A Guide to Single Channel Flame Photometer Analysis

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Group **Period**	**I**	**II**											**III**	**IV**	**V**	**VI**	**VII**	**VIII**
	1A																	**8A**
1	1 **H** 1.008	**2A**											**3A**	**4A**	**5A**	**6A**	**7A**	2 **He** 4.003
2	3 **Li** 6.939	4 **Be** 9.0122											5 **B** 10.811	6 **C** 12.011	7 **N** 14.007	8 **O** 15.999	9 **F** 18.998	10 **Ne** 20.183
3	11 **Na** 22.99	12 **Mg** 24.312	**3B**	**4B**	**5B**	**6B**	**7B**	**[---------------8B-------------]**			**1B**	**2B**	13 **Al** 26.982	14 **Si** 28.086	15 **P** 30.974	16 **S** 32.064	17 **Cl** 35.453	18 **Ar** 39.948
4	19 **K** 39.102	20 **Ca** 40.08	21 **Sc** 44.956	22 **Ti** 47.9	23 **V** 50.942	24 **Cr** 51.996	25 **Mn** 54.938	26 **Fe** 55.847	27 **Co** 58.933	28 **Ni** 58.71	29 **Cu** 63.546	30 **Zn** 65.37	31 **Ga** 69.72	32 **Ge** 72.59	33 **As** 74.922	34 **Se** 78.96	35 **Br** 79.904	36 **Kr** 83.8
5	37 **Rb** 85.47	38 **Sr** 87.52	39 **Y** 88.905	40 **Zr** 91.22	41 **Nb** 92.906	42 **Mo** 95.94	43 **Tc** [97]	44 **Ru** 101.07	45 **Rh** 102.91	46 **Pd** 106.4	47 **Ag** 107.87	48 **Cd** 112.4	49 **In** 114.82	50 **Sn** 118.69	51 **Sb** 121.75	52 **Te** 127.6	53 **I** 126.9	54 **Xe** 131.3
6	55 **Cs** 132.91	56 **Ba** 137.34	57* **La** 138.91	72 **Hf** 178.49	73 **Ta** 180.95	74 **W** 183.85	75 **Re** 186.2	76 **Os** 190.2	77 **Ir** 192.2	78 **Pt** 195.09	79 **Au** 196.97	80 **Hg** 200.59	81 **Ti** 204.37	82 **Pb** 207.19	83 **Bi** 208.98	84 **Po** 209	85 **At** 210	86 **Rn** 222
7	87 **Fr** 215	88 **Ra** 226.03	89** **Ac** 227.03	104 **Rt** [261]	105 **Db** [262]	106 **Sg** [266]	107 **Bh** [264]	108 **Hs** [269]	109 **Mt** [268]	110 [271]	111 [272]	112 [277]		114 [289]		116 [289]		

*** Lanthanides**	58 **Ce** 140.12	59 **Pr** 140.91	60 **Nd** 144.24	61 **Pm** 145	62 **Sm** 150.35	63 **Eu** 151.96	64 **Gd** 157.25	65 **Tb** 158.92	66 **Dy** 152.5	67 **Ho** 164.93	68 **Er** 167.26	69 **Tm** 168.93	70 **Yb** 173.04	71 **Lu** 174.97
**** Actinides**	90 **Th** 232.04	91 **Pa** 231	92 **U** 238	93 **Np** 237.05	94 **Pu** 239.05	95 **Am** 241.06	96 **Cm** 244.06	97 **Bk** 249.08	98 **Cf** 251	99 **Es** 252.08	100 **Fm** 257.1	101 **Md** 258.1	102 **No** 259.1	103 **Lr** 262.11

Flame Photometer technology is an inherently simple and reliable methodology which has been applied to a range of products encompassing many fields of applications.

The aim of this booklet is to introduce the reader to the simple technique of low temperature flame photometry and to discuss in some detail some of the many and varied applications in which the technique can be utilised.

Contents

Introduction

The estimation of the alkali metals by flame photometry is by far its most important application in routine chemical analysis. For this widespread requirement, low temperature flame photometry provides the most reliable and convenient procedure available.

This technique has considerable appeal in the clinical chemistry field as it provides a rapid and reliable means of estimating sodium, potassium and lithium in body fluids. The methodologies of such analyses are well known and indeed well established and shall therefore not be discussed in this booklet.

However the estimation of the alkali and alkaline earth metals is commonly required in a sample matrix which does not lend itself to simple and direct analysis involving only a dilution step, e.g. sodium in fuel oil. This booklet therefore aims to guide the user towards making successful determinations in a wide variety of sample media by giving in detail a large selection of procedures using flame photometry from which other methodologies may be appended with relative ease.

Principles of Flame Photometry

The basis of low temperature flame photometry is the same as that of the simple quantitative analytical flame test. This exploits the fact that compounds of the alkali and alkaline earth metals are thermally dissociated into atoms at the temperature of a Bunsen burner flame and that some of these atoms produced are further excited to a higher energy level. When these ‘excited’ atoms return to the ground state, they emit radiation, which for the elements of these two groups lies mainly in the visible region of the electromagnetic spectrum.

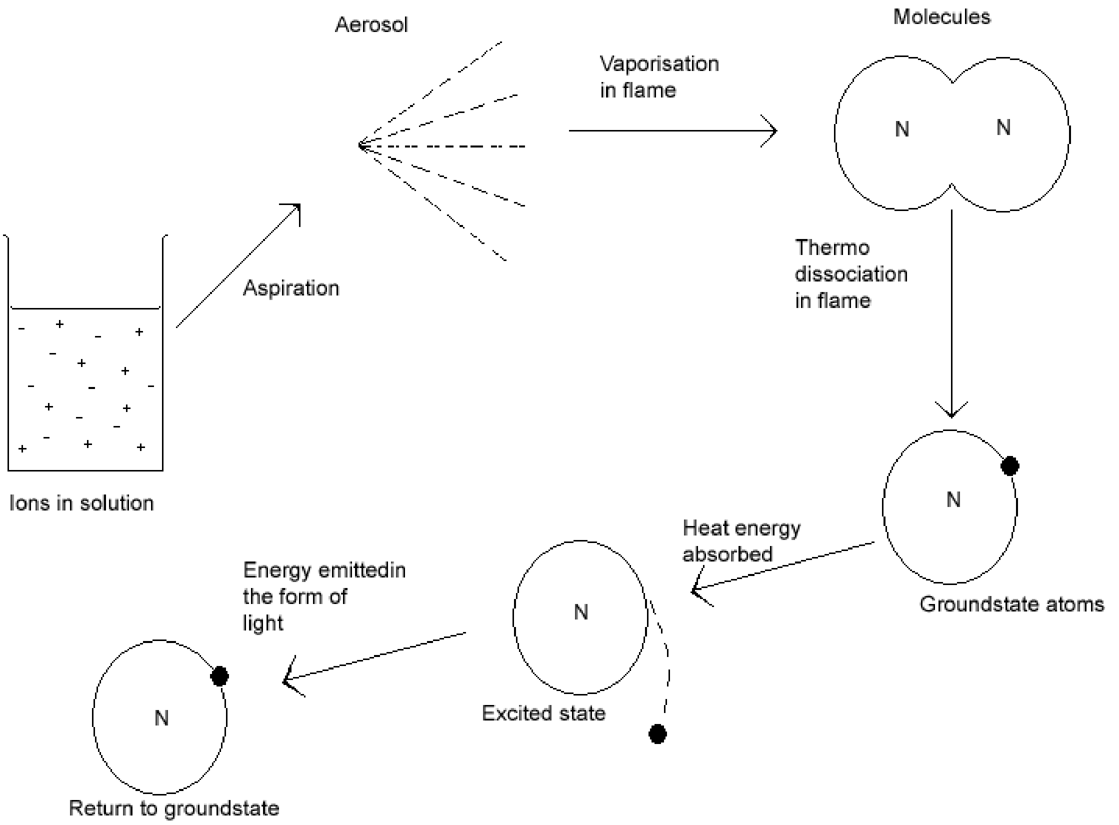

Fig. 1. The process involved in flame photometry.

The wavelength of the light emitted from the flame is characteristic of the particular element.

