Asian Journal of Chemical Sciences

2(2): 1-7, 2017; Article no.AJOCS.32418

Building a Visible Light Spectrophotometer From NaI Detector

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Authors' contributions

This work was carried out in collaboration between all authors. Author KMSA designed the study, performed the statistical analysis, wrote the protocol, wrote the first draft of the manuscript and managed the literature searches. Authors LAJ and PHS managed the analyses of the study. All authors read and approved the final manuscript.

Article Information

DOI: 10.9734/AJOCS/2017/32418
Editor(s):
(1) Aleksandra Sentkowska, Heavy Ion Laboratory, University of Warsaw, Poland.
Reviewers:
(1) T. S. Verma, University of Botswana, Botswana.
(2) M. E. Medhat, Nuclear Research Center, Cairo, Egypt.
(3) Theocharis S. Kosmas, University of Ioannina, Greece.
Complete Peer review History: http://prh.sdiarticle3.com/review-history/18555

Original Research Article

Received 25th February 2017
Accepted 4th April 2017
Published 7th April 2017

ABSTRACT

A fluorescence spectrometer is used in the Measurement of a broad range of applications. The main parts of spectrometers are the photocathode connected to photomultiplier and the multichannel scalar (MCS) for Lifetime Measurements in Nanosecond to Seconds Range and multichannel analyzer (MCA) for the intensity and wavelength of UV/V light.

There is a wide range of sensors for light sensing applications: from a photomultiplier tube which gives a large voltage pulse for every photon it detects, to cooled thermopiles that absorb kilowatts of power.

The aim of this work is to develop and introduce Single Photon Detector as fluorescence spectrometer and chemiluminescence kinetics. For this purpose, the NaI crystal is removed gently from the detector cavity, used in gamma spectroscopy, so that the bare photocathode remained sealed to the photomultiplier. The capability of the system is mainly dependent on the sensitivity of the photocathode. The system is investigated to find a calibration curve of standard solutions. First,

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applying UV/V atomic absorption spectroscopy and measuring the time course of fluorescence signal from chemiluminescence absorbance. The results obtained are very encouraging to move forward towered more applications.

Keywords: NaI scintillation detector; photomultiplier tube; fluorescence spectroscopy; chemiluminescence kinetics.

1. INTRODUCTION

Optical instruments or light sensing devices are used to measure the intensities and wavelengths of the visible region of the electromagnetic spectrum (for instance eye is an essential part of the instrument for the eye collects the light from the grating and focuses the many colors on the retina). In general, fluorescence investigations are conducted with radiation having wavelengths ranging from the ultraviolet to the visible regions of the electromagnetic spectrum (250 to 700 nanometers) [1].

A fluorescence spectrometer, in the global, is used in the Measurement of a broad range of applications. The main parts of spectrometers are the photocathode connected to photomultiplier and the multichannel scalar (MCS) for Lifetime Measurements in Nanosecond to Seconds Range and multichannel analyzer (MCA) for the intensity and wavelength of UV/V light.

There is a wide range of sensors for light sensing applications: from a photomultiplier tube which gives a large voltage pulse for every photon it detects, to cooled thermopiles that absorb kilowatts of power.

With most fluorescence spectroscopy it is possible to record both excitation and emission spectra. An emission spectrum is the wavelength distribution of an emission measured at a single constant excitation wavelength. Conversely, an excitation spectrum is the dependence of emission intensity, measured at a single emission wavelength, upon scanning the excitation wavelength. Such spectra can be presented on either a wavelength scale or a wavenumber scale.

For an ideal instrument, the directly recorded emission spectra would represent the photon emission rate or power emitted at each wavelength, over a wavelength interval determined by the slit widths and dispersion of the emission monochromatic.

