

Development of Differential Colorimetric Analyzer for the Measurement of Iron in Water Solution

Pravin K. Bhadane, Suchita P. Bhangale

Department of Electronics, Nowrosjee Wadia College, Pune (India)

Abstract— An equipment has been developed for the analysis of iron in waste water. The photoelectric section of the equipment has been developed on the basis of Beer-Lambert law. Monochromatic light emitting diode (LED) with domed lense was used as source of light. The intensity of incident light beam was varied by the software controlled pulse width modulator. The detector was designed by using sensitive photoconductive light dependent resistor (LDR). Operational amplifier based difference amplifier interfaced to the microcontroller (μC) has been used for the measurement of relative absorption. The instrumental and the experimental errors introduced due to the absorption by cuvette and the reagent respectively were compensated by the software techniques. In addition to above functions, the software performs the operations of calculation of absorptivity coefficient, detection of analyte species and estimation of concentration of analyte. The developed equipment has been successfully used for the analysis of iron in waste water. The software program can be easily modified for the similar study of other analytes.

Index Terms—Beer-Lambert law, LDR, LED, Iron, Microcontroller.

I. INTRODUCTION

Iron in its reduced and oxidized state has positive charges of two and three respectively [1]. The charge on iron helps him to carry the condensed gases in some processes. For example, iron in hemoglobin helps to carry the oxygen during blood circulation. Similarly, iron is found in enzymes which are involved in oxidation – reduction reaction for making amino acids and hormones. The absorption of iron in the intestine of human beings takes place with the help of mucosal ferritin and transferrin protein molecules [2]. The losses of iron in human body occur via bleeding and urine. If absorption cannot compensate for losses due to low dietary intakes, then iron deficiency is detected. Women are especially victim to iron deficiency due to repeated blood losses during menstruation [3, 4]. The iron deficiency is characterized by decrease in the quantity of iron being transported within the body. It is necessary to measure the absorption and concentration of iron for the diagnosis of its deficiency [5]. The measurement of concentration can be achieved with the help of μC based automated colorimeter. Generally, it is equipped with LED, LDR, and supporting electronic devices [6, 7]. The aims of the present work were (i) to design and construct electronic circuit of the MSP μC based differential photoelectric colorimeter, and (ii) development of software for the control and measurement of the incident and transmitted light respectively, determination of characteristic wavelength (λ_{max}) and absorptivity coefficient, calculation of absorption and estimation of

concentration. The rest of article is organized as follows: The Beer- Lambert method of photoelectric analysis is discussed in section II and design of electronic circuit and its description is discussed in section III. The results are discussed in section IV. The steps in software design are discussed in section V. Finally, the conclusions and future scope are discussed in section VI.

II. BEER-LAMBERT METHOD

LED produces a beam of monochromatic light which passes through the solution placed in the cuvette. The light transmitted by the solution falls on the LDR. The resistance of the LDR is inversely proportional to the intensity of light falling upon it. The change in resistance of LDR is measured by the microcontroller unit and converted into intensity. The Beer – Lambert method requires the intensity of the incident beam, I_0 , and transmitted light, I , for the calculation of absorption [8]. If some of the incident light I_0 , is absorbed by the solution, then intensity of the beam reaching the LDR, I , will be less than I_0 . The difference amplifier subtracts the two intensities and the difference is applied to the ADC section of the microcontroller. The μC converts the difference into either transmittance (T) or absorbance (A). The fraction of I_0 that passes through the solution is called Transmittance (T) and is given by,

$$T = \frac{I}{I_0} \quad (1)$$

The absorption ‘A’ can be obtained from the transmittance given by,

$$A = -\log_{10} T, \quad A = \log_{10} \frac{I_0}{I} = \log_{10} \left(\frac{I_0}{I} \right) \quad (2)$$

T is also given by the Beer’s law [8],

$$T = 10^{-\epsilon d C} \quad (3)$$

Where, ϵ = absorptivity coefficient, d = path length of light beam (cm); C = concentration of absorbing analyte. The logarithm of (3) yields the relation between ‘A’ and ‘C’.