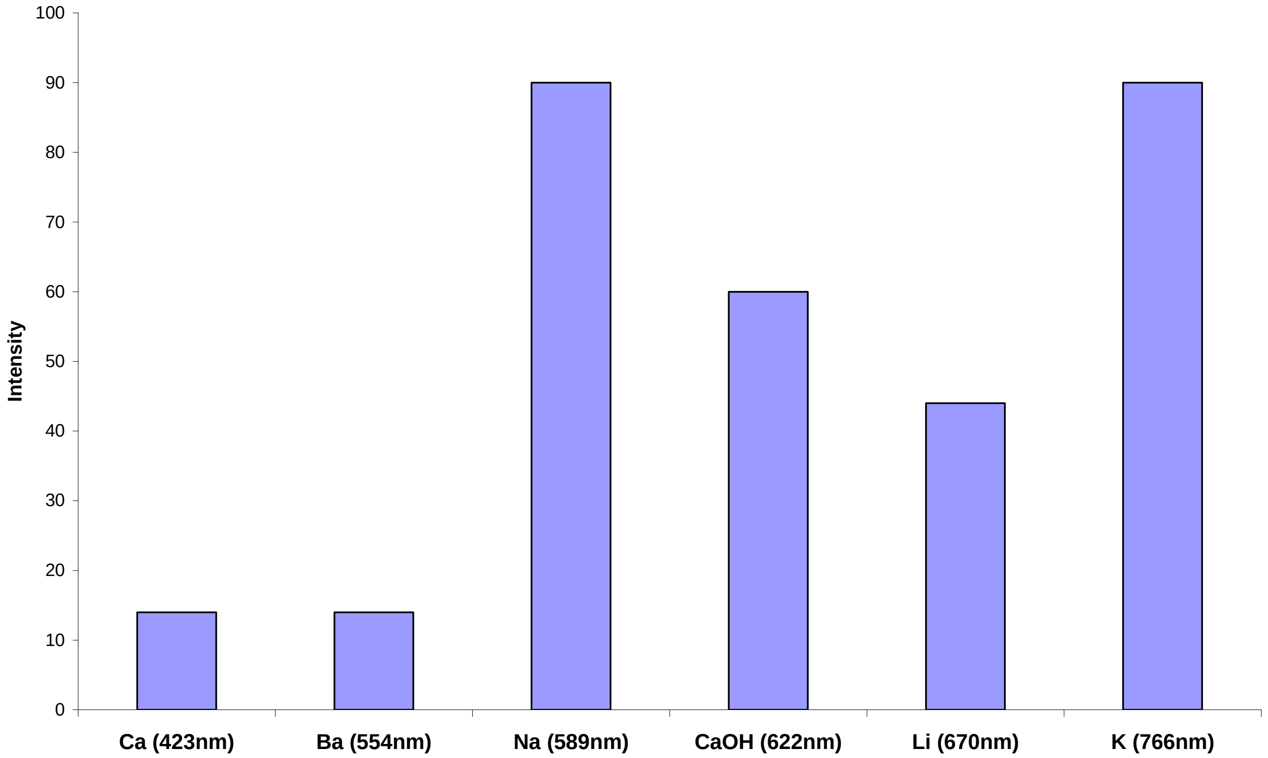

Fig. 2. Intensities of emissions of the elements at equal concentrations and their wavelengths.

The intensity of this light is, in most cases, proportional to the absolute quantity of the species present in the flame at any moment, i.e. the number of atoms returning to the ground state is proportional to the number of atoms excited, i.e. the concentration of the sample.
N.B. This relationship applies only at low concentrations.

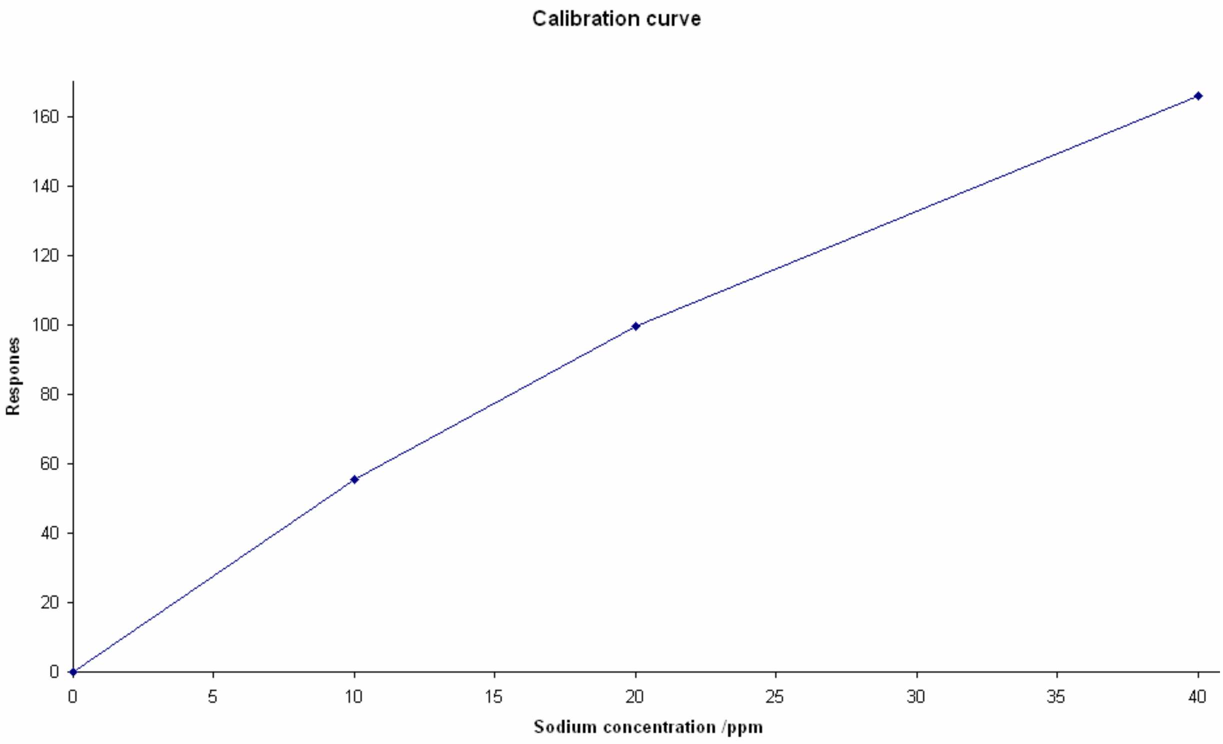

Fig. 3. An example of a calibration curve for an industrial measurement of sodium concentration.

The emitted radiation is isolated by an optical filter and then converted to an electrical signal by the photo detector

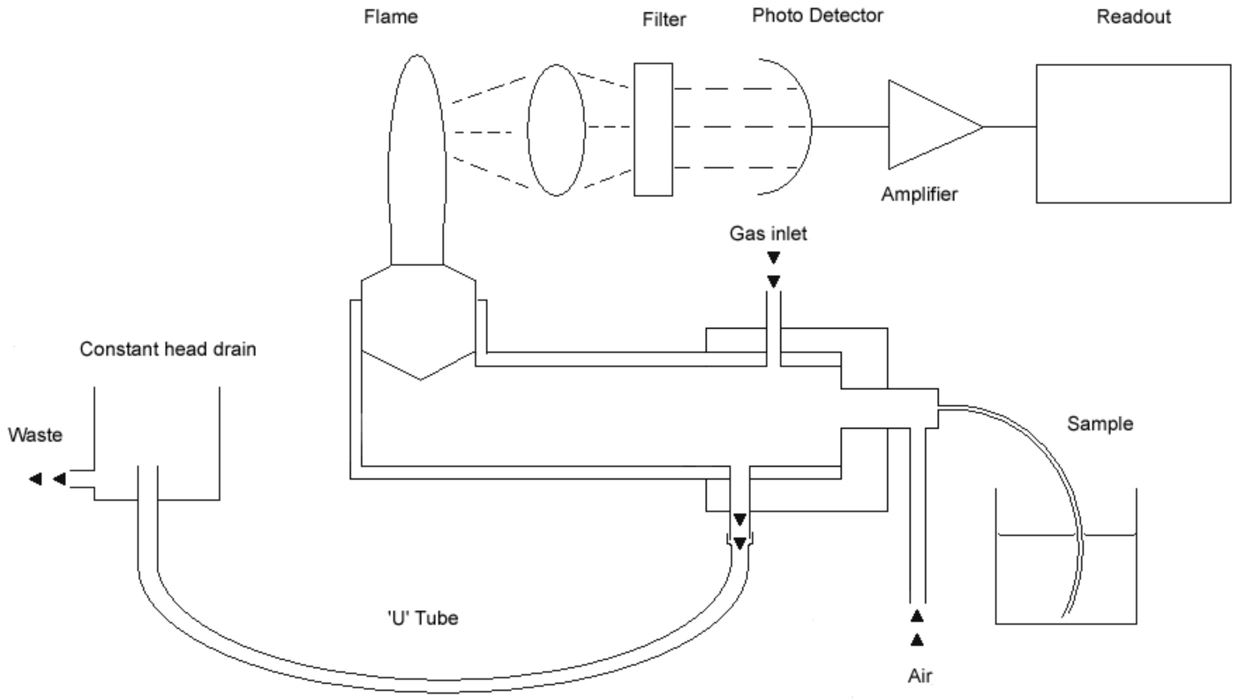

Fig. 4. Basic components of a simple flame photometer

A simple flame photometer consists of the following basic components:

1. A flame that can be maintained in a constant form and at a constant temperature:- “**The Burner**” (see Fig. 4)
2. A means of transporting an homogeneous solution into the flame at a steady rate:- “**Nebuliser and mixing chamber**” (see Fig. 4)
3. A means of isolating light of the wavelength to be measured from that of extraneous emissions:- “**Simple colour filters**” (interference type) (see Fig. 4)
4. A means of measuring the intensity of radiation emitted by the flame:- “**Photo Detector**” (see Fig. 4).

