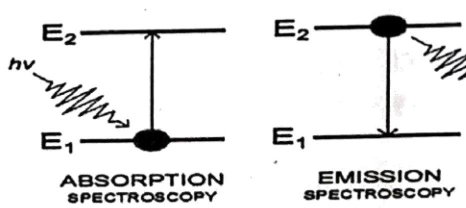
Estimation of $Ca^{2+}/Na^{+}/K^{+}$ in Soil by Flame Photometry*

1. Importance of the experiment: Approximately 7% of the total cultivable land across the globe is salt-affected triggering a great damage to agriculture. Salinity stress is a limitation to the productivity of agricultural crops worldwide, and it refers to the excessive amount of soluble salts in the root zone which induce osmotic pressure and ion toxicity in the growing plants on critical biochemical processes. Saline soil is composed of dissolved salts such as NaCl, Na_2SO_4, $MgSO_4$, $CaSO_4$, $MgCl_2$, KCl, and Na_2CO_3, each of which contributes to salinity stress. Among these ions, excess Na^+ ion concentration has the most adverse effects on plant growth by its detrimental influence on plant metabolism inhibiting enzyme activities. On the other hand, deficiency of Ca^{2+} ion results in distorted growth exhibited by new tissue such as root tips, young leaves and shoot tips. Blossom end rot of tomatoes is a classic case of calcium deficiency. Calcium deficiency can arise if levels in the fertilizer solution are less than 40-60 ppm and/or K^+, Mg^{2+} or Na^+ levels are too high.

2. Concept: Flame atomic emission spectrometry, also called as flame photometry, is a fast, simple, and sensitive analytical method for the determination of trace metal ions in solution. This method is suitable for those metals which are easily excited to higher energy levels at flame temperature. The compounds of alkali and alkaline earth metals dissociate into atoms when introduced in flame, and some of these atoms further get excited to even higher energy levels. But being unstable there, these atoms emit radiations while returning back to the ground state which generally lie in visible region of the spectrum where each alkali/alkaline earth metal has a specific wavelength. The intensity of emission is directly proportional to the number of atoms returning to the ground state and the light emitted is in turn proportional to the sample concentration. On the other hand, non-metals generally do not produce isolated neutral atoms in a flame and thus not suitable for determination by flame emission spectroscopy. Flame photometry is an empirical method of analysis, i.e. you must calibrate the method carefully. Many different experimental variables affect the intensity of light emitted from the flame. Therefore, careful and frequent calibration is necessary for good results.



Element	Emitted wavelength (nm)	Colour
Ca	622**	Orange
Na	589	Yellow
K	766	Violet
Li	670	Red

3. Applications: Flame photometry could be applied to analyze alkali and alkaline earth metals qualitatively as well as quantitatively. Presence of some group II elements is critical for soil health. Therefore, after analyzing the soil, it could be supplied with specific fertilizers to improve the soil quality for better cultivation. The concentration of $Ca^{2+}/K^{+}/Na^{+}$ ions are determined by diluting and aspirating the samples into the flame. This technique is also extremely useful to determine the concentrations of various metals in soft drinks, fruit juices and alcoholic beverages.

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*One page write-up is common for both the flame photometry experiments.

**Calcium is measured by using the calcium hydroxide band emission at 622 nm. However, the main atomic emission occurs at 423 nm.

Expt. No 8 **Date:** 08.09.2018

Experiment	**Estimation of Ca^{2+} in Soil by Flame Photometry**
Problem definition	Deficiency of Ca^{2+} ion in soil results in distorted plant growth. Calcium deficiency can arise if levels in the fertilizer solution are less than 40-60 ppm and/or K^{+}, Mg^{2+} or Na^{+} levels are too high.
Methodology	Trace amount of calcium in soil can be determined by flame photometry at a wavelength of 423 nm, and its intensity is proportional to its concentration.
Solution	After careful calibration of photometer with Ca^{2+} solution of known concentration prepared using the stock solution, the spectral line intensity of unknown Ca^{2+} solution is correlated with its concentration.
Student learning outcomes	Students will learn to a) Calibrate the flame photometer using diluted concentrations of known Ca^{2+} solution prepared from stock solution. b) To correlate the Ca^{2+} concentration (in ppm) in the given unknown solution.

