternative to test the hypothesis that a concentrated material provides the expected advantage, we formulate the following requirements:

1) The luminescent center should be capable of fast (high oscillator strength f) optical transition and absence of nonradiative processes. The Ce^{3+} ion fulfills these conditions, probably better than any other. Its optical transition, being of d–f type, is fast and efficient. Since there is only one f electron the f–f electronic structure does not overlap with the d–f structure so that competing and slow f–f transitions are lacking.

2) Another requirement concerns the lack of concentration quenching, assuring efficient luminescence at stoichiometric composition. The pentaphosphate lattice, [8], known for its remarkably isolating qualities, [9], is a good choice. Normally lack of concentration quenching seems to require a large Stokes shift, [10], which has the additional advantage that it reduces self-absorption, an unwanted effect in scintillators. Curiously enough, while concentration quenching is totally absent in Ce-pentaphosphates, its Stokes shift is rather small and self-absorption is quite evident. These features are described but not explained in this paper.

3) Finally another requirement is that the ion-lattice combination provides the presence of a filled level in the forbidden gap and/or intra-ionic transitions below energy gap likely to create a local binding potential for the hole, [11]. This is necessary for stability of the 4 + charge state, which ensures that Ce ions will intercept at least part of the energy deposited in the material. It is also likely to create an efficient mechanism of transferring holes from the nearby ions thus enhancing the "prompt" component in the scintillation signal.

The most notable feature of the Ce-pentaphosphate is its high speed, which prompted a suggestion of application as an electron beam indexing phosphor, [12]. We report results of our studies on Ce$_x$La$_{1-x}$P$_5$O$_{14}$, Ce-La-pentaphosphates. These studies constitute a part of the larger project aimed at establishing the ultimate, physical limitations of scintillation performance of Ce-materials, [3], [7]. The preliminary results on Ce-La-pentaphosphates were presented and published elsewhere, [13]. The conclusion was that those materials constitute a relatively simple case lacking complications typical of other Ce-materials. This

makes them primary candidates for studies of basic physical mechanisms of scintillation, [14].

II. MATERIALS AND EXPERIMENT

Samples of Ce-La-pentaphosphates were grown in our lab using a method described by Plattner *et al.*, [8]. They were rather small monocrystals (typically 3×7 mm platelets about 2-mm thick), but of reasonably good optical quality. With one exception, (a total scintillation light output as a function of Ce concentration), we will limit presentation of experimental data to two samples, CeP_5O_{14} and LaP_5O_{14}:Ce (0.05 mol% Ce). These two samples represent extreme cases and are illustrative of tendencies characteristic of the whole set.

Luminescence spectra were measured using a 0.25 m Jarrell-Ash 82-410 monochromator with 1180 grooves/mm grating blazed at 300 nm (second order). The monochromator was equipped with the Hamamatsu R2059 dry-ice cooled photomultiplier tube with quartz window. For luminescence excitation and transmission spectra a second, double monochromator was used (0.22 m Spex 1680) with Xe lamp. Optically excited decays were measured using the Stanford Research SR270 boxcar averager with 2 ns gate. Exciting light was provided by the FL2000 Lambda-Physik dye laser pumped by an N_2 laser operated at 30 Hz and producing pulses of about 3 ns duration time. The dye laser light was frequency doubled using a KDP crystal. Steady state spectra (luminescence, luminescence excitation and transmission) were measured using a current-voltage amplifier (Keithley 427). In most experiments the sample was mounted on the cold finger of a CTI Cryogenics closed cycle He cooler, providing variable temperatures between 23 K and 600 K. Placement of the sample avoided direct reflection of exciting light into the monochoromator. Energy spectra were measured using a standard System 100 Canberra set-up. Decays under ionizing γ and β rays from [207]Bi and Ru/Rh sources were measured using the same equipment adapted for the synchronous photon counting experiment using the method described earlier, [15], [16]. All experiments were controlled by personal computers where data were stored for subsequent analysis.

