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PROSPECTS FOR NEW INORGANIC SCINTILLATORS*

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ABSTRACT

Although scintillation has been discovered in many inorganic crystals during the past 40 years, there is continued interest in finding compounds with improved scintillation properties. However, the testing of candidate compounds has thus far been limited to those available as crystals of suitable quality and size. We describe a method using synchrotron x-radiation for measuring the fluorescence properties of powders, which makes accessible a much larger number of compounds. We present results for 85 compounds tested by this method. Scintillation from several compounds was discovered, notably CeF_3 and PbCO_3 .

1 BACKGROUND

Since the discovery of scintillation in $\text{NaI}(\text{Tl})$ by Hofstadter in 1948 [1, 2], scintillation in many other inorganic compounds such as pure NaI , $\text{CaF}_2(\text{Eu})$, $\text{LiI}(\text{Eu})$, $\text{CsI}(\text{Tl})$, $\text{CsI}(\text{Na})$, CsF , CaWO_4 , CdWO_4 , $\text{Bi}_4\text{Ge}_3\text{O}_{12}$ (BGO), BaF_2 , Gd_2SiO_5 (GSO), and pure CsI has been discovered and is now used for a variety of applications in nuclear detection and measurement [3]. However, even when the "best" scintillator is used, its effectiveness for the application is often limited by detection efficiency, light yield, speed of response, ruggedness, or cost. A notable example is gamma ray detection above 400 keV, where all known scintillators are far from ideal. While $\text{NaI}(\text{Tl})$ has the best energy resolution, BGO has the best stopping power, and BaF_2 has the best timing resolution, each is lacking in the other properties. Ideally, we would design a gamma-ray scintillator combining the best of all three properties using theoretical considerations or at least empirical rules. Until recently, however, all known scintillation has been found experimentally by testing compounds available as crystals of suitable quality and size.

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2 A NEW SEARCH METHOD

Our goal has been to expand the search for scintillation to include the thousands of compounds that are not readily available as crystals. We postulated that by using the high intensity and short time duration (< 1 ns) of recently available x-ray synchrotron sources, a scintillator in powdered form would exhibit intense enough fluorescent emissions to be detectable with a phototube (Figure 1) and that useful measurements of fluorescence lifetime, wavelength, and intensity could be made. The costly and time-consuming step of growing crystals would then be limited to the small fraction of compounds that looked promising in powdered form. When a crystal sample is shown to have fluorescent emissions whose intensity depends on the amount of ionizing energy deposited in the bulk of the crystal, then the compound is established as a scintillator.

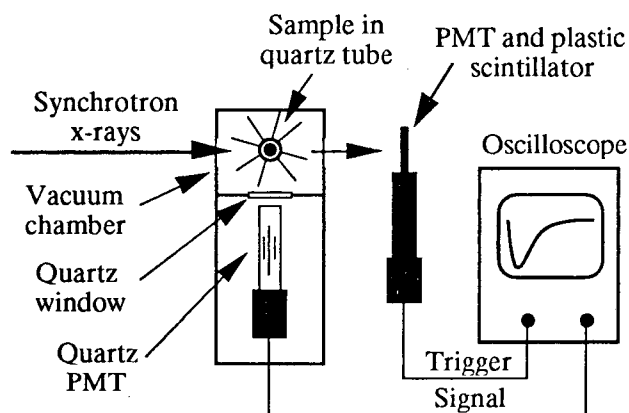


Figure 1. Experimental method for using synchrotron x-rays to measure the fluorescence intensity and lifetime of compounds in powdered form.

It is difficult to use beta- or gamma-ray interactions to investigate the fluorescence properties of powders because only light from a thin outer layer can escape and reach the photodetector. Powders consist of many tiny crystals and almost all the light produced at a depth

much greater than the crystal size is scattered many times and absorbed.

In preliminary measurements at the Stanford Synchrotron Radiation Laboratory (SSRL) in November and December of 1988, we used a 5 to 20 keV white x-ray beam to screen 60 compounds and crystals, and found that CeF_3 has a fluorescence lifetime less than 50 ns. Fortunately, Optovac, Inc. (North Brookfield, MA) had previously grown excellent crystals for another purpose, and these readily exhibited a gamma-ray photopeak when coupled to a phototube [4, 5].