Brief History

As a direct result of the work done by Kirchhoff and Bunsen in the early 1860’s, the possibility of using the characteristic radiation emitted by atoms excited in flames for quantitative analysis was realised. Soon after, an instrument was developed for the quantitative analysis of sodium in plant ash using a Bunsen flame.

The major problems experienced over the next 60 years were that of finding a reproducible method of introducing a sample into a flame and to then find a convenient technique to measure the emission intensity

These difficulties were largely overcome by Lundegardh in the 1920’s whose apparatus included a nebuliser which enabled the sample to be presented to the air / acetylene flame in aerosol form. The emission was dispersed by a quartz prism spectrograph and recorded photographically. Precision was typically 5-10%.

Greater convenience resulted from the introduction of simple coloured filters for wavelength selection, together with photocell/galvanometer combinations for measuring intensities directly. As a result simple and inexpensive instruments of this type using air/coal gas or air/acetylene flames became widely available from the late 1940’s for the determination of sodium, potassium, lithium and calcium.

Work on many other elements then became possible with the use of grating spectrometers equipped with photomultiplier detectors through to the development of atomic absorbtion in the late 1960’s which restricted the use of flame emission.

However, there is a widespread requirement for the estimation of the alkali metals and for this purpose low temperature flame photometry provides the most reliable and convenient procedure available.

Flame Photometer Operating Data

The analysis of alkali and alkaline earth metals by flame photometry has two major advantages.

1. Their atoms reach the excited state at a temperature lower than that at which most other elements are excited.

2. Their characteristic wavelengths are easily isolated from those of most other elements due to wide spectral separation.

Table 1 shows the temperature achieved by a variety of gas mixtures:

Mixture	**Temperature (°C)**
Acetylene/oxygen	3,100-3,200
Acetylene/nitrous oxide	2,900-3,000
Natural gas/oxygen	2,700-2,800
Hydrogen/Oxygen	2,500-2,700
Acetylene/air	2,100-2,400
Hydrogen/air	2,000-2,100
Propane/air	1,900-2,000

Butane/air	1,300-1,900
Natural gas/air	1,700-1,800

Table 1.

The analysis of Na, K, Li, Ba and Ca are typically determined at low temperatures, i.e. 1500-2000°C, therefore suitable mixtures are propane/air, butane/air and natural gas/ air.

Characteristic Wavelengths of the Elements

It is common knowledge that when sodium is introduced into a flame it emits a radiation in the yellow region of the visible spectrum. Table 2 gives details of the measurable atomic flame emissions of the alkali and alkaline earth metals in terms of the emission wavelength and the colours produced.

Element	**Emission Wavelength (nm)**	**Flame Colour**
Barium (Ba)	554	Lime Green
Calcium (Ca)	622*	Orange
Lithium (Li)	670	Red (Carmine)
Potassium (K)	766	Violet
Sodium (Na)	589	Yellow

Table 2.

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

The other elements of group I and II in the periodic table also give a characteristic and sometimes intense band of emitted radiation when introduced into a flame (see Table 3). However, if these are to be utilised analytically then factors such as the need for higher flame temperatures and measurements outside the visible spectrum should be considered.

Element	**Emission Wavelength (nm)**	**Flame Colour**
Caesium (Cs)	852	**
Magnesium (Mg)	285	*
Rubidium (Rb)	780	Violet
Strontium (Sr)	461	Scarlet

Table 3.

* The main emission peak for Mg is outside the visible spectrum.
**The main emission peak for Cs is outside the visible spectrum, however, modern detectors are sensitive enough to enable Cs and Rb to be measured in the absence of interferents, e.g. potassium.

NOTE: Berylium may be determined by flame photometry by virtue of emission at 471 nm due to the molecular band emission from the monoxide (BeO).

What you will need

The list given in this section outlines the basic equipment and reagents required to perform analysis by low temperature flame photometry.

1. Flame Photometer.
2. Regulated gas supply.
3. Mains regulated air supply.
4. Power supply (90—250V ac).
5. Stock standard solutions, e.g. 1000 ppm Na, K, Li, etc.
6. Volumetric flasks for standard and sample dilution.
7. Glass beakers or suitable sample vessels.
8. Graph paper or software package to plot calibration graph.

NOTE: For medical systems a dilutor is commonly used while in many industrial applications sample preparation involves the use of, e.g. centrifuge, muffle furnace, filtration equipment, etc.
Medical sample concentrations are commonly expressed in mMoldm^{-3} in contrast to ppm (parts per million) which is the typical unit used for industrial analysis. The relationship between the two is as follows.

$$\text{ppm} = A \times \text{mMoldm}^{-3} \quad \text{and} \quad \text{mMoldm}^{-3} = \frac{\text{ppm}}{A}$$

where A=atomic mass of the atom being analysed.

Preparing for Analysis

1. Initial Precautions

It is of the utmost importance to become familiar with the flame photometer and ancillary equipment prior to analysis. If familiarity is not achieved, inaccuracy of results or even a hazard to safety could result. Therefore, always read the instrument's instruction manual. There are several practical points which should be adhered to to achieve the required accuracy in your analysis.

1. Avoid handling samples with fingers. This leads to serious contamination, e.g. if a finger is immersed in 20 mls of deionised water the resulting Na' concentration will exceed that of a 10 ppm standard.

2. Standards should be stored in sealed vessels and in high concentrations, i.e. store the standards as a stock 1000 ppm solution and prepare dilutions when required. The long term storage of low concentration standards is not recommended due to degradation of the ionic species.

3. All analyses involve the use of a diluent which is almost always deionised water. This should be of the highest quality for accurate flame analysis. Sodium, potassium and calcium are present in high concentrations in tap water and thus efficient deionisation is essential if any of the common flame analytes are to be determined.

4. Species that cause interference should be removed from samples or the equivalent concentration of interferent should be present in the standards so as to avoid erroneous results, e.g. if a sample of approximately 10 ppm Na contains approximately 1000 ppm Ca^{2+} then Na^+ analysis can only be achieved by removing Ca^{2+} using oxalate/oxalic acid or by ensuring all standards contain 1000 ppm Ca^{2+}
   Always follow a well documented analytical procedure (see Application Methods) which should contain information pertaining to interference removal when applicable.

5. Standards and samples should not be exposed to the atmosphere for long periods due to contamination from airborne particles and the evaporation of the solvent leading to elevated concentrations.

When in doubt about equipment or application the operator should contact the manufacturer for advice.

2. Sample Preparation

The sample, if not already a solution, must be converted to a media which is suitable for direct introduction into the flame photometer i.e. the sample should be aqueous with no solid matter present.
This is achieved by:

1. Extracting the salts from solid samples using deionised water or suitable extractants, e.g. saturated $CaSO_4$ for sodium in soil. Extraction is made more successfully using a blender macerator or shaking machine.

2. If the sample is organic then the organic material should be removed by ashing. The remaining oxides are then dissolved using strong acids.

3. Filtration/centrifugation to remove solid debris.

When aqueous, the sample can then be diluted to a **known,** accurately measured volume using deionised water. If it is a concentrated sample then the dilution ratio should be increased. If the sample concentration is low then a small volume of diluent and initial extractant should be used.

3. Standard Preparation

When preparing standards always observe the following:

1. Standards must always contain the constituents that are present in the samples in the same concentration ratios (element being sensed not included), i.e. if samples are prepared in 0.05M HCl then the standards should also contain 0.05M HCl.

2. Always ensure that the standards encompass the expected range of sample concentrations.

3. Standards should be prepared so as to ensure that the region in which measurements are made coincide with the concentrations that produce the optimum performance from the flame photometer, i.e.:

   when measuring **sodium** the top standard is ideally 10 ppm;
   when measuring **potassium** the top standard is ideally 10 ppm;
   when measuring **calcium** the top standard is ideally 100 ppm;
   when measuring **barium** the top standard is ideally 1000 ppm.

