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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 29875-2013

Determination of lead, arsenic, mercury in phosphate rock and concentrate

磷矿石和磷精矿中铅、砷、汞含量的测定

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Table of Contents

1 Scope

This standard specifies methods for determining the lead, arsenic and mercury contents of phosphate rock and phosphate concentrate.

It applies to the determination of lead contents above 0.001 0 %, arsenic contents above 0.000 2 % and mercury contents above 0.000 01 % in phosphate rock and phosphate concentrate.

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.

The documents listed are GB/T 602 (chemical reagents - preparation of standard solutions for impurity determination), GB/T 603 (chemical reagents - preparation of preparations and products used in test methods), GB/T 6003.1 (test sieves - technical requirements and testing - Part 1: test sieves of metal wire cloth), GB/T 6682 (water for analytical laboratory use - specification and test methods) and GB/T 9723 (chemical reagents - general rules for flame atomic absorption spectrometry).

3 Test sample

The test sample passes a 125 μm test sieve complying with GB/T 6003.1, is dried at 105 °C to 110 °C for 2 h or more, and is then cooled to room temperature in a desiccator.

4.1 General

Unless otherwise stated, only reagents recognised as analytical grade or better are used in the analysis, and the water used meets the requirements of grade two water in GB/T 6682.

Where no preparation method is given for a reagent or solution used in the standard, it is prepared according to GB/T 602 and GB/T 603.

4.2 Determination of lead content - atomic absorption spectrometry

Summary of the method: the test sample is dissolved with hydrochloric acid and nitric acid; in dilute nitric acid medium and with an acetylene-air flame, the absorbance of the sample solution is measured with an atomic absorption spectrometer at a wavelength of 283.3 nm, background interference is subtracted at the same time, and the lead content of the phosphate sample is obtained by the working curve method.

Reagents and solutions: hydrochloric acid (guaranteed reagent); nitric acid (guaranteed reagent); nitric acid solution 1+1; lead standard solution 1 000 μg/mL; lead standard solution 100 μg/mL, prepared by transferring 10 mL of the 1 000 μg/mL solution to a 100 mL volumetric flask, adding 10 mL of the nitric acid solution and diluting to the mark with water, so that 1 mL of the solution contains 100 μg of lead.

Apparatus: an atomic absorption spectrometer fitted with a lead hollow cathode lamp and complying with GB/T 9723.

Analytical procedure: 1 g of the test sample is weighed to the nearest 0.000 1 g into a 150 mL beaker and a blank test is run at the same time. The sample is wetted with a little water, 15 mL of hydrochloric acid and 5 mL of nitric acid are added carefully, the beaker is covered with a watch glass and heated gently on a hot plate, evaporated almost to dryness, then removed and cooled. Ten millilitres of the nitric acid solution and a little water are added, the

soluble salts are dissolved by heating, the solution is cooled to room temperature and transferred to a 100 mL volumetric flask, diluted to the mark with water and mixed; it is then filtered dry, the first small portion of filtrate is discarded and the rest is collected for use. With reference to the instrument manual the working parameters are optimised, an acetylene-air flame and the lead hollow cathode lamp are used, and at 283.3 nm, with water as the zero, the absorbances of the sample solution and of the blank solution are measured in background correction mode; the corresponding lead mass concentration is read from the working curve.

Plotting of the working curve: 0.0 mL, 1.0 mL, 3.0 mL, 5.0 mL, 7.0 mL and 10.0 mL of the 100 μg/mL lead standard solution are transferred to a set of 100 mL volumetric flasks, 10 mL of the nitric acid solution is added to each, and each is diluted to the mark with water and mixed; the lead concentrations of this series are 0.0 μg/mL, 1.0 μg/mL, 3.0 μg/mL, 5.0 μg/mL, 7.0 μg/mL and 10.0 μg/mL. Their absorbances are measured in background correction mode under the same conditions as the sample solution, the reagent blank absorbance is subtracted, and the curve is plotted with lead mass concentration on the abscissa and absorbance on the ordinate.

Calculation of the result: the lead content is expressed as the mass fraction and is obtained from the lead mass concentration read off the working curve, in micrograms per millilitre, and the mass of the test portion, in grams. The arithmetic mean of parallel determinations is taken as the final result.