Chemiluminescence (CL) has a powerful application in analytical chemistry, mainly in the area of flow injection analysis. The use of CL as a detection principle permits quantitative determination of various compounds at low concentrations [2].Chemiluminescence (CL) is defined as the production of electromagnetic radiation (ultraviolet, visible or infrared) observed when a chemical reaction yields an electronically excited intermediate or product, which either luminesces or donates its energy to another molecule, which then luminesces [3].

The aim of this research is to develop and introduce a visible light spectrophotometer. For this purpose, the front aluminum face of the NaI (2"×2") detector is removed by turning machine. The NaI crystal, used for gamma spectroscopy, is removed gently from the detector cavity so that the bare photocathode, facing the light source, remained sealed to the photomultiplier. Photocathode is then covered by a black rubber sheet with a central aperture of diameter ~5 mm as an active area of light detection.

The developed system is investigated by the basic principles of spectrophotometry of standard solutions which used in reading off the unknown concentrations. Here a total signals emitted by standard chemical agent molecules are considered by multi-channel scaler (MCS).Or the number of photons detected during a specified integration time and the use of light transition and absorption to measure the concentration of chemicals in solution.

1.1 Building an Instrumentation for Fluorescence Spectroscopy

NaI(Tl) scintillation detectors are widely used in radiation spectroscopy: in nuclear medicine, for environmental monitoring and in many other applications. Even though it has poor resolution, a mutual spectral interference of environmental samples has been resolved [4]. For example but not limited to, it is highly efficient in the low level activity NORM samples [5,6].

The available electronics, of the developed fluorescent spectrometer, were based on separate components installed in Nuclear Instruments Module (NIM) bins. A schematic diagram of a typical general purpose fluorescence spectrometer is shown in Fig. 1. The detection system is composed of a detector, signal processor electronics, and data output display device such as multi-channel analyzer (MCA) and multi-channel scaler (MCS). The main part of the system is the Photomultiplier tube. PMT is a sensitive special vacuum tube that is capable of converting the incoming light photons absorbed by the surface of the photocathode into a very large number of electrons. PMT, supplied by high-voltage power 1,000 to 3,000 volts DC, is regulated to an appropriate operating Voltage. Photons incident on the photocathode get converted into electrons through the process of photoelectric effect. The electron is then made to accelerate and multiplied by structure of dynodes until these electrons reach the last dynode (called anode) where the resulting electrical pulse signal is measured by other electronic signal processor [7].

Photomultiplier tubes are generally used to detect the lowest light levels where the application demands the superior sensitivity of the photocathode. The photocathode can be made of a variety of materials, with different properties. The photocathode material is usually a mixture of alkali metals, which make the PMT sensitive to photons throughout the visible region of the electromagnetic spectrum. Typically the materials have low work function and are therefore prone to thermionic emission, causing noise and dark current, especially the materials sensitive in infrared [7].

The available photocathode in our laboratory (University of Zakho-Kurdistan region-Iraq) is Bialkali (Sb-K-Cs, Sb-Rb-Cs): cesium-activated antimony- potassium or antimony- rubidium alloy. It is a transmission type and has similar characteristics of Sb:Cs, with higher sensitivity and lower noise. Its favorable response to a NaI:Tl scintillator flashes, of wavelength 400-420 nm, make such types widely used in gamma spectroscopy and radiation detection, as shown in Fig. 2. It is limited to sensing ultra-violet to near infrared wavelengths (170 to 650 nm). Fig. 2 Shows the variation of quantum efficiency as a function of electromagnetic wavelengths of a bialkali ETI9266 PMT. where the used PMT is that of the curve (W) with UV glass [8].

Electronic counting connected to the PMT output are: Preamplifier and Amplifier, formulate linearly and amplifying PMT output to a maximum of 10 volts. Analog to Digital Converter (ADC) and Time-to-Amplitude Converter (TAC) convert an analog pulse into a digital form which can perform Pulse Height Analysis (PHA). This is merely counting the number of events with a certain pulse height. The output of the PHA can then be stored using either MCA or MCS which is subsequently display the spectrum on a screen [9].

