

# Development of inorganic scintillators

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## Abstract

A review is presented on different lines of scintillator research in relation to the different applications and requirements. Applications of inorganic scintillators are found in very different fields. Often the scintillator requirements are correspondingly different. In a number of cases there is a strong interest in scintillators with a fast response and a high light yield. E.g. for positron emission tomography the materials  $\text{Lu}_2\text{SiO}_5(\text{Ce})$  and  $\text{LuAlO}_3(\text{Ce})$ , both with a relatively high density, are studied. For small gamma cameras with a good position resolution, e.g. for emission mammography, the less dense material  $\text{YAlO}_3(\text{Ce})$  is of interest. For thermal-neutron detection neutron-gamma ray discrimination is occasionally very important. Here  $\text{BaLiF}_3$ , with both a very fast and a relatively slow luminescence component, and  $\text{BaLiF}_3(\text{Ce})$  are interesting new materials. And finally, for high-energy physics the crucial combination is a fast response and a short radiation length. Here all attention is focussed on the dense, high-atomic-number material  $\text{PbWO}_4$ , a scintillator which has a very low light yield.

## 1. Introduction

Inorganic scintillators play an important role in many sectors of fundamental research, in almost all medical diagnostic imaging modalities that use X-rays or gamma rays, and in many industrial measuring systems [1,2]. This is explained by the comparatively good detection efficiency of inorganic scintillators for hard radiation. In the different applications the detector requirements differ considerably and consequently the scintillator requirements also differ. Yet, basic requirements for many applications are a fast response (10–100 ns), a high light yield ( $\geq 25000$  photons per MeV), a high density  $\rho$ , and a high atomic number  $Z$  (photoelectric absorption  $\propto \rho Z^3$ ). In addition, there are many other requirements, of which the possibility to grow large crystals ( $> 1 \text{ dm}^3$ ) and a modest price per  $\text{cm}^3$  are very important.

In studying the specifications of the more traditional commercially available inorganic scintillators [1], one arrives at the conclusion that there are roughly two groups of scintillators: on the one hand, we have  $\text{NaI}(\text{Ti})$ ,  $\text{CsI}(\text{Ti})$ ,  $\text{CsI}(\text{Na})$ ,  $\text{BGO}$ , and  $\text{CdWO}_4$ , with light yields  $\geq 10^4$  photons per MeV, but decay times of  $> 200$  ns. On the other hand, we have  $\text{BaF}_2$ ,  $\text{CsF}$ ,  $\text{CeF}_3$ , and  $\text{CsI}$ , with decay times in the range  $\sim 1$ –30 ns, but light yields  $\leq 2000$  photons per MeV.  $\text{GSO}(\text{Ce})$ , with a 60 ns decay time and 8000 photons per MeV, is an intermediate case. A scintillator that meets the basic requirements of both

a fast response and a high light yield is not found, not to mention high density and high  $Z$ . In consequence of this a large number of groups is performing scintillator research.

There are essentially four mechanisms of interest for fast scintillators. (a) A fast response is obtained in a crystal like  $\text{CsI}$ . The scintillation mechanism is not exactly known. There are several models [3,4]. The light yield is low and this case will not be considered any further. (b) Allowed  $5d$ – $4f$  transitions in lanthanide ions [5,6]. This is the most promising mechanism. Some developments will be discussed below. (c) Cross luminescence, also called core-valence luminescence [7,8]. It gives the fastest response. However, the light yield is low. A new application in neutron detection will be discussed. (d) Quenched luminescence can also have a short decay time, however, at the cost of the light yield, which can become very low. A striking example is  $\text{PbWO}_4$  which, in spite of the very low light yield, has recently been selected for application in the electromagnetic calorimeter of the CMS experiment at CERN [9,10]. We will report on this case as well.

Before turning to the discussion of the development of fast scintillators, some remarks are in order on the use of the traditional scintillators  $\text{NaI}(\text{Ti})$  and  $\text{CsI}(\text{Ti})$ . The former are used on a large scale in gamma cameras [11]. Recently very large,  $\sim 0.5 \times 1.0 \text{ m}^2$ , cameras have become commercially available (CRISMATEC). Thus, really large-area position-sensitive detection of radiation

in the range  $\sim 100$ – $400$  keV has become possible. CsI(Tl) is used for X-ray imaging in medical diagnostics. Often for the position-sensitive detection of the scintillation light an image intensifier is used in combination with CCD readout [11]. Energies are  $< 100$  keV. A very interesting development will be the replacement of the bulky image intensifier system by a thin, large area,  $40 \times 40$  cm<sup>2</sup>,  $2000 \times 2000$  pixel, amorphous silicon sensor [12,13]. CsI(Tl) can be evaporated directly onto this sensor. Both developments are of great interest for other fields as well.