$$\log_{10} T = \log_{10} (10^{-\epsilon d C}) = -\epsilon d C$$

$$A = -\log_{10} T = \epsilon d C \quad (4)$$

Each analyte has particular characteristics wavelength (λ_{max}) at which it absorbs maximum light (A_{max}). The λ_{max} can be obtained by varying the wavelength of light. In automated colorimeter it is easy to change the wavelength and to find the A_{max} from the analysis of wavelength versus absorption graph. From the graph, it is possible to determine the exact wavelength corresponding to the maximum absorbance (A_{max}). The absorbance, ‘A’, is also called optical density (OD). Since ϵ and d are constant, they can be combined into a new constant, k . The substitution of ‘k’ into (4) results into (5) which is similar to the equation of line (6)

$$A = kC$$

$$(5) \quad \text{Light Dependent Resistor}$$

$$y = mx + b$$

$$(6)$$

Comparison of (5) and (6) gives,

$$y = A, x = C, m = k \text{ and } b = 0.$$

Beer's law then states that a plot of absorbance versus concentration will give a straight line passing through the origin (see Fig. 1). The slope of the line depends upon the type of analyte and it is required for the calculation of unknown concentration.

To find the concentration of unknown solution, it is necessary to plot the calibration curve [9]. The slope of the curve gives value of 'k'. The concentration can be obtained by substituting 'k' into (5),

$$C = A_{\max} / k.$$

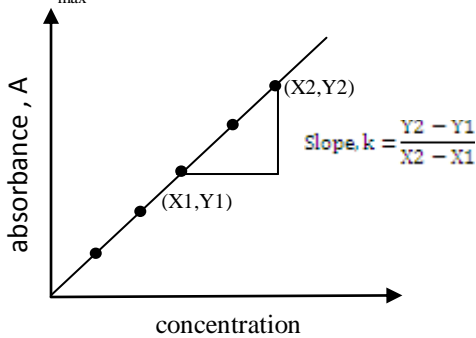


Fig.1. Plot of concentration versus absorbance (A)

III. ELECTRONIC CIRCUIT DESIGN

The block diagram of differential colorimeter is shown in Fig. 2. The important blocks are PWM, cuvette and difference amplifier. The complete circuit diagram including values of components is shown in Fig. 3. It comprises four main sections- photoelectric sensor, microcontroller, signal display unit, and power supply. Functions of each unit are discussed below.

A. Photoelectric Sensor

It is based on Beer-Lambert law [8], which deals with relationship between transmittance of light and concentration of an absorbing species. It consists of four units: LED, Cuvette, LDR, and Difference amplifier, as shown in Fig. 3. The details of each unit are given below.

Light Emitting Diode

It is the source of monochromatic light. As the name indicates, it emits visible light when its P-N junction is forward-biased. The colour of the emitted light depends on the type of material used in making the LED. For example, GaP and GaAsP produce red and yellow light respectively [6]. LEDs are manufactured with domed lenses in order to improve the focus of light beam. They are always encased in order to protect their delicate wires and it has life more than 10,000 hours. Since LEDs operate at dc voltage levels from 1.6V to 3.3V, they can be powered by the ordinary laboratory dc power supply. In the present work, red (660nm), green (540), blue (460 nm), and yellow (590 nm) color. LEDs are used as a source of light.

It is used for the measurement of intensity of light. The resistance of an LDR is very high in dark, but when it is illuminated with sun-light then resistance drops dramatically. In the present study, separate LDRs are used for the reference and sample solution. The variation in resistance (R) with intensity (I) is given by,

$$\log_{10} (R) = -K \log_{10} (I) + C \quad (7)$$

Where, K and C are constants which are dependent on wavelength and physical dimensions [8]. LDR has the advantages of long life and it requires simple electronics.

Cuvette

It is made from the easily available, low cost glass material. LED is fitted at one side of cuvette and its light is made to pass through the sample placed in cuvette. The transmitted light passed through the sample, struck the opposite side of the cuvette where the LDR is mounted.

