10.3 Determination of calcium, copper, iron, magnesium, manganese, potassium, sodium and zinc – using Atomic Absorption Spectrometry

Reagents and materials

Use only reagents of recognized analytical grade.

- Water, complying with at least grade 3 in accordance with ISO 3696.
- Concentrated hydrochloric acid. c(HCl) = 12 mol--- (p = 1.19 g/ml).
- Hydrochloric acid, c(HCl) = 6 mol/l.
- Dilute hydrochloric acid. c(HCl) = 0.6 mol/l.

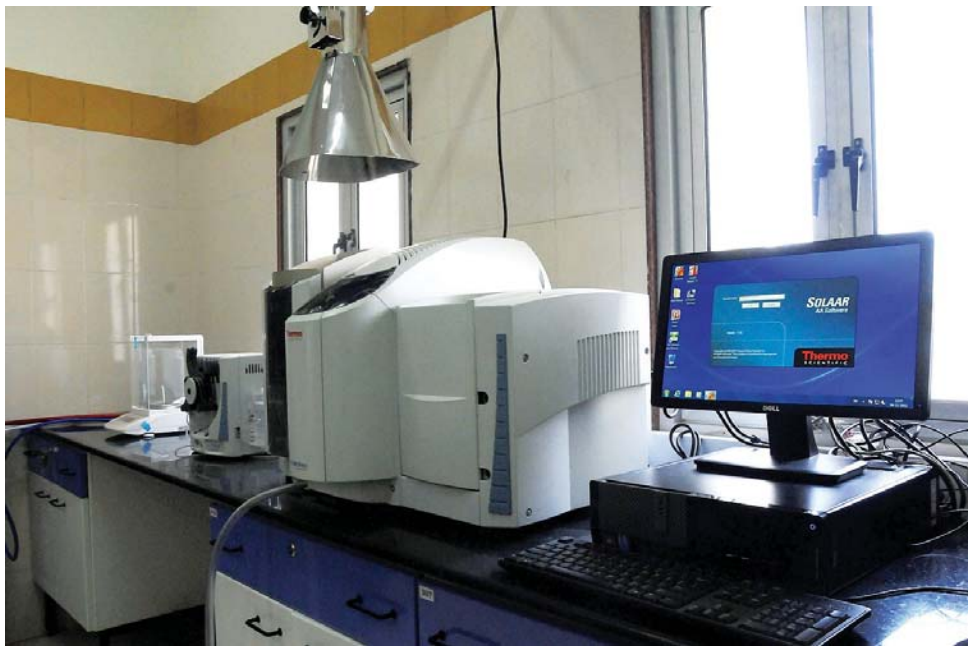

Fig. 10.3 Atomic Absorption Spectrophotometer (AAS)

- Lanthanum nitrate solution - Dissolve 133 g of $La(NO_3)_3.6H_2O$ in 1 litre of water.
- Caesium chloride solution - Dissolve 100 g of CsCl in 1 litre of water.

Stock solution of Cu, Fe, Mn and Zn

Mix 100 ml of water and 125 ml of concentrated hydrochloric acid in a 1 litre volumetric flask.

Weigh out the following:

- 392.9 mg of copper (II) sulfate pentahydrate ($CuSO_4.5H_2O$)
- 702.2 mg of ammonium iron (II) sulfate hexahydrate [$(NH_4)_2SO_4FeSO_4.6H_2O$]
- 307.7 mg of manganese sulfate monohydrate ($MnSO_4.H_2O$)
- 439.8 mg of zinc sulfate heptahydrate ($ZnSO_4.7H_2O$)

Transfer the weighed salts to the volumetric flask and dissolve them in water. Dilute to the mark with water.

The contents of Cu, Fe, Mn and Zn of this stock solution each are 100 µg/ml.

Note : Ready-prepared commercially available solutions may be used.

Standard solution of Cu, Fe, Mn and Zn

Dilute 20.0 ml of the stock solution with water to 100 ml in a volumetric flask. The contents of Cu, Fe, Mn and Zn of this solution are each 20 µg/ml. Prepare the solution fresh on the day of use.

Stock solution of Ca, K, Mg and Na

Weigh out the following:

- 1.907 g of potassium chloride (KCl)
- 2.028 g of magnesium sulfate heptahydrate ($MgSO_4 7H_2O$)
- 2.542 g of sodium chloride (NaCl).

Transfer the weighed salts to a 1 litre volumetric flask.

Add 50 ml of hydrochloric acid (6 mol/l) to a beaker. Weigh into the beaker 2.497 g of calcium carbonate ($CaCO_3$).

Standard solution of Ca, K, Mg and Na

Dilute 25.0 ml of stock solution with dilute hydrochloric acid (0.6 mol/l) to 250 ml in a volumetric flask.

The contents of Ca, K and Na of this solution are 100 µg/ml each; the content of Mg of the solution is 20 µg/ml.

Prepare the solution fresh in the week of use and store it in a polyethylene bottle.

Lanthanum / caesium blank solution

Add 5 ml of lanthanum nitrate solution, 5 ml of caesium chloride solution and 5 ml of hydrochloric acid (6 mol/l) to a 100 ml volumetric flask. Dilute to the mark with water.

