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### Authors

Li, H Harold  
Driewer, Joseph P  
Han, Zhaohui  
[et al.](#)

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## Two-dimensional high spatial-resolution dosimeter using europium doped potassium chloride: a feasibility study

H. Harold Li<sup>1,2</sup>, Joseph P. Driewer<sup>3</sup>, Zhaohui Han<sup>4</sup>, Daniel A. Low<sup>5</sup>, Deshan Yang<sup>1</sup>, and Zhiyan Xiao<sup>1</sup>

<sup>1</sup>Department of Radiation Oncology, Washington University School of Medicine, St. Louis, MO, USA

<sup>2</sup>Center for Materials Innovation, Washington University, St. Louis, MO, USA

<sup>3</sup>Department of Radiation Oncology, University of Nebraska Medical Center, Omaha, NE, USA

<sup>4</sup>Department of Radiation Oncology, Brigham and Women's Hospital and Harvard Medical School, Boston, MA, USA

<sup>5</sup>Department of Radiation Oncology, University of California, Los Angeles, CA, USA

### Abstract

Recent research has shown that KCl:Eu<sup>2+</sup> has great potential for use in megavoltage radiation therapy dosimetry because this material exhibits excellent storage performance and is reusable due to strong radiation hardness. This work reports the authors' attempts to fabricate 2D KCl:Eu<sup>2+</sup> storage phosphor films (SPFs) using both a physical vapor deposition (PVD) method and a tape casting method. X ray diffraction analysis showed that a 10 μm thick PVD sample was composed of highly crystalline KCl. No additional phases were observed, suggesting that the europium activator had completely been incorporated into the KCl matrix. Photostimulated luminescence and photoluminescence spectra suggested that F (Cl<sup>-</sup>) centers were the electron storage centers post-xray irradiation and that Eu<sup>2+</sup> cations acted as luminescence centers in the photostimulation process. The 150-μm thick casted KCl:Eu<sup>2+</sup> SPF showed sub-millimeter spatial resolution. Monte Carlo simulations further demonstrated that the admixture of 20% KCl:Eu<sup>2+</sup> and 80% low Z polymer binder exhibited almost no energy dependence in a 6 MV beam. KCl:Eu<sup>2+</sup> pellet samples showed a large dynamic range from 0.01 cGy to 60 Gy dose-to-water, and saturated at approximately 500 Gy as a result of KCl's intrinsic high radiation hardness. Taken together, this work provides strong evidence that KCl:Eu<sup>2+</sup> based SPF with associated readout apparatus could result in a novel electronic film system that has all the desirable features associated with classic radiographic film and, importantly, water equivalence and the capability of permanent identification of each detector.

### Keywords

radiation therapy dosimetry; dosimeter; storage phosphor

## 1. Introduction

Two-dimensional radiation therapy dosimeters require sub-millimeter spatial-resolution due to the complex dose distributions and high dose gradients associated with modern radiation therapy treatment modalities such as intensity modulated radiation therapy (IMRT) (Low *et al.*, 2011). Film, either radiographic (Pai *et al.*, 2007) or radiochromic (Lynch *et al.*, 2006), has been the detector of choice for high spatial-resolution IMRT dose distribution verification. Film can be inserted in any orientation in a phantom mimicking a patient's anatomy and has unparalleled high spatial-resolution that is essential for verification of steep dose gradients. While phantom integration and high spatial-resolution are clearly desirable characteristics, a dosimeter should also be reusable so that response variation from pixel to pixel can be quantified and therefore corrected. More importantly, reusability is central to building medical physicists' confidence in dosimetric measurements through the acquisition of benchmarking datasets and through long-term, repeated use and performance monitoring. The lack of reusability has contributed to the relegation of film to relative dosimeter. Quantitative measurements with film would require the acquisition of a sensitometric curve for each measurement; yet even this practice carries with it a questionable assumption that individual films from a single batch and individual pixels on the same sheet share a common response (Hartmann *et al.*, 2010).

In 2005, Olch (Olch, 2005) showed that BaFBr:Eu<sup>2+</sup>-based computed radiography (CR) storage phosphor films (SPFs) had the potential to be used for two-dimensional megavoltage radiation therapy dosimetry. However, BaFBrI has a high Z number ( $Z_{\text{eff}} = 49$ ) which leads to a strong photon energy dependence and consequently unacceptable measurement accuracy. Also, BaFBrI or CsBr-based SPFs were designed for diagnostic radiology where radiation doses are on the order of  $\mu\text{Gy}$ -mGy (Batentschuk *et al.*, 2009). For radiation therapy, a typical fractionated dose is 2 Gy. Therefore, a reusable dosimeter with tissue-like response, high spatial-resolution, and excellent radiation hardness properties is needed for quantitative radiation therapy dosimetry but is not currently available.

Recent work (Han *et al.*, 2009; Driewer *et al.*, 2011) has shown that KCl:Eu<sup>2+</sup> has great potential for use in megavoltage radiation therapy dosimetry because this material exhibits excellent storage performance and is reusable due to strong radiation hardness. Hansel *et al.* (Hansel *et al.*, 2014; Hansel *et al.*, 2013) report that KCl:Eu<sup>2+</sup> samples provide excellent signal even after they were given doses of up to 200 kGy at the Louisiana State University Synchrotron facility. Functionally, KCl:Eu<sup>2+</sup> relies on a mechanism of photostimulated luminescence (PSL), similar to BaFBr:Eu<sup>2+</sup>. Irradiation of the material produces electron-hole pairs that are stored in metastable energy traps. The spatial distribution of these trapped charge carriers forms a latent image whose information can be read out by stimulating the trapped charge carriers to recombine and release PSL photons proportional to the locally deposited dose. Charges remaining trapped after readout can be erased with a bright, broadband light and the material can be used again in the same manner.

Although KCl has a higher effective Z than water, i.e.  $Z=18$  vs. 7, Monte-Carlo simulations demonstrate that reducing the dimensions of the KCl:Eu<sup>2+</sup> dosimeter to a few microns would convert a Burlin-cavity dosimeter to a Bragg-Gray dosimeter (Zheng *et al.*, 2010),

thus providing minimal energydependence for megavoltage therapeutic beam dosimetry. Future development would involve the creation of a large-area KCl:Eu<sup>2+</sup>-based storage phosphor film using modern thin film techniques, for example, tape casting (Li et al., 2002; Mistler and Twiname, 2001) or physical vapor deposition (Hell *et al.*, 2008; Schmitt *et al.*, 2002; Leblans *et al.*, 2000).