1.2 Flow Injection System

Ethylene glycol is a clear, colorless, odorless, relatively non- volatile, viscous liquid. All chemicals and reagents used were of analytical grades. Distilled water was used for preparation of all solutions. The antifreeze solution containing (50%) of ethylene glycol was used to prepared (5000)ppm as stoke solution by take (1.0ml) of antifreeze solution and dissolving it in D.W. The resulting solution was diluted to (100ml) in a volumetric flask. Other solutions were prepared by serial dilution of the stock solution [10].

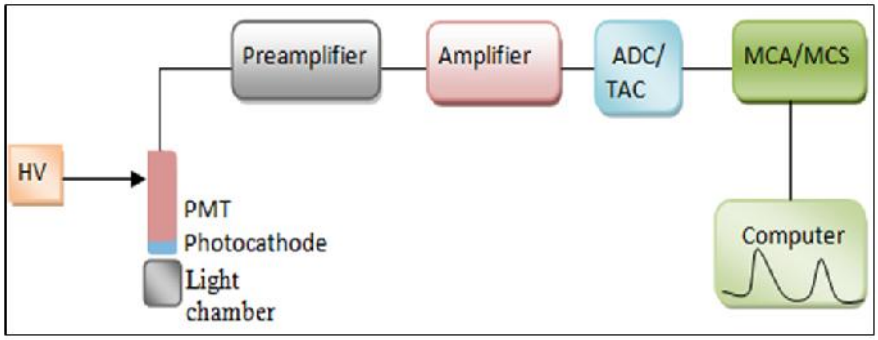

Fig. 1. Block diagram of a typical Fluorescence Spectroscopy

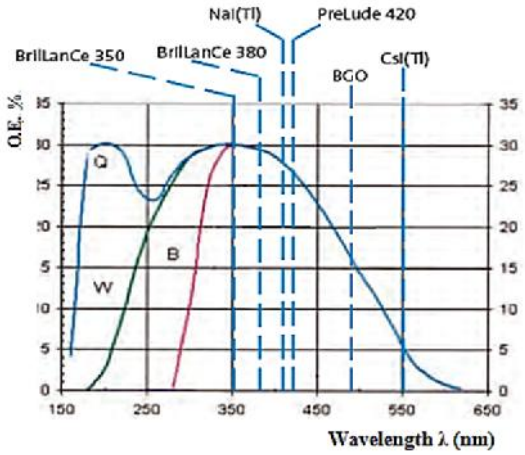

Fig. 2. Maximum emission points of various scintillators compared to quantum efficiency of a bialkali ETI9266 PMT with (B) Borosilicate, (W) UV glass and (Q) Quartz face plates (Q.E. data courtesy of Electron Tubes, Inc.)

The flow system used for the determination and chemiluminescence (CL) detection of ethylene glycol (EG), shown schematically in Fig. 3, is (Knauer D-14163) with variable sample volumes. Three peristaltic pumps (Watson marlow205u of 8 channels, variable speed) were used to drive the carrier and the reagent streams through the flow system. Each stream was pumped at a constant flow rate using silicone rubber tubes (0.8 mm I.D.). A needle valve is used to inject the sample (EG solution) into flowing carrier streams. At the entrance of the flow cell the reagent and lucignine solution are mixed to produce CL.

The mixing place of the flow cell was considered in front of the developed spectrophotometer detector. The light source which was blocked is positioned either perpendicular to the cell-detector path, for light absorption spectrum, or in the same path, for emission spectra. The flow cell and the light source are assembled inside a closed chamber as shown in Fig. 4. The light source used is a blue LED (HK-F5050X30-X-X-12) of peak wavelength 465-470 nm and the spectral bandwidth at Full Width at Half Maximum (FWHM) 40 nm [11].

