

LabVIEW Controlled Flame Atomizer for Atomic Absorption Spectrophotometer

Bharati Sagi, Hari Krishna M., and S. Raja Rathnam

Abstract—Concentration of majority of metal atoms is measured using AAS (Atomic Absorption Spectrophotometer) by measuring the absorption of radiant energy by free metal atoms in the sample. The present paper emphasizes on controlling and monitoring of factors effecting this free atom concentration in flame AAS (FAAS) using Lab VIEW and an AVR based embedded system. These factors include flame temperature, fuel-oxidant aspiration rate, and fuel-oxidant ratio of the flame. These parameters are controlled and monitored by a 32-bit AVR microcontroller has been used to interface the instrument under control i.e. AAS to Lab VIEW through serial interface protocol RS 232. The purpose of this work is for the further perfection of the atomic absorption method of analysis by introducing a feedback between the result of analysis of any sample and the procedure of analysis of this sample. It was possible only by introducing the principle of self-tuning. The complete system monitors all safety interlocks and controls the required process parameters with the pre-set values and accomplishes appropriate flame conditions for uniform sample atomization.

Index Terms—Atomic absorption spectrophotometer, DSC module, flame controller module, lab VIEW, RS 232, flame controller module.

I. INTRODUCTION

In flame atomic absorption spectroscopy (FAAS), the measurement of absorption of radiation beam at characteristic resonant spectral line for a particular element yields a measure of the concentration of that element in an original sample solution [1]. Presently, the most common technique for atomizing an element for the purpose of the absorption measurement, is by introducing a liquid sample solution of the element of interest into a gas burner wherein droplets of the solution are vaporized and the elements ultimately atomized [2], so as to form in the path of the apparatus radiation beam, a substantial quantity of the element of interest in its atomic state.

In order to effect appropriate burning of the element containing solution, the liquid sample must be converted into a fine spray then mixed with a fuel and oxidant gas before introducing into burner. Fine spray is achieved through nebulizer. The sample laden gas or oxidant (Air/acetylene N_2O) then passes into the burner chamber where it is mixed with fuel acetylene (C_2H_2). It is then introduced into the burner head where it is ignited.

The sensitivity of the absorption measurement is dependent on many factors, one of which being the flame condition of the burner i.e., the leanness or richness of the

fuel-oxidant mixture [3], [4]. To achieve optimum flame conditions, few system requisites should be maintained which include appropriate fuel and oxidant pressures, drain level, fuel and oxidant flow rates, proper burner position, presence of pilot flame and soon [5], [6]. According to the sample selected, fuel-oxidant flow rates should be set to establish required flame conditions. All these settings and adjustments should be achieved by operating and controlling different mechanical elements like solenoid valves, proportional valves *et al.* It would be tedious and time taking job to do this manually and would result in errors.

II. MAIN OBJECTIVES

The objective of developing automatic flame controller is to eliminate operator free adjustments there by reducing errors. Further the automatic gas controller should allow for the programmability of optimum analysis parameters e.g., selection of proper fuel-oxidants, the optimum fuel-oxidant flow rates *et al.* [7] should be maintained constant for each measurement even on different instruments and can be stored in discs for further usage. It is therefore a primary requirement of this project to provide a control system which will operate and control the gas box apparatus with respect to pre-programmed parameters regardless of the instrument system in which it is employed. Another objective of the project is to provide the user, run time process visualization on graphical environment so that the user can visualize and understand the process configurations, failures if any and can analyze the process, also should enable the user to change the process configurations in any condition.

III. DESIGN AND DEVELOPMENT

The sample atomizer in flame atomic absorption spectrometer will employ flame temperatures varying from 2000 °C to 3000 °C for atomizing different metal elements present in a sample. In this 2000 °C to 2500 °C temperatures can be achieved by using Air-Acetylene combination and 2500 °C to 3000 °C temperatures can be achieved by using N_2O -Acetylene combination [8]. Flame controller model is shown in Fig. 1.