III. RESULTS

Fig. 1 presents luminescence spectra of two crystals: CeP_5O_{14} and LaP_5O_{14}:Ce (0.05 mol% Ce). As expected, in the case of diluted Ce the spectra consist of two peaks, at 310 and 330 nm, with no change in positions and relative intensities between 24 and 400 K. The energy difference between those peaks, (1960 cm^{-1}), is characteristic for the spin-orbit splitting of $^2F_{7/2}$ and $^2F_{5/2}$ states of the f^1 configuration on which luminescence transitions are terminated. For concentrated Ce-pentaphosphate the spectra are similar and differences can be explained by increased self-absorption, which shifts the peaks and distorts the spectra. The effect is stronger at higher temperatures. At low temperature two peaks are clearly resolved (312 and 332 nm). The low energy peak position is close to
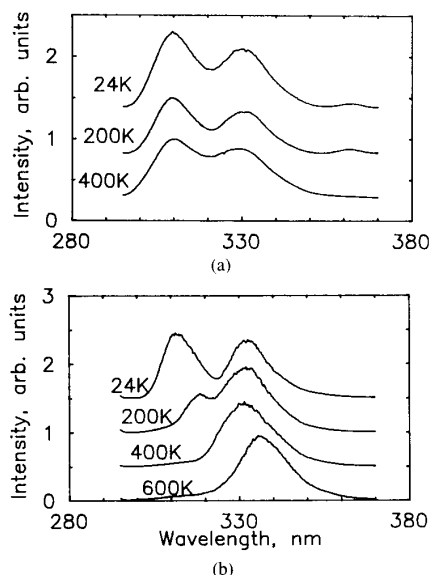

Fig. 1. Luminescence spectra of a) LaP_5O_{14}:0.05% Ce and b) CeP_5O_{14}, under 250 nm cw excitation (Xe lamp) for different temperatures (24 K, 200 K, 400 K and 600 K). Spectra were not corrected for the spectral sensitivity of the system. Baselines for different spectra are shifted for clarity of presentation.

that for diluted Ce while relative intensities and peak positions of the high energy peak change consistently with the assumption of increased self-absorption.

The close similarity between emission spectra of Ce-doped La-pentaphosphate and Ce-pentaphosphate is not unexpected. In mixed Ce-La-pentaphosphates La^{3+} ions will substitute for Ce^{3+} ions but, since they have no f electrons, they will remain inactive. On the other hand the crystal field seen by any of the active Ce^{3+} ions should not change, even for a very low Ce content. This is because Ce-La substitution does not affect nearest and next nearest neighbors of Ce^{3+} and changes in the next coordination spheres are not significant (1.03 Å ionic radius C^{3+} ions being substituted by 1.02 Å La^{3+} ions, [17]). The same should be true for excitation (or absorption) spectra.

In Fig. 2 excitation spectra of two emission bands of LaP_5O_{14}:Ce are shown for 26 K, 200 K, and 400 K. The spectra for two emission bands are very close as expected. Positions of peaks, (240 nm and 295 nm), are slightly temperature dependent especially for the high energy peak. The peaks can be assigned to the crystal field split d states of Ce^{3+} ion. Excitation spectra of CeP_5O_{14}, (Fig. 3), show strong shifts and distortions due to a very high absorption (allowed f–d transition and high concentration of ions). A simple model involving the imaging of the light emitting region on the slit of the monochromator explains the origin of those distortions, [14]. The model generates a distortion of the excitation spectrum with the maximum of the true spectrum replaced by a minimum of the measured spectrum. With this in mind the positions of excitation bands in Ce-pentaphosphate are found to be
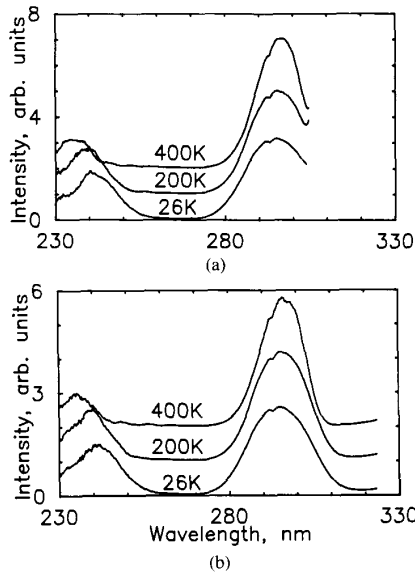
Fig. 2.   Excitation spectra of LaP_5O_{14}:0.05% Ce for two emission wavelengths, a) -312 nm, b) -333 nm, and for different temperatures (26 K, 200 K and 400 K), baselines for different spectra are shifted.