This work reports the authors' attempts to fabricate 2D KCl:Eu<sup>2+</sup> SPFs using both a physical vapor deposition (PVD) method and a tape casting method in a laboratory setting. In conjunction with numerical simulation data and the bulky material's large dynamic range data, this work provides strong evidence that KCl:Eu<sup>2+</sup>-based SPFs with associated readout devices could result in novel electronic film systems, combining water equivalence and the capability of permanent identification of 2D films with other desirable features associated with classic radiographic film.

## 2. Materials and methods

### 2.1 Physical vapor deposition

High purity KCl (99.995%, Sigma-Aldrich) particles and reagent grade EuCl<sub>3</sub>·6H<sub>2</sub>O particles were mixed at a 500 ppm Eu<sup>2+</sup> concentration. A thin layer of KCl:Eu<sup>2+</sup> was subsequently deposited on a substrate of borosilicate glass (e.g., laboratory slides) with a physical vapor deposition system (Kurt J. Lesker, Clairton, PA). During evaporation, the substrate was rotated in order to maximize active layer's homogeneity. The distance from the source to the substrate was fixed at 20 cm in the current system. The substrate holder had an optional heating element with a maximum achievable temperature of 300°C. Raw materials were placed in evaporation source holders, i.e. tungsten or tantalum boats or aluminum oxide crucibles, which were resistively heated under vacuum, for example 10<sup>-6</sup> Torr. The deposition rate was controlled by a PID controller, which adjusted current through the thermal sources upon receiving feedback from a deposition rate monitor.

### 2.2 Tape casting

The tape casting device shown in Figure 1 was designed and constructed for prototyping purposes. A linear slide (Velmex, MA6000 series Unislide, Bloomfield, NY) fitted with a custom aluminum pusherbar was utilized to move the casting head. The slide was positioned parallel to a 30 cm × 45 cm vacuum plate (Paul N. Gardner Company, Inc., Pompano Beach, FL) under a fume hood. The weight of the slide and the vacuum plate was sufficient to prevent relative motion of the two devices during casting. The fume hood vacuum line was connected to the vacuum plate. Seven mil polyethylene sheets (DuPont Melenix 454, KRS Plastics, Tabor City, NC) served as the substrates for this study. The linear slide was controlled by a programmable stepper motor controller.

### 2.3 Spectroscopic measurements

The stimulation spectra of x-ray irradiated KCl:Eu<sup>2+</sup> samples measure the PSL intensity as a function of stimulation wavelength. The stimulating light was a 150 W, UV enhanced Xe arc lamp (Newport, Stratford, CT). The stimulation spectra were obtained by scanning a monochromator (Cornerstone 130, Newport) between 500 and 750 nm in 1 nm increments,

while the PSL signals were collected using a photomultiplier tube (R1924A, Hamamatsu, Bridgewater, NJ) and a narrow bandpass filter FB420-10 (420 nm wavelength with 10 nm full-width-half-maximum, FWHM, Thorlabs, Newton, NJ). The bandpass filter separates the PSL light from the stimulation light. In addition, a second monochromator was used between the sample and the PMT in order to obtain the PSL emission spectra when the sample was stimulated using a 560 nm light provided by the Xe lamp. For unirradiated samples, photoluminescence (PL) emission spectra were measured using a 340 nm UV excitation light provided by the Xe lamp filtered by a 340 nm band pass filter with 10 nm FWHM.

#### 2.4 X ray diffraction measurements

X-ray diffraction spectra were obtained using a Rigaku Giegerflex D-MAX/A Diffractometer using Cu-K $\alpha$  radiation. The step size for each of the following spectra was 0.01 and the dwell time was 1 s per step. The operating power used was 35 kV and 35 mA.

#### 2.5 Storage phosphor film readout

A precision tilter that holds the SPF in place was fit to a 2-D array of linear slides positioned in-line with the laser light as shown in Figure 2. The film was scanned in front of the laser, which was turned on for a line scan and blocked when the slides moved to the next row. Spatial resolution was determined by the range of travel between pulses that triggered data acquisition.

#### 2.6 Pellet sample

The mixed powders were pressed at 2200 lbs force in a hydraulic press (Carver Inc., Wabash, IN) using an evacuable pressing die to 6 mm in diameter and 1 mm thick. The pellets were sintered at 710 °C for 3 h in air and allowed to cool naturally to 300 °C followed by a rapid cooling to room temperature. Pellet samples were wrapped in plastic wrap to mitigate the adverse effects of moisture on the material.

#### 2.7 Monte Carlo simulations

To understand the response of the KCl:Eu<sup>2+</sup>-binder mixture to clinically relevant cases, Monte Carlo (MC) simulations were employed. The approach was similar to that of Palm's work (Palm *et al.*, 2004; Palm and LoSasso, 2005). The MC code was DOSRZnrc in EGSnrcMP (Kawrakow *et al.*, 2003). We simulated the passage of the circular 6 MV photon beams incident on a 20 cm radius, 30 cm deep right cylindrical water phantom. The photon beam was modeled as emitting from a point source. The incident energy spectrum was obtained from published data for a 6 MV beam from a common linear accelerator (Varian) (Sheikh-Bagheri and Rogers, 2002). The calculations were run with sufficient histories to achieve less than 0.5% statistical uncertainty. The binder was modeled as polyethylene terephthalate or C<sub>6</sub>H<sub>10</sub>O<sub>4</sub>, and the film thickness was 0.2 mm (Li *et al.*, 2007).

### 3. Results

The x ray diffraction spectra (Figure 3) show that a 10  $\mu$ m- thick PVD sample is composed of highly crystalline KCl. The peaks at 28.494° and 40.797° correspond to KCl's (200) and

(220) planes, respectively (Powder Diffraction File 00-001-0786). No other phases are observed, suggesting that europium activator was completely incorporated into the KCl matrix during deposition.