## 2. 5d–4f transitions in lanthanides

The fast, allowed, 5d–4f transition in lanthanide ions,  $\text{Ln}^{3+}$ , offers an attractive possibility to create new scintillators with a fast response [5,6]. The basic idea is that the  $\text{Ln}^{3+}$  ion is placed in a host crystal with such a configuration that both the 4f and the lowest 5d levels are in the gap between the valence and the conduction band (energy difference  $E_g$ ). Interaction of radiation with the crystal will result in a large number of thermalized electron–hole pairs. An energy per pair of  $\sim 2$ – $3E_g$  is required. An electron and a hole can move independently or as a pair (exciton) towards the  $\text{Ln}^{3+}$  ion and transfer

their excitation energy to the dopant. It is also possible that energy is transferred to the  $\text{Ln}^{3+}$  ion from an emission centre of the host by multipole–multipole interaction. Both cases can result in an excited 5d state and subsequently fast emission of a photon, provided that the 5d–4f transition is not quenched. The crucial question is how efficiently the transfer of the excitation energy to the  $\text{Ln}^{3+}$  ion can be realized. Many mechanisms can prevent this, e.g. trapping of electrons and holes in the regular lattice or by defects, followed by luminescence with a very long decay time, or radiationless recombination. We will focus our attention on the most promising case,  $\text{Ce}^{3+}$ , which has fluorescence decay time  $\tau$  of  $\sim 30$  ns. For  $\text{Pr}^{3+}$  and  $\text{Nd}^{3+}$ , see Ref. [6].

In Table 1 results are presented on a large number of  $\text{Ce}^{3+}$ -doped inorganic scintillators. The  $\text{Ce}^{3+}$  4f state is well shielded and therefore hardly affected by the host lattice. An electron in the 5d state, on the contrary, experiences a strong interaction. The average 5d–4f energy difference is 6.0 eV for the free ion,  $\sim 5.6$  eV for  $\text{Ce}^{3+}$  in fluorides,  $\sim 4.7$  eV in chlorides, and  $\sim 4.7$  eV in oxides. Furthermore, there is a splitting of the 5d levels by the crystal field. A crystal field with a low point symmetry at the  $\text{Ce}^{3+}$  site will split the 5d level into 5 sublevels. The stronger the crystal field and the lower the symmetry, the stronger the splitting is and the more

Table 1  
Compilation of Ce-doped scintillator data at 293 K

	Ce conc. (mol%)	$\lambda$ (nm)	Light yield (photons/MeV)	$\tau$ (ns)	Density (g/cm <sup>3</sup> )	$X_0$ (cm)
CaF <sub>2</sub>	0.1	320,335	2400	350	3.2	
	5	370	550	50		
BaF <sub>2</sub>	0.2	310,325	7000	60,600	4.9	2.0
	4.5	340,365	2200	50		
LaF <sub>3</sub>	10	300,350	2000	5,30	5.9	
CeF <sub>3</sub>	100	310,340	$\sim 4000$	5,30	6.2	1.7
CsY <sub>2</sub> F <sub>7</sub>	5	335	1400	32		
CsGd <sub>2</sub> F <sub>7</sub>	20	335	$\sim 1600/5800$	15–25/ $\sim 3000$	5.5	
K <sub>2</sub> LaCl <sub>5</sub>	10	370	32000/18000	140–10000/long	2.9	
Cs <sub>2</sub> NaLaCl <sub>6</sub>	$\sim 1$	$\sim 390$	3000/3000/3000	80/600/ $> 4500$	3.2	
Cs <sub>2</sub> NaCeCl <sub>6</sub>		400	6000/3000/3000	60/400/ $> 1000$	3.2	
Cs <sub>2</sub> NaLuCl <sub>6</sub>	1	$\sim 390$	1500/2000/20000	80/300/ $> 4500$	3.7	
YAlO <sub>3</sub> (YAP)	0.2	360	15000/1000	$\sim 30/10000$	5.6	2.9
GdAlO <sub>3</sub> (GAP)	1	335,358	$\sim 700/5300/3000$	30/180/long	7.5	1.5
LuAlO <sub>3</sub> (LuAP)	$< 1$	$\sim 365$	$\sim 12000/12000$	$\sim 17.5/ > 10000$	8.3	1.1
Y <sub>3</sub> Al <sub>5</sub> O <sub>12</sub> (YAG)	1	550	9000	65	4.6	
Gd <sub>3</sub> Sc <sub>2</sub> Al <sub>3</sub> O <sub>12</sub>	3	560	$\sim 2000$	$< 1000$	5.6	
Lu <sub>3</sub> Al <sub>5</sub> O <sub>12</sub>	1	300,510	3000/11000	$\sim 100/long$	6.7	
Y <sub>2</sub> SiO <sub>5</sub> (YSO)		420	10000	37,82	4.5	
Gd <sub>2</sub> SiO <sub>5</sub> (GSO)	0.5	440	8000/1000	60/600	6.7	1.4
Lu <sub>2</sub> SiO <sub>5</sub> (LSO)	0.1	420	25000	40	7.4	1.1
LuPO <sub>4</sub>		350	18000	24	6.5	
GS1	a	390	1500	70,1000	2.5	