Difference Amplifier

The light produced by LED is passed through the solution. The solution absorbs different amount of light depending on the type and concentration of material in the solution. LDR is mounted on the other side of the cuvette. As the transmitted light struck the LDR, its resistance changes, and current passing through it also changes. The difference amplifier subtracts the absorption due to blank from the sample one. An operational amplifier (OP-AMP) is configured as difference amplifier (DA). The IC CA3140 operational amplifier is selected to design the DA because of its very low input bias current (20 pA) and the input offset voltage (5 mV). The sensitivity of DA can be controlled by varying the feedback resistance. Some solutions may have very strong absorption characteristics, resulting in less transmitted light and less current in LDR. To amplify it to a sufficient level, small value of feedback resistor is required. While at the same time to measure the small currents corresponding to the highly diluted solutions, a large feedback resistance is required. To balance the need of different gain values, variable resistor of value 1 MΩ is used in the feedback [6].

B. Microcontroller Unit

The MSP430f2013 is a low power, high performance 8-bit microcomputer with 2Kbyte of flash programmable, and erasable read only memory. The device is manufactured using high density integrated technology. The on-chip flash allows the program memory to be reprogrammed in system or by external memory programmer. By combining the 8-bit CPU with flash and ADC on a monolithic chip, the microcontroller is a powerful microcomputer, which provides a highly flexible and cost effective solution to embedded system applications [10].

C. Signal Display Unit

The values of absorption and concentration are displayed on 16 x 2 alphanumeric liquid crystal display (LCD) based

on HD44780 controller. The backlight feature of the LCD makes it readable even in low light conditions. The LCD is used here in 4-bit mode to save the microcontroller's port pins. Usually the 8-bit mode of interfacing with a

microcontroller requires eleven pins, but in 4 bit mode the LCD can be interfaced to the microcontroller using only seven pins.