Flow cell used in this work was made by winding the length of a plastic tubing (0.8mm I.D.) to form a coil with about 60µl volume, in a way that the reagent and lucigenine solutions are mixed exactly at the entrance of the cell. FWHM Peak height of the visible absorbance spectrum was measured for each EG sample concentrations (varied from 10% to 100%) by MCS.

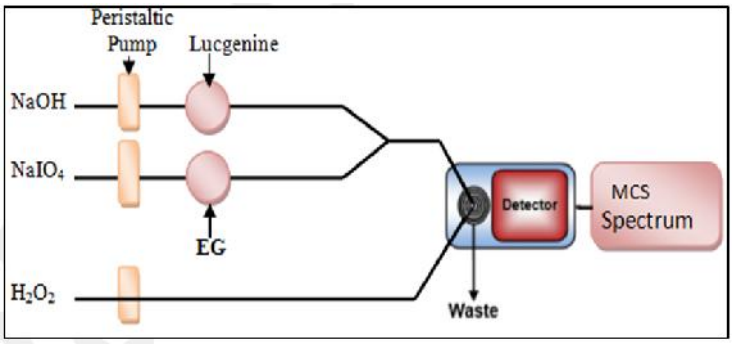

Fig. 3.The flow injection analysis used for CL determination of EG

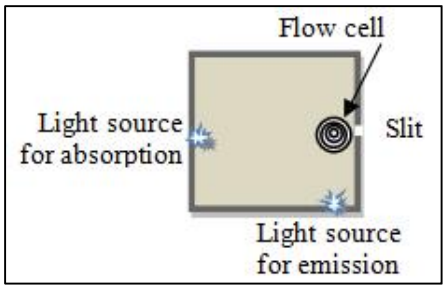

Fig. 4. The flow cell and the light source assembled inside a closed chamber

2. EXPERIMENT

The flow injection system is prepared for the chemiluminescence (CL) absorption phenomena of ethylene glycol (EG). Each standard solution is prepared as accurately as possible in identically the same fashion. The only difference between them being is their (10% - 100%) concentrations. Conditioning of flow injection system (Fig. 3), involved injection of initial concentration of 0.001 mol/L lucigenin into the carrier stream (0.5 mol/L NaOH) which was mixed with a combined 15 mg/L ethylene glycol and 0.007 mol/L sodium periodate solution, which are previously merged and trapped for five minutes in a reaction coil by stopping a peristaltic pump. The resulted stream is then mixed with 0.3 mol/L H_2O_2 solution at the entrance of the flow cell. The mixing position of the flow cell is fixed inside the chamber in front of the detector connected to fluorescence spectrophotometer system, as in Fig. 4.

The Fluorescence Spectroscopy is adjusted for the measurement of the evolution of the light source emission spectrum with time. This is a resolving single photon pulses selected from a limited time interval which is referred to the Time Resolved Emission Spectra (TRES). For this purpose, the available model MCD-2E, PC-card MCA with 2-Input MCS is used. Among all features of this model, it is a programmable sweep/memory with range input: 256, 512, 1k... 128k channel and Dwell time input: 1 µs to 160s, Programmable in steps of N x 10^M x1 µs (N= 1... 16; M= 0... 7).In this experiment, the MCD-2E is configured as single MCS, 1 s dwell time, range 1024 channel, one complete sweep and internal clock [12].

3. RESULTS AND DISCUSSION

In the present work ethylene glycol were determined by a method using Lucigenine – H_2O_2-$NaIO_4$ in basic media as sensitizer. To obtain the optimum values of chemiluminescence intensity for the determination of EG, all chemical and physical parameters affecting the flow system were examined. Their values are given in Table 1.

The analytical signal was calculated as sample output minus blank (change in CL intensity ΔI). As light passes through a sample, its power decreases as some of it is absorbed. This attenuation of radiation is described quantitatively by Intensity of light transmitted through the sample solution I_T and Intensity of the incident light I_0. The more common unit for expressing the attenuation of radiation is absorbance or optical density of the EG(A), which defined as:

$$A = -\log \frac{I_T}{I_0}$$

Common quantitative analyses to establish the linear relationship between absorbance and analyst's concentration C is the Beer-Lambert law [1].

$$A = -\log \frac{I_T}{I_0} = \varepsilon bC$$

where ε is the molar absorptivity in $cm^{-1}M^{-1}$ and b is the sample's thickness in cm.