4. Four standards should be prepared to enable an accurate calibration plot to be drawn.

NOTE: that the blank used should contain all the constituents of the standard solutions except the element that gives rise to the emission.

Making a Measurement

1. Prepare standard and sample solutions (see section 6).

2. Power up the flame photometer in accordance with the instruments instruction manual

3. Set blank with the diluent used for sample and standard preparation. This is usually deionised water

4. Aspirate the top standard and adjust the display to the required reading.

5. Re-check the blank and standard settings.

6. Aspirate the prepared standards in increasing concentrations and record their stable display readings.

7. Plot a graph of display reading (intensity) against standard concentration on linear graph paper

8. Aspirate the unknown solution and record the stable display reading.

9. Determine the sample concentration by interpolation from the calibration plot.

10. Operate the instrument shutdown procedure.

NOTE: When using clinical flame photometers a calibration curve is not required as the display is calibrated in direct concentration units, therefore only one standard is required to enable this calibration to be performed.

Applications Methods

The following applications methods will enable the user to determine alkali and alkaline earth metal concentrations in a wide variety of sample media.

It is envisaged that, from the information given in this section, further applications methods will be appended with relative ease.

The procedures detailed in this section are as follows:

1. Determination of alkalis in cement
2. Determination of calcium in beer
3. Calcium in biological fluids
4. Estimation of calcium in milk
5. Determination of potassium in fertilizers
6. Determination of potassium in plant material
7. Determination of available potassium in soils
8. Sodium and potassium in silicates, minerals and ores
9. Determination of sodium in raw pine oil
10. Determination of exchangeable sodium in soils
11. The flame photometric determination of sulphate
12. The determination of sodium in fuel oil
13. The determination of potassium in mixtures containing plant derived resins
14. The determination of lithium in greases
15. The simple flame photometric determination of barium
16. The simple flame photometric determination of calcium
17. The determination of calcium in fruit juice
18. The determination of potassium in glass
19. The determination of sodium in glass
20. Sodium in straw by flame photometry
21. Determination of sodium and potassium in fruit juice
22. The determination of calcium in biscuits

Determination of Alkalis in Cement

This method describes how soda and potash may be estimated in cement using a flame photometer. The addition of aluminium nitrate solution suppresses the interference caused by concentrations of lime up to 350mg/litre which is the equivalent concentration for cement, allowing for the effect of silica and alumina already present in solution.

For materials other than cement, the addition of aluminium nitrate can be adjusted according to the lime concentration expected. 30ml of the solution is sufficient for a CaO concentration of 1 g/litre. Excess of aluminium nitrate is not harmful.

Equipment Required:

1. Single channel Flame Photometer
2. Balance weighing ±0.0005g
3. Whatman No. 40 filter paper
4. Sodium chloride (analar)
5. Potassium chloride (analar)
6. Concentrated HCl
7. Aluminium (high grade)
8. Concentrated HNO_3

Reagent Preparation:

Standard Solution: This contains 10mg/litre K_2O and 5mg/litre Na_2O. Dissolve 0.7915g KCl (analar) and 0.4715g NaCl with distilled water in the same 1 litre volumetric flask. Dilute to the mark with distilled water and mix well by inversion. This solution contains 500mg/ litre K_2O. Dilute exactly 20mls of this solution in a 1 litre volumetric flask with distilled water and add 10ml of concentrated HCl very slowly. Dilute to the mark with distilled water.

This is the universal standard containing 10mg/litre K_2O and 5mg/litre Na_2O and can be used for all purposes.

Aluminium Nitrate Solution: Dissolve 30g of aluminium known to have a low alkali content, (high grade) in 400ml of 50% HNO_3. Add a few drops of mercuric chloride to aid solution of aluminium. When the aluminium has dissolved and the solution cooled dilute to 1 litre with distilled water.

Method:

1. Mix 0.5g cement with 20-30ml of distilled water, add 5ml of concentrated HCl and dilute to about 50ml.
2. Heat the solution to boiling.
3. Filter the solution through a 9cm No. 40 Whatman filter paper into a 500ml volumetric flask.
4. Wash the filter paper 6 times with hot water
5. Add 10ml of the aluminium nitrate solution.
6. Cool the solution and dilute to the mark with distilled water
7. Prepare a series of standards by dilution of the universal standard ensuring that each prepared standard contains the equivalent of 1 ml of concentrated HCl per 100mls.
8. Aspirate the set of standards directly into the Flame Photometer and plot a calibration curve of Na_2O concentration vs meter reading.

9. Aspirate the cement sample solution and read off the Na_2O concentration from the calibration curve.

10. Repeat stages 8 and 9 using the potassium interference filter to determine the K_2O concentration in the cement sample.

11. Calculate the alkali content of the cement by multiplying the mg/l K_2O and Na_2O present in the sample solution by the dilution factor.

The Determination of Calcium in Beer

The determination of calcium in beer may be carried out by preliminary separation of calcium as oxalate followed by the addition of perchloric acid, which releases the calcium from the inhibiting effect of the oxalate.

Equipment Required:

1. Single channel flame photometer with calcium filter
2. Graduated stoppered centrifuge tube
3. Standard calcium solution 100 mg/l (in 0.2 molar perchloric acid)
4. 1 % ammonium oxalate solution
5. 0.5% ammonium oxalate solution
6. 25% ammonia solution
7. 4M perchloric acid

Method:

1. Pipette 5ml beer into a 10ml graduated stoppered centrifuge tube.
2. Add 5mls of 1 % ammonium oxalate and 3 drops ammonia solution.
3. Shake, and allow to stand for 30 minutes.
4. Centrifuge at 2000 rpm for 2 minutes.
5. Decant the supernatant and allow the tube to drain inverted for 30 seconds.
6. Add 0.5ml of 4M perchloric acid and shake.
7. Heat for 1 minute in a boiling water bath.
8. Cool and dilute to the mark with distilled water.
9. Calibrate the flame photometer using a 100 ppm Ca^{2+} standard containing 50mls 4M $HClO_4$ per litre from which 60 and 40 ppm Ca^{2+} solutions have been prepared.
10. Aspirate the sample directly into the flame photometer.
11. The calcium concentration is then calculated by multiplying the flame photometer reading on the calibrated scale by the dilution factor.

Calcium in Biological Fluids

Calcium is precipitated out of the sample with an ammonium oxalate/oxalic acid mixture which also acts as a pH buffer. The mixture is then centrifuged and the supernatant fluid decanted off to be used for sodium and potassium determination if required. The precipitate is dissolved in perchloric acid and then aspirated into the calibrated flame photometer.

Equipment Required;

1. Single channel flame photometer
2. Balance weighing to ±0.0005g

Reagents Preparation:

Ammonium Oxalate/Oxalic acid mixture :

1. Dissolve 12.6g $(COOH)_2 2H_2O$ to1 litre distilled water in a volumetric flask. This is 0.1 M oxalic acid.

2. Dissolve 12.2g $(COONH_4)_2 H_2O$ to 1 litre of distilled water in a volumetric flask. This is 0.1 M ammonium oxalate.

3. To 5ml of 0.1 M oxalic acid in a 100ml volumetric flask add 0.1 M ammonium oxalate up to the mark.

0.05M Perchloric acid.

Half fill a 1 litre volumetric flask with distilled water, add 5mls of 60% perchloric acid and dilute to the mark.

Standard Preparation:

1. Dissolve 1.834g $CaCl_2.2H_20$ (analar) in 0.05M perchloric acid and dilute to 500mls. This is the calcium 1000mg/l stock standard.

2. Dilute the stock standard 1:10 with 0.05M perchloric acid. This is the calcium 100mg/l standard.

3. A range of standards is now prepared by successive dilution of the 100mg/l standard with 0.05M perchloric acid.

Method:

1. Pipette 2mls of serum into a rounded centrifuge tube and add 3mls of Ammonium oxalate/oxalic acid mixture.

2. Mix and stand for 30 minutes.

3. Centrifuge well and decant the supernatant fluid and drain the sample.

4. Add 4ml of 0.05M perchloric acid to the pellet, vortex or shake vigorously to dissolve the precipitate fully. The sample is now ready for analysis.

5. Treat 2ml of the calcium 100mg/l standard in the same way as the sample, i.e. stages 1-4, to obtain a control.

6. Select the calcium filter on the flame photometer.

7. Aspirate the range of calcium standards into the single channel flame photometer and plot meter reading vs concentration.

