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GB/T 5121.8-2024

Methods for chemical analysis of copper and copper alloys - Part 8: Determination of oxygen, nitrogen and hydrogen contents

铜及铜合金化学分析方法 第8部分：氧、氮、氢含量的测定

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1 Scope

Part 8 of China's national series on the chemical analysis of copper and copper alloys, covering oxygen, nitrogen and hydrogen. These three gases are measured separately from every other element because they behave differently in the metal and because their effects are out of all proportion to their concentration. Oxygen in copper forms cuprous oxide at the grain boundaries, and when such copper is heated in a reducing atmosphere - which is to say, brazed or welded - hydrogen diffuses in, reduces the oxide and generates steam that cannot escape, cracking the metal from within. That is hydrogen embrittlement, and it is why oxygen-free copper exists as a separate product. Hydrogen itself causes porosity in castings as it comes out of solution on freezing. The methods are inert gas fusion with infrared or thermal conductivity detection, and the document does not apply to copper alloys containing more than 1.0 per cent zinc.

This document describes methods for determining the oxygen, nitrogen, hydrogen content in copper and copper alloys, including the determination of oxygen and nitrogen content using the inert gas melting-infrared absorption/thermal conductivity method, as well as the determination of hydrogen content using the inert gas melting-infrared absorption or thermal conductivity method. This document is applicable to the determination of oxygen, nitrogen, hydrogen content in copper and copper alloys. Method 1 is applicable to the individual or simultaneous determination of oxygen and nitrogen content in copper and copper alloys, while Method 2 is applicable to the determination of hydrogen content in

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copper and copper alloys. The determination range is shown in Table 1. This document does not apply to the determination of oxygen, nitrogen, hydrogen content in copper and copper alloys with a zinc content greater than 1.0%.

2 Normative references

The following documents, through normative references in this document, constitute essential provisions of this document. For dated references, only the edition cited applies; for undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 6682 Water for analytical laboratory use - Specification and test methods YS/T 668 The sampling method of physical and chemical testing for copper and copper alloy

3 Terms and definitions

This document does not contain any terms and definitions that require definition.

4 Method 1.Determination of oxygen and nitrogen content

by inert gas fusion-infrared absorption/thermal conductivity method

4.1 Principle The prepared sample is placed in a graphite crucible (a nickel basket is used when the sample contains refractory components) and heated to melt under an inert atmosphere, releasing oxygen and nitrogen. Oxygen is released as carbon monoxide, which, along with the inert gas, passes through a rare-earth copper oxide furnace, oxidizing most of the carbon monoxide to carbon dioxide. The carbon dioxide and remaining carbon monoxide are introduced into an infrared detector for measurement. The computer system calculates the oxygen content based on the sample mass and signal intensity. After the carbon monoxide, carbon dioxide, water in the gas path are absorbed and separated, nitrogen gas is introduced into a thermal conductivity detector via an inert carrier gas flow, outputting a signal. The computer system calculates the nitrogen content based on the sample mass and signal intensity.

4.2 Reagents or materials Unless otherwise specified, only reagents confirmed to be of analytical grade are used in the analysis.

4.2.1 Water, GB/T 6682, grade II or higher purity.

4.2.4 Glacial acetic acid (rho =

4.2.5 Anhydrous ethanol or acetone.

4.2.6 Mixed acid. Nitric acid (4.2.2), phosphoric acid (4.2.3), glacial acetic acid (4.2.4) are mixed in a volume ratio of 10.28.62.

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4.2.7 Standard substances/standard samples. Oxygen and nitrogen standard substances/standard samples of copper and copper alloys, or other applicable standard substances/standard samples.

4.2.8 Nickel basket. 1 g, purity not less than 99.9%, oxygen content not greater than 0.0010%, nitrogen content not greater than 0.0002%. Except for the no-wash nickel basket, the surface should be cleaned as follows before use. Soak in a mixed acid (4.2.6) for 5 minutes; then quickly remove and rinse with running water for 2 ~ 3 minutes. After cleaning with anhydrous ethanol or acetone (4.2.5), soak in fresh anhydrous ethanol or acetone (4.2.5) for later use. Dry with cold air before use.