Principle:

Estimation of calcium is based on flame emission spectroscopy, also called as flame photometry which deals with the excitation of electrons from ground state to higher energy state and coming back to its original state with the emission of light. Trace amount of calcium can be determined at a wavelength of 423 nm, and its intensity is proportional to its concentration. After careful calibration of photometer with solution of known compositions, it is possible to correlate the spectral line intensity of unknown solution with its concentration.

Reagents: (a) 1000 ppm of Calcium stock solution (400 ppm of Ca^{2+} in 1000 ppm $CaCO_3$; then 1000 ppm of Ca^{2+} = 2497 ppm $CaCO_3$) prepared by dissolving 2497 mg of $CaCO_3$ in 1-2 mL conc. HCl and diluting to 1L in a std. flask accurately. **(b)** 4 different concentrations of calcium solution (100 – 200 ppm) and given unknown sample solution prepared from 1000 ppm stock solution were made up to 50 mL standard flasks using distilled water.

Apparatus: Burette, graduated pipette, beakers, rubber bulb, standard volumetric flasks (50 mL), flame photometer and its accessories.

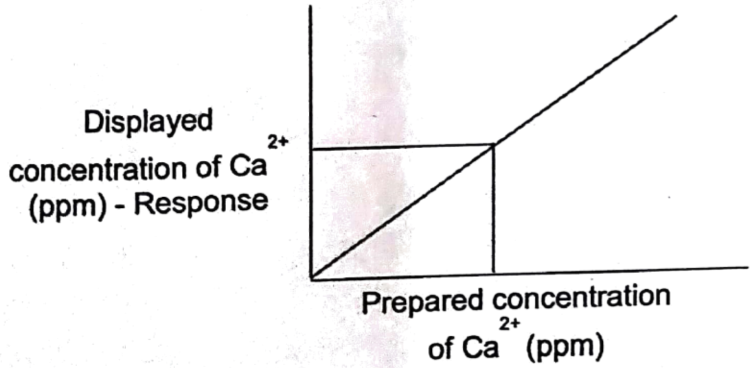

Table 1: Experimental Data

S. No	Concentration (X axis)	Response (Y axis)
1.	20 ppm	14.06
2.	40 ppm	39.23
3.	60 ppm	56.65
4.	80 ppm	73.40
5.	Unknown	50.25

Result:

The amount of calcium present in the given water sample = 55 ppm

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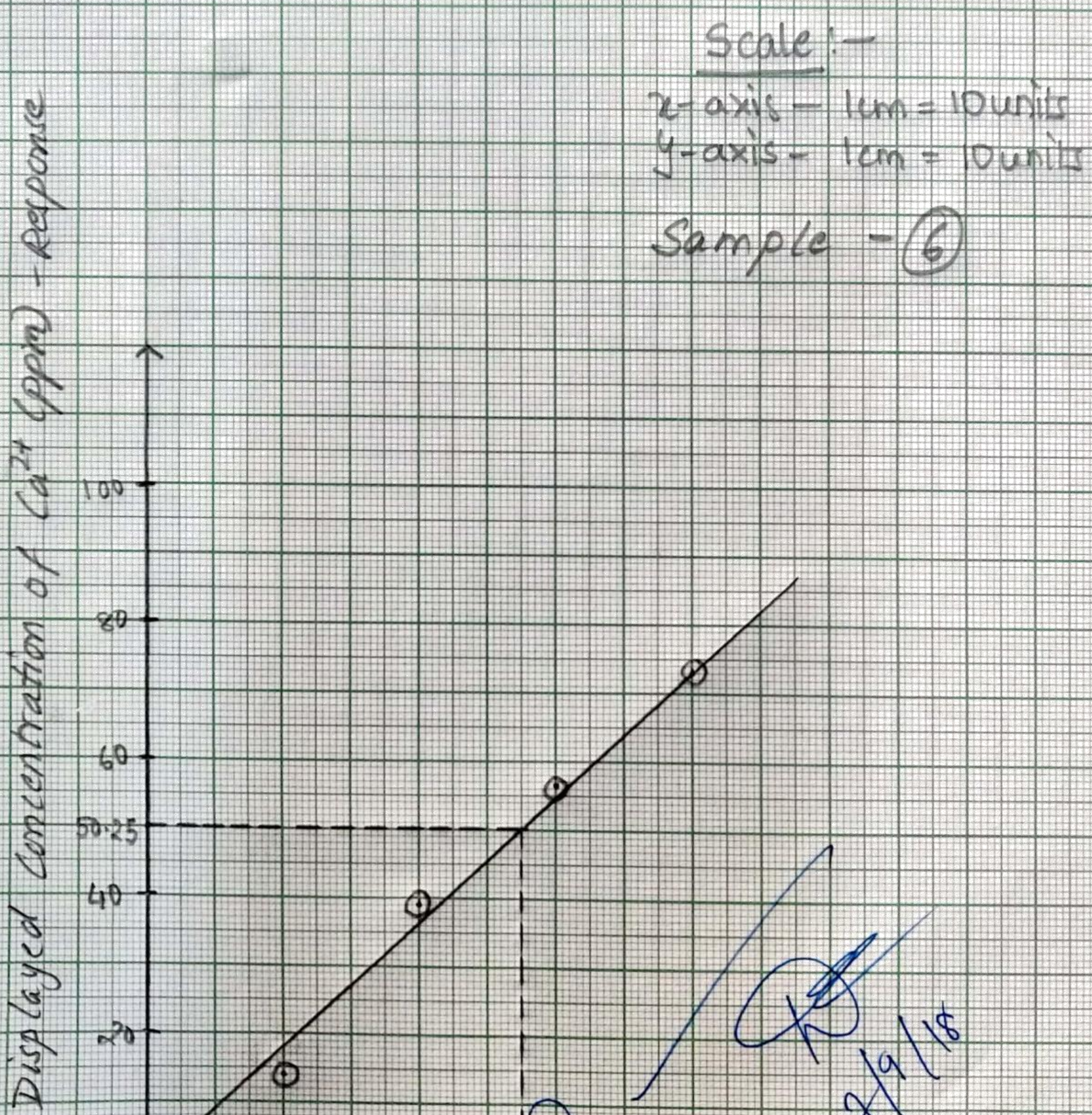
Evaluation of Result:

Sample number	Experimental value	Actual Value	Percentage of error	**Marks awarded**
6				

<u>Procedure: Setting the flame photometer for the estimation of Calcium</u>

1) Start the electrical supply and switch on the air supply. Stabilize the air and the needle should be steady at the mark to maintain about 0.5 Kg/cm^2 pressure.
2) Switch on the gas and maintain the gas fuel mixture so that blue flame is seen through the viewing window.
3) Adjust the flame so that the flame is seen as 5 cones each in two parallel rows.
4) Follow the operating instructions of the instrument.
5) Following the instructions seen on the screen, carry out calibration using the four different diluted solutions of Calcium. Stop when "CALIBRATION OVER" display comes.
6) Then select "SAMPLE" from the menu.
7) Each one of the solutions should be aspirated (wait 10 seconds). Sample and the concentration reading displayed by the instrument should be noted down.
8) A plot should be made having prepared concentration (x-axis) *vs* displayed concentration (y-axis).
9) Record the concentration of given unknown calcium from the plot and report the result.



Scale :-
x-axis - 1cm = 10units
y-axis - 1cm = 10units
Sample - 6
Displayed Concentration of Ca^{2+} (ppm) - Response
Prepared Concentration of Ca^{2+} (ppm)
100
80
60
50.25
40
20
20
40
55
60
80
100
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