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Inorganic chemicals for industrial use - Determination of impurity elements - Inductively coupled plasma optical emission spectrometry (ICP-OES)

无机化工产品 杂质元素的测定 电感耦合等离子体发射光谱法(ICP-OES)

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Warning

Some of the reagents used in this test method are toxic or corrosive and shall be handled with care. Any splash on the skin shall be rinsed at once with water, and severe cases shall receive immediate medical treatment. The method uses high-pressure argon cylinders, which shall be handled according to the safety rules for high-pressure cylinders. Once the plasma is lit, the torch compartment door shall not be opened, so as to avoid injury from high-frequency radiation. Care shall be taken with the electrical supply.

1 Scope

This standard specifies the principle, the reagents, the instruments and equipment, the analytical procedure, the precision and the recovery for determining metallic and non-metallic impurity elements in inorganic chemicals for industrial use by inductively coupled plasma optical emission spectrometry (ICP-OES).

It applies to the determination, by inductively coupled plasma optical emission spectrometer with direct injection, of liquid samples of inorganic chemicals for industrial use containing several impurities, or of test solutions from which the matrix has been removed.

2 Normative references

The following documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition, including all amendments, applies.

GB/T 4470 Terms for flame emission, atomic absorption and atomic fluorescence spectrometric analysis · GB/T 4842 Argon · GB/T 6379.2 Accuracy (trueness and precision) of measurement methods and results - Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method · GB/T 6682-2008 Water for analytical laboratory use - Specification and test methods · HG/T 3696.2 Inorganic chemicals for industrial use - Preparation of standard and reagent solutions for chemical analysis - Part 2: Preparation of standard solutions for impurity determination

3 Terms and definitions

The terms and definitions given in GB/T 4470, together with the following, apply to this document.

plasma: ionised gas with a degree of ionisation greater than 0.1 % in which the positive and negative charges are equal.

high frequency generator: high-frequency power source that supplies high-frequency energy to the coupling coil and to the plasma.

plasma torch: device that sustains a stable discharge in the ICP, normally made of three concentric quartz tubes, the outer one carrying the coolant gas, the middle one the auxiliary gas and the inner one the carrier gas.

incident power: net power delivered by the high frequency generator to the coupling coil and the plasma.

observation height: height of the exposed part of the plasma torch, that is the distance from the centre of the observation zone to the top of the coupling coil.

coolant gas: gas flowing between the outermost and the middle tube of the torch, which cools the torch and sustains the plasma.

auxiliary gas: gas flowing between the middle and the central tube of the torch, which lights the plasma and keeps the hot base of the ICP at a distance from the central and middle tubes, protecting their tops - and above all the mouth of the central tube - from melting or overheating, and reducing the build-up of salts carried by the aerosol at that mouth. It also raises the ICP and so changes the observation position.

carrier gas: gas flowing in the central tube of the torch, which nebulises the liquid into an aerosol and carries that aerosol into the plasma.

washing time: time for which the sample introduction system is rinsed with the sample solution before exposure.

4 Principle

The liquid sample is carried by the carrier gas into the nebulising system and, as an aerosol, enters the axial channel of the plasma, where the high temperature and the inert gas evaporate, atomise, ionise and excite it completely. The characteristic spectral lines emitted by the elements present pass through the dispersing system to the spectral detector, which performs the qualitative, semi-quantitative and quantitative analysis from those characteristic spectra.

5 Reagents

Unless otherwise stated, the reagents used are of analytical grade or better. Sample treatment commonly uses inorganic acids such as hydrochloric, nitric, perchloric and hydrofluoric acid; these shall be checked before use and shall so far as possible be free of the metallic elements to be determined.

The laboratory water shall meet at least the grade two specification of GB/T 6682-2008.

Stock standard solutions: the stock standard solution of each element to be analysed is prepared according to HG/T 3696.2; alternatively a mixed solution or a single-element solution of certified national reference materials of the corresponding concentration may be used and diluted to the concentration required. The preparation of multi-element standard solutions is described in Annex A.

6 Instruments and equipment

Inductively coupled plasma atomic emission spectrometer, comprising the sample introduction system, the excitation source, the optical system, the detection system and the data processing system.

7 Analytical procedure

Choice of the measuring conditions. The wavelengths of the analytical lines of the elements to be determined are given in Annex B. The incident power is chosen for the characteristics of the sample and the conditions of the instrument, normally between 0.8 kW and 1.6 kW. The observation height, measured from the top of the induction coil to the measuring axis, is normally 14 mm to 18 mm; for a single element the optimum height for that element is chosen, and for several elements an intermediate height. The solution uptake rate is normally 0.6 mL/min to 2 mL/min. The optimum flow of each gas is set according to the torch and the requirements of the analysis, and the argon used shall meet GB/T 4842. The washing time and the exposure time are determined by the instrument and by the

requirements of the analysis.

Elimination of interferences. A suitable separation method and a suitable choice of spectral line, observation height, incident power and carrier gas flow keep some of the interference effects in the source within a defined level. Spectral interferences may be corrected by methods based on interference coefficients, by separating the matrix, or with the software supplied by the instrument maker; non-spectral interferences shall be corrected by matrix matching, by the standard addition method or similar. The medium and the acidity of the sample solution and of the standard solution shall be kept as close as possible, so that the uptake rate and the nebulisation efficiency of the instrument stay stable and the physical interference from the sample is removed.

Laboratory equipment. General cleanliness of the laboratory matters for micro and trace analysis. Vessels used to digest solids are normally of polytetrafluoroethylene (PTFE) or of fluoroplastic (PFA, tetrafluoroethylene with perfluorinated alkyl side chains). Before use, vessels are normally soaked for several hours in 20 % to 30 % nitric acid solution, rinsed clean with water and dried in a thermostatic oven.

Sample pretreatment methods. Open-vessel digestion, in which sample and reagents are heated in an open vessel over a flame, on a hotplate or in a furnace, is the most widely used; it makes modest demands on equipment and allows many samples to be digested at once, but is slow, more prone to contamination and less accurate and precise.
Closed-vessel digestion carries out the wet digestion with acid or other reagents in a sealed vessel, generally of PTFE, under heat and pressure. Microwave digestion works in a sealed pressurised vessel under microwaves, normally at 2 450 MHz: the mixture of sample and acid absorbs the microwave energy, the boiling point of the inorganic acid rises, its oxidising reactivity increases, and the surface layer of the sample is stirred and broken open so that fresh surface keeps meeting the acid until digestion is complete, after which the excess acid is driven off. Alkali metal fusion mixes the sample with any of various alkali metal fluxes - lithium metaborate, lithium tetraborate, sodium carbonate, sodium hydroxide, sodium peroxide, the corresponding potassium salts and alkali metal fluorides, especially potassium hydrogen fluoride - and fuses it at high temperature. Separation and preconcentration removes possible matrix effects and interferences while concentrating the analyte and so lowering the limit of quantification; the main methods are solvent extraction, ion exchange and coprecipitation or adsorption.

Requirements for the liquid solution. After treatment the sample is made up to a suitable volume according to the content of the elements to be determined, giving the sample solution. The amount taken depends on the mass concentration of those elements in the sample and on the detection limit of the method; the mass concentration of the element in the sample solution shall be at least three times its detection limit. The detection limit is determined by the method of Annex C.

Qualitative analysis. The presence of an element is judged from three or more of its