<sup>a</sup> GS1 = (SiO<sub>2</sub>)<sub>0.55</sub>(MgO)<sub>0.24</sub>(Al<sub>2</sub>O<sub>3</sub>)<sub>0.11</sub>(Li<sub>2</sub>O)<sub>0.06</sub>(Ce<sub>2</sub>O<sub>3</sub>)<sub>0.04</sub> glass. For references see Ref. [6].

the lowest 5d level is pushed towards the 4f ground state. The 5d–4f luminescence being due to transitions between the lowest 5d state and the 4f ground state, the emission wavelengths change correspondingly. This explains the different emission wavelengths in Table 1.

Fluorides, oxides and chlorides have a bandgap  $E_g$  of, respectively,  $\sim 8$ –11,  $\sim 6$ –8, and  $\sim 5$ –7 eV.  $\text{Ce}^{3+}$  “fits” in all of these. We mentioned already that the energy required to produce a thermalized electron–hole pair is  $\sim 2$ – $3E_g$ . Consequently, the number of electron–hole pairs  $n_{e-h}$  produced per unit of absorbed energy (MeV) is highest if  $E_g$  is smallest. Then, considering that the light yield is proportional to  $n_{e-h}$ ,  $\text{Ce}^{3+}$  in an oxide or chloride host seems most promising for our purpose.

It is relatively easy to lose energy, and therefore difficult to transfer the absorbed radiation energy efficiently to the  $\text{Ce}^{3+}$  centres. This is reflected by the data in Table 1. For most cases the light yield (number of photons/MeV) is low, i.e. much lower than could be expected on the ground of the  $n_{e-h}$  number, and often the response time is long. All fluorides have a low light yield; the chlorides and oxides have on the average a much higher yield. Apparently, if the  $\text{Ce}^{3+}$  5d–4f energy difference fits better inside the gap, it is more likely that electron and hole are efficiently transported to the  $\text{Ce}^{3+}$  ion for energy transfer. In connection with this one should also notice the relatively small cerium concentrations of 0.2,  $< 1$ , and 0.1 mol% in, respectively, YAP, LuAP, and LSO. Direct cerium excitation by radiation is negligible at these concentrations. There seems to be, fast, relatively efficient transport to the  $\text{Ce}^{3+}$  centres.

In Table 1 we included some new results on chlorides ( $\text{Cs}_2\text{NaLnCl}_6$ ,  $\text{Ln} = \text{La}(\text{Ce}), \text{Ce}, \text{Lu}(\text{Ce})$ ) [14]. The work was initiated because of the high  $\text{K}_2\text{LaCl}_5(\text{Ce})$  light yield and the excellent energy resolution that could be obtained with this material (5% FWHM at 662 keV) [15]. In general, for chlorides,  $\rho \leq 4 \text{ g/cm}^3$ , so for a high-density  $\text{Ce}^{3+}$  scintillator a chloride is not the obvious material. Yet, for special applications, lower densities can be of interest. Unfortunately, only a small fraction of the luminescence intensity of these new materials is emitted rapidly. Research on chlorides will be continued.

