C 141 (Experiment No._____)

NAME:_____ BATCH:_____

ROLL NO:_____ SEM:_____

SIGNATURE:_____ DATE:_____

DETERMINATION OF ALKALI METALS (Na & K) IN SOIL, USING FLAME PHOTOMETER

AIM

To determine the amount of alkali metals (Na & K) in soils using flame photometric method.

THEORY

Flame Photometry, now more properly called "Flame Atomic Emission Spectrometry", is used to analyze the metal in all types of sample solutions of minerals, metals, ferro alloys. In a typical flame photometric experiment, a solution containing the relevant substance to be analyzed is aspirated into the burner and dispersed into the flame as a fine spray. This process is called **Nebulization**.

When a solution of metallic salt is sprayed on to a flame, fine droplets are formed. Due to the thermal energy of the flame, the solvent in the droplets evaporates, leaving the fine residue, which is converted to the neutral atoms. These neutral atoms are converted into the excited state atoms by the thermal energy of the flame. As the excited state is not stable, these excited atoms return to the ground state, with the emission of radiation of specific wavelength. The wavelength of the radiation emitted is characteristic of the element, and is used to identify the element (Qualitative Analysis). The intensity of the radiation emitted depends upon the concentration of the element analyzed (Quantitative Analysis).

Fraction of free atoms thermally excited = $N^* / N_o = A\, e^{-\Delta E/kT}$

where N^* = no. of atoms in excited state

N_o = no. of atoms in ground state

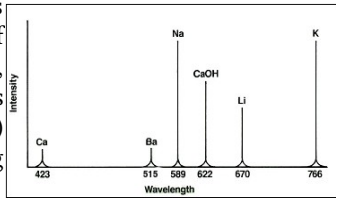
A = Constant for element

ΔE = difference in energy level of excited and ground state

k = Boltzmann constant

T = Flame Temperature

The wavelength of light emitted depends upon the difference in the energy levels of atoms in the excited and ground state. Since atoms of each element have a specific wavelength of radiation, the color of the emitted radiation is different for different elements. For example, alkali metal elements like 'Na' emits yellow (589nm), 'K' emits purple (767nm), 'Li' emits violet (670nm). Alkaline earth metals like 'Ca' emits brick red (422nm, 554nm and 626nm) colored radiations. The wavelength of the radiation emitted is given by the following equation:



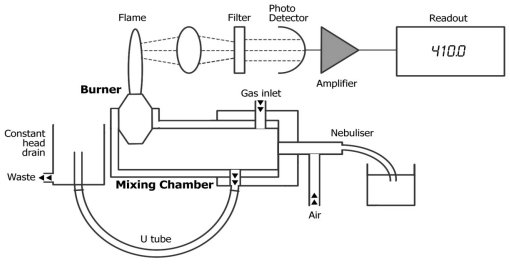
$$\text{Wavelength of light emitted } (\lambda) = \frac{hc}{E_2 - E_1}$$

where h = planck's constant

c = velocity of light

E_2, E_1 = energy levels of excited and ground states respectively.

The intensity of the radiation emitted depends upon the concentration of the element present in the solution. Higher the concentration, more is the flame intensity and lower the concentration, less is the flame intensity. The intensity of the spectral emission line is given by the following equation:



Intensity of spectral emission line

$$(I_v) = \frac{V\, A_T\, h\, r_o\, N_o\, g_a}{B(T)}\, e^{-E/kT}$$

where E= energy of excited state ; k = Boltzmann constant

T = absolute temperature ; h = Planck's constant

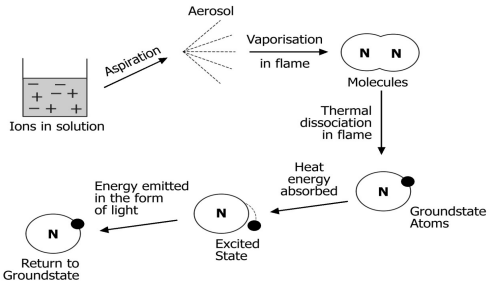
r_o = freq. of radiation ; V= flame volume (aperture ratio)

A_T = no. of transitions each excited atom undergoes per second

N_o = no. of free metal atoms in ground state per unit volume

g_a = statistical wt. of excited atomic state

B (T) = partial function of the atom overall states



MATERIALS REQUIRED

Flame photometer with cuvette cells, compressor and flame, Weighing balance, standard volumetric flasks (100 ml, 50 ml); Burette; Graduated pipette (25ml / 10ml / 5 ml), 1000 ppm NaCl/KCl Stock solution , 1000 ppm $CaCO_3$/ $LiCO_3$ Distilled Water.

PROCEDURE

Step 1: Calibration of flame photometer

1. Prepare the standard solutions of 1 ppm, 9 ppm (for low concn. Mode) and 10 ppm, 50 ppm, 100 ppm (for high concn. mode) in 100ml / 50 ml volumetric flasks from the stock solution of NaCl / KCl.
2. Similarly prepare the standard solutions for Ca / Li in low and high concentrations using the stock solution of $CaCO_3$/$LiCO_3$.
3. Switch on the instrument by following the instructions written on the instrument and choose a setup number.
4. Set the instrument to low or high concentration mode and start calibration in each mode by aspirating the standards. Use distilled water as blank.
5. Now, after calibration, aspirate the standards and check whether they are in proper range and note the readings displayed.
6. From the supplied unknown solution of Na / K and Ca / Li, prepare the sample solutions in low or high concentration modes in 100 ml /50 ml volumetric flask or by diluting them 100,200, 300 times (A, B, C etc.) and again calibrate the instrument with the sample solutions by choosing different setup number.
7. Now the samples are aspirated and analyzed under flame photometer and the readings are noted.
8. The concentrations of the respective solutions (A, B & C) for Na/K and Ca/Li are multiplied by the respective dilution factors to get the mean concentration of Na/K and Ca/Li which gives the original concentration of Unknown Solution.
9. Save your calibration file in the directory and note the set up number for low and high concn. Modes.

Table – I: Calibration of device using Standard solutions

S.No.	Dilution Factor (i)	Concentration of Standard soln. (ppm)		Flame Photometer Reading (ppm)	
		Entered Na / K (p / q)	Displayed Na / K (x / y)	Na = [x * i]	K = [y * i]
1		1			
2		9			
3		10			
4		50			
5		100			
6		Blank (H_2O)			

Table – II: Determination of Concentration of Unknown Soln for Na/K

S.No.	Dilution Factor (i)	Concentration of soln. (ppm)		Flame Photometer Reading (ppm)	
		Entered Na / K (x)	Displayed Na / K (y)	Na = [y * x * i]	K = [y * x * i]
1					
2					
3					

Mean Concentration of Na (ppm) = _____

Mean Concentration of K (ppm) = _____

OBSERVATIONS AND CALCULATIONS

Write the setup file number method chosen for the experiment for each step.

Draw tabular form for Ca / Li solution.

Show each and every calculation for the preparation of solutions with proper formulas, units etc.

Note

Prepare the solutions according to the flasks given with proper care.

Calibration can be done either both Na / K and Ca/Li or individual Na / K and Ca / Li separately.

Always use distilled water to avoid contamination. Standards and samples should not be exposed to the atmosphere for long periods due to contamination from air-borne particles to avoid error.

RESULTS AND COMMENTS

Solution	Concentration in PPM			
	Na	K	Ca	Li
Unknown Soln. 1			------	
Unknown Soln. 2				