Permissible difference: the relative deviation of parallel analytical results is not greater than the permissible values of Table 1, which gives 30 % for a lead content below 0.002 0 %, 20 % for a lead content from 0.002 0 % to 0.010 % and 10 % for a lead content above 0.010 %.

4.3.1 Determination of arsenic content - atomic fluorescence spectrometry (referee method)

Summary of the method: the test sample is dissolved with hydrochloric acid and nitric acid; under acidic conditions trivalent arsenic reacts with potassium borohydride to form arsine, which is carried by the carrier gas (argon) into a quartz atomiser where it decomposes to atomic arsenic. Under the irradiation of a special arsenic hollow cathode lamp the ground state arsenic atoms are excited to a high energy state and, on returning to the ground state, emit fluorescence of characteristic wavelength; within a certain concentration range the fluorescence intensity is proportional to the arsenic content, and the arsenic content of the phosphate sample is obtained by the working curve method.

Reagents and solutions: hydrochloric acid and nitric acid (guaranteed reagent); hydrochloric acid solutions 1+1 and 5+95; potassium hydroxide solution 5 g/L; potassium borohydride solution 10 g/L, prepared by dissolving 1 g of potassium borohydride in 100 mL of the

potassium hydroxide solution and used freshly prepared; ascorbic acid solution 50 g/L, freshly prepared; thiourea solution 50 g/L; arsenic standard solution 1 000 μg/mL, from which are prepared in turn a 50 μg/mL solution (10.0 mL diluted to 200 mL), a 1 μg/mL solution (10.0 mL of the 50 μg/mL solution diluted to 500 mL) and a 100 ng/mL solution (10.0 mL of the 1 μg/mL solution diluted to 100 mL).

Apparatus: an atomic fluorescence spectrometer fitted with an arsenic hollow cathode lamp.

Analytical procedure: the test portion is weighed according to Table 2 to the nearest 0.000 1 g into a 150 mL beaker and a blank test is run at the same time. The sample is wetted with a little water, 15 mL of hydrochloric acid and 5 mL of nitric acid are added carefully, the beaker is covered with a watch glass, heated gently on a hot plate and evaporated to 1 mL to 2 mL, then removed and cooled to room temperature; a note warns that the sample solution is not to be evaporated to dryness, since the result would then be low. Ten millilitres of the 1+1 hydrochloric acid solution and a little water are added, the soluble salts are dissolved by heating, the solution is cooled to room temperature and transferred to a 100 mL volumetric flask, 10 mL of the ascorbic acid solution and 10 mL of the thiourea solution are added, the flask is diluted to the mark with water, mixed and left to stand for 30 min; the solution is then filtered dry, the first small portion of filtrate is discarded and the rest is collected. With the instrument parameters optimised, the 5+95 hydrochloric acid solution is used as the carrier and the potassium borohydride solution as the reductant, and the fluorescence values of the sample solution and of the blank solution are measured in turn.

Table 2 grades the test portion mass by arsenic content: 0.2 g to 0.5 g for an arsenic content of 0.000 2 % to 0.001 0 %, 0.1 g to 0.2 g for 0.001 0 % to 0.002 0 %, and 0.05 g to 0.1 g above 0.002 0 %.

Plotting of the working curve: 0.0 mL, 1.0 mL, 2.0 mL, 5.0 mL, 10.0 mL and 20.0 mL of the 100 ng/mL arsenic standard solution are transferred to a set of 100 mL volumetric flasks, 10 mL of the 1+1 hydrochloric acid solution, 10 mL of the ascorbic acid solution and 10 mL of the thiourea solution are added to each, and each is diluted to the mark, mixed and left for 30 min; the arsenic concentrations of the series are 0.0 ng/mL, 1.0 ng/mL, 2.0 ng/mL, 5.0 ng/mL, 10.0 ng/mL and 20.0 ng/mL. The fluorescence values are measured under the same conditions as the sample solution, the reagent blank value is subtracted, and the curve is plotted with arsenic mass concentration on the abscissa and fluorescence value on the ordinate.

Calculation and permissible difference: the arsenic content is expressed as the mass fraction and is obtained from the arsenic mass concentration read off the working curve, in nanograms per millilitre, and the mass of the test portion, in grams; the arithmetic mean of parallel determinations is the final result. Table 3 gives the permissible relative deviation as 30 % for an arsenic content below 0.000 5 %, 20 % for 0.000 5 % to 0.002 0 % and 10 % above 0.002 0 %.