Fig. 3.   Excitation spectra of CeP_5O_{14} for two emission wavelengths, a) -312 nm, b) -333 nm, and for different temperatures (24 K, 200 K, and 400 K). Baselines for different spectra are shifted.
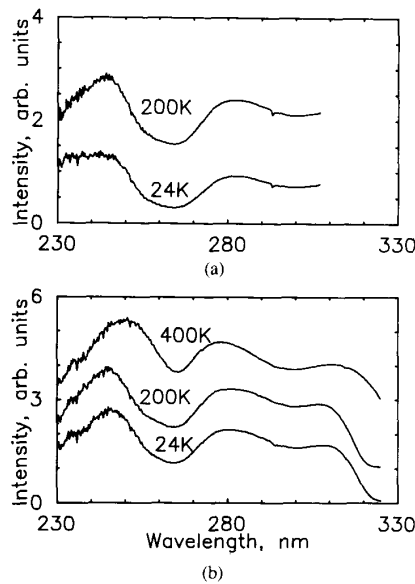
295 and 245 nm, reasonably close to those of Ce-La-pentaphosphate.

In Fig. 4 transmission spectra of LaP_5O_{14}:Ce are shown for 26 K, 200 K, and 400 K. Because of a very high absorption, transmission spectra of CeP_5O_{14} are strongly distorted and not very meaningful, (see Fig. 2 in [13]). The LaP_5O_{14}:Ce spectra show a minimum in transmission around 295 nm, confirming that the absorption edge is

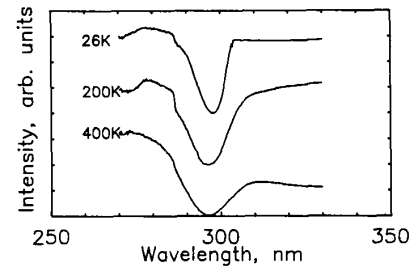Fig. 4.   Transmission spectra of LaP_5O_{14}:0.05% Ce for different temperatures (26 K, 200 K and, 400 K). Spectra were corrected for the spectral distribution of the light from the Xe lamp. Baselines for different spectra are shifted.

determined by the f–d transition on Ce ion and that there is no contribution from other impurities.

In Fig. 5 the decay of luminescence is shown for CeP_5O_{14} under ionizing excitation from a Ru/Rh radioactive source (β and γ radiation) at room temperature. Within experimental error decays under ionizing and optical (below bandgap) excitations are the same and, to a good approximation, single-exponential. As reported earlier, [13], decay times of concentrated Ce-pentaphosphates show a very peculiar increase with temperature. In Fig. 6 we show temperature dependencies of decay times (CeP_5O_{14} and LaP_5O_{14}:Ce) and total light output under optical excitation. It is interesting to note that the effect is absent for LaP_5O_{14}:Ce (decay time is about 20 ns from 24 to 550 K) and, also, that the light output for CeP_5O_{14} practically does not change with temperature. It is remarkable that there is absolutely no indication of thermal quenching up to 550 K for both CeP_5O_{14} and LaP_5O_{14}:Ce.

In Fig. 7 we present energy spectra for BGO and CeP_5O_{14} under [207]Bi excitation. The light output of Ce-pentaphosphate is about half that for BGO. Assuming that for BGO it is 8200 photons/MeV, [18], [19], we estimate the light output of CeP_5O_{14} to be close to 4000 photons/MeV. As shown in Fig. 8 the light output of Ce-La-pentaphosphate is, to a good approximation, a linear function of Ce fraction. The implications of these results for the model of scintillation process in a Ce-pentaphosphate will be discussed in the next section.

