

DETERMINATION OF MANGANESE IN GRAPE WINE USING LIGHT EMITTING DIODE AS LIGHT SOURCE.

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Abstract:

Many methods are available for determination of the manganese in different matrices. For example there are, X-ray fluorescence, voltammetry, atomic absorption spectrometry (AAS) and UV-VIS spectrophotometry. Atomic absorption spectrometry is a technique widely used for quantification of manganese, but the apparatus used is more expensive than UV-VIS spectrophotometry. Colorimetric method, Periodate colorimetry, persulphate spectrophotometry, Formaldoxime (FAD) method and Pyridylazonaphthol (PAN) etc. but all these methods are quite expensive and require expert handling. However this method is very simple and very easy to perform. But it doesn't have its limitations. In this paper we describe an application of LED's to the determination of Mn in Wine samples. A simple apparatus using LED's as light source is described. The advantages of this apparatus are its low cost, versatility, and easy to build characteristics.

Index terms: LED (light emitting diode), Mn, Grape wine, PD Amplifier, LF 356 IC.and digital multimeter.

Introduction:

Manganese is essential element in all known living organisms. Many types of enzymes contain manganese. Manganese is the fifth most abundant metal in the earth's crust and also it is considered to be 12th most abundant element in the biosphere. It is widely spread in soil, sediment, water, and biological materials. Although manganese is essential trace element for human body and other species of the animal kingdom as well as for plants. It is serving synthesis and activation of several enzymes including kinases, phosphatases and oxidoreductases. It plays an important role in the brain. However, excessive levels of this metal are harmful to the body, causing toxicity to the respiratory, cardiac and reproductive systems. The average human body contains about 12 milligrams of manganese. We take in about 4 milligrams each day from such foods as nuts, bran, wholegrain cereals, tea, and green vegetables. Without it bones grow spongier and break more easily. It is also essential for utilization of vitamin B1. In man chronic manganese excess affects the central nervous system, with the symptoms resembling those of Parkinson's disease. This is the reason why manganese belongs to highly toxic heavy metals.

The human body contains about 10-20 mg of Mn of which 5-8 mg are turned over daily. Manganese deficiency in human body is very rare because of its widespread presence in the human diet. Manganese deficiency has been related with skeletal abnormalities, ataxia, and alterations of reproductive function as well as lipid and carbohydrate metabolism, osteoporosis.

Various techniques have been utilized for detection of manganese including X-ray fluorescence, voltammetry, atomic absorption spectrometry (AAS) and UV-VIS spectro-photometry. Atomic absorption spectrometry is a technique widely used for quantification of manganese, but the apparatus used is more expensive than UV-VIS spectrophotometry.

In this paper we describe an application of LED'S to the determination of manganese in grape wine samples. A simple apparatus using LED'S as light source is described. The advantages of this apparatus are very low cost, versatility and easy to build characteristics and also with easy available chemicals and instrumentation, simple and direct application to real samples.

LED'S with different wave lengths provide an inexpensive and easy to use light source in the visible region. A photometer operating with LED'S is cheap and versatile for the analysis of many different kinds of samples.

LED's have characteristic wavelengths emission maximum and band widths. The LED's have some disadvantage, since without monochromatic or narrow band filters, the measured intensive are due to all the light reaching the deviator, regardless of its wave length. As is well known, this usually gives rise to deviations from the Beer's law. In this article the LED used is LF 356 IC. The specifications of LF356 IC and information is as given bellow.

LF356 IC specifications and information:

- Low input bias current: 30 PA
- Low input offset current: 3 PA
- Low input offset voltage: 1.0 mv
- Temp compensation of input offset voltage: 3.0 v/c
- Low input noise current 0.001PA/HZ
- High input Impedence: 1012 Ω
- High common mode rejection ratio: 100 db
- Large DC voltage gain: 106 db

Applications: LF Series is suggested for all general purpose FET input amplifier requirements where precision and frequency response flexibility are of prime importance.

Experimental Procedure

For our instrument, a high brightness yellow LED was obtained from a local supplier. A regulated 12V power supply together with a series combination of a 1000 Ω resistor and a 10k Ω potentiometer was used as current source for the LED. The current can be adjusted in the 1-20 mA range, assuming a $V_f = 1.8\text{v}$. A digital multimeter was included to adjust the current available and must always be included to avoid permanent damage to the LED due to an excessive current passing through it. For a flexible operation, the emitted light was conducted through a fiber optic. A Polymeric type fiber with a 1 mm diameter was selected because of its low cost and low loss at the working wavelengths. The fiber is covered with a black polyethylene cladding. A homemade plastic coupling was used to join the fiber to the LED. Figure (1) shows the Circuit diagram of the photo detector, LED, fibre optic and power supply. The detector used was a light to voltage optical sensor this is a very small combining a photodiode and a trans impedance amplifier with a 37 M Ω integrated feedback resistor on a single IC. In appearance it is similar to a conventional low power transistor also having three leads used as ground, power supply and output.