The mean of three injections for each EG concentration were used. As the absorbed intensity peaks obtained are Gaussian, MCD-2E calculates their counts by Full Width at Half Maximum (FWHM) to obtain counts x10^7fixing FWHM at 27 s for all peaks.

Table 1. Optimum conditions for determination of EG

Parameters	Optimum value
H_2O_2	0.3 mol/L
Periodate concentration	0.007 mol/L
Lucigenin concentration	$1.0x10^{-3}$mol/L
NaOH concentration	0.45 mol/L
Flow-rate	5 ml/min
Reaction time (stopped time)	5 min
Reagent volume	30µL

Fig. 5 shows the corresponding bell-like curve, or the normal curve fitting, for the calibration graph obtained for EG by plotting the graph of absorbance A vs. EG concentration. As the performance characteristics must be or expected, the CL absorbance increases with increasing EG concentration up to 50-60% concentration with a correlation coefficient 0.914. While the absorbance decreases for higher concentration. In other words, the CL intensity decreases with increasing EG concentration up to 60% then increases for higher concentration. The obtained results are in good agreement with other previous works [13].

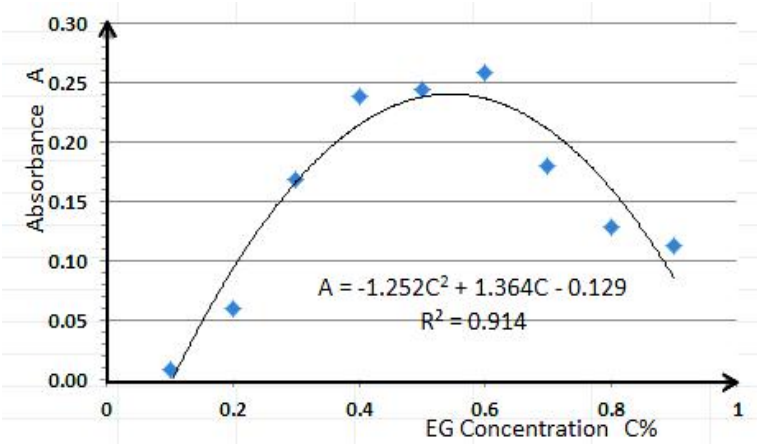

Fig. 5. Calibration graph for the variation of the Absorbance verses ethylene glycol Concentrations ($A = -1.252C^2 + 1.364C - 0.129$ with $R^2 = 0.914$) for a standard chemical solution

Finally, it is worth mentioning that, to improve the system for other spectrometric applications, the excitation and emission monochromator must be designed with plane gratings for accurate focus at all wavelength and minimum stray light. Such an improvement will be addressed in a future work.

4. SUMMARY AND CONCLUSIONS

The measurement of light absorption is a fundamental aspect of fluorescence spectroscopy. The developed Light Spectrophotometer applied to the determination of EG in antifreeze samples shows a good performance. The photocathode, bialkali ETI9266 PMT used is susceptible to ultra-violet to near infrared wavelengths and suitable for the determination and chemiluminescence (CL) detection. The proposed technique offers the advantages of simplicity, rapidity, and good sensitivity for the determination of EG.

This work established a relationship between the chemiluminescence intensity and EG concentration, pointing out that the emission intensity depends on the concentration of the sample (analyte) and distinguishes the speed of the chemiluminescence reaction by monitoring of the transient light emission. The system can be applied for any other chemiluminescence reaction.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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Peer-review history:
The peer review history for this paper can be accessed here:
http://prh.sdiarticle3.com/review-history/18555