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# A versatile low-cost laboratory apparatus for testing triboluminescent materials

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#### ABSTRACT

The design and fabrication of a simple low-cost laboratory apparatus used to test triboluminescent properties of materials is reported. This unique experimental system consists of an impact chamber attached to the bottom of an adjustable drop tower. A 1.25 in. steel ball with a mass of 130 g is dropped from a known height in the tower impacting the pile of powdered triboluminescent sample at low velocities (<10 ms<sup>-1</sup>). Any generated triboluminescence is then recorded using a photodiode and amplifier and recorded on an oscilloscope. The resulting triboluminescence data is analyzed using specially written LabVIEW programs. The details of the experimental setup, techniques, and results for several common inorganic and organic triboluminescent phosphors are discussed in this paper.

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#### 1. Introduction

In 1888, Wiedemann and Schmidt defined triboluminescence (TL) as the emission of light produced by mechanical action. Since that time, scientists have been trying to fully understand and take advantage of this fascinating phenomenon. It has been estimated that 30% of organic crystals and 50% of inorganic crystals are triboluminescent [1]. While no complete theory exists, the triboluminescent spectrum and decay times have been measured for thousands of crystals. Extensive research has also been completed using TL as the active element for impact sensors [2–8].

Recently, Hollerman et al. have been investigating the properties of zinc sulfide doped with manganese (ZnS:Mn), which emits strong TL when stressed or struck [4–8]. Using a custom-built drop tower, Bergeron et al. measured the

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triboluminescent emission spectrum of ZnS:Mn generated during low velocity (few ms<sup>-1</sup>) impacts [7,9]. In addition, Bergeron et al. found that the intensity of the emitted triboluminescent light is proportional to projectile velocity or kinetic energy [10].

The majority of known materials emit TL with low intensities [11]. Over the past half-century, approximately 40 materials have been reported that contain sufficient light emission to be easily observed with the naked eye [11,12]. Europium tetrakis (dibenzoylmethide) triethylammonium (EuD<sub>4</sub>TEA) is one of the brightest TL phosphors currently available [12]. It exceeds the TL emission of even ZnS:Mn, which is one of the most studied TL materials [2]. This leads to the question, how can researchers systematically compare the triboluminescent emission yield for different materials? The authors have successfully designed and fabricated a simple drop tower to measure and compare the triboluminescent emission properties for any number of materials.

For many luminescent materials, the reduction in light intensity from the cessation of excitation can be written as:

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$$I = I_0 \exp\left\{-\frac{t}{\tau}\right\},\tag{1}$$

where I is the fluorescent light intensity (arbitrary units),  $I_0$  is the initial fluorescent light intensity (arbitrary units), t is the total elapsed time since cessation of excitation (s), and  $\tau$  is the fluorescent decay time (s).

The time needed to reduce the light intensity to  $e^{-1}$ (36.8%) of its original value is defined as the fluorescence decay time ( $\tau$ ). Luminescence can be divided into two groups: fluorescence and phosphorescence. Phosphorescence, also known as delayed emission, has a much longer decay lifetime than fluorescence. Typical phosphorescence lifetimes can vary from 100 ms up to many seconds in duration, while the lifetimes of fluorescent materials usually range from 1 ns to 10 ms. More specifically, phosphorescence is the emission of light from a triplet excited state, which is where the electron in the ground state and excited state has the same spin orientation. The triplet state is due to the fact that the spin flips take more time to complete. Fluorescence occurs in singlet states, with the excited electron having the opposite spin of the paired ground state, resulting in a short emission time for photons. The fluorescence decay time is unique to each material. It might be possible to use the fluorescence decay time as an indicator to gauge the production of TL from impacts. Some materials like ZnS:Mn,Cu have multiple decay times. Eq. (1) would be valid over a limited range of times.

#### 2. Experimental apparatus

Fig. 1a shows a schematic diagram of a specially designed drop tower, which is based on previous related designs as described in Refs. [4–6]. The tower was constructed from 0.75 in. (19 mm) thick medium density fiberboard, a 0.25 in. (6 mm) thick plexiglass plate, and a common 1.25 in. (32 mm) diameter piece of PVC pipe. The pipe has holes drilled in uniform increments denoting the net distance of the drop. A simple pin is used as the release

mechanism and to set the drop height to the desired distance.

A measurement begins by placing a pile of sample powder under the plexiglass plate as shown in Fig. 1a. The powder is spread out so that it has a minimum thickness and is positioned around the center of the tube. A 130 g steel ball is positioned on a pull pin a distance of 42 in. (1.1 m) above the material. The pin is pulled and the ball falls and impacts with the sample material producing TL. After each test, the drop tube is removed, the ball is cleaned, and the sample powder is redistributed near the center of the target area [9].

To determine the triboluminescent yield for a given sample, a United Detector photodiode (PD) is positioned under the plexiglass plate 0.875 in. (2.25 cm) below the sample. A Melles Griot large dynamic range linear amplifier (often set to a gain of 200  $\mu A$ ) increases the signal amplitude. A Tektronix 2024B oscilloscope records the signal in single sequence mode. Table 1 shows typical oscilloscope settings used for most of the tested material measurements. Usually for materials like EuD\_4TEA, the oscilloscope needs to be set on 2 or 4 volts per division. Conversely, TL signals from impacts on ZnS:Cu requires the Melles Griot amplifier to be set to 20  $\mu A$ . Once the signal is acquired, it is analyzed using custom LabVIEW program that integrates the area under the curve and calculates the decay time for the particular emission.

Knowing the decay time and relative intensities of materials is useful; however, how does one characterize materials with similar triboluminescent yields or decay times? One way is to measure the triboluminescent emission spectrum emitted at impact. To do this, the PD is replaced with a cosine corrector. A computer program called SpectraSuite by Ocean Optics is then used to control a S2000 spectrometer. Following the same procedures as described above, a triboluminescent spectrum is recorded with an integration time set to 0.1 s using the apparatus shown in Fig. 1b. After the measurements are completed, SpectraSuite-generated files are processed and the emission peak locations and

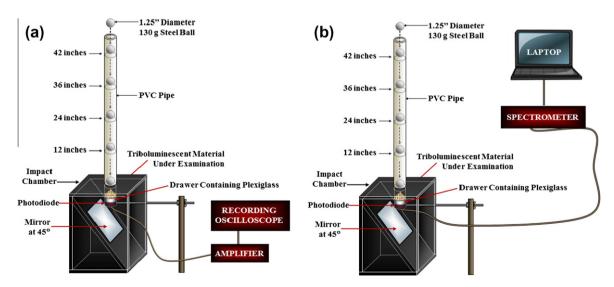


Fig. 1. Schematic diagram of the specially designed drop tower used to measure the triboluminescent: (a) integrated light yield, and (b) visible spectrum.

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