In January of 1989 we studied 85 powdered compounds using a 22 keV monochromatic x-ray beam at the National Synchrotron Light Source (NSLS) at the Brookhaven National Laboratory. These included 14 known scintillators and as many heavy-atom, dense ($> 5 \text{ gm/cm}^3$) compounds as could readily be purchased (all powders tested at SSRL were also tested at NSLS). Most were white, but a number of colored powders were also included.

Approximately 0.2 ml of each powder was placed in a cylindrical suprasil "quartz" vial 5 mm in diameter with 0.37 mm wall thickness, and the tube was sealed with a plastic cap provided by the manufacturer for that purpose. A 20 keV x-ray beam is not significantly attenuated by the wall of the tube (attenuation length 1.2 mm).

The vial was placed in the pulsed synchrotron x-ray beam and a Hamamatsu R2059 phototube with a "quartz" (fused silica) window was used to detect any fluorescent emissions. The x-ray beam was collimated to 3 x 10 mm, aimed so that a portion missed the edge of the tube and was detected by a plastic scintillator and phototube detector which provided a trigger pulse [6] for the oscilloscope and the fluorescent lifetime measurements described in the next section.

3 RESULTS

3.1 Total fluorescent output measurements

While differences in light collection efficiency among the powdered samples prevented us from measuring the total fluorescent output as accurately as with a clear crystal, an order of magnitude estimate can be made. With the 22 keV NSLS beam exciting the sample, the single photoelectron rate R was measured using a Hamamatsu quartz window R2059 phototube. This tube has a quantum efficiency that nominally peaks above 25% at 400 nm and falls to 5% at 160 nm and 580 nm. Note that this measurement does not depend on the fluorescent lifetime because it counts all photoelectrons without regard to the particular x-ray pulse that produced them.

To obtain the observed fluorescent intensity I , the rate R was (i) corrected for the average phototube background rate R_0 , (ii) adjusted for the electron current B in

the synchrotron ring during the measurement, and (iii) normalized to a value of 1000 for BGO:

$$I = 1000 \frac{B(\text{BGO})}{B(\text{sample})} \frac{[R(\text{sample}) - R_0]}{[R(\text{BGO}) - R_0]}$$

$R_0 = 40/\text{s}$ and $R(\text{BGO}) = 86,000/\text{s}$, which was much less than the beam repetition rate of 1.8 MHz. Due to variations in R_0 , we felt that only values of R above $2R_0$ were reliable indications of fluorescent emissions. Thus the sensitivity of the experiment was 40/s, or 0.05% of BGO. Table 1 lists the compounds whose fluorescence was above that value and Table 2 lists the compounds whose fluorescence was below that value.

The samples of compounds listed in Tables 1 and 2 had a range of purity levels and in some cases the impurities could have significantly effected the observed emissions, either by acting as fluorescent emitters or by acting to quench the fluorescence. Consequently, some of the compounds listed in Table 2 might be useful scintillators, but impurities prevented the observation of fluorescent emissions.

Because this method is primarily sensitive to fluorescent emissions from the surface of the powder, it can lead to "false positives," as some compounds absorb their own fluorescent emissions (radiation trapping) and therefore do not scintillate.

3.2 Correcting the total fluorescent output for absorption

The compounds tested were in the form of powders, consisting of many tiny crystals typically 1 μm in size. Light produced at a depth that is much greater than the size of these microcrystals is scattered many times by Fresnel reflection and total internal reflection at the interfaces between the crystal surface and the surrounding air. This increases the effective path length, and reduces the linear absorption length for fluorescent photons by a large factor. As a result, photons escaping from the powder are those produced in the outermost surface layer. As we shall describe below, 20 keV x-rays interact at a typical average depth of 50 μm while only light from the outermost 10 μm was observed.

In the following analysis, we convert the observed intensity I to the intensity I_0 that would be observed if all the fluorescence were produced at the surface of the powder. The powder is modeled as having a linear absorption coefficient η (cm^{-1}) for fluorescent photons and a linear absorption coefficient μ (cm^{-1}) for x-rays. The coefficient μ is given by $\mu = \sigma\rho$, where σ is the x-ray cross section (cm^2/gm) and ρ is the density (gm/cm^3). The absorption of fluorescent photons is used rather than narrow-beam linear attenuation because for an isotropic emitter, the randomization of angle by scattering does not reduce the flux in terms of photons per second per steradian.