An oxide scintillator that is increasingly being used is YAP(Ce). It is particularly of interest for low-energy gamma cameras; see e.g. Ref. [16]. YAP(Ce) is commercially available from Preciosa Crytur.

A field that is presently drawing much attention is positron emission tomography (PET) [2]. Scintillator requirements are (a) a very good 511 keV detection efficiency for efficient coincidence measurements and to reduce parallax, (b) a good energy resolution to reduce events resulting from Compton scattering in the body, and (c) a good time resolution to reduce random coincidences and dead time, and to use time-of-flight information. Two of the scintillators in Table 1 are of interest: LSO(Ce) and LuAP(Ce).

LSO(Ce) was discovered about 6 years ago [17] and much work has been carried out on this material [18–22]. Recently, Siemens-CTI has acquired patent rights. It is difficult to produce the LSO(Ce) scintillator in large-size crystals with a homogeneous light production, and furthermore, the light yield is strongly non-proportional below 100 keV. Consequently, the energy resolution is poor. Although 7.5% FWHM at 662 keV has been realized, a resolution in the range 10–20% is rather normal for large crystals. Other problems are a strong afterglow and an intrinsic count rate of  $300 \text{ s}^{-1} \text{ cm}^{-3}$  due to  $^{176}\text{Lu}$ . However, in PET the latter does not pose a problem as coincidences are recorded.

LuAP(Ce) has been introduced more recently [23]. The development has been hampered by the fact that one gets easily  $\text{Lu}_3\text{Al}_5\text{O}_{12}(\text{Ce})$  instead of  $\text{LuAlO}_3(\text{Ce})$ , or a mixture of both. A lot of work has been carried out since the introduction [24–29]. This material has a higher density and a shorter lifetime for the fast luminescence than LSO(Ce). However, the light yield of the fast component is only about 50% of that of LSO(Ce) and it has a slow component as well; see Fig. 1. At present the main problem is the strong self-absorption, which is independent of the Ce concentration. This applies to crystals grown by the Czochralski method [27,28] as well as those grown by the Bridgman method [29]. The problem is not yet understood. Analogous to LSO(Ce), LuAP(Ce) has an intrinsic count rate of  $\sim 300 \text{ s}^{-1} \text{ cm}^{-3}$  and it has an afterglow.

Clearly, a lot of work has to be done on LSO(Ce) and LuAP(Ce) before we can decide how competitive they are with BGO, the scintillator most frequently used for PET at the moment. In Table 2 we summarize the most important parameters.

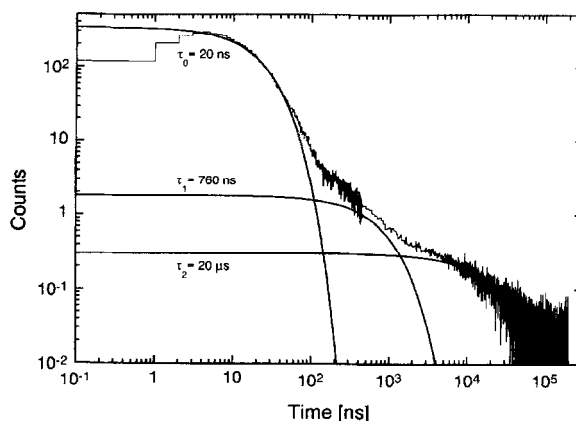


Fig. 1. Luminescence decay spectra of an  $\text{LuAlO}_3(\text{Ce})$  crystal, 1 mm thick, Ce concentration 0.75 mol%. The response is dominated by the 20 ns and the 20  $\mu\text{s}$  component, which have about equal intensity.

A serious disadvantage of LSO(Ce) and LuAP(Ce) is the high price of  $\sim \$100 \text{ cm}^{-3}$ , due to Lu. Application in a large PET system will easily bring costs to the  $\$10^6$  level.

### 3. Core-valence luminescence

Core-valence luminescence (CVL) results from the production of a hole in the outer core band of an ionic crystal [7,8]. This hole is filled very fast by an electron from the valence band ( $\tau \approx 1 \text{ ns}$ ). If the energy difference is less than  $E_g$ , the process involves the emission of a photon, usually in the UV region. Light yields are of the order of 1000 photons per MeV of absorbed radiation energy. In general, other luminescence processes occur as well. The best-known CVL case is that of  $\text{BaF}_2$ . In addition to CVL it has self-trapped-exciton (STE) luminescence.