IV. DISCUSSION

We divide the discussion of our results into two subsections, dealing respectively with photoluminescence of Ce-La-pentaphosphates under optical excitation and scintillation under ionizing excitation.

A. Photoluminescence

Except for some peculiarities in luminescence spectra for high temperatures and temperature dependencies of decay times, the spectroscopy of concentrated Ce-pentaphosphates is relatively simple. This is evidenced by experimental results presented in the previous section, which are entirely determined by properties of the Ce^{3+} ion. Low temperature luminescence, luminescence excitation
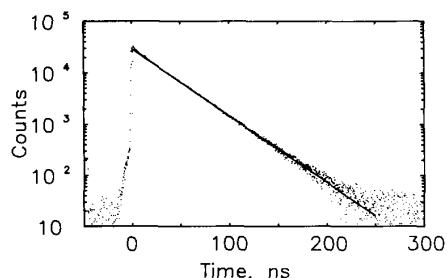
Fig. 5. Scintillation decay for CeP_5O_{14} under Ru/Rh ionizing radiation excitation. Sharp peak at zero time is due to Cerenkov radiation. Solid line shows the best, single exponential fit to experimental points with decay time of 33. 4 nsec.
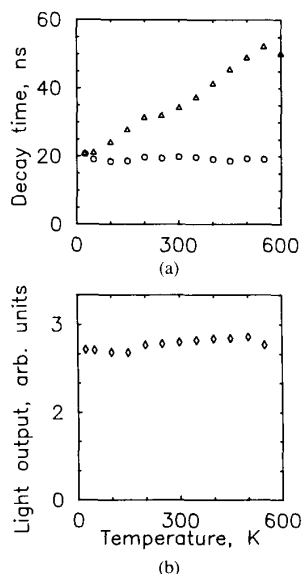

(a)

(b)

Fig. 6. Temperature dependencies of decay times and the total light output under optical excitation. a) o – LaP_5O_{14}:Ce; Δ – CeP_5O_{14}. Decay times for short and long wavelength emission bands are very close. Excitation wavelength 270 nm. b) Experimental points are for CeP_5O_{14}; excitation wavelength 250 nm.

Fig. 7. Energy spectra of BGO and CeP_5O_{14} under ^{207}Bi excitation. Compare BGO photopeak at channel 870 and Ultraphosphate at channel 450.

Fig. 8. Total, room temperature light output of Ce-La-pentaphosphates, $Ce_xLa_{1-x}P_5O_{14}$, as a function of Ce fraction x.

and transmission spectra can easily be explained on the basis of electronic structure of the Ce^{3+} ion which, interestingly enough, combines properties of a transition metal and a rare earth ion. There is no evidence of nonradiative quenching (with increasing temperature or concentration) and, at least for low temperatures, the measured decay time is close to the radiative lifetime of the Ce^{3+} ion. The radiative lifetime, τ (in ns), for any transition can be calculated using [20]:

$$\tau = 1.5 \cdot 10^{-5} \cdot \frac{\lambda^2}{f \cdot \frac{1}{9}(n^2 + 2)^2 \cdot n} \tag{1}$$