Depending on the sample element and its atomizing temperature value mode of operation should be selected as Air mode or N_2O mode. In air mode Air is selected as combustion element and in N_2O mode N_2O is selected as combustion element. In both cases Acetylene is the fuel. The selection of the oxidants is accomplished using solenoid valves connected to respective oxidant cylinders. Solenoid valves operate like switches which when excited they will change their state from OFF to ON (Normally open) or ON to

OFF (Normally close).

By varying flow rates of fuel-oxidants any particular temperature of the flame can be set depending on the sample being selected for analysis. Flow rates can be set using proportional valves. A flow rate of 0 to 3 liters per minute can be varied with a voltage of 0 to 5V.

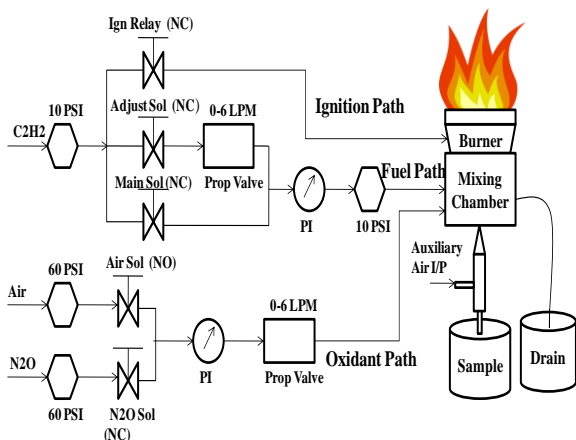


Fig. 1. Flame atomizer in AAS.

Along with the flow rates proper pressures should be maintained for fuel and oxidants for proper uniform flame generation at the burner tip. As per the standards these are 50 PSI for oxidants and 15 PSI for fuel C₂H₂. These pressures are pre-set at the pressure switches which will be monitored continuously.

8051 based embedded system is designed to acquire these signals from the sensors at respective nodes and to send back the control signals to the corresponding valves in the system. Auto ignition for the burner is achieved by an auxiliary path of fuel and oxidant (air). To do so there is a need to detect the presence of flame at the burner tip using a flame sensor.

In a process to establish required flame conditions the initial step is to check all the interlocks. Interlocks are acceptable conditions of the gas box system to generate flame under safe operation of the system [9]. These interlocks are the inputs to the controller.

These interlocks include

- Pre and post pressure switch outputs which should be high indicating the presence of fuel or oxidant without any leakage.
- Flame sensor output (0/1). 0 indicates absence of flame and 1 indicates the presence of flame at the burner.
- Drain sensor output (0/1). It is a level sensor to sense the presence of required distilled water to clean the burner assembly after the analysis of the sample.

After checking the interlocks the controller will operate the solenoid valves to perform the ultimate task. There are 6 solenoid valves in which two are for oxidants and remaining four for fuel path. The appropriate solenoid valves will be operated according to required flame conditions for the sample. These are outputs from the controller to the system and also the controller will operate the proportional valves to set the required flow rates for fuel and oxidant.

The above discussed tasks are achieved with LabVIEW 2010 and microcontroller ATMEGA3V.

The implementation of the above discussed tasks is developed in two stages.

A. Development of ATMEGA32V based data acquisition hardware to acquire signals from different nodes of gas box system and to feedback control signals to appropriate nodes to accomplish required control action to operate the instrument in a way to achieve ultimate system performance. This is as represented in Fig. 2.