Table 1 Measured luminosity of powders using 22 keV synchrotron x-radiation^a

Compound	Formula	ρ (gm/cm ³)	Color of powder	Purity (%)	I	Lifetime (see note b)	μ^{-1} (mm)	I_0
Known scintillators:								
NE102A	CH	1.032	white		28	fast	28.	2480 ^c
NE102A	CH	1.032	white		28	fast	28.	2480 ^c
Pilot U	CH	1.032	white		27	fast	28.	2470 ^c
Strontium fluoride	SrF ₂	4.2	white	99.99	1180	≈200 ns	0.055	2400
Barium fluoride	BaF ₂	4.89	white	99.995	410	fast + slow	0.092	1300
Bismuth germanate	Bi ₄ Ge ₃ O ₁₂	7.1	white	99.99995	1000	≈250 ns	0.021	1000
Calcium tungstate	CaWO ₄	6.1	white	99.995	600	slow	0.038	920
Zinc tungstate	ZnWO ₄	6 ^e	white	98	370	slow	0.036	540
Cerium fluoride	CeF ₃	6.2	white	99.99	59	fast	0.069	150
Cadmium fluoride	CdF ₂	6.6	white	99.99	35	fast ^d	0.110	130
Zinc oxide	ZnO	5.6	white	99.999	49	slow	0.059	110
Europium oxide	Eu ₂ O ₃	7.4	white	99.99	50	slow	0.038	77
Aluminum oxide	Al ₂ O ₃	4.0	white	99.99	1.3		1.24	52
Germanium oxide	GeO ₂	6.2	white	99.999	1.4	slow	0.056	2.9
Compounds whose x-ray excited fluorescent lifetimes were not previously known:								
Lutetium fluoride	LuF ₃	8.3	white	99.99	170	slow	0.028	210
Strontium tungstate	SrWO ₄	6.2	white	98	96	slow	0.031	125
Hafnium oxide	HfO ₂	9.7	white	99.9	111	slow	0.020	110
Lead carbonate (basic)	2(PbCO ₃)• Pb(OH) ₂	6.1	white	99.999	73	fast ^d	0.024	80
Europium fluoride	EuF ₃	6 ^e	white	99+	22	slow	0.051	43
Hafnium fluoride	HfF ₄	7.1	white	99.9	27	fast + slow ^d	0.034	37
Lead chloride	PbCl ₂	5.9	white	99.999	24	fast ^d	0.026	28
Lead tungstate	PbWO ₄	8.2	white	99.998	39	fast ^d	0.019	36
Lutetium oxide	Lu ₂ O ₃	9.4	white	99.9	18	fast + slow	0.021	18
Hafnium bromide	HfBr ₄	6 ^e	white	99.7	14	fast + slow	0.031	18
Zinc selenide	ZnSe	5.4	yellow	99.99	8.6	slow	0.043	15
Lead zirconate	PbZrO ₃	7.0	white	99.99	10.3	slow	0.021	10
Hafnium chloride	HfCl ₄	6 ^e	white	98	5.4	slow	0.046	9.6
Zinc fluoride	ZnF ₂	5.0	white	99	2.6	fast	0.083	7.7
Lanthanum oxide	La ₂ O ₃	6.5	white	99.99	2.8	fast	0.060	6.2
Ytterbium oxide	Yb ₂ O ₃	9.2	white	99.9	5.6	fast ^d	0.023	5.8
Erbium fluoride	ErF ₃	7.8	lt. pink	99.99	3.2		0.034	4.5
Lead bromide	PbBr ₂	6.7	white	99.999	4.3	fast	0.021	4.3
Bismuth sodium iodide	see note f	6 ^e	purple	see note f	2.4	slow	0.041	3.9
Lead perchlorate	Pb(ClO ₄) ₂ • 3H ₂ O	2.8	white	99	0.8	fast	0.091	2.6
Lead selenate	PbSeO ₄	6.4	white	99.9	1.6	fast	0.026	1.8
Bismuth borate	BiBO ₃	6 ^e	white	99	1.5	fast	0.025	1.7
Silver iodide	AgI	6.0	yellow	99.9	0.6		0.081	1.7
Ytterbium fluoride	YbF ₃	8.2	white	99.99	1.3	slow	0.030	1.6
Bismuth fluoride	BiF ₃	5.3	white	99.999	1.3	fast	0.018	1.1
Lead fluoride	PbF ₂	8.2	white	99.99	0.9	fast	0.017	0.8

^aIn the 160- 580 nm wavelength range and relative to BGO = 1000.

