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GB/T 20899.12-2016

Methods for chemical analysis of gold ores - Part 12: Determination of arsenic, mercury, cadmium, lead and bismuth contents - Atomic fluorescence spectrometric method

金矿石化学分析方法 第12部分：砷、汞、镉、铅和铋量的测定 原子荧光光谱法

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1 Scope

This part of GB/T 20899 lays down the method for determining the arsenic, mercury, cadmium, lead and bismuth contents of gold ores.

The part applies to the determination of arsenic, mercury, cadmium, lead and bismuth in gold ores. The determination range is 0.000 04 % to 0.20 %.

2 Summary of the method

The test portion is dissolved in a mixed acid of hydrochloric acid and nitric acid in a water bath, or dissolved with hydrochloric acid, nitric acid, perchloric acid and hydrofluoric acid. A reducing agent brings the As(V) in the test solution to As(III) and the Cd(IV) to Cd(II); lead, mercury and bismuth need no prereduction. A masking agent masks the interfering elements in the test solution. At a set acidity the test solution and a potassium borohydride solution pass through a hydride generator and produce hydrides, which the carrier gas takes into a quartz tube where they are atomized; the fluorescence intensity is measured on an atomic fluorescence spectrometer and the arsenic, mercury, lead and bismuth contents are calculated by the working curve method.

The oxidation states written in this clause are reproduced as printed in the standard,

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including the reduction of Cd(IV) to Cd(II).

3 Reagents

Unless otherwise stated, only reagents confirmed to be of analytical grade and doubly deionized water are used in the analysis.

The concentrated acids listed are hydrochloric acid of density 1.19 g/mL, nitric acid of density 1.42 g/mL, hydrofluoric acid of density 1.18 g/mL, perchloric acid of density 1.76 g/mL and sulfuric acid of density 1.84 g/mL, all of guaranteed grade.

The diluted acids listed are hydrochloric acid (1+1), (1+19) and (1+49), each diluted from the concentrated hydrochloric acid; nitric acid (1+4), diluted from the concentrated nitric acid; sulfuric acid (1+4), diluted from the concentrated sulfuric acid; and a mixed acid (1+3+4), made up by mixing 1 volume of nitric acid, 3 volumes of hydrochloric acid and 4 volumes of water, to be prepared immediately before use.

Two sodium hydroxide solutions are listed, at 5 g/L and at 200 g/L. Potassium borohydride solution I at 20 g/L is prepared by weighing 10.0 g of potassium borohydride and dissolving it in 500 mL of the 5 g/L sodium hydroxide solution, immediately before use. Potassium borohydride solution II at 20 g/L is prepared in the same way, with 5 g of potassium ferricyanide added and dissolved, again immediately before use.

Further reagents listed are a thiourea-ascorbic acid solution at 100 g/L, oxalic acid at 80 g/L, cobalt chloride hexahydrate solution at 0.5 g/L, sodium pyrophosphate at 20 g/L, potassium sulfate at 100 g/L and barium chloride at 50 g/L.

Standard stock solutions at 100 μg/mL are prepared for arsenic from arsenic trioxide, for mercury from mercuric chloride, for bismuth from bismuth metal, for lead from lead metal and for cadmium from cadmium metal, the metals being of mass fraction not less than 99.99 %; each is dissolved as prescribed and made up to the mark in a 1 000 mL volumetric flask.

From these stock solutions the standard working solutions are prepared by successive dilution in volumetric flasks with addition of nitric acid or hydrochloric acid: a combined arsenic and bismuth standard solution I at 2 μg/mL and a solution II at 100 ng/mL; a mercury intermediate solution at 2 μg/mL, a mercury standard solution I at 100 ng/mL and a solution II at 10 ng/mL; a lead standard solution I at 2 μg/mL and a solution II at 100 ng/mL; and a cadmium intermediate solution at 2 μg/mL, a cadmium standard solution I at 100 ng/mL and a solution II at 10 ng/mL.

4 Apparatus

An atomic fluorescence spectrometer fitted with hollow cathode lamps for arsenic, mercury, cadmium, lead and bismuth. The detection limit, the precision and the correlation coefficient

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of the working curve are to meet the provisions of the metrological calibration procedure.

5 Test sample

5.1 The particle size of the test sample is not to exceed 0.074 mm.

5.2 The test sample is dried at 100 °C to 105 °C for 1 h and then cooled to room temperature in a desiccator.

6 Analytical procedure

6.1 The test portion is weighed out from the test sample according to Table 1, on the basis of the content of the element to be determined, to the nearest 0.000 1 g. Two determinations are carried out independently and the mean of the two is taken.