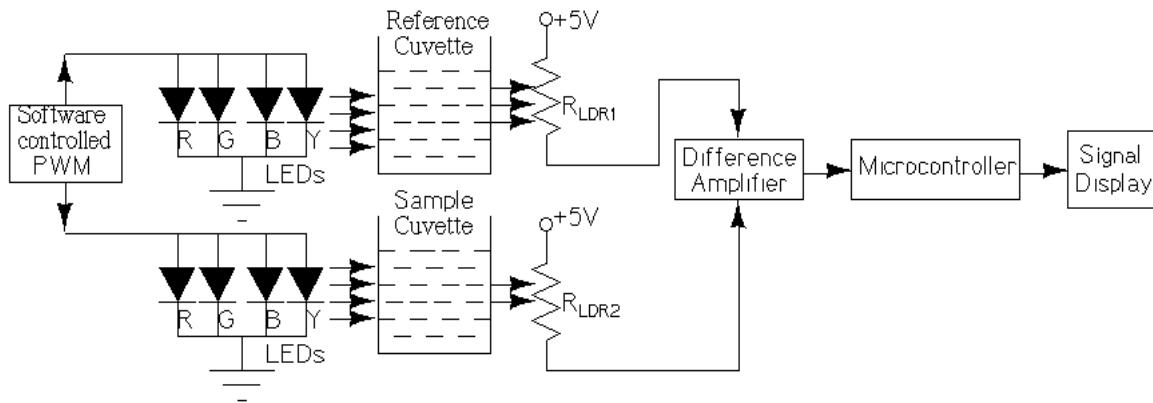


Fig.2. Block diagram differential colorimetric analyzer

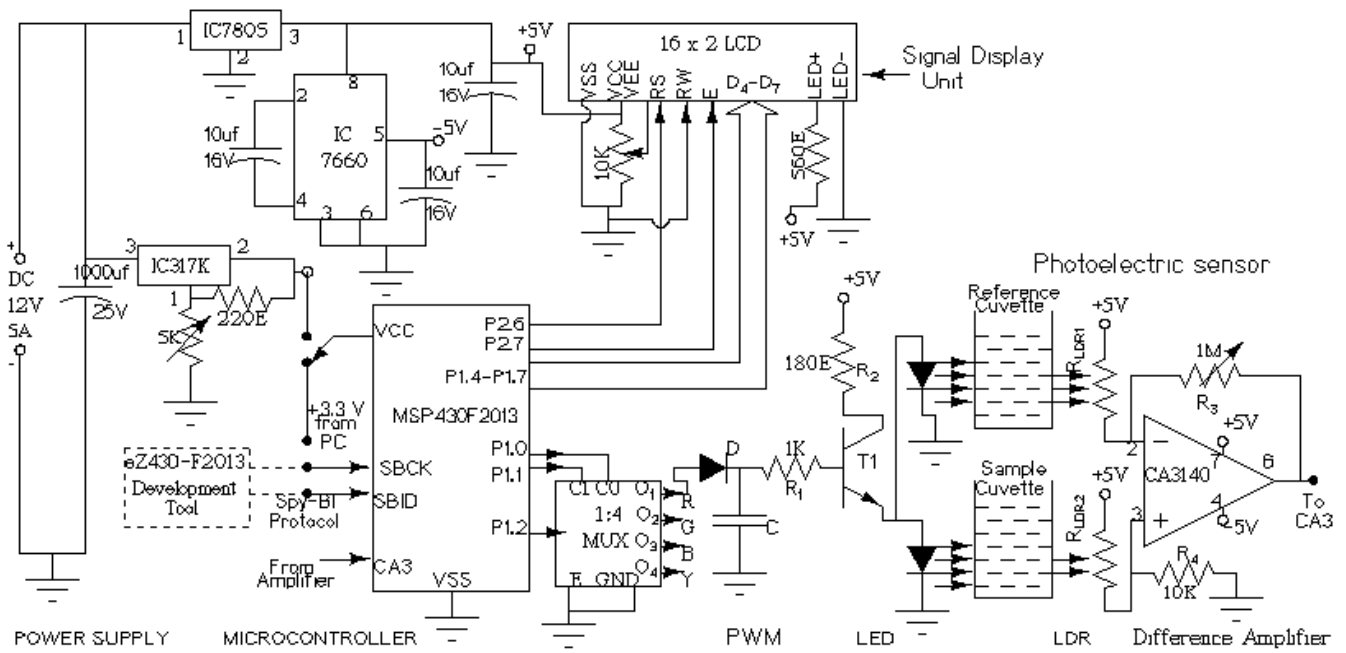


Fig.3. Circuit diagram differential colorimetric analyzer

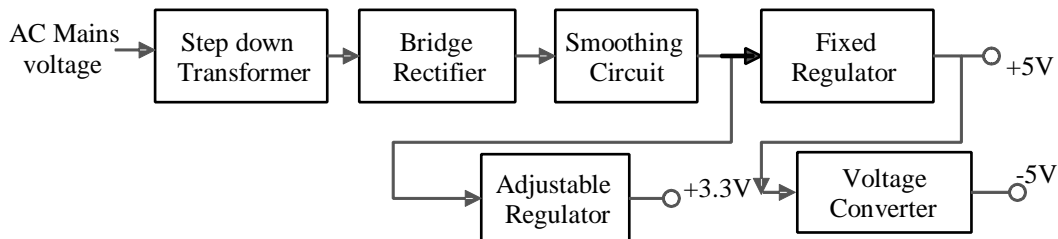


Fig. 4. Regulated dc power supply

D. Power Supply Unit

The power supply is designed to convert high AC mains voltage to a suitable low dc voltage supply required for the electronic circuits [6]. A power supply can be broken down into a series of functional blocks as shown in Fig. 4. The function of blocks is discussed below. Transformer steps down the high voltage AC mains to low voltage AC. Rectifier converts AC to DC, but its DC output is pulsating. An electrolytic capacitor works as smoothing device. It removes the unwanted ac ripple signal and makes the dc signal steady. Regulator maintains the dc voltage constant. IC 7805 is a fixed regulator to provide +5V. The IC 317 is the adjustable regulator to supply constant +3.3V. The positive dc voltage is converted into negative by the voltage convertor IC 7660.

