CHAPTER 4

RESULTS AND DISCUSSION
Section 4.1
CALIBRATION

The individual blocks of the computer based measurement system for potassium ion are designed and constructed. The necessary software is developed in C language. These details are already presented in chapter 2 and 3 respectively.

Before using the system, the electrodes must be calibrated by measuring a series of known standard solutions, made by serial dilution of the 0.1M KCl standard solution. In the present study, the system is calibrated at four concentrations of standard KCl solutions – 0.1M, 0.01M, 0.001M and 0.0001M.

Preparation of the standard solutions:

To prepare the standard solutions, primarily, make a stock solution of 0.1M KCl which is prepared by dissolving 745.5 milli grams of potassium chloride salt in 100 ml of distilled water.

To prepare 0.01M KCl solution take 10 ml of 0.1M KCl solution and dilute it to 100 ml with distilled water. For 0.001M KCl solution take 10 ml of 0.01M KCl solution and dilute it to 100 ml with distilled water. Similarly for 0.0001M KCl solution take 10 ml of 0.001M KCl solution and dilute it
to 100 ml with distilled water. 2 ml of 2.5 Molar NaCl buffer solution (ISAB) should be added to each 100 ml standard and mix thoroughly to compensate for different activity coefficients between samples and standards. The standard solutions are prepared in the laboratory in accordance with the accepted principles of analytical chemistry\textsuperscript{58,59}.

After preparing the standard solutions, run the software program of the system for calibration. When the execution of the program starts, a user menu is displayed on the CRT screen of the computer as shown below.

**Main Menu**

1. ADC Testing
2. Potassium Measurement
3. Temperature Measurement
4. Calibration of the system
5. Quit

Select the option ‘4’ for calibration of the system by pressing key number 4 on the keyboard and press ‘Enter key’. Now the system is ready to calibrate the system.

The calibration menu itself guides the user to calibrate the system which is shown below (which appears on the Monitor of the PC).

**CALIBRATION OF THE SYSTEM**

**FOR CALIBRATION PREPARE 4 STANDARDS OF KCl SOLUTIONS OF CONCENTRATIONS 0.1M, 0.01M, 0.001M & 0.0001M**
(Rinse the electrodes with distilled water thoroughly and blot dry with tissue paper before dipping the electrodes in the solutions every time)

**DIP THE ELECTRODES IN 0.0001M CONCENTRATION KCL SOLUTION**

After a stable reading press any key on the keyboard to switch on to next step.

**DIP THE ELECTRODES IN 0.001M KCL STANDARD SOLUTION**

After a stable reading press any key on the keyboard to switch on to next step.

**DIP THE ELECTRODES IN 0.01M KCL STANDARD SOLUTION**

After stable reading press any key on the keyboard we can see the slope of the electrode in milli volts/decade for the first two standard solutions.

**DIP THE ELECTRODES IN 0.1M KCL STANDARD SOLUTION**

The average slope of the potassium ion sensitive electrode is computed and displayed on the screen. And it automatically switches to main menu after pressing the enter key on the keyboard.

Now the calibration of the system is completed and the system is ready to measure the potassium ion concentration of the sample. The slope of the electrode tells the sensitivity of the potassium ion sensitive electrode.
After making the appropriate adjustments both in the hardware and software and also following the calibration procedure as mentioned earlier, the instrument is tested with the standard solutions of potassium. The results of measurements are presented in Table 4.1. The measurements are made for these samples using a Flame photometer of Elico Make and the results are presented in the same table. The results of the present study are in good agreement with those obtained from the Flame photometer (measuring range 1 to 100 parts per million (ppm)).
<table>
<thead>
<tr>
<th>Standard values</th>
<th>Present study</th>
<th>Flame Photometer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Conc. in Molarity (M)</td>
<td>Conc. in ppm</td>
<td>Conc. in ppm</td>
</tr>
<tr>
<td>0.1M (KCl)</td>
<td>3910.00</td>
<td>3925.64</td>
</tr>
<tr>
<td>0.01M (KCl)</td>
<td>391.00</td>
<td>430.10</td>
</tr>
<tr>
<td>0.001M (KCl)</td>
<td>39.10</td>
<td>39.10</td>
</tr>
<tr>
<td>0.0001M (KCl)</td>
<td>3.91</td>
<td>4.30</td>
</tr>
</tbody>
</table>
MEASUREMENT OF POTASSIUM ION IN SOILS

The system is used to measure the potassium ion in soil samples collected from the fields of Anantapur area. The samples are prepared for measurement following the procedure cited in the literature\textsuperscript{60}.

**Soil sample preparation:**

After removing any stones or fresh organic material (roots, twigs, leaves, worms, insects etc), and breaking up any large lumps, soil samples must be air dried by laying out in a thin layer on metal or plastic trays in a current of air at no more than 30 °C until dry. Then they must be crushed in a pestle and mortar to pass through a 2 mm sieve. About 200 g of material should be sufficient for duplicate analysis and storage.

Weigh accurately 50 g of dry soil sample and add 100 ml of de-ionized water and shake vigorously for 30 seconds to ensure good dispersion, then leave to stand for 15 mins. After this time, shake again for 5 seconds, then repeat this procedure three times before finally allowing to settle. When the supernatant is clear, decant or pipette 50 ml and mix with 1 ml of buffer solution in a plastic beaker.
Measurement:

Follow the instructions in the computer interface software and/or electrode operating instructions to measure a series of samples and record the results. Briefly, it is important to note that the electrodes must be washed and dried between each sample to avoid cross contamination and sufficient time must be allowed (2 or 3 minutes), before taking a reading after immersion, to permit the electrode signal to reach a stable value. For the highest precision, frequent recalibration is recommended.

The results of the measurement of potassium ion, in soil samples are presented in Table 4.2. They are compared with the measurements taken from the Flame Photometer of ELICO’s make (Model No.CL360). The results are in good agreement with those obtained from the Flame photometer. The measuring range of the present system is 0.0001M/3.91 ppm to 0.1M/39100 ppm with an accuracy of ±1%.
<table>
<thead>
<tr>
<th>Sl.No.</th>
<th>Samples</th>
<th>Present study</th>
<th>Flame Photometer</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Conc. in M</td>
<td>Conc. in ppm</td>
</tr>
<tr>
<td>1.</td>
<td>Soil sample 1 (R₁ T₇)</td>
<td>0.00033</td>
<td>12.81</td>
</tr>
<tr>
<td>2.</td>
<td>Soil sample 2 (R₂ T₂)</td>
<td>0.00025</td>
<td>10.23</td>
</tr>
<tr>
<td>3.</td>
<td>Soil sample 3 (R₃ T₉)</td>
<td>0.00030</td>
<td>12.10</td>
</tr>
<tr>
<td>4.</td>
<td>0.002M (KCl)</td>
<td>0.002</td>
<td>78.20</td>
</tr>
</tbody>
</table>

The system is quite successful in the measurement of potassium ion in the given samples.
Suggestions for the improvement of present system

1. The system can be made a dedicated one by replacing the personal computer with a microcontroller with appropriate hardware.

2. The two electrodes ion sensitive and reference electrodes (glass make) can be replaced by combined potassium ion sensitive electrode of polymer make so that the durability of the electrodes may be improved.

3. The accuracy and resolution of the system can be improved by using 16 bit or higher resolution analog to digital converter.

4. The availability of the input channels of the instrument can be used to measure other parameters like pH, conductivity and other ion measurements etc.