# A low-cost thermoluminescence system for use with .net computing environments

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Abstract. Many branches of scientific and industrial research require precise instrumentation for control and measurement that tend to be prohibitively expensive. In the current global economic climate, the funding to procure such equipment is fast dwindling. Rather than face a gradual downturn in research activity as a result of equipment procurement difficulties, an alternative is to design and build low-cost instruments. Our current institutional interest in the synthesis and characterization of phosphors, polymers and nano-materials indicates a variety of instruments, one of which is the use of thermoluminescence (TL) system. This instrument requires precise control and measurement of experimental parameters, particularly the sample excitation temperature and output intensity. In the present paper we describe the design and construction of an ultra-low cost TL instrument that allows automatic control of various steps of the experiment while logging instantaneous intensity output. We present preliminary results and indications that demonstrate the versatility for temperature sequencing, range and heating control of the sample over the temperature range of 23 to  $400^{\circ}$ C. A comparable instrument in the institution operates at a maximum ceiling of  $300^{\circ}$ C. Additional refinements enable the sample temperature to be held constant at any temperature in this range to within  $\pm 0.5$  °C with the aid of a software tuned proportional-integral-derivative (PID) controller. Intensity measurements are made using a temperature compensated, large area photo-diode operated in photovoltaic mode and covering a wavelength range 400 nm to 1100 nm. The various interfaces such as the universal serial bus (USB) protocol handling, the Visual Basic.NET control program, the microcontroller firmware code written in the C-language have been developed simultaneously. A record of each experimental run is logged to a disk file in a format that allows direct import into spreadsheets and analysis programs. Finally, we draw comparisons between the final TL system and the existing commercial TL system for a standard reference sample.

## 1. Introduction

Thermoluminescence is the emission of light from an insulating or semiconducting material in response to an absorption of ionizing energy as it gets heated from a low temperature to a higher temperature [1]. Commonly encountered ionizing radiation are  $\alpha$ -particles,  $\beta$ -particles,  $\gamma$ -rays, X-rays and neutrons. Thermoluminescence is not to be confused with the glowing observed in incandescence and luminescence [2] in response to vigorous heating to high temperatures. The temperatures involved in TL are far lower, typically less than 400°C, generally insufficient to heat a material to glow when seen with the naked eye. The response emissions follow a distribution that is characteristic of the sample. In principle, all that a TL instrument need do is vary the temperature of a sample between two thresholds while monitoring the intensity of the light output from the sample. In practice, however, there are many technical challenges

that must be identified and solved. The control of temperature over wide ranges with good measurement resolution and accuracy for the small dimensioned samples can be difficult, more so if the samples are in powder form. Secondly, the nonlinearity and output drift, spectral correlation errors in the optical detector all require careful characterization and compensation. At the very least, the temperature response of the sensor itself over the instrument operating range should be known. Several designs of TL instruments have been proffered over the last two decades at varying levels of complexity and proclaimed ease of usage - leading to solutions ranging from relatively low-tech to high-tech. Neelamegam et al [3] developed a system that permits recording TL data based on a legacy microprocessor, the 6502 from Motorola [4]. The version created by Molina et al [5] allows arbitrary heating profiles that includes logarithmic heating. Bhatnagar et al [6] catered for automatic control of heating with the added use of lightemitting diodes for additional sample excitation. Lyamayev [7] created a heating and cooling system with wide range of control, finer temperature regulation, simplicity and low cost. More recent contributions have attempted to employ advancements in embedded controllers [8]. The designs above have mostly relied on the classical, hard to find thermionic emission PM tube that is notoriously sensitive to temperature variations, exhibiting a noise figure that can only be kept low by careful cooling, usually at cryogenic temperatures [9]. Quilty et al [10], however, used platinum thermopile (PT100) resistors as both heating and sensing. Solid-state PM devices, though expensive, are now on the market, but TL instrument designs using them are yet to be seen. In addition, almost all the foregoing designs used third-party hardware proportionalintegral or proportional-integral-derivative (PID) control of the heating element, a detraction from true low-cost. Others used "bang-bang" servos - our own experiments with the "openloop" have shown that for the heater small "plant", the techniques of temperature regulation used often could not reproduce the reported accuracy, for example [3]. An additional difficulty in duplicating the work is that the code, hardware specifics and other beneficent aspects are described heuristically or are protected outright. A TL instrument is clearly a versatile tool with potential in the applied sciences and feasible, custom built alternatives have been sufficiently demonstrated.