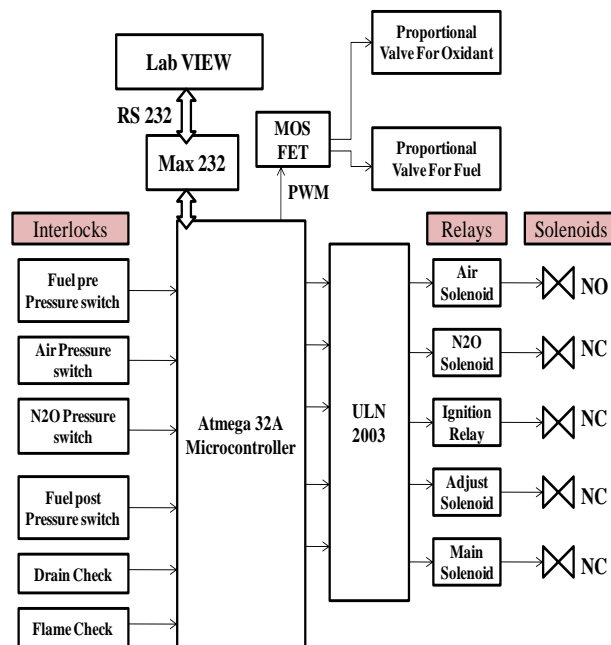


Fig. 2. Block diagram of auto flame controller.

This hardware includes:

- Required power-up for the system.
- Inputs from the pressure switches, flame sensor and drain sensor.
- Set of relays to switch appropriate solenoid valves to achieve required action sequence.
- A PWM and mos-fet set up to control position of orifice in proportional valve.
- RS232 interfacing module to establish serial communication between microcontroller and LabVIEW in personal computer.
- A 16*2 LCD interface to indicate the process status.

National instruments graphical programming environment LabVIEW DSC (Data Logging & Supervisory Control) is used to develop a user interface to monitor and control overall process parameters on LabVIEW front panel. Its functions include

- Displaying process status in different stages.
- Selection of sample mode of operation.
- Setting appropriate process parameters to microcontroller.
- Enabling user to change process configurations if required.

IV. HARDWARE IMPLEMENTATION

To maintain the factors affecting the flame conditions as per the standards, the corresponding hardware elements in flame controller are monitored and controlled through LabVIEW integrated with an embedded system.

The present work has been developed in two modules:

- Designing and developing a hardware consisting of an Embedded based data acquisition and control module.

- Development of LabVIEW based flame controller using DSC module

The block diagram of auto flame controller is shown in the Fig. A preview of this project is shown in Fig. 3.

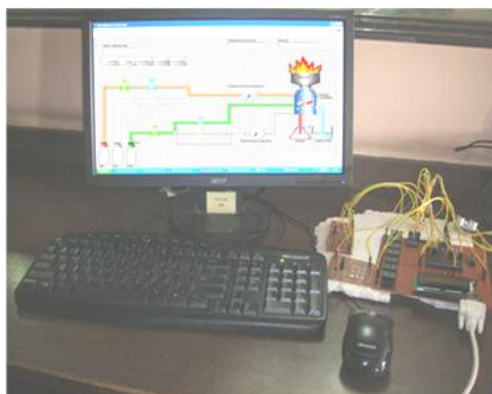


Fig. 3. Snap shot of implemented project.

The following instrument interlocks are the inputs to the microcontroller at Port A.

- Fuel C₂H₂ pre pressure switch – Normally Closed (0 or 5V).
- Fuel C₂H₂ post pressure switch – Normally Closed (0 or 5V).
- Air Pre pressure switch – Normally Open (0 or 5V).
- N₂O pre pressure switch – Normally Closed (0 or 5V).
- Oxidant post pressure switch – Normally Closed (0 or 5V).
- Flame sensor output - (0-5 V).
- Drain level - (0 or 1 logic level).

The outputs from Port D of microcontroller are connected to respective solenoid relays through a Darlington pair of transistor IC ULN 2003.

The set of solenoid relays so connected are shown in Fig. 2.

The flow rates adjustment for fuel and oxidant proportional valves is achieved through PWM (pulse width adjustment) by the microcontroller.

Hardware to LabVIEW interface is accomplished using RS 232 communication interface through MAX 232 DB9 connector.