where if is the oscillator strength of the transition, λ designates luminescence wavelength in nanometers and n strands for refractive index. There are in the literature
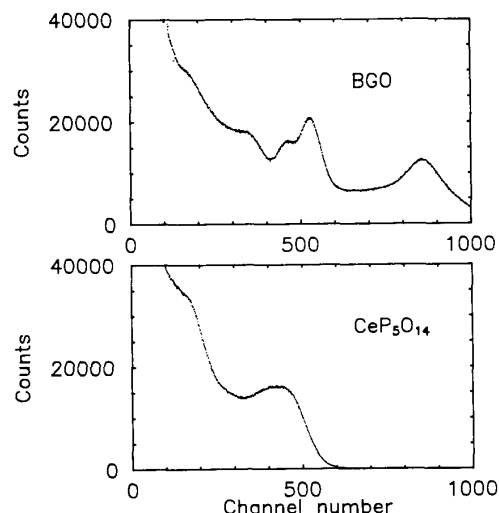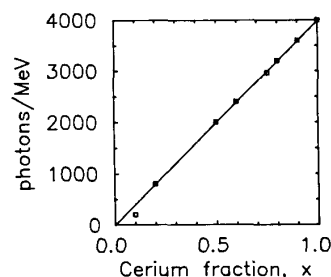
several, slightly different definitions of the oscillator strength. If we use the quantity $P = 0.02$ listed in [21], then the proper value of f to be used in (1) is 0.014. Taking λ as 320 nm and n equal to 1.6 we calculate τ from (1) to be 30 ns, in reasonable agreement with the measured decay time for Ce-doped La-pentaphosphate and the low temperature decay time of concentrated Ce-pentaphosphate, see Fig. 6.

The pecularities of Ce-pentaphosphate spectroscopy, differentiating this material from the lightly Ce-doped La-pentaphosphate, can be summarized as follows:

1) Although the total light output of both crystals does not change with temperature the concentrated material shows pronounced changes in relative intensities between short and long wavelength emissions up to 300 K. For higher temperatures there is only one (long wavelength) band left, for which distinct changes in band shape and position can now be observed, as shown by Fig. 1;

2) Decay times for Ce-pentaphosphate increase with temperature while for Ce-doped La-pentaphosphate they remain constant up to 550 K.

Thermally stimulated increases of decay times have been observed and explained in different ways before, [22]–[24]. More detailed discussion of some of those models is given by Berman, [14]. Generally, however, they do not explain the correlation between changes in luminescence spectra and decay times.

We propose a different explanation, in which we assume that spectral redistribution, caused by increased self-absorption, is actually responsible for the increase of decay times by way of radiation trapping. Significant increases of lifetime due to self-absorption have been observed for resonance radiation in both gases and solids [25]–[27]. In the pentaphosphate the situation is different because the process occurs between the two emission/absorption bands.

We simplify our model by assuming that the excited Ce ion can emit a photon (on average after time τ, the radiative lifetime) with two different energies but equal probabilities (high energy and low energy emission bands). If the photon is emitted with higher energy, it has probability ξ of being absorbed (by a different ion) and probability $1 - \xi$ of leaving the crystal. After the photon, emitted by one ion, is absorbed by the second ion, the cycle can repeat (after 2τ) and so on. The photon emitted with lower energy is free to leave the crystal. All of those possibilities will contribute to the decay of the initially excited ion, therefore they will contribute to the rate (hence decay time) of luminescence. It is obvious that the contribution of self-absorption will lengthen the decay process. The effective decay time, τ_d, is expected to be some function of ξ, the redistribution parameter, which in our approach can be found from the luminescence spectra. The intensities of the long wavelength band, I_1, and short wavelength band, I_2, are

$$I_1 \sim \frac{1}{2} + \frac{1}{4} \cdot \xi + \frac{1}{8} \cdot \xi^2 + \cdots = \frac{1}{2 - \xi}$$

$$I_2 \sim \frac{1}{2} \cdot (1 - \xi) + \frac{1}{4} \cdot (1 - \xi) \cdot \xi + \cdots = \frac{1 - \xi}{2 - \xi}. \quad (2)$$

The decay time, τ_d, is given by the following equation:

$$(\tau_d)^{-1} = \frac{1}{2} \cdot \frac{1}{\tau} + \frac{\xi}{4} \cdot \frac{1}{2\tau} + \cdots +$$
$$+ \frac{1 - \xi}{2} \cdot \frac{1}{\tau} + \frac{(1 - \xi)\xi}{4} \cdot \frac{1}{2\tau} + \cdots \quad (3)$$

which can be used to find τ_d numerically for every value of ξ. (3) can be rewritten as

$$\frac{1}{\tau_d} = \frac{1}{\tau}\left[1 - \frac{\xi}{4} - \frac{\xi^2}{24} - \frac{\xi^3}{96} - \cdots\right]. \quad (4)$$