8. Aspirate the sample solution into the flame photometer and from the graph determine the calcium concentration.

9. Aspirate the control into the flame photometer and record the meter reading.

Calculation

$$\text{Concentration read from graph X } \frac{\text{100mg/l standard meter reading}}{\text{control meter reading}}$$

Estimation of Calcium in Milk

Equipment required:

1. Single channel flame photometer
2. Calcium filter
3. Accurate balance

Reagent Preparation:

Dilute concentrated hydrochloric acid 1:4 with distilled water.

CAUTION: ALWAYS ADD ACID TO WATER WITH GREAT CARE. NEVER ADD WATER TO CONCENTRATED ACID.

Blank Preparation:

2.5ml of diluted hydrochloric acid, 2.0ml 10% lanthanum chloride, make up to 100ml with distilled water to give reagent blank.

Standard Preparation:

Place 1.249gm A.R. calcium carbonate in approximately 50ml H_20, adding dropwise concentrated hydrochloric acid until calcium carbonate is dissolved (should take about 10ml). This will give 500 ppm stock calcium standard.

The stock solution should be diluted to give standards of 2.5, 5.0, 7.5 and 10 ppm Ca^{2+}

Sample Preparation:

Place 4gm (accurately weighed) of milk in a dry silica crucible. Ash sample at 500- 525°C oven. When cool, dissolve ash in 5ml of 1:4 diluted concentrated hydrochloric acid. Transfer to 100ml volumetric flask; make up to mark. Filter through Whatman filter paper, pipette 50ml of filtrate into 100ml volumetric flask. Add 2ml of 10% lanthanum chloride. Make up to mark with distilled water

Method:

1. Spray reagent blank, set zero.
2. Spray 10 ppm standard set full scale.
3. Reset zero.
4. Spray intermediate standards, note readings, construct calibration curve.
5. Spray test samples and note readings.

This gives you calcium in parts/million of calcium for milk. If percent calcium required multiply Ca^{2+} ppm sample reading by 0.025 = percent Ca^{2+} in milk. Normal level for milk is approximately 0.11%.

Determination of Potassium in Fertilizers

Equipment Required:

1. Single channel flame photometer
2. Balance accurate to ±0.0005g
3. Filter paper
4. Potassium chloride (analar)

Method:

1. Prepare a series of potassium standards covering the range 0-10 ppm using potassium chloride diluted with distilled water.
2. Weigh out accurately 10g fertilizer and add 300ml distilled water. Shake for 30 minutes.
3. Transfer the solution to a 500ml volumetric flask and dilute to the mark using distilled water.
4. Filter the solution.
5. If necessary dilute the filtrate to produce an expected concentration within the range 0-10 ppm potassium.
6. Aspirate the 10 ppm standard into the flame photometer and adjust the controls to give a suitable display reading. Aspirate each of the other standards in turn, record their readings and plot a curve of intensity against concentration of potassium.
7. Aspirate the sample into the flame photometer and record the result.
8. From the graph obtain the concentration of potassium in the sample.
9. The percentage potassium contained in the fertilizer can be determined taking into account the dilution factor

The Determination of Potassium in Plant Material

Equipment Required:

1. Single channel flame photometer
2. Balance weighing to ±0.0005g
3. Water bath and oven
4. 10 Molar Hydrochloric acid
5. Potassium chloride (analar)

Method:

1. Completely oxidise at 500-525°C 2-3g of the ground oven dried sample.
2. Dissolve the ash in 10 molar Hydrochloric acid and allow it to digest on a steam bath for 20 minutes.
3. Evaporate the solution to dryness and heat the residue in an oven for 30 minutes at 105°C.
4. Allow to cool, return to the water bath and add 10ml 10 molar Hydrochloric acid. Warm to dissolve.
5. Transfer 2mls to a 100ml volumetric flask and dilute to the mark with distilled water.
6. Prepare a 5 ppm potassium standard by dissolving KCl (analar) in an HCl/water mixture containing 2ml conc. HCl per 100ml.
7. Prepare a calibration curve using progressive dilutions of the 5 ppm standard using the HCl/water mixture as diluent.
8. Aspirate each standard into the flame photometer in turn, and plot meter reading against potassium concentration
9. Aspirate the sample solution and from the graph determine the potassium concentration.
10. The amount of potassium in the plant material sample can be calculated taking into account the dilution factor.

The Determination of Available Potassium in Soils

Equipment Required:

1. Single channel flame photometer
2. Whatman No. 30 filter paper
3. Potassium chloride (analar)
4. Balance weighing to ±0.0005g
5. Ammonium acetate
6. Acetic acid

Method:

1. Prepare 0.5M aqueous solution of ammonium acetate/acetic acid by taking 38.55g ammonium acetate and dissolving it in 29mls of glacial acetic acid and diluting to 1 litre with distilled water

2. Accurately weigh 10g of soil and transfer to a plastic bottle together with 50ml of acetate/acetic acid solution.

3. Stopper the bottle and shake using an automatic shaker for 30 minutes.

4. Remove from the shaker, allow to stand for several minutes. Filter the solution through a Whatman No. 30 filter paper

5. Prepare standard potassium solutions to cover the range 0-100 ppm as follows: Accurately weigh 1.907g potassium chloride in about 50mls ammonium acetate/ acetic acid solution, Transfer to a 500ml volumetric flask and dilute to the mark with ammonium acetate/acetic acid solution. This solution contains 2000 ppm potassium. Transfer 25mls of this stock solution into a 500ml volumetric flask and dilute to the mark with ammonium acetate/acetic acid solution. This is the 100 ppm potassium solution. From this prepare 80, 60, 40 and 30 ppm standards using the CH_3COONH_4/CH_3COOH solution as diluent.

6. Aspirate these standards into the flame photometer and construct a calibration graph of meter reading against potassium concentration.

7. Determine the potassium content of the soil extract by spraying the solution without further dilution into the flame photometer and reading off from the calibration graph the potassium concentration.

Sodium and Potassium in Silicates, Minerals and Ores

Equipment Required:

1. Single channel flame photometer
2. Accurate balance
3. Platinum crucible
4. Volumetric glassware.
5. 48% Hydrofluoric acid
6. Concentrated Sulphuric acid (analytical quality)
7. Hydrochloric acid

Sample Preparation:

Transfer 1 gm of sample to 30ml platinum crucible, moisten the sample with about 5 drops of water and an equal volume of concentrated sulphuric acid and add 5 ml hydrofluoric acid.

Place the platinum dish in a porcelain dish. Heat the porcelain dish on a hot plate at a rate that will slowly evolve white fumes of sulphuric acid without boiling or splattering. When the sample is almost dry add a second portion of H_2S04 and HF and repeat the slow evaporation. Repeat a third time to make sure that the rock is completely decomposed. On the third evaporation take care to evaporate the sample to complete dryness driving off as much Fluoride as possible. There is a tendency to splatter at this point and the heat must be carefully controlled. Use hot 1:9 HCl to transfer the metallic sulphates to a 250ml beaker. About 175mls of hot acid are sufficient with heating and stirring using a glass rod. Transfer the solution to a 200ml volumetric flask, cool and fill to the mark with 10% (V/V) hydrochloric acid. This provides a 0.5% solution of the rock sample in 10% (V/V) HCl. Having prepared the sample in this way a further dilution may be necessary prior to presentation of sample to nebuliser.

Standard Preparation:

Prepare a series of sodium and potassium standards from the chlorides (10, 6, 4 and 2 ppm) ensuring that each standard contains the same concentration of HCl as the sample.

Method:

1. Aspirate the standards in increasing concentration into the flame photometer.
2. Plot a calibration curve of intensity vs concentration.
3. Aspirate the unknown solution and record the display reading (intensity).
4. Read off the sample concentration from the calibration curve.

The Determination of Sodium in Raw Pine Oil

This method is used to determine sodium in raw pine oil from a minimum concentration of 1 ppm. It involves conversion of the sample into an inorganic residue by ashing. The residue dissolved in sulphuric acid is diluted with distilled water, and sodium is determined using a flame photometer.

Equipment Required:

1. Single channel flame photometer
2. Small crucible (nickel or porcelain)
3. Bunsen burner, tripod, etc
4. Accurate balance weighing to ±0.0005g
5. Anhydrous sodium sulphate (analar)
6. Concentrated sulphuric acid
7. Distilled water

Sample Preparation:

1. Weigh accurately 1 g of raw pine oil into a small clean crucible.
2. Heat the sample under a Bunsen burner in a fume cupboard. Ignite the vapours and ensure the sample burns freely. Re-ignition may be necessary before only a char remains.
3. Transfer the crucible to a muffle furnace pre-heated to 500-525°C and maintain this temperature until all carbon is burned off.
4. Allow the crucible to cool and wash the ash off the sides with a little distilled water.
5. Add 1 ml of concentrated sulphuric acid to fully dissolve the ash.
6. Carefully add distilled water to the crucible and transfer the solution and washing to a 100ml volumetric flask.
7. Dilute to the mark using distilled water.

