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HS

**INDUSTRY STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

HS/T 62-2019

**Determination of copper content in printed circuit board waste
and scrap -- Wavelength dispersive X-Ray fluorescence
spectrometry**

(English Translation)

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Foreword

This standard was drafted in accordance with the rules given in HS/T 1-2011 and HS/T 39-2013.

This standard was proposed by the Customs Administration Department of the General Administration of Customs of the People's Republic of China.

This standard is under the jurisdiction of the Department of Policies and Regulations of the General Administration of Customs of the People's Republic of China.

Appendix A and Appendix B of this standard are informative appendices.

This standard was drafted. Chongqing Customs, People's Republic of China.

1 Scope

This standard specifies test methods for quantitative analysis of copper content in PCB waste and scrap.

This standard is applicable to the determination of copper content in PCB waste and scrap. The measurement range is 10.0 g/100g ~ 56.0 g/100g.

2 Normative references

The following documents are essential for the application of this document. For dated references, only the dated version applies to this article Pieces. For undated references, the latest version (including all amendments) applies to this document.

GB/T 6682 National Standard for Analytical Laboratory Water JY/T 016 General rules for wavelength-dispersive X-ray fluorescence spectroscopy

3 Principle

The crusher and ring grinder are used to crush and pre-process the PCB. After homogenization, the muffle furnace is used for burning. After burning, the ball is ground.

The instrument performs secondary pulverization, and then prepares as a fuse. The measured value is obtained by X-fluorescence quantitative analysis, and then the weighing value of the sample before and after burning, the elemental ratio of copper in copper oxide and X-fluorescence quantitative measurement calculate the copper content in the PCB.

4 Reagents and materials

Unless otherwise specified, the reagents used in this method are analytical grade, and the water is grade III water specified in GB/T 6682.

4.1

Lithium tetraborate (LiB_4O_7).

4.2

Lithium metaborate (LiBO_2).

4.3

LiBr . Prepared as a solution with a mass fraction of 18 g/100g.

4.4

Lithium nitrate (LiNO_3). formulated as a solution with a mass fraction of 22 g/100g.

4.5

Corundum crucible. 100 mL volume.

4.6 Platinum-gold alloy crucible (95% P 5% Au)

4.7 Desiccator. An effective desiccant is included.

4.8 Reference materials. copper oxide (CuO), silicon dioxide (SiO_2), aluminum oxide (Al_2O_3), iron oxide (Fe_2O_3), oxygen Calcium (CaO), magnesium oxide (MgO).

5 Equipment

5.1

Wavelength dispersion X-ray fluorescence spectrum. The composition and performance of the instrument should meet the requirements of JY/T 016.

5.2

Pulverizer. Sieve with 5 cm hole size.

5.3

Ring grinder. with tungsten carbide grinding head.

5.4

Ball mill. The maximum speed is not less than 500 rpm.

5.5

Electronic balance. Sensitivity 0.1 mg.

5.6

Muffle furnace. the maximum operating temperature is not less than 850 °C, and the cavity volume is not less than 30 L.

5.7

Automatic temperature control oven. The maximum use temperature is not lower than 110 °C.

5.8

Melt sample machine. The maximum operating temperature is not less than 1150 °C, with a crucible.

5.9

Standard sieve. 100 mesh.

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5.10

Microsampler.

6.1.1 Crush

A 1 kg sample was weighed and pulverized into an irregular block and powder with a maximum diameter of 5 cm by a pulverizer (5.2), and recorded as sample a.

6.1.2 Ring grinding

The sample a was put into a ring mill, and the obtained powder was passed through a 100-target sieve, and recorded as sample b.

6.1.3 Burn

The following steps are started, and two samples are tested in parallel.

6.1.3.1

Take sample b in an oven at 101 °C ~ 105 °C, dry it for 2 hours, take it out, cool to room temperature in a desiccator, and weigh about 3 g.

Record the weight as m1 (to the nearest 0.1 mg).

6.1.3.2

After weighing, put it into the corundum crucible (4.5) and place it in a muffle furnace for burning. The burning temperature is 775 °C ~ 825 °C. The burning time for 10 h. After taking it out, cool it to room temperature in a desiccator and weigh it. Record the weight as m2 (accurate to 0.1 mg).

6.1.4 Ball milling

The sample c was ball-milled. The rotation speed was set to 500 r/min and the ball-milling time was 2 minutes. The sample obtained by the ball-milling was recorded as sample d.

6.1.5 Fuse

6.1.5.1

Put sample d in an oven at 101 °C ~ 105 °C, dry it for 2 hours, and take it out. Cool it in a desiccator to room temperature before weighing.

6.1.5.2

Prepare lithium tetraborate and lithium metaborate as a mixture of lithium borate in a ratio of 67.33; weigh 0.4 g of dry sample d (standard to the nearest 0.1 mg), weigh 6 g of lithium borate mixed flux (accurate to 0.1 mg), mix uniformly, and drop in lithium bromide with a micro sampler.

The solution and lithium nitrate solution are each 1 mL. All weighing and mixing processes should be completed in a platinum-gold alloy crucible (4.6).

6.1.5.3

Put the crucible (4.6) with the sample into the melting sample machine. The recommended melting temperature is 1050 °C and the melting time is 10 minutes.