V. EXPERIMENT & RESULT

The software for the present work is implemented in two stages.

- Checking the interlocks
- Ignition flame establishment
- Main flame establishment

The flame controller module should establish requires flame only after passing all the interlocks. The process configuration is represented by a flow chart shown below in Fig. 4.

- The status of the instrument passes a particular digital value to the microcontroller and the microcontroller pass that status to Lab VIEW through serial port read buffer.
- A case structure is developed in Lab VIEW block diagram in which each status is identified and Lab VIEW displays the corresponding error at its front panel.

- After obtaining a satisfying status of the instrument, the flame controller solenoids are operated to establish a pilot flame. The sequence of operations to obtain auto ignition are indicated in the process flow chart given below.

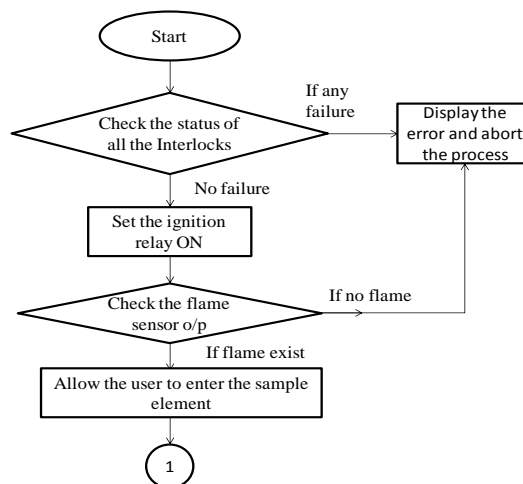


Fig. 4. Flowchart for establishing ignition flame.

The flame controller instrument is virtually designed on LabVIEW front panel using 3D controls from DSC module as shown below in Fig. 5. This VI front panel represents the flame controller module described in Fig. 1.

This system is designed using the DSC module controls like pipes and valves to represent the corresponding fuel oxidant paths, solenoids and in the actual flame controller instrument. After switching on the instrument, the system checks for all the interlocks. If any error obtained at any of the interlocks, it is displayed and the system operation hangs and waits for safe shut down of the instrument.

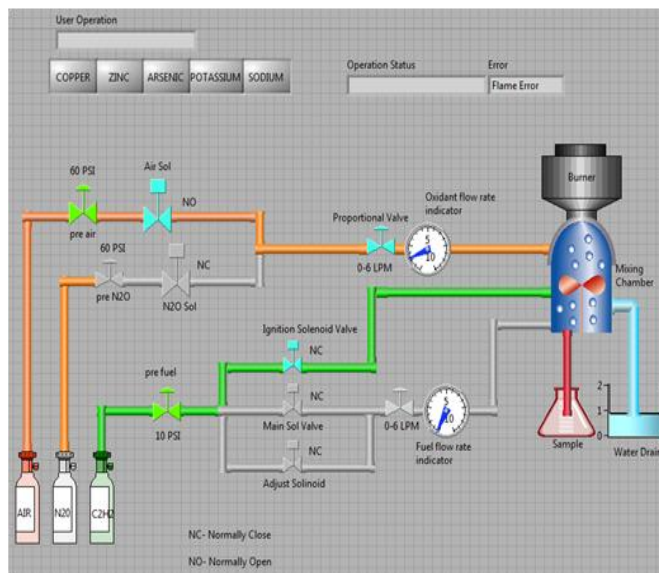


Fig. 5. Error display on LV front panel.

If there is no failure, the system operates the ignition solenoid and ignition relay through a relay and mosfet set up. After few seconds, the system checks for pilot flame. If the flame is not established a flame Error is displayed, indicating trouble with the instrument and it needs assistance.

If expected flame is established, system indicates that "Ignition Flame is Established". Now the system will ask the

user to enter the sample element the user wants to analyze.