Table 2

Comparison of scintillators for PET application

	BGO	LSO(Ce)	LuAP(Ce)
Light yield (ph/MeV)	9000	25 000	12 000/12 000
Max. emission (nm)	480	420	365
Decay time (ns)	300	40	18/long
Density ( $\text{g/cm}^3$ )	7.13	7.4	8.3
Mean free path			
511 keV (cm)	1.06	1.13	1.05
Photofraction (%)	41	33	30

At present feasibility studies for the construction of a high-intensity European spallation source (ESS) are in progress. For thermal neutron detection at this facility, large-area position-sensitive detectors with a time response in the 10–100 ns range will be needed. The CVL mechanism offers an interesting possibility for this purpose.

At present  $\text{LiI}(\text{Eu})$  crystals, Li containing glass, and  $^6\text{Li}$  admixed  $\text{ZnS}(\text{Ag})$  powder scintillators are used for thermal neutron detection. Detection is based on the reaction  $^6\text{Li} + n \rightarrow ^3\text{H} + ^4\text{He}$ . The two charged particles, which have a total kinetic energy of 4.8 MeV, produce the scintillation light in the scintillator. The use of relatively low Z materials is important to minimize the sensitivity for the gamma ray background, which is generally present at facilities where thermal neutrons are produced (reactor or spallation source).

A CVL scintillator which contains  $^6\text{Li}$  is  $\text{BaLiF}_3$ . Analogous to  $\text{BaF}_2$  this material shows both CVL and STE luminescence. The time response is shown in Fig. 2(b): a 0.8 ns CVL component and an 8  $\mu\text{s}$  STE component. An interesting aspect of CVL is that it is fully quenched if excitation occurs by heavy, charged, particles such as  $^3\text{H}$  and  $^4\text{He}$ . Consequently, upon interaction with thermal neutrons we observe only the STE luminescence, while interaction with gamma rays results in both CVL and STE luminescence. Then, by means of two discriminators (one “low level” that gives a trigger independent of the presence of CVL, and one “high level” that triggers only if CVL is present) and some NIM logic instrumentation, we can easily veto the gamma-radiation events. In Fig. 3 we show the power of this method. With the veto “on” we

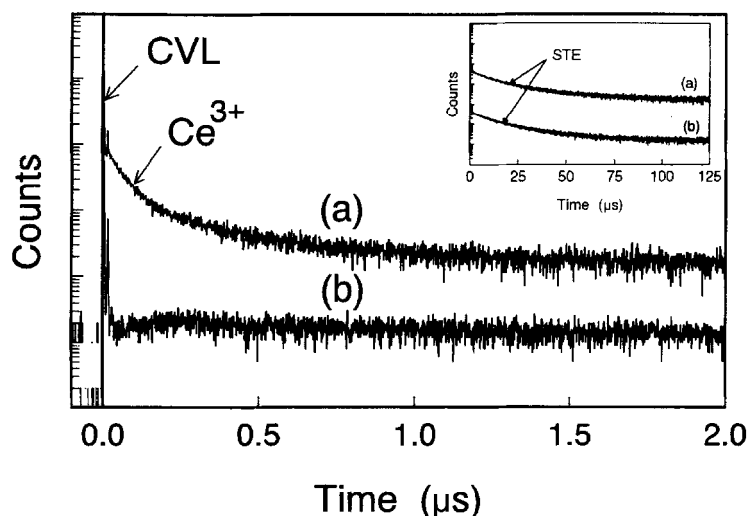


Fig. 2. Luminescence decay spectra of (a)  $\text{BaLiF}_3(\text{Ce})$  and (b)  $\text{BaLiF}_3$  under gamma-ray excitation. The 0.8 ns CVL shows up as the very intense narrow peak on the left (the second peak is an artifact). The 8  $\mu\text{s}$  STE luminescence is also shown on a longer time scale in the inset. Ce doping clearly results in a third component with a fast response, well below 100 ns.