IV. RESULTS AND DISCUSSION

The concentration of unknown solution is determined from the calibration curve [10]. In order to draw the curve, the first step is to find wavelength at which maximum absorption occurs. This wavelength is called the characteristics wavelength (λ_{max}). Five different concentrations of FAS (ferric ammonium sulphate) with salicylic acid solutions were prepared. Using highest concentration solution of FAS, λ_{max} was determined and it was found to be 540 nm (see Table I). The graph of wavelength verses absorbance is shown in Fig. 5

Table I. Absorbance values at different wavelengths

ml of 0.001M FAS solution	Total Volume, ml	Concentration of FAS /10 ⁻⁴ M	Absorbance at 540nm
5	25	2	0.17
10	25	4	0.36
15	25	6	0.55
20	25	8	0.72
25	25	10	0.88
Sample solution	25	Unknown	0.42

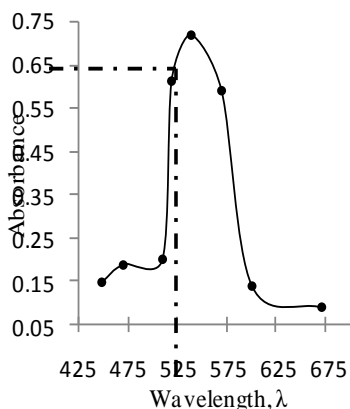


Fig. 5 Graph of wavelength (nm) versus absorbance.

The plot of λ versus absorbance is shown in Figure.5. It is revealed from that the maximum absorbance is at 540 nm, which is the wavelength (λ_{max}) of green colored light. In next step the absorbance values were measured for the series of known standard solutions [11]. At λ_{max} , absorbance of different concentration solutions of FAS as well as unknown concentration solution of FAS was measured and is depicted in the Table II. The calibration curve of concentration versus absorbance is shown in Fig. 6.

Table II. Absorbance by standard solutions of different concentrations

Wavelength, λ (nm)	Absorbance
450	0.15
470	0.19
510	0.2
520	0.61
540	0.72
570	0.59
600	0.14
670	0.09

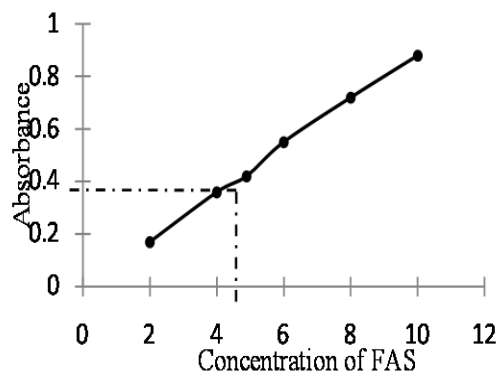


Fig. 6. Calibration curve of concentration versus absorbance

For a given unknown solution the measured absorbance is 0.42. The concentration of unknown solution is determined from the calibration curve as shown in the above Fig. 6. The value of unknown concentration from the curve is found to be 4.89 M.

V. SOFTWARE DESIGN

The source program is written in C- language and compiled using IAR IDE. The flow-chart shown in Fig.7. The hex file is burnt into the microcontroller with the use of eZ430f2013 development tool. The software has the functions to: adjust the zero absorption for blank solution, measure absorption of sample solution, find the characteristics wavelength, and to find the concentration.