Once after getting the request to “Enter the Sample”, the user can select the sample element that he is going to analyze under AAS on the Lab VIEW front panel.

Now the task of the system is to establish a specific main flame required to burn the selected sample element. To do so, the instrument used two oxidants Air and N₂O.

Both oxidants in combination with acetylene flame establish flames of 2000 °C to 2400 °C and 2400 °C to 2900 °C respectively. These temperatures can even be pr éciséd to suit a particular sample element by proper fuel-oxidant ratios and their flow rates.

All these parameters are indicated in the table given below Units.

TABLE I: FUEL-OXIDANT RATIOS AND REQUIRED FLOW RATES FOR SPECIFIC SAMPLE

S.NO	Sample	Oxidant Required for combustion	Fuel Flow rate (LPM)	Oxidant Flow rate (LPM)	Absorbing wavelength (nm)
1.	Copper	Air (Medium)	1	1.5	324
2.	Zinc	Air (Lean)	1	1	214
3.	Arsenic	N ₂ O (Medium)	4	1.5	194
4.	Sodium	Air (Lean)	1	1	589
5.	Potassium	Air (Lean)	1	1	766
6.	Aluminum	N ₂ O (Fuel Rich)	4.5	1	310
7.	Iron	Air (Lean)	1	1	248
8.	Lead	N ₂ O (Lean)	4	1	217
9.	Mercury	Air(Lean)	1	1	253
10.	Barium	N ₂ O (Fuel Rich)	4.5	1	554

These parameters are pre set in Lab VIEW, and it will pass the appropriate parameters to the instrument after sample is selected from the Lab VIEW front panel. Depending on the sample element and the corresponding oxidant selected, the system functions in two different modes.

Air mode: In which the set of solenoids to be energized are as follows.

- Air solenoid ON
- Main solenoid ON
- Adjust solenoid ON

While the other solenoids kept closed.

N₂O mode: In which the set of relays to be energized are as follows.

- N₂O solenoid ON
- Main solenoid ON
- Adjust solenoid ON

For setting the required flow rates for fuel and the oxidant, an appropriate duty cycle is chosen from the microcontroller. The flow chart showing the sequences of these operations is given below in Fig. 6. Corresponding Lab VIEW front panel is shown below in Fig. 7.

This system provides the analysis for 5 sample elements.

- Copper
- Arsenic
- Potassium
- Zinc and
- Sodium

It can be observed that the system is adjusting its fuel-oxidant paths specific to sample selected. The amount of fuel required for each combustion process is preset through

Lab VIEW as per the standards, which are indicated on the flow indicators on the front panel.

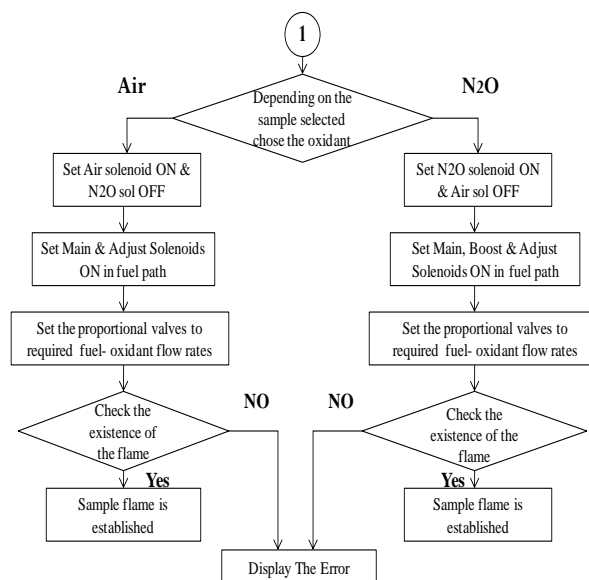


Fig. 6. Flow chart for main flame.

