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Replacing GB/T 13748.13-2005

Methods for chemical analysis of magnesium and magnesium alloys - Part 13: Determination of lead, calcium, potassium and sodium contents - Flame atomic absorption spectrometry

镁及镁合金化学分析方法 第13部分：铅、钙、钾、钠含量的测定 火焰原子吸收光谱法

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Table of Contents

Foreword

The sample was processed into fragments with a thickness of no more than 1 mm.

6 Determination of lead content

6.1 Method Overview The sample was dissolved in hydrochloric acid, and lead was enriched by co-precipitation using ferric hydroxide as a carrier. In a hydrochloric acid medium, a lean-burn air-acetylene flammable material was used for separation. The absorbance of lead was measured at a wavelength of 283.3 nm using a flame atomic absorption spectrometer. The mass concentration of lead was determined from the working curve, and the concentration was calculated. The mass fraction of lead was obtained.

6.2 Reagents Unless otherwise specified, only reagents confirmed to be of analytical grade and grade II water as specified in GB/T 6682 shall be used in the analysis.

6.2.1 Hydrochloric acid (1 1). Transfer 500 mL of hydrochloric acid (rho=

1.19 g/mL, analytical grade) to 500 mL of water and mix well.

6.2.2 Ammonia (1 1). Transfer 100 mL of ammonia (rho=

0.90 g/mL, analytical grade) to 1000 mL of water and mix well.

6.2.3 Iron solution (1 mg/mL). Weigh

0.484 g of ferric chloride hexahydrate (FeCl3·6H2O), add 5 mL of hydrochloric acid (6.2.1), and dilute with water. Dissolve and dilute to 100 mL, then mix well.

6.2.4 Ammonia-Ammonium Chloride Washing Solution. Weigh 135g of ammonium chloride, dissolve it in 500mL of water, add 48mL of ammonia solution (6.2.2), and transfer to 1000mL of [preparation solution]. Dilute the solution to the mark with water in a volumetric flask and mix well; then transfer 10 mL of the solution to a 500 mL volumetric flask, dilute to the mark with water, and mix well.

6.2.5 Lead Standard Stock Solution (1 mg/mL). Weigh 1.0000 g of lead (wPb >= 99.99%), place it in a 400 mL beaker, and add... 30 mL nitric acid (1.2), cover with a watch glass, heat slowly until completely dissolved, boil for 5 minutes, cool, transfer to a 1000 mL volumetric flask, and use... Dilute with water to the mark and mix well. 1 mL of this solution contains 1 mg of lead. Alternatively, use a certified standard solution.

6.2.6 Lead Standard Solution A (100 μg/mL). Transfer

25.00 mL of lead standard stock solution (6.2.5) to a 250 mL volumetric flask, add... 1 mL of hydrochloric acid (6.2.1) is diluted to the mark with water and mixed well. 1 mL of this solution contains 100 μg of lead.

6.2.7 Lead Standard Solution B (20 μg/mL). Transfer

10.00 mL of lead standard stock solution (6.2.5) to a 500 mL volumetric flask, add... 2 mL of

hydrochloric acid (6.2.1), diluted to the mark with water, and mixed well. 1 mL of this solution contains 20 μg of lead.

6.2.8 Phenolphthalein ethanol solution (1 g/L).

6.3 Test Procedure

6.3.1 Sample Weigh 2.50g of sample (Chapter 5), accurate to 0.0001g, and record it as m0.

6.3.2 Parallel Tests Perform the experiment twice in parallel and take the average value.

6.3.3 Blank Test A blank test was performed along with the sample.

6.3.4 Measurement

6.3.4.1 Place the sample in a 250mL beaker, cover with a watch glass, add approximately 5mL of water, and then add 35mL of hydrochloric acid (6.2.1) in portions. Allow to steep. After the strong reaction stops, heat until completely dissolved, then cool.