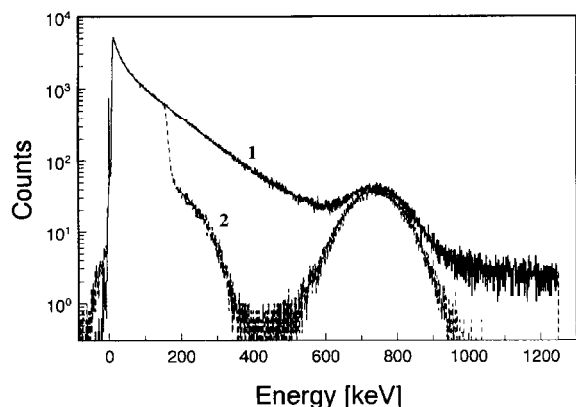


Fig. 3. Neutron spectrum of a Pu-Be source, recorded with BaLiF<sub>3</sub>. Spectrum 1 includes the gamma-ray background. In spectrum 2 gamma rays were vetoed using the CVL response. The neutron peak at 750 keV is well resolved.

resolve the neutron peak from a Pu-Be source, surrounded by paraffin wax and 6 cm of lead, almost free from background. A gamma-ray suppression factor of  $>10^6$  could be obtained [30].

Obviously, the thus obtained thermal-neutron detector is not fast, the STE-response time being 8  $\mu$ s. The remedy is sought in doping with Ce<sup>3+</sup> ions [31]. In Fig. 2(a) we see the time response of BaLiF<sub>3</sub>(Ce). There is a clear fast Ce response. However, as demonstrated in the inset in Fig. 3, the slow STE component is still present. Increase of the Ce concentration, which is only  $\sim 0.1$  mol% in the case of Fig. 2(a), may help to reduce the slow STE component further. Work is in progress.

#### 4. PbWO<sub>4</sub> scintillator for CMS

PbWO<sub>4</sub> was recently selected to become the scintillator of the electromagnetic calorimeter of the CMS experiment at LHC, primarily because of its high density of 8.2 g/cm<sup>3</sup> and  $X_0 \approx 0.9$  cm (see e.g. Ref. [9]). Other requirements, like good mechanical properties, luminescence stability versus time and temperature, and an acceptable cost, are favourable as well. A large number of 110 000 scintillator blocks of  $\sim 2.0 \times 2.0 - 2.4 \times 2.4 \times 23$  cm<sup>3</sup> are needed, representing a volume of 13 m<sup>3</sup>.

The average response time is fast ( $\tau \approx 10$  ns), but the light yield is very low:  $\sim 150$ – $200$  photons/MeV. This is on the very edge of what is feasible. The low light yield is due to a strong quenching effect. At  $-150^\circ\text{C}$  the intensity is more than an order of magnitude higher.

There are several luminescence mechanisms. We encounter a fast blue emission, which is usually ascribed to the regular WO<sub>4</sub> group, but the Pb<sup>2+</sup> ion seems to play

a role as well. A green emission is due to the defect WO<sub>3</sub> group, possibly with an F centre nearby. Furthermore, there is a red emission due to Pb<sup>3+</sup>. The average decay time of 10 ns is a mixture of very fast ( $\sim 5$  ns) and somewhat slower (20–50 ns) decays. Furthermore, there is a weak slow component ( $> 1$   $\mu$ s). The luminescence spectrum and the decay contributions depend strongly on the starting material, the growth conditions, the thermal treatments after growth, and on doping. After painstaking crystal growth studies it seems now possible to grow crystals that meet the requirement of time response.

Another aspect that is very important is radiation hardness. The radiation tolerance should be  $> 5$  Mrad. Also with respect to this point we are close to a solution. However, some more studies have yet to be carried out.

#### 5. Conclusions

For a variety of applications new scintillators are being studied. As illustrated by the BaLiF<sub>3</sub> case, new applications continue to turn up and the interest in the introduction of new fast inorganic scintillators will continue as well. The emphasis of scintillator research will be on the 5d–4f transitions of Ce<sup>3+</sup>-doped scintillators. A high light yield is needed in the visible region and the time response should be in the 25–100 ns range. In addition to favourable specifications, the cost should be modest.

From the cases discussed it becomes clear that it takes some time before the specifications of a potential scintillator are brought in agreement with the requirements of a specific application. In particular, in the case of LSO(Ce) and PbWO<sub>4</sub>, we are confronted with a period of more than five years of research. Although this seems to be a pessimistic statement, one should realize that it also demonstrates that we are more and more able to control the basic mechanisms and the technology of inorganic scintillation crystals.

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