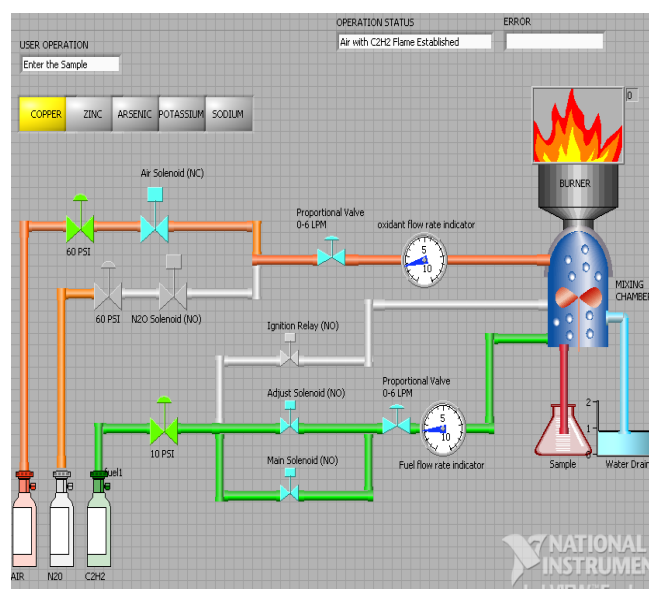


Fig. 7. Sample flame for copper element.

It can be observed that the system is adjusting its fuel-oxidant paths specific to sample selected. The amount of fuel required for each combustion process is preset through Lab VIEW as per the standards, which are indicated on the flow indicators on the front panel.

When the auto flame controller system is integrated and run with a single beam FAAS the following sample graphs are obtained.

Concentration Vs Absorption graphs with standard solutions and samples solution of unknown concentrations are plotted on a single XY graph in LabVIEW.

A good correlation is observed between standard[7], [8] and the sample graphs.

Histerisis is minimized with the use of microcontroller PWM for setting the flowrates.

The graph below shows:

X axis: Concentration (parts per million- ppm)

Y axis: % Absorption

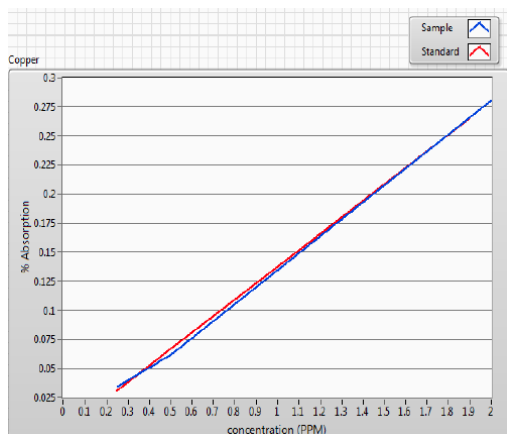


Fig. 8. Sample graph VS Standard graph for copper.

VI. CONCLUSION

The present work is an initial effort made towards designing an automated test and control systems for complicated instrument like AAS. Designing of such automated measurement and control systems for even more complicated AAS systems such as dual chamber sampling systems, automated multi sampling systems can be made easy, user friendly, efficient and reliable.

The proposed system is verified through simulation and is implemented in a prototype. Experimental results are compared with the conventional method. Initial results are found to be satisfactory. Further the results also indicate that this system has stable performance and can meet the designing requirements. This initiative is a preliminary work towards the development of complete automation system for Atomic Absorption Spectrometer.

The performance of the system can be enhanced by developing the features like self adjustable fuel-oxidant flow rates, burner position etc with respect to standard values.