In this article, we report on work - both ongoing and achieved - to construct a low-cost TL system based on the PIC18F2520 for both solid and powdered samples. The work relies on an original, low-noise and high sensitivity photo-diode sensor and conditioning circuitry that has not been reported before. Also, rather than rely heavily on hardware controller principles, the PID controller is implemented in controller firmware, with a resistive heating element directly driven by a MOSFET using pulse-width modulation (PWM) at 10 kHz. The temperature level is fed back using a k-type thermocouple. A few unique refinements extend the range of temperature control and measurement resolution stated above. For example, a rewound stepdown toroidal mains transformer outputting +180V/0.6A to drive the heater, enabling a wider temperature range for 0–100% duty cycles. The TL instrument is interfaced as full-speed to a Windows.net computer on the Universal Serial Bus 2 (USB 2.0) from which it derives power for the low voltage interfaces and amplifiers. The user commands are initiated on a program written in Microsoft's .net frameworks 1.0–4.0 [11]. Response data is sent to a personal computer for further processing through the USB 2.0 and stored in a file format that enables direct import into analysis environments like Microcal Origin 6, MS Excel, etc.

# 2. Description of the TL system

## 2.1. Overall system description

The designed TL system, Figure 1, consists of aluminium sample holder, a type k-thermocouple (chromel-alumel) for temperature feedback, a temperature-compensated, large area photodiode, several conditioning amplifiers, a PIC18f2520 microcontroller, the USB 2.0 interface and the multi-rail power supplies.



Figure 1. Block diagram of the TL system. The thermocouple is labeled "t/c".

#### 2.2. Temperature and output intensity measurement

The thermocouple circuit was constructed around the low-cost LM324N quad operational amplifier (OPAmp) shown Figure 2. Unfortunately the LM324 has no direct offset adjustment can exhibit input offsets of up to 7.0mV which, when combined with high forward gain, can produce substantial output error. Therefore, external offset trimming and room-temperature compensation using the LM35CZ device have been used to guarantee temperature measurement accuracy. The sample holder is an aluminium block of  $2.5 \times 1.7 \times 0.5$  cm<sup>3</sup> to which the k-type thermocouple was been affixed using a pressing metal plate and screws. A convenient 100W resistive heating source was implemented simply using a commercial pen-type soldering iron with a tip long and narrow enough to be inserted tightly into a compatible hole drilled into the sample holder. The photometer circuit, Figure 3, employs two OPA111BM OPAmp suitable where very low noise  $(7nV/\sqrt{Hz})$ , low input offset  $(\pm 50\mu V)$ , low drift  $(0.5\mu V/^{\circ}C)$  and low bias current ( $\pm 0.5$ pA) amplifiers are necessary [12]. This is mainly because extremely high gain is required to sense very low intensity counts. Furthermore, the BPW21R PN photodiode [13] is operated in the photovoltaic mode which has much higher intensity sensitivity than the photoconductive mode [14]. Solid state photodiodes also have sensitivity to temperature. The approach used here to assure repeatability is to preheat the diode before running baseline or sample characterization. This was done using a slightly overdriven TIP41C transistor bolted onto the photodiode array to maintain the detector temperature at around 30°C. The OPAmp conditioning circuits produce outputs between 0 and 5 volts to exploit the maximum resolution of the 10-bit analog to digital converter (ADC) in the PIC182520. The heating source, the +180V high-tension supply for the PWM controller and the sample holder were enclosed in a metal box measuring  $10 \times 10 \times 15$  cm<sup>3</sup>. The top lid of the box was hinged to allow sample access on the holder below. The photodiode was mounted on a block of aluminium on the underside of the lid to allow it to aim directly at a sample below when the lid was closed. A further precaution against stray light is to glue using Q-bond a hollow metal tube on the aluminium block that is directed at the sample. The conditioning circuits and the interfaces were in a separate metal box attached to the sampling arrangement by shielded wires, each wire not exceeding 20cm in length.

### 2.3. The software PID temperature controller

In PID temperature control the drive control signal, u(t), to the heating element is derived from the feedback from past and present temperature [15]. The controller first determines the error difference signal e(t) that indicates the difference between the target and the current temperature. It then generates a control signal that is a sum of three quantities, one proportional



Figure 2. Temperature measurement circuit.