Figure 4 shows PSL stimulation spectra for a 1 mm- thick pellet sample and a 10  $\mu\text{m}$ - thick PVD sample. Both the shape and peak position agreed with each other, indicating that same type of electron storage centers were created in the samples, namely  $\text{F}(\text{Cl}^-)$  center. Figure 5 shows PSL emission spectra and PL emission spectra for both samples. The overall good agreement between the spectra for the different excitation processes (i.e., PL and PSL) suggest that  $\text{Eu}^{2+}$  cation acts as the photostimulated luminescence center in the PSL process of both samples via  $4f^65d^1 \rightarrow 4f^7 (^8\text{S}_{7/2})$  transition, which means that the emission is largely due to the promotion of an  $\text{Eu}^{2+}$  electron to the  $4f^65d^1$  excited state and then radiative decay to the  $4f^7$  ground state. For the PSL process, the energy came from electronhole recombination during photostimulation post-xray irradiation, while for the PL process, the energy came directly from the 340 nm UV stimulation light. For the PVD sample, the relative peak shift between PL and PSL was probably due to the interference of the glass substrate. Note that stimulation light for the PSL stimulate spectrum measurement is monochromated and has a power on the order of  $10^{-6}$  W as compared to  $10^{-2}$  W of a laser used in the commercial CR reader, suggesting excellent storage performance of the  $\text{KCl}:\text{Eu}^{2+}$  samples.

Figure 6 shows a KV-xray image of a lead spatial-resolution pattern taken by the  $\text{KCl}:\text{Eu}^{2+}$  tape casted sample. It is evident that sub-millimeter resolution can be achieved by the  $\text{KCl}:\text{Eu}^{2+}$  SPF.

The Monte Carlo simulated dose distribution profiles for the depths of 10 cm and 20 cm are shown in Figure 7 for a 22.6 cm diameter field (the equivalent circular field of a  $20 \times 20 \text{ cm}^2$  square field) normalized at the center of the field. This figure shows that the strong overresponse of  $\text{BaFBr}_{0.85}\text{I}_{0.15}:\text{Eu}^{2+}$  to the low-energy scattered photons causes the profile shape to be distorted by up to 15%. The pure  $\text{KCl}:\text{Eu}^{2+}$  overresponds significantly less as evidenced by the closer agreement to the actual dose profile, with a maximum of 4.0% difference in the peripheral region where low-energy scattered photons are dominant. The admixture of 20%  $\text{KCl}:\text{Eu}^{2+}$  and 80% binder comes closest to the actual dose profile, with a maximum difference of only 1.6%. The leakage dose was not modeled here, but would add a small amount of high-energy photons outside the portal. The overresponse to the scattered photons would cause dose measurement errors in complex IMRT dose distributions with spatially varying primary-to-scattered photon ratios (Kirov *et al.*, 2006; Palm *et al.*, 2004; Palm and LoSasso, 2005)

Figure 8 shows the normalized PSL intensity vs. dose-to-water measured using a pellet sample.  $\text{KCl}:\text{Eu}^{2+}$  shows a linear response from zero to 60 Gy, and eventually saturates at about 500 Gy. Li et al. (Li *et al.*, 2013) recently reported that the same 1 mm- thick pellet sample is capable of detecting a dose-to- water of 0.01 cGy, suggesting that  $\text{KCl}:\text{Eu}^{2+}$  material yields a large dynamic range with comparable storage performance as traditional CR materials but superior radiation resilience.

## 4. Discussion and conclusion

Computed radiography (CR) has been successful in replacing radiographic film in digital diagnostic imaging. However, due to the high effective Z and low radiation hardness of common CR materials, use of CR in radiation therapy dosimetry has been thus far unsuccessful. Recent research seems to have found an excellent storage phosphor material, KCl:Eu<sup>2+</sup>, with the potential to become the physical foundation of a novel, reusable, high spatial-resolution, phantom insertable planar dosimeter. Although KCl has a much lower effective Z than conventional CR materials, its Z number is still larger than that of water. One way to maximize the waterlike response of the KCl:Eu<sup>2+</sup> dosimeter is to reduce the dimensions of the sensitive volume to a few microns. It is well known that the energy dependence of a dosimeter is largely the result of photoelectric interactions of low energy scattered photons with the dosimetry material, especially with those lower than 100 keV according to Palm's MC stimulations (Palm *et al.*, 2004). The projected maximum range for, say, a 40 keV secondary electron generated by photoelectric interactions in water is about 20  $\mu\text{m}$ . Because the range in a medium is roughly independent of Z for electrons and positrons (Attix, 2004) the above range in KCl:Eu<sup>2+</sup> reduces to about 10  $\mu\text{m}$  when scaled by KCl's physical density of 1.987 g/cm<sup>3</sup>. If the dosimeter size is of the order of a few microns, it is highly probable that secondary electrons created in the active layer, so-called starters, deposit a significant fraction of their energy outside the dosimeter's sensitive volume, reducing the energy dependent measurement artifact. With the increase in dosimeter or cavity dimension, the range of the above starters becomes smaller than the cavity size, and thus starters become insiders. As a result, increased energy dependence occurs. Zheng *et al.* (Zheng *et al.*, 2010) confirmed this suggestion through Monte Carlo simulations. This reasoning also explains why AgBr radiographic film, with a sensitive layer on the order of a few microns thick, did not show a strong energy-dependence as would be expected due to its high effective Z of 43 (Low *et al.*, 2011).

Our data (Figures 3, 4, 5) demonstrates that micron-thick KCl:Eu<sup>2+</sup> materials can be successfully fabricated using a physical vapor deposition (PVD) method. PVD is based on the concept that all materials exhibit a finite vapor pressure (Mahan, 2000). The material to be deposited either sublimates or evaporates from a source and condenses onto a substrate to form a thin film. There is no limit on source shape and deposition rate, and thickness is easily controlled, ranging from tens of angstroms to tens of microns. Physical vapor deposition leads to the best results when phosphor crystals with high crystal symmetry are used as the source. Fortunately, potassium chloride (KCl) belongs to this group (Mahan, 2000) and is one of a class of compounds, molecular solids, whose vapors consist of particles having stoichiometric composition (or are at least composed primarily of such molecules). Therefore, stoichiometric europium doped potassium chloride thin films can be obtained by direct vaporization of these compounds.

An alternative approach to fabricating a waterlike KCl:Eu<sup>2+</sup> SPF is using the classic tape casting method. Tape casting has been the main method to create BaFBr:Eu<sup>2+</sup> detectors, thickness ranging from 100  $\mu\text{m}$  to 300  $\mu\text{m}$ , for computed radiography where the routine phosphor particle size is between 5 to 10  $\mu\text{m}$  on average (Leblans *et al.*, 2011). The introduction of binder material, with a low atomic number very close to that of water,

partially absorbs secondary electrons generated by the interaction between low energy scattered photons and a  $\text{KCl:Eu}^{2+}$  particle and prevents them from reaching other  $\text{KCl:Eu}^{2+}$  particles and thus degrading the energy response. However, it does not affect the primary contribution to dose signal from electrons generated in a phantom or tissue. The thickness of a SPF cast from particles of this size may be, for example, 100  $\mu\text{m}$ ; however, the action of the binder will lower the effective thickness to a value in the neighborhood of the size of an individual phosphor particle, thus reducing energy dependence (Li, 2012). The data shown in Figures 1, 6, 7 demonstrate that a thick  $\text{KCl:Eu}^{2+}$  SPF provides sub- millimeter spatial-resolution and potentially a nearly water-equivalent response.

