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Determination of phosphorus content in detergents - Inductively coupled plasma mass spectrometry

洗涤剂中磷含量的测定 电感耦合等离子体质谱法

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1 Scope

This document describes the method for the determination of the phosphorus content of detergents by inductively coupled plasma mass spectrometry.

This document applies to the determination of total phosphorus in phosphate-free and low-phosphate detergents.

4 Principle of the method

The test sample is digested and then measured by inductively coupled plasma mass spectrometry; identification is by the mass-to-charge ratio of the specific mass number of the element, and quantification relies on the fact that the ratio of the mass spectrometric signal of the element to be determined to the mass spectrometric signal of the internal standard element is proportional to the concentration of the element to be determined.

5 Reagents and materials

Unless otherwise stated, only reagents of guaranteed grade are used.

5.1 Water, grade 1 of GB/T 6682.

5.2 Dilute nitric acid solution, 5 %; take 50 mL of nitric acid and dilute with water to 1 L.

5.3 Argon, high-purity argon of purity greater than 99.999 %.

5.4 Helium, high-purity helium of purity greater than 99.999 %.

5.5 Reference material of the target element phosphorus, 1 000 micrograms per millilitre, a certified reference material or reference sample being used.

5.6 Reference material of the internal standard element scandium, 1 000 micrograms per millilitre, a certified reference material or reference sample being used.

5.7 Phosphorus standard working solutions. Take a suitable quantity of the phosphorus stock solution of 1 000 micrograms per millilitre and dilute it stepwise with the 5 % dilute nitric acid solution (5.2) to make up a series of standard solutions. The concentration range of the phosphorus standard series may also be adjusted to suit the mass concentration of phosphorus in the sample solution. The mass concentrations of the phosphorus standard series working solutions are given in Table A.1 of Annex A.

5.8 Scandium internal standard solution. Take a suitable quantity of the internal standard scandium stock solution of 1 000 micrograms per millilitre, mix, and prepare an internal standard working solution of suitable concentration with the 5 % dilute nitric acid solution (5.2). The mass concentration of the scandium internal standard working solution is given in Table A.2.

6 Apparatus

6.1 Inductively coupled plasma mass spectrometer (ICP-MS).

6.2 Electronic balance, with a scale division of 0.1 mg.

6.3 Microwave digestion system, fitted with polytetrafluoroethylene digestion vessels of 50 mL.

6.4 Pressure digestion system, fitted with polytetrafluoroethylene digestion vessels of 50 mL.

6.5 Hotplate, from room temperature to 200 degrees Celsius.

6.6 Ultrasonic water bath.

7 Analytical procedure

7.1 Preparation of the test sample. Powder samples are stirred until uniform and set aside; liquid samples are shaken until uniform and set aside.

7.2.1 Microwave digestion. Weigh 0.2 g to 0.5 g of the sample, to the nearest 0.001 g, into the digestion vessel, add 5 mL of nitric acid, cover and leave to stand for 1 h, then tighten the vessel cap and digest following the operating procedure of the microwave digestion system; the reference conditions of the digestion system are given in Table B.1 of Annex B. After cooling, remove the vessel and open the cap slowly to vent it, rinse the inner cover with a little water, place the digestion vessel in an ultrasonic water bath or on a hotplate and drive off the brown fumes until colourless; then remove the vessel, transfer the digest to a 50 mL volumetric flask, make up to the mark with water and mix; carry out a blank test at the same time.

7.2.2 Pressure digestion. Weigh 0.2 g to 0.5 g of the sample, to the nearest 0.001 g, into the digestion vessel, add 5 mL of nitric acid, cover and leave to stand for 1 h, then tighten the stainless steel jacket and digest following the operating procedure of the pressure digestion system; the reference conditions are given in Table B.1. Allow to cool naturally to room temperature, then slowly slacken the stainless steel jacket, remove the digestion vessel, rinse the inner cover with a little water, place the vessel in an ultrasonic water bath or on a hotplate and drive off the brown fumes until colourless. Transfer the digest to a 50 mL volumetric flask, make up to the mark with water and mix; carry out a blank test at the same time.

7.3 Instrument working programme. Optimize the instrument operating conditions, the reference operating conditions being given in Table B.2, so that sensitivity and the formation of oxides and doubly charged species meet the requirements of the determination. Once the instrument tuning meets those requirements, write the determination method and select the phosphorus isotope and the scandium internal standard element. Interference correction equations may be used to correct the results; instrument software generally includes such equations.

7.4 Calibration curve. Inject the phosphorus standard series working solutions together with the scandium internal standard working solution into the inductively coupled plasma mass spectrometer and measure the signal responses of the target element phosphorus and of the internal standard scandium; plot the calibration curve with the concentration of phosphorus as abscissa and the ratio of the phosphorus signal response to the scandium signal response as ordinate.

7.5 Determination of the solutions. Under the same working conditions of the instrument, mix the blank solution and the sample solution to be tested separately with the scandium internal standard solution and inject them into the inductively coupled plasma mass spectrometer, obtaining for each the ratio of the signal response of phosphorus to that of the internal standard scandium; calculate the concentration of phosphorus in the sample solution from the calibration curve. If the phosphorus content of the sample solution is too

high, dilute it and determine it again.

7.6 Expression of the result. The mass fraction of phosphorus in the test sample, expressed as diphosphorus pentoxide, is calculated by Formula (1), in which the mass fraction is given as a percentage; the mass concentration of phosphorus in the sample solution to be tested and in the blank solution is in micrograms per litre; V is the volume to which the sample solution is made up, in millilitres; 2.291 3 is the factor converting the mass of phosphorus to the mass of diphosphorus pentoxide; f is the dilution factor of the test sample; 0.000 1 is the factor converting milligrams per kilogram to a percentage; m is the mass of test sample weighed out, in grams; and 1 000 is the factor converting millilitres to litres. The arithmetic mean of two parallel determinations is taken as the result, expressed to two significant figures.

8 Precision

The absolute difference between two independent results obtained under repeatability conditions shall not be greater than 15 % of their arithmetic mean.

9 Detection limit and quantification limit of phosphorus, expressed as diphosphorus pentoxide

The detection limit and the quantification limit of phosphorus, expressed as diphosphorus pentoxide, are given in Table 1: a detection limit of 0.000 02 % and a quantification limit of 0.000 06 %. Note: these are calculated for a sample weight of 0.2 g and a made-up volume of 50 mL.