NOTE: If carbon is present the solution should be filtered. However, all carbon should be removed by correct heating (stages 2 and 3).

Standard Preparation:

Stock Standard (1000 ppm Na): Dissolve 1.5435g of anhydrous sodium sulphate in distilled water and dilute to 500ml. Store in an air tight polythene bottle.

Working Standard (5 ppm Na): Dilute 5ml of the stock standard to about 900mls with distilled water. Add 10ml of concentrated sulphuric acid and dilute to the mark with distilled water

Prepare 3, 2 and 1 ppm Na standards by dilution of the working standard ensuring that each contains the equivalent of 1 ml concentrated H_2SO_4 per 100mls.

Method:

1. Aspirate the standards into the flame photometer. Plot a standard curve of sodium concentration against the meter reading.
2. Aspirate the sample solution into the flame and record the meter reading.

3. From the graph read off the sample sodium concentration.
4. To calculate sodium concentration in mg/g of the raw pine oil divide the concentration read off the graph by 10.

NOTE: Calcium may be determined using the same sample solution. However, a calcium interference filter is required for the flame photometer

The Determination of Exchangeable Sodium in Soils

Equipment Required:

1. Single channel flame photometer
2. Balance weighing to ±0.0005g
3. Centrifuge
4. Sodium chloride (analar)
5. 1 molar ammonium acetate solution

Method:

1. To 10g soil add 200ml of 1 molar neutral ammonium acetate solution. Allow to stand for 18 hours stirring occasionally.
2. Withdraw and centrifuge the suspension.
3. Transfer 5ml of the supernatant liquid to a 50ml volumetric flask and dilute to the mark.
4. Prepare a 5mg/l sodium standard in 1 molar neutral ammonium acetate solution.
5. Prepare from the above 2.5mg/l and 1.25mg/l Na standards in 1 molar ammonium acetate solution.
6. Aspirate the 5mg/l standard into the flame photometer and adjust the display to a suitable reading.
7. Aspirate the other standards and plot a standard curve of meter reading vs concentration.
8. Aspirate the sample and record its meter reading.
9. Read off the concentration of the sample from the standard curve.
10. Multiply the mg/l sodium value by 2 to give mg sodium per 10g soil.

The Flame Photometric Determination of Sulphate

The sulphate present in the sample is dissolved in water and precipitated out of solution using an excess of barium chloride. The precipitate is removed by centrifugation and is then dissolved in ammonium Ethylyenediaminetetra-acetate. The Ba^{2+} complex formed is then aspirated directly into the flame and the sulphate content of the sample calculated from the barium concentration recorded. Samples must be free of anions that produce insoluble barium salts other than sulphate.

Equipment Required:

1. Single channel flame photometer
2. barium interference filter
3. An accurate balance weighing to ±0.0005g
4. Centrifuge
5. Sodium sulphate (analar)
6. Concentrated hydrochloric acid
7. Barium chloride solution (1 % W/V)

Reagents Preparation:

Ammonium Ethylenediaminetetra-acetate solution: Dissolve 5g of Ethylenediaminetetra-acetic acid in 100ml deionised water add 50ml of ammonia solution (sp gr 0.88) and dilute to 500ml with deionised water

Preparation of Stock Standard:

Dissolve 0.6713g sodium sulphate (analar) in deionised water and dilute to 100mls. This is 2000 ppm SO_4^{2-} stock standard.

Method:

1. Accurately weigh a sample containing not more than 0.2g of sulphate as SO_4^{2-}, Dissolve in deionised water and transfer the solution to a 100ml volumetric flask.
2. Add 10ml of concentrated HCl. Mix thoroughly and dilute to the mark with deionised water.
3. Pipette 5ml of the sample solution into a 10ml calibrated centrifuge tube and add 5ml 0.1 % $BaCl_2$. Mix the solution well and spin in a centrifuge for three minutes at 4000 rpm.
4. Discard the supernatant liquid.
5. Re-suspend the pellet in 10ml of deionised water and spin again for 3 minutes at 4000 rpm.
6. Discard the supernatant liquid.
7. Add ammonium ethylenediaminetetra-acetate solution and stir until the pellet completely dissolves. Use gentle heat if necessary.
8. Dilute exactly up to the 10ml calibrated mark with the ammonium ethylenediaminetetra-acetate solution.
9. Subject a 5ml portion of the stock sulphate standard to identical treatment (i.e. stages 1 -9). This yields a final solution containing 1000 ppm SO_4^{2-}.
10. Using the ammonium ethylenediaminetetra-acetate solution as diluent prepare additional standards of 600, 300 and 100 ppm SO_4^{2-} from the stock standard.

11. With the barium filter in place aspirate the standards into the flame in increasing concentrations.

12. Plot a calibration curve of [Ba^{2+}] vs meter reading on linear graph paper.

13. Aspirate the sample into the flame and from the graph constructed interpolate the barium, hence sulphate, concentration in the sample solution.

Calculation:

To obtain the mass of sulphate in the original sample in mg divide the sample sulphate concentration from the graph by 5.

The Determination of Sodium in Fuel Oil

The sample is burned in air in a silica crucible on a hot plate and the residue ashed in a muffle furnace at 525°C. The ash is dissolved in sulphuric and nitric acids and the sodium content is determined using a flame photometer. This method is suitable for concentrations of sodium from 1 ppm upwards.

Equipment Required:

1. Single channel flame photometer
2. Silica crucible
3. Hotplate
4. Muffle furnace
5. Accurate balance
6. Concentrated sulphuric acid
7. Concentrated nitric acid

Reagent Preparation:

1000 ppm sodium stock standard solution: Dissolve 1.5435g anhydrous sodium sulphate (analar) in distilled water and dilute to 500ml. Store in an airtight polythene bottle.

Working Standards Preparation:

Prepare 5 ppm sodium by diluting 5ml of the stock standard to 500ml with deionised water in a 1 litre volumentric flask. Add 20ml of concentrated H_2S0_4 and 20ml of concentrated HNO_3 and mix well. Dilute to the mark with deionised water.

CAUTION: Never add water directly to concentrated acids. HNO_3 and must be handled with great caution.

From the acidified 5 ppm sodium standard prepare a series of diluted standards ensuring that each contains the equivalent of 20ml concentrated HNO_3 and 20ml concentrated H_2S0_4 per litre.

Method:

1. Heat the sample for 30 minutes at 60°C. Shake well.
2. Transfer about 10g into a clean, dry vessel and leave to cool to room temperature.
3. Weigh 10g accurately into a clean, dry silica crucible.
4. Heat the crucible and contents on a hot plate and ignite the oil. Re-ignite if necessary to produce a solid residue. Perform this step in a fume hood.
5. Transfer the crucible to a muffle furnace heated to 500°C and maintain the temperature until all of the carbon is burned off
6. Allow to cool. Rinse down any ash from the side of the crucible with a few ml of deionised water.
7. Add 1 ml of sulphuric acid (conc.) and 1 ml of nitric acid (conc.).

8. Replace on the hot plate and heat until fumes of SO_3 evolve.

9. Allow to cool. Add 10ml of deionised water.

10. Wash the contents of the crucible into a 50ml volumetric flask and dilute to the mark with deionised water. Carry out the test as soon as possible.

11. Aspirate the series of standards into the flame photometer in increasing concentrations using deionised water, containing the equivalent of 20mls of concentrated H_2S0_4 and 20mls concentrated HNO_3 per litre, as the blank solution.

12. Plot the concentration of the standards against the meter reading obtained to attain the calibration curve.

13. Aspirate the sample solution into the flame and from the meter reading obtained interpolate the sample concentration.

N.B. Dilute the sample if necessary using the blank solution to bring the sodium concentration within the standard range.

Calculation:

To obtain the weight of sodium in mg present in the original 10g of sample multiply the sample concentration reading obtained by 0.05.

The Determination of Potassium in Mixtures Containing Plant Derived Resins

This method is used to determine potassium in the presence of plant derived resins. The sample mixture is expected to contain about 10% K_2C0_3.

The sample is firstly converted into an inorganic residue by ashing, the residue dissolved in hydrochloric acid is diluted with distilled water and potassium is determined using a flame photometer.