Despite promising data, significant research and development remains to go from bench to clinic. These efforts include, for example, encapsulation against ambient moisture and creation of particles of a few microns for tape casting.  $\text{KCl:Eu}^{2+}$ , similar to  $\text{CsBr:Eu}^{2+}$ , is hygroscopic.  $\text{CsBr:Eu}^{2+}$  is the basis of a novel needle-crystalline CR detector created by PVD method (Hell *et al.*, 2008; Schmitt *et al.*, 2002; Leblans *et al.*, 2000). Modern protective coating technology could be used to overcome this so that after coating, the dosimeter will not be affected by ambient humidity. In a recent patent (Leblans *et al.*, 2002), for example, a  $\text{CsBr:Eu}^{2+}$  screen was prepared with protective coatings. The screen remained intact after it was submerged in water for 24 hours. Furthermore, it showed excellent resistance to abrasion.  $\text{KCl}$ 's deliquescence also demands moisture-free powder processing techniques in order to create particles with dimensions of a few microns. These particles will subsequently be mixed with compatible binder materials to form a homogenous, low  $\text{KCl:Eu}^{2+}$ -to-binder ratio slurry or lacquer for tape casting, thus leading to minimized energy dependence.

In conclusion, it is feasible to fabricate  $\text{KCl:Eu}^{2+}$  storage phosphor film with either physical vapor deposition method or tape casting method. With further development the  $\text{KCl:Eu}^{2+}$ -based dosimeter could become a versatile and durable tool with a wide range of applications in the modern radiation oncology clinic.