6.3.4.2 Add 4 mL of iron solution (6.2.3), and dilute with water to approximately 50 mL. Add 4 drops of phenolphthalein ethanol solution (6.2.8) while stirring. Add ammonia water (6.2.2) until the solution turns purple-red, then add an excess of 1 mL. Add 10 mL of ammonia-ammonium chloride washing solution (6.2.4) and let stand for 2 hours.

6.3.4.3 Filter using medium-speed quantitative filter paper, and wash the precipitate four times with ammonia-ammonium chloride washing solution (6.2.4). Place the precipitate in a 25 mL beaker, add... Dissolve the precipitate in hydrochloric acid (6.2.1) (hydrochloric acid temperature. 60°C~80°C), then transfer the solution to a 25mL volumetric flask and dilute to the mark with water. Mix well.

6.3.4.4 At a wavelength of 283.3 nm using a flame atomic absorption spectrometer, with an air-acetylene lean flame and zeroed with water, compare with the corresponding series... The absorbance of lead was measured simultaneously with the standard solution. After subtracting the absorbance of the blank test solution accompanying the sample, the mass of lead was determined from the working curve. Concentration (rhoPb).

6.3.5 Plotting the Working Curve

6.3.5.1 Based on the mass fraction of lead in the sample, the standard solutions for the working curve series are prepared as follows:

---When the lead mass fraction is 0.0010%~0.0040%. Transfer 0mL, 1.00mL, 2.00mL, 3.00mL, 4.00mL, and 5.00mL. Lead standard solution B (6.2.7) was placed in six 25 mL volumetric flasks, and 5 mL of hydrochloric acid (6.2.1) was added to each flask. The solutions were then diluted with water to the specified concentrations. Scale, mix well.

---When the lead mass fraction is >0.0040%~0.010%. Transfer 0 mL,

3.00 mL. Lead standard solution A (6.2.6) was placed in six 25 mL volumetric flasks, and 5 mL of hydrochloric acid (6.2.1) was added to each flask. The solutions were then diluted with water to the specified concentrations. Scale, mix well.

6.3.5.2 The series of standard solutions were analyzed using an air-acetylene lean flame at a wavelength of 283.3 nm on a flame atomic absorption spectrometer, with water added. Zero, the absorbance of lead is measured. The x-axis represents the mass concentration of lead, and the y-axis represents the absorbance minus the absorbance of the "zero concentration" solution. Plot the working curve.

6.5 Precision

6.5.1 Repeatability The measured values of two independent test results obtained under repeatability conditions, within the range of the average values given in Table 2, represent the two test results. The absolute difference does not exceed the repeatability limit (r), and the number of cases exceeding the repeatability limit (r) does not exceed 5%. The repeatability limit (r) is calculated using linear data from Table 2. It can be obtained by interpolation or extension.

6.5.2 Reproducibility The measured values of two independent test results obtained under reproducibility conditions, within the range of the average values given in Table 3, represent the two test results. The absolute difference should not exceed the reproducibility limit (R), and the number of cases exceeding the reproducibility limit (R) should not exceed 5%. The reproducibility limit (R) is determined according to the data in Table 3. Obtained by linear interpolation or extrapolation.

7 Determination of calcium content

7.1 Method Overview The sample was dissolved in hydrochloric acid and, in the presence of lanthanum chloride, analyzed by flame atomic absorption spectrometry at a wavelength of 422.7 nm using an air-acetylene-rich flame retardant. The absorbance of calcium is measured by flame, and the mass concentration of calcium is found according to the working curve, and the mass fraction of calcium is calculated.

7.2 Reagents Unless otherwise specified, only reagents confirmed to be of analytical grade and grade II water as specified in GB/T 6682 shall be used in the analysis.

7.2.1 Hydrofluoric acid (rho=1.14g/mL).

7.2.2 Hydrogen peroxide (rho=1.10g/mL).

7.2.3 Nitric acid (rho=1.42g/mL).

7.2.4 Perchloric acid (rho=1.67g/mL).

7.2.5 Hydrochloric acid (1 1).