4.2.9 High-purity carrier gas (helium or argon). Purity not less than 99.999%.

4.2.10 Power gas (nitrogen, argon, or compressed air). Impurities (oil or water) not greater than 0.5%.

4.2.11 Graphite crucible. Made of high-purity or spectrally pure graphite.

4.3 Instrumentation and equipment Inert gas melting-infrared/thermal conductivity detection system (including electrode furnace, dust collection device, carrier gas purification and analytical gas conversion system, infrared detector and thermal conductivity detector, computer and software control system). If only oxygen or nitrogen content needs to be detected, an instrument equipped with a separate infrared detector or thermal conductivity detector can be selected.

4.4 Samples

4.4.1 Process the sample into round bars or strips with a diameter of 2 mm ~ 5 mm or a cross-section of 2 mm x 2 mm ~ 5 mm x 5 mm and a length greater than 40 mm, or other small pieces with suitable size and weight, according to YS/T 668.Wires and filaments can be sampled directly.

4.4.2 Use a lathe and steel file to remove surface contamination or oxide layers, exposing the fresh metal surface. Overheating shall be avoided during processing to prevent oxidation.

4.4.3 Use a special steel file, hand saw, or wire cutter to process the sample to the required weight. The processed sample shall be free of defects visible to the naked eye, such as pores and cracks.

4.4.4 Immerse the sample in a mixed acid (4.2.6) for 10 minutes; remove it; wash it quickly with water; then wash it with anhydrous ethanol or acetone (4.2.5); dry it with cold air; analyze it immediately. The prepared sample must be free from contamination before and during testing. Clean tweezers shall be used to handle the sample during the testing process. If the sample is oxidized, this step shall be repeated.

4.4.5 If only nitrogen content is being tested, procedures

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4.4.1 To

4.4.4 can be followed, or the sample can be prepared into a shaving form according to YS/T 668 for analysis.

4.5.1 Sample Weigh

0.10 g ~

2.00 g of sample (4.4), accurate to 0.0001 g.

4.5.2 Parallel test Perform two measurements repeatedly; take the average of the results.

4.5.3 Blank test Perform a blank test along with the sample (4.5.1). The blank test shall be repeated at least three times; the average value shall be used for blank compensation or subtraction. The blank value (absolute value) after compensation or subtraction shall not exceed 0.00005%. If a nickel basket (4.2.8) is used when analyzing samples, the blank test shall also include a blank in the nickel basket (4.2.8).

4.5.4 Instrument calibration

4.5.4.1 Single standard point calibration Select a standard substance/standard sample (4.2.7) whose oxygen and nitrogen contents are close to the expected contents of the sample to be tested, or make the oxygen and nitrogen masses in the standard substance/standard sample (4.2.7) similar to those in the sample to be tested by controlling the mass of the sample. Perform the single standard point calibration procedure, according to the instrument manual; repeat the analysis at least three times, taking the average value and calibrating the oxygen and nitrogen calibration curves, respectively. For the sample to be tested with unknown contents, the instrument's built-in original working curve or other similar working curves can be used to pre-analyze the sample (4.5.1) according to 4.5.5, to obtain the expected contents of oxygen and nitrogen in the sample to be tested.

4.5.4.2 Multi-point calibration Select two or more standard substances/standard samples (4.2.7). The oxygen and nitrogen content in the standard substances/standard samples (4.2.7) shall cover the oxygen and nitrogen content in the sample to be tested, or the oxygen and nitrogen mass in the standard substances/standard samples (4.2.7) shall cover the oxygen and nitrogen mass in the sample to be tested by controlling the sample mass. Perform multi-point curve calibration according to the instrument manual. Each standard substance/standard sample (4.2.7) shall be analyzed at least three times; the average value shall be used to calibrate the oxygen and nitrogen calibration curves, respectively.

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