^bFast components < 50 ns, slow components > 1 μ s.

^cOptical attenuation length $\eta^{-1} = 0.3$ mm in plastic. For all other compounds, $\eta^{-1} = 0.01$ mm.

^dSee Table 3 for lifetime values fitted to measured coincident delay curves.

^eDensity unavailable- value shown was assigned solely to permit calculation of I_0 .

^fFormula and purity unavailable from supplier.

Table 2 Powders exhibiting no detectable fluorescence (< 0.05% of BGO)

Compound name	Formula	Color
Bismuth acetate	Bi(C ₂ H ₃ O ₂) ₃	white
Bismuth antimonide	BiSb	silver
Bismuth bromide	BiBr ₃	yellow
Bismuth chloride	BiCl ₃	white
Bismuth hydroxide	Bi(OH) ₃	white
Bismuth molybdate	Bi ₂ (MoO ₄) ₃	lt. yellow
Bismuth nitrate oxide	BiONO ₃	white
Bismuth oxide	Bi ₂ O ₃	lt. yellow
Bismuth oxybromide	BiOBr	yellow
Bismuth oxychloride	BiOCl	white
Bismuth potassium iodide	K ₄ BiI ₇	dk. purple
Bismuth stannate	Bi ₂ (SnO ₃) ₃	white
Bismuth subcarbonate	Bi ₂ O ₂ CO ₃	white
Bismuth sulfate	Bi ₂ (SO ₄) ₃	white
Bismuth titanate	Bi ₂ Ti ₂ O ₇	white
Bismuth tungstate	Bi ₂ (WO ₄) ₃	white
Bismuth tungstate	Bi ₂ WO ₆	gray
Boron oxide	B ₂ O ₃	white
Cadmium iodate	Cd(IO ₃) ₂	white
Cerium oxide	CeO ₂	lt. orange
Cobalt tungstate	CoWO ₄	dk. purple
Dysprosium oxide	Dy ₂ O ₃	white
Erbium oxide	Er ₂ O ₃	lt. pink
Holmium fluoride	HoF ₃	white
Lead hydroxide	Pb(OH) ₂	white
Lead niobium oxide	Pb(NbO ₃) ₂	white
Lead nitrate	Pb(NO ₃) ₂	white
Lead orthosilicate	Pb ₂ SiO ₄	white
Lead oxalate	PbC ₂ O ₄	white
Lead silicate	PbSiO ₃	white
Lead sulfite	PbSO ₃	white
Lead tantalate	PbTa ₂ O ₆	white
Lead telluride	PbTe	silver gray
Lead titanate	PbTiO ₃	lt. yellow
Lead vandanate	Pb(VO ₃) ₂	yellow
Magnesium oxide	MgO	white
Mercury bromide	HgBr ₂	white
Mercury fluoride	HgF ₂	white
Molybdenum carbide	Mo ₂ C	black
Samarium fluoride	SmF ₃	white
Samarium oxide	Sm ₂ O ₂	white
Silicon dioxide	SiO ₂	white
Silver telluride	Ag ₂ Te	silver gray
Tantalum oxide	Ta ₂ O ₅	white
Thallium fluoride	TlF	white
Zinc gallate	ZnGa ₂ O ₄	white

The relationship between I₀ and I is given by:

$$I = \frac{I_0 f \mu}{0.324} \int_0^{\infty} e^{-\mu f x} e^{-\eta f x} dx = \frac{I_0}{0.324} \left(\frac{\mu}{\mu + \eta} \right)$$

where 0.324 is the value of $\mu/(\mu+\eta)$ for BGO which preserves the normalization of 1000 for BGO, f is the packing fraction of the powder (the ratio of the powder density to the crystal density), and x is the depth in the powder. Note that the relationship between I and I_0 does not depend on f .