REFERENCES

- [1] G. Schaldach, "Predictor of 'High performance flow flame atomic absorption spectrophotometry for interference-free trace determination,'" *Fresenius Journal Analytical Chemistry*, Spinger, vol. 350, pp. 481-486, 1994.
- [2] J. Copan, R. Balachandar, and F. Berruti, "Droplet size-velocity characteristics of sprays generated by two-phase feed nozzles," *Chemical Engineering Communications*, vol. 184, 2001.
- [3] Z. L. Fang and L. P. Dong, "Flow injection on-line coprecipitation preconcentration for electrothermal atomic absorption spectrometry," *Journal of Analytical Atomic Spectroscopy*, vol. 7, pp. 439-445, RSC Publications, 1992.
- [4] J. P. Matousek, "Aerosol deposition in furnace atomization," US Patents, Patent No: 4,201,469, 1980.
- [5] S. L. C. Ferreira and V. A. Lemos, "An automated on-line flow system for the pre-concentration and determination of lead by flame atomic absorption spectrometry," *Micro Chemical Journal*, vol. 68, issue 1, Elsevier Publications.

- [6] V. K. Nagdev and Y. F. Bukreev, "Formation of free atoms using a graphite rod atomizer in atomic absorption spectrometry," *Journal of Applied Chemistry*, vol. 3, issue 4, Spinger, pp. 1052-1055, 1980.
- [7] E. D. Frederick and G. M. Eichenbaum, "Apparatus, method and computer program product for automated high-throughput sampling and data acquisition," US Patents, Patent No: US 2004/0014227 A1, 2004.
- [8] M. Mazloum, "Study of interference in flame atomic absorption spectrometric determination of aluminium by using factorial design," *Pakistan Journal of Applied Chemistry*, Science Publications, pp. 405-407, 2002.
- [9] D. C. Stahl, "Method and system for real-time control of analytical and diagnostic instruments," 2000, US Patents, Patent. No: 6049764.



Bharati Sagi was born in Eluru, Andhra Pradesh in July 1983 in Andhra Pradesh. She received her M.Tech in Electronics & Instrumentation from the university of JNTU- Hyderabad in 2012 and B.Tech degree in Electronics & Instrumentation Engineering from Andhra University in the year 2000. Presently she is working as a Lecturer in EIE Dept, VNR Vignana Jyothi Institute of Engineering and Technology (VNR VJIET), Hyderabad, A.P, India. Previously she worked as a research associate in an analytical Instruments designing company Elico India Ltd, Hyderabad during the year 2011-12. Her research interests include Control Systems, Process Control Instrumentation and Analytical instruments. She is awarded with a gold medal by the institution VNR VJIET in the year 2011 during her M.Tech course work.



Hari Krishna Mulam was born in Gudur, Andhra Pradesh, India in 1979. He received the B.Tech degree in Electronics and Instrumentation from JNT University, Hyderabad, India in 2001 and the M.Tech degree in Instrumentation and Control Systems from JNT University Kakinada, Andhra Pradesh, India in 2010. He is currently pursuing the Ph.D. degree from Department of Biomedical Engineering, Osmania University, Hyderabad, India. He has 12 years of Teaching experience and presently working as Associate Professor in the Department of Electronics and Instrumentation Engineering, VNR VJIET, Hyderabad, India. Mr. Harikrishna Mulam is a Life Member of professional societies like ISTE (Indian Society for Technical Education) and ISOI (Instrument Society of India). He is presently working on Electrooculogram based HCI system.



S. Raja Ratnam was born in Andhra Pradesh in 1938. He received his Ph.D in Electronics & Communications Engg from Andhra University in 1977. He has 45 years of teaching and research experience under various universities and institutions. Presently he is working as a professor in Electronics & Instrumentation Dept in VNR VJIET, Hyderabad. He is a life member of few renowned organizations like IEEE, ISTE and ISI. Prof Raja Rathnam has received J.C. Bose Award in 1977 for the Indigenous design of VHF Forward Scatter communication system for "Himalayan Radio Propagation Unit" (of DRDO). Received The Andhra Pradesh State Award For The Best University Teacher for the year 1996-97. He is the founder of Department of Instrumentation in Sri Krishna Devaraya University and VNR Vignana Jyothi Institute of Engineering and Technology.