A diagram of the photometer is shown in fig (2). The voltage signal produced by the incident light incident on the photo diode was read by the digital multimeter. Thus intensity data are the primary data obtained. To transform these data into absorbance, a logarithmic transformation is needed. Taking into account the well-known relation of the primary data into the final absorbance values as shown in equation (1)

$$A = \log \left(\frac{E_0 - E_\infty}{E - E_\infty} \right) \text{----- (1)}$$

E = is the potential measured with the sample placed into cell holder.

E_0 = is the potential measured with the reference solution. And

E_∞ = is the potential in the absence of light.

A present limitation of LED applications in the photometry is that LEDs for only a few visible wavelengths are available so that in principle only species absorbing these can be investigated.

HAZARDS: There are no significant hazards associated with the use of LED's. LED's do not contain toxic material like mercury which is used in fluorescent lamps.

Figure (1): Circuit diagram me of the photo detector, LED, fibre optic and power supply

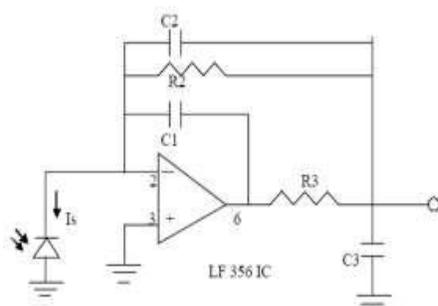


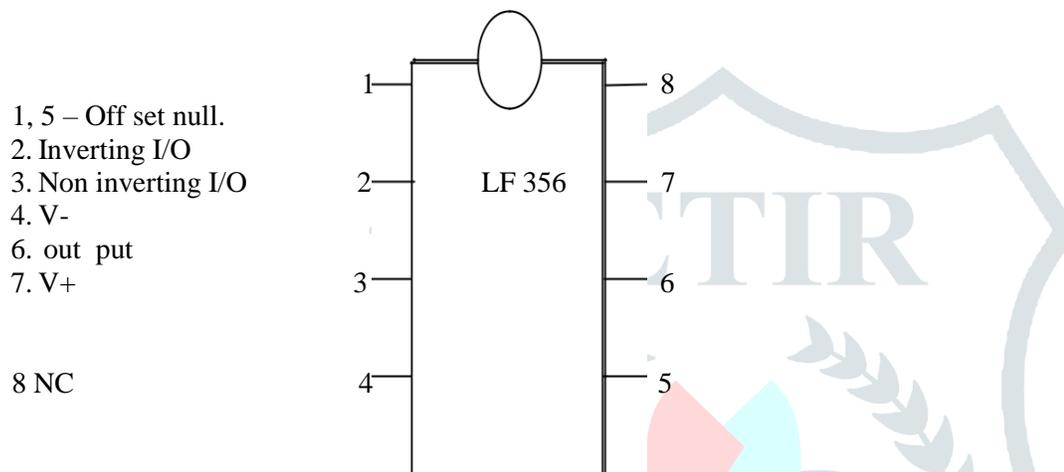
Fig (2) feedback PD amplifier

$$R2 = 37 \text{ M } \Omega ; R3 = 1\text{K } \Omega;$$

$$C1 = C2 = 0.5 \text{ pf};$$

$$C3 = 10 \text{ nf};$$

Fig (3) Pin diagram of LLF 356 IC



Preparation of wine sample:

50 mL of wine samples were transferred into a 100 mL beaker and then heated to remove ethanol. The samples were nitrated by heating until there was no smoke volatilizing with 2 mL of concentrated sulphuric acid and 2 mL of concentrated nitric acid. After cooling, the solutions were transferred into a 50 mL volumetric flask and diluted to the marks with water.

A suitable volume of Mn (VII) working solution was transferred into a 10 mL colorimetric tube with a glass stopper. Then, 1.5 ml H_2SO_4 solution and 1.5 ml acid fuchsin solution were added. The solution was diluted to the mark with water and kept aside for 10 min at room temperature. The reagent blank solution which was absence of Mn (VII) was prepared as above. The absorbance “a” of the solution was measured at 550 nm with 1 cm cells by using reagent blank solution as reference. . The solutions thus prepared are taken into cuvette, and then the light from the LED is passed through the solutions. The readings should be taken from the digital multimeter. The apparatus should be arranged as per the circuit diagramme (1).