Apparatus

- Analytical balance, capable of weighing to the nearest 0.1 mg.
- Incineration dishes of platinum, quartz or porcelain, free from potassium and sodium with a smooth un-detached inner surface, upper internal diameter 4 to 6 cm, lower internal diameter 2 to 2.5 cm, and a height of about 5 cm. Before use, boil with hydrochloric acid.
- Glassware of hard borosilicate glass.
- Electric hot plate or gas burner.
- Boiling water bath.
- Electric muffle furnace, capable of being maintained at (550 ± 15ºC)
- Atomic absorption spectrometer (Fig. 10.3). Suitable for measuring at the wavelengths specified and provided with an air-acetylene flame and a facility for correction, or measurement of background absorption.
- Hollow cathode lamps or electrode-less discharge lamps for the determination of Ca, Cu, Fe, K, Mg, Mn, Na or Zn.
- Filter paper which does not release minerals.

Procedure

Detection of presence of organic matter

Heat a spatula with some test sample in a flame.

If the test sample melts without smoke, little organic matter is present.

If the test sample changes in color and melting does not occur, the test sample contains organic matter.

Test portion

Depending on the expected content, weigh 1 to 5 g of the prepared test sample to the nearest 1 mg into an incineration dish.

If the test sample contains organic matter, proceed for dry ashing.

If the test sample contains little or no organic matter, proceed for decomposition.

Dry ashing

Heat the incineration dish on a hot plate or over a gas burner until the test portion has been completely carbonized in fume hood chamber (Fig. 10.4). Avoid burning the test portion.

Transfer the dish to the muffle furnace, which has already been at a temperature of 550ºC for 15 min. Ash the sample for 3 h at this temperature.

Allow the sample to cool down then moisten the contents of the dish with 2 ml of water. If many carbon particles are present, dry the dish over the water bath.

Ash for another 2 h in the muffle furnace set at 550ºC.

Allow to cool down then add 2 ml of water.

Decomposition

While swirling, add 10 ml of hydrochloric acid (6 mol/l), first drop wise until effervescence (possible development of carbon dioxide) has ceased, then faster. Swirl and heat the contents of the dish until almost dry. While drying, take care to avoid loss by splattering.

Dissolve the residue by heating with 5 ml of hydrochloric acid (6 mol/l) and transfer the solution quantitatively with some 5 ml portions of water to a 50 ml volumetric flask.

Allow to cool, then dilute to the mark with water and mix. Allow the particles to settle and filter the solution if it is not clear after 4 h.

Fig. 10.4 Fume hood chamber

Blank solution

Prepare for each measuring series a blank solution by carrying out the procedure according to test portion, dry ashing and decomposition without the test sample.

Determination of copper, iron, manganese and zinc

Measuring conditions

Adjust the atomic absorption spectrometer in accordance with the manufacturer's instructions. Optimize the response of the instrument for measurement with the air-acetylene flame. For the determination of Cu, Fe, Mn and Zn set the following wavelengths:

Cu: 324.8 nm;
Fe: 248.3 nm;
Cu: 279.5 nm;
Cu: 213.8 nm

Preparation of calibration curve

Prepare a series of appropriate calibration solutions by diluting the standard solution with dilute hydrochloric acid (0.6 mol/l).

Measure the absorbance of the hydrochloric acid (0.6 mol/l). Measure the absorbance of the calibration solutions and subtract the absorbance measured for hydrochloric acid (0.6 mol/l).

Draw a calibration curve by plotting the corrected absorbance against the respective contents of Cu, Fe, Mn and Zn.

Measurement of test solution

Measure parallel to the calibration solutions, under identical circumstances, the absorbance of the test solution and the blank solution. Subtract the latter absorbance from the first absorbance.

If necessary, dilute a quantity of the test solution and blank solution with dilute hydrochloric acid (0.6 mol/l) to obtain an absorbance in the linear part of the calibration curve.

Proceed in accordance with expression of results

Determination of calcium, magnesium, potassium and sodium

Measuring conditions

Adjust the atomic absorption spectrometer in accordance with the manufacturer's instructions. Optimize the response of the instrument for measurement with the air-acetylene flame. For the determination of Ca, K, Mg and Na set the following wavelengths:

Ca : 422.6 nm;
K : 766.5 nm;
Mg : 285.2 nm;
Na : 589.6 nm

Preparation of calibration curve

Dilute the standard solution with water. Add per 100 ml of diluted standard solution 5 ml of lanthanum nitrate solution, 5 ml of caesium chloride solution and 5 ml of hydrochloric acid (6 mol/l). Choose the dilutions so that appropriate calibration solutions are obtained.

Measure the absorbance of the lanthanum / caesium blank solution.

Measure the absorbance of the calibration solutions and subtract the absorbance measured for the lanthanum / caesium blank solution.

Draw a calibration curve by plotting the corrected absorbance against the respective contents of Ca, K, Mg and Na.

Measurement of test solution

Dilute a quantity of the test solution and blank solution with water. Add per 100 ml of diluted solution 5 ml of lanthanum nitrate solution, 5 ml of caesium chloride solution and 5 ml of hydrochloric acid (6 mol/l).

Measure parallel to the calibration solutions, under identical circumstances, the absorbance of the diluted test solution and the diluted blank solution. Subtract the latter absorbance from the first absorbance.

If necessary, dilute a quantity of the test solution and blank solution with lanthanum / caesium blank solution to obtain an absorbance in the linear part of the calibration curve.

Expression of results

Calculate the content of each of the elements calcium, copper, iron, magnesium, manganese, potassium, sodium and zinc starting from the calibration curve and taking into account the mass of the test portion and the dilutions applied.

Express the result in milligrams per kilogram or grams per kilogram.

Reference: IS: 15121: 2002, ISO 6869: 2000.