## Acknowledgments

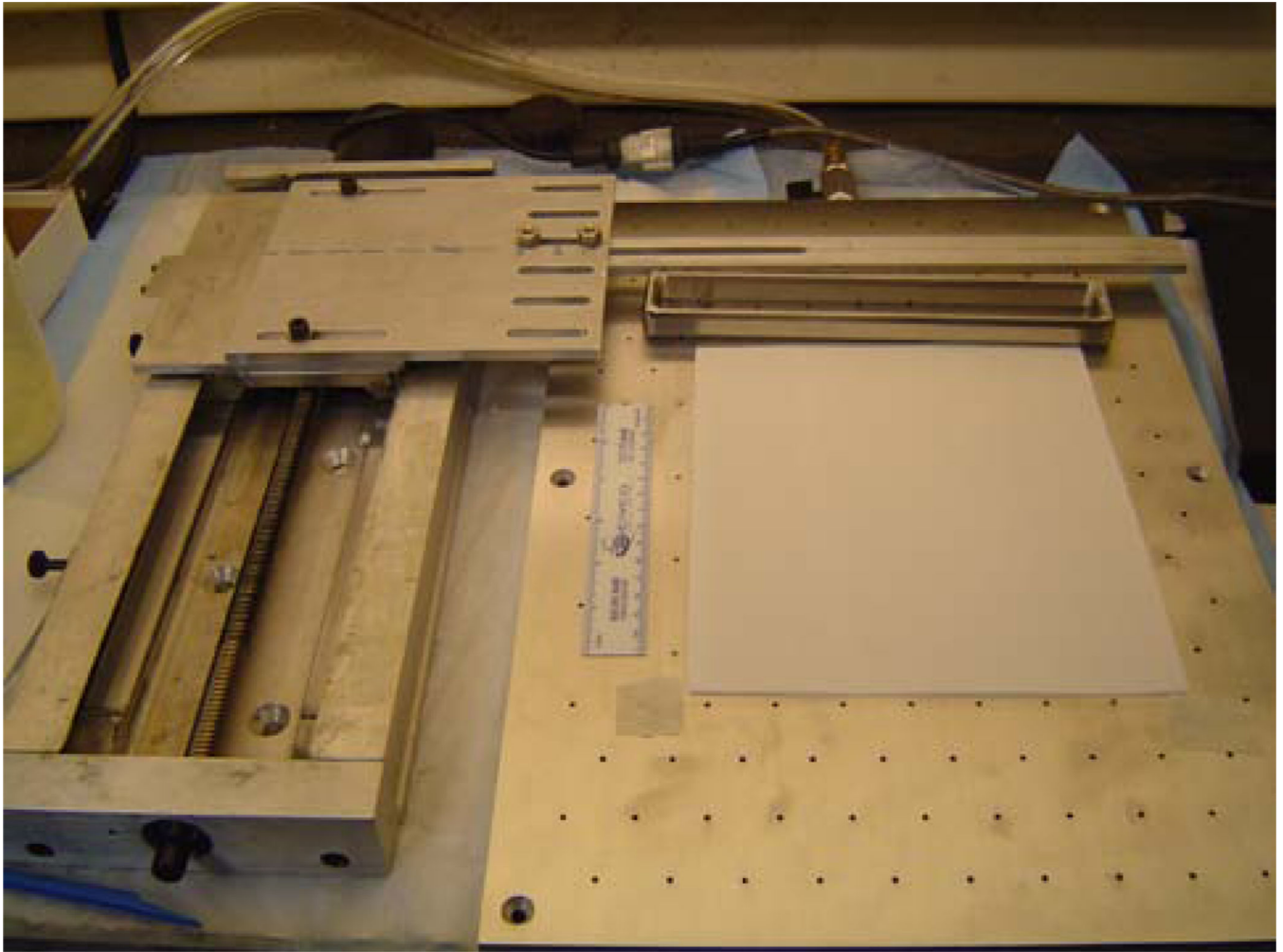
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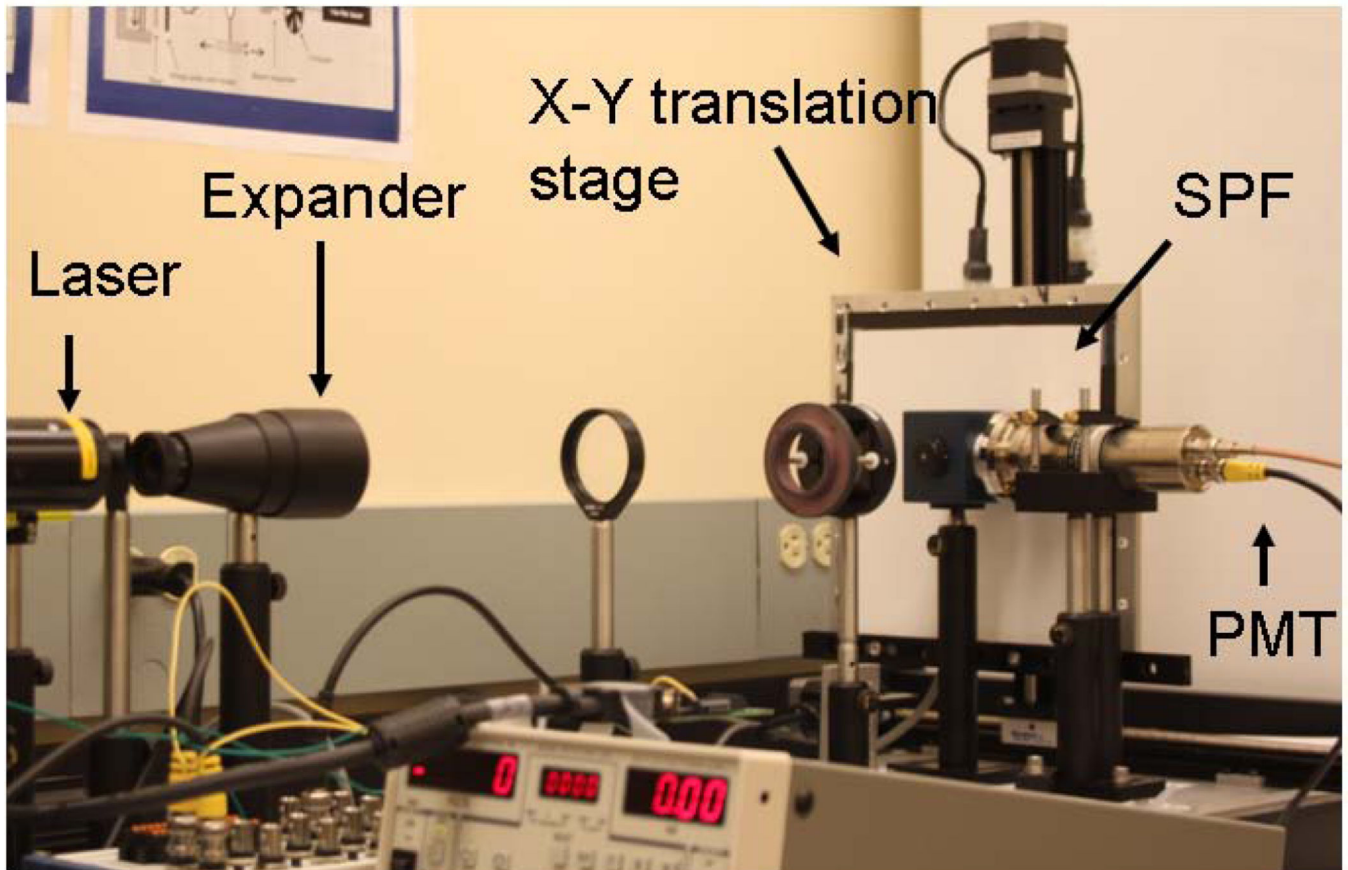
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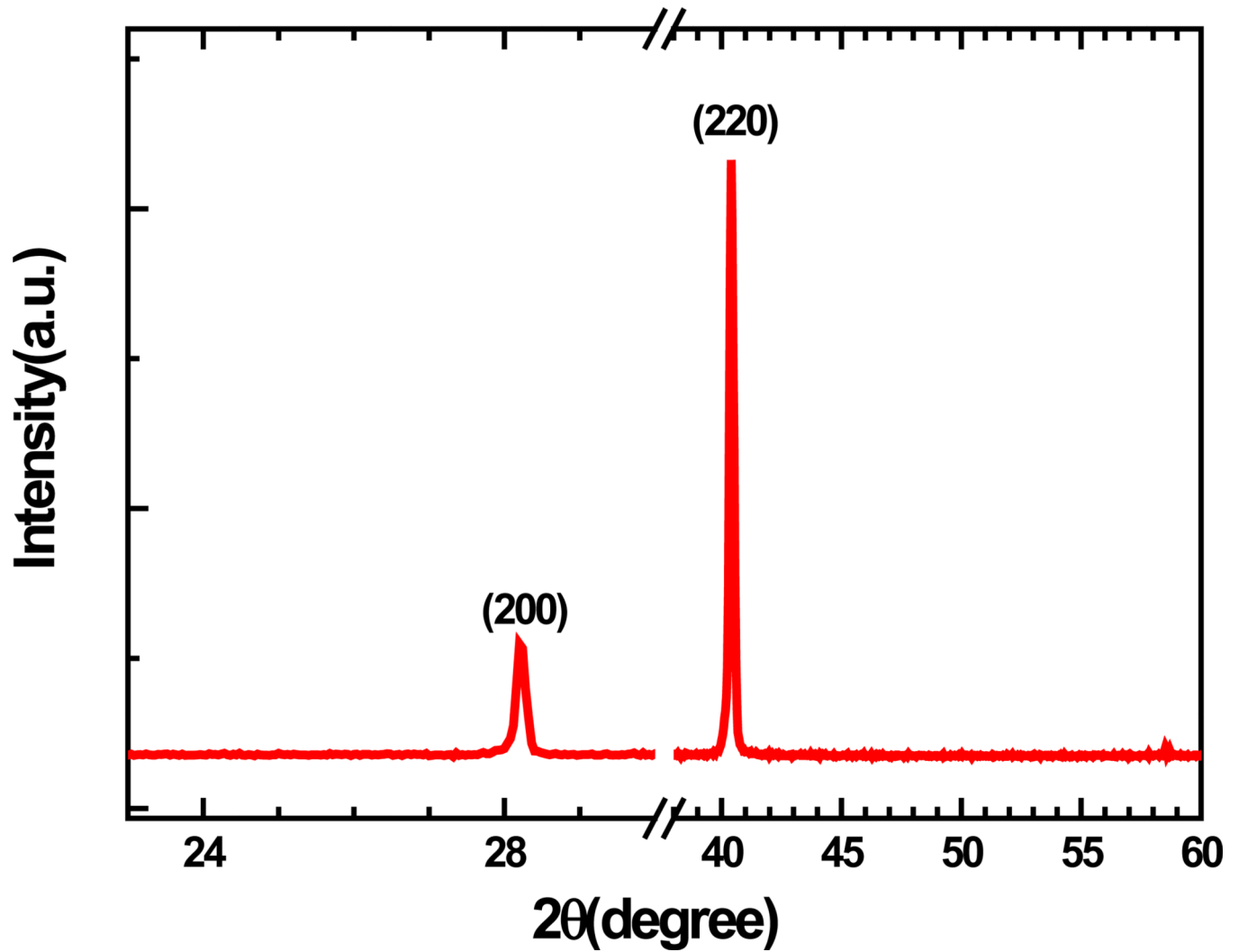