Filling and wiping cuvettes:

Glass cuvettes are used to hold either samples or blank solutions. These cuvettes should be filled with the solution. So that the internal light beam passes entirely through the liquid or not through the air. Kim wipe tissue papers should be used to remove any droplets or finger print marks on the outside of the cuvettes

Inserting cuvettes:

Cuvette should be gently inserted into the sample holder itself so that any imperfections in the glass cuvettes are in the same position, relative to the light source, each time cuvette is inserted. This minimizes errors due to flows in the glass cuvettes. For the most accurate quantitative work, a single cuvette is used for all reading of blanks and samples. Cuvette should be removed as soon as the reading is recorded to prevent damage to the photodiode. The word “SAMPLE” refers to the various blue test or sample solutions. The word “BLANK” refers to a cuvette filled with double distilled water that is the solvent for the different solutions.

Sample collection: The grape wine samples are collected from the local suppliers. All chemicals were of analytical reagent grade and all the solutions were prepared with doubly distilled water. A manganese (VII) stock standard solution (*ca.* 0.1 g L^{-1}) was prepared by KMnO_4 and calibrated by sodium oxalate. Working standard solutions were prepared by appropriate dilutions when required. Acid fuchsin solution (0.1 g L^{-1}) and sulphate solution (0.2 mol L^{-1}) were used

Methodology:

Many methods are available for determination of the manganese in different matrices. For example there is X-ray fluorescence, voltammetry, atomic absorption spectrometry (AAS) and UV-VIS spectro-photometry. Atomic absorption spectrometry is a technique widely used for quantification of manganese, but the apparatus used is more expensive than UV-VIS) spectrophotometry, Colorimetric method etc. but all these methods are quite expensive and require expert handling. However this method is very simple and very easy to perform. But it doesn't have its limitations. In this paper we describe an application of LED's to the determination of Mn in ground water samples. A sample apparatus using LED's as light source is described. The advantages of this apparatus are its low cost, versatility, and easy to build characteristics, easy available chemicals and instrumentation, simple and direct application to real samples.

HAZARDS: There are no significant hazards associated with the use of LED's. LED's do not contain Toxic material like mercury which is used in fluorescent lamps.

RESULTS AND DISCUSSION:

We present here a low cost photometric apparatus based on LED technology and described an under graduate laboratory experiment for determining the manganese in grape wine. Since LED research is an active in continuously expanding area, the implementation LED's in new instrumentation seems quite meaningful. In this experiment a calibration curve was created by plotting absorbance vs concentration in excel. The method was used for the determination of manganese in several kinds of wine. The results show that the method is easy,

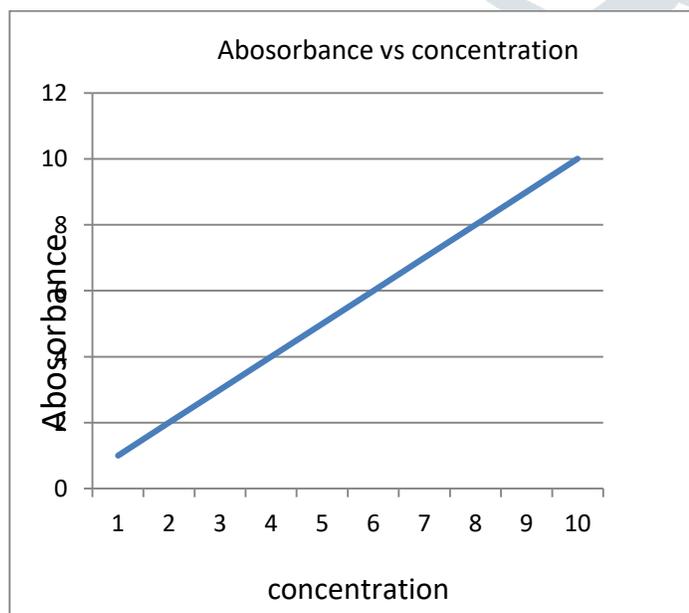
rapid and inexpensive. The Beer's law states that absorbance and concentration are directly proportional to each other experimentally it was true over narrow concentration ranges and in dilute solutions. The standard solutions in narrow concentrations will exhibit linearity. Graph of the Absorbance vs concentration is shown in fig (4). In this method also showed that calibration curve is linear drawing absorbance vs concentration and it followed Beer's law. Hence we adopted this method to determine manganese in grape wine.

Table No (1) shows the concentration and absorbance values table.

For reference Solution $E_0 = -209.6$ mv

S.No	Concentration $\times 10^{-2}$ M	Voltage		Absorbance
		E	E_∞	
1.	1	-192.1	1.5	0.0374
2.	2	-176.4	1.5	0.0743
3.	3	-161.4	1.5	0.1125
4.	4	-148.1	1.5	0.1495
5.	5	-135.6	1.5	0.1874
6.	6	-124.5	1.5	0.2240
7.	7	-114	1.5	0.2618
8.	8	-104.4	1.5	0.2995
9.	9	-95.7	1.5	0.3368
10.	10	-87.7	1.5	0.3741

Fig(4): Graph of the Absorbance vs concentration.



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