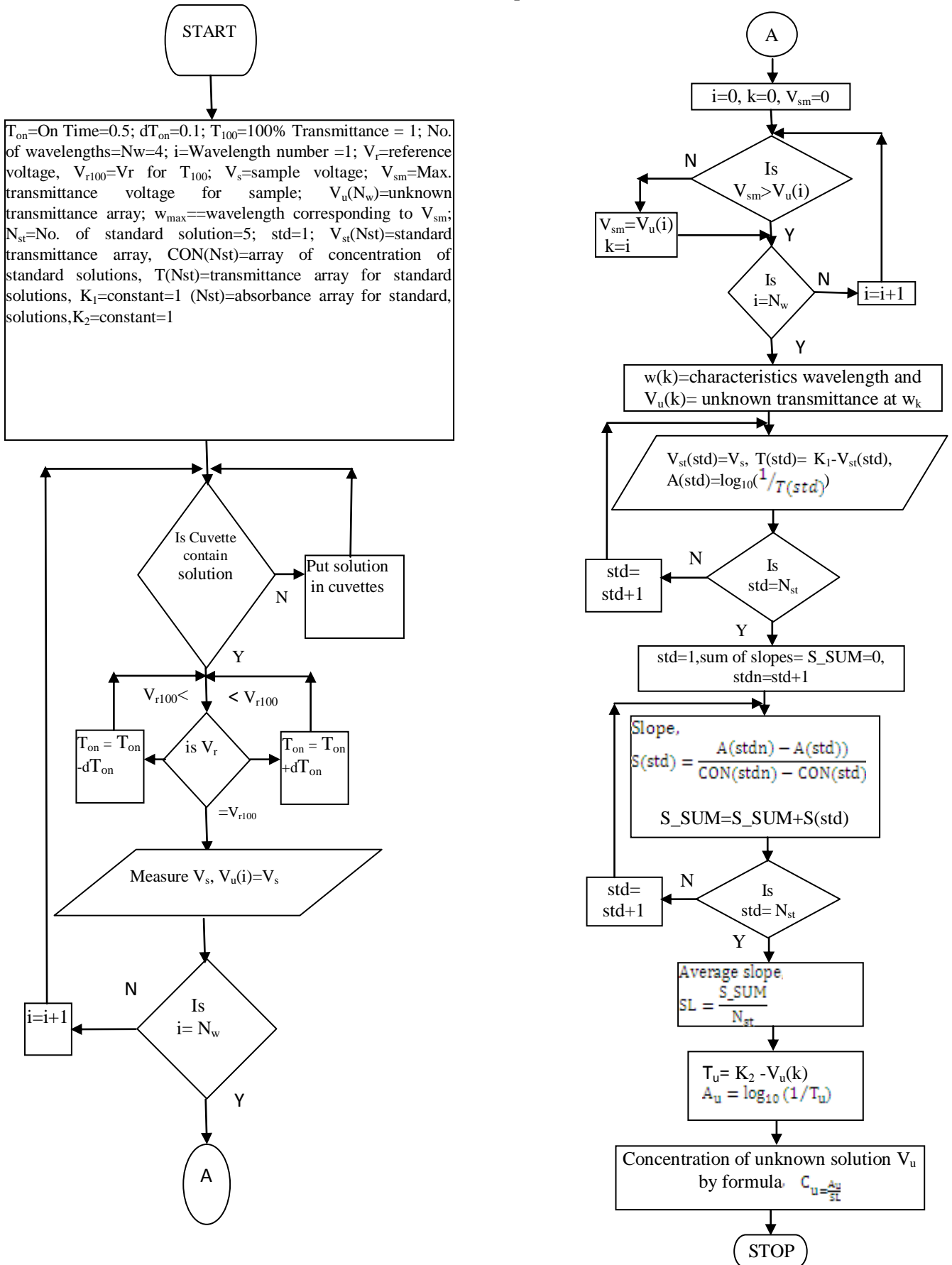


Fig. 7. Flow chart of the differential colorimetric analysis

VI. CONCLUSIONS

Differential colorimeter is a new kind of inexpensive instrument that automatically measures the absorption and concentration. The absorption due to no-analyte solution is subtracted from the analyte one. This compensates instrumental as well as experimental errors introduced due to cuvette and reagent respectively. The gain of difference amplifier can be set for the low and high sensitivity for the concentrated and dilute solutions respectively. The developed software program has been successfully implemented for the determination of characteristics wavelength, absorptivity coefficient and concentration. The developed instrument has been successfully used for the estimation of iron in water solution. Similarly, it can be used for the measurement of concentration of some other metal compounds.

Future scope: The differential colorimeter will be interfaced to the personal computer. Software program will be developed for the visualization of graphs. A comparative study of different set of observations will be carried out by graphical analysis.

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ABOUT AUTHORS



Dr. Pravin K. Bhadane is Head of the Electronics Department at Nowrosjee Wadia College, Pune (India). He has obtained the M.Phil. and Ph.D. degrees from University of Pune. He is author of fifteen research papers and four books. His research interest is in the field of analytical instrumentation.



Prof. (Mrs) Suchita P. Bhangale is working as an Assistant Professor of Electronics at Nowrosjee Wadia College, Pune (India). She has done M.Sc. in Electronics and now pursuing her Ph.D. work under the supervision of Dr. P. K. Bhadane. She has published more than three research papers in the international journals.