**Figure 1.** House-made casting device and a test  $15 \times 15 \text{ cm}^2$   $\text{KCl:Eu}^{2+}$  storage phosphor film. A homogenous suspension containing  $\text{KCl:Eu}^{2+}$  storage phosphor particles, liquid vehicle and polymer binder was formed and subsequently cast by doctor-blade onto a polyethylene terephthalate substrate to form a  $150 \text{ }\mu\text{m}$ - thick  $\text{KCl:Eu}^{2+}$  film with coating weight of  $28 \text{ mg/cm}^2$ .

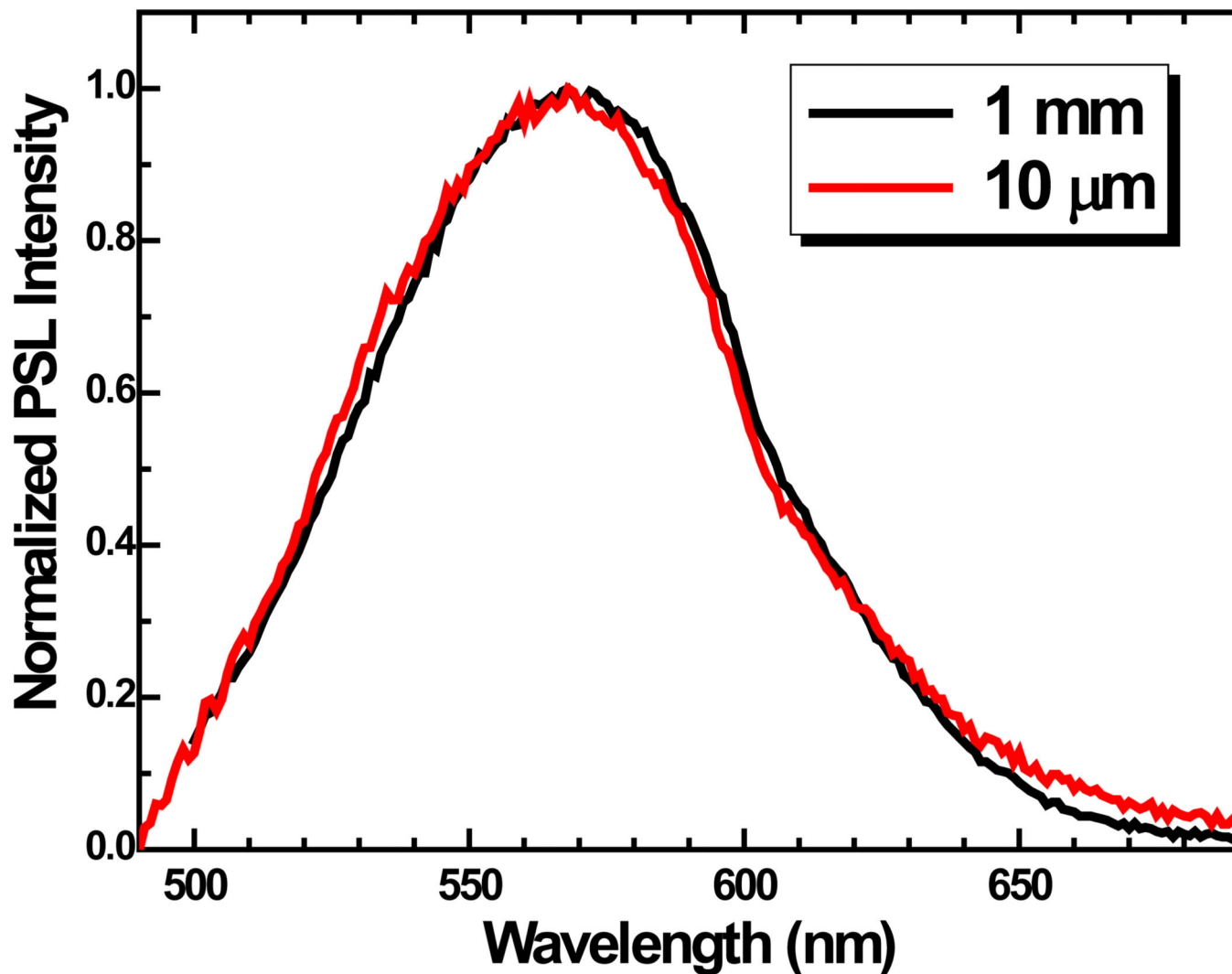


**Figure 2.**

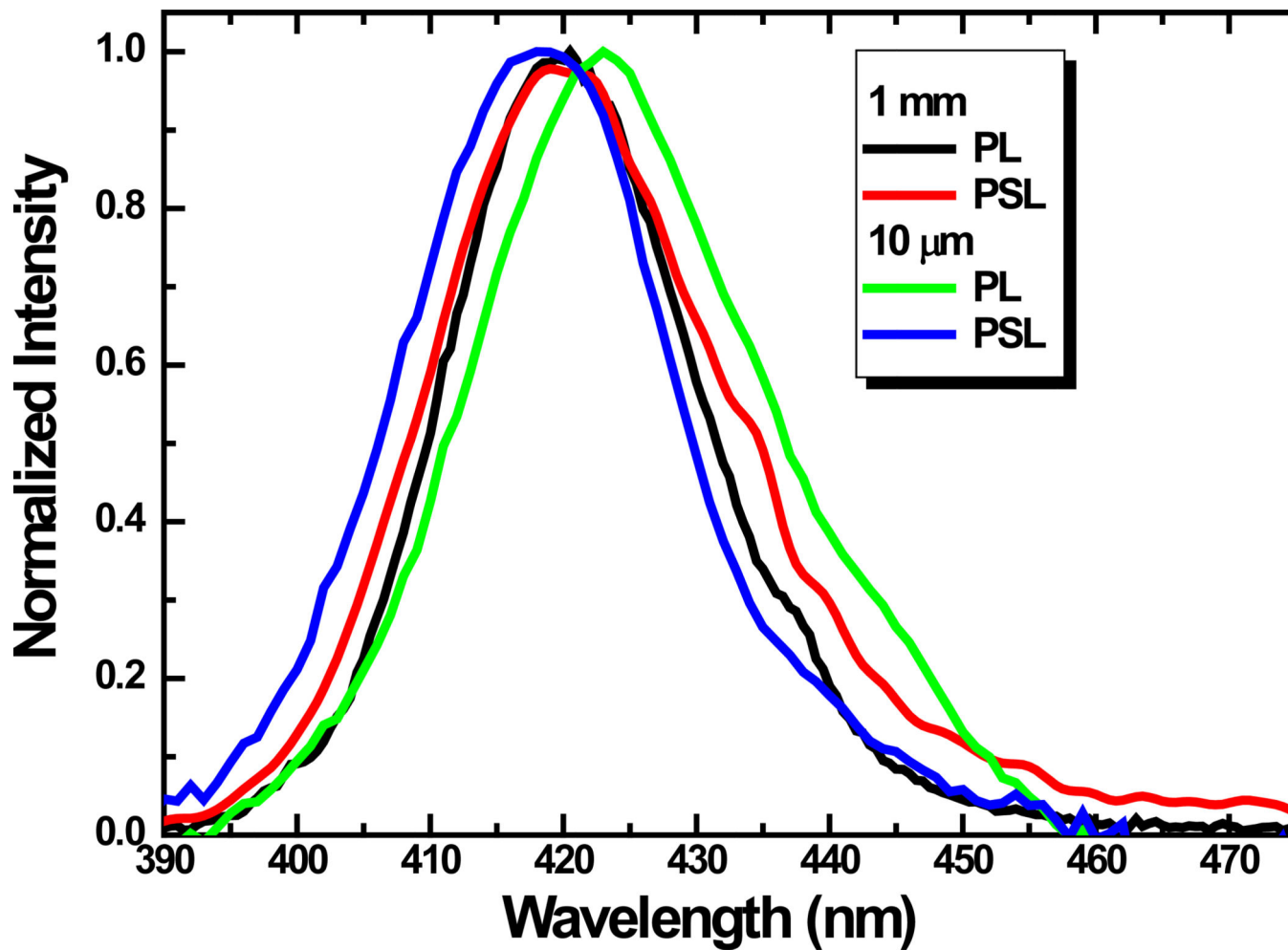
A laboratory 2D scanner for  $\text{KCl:Eu}^{2+}$  storage phosphor film (SPF) readout. The scanning speed is up to 7.5 cm/sec. A 17 cm x 17 cm scan at 0.5 mm pixel size may be performed and processed in under 20 minutes. Note that a commercial CR reader takes