Figure 3. Photometer circuit.

to e(t), another dependent on the time accumulated (integral) error and another dependent on how fast the error is changing with time i.e. the error derivative. Mathematically,

$$u(t) = K_p e(t) + \frac{K_p}{T_i} \int_0^t e(t)dt + K_p T_d \frac{de(t)}{dt},$$
(1)

where  $K_p$  is the proportional gain,  $T_i$  and  $T_d$  are the integral and derivative time constants respectively. To implement Equation 1  $T_i$  and  $T_d$  were first determined using Ziegler and Nichols open-loop tuning method [16, 17], essentially by driving the MOSFET at 10 kHz in open-loop and graphically determining the response times associated with "unit stepping" the duty cycle from 10% to 95% and steady-state. Figure 4 shows the PWM actuator used. A stable, optimal closed-loop controller was then found. Converting the system to z-transform space allowed digital implementation on the PIC18f2520 micro-controller using methods that are well covered in [18]. Linear heating was possible by ramping the control input from the software through the output range and achieved up to 20°C/min at resolutions of 0.5 °C. Heating rate is known to be significant on observed results [19].



Figure 4. Diagram of the PWM heater driver.

#### 2.4. Low-voltage power supply scheme

The amplifiers are powered from a flyback inverter built around a small flyback transformer and a 555-astable PWM driven in closed-loop between 115 kHz and 300 kHz. The USB 2.0 power load was configured as a low-power device requiring at most 100 mA (500 mW). The high frequency design allows the small overall dimensions while delivering a power efficiency of up to about 70% with good regulation (300 mW) and low output ripple. The latter is especially important for the low-noise, high-gain photometer circuit. The circuit was first simulated in LT Spice [20] and then verified by building. The use of common components improves repeatability and cost thereby making the design attractive.



Figure 5. Dual-rail amplifier power supply, giving  $\pm 9.26$ V with the 200 $\Omega$  loads shown.

# 2.5. USB data interface

User commands, instrument status and sensor response data are exchanged between the target PC and the TL system using USB 2.0 as a virtual communications port (VCP). The graphical user interface (GUI) was written in Visual Basic.NET and supports Microsoft's frameworks. It was successfully tested on operating systems ranging from Windows 98 to Windows 7. The role of the GUI is twofold. First, it provides a center from which commands may be issued e.g. the heating regime, handling calibrations and generally making the GUI friendlier, see Figure 6. Second, it logs the actual temperature and intensity to a file in a format that can easily be exported to third party spreadsheet applications.

## 3. Experimental Results

The initial tests of the TL instrument were carried out to establish the temperature profile of the photometer. This was done several times by running the sample holder empty from room temperature to about 350°C and logging the intensity counts. This established the baseline performance of the instrument to enable essential program parameters to be encoded into the .net console application. The application allows subsequent baseline calibration to be done. Table 1 shows the output of a sample run done for a polyethylene sample.

Parameter	Literature value [21]	Designed system
Glow peak temperature $/^{\circ}C$	385.0	388.5
Activation energy $/eV$	0.880	0.830

Table 1. Output of a sample run using a polyethylene sample.

# 4. Conclusions

The goal of creating a TL-instrument using common components has been largely met. The technical challenges were identified and simpler, alternative solutions to those in the literature were tried, tested and refined. While there remains a wide scope for further development, the basic frameworks have been put in place. The final product uses current computer interfaces with innovative peripheral components that permit ready duplication. The preliminary tests indicate considerable promise for serious scientific research, especially in environments where the performance to cost index is paramount. The scope of future work remains to implement more

Thermo-Luminescer	ice Instrume	nt Console p			
		ADC Inte	/ ensity Data	Running relative intensity count data display (Channel 0)	USB port COM3
Channel	ADC (0-1023)	Conversion			
Intensity	322	15174			
Sample Temp	112	76.3			
Photodiode Temp	137	29.6			
Room Temp	271	24.3			
Inverter rails	509	12.3			Start ADC
					Stop ADC
					Exit program
				Temperature /K	
lename: C:\Users\Oc	:aya\Docum	ents\solar.txt (D	lefault)		
B port has been set.	The ADC m	ay now be star	ted.		

Figure 6. TL glow-curve of a polymer sample in the .net program console.

accurate temperature compensation of the photodiode, wider detection range, and measurement resolution and accuracy. The gradual appearance of solid state photomultipliers offers further potential to improve the intensity detection apparatus significantly, without driving costs too much higher.

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