Equipment Required:

1. Single channel flame photometer
2. Small crucible (nickel or porcelain)
3. Bunsen burner, tripod, etc
4. Accurate balance weighing to 0.0005g
5. Potassium dihydrogen ortho phosphate (analar)
6. 10 molar hydrochloric acid

Sample Preparation:

1. Weigh accurately 1 g of the resin sample into a small clean crucible.
2. Heat the sample using a Bunsen burner in a fume cupboard. Ignite the vapours and ensure the sample burns freely. Re-ignition may be necessary before only a char remains.
3. Transfer the crucible to a muffle furnace pre-heated to 500-525°C and maintain this temperature until all the carbon is burned off.
4. Dissolve the ash in hydrochloric acid and allow it to digest on a steam bath for about 20 minutes.
5. Evaporate the solution to dryness and heat the residue in an oven for 30 minutes at 105°C.
6. Return to the water bath and add 20ml of 10 molar HCl, warm to dissolve.
7. Transfer 2ml of the solution to a 1 litre volumetric flask containing approximately 200ml distilled water. Dilute to the mark with distilled water. The sample is now ready for analysis.

Standard Preparation:

Dissolve 3.481 g of potassium hydrogen ortho phosphate (previously dried for one hour at 105°C) in a 1 litre volumetric flask and dilute to the mark with distilled water. This is the 1000mg/l potassium standard.

Prepare from the stock standard 10, 6, 4 and 2mg/l potassium standards ensuring that each standard contains the equivalent of 2mls 10 molar HCl per litre.

Method:

1. With the potassium interference filter selected aspirate the standards into the flame photometer.
2. Plot a graph of meter reading vs concentration of potassium.
3. Aspirate the sample solution into the flame and record the meter reading.

4. From the graph read off the sample potassium concentration.

Calculation:

To calculate potassium concentration in mg/g of the resin mixture multiply the concentration of the sample from the graph by 10.

The Determination of Lithium in Greases

This method is suitable for the determination of lithium in conventional soap thickened greases. It may be used for both new and used samples but with less accuracy in the latter case.

Equipment Required:

1. Single channel flame photometer
2. Accurate balance weighing to ±0.0005g
3. Muffle furnace
4. Platinum crucible
5. lithium standard (1000 ppm)
6. Hydrochloric acid (conc.)

Method:

1. Weigh accurately about 1gm of grease in a platinum crucible.
2. Ignite by gentle heating over a Bunsen. When all volatile matter has burned off, transfer to a muffle furnace at 550°C and heat until all carbon is removed.
3. Allow to cool, cover the crucible with a watch glass. Add carefully 2 ml concentrated HCl, using the watch glass to retain splashes.
4. When the reaction has ceased wash splashes from watch glass into the crucible with a little deionised water and heat to dryness on a water bath.
5. Dilute (quantitatively) the sample with lithium blank until the expected concentration falls within the 0-10 ppm range.
6. From the stock lithium standard prepare 10, 7.5, 5 and 2.5 ppm standards.
7. Aspirate the standards into the flame photometer and plot a calibration curve of intensity vs concentration.
8. Aspirate the unknown solution into the flame and record the intensity value.
9. Interpolate the sample lithium concentration from the standard curve.
10. Adjust the sample concentration by taking account of the dilution ratio.

NOTE: If the sample contains significant concentrations of other cations then prepare standard solutions containing similar concentrations of these elements.

The Simple Flame Photometric Determination of Barium

This method is suitable for aqueous samples only which contain no interferent ions, e.g. SO_4^{2-}, K^+, etc.

Method:

1. From a 3000 ppm Barium standard prepare standards of 1000, 750, 500 and 250 using deionised water as diluent.

2. For lower concentration samples a set of Ba standards of 300, 200, 100 and 50 should be prepared. Do not set a lower top standard than 300 due to insensitivity of the technique to low Ba concentrations.

3. Aspirate the standards into the flame photometer and record the stable readings.

4. Plot a graph of intensity vs concentration on linear graph paper.
5. Aspirate the aqueous sample.

6. From the calibration graph obtain the sample concentration.

If there are interferent ions present Barium should be precipitated as Barium Sulphate, dissolved in ammonia EDTA and then analysed.

The Simple Flame Photometric Determination of Calcium

If aqueous samples of calcium are to be analysed which contain no interferent ions the following method should be employed.

Method:

1. From the 1000 ppm calcium stock standard prepare diluted standards of 100, 75, 50 and 25 ppm using deionised water as diluent.

2. If the samples to be analysed are of lower concentration then prepare 50, 40, 20 and 10 ppm standards. A top standard of less than 50 ppm is not entirely suitable due to low temperature flame photometry being insensitive to calcium at low concentrations.

3. Aspirate the standards into the flame photometer and record the stable readings.

4. Plot a graph of intensity vs concentration on linear graph paper.

5. Aspirate the aqueous sample.

6. From the calibration graph obtain the sample concentration.

If there are interferent ions present, calcium should be precipitated as calcium oxalate, dissolved in $HClO_4$ and then analysed.

The Determination of Calcium in Fruit Juice

The determination of calcium in fruit juice may be carried out by preliminary separation of calcium as oxalate formed by the addition of perchloric acid, which releases the calcium from the inhibiting effect of the oxalate.

Equipment Required:

1. Single channel flame photometer with calcium filter
2. Graduated stoppered centrifuge tube
3. Standard calcium solution 100 mg/l (containing perchloric acid)
4. 1 % ammonium oxalate solution
5. 0.5% ammonium oxalate solution
6. 25% ammonia solution
7. 4M perchloric acid

Method:

1. Pipette 5 ml fruit juice into a 10 ml graduated stoppered centrifuge tube.

2. Add 5 mls 1 % ammonium oxalate and 3 drops ammonia solution.

3. Shake, and allow to stand for 30 minutes.

4. Centrifuge at 2000 rpm for 2 minutes.

5. Decant the supernatant and allow the tube to drain inverted for 30 seconds.

6. Add 0.5 ml 4M perchloric acid and shake.

7. Heat for 1 minute in a boiling water bath.

8. Cool and dilute to the mark with distilled water.

9. Calibrate the flame photometer using a 100 ppm Ca^{2+} standard containing 50 mls 4M $HClO_4$ per litre from which 75, 50 and 25 ppm Ca^{2+} solutions have been prepared.

10. Aspirate the sample directly into the flame photometer.

11. The calcium concentration is then calculated by multiplying the flame photometer reading on the calibrated scale by the dilution factor.

The Determination of Potassium in Glass

Equipment Required:

1. Single channel flame photometer
2. Platinum crucible
3. Accurate balance weighing to 0.0005g
4. Sodium carbonate (analar)
5. 1000 ppm K flame photometer standard
6. Sodium hydroxide
7. Sodium chloride
8. Glacial acetic acid
9. Hydrochloric acid

Reagent Preparation:

Buffer Solution Dissolve 20g NaOH in 300 ml deionised water in a 600 ml beaker When completely dissolved add 30 ml glacial acetic acid. Add 29g NaCl and dissolve. Cool to ambient temperature and dilute to 1 litre in a volumetric flask with deionised water.

Blank Preparation:

To a 1 litre volumetric flask add 150 ml buffer solution and dilute to 1 litre using deionised water.

Standard Preparation:

To 10 mls of 1000 ppm K standard in a 1 litre volumetric flask add 150 ml buffer solution. Adjust to pH 7.5 with HCl. Dilute to the mark with deionised water. This is 10 ppm potassium. From the 10 ppm K standard prepare 7.5, 5 and 2.5 ppm standards using the blank solution as diluent. Ensure all standards have a pH value of 7.5.

Sample Preparation:

Fuse an accurately weighed powdered sample with 2g sodium carbonate in a platinum crucible. When a clear fused melt is obtained continue heating for approximately 1 minute. Remove the crucible from the burner and pour the contents onto a cooling plate.

Place the crucible lid and cooled melt into a 400 ml beaker, add 75 ml buffer solution and dilute to 200 ml using boiling deionised water

Place the beaker on a water bath for 30 minutes to loosen the melt from the crucible. Remove crucible and lid from beaker. Filter the solution into a 1 litre volumetric flask ensuring that deionised water washings from the beaker are also filtered.

Dilute to 300 ml and adjust the pH to 7.5 with 1:3 HCl. Dilute to the mark with deionised water

Method:

1. Set the flame photometer up as in its instruction manual.
2. Set the zero using the blank solution.
3. Aspirate the standards into the instrument recording each displayed value.

4. Construct a calibration plot of concentration vs. meter reading on linear graph paper

5. Aspirate the sample solution and record the stable display. (If the value is higher than that of the top standard, dilute sample with blank until the value falls in the range of the calibration curve.)

