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Methods for Chemical Analysis of Tin-Lead Solders – Part 11:

Determination of Phosphorus Content – Crystal Violet

Phosphorus-Vanadium-Molybdenum Heteropoly Acid

Spectrophotometry

锡铅焊料化学分析方法

第 11 部分：磷量的测定结晶紫-磷钒钼杂多酸分光光度法

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Foreword

GB/T 10574 Methods for Chemical Analysis of Tin-Lead Solders can be divided into the following 14 parts:

- Part 1: Determination of Tin Content – Potassium Iodate Titrimetric Method;
- Part 2: Determination of Antimony Content - Malachite Green Spectrophotometric Method and Potassium Bromate Titration;
- Part 3: Determination of Bismuth – Flame Atomic Absorption spectrometry;
- Part 4: Determination of Iron Content - 1,10-Phenanthroline Spectrophotometry;
- Part 5: Determination of Arsenic Content – Arseno-Antimono-Molybdenum Blue Spectrophotometry;
- Part 6: Determination of Copper Content – Flame Atomic Absorption spectrometry;
- Part 7: Determination of Silver Content – Flame Atomic Absorption Spectrometry and Potassium Thiocyanate Potentiometric Titration;
- Part 8: Determination of Zinc Content – Flame Atomic Absorption Spectrometry;
- Part 9: Determination of Aluminum Content – Electrothermal Atomic Absorption Spectrometry;
- Part 10: Determination of Cadmium Content – Flame Atomic Absorption Spectrometry and Na₂EDTA Titration;
- Part 11: Determination of Phosphorus Content – Crystal Violet Phosphorus-Vanadium-Molybdenum Heteropoly Acid Spectrophotometry;
- Part 12: Determination of Sulfur Content - High-Frequency Combustion Infrared Absorption Spectrometry;
- Part 13: Determination of Antimony, Bismuth, Iron, Arsenic, Copper, Silver, Zinc, Aluminum, Cadmium, Phosphorous and Gold Contents - Inductively Coupled Plasma Atomic Emission Spectrometric Method;
- Part 14: Determination of Tin, Lead, Antimony, Bismuth, Silver, Copper, Zinc, Cadmium and Arsenic Content - Optical Emission Spectrometry.

This Part belongs to Part 11 of GB/T 10574.

This Part was drafted as per the rules specified in GB/T 1.1-2009.

Methods for Chemical Analysis of Tin-Lead Solders – Part 11:

Determination of Phosphorus Content – Crystal Violet

Phosphorus-Vanadium-Molybdenum Heteropoly Acid

Spectrophotometry

1 Scope

This Part of GB/T 10574 specifies the determination of phosphorus content in the tin-lead solders.

This Part is applicable to the determination of phosphorus content in the tin-lead solders. The measurement range is 0.0005% ~ 0.0100%.

This Part belongs to the arbitration analysis method.

2 Normative References

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) are applicable to this document.

GB/T 8012 Casting Tin-Lead Solders

3 Method Summary

The test materials shall use nitric acid and hydrochloric acid to decompose; then in the sulfuric acid medium, use hydrochloric acid-hydrobromic acid to volatilize and remove the tin, arsenic and antimony, the lead is precipitated as lead sulfate. Take the test solution separately and adjust the acidity, then in the presence of polyvinyl alcohol, phosphorus-vanadium-molybdenum heteropoly acid and crystal violet form a purple-red ion complex; measure its absorbance at a wavelength of 545nm of spectrophotometer.

the operation for twice. The sulfuric acid continues to smoke till the residual sulfuric acid volume is 2mL~3mL; take off and cool off. Use small amount of water to wash the beaker wall, boil for 2min; cool off to the room temperature; use water to transfer into 50mL volumetric flask and dilute to the scale; mix evenly. Use a slow quantitative filter paper to dry filter in a 100mL beaker; discard the initial portion of the filtrate; respectively take 10.00mL of filtrate into 100mL volumetric flask.

6.4.3 Add one drop of p-nitrophenol solution (4.7); add 5.0mL of sodium hydroxide solution (4.6); use sulfuric acid (4.5) to adjust till the yellow color fades; add 10.0mL of sulfuric acid solution (4.5); dilute with water to about 60mL; mix evenly. Under shaking condition, slowly add 10mL of molybdenum-ammonium vanadate mixture (4.10); mix evenly, stand for 10min. Add 2.0mL of polyvinyl alcohol solution (4.11), mix evenly. Add 4.0mL of crystal violet solution (4.12); dilute with water to the scale; mix evenly. Place for 1h.

6.4.4 Transfer partial test solution into 1cm cuvette; take the blank test solution of the test materials as the reference; measure its absorbance at the wavelength of 545nm of the spectrophotometer. Find the corresponding amount of phosphorus from the working curve.

6.5 Drawing of working curve

6.5.1 Pipette 0mL, 1.00mL, 2.00mL, 4.00mL, 6.00mL, 8.00mL, 10.00mL of phosphorus standard solution (4.14) into a group of 100mL volumetric flasks; dilute with water to about 20mL; add 1.2mL of sulfuric acid (4.4), the following procedures shall take as 6.4.3.

6.5.2 Under the same conditions of measuring the test materials, take reagent blank as the reference; measure the absorbance of serial standard solutions. Take phosphorus quantity as abscissa, absorbance as the ordinate, then draw the working curve.

7 Calculation of Analytical Results

The phosphorus quantity shall be calculated by phosphorus mass fraction w_p , the value shall be expressed in %; calculate by Formula (1):

$$w_p = \frac{m_1 \cdot V \times 10^{-6}}{m \cdot V_1} \times 100 \quad \dots\dots\dots (1)$$

Where:

m_1 – mass of phosphorus checked from the working curve, in μg ;

V – total volume of test solution, in mL;

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