6.1 Table 1 is arranged as four blocks, one for arsenic together with bismuth, one for mercury, one for lead and one for cadmium. Each block carries five columns of content ranges of the element to be determined, expressed as mass fractions in per cent and running from the lowest range up to the top of the determination range. For each content range the table fixes the mass of the test portion in grams, the make-up volume in millilitres, the aliquot volume in millilitres, the dilution volume in millilitres and which of the two working curves, I or II, is to be used. A footnote states that the figure in brackets in the dilution volume row gives the volume ratio of a second dilution. The individual figures of the table are not reproduced here.

6.2 A blank test is carried out along with the test portion.

6.3.1 Preparation of the solution for arsenic, mercury and bismuth: the test portion is placed in a colorimetric tube, the size of the tube being chosen from the make-up volume given in Table 1, 10 mL of the mixed acid is added, the tube is heated on a water bath for 2 h, and after cooling the solution is diluted to the mark with water, shaken up and left to stand.

6.3.2 Preparation of the solution for arsenic, bismuth, lead and cadmium: the test portion is placed in a 150 mL polytetrafluoroethylene beaker; 6 mL of hydrochloric acid, 3 mL of nitric acid, 3 mL of hydrofluoric acid and 2 mL of perchloric acid are added, the beaker is covered with a watch glass and heated at low temperature until most of the test portion has decomposed, then the temperature is raised to not more than 220 °C until decomposition is complete and the solution has evaporated almost to dryness. The beaker is taken off and cooled, a small amount of hydrochloric acid (1+1) is added to dissolve the soluble salts, the solution is transferred to a volumetric flask, the size of the flask being chosen from the make-up volume given in Table 1, diluted to the mark with hydrochloric acid (1+19), shaken up and left to stand.

6.3.3 Aliquots of the solution from 6.3.1 and of the solution from 6.3.2 are taken according to Table 1 into volumetric flasks of the corresponding volume. The fluorescence intensity of

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each element in the test solution is measured by the same method used to plot the working curve, the blank fluorescence intensity is subtracted, and the mass concentration of the element determined is read from the corresponding working curve; Annex A is cited for reference. A note states that volumetric flasks, pipettes, colorimetric tubes, polytetrafluoroethylene beakers and polytetrafluoroethylene watch glasses are to be soaked for 12 h in the nitric acid (1+4) solution and cleaned before use.

6.4.1 Working curves for arsenic and bismuth: aliquots of 0.00 mL, 0.50 mL, 1.00 mL, 2.00 mL, 4.00 mL and 5.00 mL of the arsenic and bismuth standard solution I, and aliquots of 0.00 mL, 1.00 mL, 2.00 mL, 4.00 mL, 8.00 mL and 10.00 mL of the arsenic and bismuth standard solution II, are taken into 100 mL volumetric flasks. To each are added in turn 5 mL of hydrochloric acid and 5 mL of the thiourea-ascorbic acid solution; the flask is made up to the mark with water, shaken up and left for 30 min before measurement. The two sets form the arsenic and bismuth working curve I and the arsenic and bismuth working curve II. The standard solutions are measured with potassium borohydride solution I as the reducing agent and hydrochloric acid (1+19) as the carrier stream. The working curves are plotted with the fluorescence intensity of the standard solutions on the vertical axis and the concentration of the arsenic and bismuth solutions on the horizontal axis.

6.4.2 Working curve for mercury: aliquots of 0.00 mL, 1.00 mL, 2.00 mL, 4.00 mL, 8.00 mL and 10.00 mL of the mercury standard solution I, and the same series of aliquots of the mercury standard solution II, are taken into 100 mL volumetric flasks. To each are added in turn 5 mL of hydrochloric acid and 5 mL of the thiourea-ascorbic acid solution; the flask is made up to the mark with water and shaken up before measurement. The two sets form the mercury working curve I and the mercury working curve II. The standard solutions are measured with potassium borohydride solution I as the reducing agent and hydrochloric acid (1+19) as the carrier stream. The working curve is plotted with the fluorescence intensity of the standard solutions on the vertical axis and the concentration of the mercury solutions on the horizontal axis.

6.4.3 Working curve for lead: aliquots of 0.00 mL, 0.50 mL, 1.00 mL, 2.00 mL, 4.00 mL and 5.00 mL of the lead standard solution I, and aliquots of 0.00 mL, 1.00 mL, 2.00 mL, 4.00 mL, 8.00 mL and 10.00 mL of the lead standard solution II, are taken into 100 mL volumetric flasks. To each is added 4 mL of the oxalic acid solution and the flask is made up to the mark with hydrochloric acid (1+49), shaken up and measured. The two sets form the lead working curve I and the lead working curve II. The standard solutions are measured with potassium borohydride solution II as the reducing agent and hydrochloric acid (1+49) as the carrier stream. The working curve is plotted with the fluorescence intensity of the standard solutions on the vertical axis and the concentration of the lead solutions on the horizontal axis.

6.4.4 Working curve for cadmium: aliquots of 0.00 mL, 1.00 mL, 2.00 mL, 4.00 mL, 8.00 mL and 10.00 mL of the cadmium standard solution I, and the same series of aliquots of the

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