6. Multiply the potassium concentration of the sample by the dilution factor

The Determination of Sodium in Glass

Equipment Required:

1. Single channel flame photometer
2. Platinum crucible
3. Accurate balance weighing to ±0.0005g
4. Potassium carbonate (analar)
5. 1000 ppm Na flame photometer standard
6. Potassium hydroxide
7. Potassium chloride
8. Glacial acetic acid
9. Hydrochloric acid

Reagent Preparation:

Buffer Solution - Dissolve 28g KOH in 300 ml deionised water in a 600 ml beaker. When completely dissolved add 30 ml glacial acid. Add 37g KCl and dissolve. Cool to ambient temperature and dilute to 1 litre in a volumetric flask with deionised water.

Blank Preparation:

To a 1 litre volumetric flask add 150 ml buffer solution and dilute to 1 litre using deionised water.

Standard Preparation:

To 10 mls of 1000 ppm Na standard in a 1 litre volumetric flask add 150 ml buffer solution. Adjust to pH 7.5 with HCl. Dilute to the mark with deionised water. This is 10 ppm potassium. From the 10 ppm K standard prepare 7.5, 5 and 2.5 ppm standards using the blank solution as diluent. Ensure all standards have a pH value of 7.5.

Sample Preparation:

Fuse an accurately weighed powdered sample with 2g potassium carbonate in a platinum crucible. When a clear fused melt is obtained continue heating for approximately 1 minute. Remove the crucible from the burner and pour the contents onto a cooling plate.

Place the crucible lid and cooled melt into a 400 ml beaker, add 75 ml buffer solution and dilute to 200 ml using boiling deionised water.

Place the beaker on a water bath for 30 minutes to loosen the melt from the crucible. Remove crucible and lid from beaker. Filter the solution into a 1 litre volumetric flask ensuring that deionised water washings from the beaker are also filtered.

Dilute to 300 ml and adjust the pH to 75 with 1:3 HCl. Dilute to the mark with deionised water

Method:

1. Set the flame photometer up as in its instruction manual.
2. Set the zero using the blank solution.
3. Aspirate the standards into the instrument recording each displayed value.

4. Construct a calibration plot of concentration vs. meter reading on linear graph paper.

5. Aspirate the sample solution and record the stable display. (If the value is higher than that of the top standard, dilute sample with blank until the value falls in the range of the calibration curve.)

6. Multiply the sodium concentration of the sample by the dilution factor

Sodium in Straw by Flame Photometry

Sodium hydroxide is added to cellulose animal feeds to improve digestibility. The sodium concentration is determined using a flame photometer.

Equipment Required:

1. Single channel industrial flame photometer
2. Balance weighing to ±0.0005g
3. Filtration apparatus (or centrifuge)
4. Volumetric glassware
5. 1000 ppm sodium flame photometer standard
6. 1 Molar ammonium chloride/l Molar NH_4OH solution
7. Deionised water

Reagent Preparation:

Extractant solution: Dissolve 53.49g $NH_4Cl$with deionised water in a 1 litre volumetric flask. In a fume cupboard add to this solution 55 mls of ammonia (SG= 0.88). Dilute to the mark with deionised water.

Standard Preparation:

Dilute the 1000 ppm Na standard 1:100 with deionised water giving a 10 ppm top standard. From this prepare a 6, 4 and 2 ppm standard, by dilution with deionised water.

Sample Preparation:

1. Weigh accurately 3g straw and place in a 100 ml volumetric flask.
2. Add 80 mls of extractant solution and shake vigorously.
3. Stand for at least 30 minutes intermittently shaking the solution.
4. Filter or centrifuge to remove solid matter (Note: the solid must be washed with extractant solution and washings collected with supernatant). In a 100 ml volumetric flask dilute the washings and supernatant to the mark with deionised water
5. Dilute this solution 1 in 100 with deionised water. The sample is ready for analysis.

Method:

1. Set up the flame photometer as outlined in its instruction manual.
2. Aspirate the standards into the flame photometer noting their readings.
3. Plot meter reading vs. Na concentration.
4. Aspirate the sample and note the meter reading.
5. From the graph interpolate the sample concentration.

Calculation:

To obtain the result in % Na in the original straw sample divide the sample concentration in ppm by 3.

NOTE: If a 410 flame photometer is used and the 10 ppm standard set to 10.0, the calibration scale will be linear, i.e. 6 ppm standard will read 6.0. Therefore, the ppm Na in the sample can be read directly from the display. If calibration is non linear then to obtain direct readout dilute all standards and samples 1 in 2 with deionised water and multiply the reading by 2.

The Determination of Sodium and Potassium in Fruit Juice

This method involves the dilution of the fruit juice sample with water followed by filtration and finally aspiration directly into a flame photometer.

Equipment Required:

1. Single channel flame photometer (industrial)
2. Filtration apparatus
3. Volumetric glassware
4. 1000 ppm sodium and potassium standards
5. Deionised water

Method:

1. Prepare by dilution of the stock standards 20, 15, 10 and 5 ppm Na and K standards. Deionised water is the blank solution.
2. To 10 mls of the fruit juice add 50 mls deionised water.
3. Filter this solution through an ashless filter paper (e.g. Whatman 540) into a litre volumetric flask. Ensure that the solid particles retained by the paper are washed thoroughly and washings directed into the same 1 litre flask. Dilute to the mark with deionised water, stopper the flask and mix by inversion.
4. Set up the flame photometer as outlined in its instruction manual for sodium.
5. Set blank to zero, i.e. deionised water.
6. Aspirate the standards and record their stable readings.
7. Plot a graph of meter reading against standard concentration.
8. Aspirate the sample solution into the flame photometer
9. Record the meter reading and from the graph read off its sodium concentration.
10. Adjust the filter position to select the potassium filter and repeat stages 5 to 9 for potassium.

Note that if the Na and K concentrations in the fruit juice are outside the range of standards the sample should be diluted accordingly.

Calculation:

Multiply the concentration of Na and K obtained from the graph by the dilution factor, i.e. x 100, to express the result in ppm or mg/l of Na or K in the original fruit juice.

The Determination of Calcium in Biscuits

Calcium is extracted with lithium acetate solution and analysed directly by flame photometry.

Equipment Required:

1. Single channel flame photometer
2. Calcium interference filter
3. Accurate balance
4. Volumetric glassware
5. Filtration apparatus
6. 1000 ppm Ca2+ solution
7. Extractant solution
8. Deionised water

Extractant Solution:

Prepare 0.8 Molar lithium chloride -0.2 Molar lithium acetate solution in deionised water. This solution diluted 1 in 2 with deionised water is the blank solution.

Sample Preparation:

To 5g of dried, accurately weighed, sample in a sealable glass container add 250 ml of extractant solution. Seal and shake vigorously for 15 minutes (intermittently), stand the solution for 2 hours (or overnight if convenient).

Filter the solution through a Whatman No. 6 filter paper into a 500 ml volumetric flask and dilute to the mark with deionised water.

Standard Preparation:

From the 1000 ppm Ca^{2+} standard prepare a 100 ppm Ca^{2+} by adding 10 ml of the stock standard to 50 ml of extractant solution in a 100 ml volumetric flask and diluting to the mark with deionised water. Prepare also a 75, 50 and 25 ppm Ca^{2+} solution ensuring each standard contains a 1 in 2 dilution of the extractant solution.

Method:

1. Set up the flame photometer as outlined in its instruction manual.
2. Select the calcium filter position.
3. Aspirate the blank solution (1 in 2 dilution of extractant solution) and set to zero.
4. Aspirate the calcium standards and record their stable readings.
5. Plot on linear graph paper meter readings against calcium concentration.
6. Aspirate the sample and note its reading. The calcium concentration can now be obtained from the graph. Multiply by dilution factor
7. Repeat with further samples.

N.B. If the calcium concentration of the sample is outside the standard range then dilute with the blank solution.

The method of extraction is suitable for most samples, however if a more vigorous technique is required operate the extraction using an HCl/CH_3COOH solution followed by autoclaving for 20 minutes at 20 psi and adjusting the pH to 4.5 with NH_3 solution.

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Standard concentration conversion tables

$$\text{ppm}=\text{A X mMoldm}^{-3} \quad \text{and} \quad \text{mMoldm}^{-3}=\frac{\text{ppm}}{\text{A}}$$

where A=atomic mass of the atom being analysed.

	Atomic mass /amu	1000ppm Standard	140mmol/L Standard
Sodium	22.99	43.7mmol/L Na	3206ppm Na (3206mg Na /L)

	Atomic mass /amu	1000ppm Standard	5.0mmol/L Standard
Potassium	39.102	25.6mmol/L K	196ppm K (196mg K /L)

	Atomic mass /amu	1000ppm Standard	1.5mmol/L Standard	3M Standard
Lithium	6.939	144mmol/L Li	10ppm Li (10mg Li /L)	20817ppm Li (20.817g Li /L)

	Atomic mass /amu	1000ppm Standard
Calcium	40.08	25mmol/L Ca