The ratio of intensities emitted in short and long wavelength bands can be found from (2)

$$\frac{I_2}{I_1} = 1 - \xi. \quad (5)$$

Therefore, if we know the ratio of intensities we can find the redistribution parameter ξ, (or probability of self-absorption) and then, using (4) the decay time, $\tau_d \cdot \xi$ is changing with temperature, since the overlap between absorption and emission spectra, and hence the probability of self-absorption, increases strongly with temperature. Parameters ξ for different temperatures, calculated from luminescence spectra (after decomposition into two Gaussian components and proper normalization), are presented in Table I along with decay times, calculated using (4) assuming $\tau = 20$ ns. The agreement between the experimental decay times and model calculations is reasonable up to about 250 K. Above this temperature, the short wavelength emission band is practically gone, and the edge of absorption is now beginning to distort the long wavelength emission band.

Radiation trapping plays thus an important role in determining the decay time of concentrated Ce-materials. Certainly Ce-pentaphosphates, with their unusually small Stokes shifts of only 1640 cm^{-1} are very vulnerable. Generally, Ce-materials have much larger Stokes shifts and the effect is expected to be much less conspicuous which is clearly good news for applications.

B. Scintillation

We will find it useful to review some basic concepts concerning the detection of light generated in a volume V_{exc} of a material of volume V, containing n luminescent centers per unit volume. Under γ-excitation we have to distinguish between two steps. As long as the dimensions of the sample are small compared to the attenuation length, the γ-photons create elementary excitations, (electron-hole pairs), pretty much uniformly ($V_{exc} = V$). In the next step each luminescent center collects as much as possible of the elementary excitations from V_{exc}. The total light output will be proportional to:

$$\Omega = n \cdot V_{exc} \cdot \omega, \quad (6)$$

where ω is now a "characteristic volume" specific for the given luminescence center. The characteristic volume ω has therefore the meaning of a volume from which the center is able to collect the energy deposited by a γ-photon by any means (energy transfer or any process involving charge transport or diffusion). The parameter Ω can be interpreted as a fraction of the total volume excited by the γ-photon, "controlled" by emitting centers. Therefore the efficiency of a scintillator defined as, [3]

$$\eta = n_p / n_{e-h} \quad (7)$$

where n_p is the number of scintillation photons and n_{e-h} is the number of elementary excitations (electron-hole pairs) produced in the unit volume of the scintillator can be expressed as:

$$\eta = \frac{\Omega}{V_{exc}} = n \cdot \omega. \quad (8)$$

TABLE I
REDISTRIBUTION PARAMETER ξ AND DECAY TIME
τ_D (IN NS), OF CE-PENTAPHOSPHATE FOR DIFFERENT
TEMPERATURES T (IN K), CALCULATED FROM LUMINESCENT SPECTRA,
(SEE TEXT)

T	24	50	100	150	200	250
ξ	0	.125	.401	.607	.785	.900
τ_d	20	20.7	22.5	24.4	26.5	28.2

The relations (6) and (8) hold as long as $n < n_{cr}$, where n_{cr}, the critical concentration, can be, in this case, expressed as:

$$n_{cr} = \frac{1}{\omega}. \tag{9}$$

We are free to choose an elementary volume in a crystal to be the volume per molecule of the compound. In this case, for concentrated Ce-materials $n = 1$ (and $\eta = \omega$) and the lack of any saturation, as evidenced by Fig. 8, suggests that for Ce^{3+} in pentaphosphates ω_{Ce}, expressed in the same units, must satisfy the condition:

$$\omega_{Ce} < 1, \tag{10}$$

which means that the volume controlled by one Ce ion is smaller than the elementary volume. A more precise estimate is possible if we take into account the fact that the light output of Ce-pentaphosphate is 0.49 that of BGO while efficiency of BGO, determined by its low quantum efficiency is 0.16, [18]. Therefore the efficiency of Ce-pentaphosphate is 0.078. Since quantum efficiency in pentaphosphates is 1, we have

$$\eta = \frac{\Omega}{V_{exc}} = \omega_{Ce} = 0.078. \tag{11}$$

Let us now consider the situation after the shower of fast electrons and X-rays, (due to recombination of electrons and deep holes produced by the photoeffect on deep core electrons), eventually produces a cloud of thermalized electrons and holes. If we regard holes as localized, they will reside on anions and on those cations which can produce a hole binding potential or, in other words, capable of introducing a filled localized level in the forbidden energy gap ("hole traps"). Delocalization of holes would tend to change the distribution in favor of "traps", since they provide states with lower energies. However, in many materials mobilities of holes are not very high and they are very likely to "localize" themselves by a strong lattice relaxation. It is reasonable to assume that relaxation of holes is much faster than any electron-relaxing process, [28]. Since relaxed (localized) holes create a long range Coulomb potential characterized by a large electron capture cross section, electrons will form either lattice excitations (when an electron is bound by the hole on anion) or bound excitons (when an electron is bound by the hole on a "trap"). If, as in the case of Ce-pentaphosphate, the "traps" are due to ions with some internal energy structure, the bound exciton can transfer its energy to this structure, producing an ion in the excited state. Assuming that there is no energy transfer from the lattice

excitons we expect the efficiency of Ce-pentaphosphate, CeP_5O_{14}, to be

$$\eta = \frac{\omega_{Ce}}{\omega_{Ce} + 14 \cdot \omega_O}, \tag{12}$$

where ω_{Ce} and ω_O are characteristic volumes for hole capture processes for Ce and O ions, respectively. Because of the deep location of the P bands, the phosphorous ions will play no role. Assuming that

$$\omega_{Ce} = \omega_O, \tag{13}$$

we may estimate efficiency, η, and ω_{Ce} to be 0.067, which is reasonably close to the estimate, derived directly from experimental results (0.078). The open question is what happens to remaining 90% of the energy deposited by the high energy particle. The likely explanation may be in large nonradiative rates for self-trapped lattice excitons due to their much larger lattice relaxation energies. Simply speaking, these large lattice relaxation energies would result from large changes in charge distribution between ground and excited states; in the excited state electron would be localized mostly on P or Ce ions, whereas the ground state wavefunction would be localized on O ions. For Ce bound excitons both wavefunctions are localized on the same ion and relaxation is much smaller. This problem is beyond the scope of this paper.

V. CONCLUSIONS

Ce-pentaphosphate, as a relatively simple scintillating material, presents a unique opportunity to study basic physical mechanisms of the scintillation effect in concentrated Ce-systems. We have established that spectroscopically this system is determined by the electronic structure of the Ce^{3+} ion. Some pecularities, like distortions in emission spectra and temperature increase of decay time, can be explained by radiation trapping, caused by a small Stokes shift and consequent overlap between absorption and emission. Under ionizing excitation the process responsible for delivering the energy to Ce ions starts when Ce as well as oxygen ions become ionized by X-rays and fast electrons coming from the shower generated by a high energy particle. Ionization of phosphorus ions will play no role because holes located on them will be immediately filled. The capture of electrons, formation of excitons, transfer of excitonic energy to the cerium $d-f$ electronic structure, relaxation and emission of photons complete the process. The distribution of holes between oxygen and cerium ions, established in the first step of this process, determines the share of the total energy intercepted by Ce ions, hence the efficiency of the scintillator material. It seems that the large fraction of total energy used to ionize oxygen, is lost to nonradiative processes.

The most important conclusion of this paper is that the efficiency of concentrated Ce-materials is governed by the Ce/anions ratio. In materials where the number of Ce ions per anion is higher than in Ce-pentaphosphate we can expect the efficiency to be significantly improved.

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