We used known x-ray cross sections [7] and densities to determine μ and ρ , and chose a value of η for best agreement between the intensity of the known scintillators and their accepted values relative to BGO. Although η undoubtedly varies from sample to sample, we used the value $\eta^{-1} = 0.01$ mm for all the inorganic powders and the agreement was within a factor of three for BaF₂, CeF₃, and CaWO₄. The plastic scintillators required a value $\eta^{-1} = 0.3$ mm since they were in the form of small shavings and more optically transparent than the inorganic powders.

We did not correct I_0 for the quantum efficiency of the phototube because, in most cases, the emission wavelength was not measured.

3.3 Fluorescent lifetimes and wavelengths

For those compounds that exhibited measurable fluorescence, the lifetime was estimated by observing the the phototube output pulse on an oscilloscope trace (Figure 1). Pulse integration was used to reduce shot noise and this prevented accurate estimates of fluorescent lifetime components shorter than 50 ns, but their presence was readily observed. Such components are called "fast" in Table 1. The 560 ns repetition period of the x-ray beam prevented the observance of time structure for components longer than 1 μ s. These components appeared as flat "grass" on the oscilloscope and are called "slow" in Table 1.

More accurate measurements of the fluorescent lifetime were made using the delayed coincidence method [8]. A single photoelectron pulse was used to start a time-to-amplitude converter (TAC). The following x-ray burst (560 ns later) produced a trigger pulse from the plastic scintillator that stopped the TAC [9]. A sum of exponentials plus a flat background were fit to the time delay distribution. Note that components with a lifetime longer than 1 μ s show no time structure, and are included in the fit as part of the background term.

The emission spectrum was measured by placing a 0.125 meter Jarrell-Ash MonoSpec Monochromator between the sample and the quartz phototube. The single photoelectron rate was recorded as the monochromator wavelength was scanned by computer control and the data were not corrected for the phototube response.

Table 3 Measured emission wavelength, relative intensities and lifetimes of fluorescent powders

Compound	Formula	ρ (gm/cm ³)	I_0	λ (nm)	Lifetimes (ns) and intensity fractions ^a		
					fast	medium	slow
Cadmium fluoride	CdF ₂	6.6	133 ^b	420	4.8 (29%)	24 (28%)	78 (43%)
Lead carbonate	2(PbCO ₃)•Pb(OH) ₂	6.7	80	475	5.6 (24%)	25 (48%)	155 (28%)
Hafnium fluoride	HfF ₄	7.1	37	350	29 (100%) ^c		
Lead tungstate	PbWO ₄	8.2	36	490	2.5 (25%)	11 (29%)	98 (46%)
Lead chloride	PbCl ₂	5.9	28	500	2.9 (41%)	20 (32%)	179 (27%)
Ytterbium oxide	Yb ₂ O ₃	9.2	6.	350	<1 (100%)		

^aFit to measured coincidence delay curves.

^bIntensity I_0 corrected for optical absorption in powder and normalized to 1000 for BGO.

^cSmall slow component included in the fit as part of the background term.

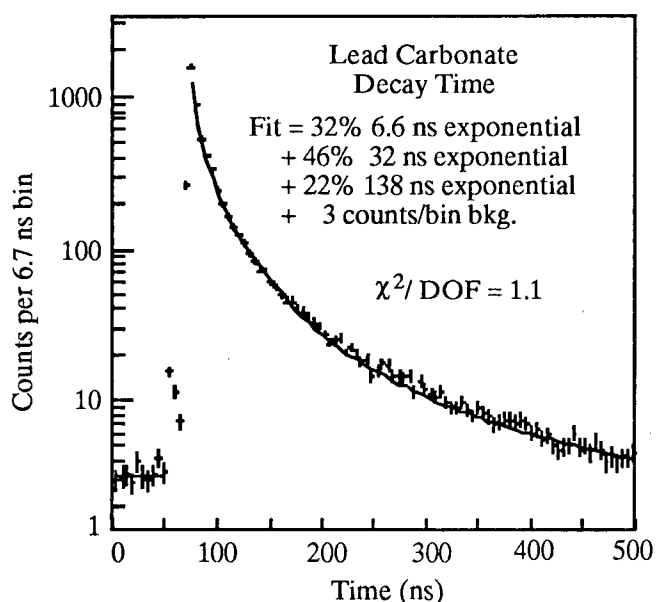


Figure 2. Exponential lifetime fit to measured fluorescent decay data from 2(PbCO₃)•Pb(OH)₂ powder. Time axis shifted by an arbitrary delay.