less than 1 minute to process a CR SPF. Data is simultaneously sampled from the signal generating PMT and a reference diode in order to correct for laser power fluctuation. Laser spot size is controlled by a lens fixed to a precision micrometer and is typically 0.1 mm.



**Figure 3.** X-ray diffraction spectra for a KCl:Eu<sup>2+</sup> thin active layer created by physical vapor deposition. Thickness: 10 microns. The peaks at 28.494° and 40.797° correspond to KCl's (200) and (220) planes, respectively.

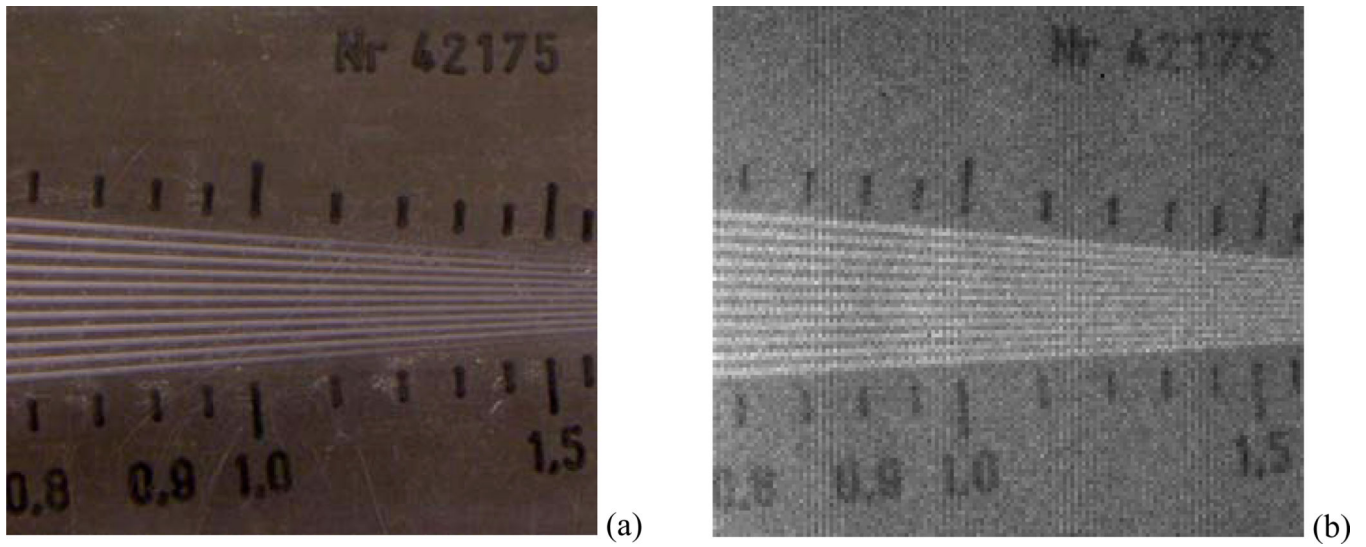


**Figure 4.** Stimulation spectra for KCl:Eu<sup>2+</sup> pellet sample and PVD sample. Both the shape and peak position agreed with each other, indicating that the same type of charge storage centers were created in both samples, namely F(Cl<sup>-</sup>) center.



