Phosphorous, calcium, magnesium and aluminium determination by Inductively Coupled Plasma-Atomic Emission Spectrophotometry (ICP-AES) [1]

Accurately weigh about 0.500 g of finely ground sample into a 100-ml PTFE beaker. Add about 10 ml of deionized water, 5 ml aquaregia (HNO_3: HCl, 3:1) and leave it in a fume hood for about 15 min. Place the beaker, if required, on a hot plate maintained at low heat for about 30 min. Control the temperature in a way that the sample do not over boil or get dried up. Ensure that the solution does not spurt. Quantitatively transfer into a 100-ml volumetric flask and make up to volume with deionized water. Dilute sample solution to bring the solution within the working range of the standard curve. Prepare a reagent blank omitting sample and dilute the blank as required.

Note: *The selection of sample size and method of sample preparation may be based on principles of methods described in the Combined Compendium of Food Additives Specifications, Volume 4. Sample weight may vary according to the concentration of element under the assay. Purity of acids and other reagents used shall be of atomic spectroscopy grade.*

Determine the elements phosphorous, magnesium, calcium and aluminium using ICP-AES technique appropriate to the specified level. Set instrument parameters as specified by the instrument manufacturer. Use analytical line for P (213.618 nm), Mg (279.078 nm), Ca (318.127), Al (396.152 nm) and deduce the concentration of element (µg/ml) in the sample. Calculate percentage as follows:

$$\% \text{ element} = (A-B) \times V \times DF / 10000 \times W$$

Where:

A is the concentration of element in the sample solution, µg/ml
B is the concentration of element in the corresponding reagent blank solution, µg/ml
V is the volume of sample made up, ml
DF is the dilution factor
W is the weight of sample, g

Convert the mineral to the corresponding oxides using the formulae:

$\%w/w\ P_2O_5 = \%\ P \times 4.583$
$\%w/w\ MgO = \%\ Mg \times 1.658$
$\%w/w\ CaO = \%\ Ca \times 1.399$
$\%w/w\ Al_2O_3 = \%\ Al \times 3.779$

[1] This method is added to the section *Inorganic Components* of Volume 4 of the Combined Compendium of Food Additive Specifications (on page 74).