Table 3 lists the peak emission wavelengths and fitted lifetimes of compounds exhibiting significant emission intensity and lifetime components less than 50 ns. Figure 2 shows the fit to the fluorescent lifetime components of 2(PbCO₃)•Pb(OH)₂ powder. Note that although the data are displayed in 6.7 ns bins, they were collected and fit in 0.33 ns bins. The agreement with similar measurements made with crystals of the natural mineral cerussite is excellent [9].

While some of the compounds listed in Table 3 have been known as fluorescent materials for some time [10, 11], this does not necessarily mean that they are useful as scintillators because (i) some compounds have radiative transitions that are excited much more efficiently by UV

radiation than by x-rays and (ii) some compounds exhibit a fluorescence after UV or x-ray excitation that originates only from the surface and photons produced by excitation deep in the crystal (such as by gamma radiation) are absorbed in the bulk of the crystal by radiation trapping and lost by non-radiative processes. For this reason, many powders listed in Table 1 require further study in crystal form before they can be established as useful scintillators. Conversely, compounds that do not fluoresce by UV excitation may still be useful scintillators because they have radiative transitions that can only be excited by more energetic radiation.

3.4 Lead carbonate, Ytterbium oxide, and Lead chloride

One interesting result is the fluorescence of PbCO₃, which we have further investigated in the form of the naturally occurring mineral cerussite to establish this compound as a scintillator [9]. Since this material decomposes on heating, hydrothermal methods may be necessary for the production of quality synthetic crystals.

Another interesting result is the high density (9.2 g/cm³) and extreme speed of the emission of Yb₂O₃, whose response was faster than the BaF₂ and plastic powders tested, and showed no detectable components longer than 1 ns. Although the light output is low relative to BGO (0.6%), the emission wavelength of 350 nm would be convenient for glass phototubes and as a scintillation crystal could be useful for detecting energetic (>100 MeV) photons in high-rate conditions. Further investigation must await the critical step of finding an interested crystal grower.

While PbCl₂ was previously known to be fluorescent [10] and crystals are commonly grown, the fast components reported here may be useful, and this material merits further study.

4 FURTHERING THEORETICAL UNDERSTANDING

The registry file of the the Chemical Abstracts Service (American Chemical Society, Columbus, OH) contains entries for 9.7 million compounds, of which about one million are inorganic. For example, lead is present in about 17,000 compounds, excluding alloys. Considering the additional possibilities presented by doping with various concentrations of other elements, the number of candidates is staggering. Even if only a tiny fraction of these turn out to scintillate, many could have very useful properties indeed.

Only a small fraction of the listed compounds can be readily purchased, even in powdered form. A better theoretical understanding of scintillation mechanisms would be of benefit in choosing which candidates are worth the effort of preparing and purifying. To this end, a number of experimental techniques are available using synchrotron vacuum UV radiation for determining the electron band structure and radiative transitions in scintillators, including absorption spectroscopy, excitation spectroscopy, and photoemission spectroscopy. If a large variety of scintillators is studied by these means, a theory may emerge to guide the way to a more systematic selection of candidate compounds. Ultimately, it may some day be possible to perform first-principle molecular orbital calculations using supercomputers to determine the stability, crystal structure, and scintillation properties of any compound.

5 CONCLUSIONS

We have shown that it is possible to use synchrotron x-radiation to measure fast fluorescent emissions in compounds not readily available in single crystal form. Of 85 compounds tested, about half were found to have fluorescence below the limit of our technique, which was about 0.05% of BGO. Others had emissions in the 0.1 to 1% range, and a few had emissions that were both fast and above 1% of BGO.

Since only a small fraction of known compounds have ever been tested for scintillation properties, we conclude that many of these may be useful scintillators.

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