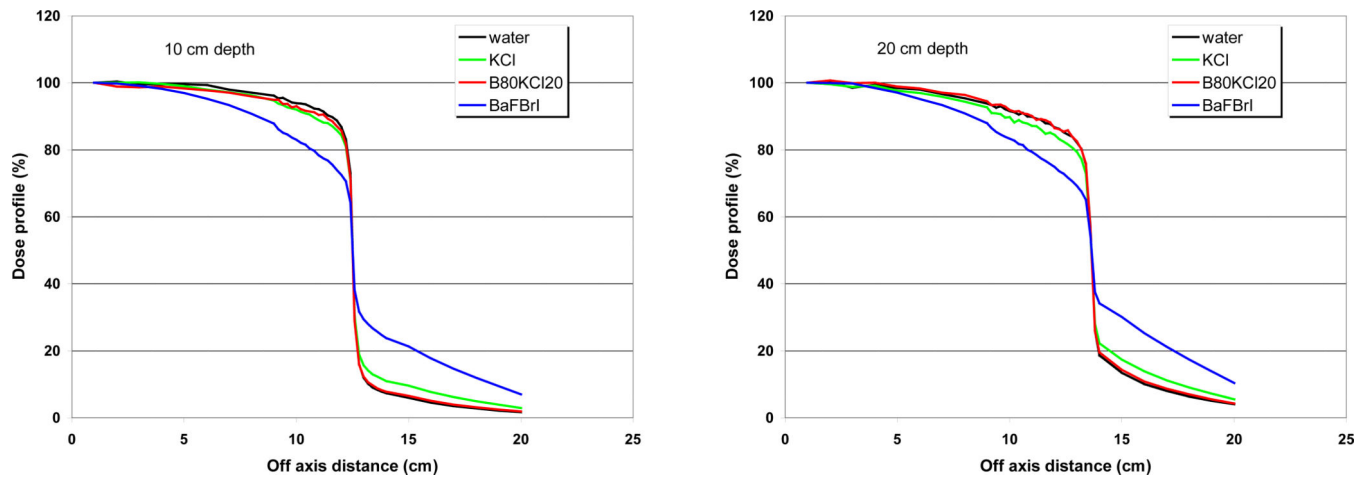
**Figure 5.**

PL (photoluminescence) and PSL emission spectra for KCl:Eu<sup>2+</sup> pellet sample and PVD sample, suggesting that Eu<sup>2+</sup> cation acts as the luminescence center in the PSL process in both samples via  $4f^65d^1 \rightarrow 4f^7 (^8S_{7/2})$  transition.



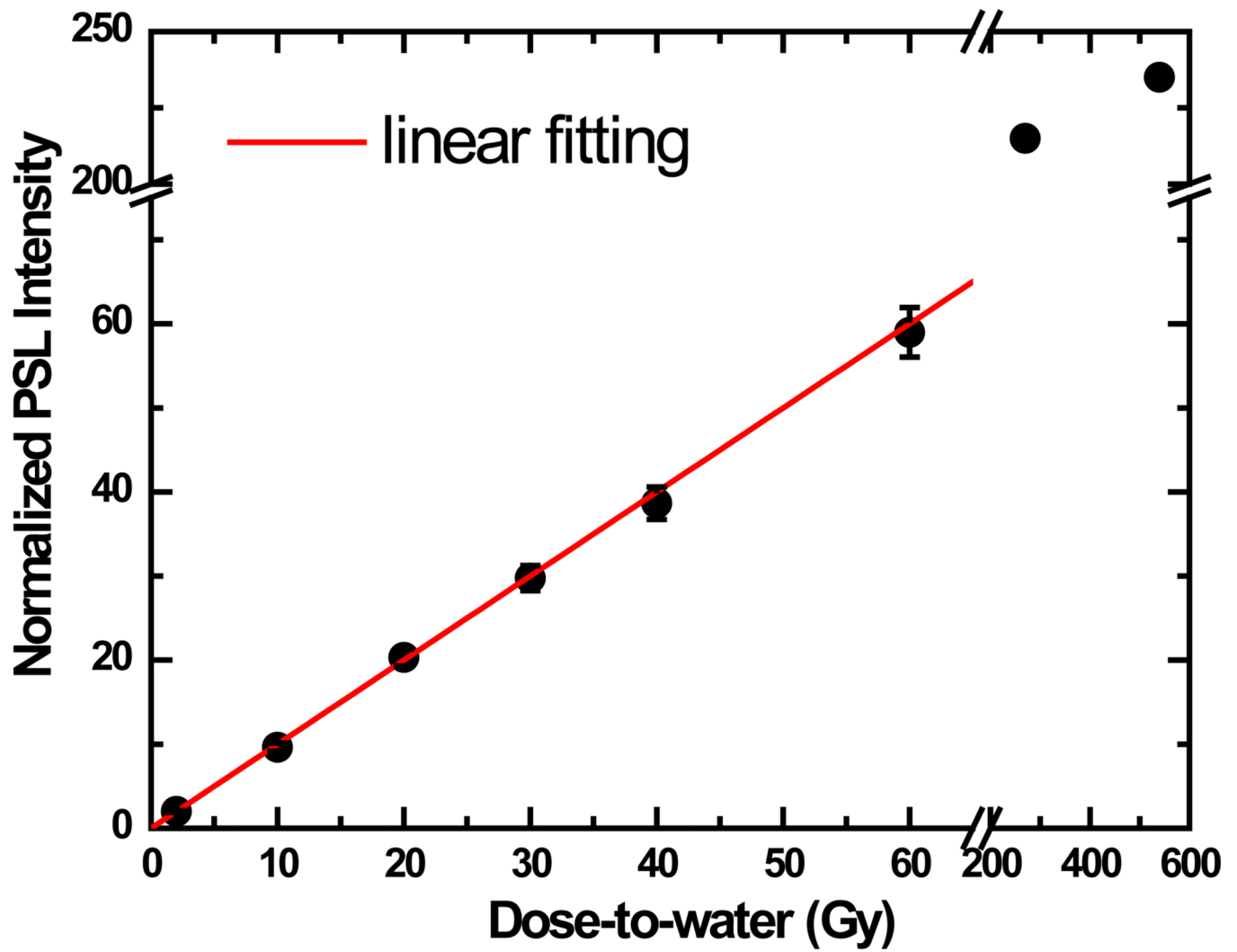
**Figure 6.**

Lead spatial resolution pattern in lp/mm (a), and raw KCl:Eu<sup>2+</sup> PSL image (b). A KV $\times$ ray image of a lead spatial-resolution pattern was taken by the prototype KCl:Eu<sup>2+</sup> SPF. The x-ray image was readout using a laboratory 2D scanner. It is evident that sub-millimeter resolution can be achieved by the SPF.



**Figure 7.** Monte Carlo simulated dose profiles at depth 10 cm (left) and 20 cm respectively for a 6 MV beam, SSD = 100 cm, 20x20 cm<sup>2</sup> field size (i.e. radius = 11.28 cm for an equivalent circular field)





**Figure 8.** Normalized PSL intensity vs. dose-to-water. KCl:Eu<sup>2+</sup> shows a linear response up to 60 Gy dose-to-water, and saturates at approximately 500 Gy.