



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

GB/T 5124.4-2017

**Hard metals -- Part 4: Determination of titanium content --
Photometric peroxide method**

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1 Scope

This part of GB/T 5124 specifies the hydrogen peroxide spectrophotometric method for the determination of titanium content in hard metals and carbides.

2 Scope of application

This part of GB/T 5124 is applicable to the determination of titanium content in mixed powders of carbides and bonded metals (without lubricant) and all grades of pre-sintered or sintered hard metals. The measuring range is above 0.2% (mass fraction).

3 Principles of the method

A yellow pertitanic acid complex is generated, and the absorbance of the complex is measured.

4 Interfering elements

Under specified conditions, elements that form colored complexes with hydrogen peroxide will interfere. For example, vanadium and molybdenum interfere quantitatively with the determination; correction can be made if the content of each element is less than 5% (mass fraction).

5 Reagents

Only reagents confirmed to be of analytical grade and distilled or corresponding pure water are used in the analysis.

5.4 High-purity titanium metal or titanium dioxide, in which the total amount of

8.2 Sample Weigh 0.1 g or 0.2 g of the sample, accurate to 0.0001 g.

8.3 Sample decomposition Place the sample in a 250 mL beaker or 100 mL~200 mL Erlenmeyer flask, add 5 g of ammonium sulfate (5.2) and 10 mL of sulfuric acid (5.7), cover with a glass watch glass, and heat to nearly boiling until completely dissolved.

Another sample dissolution method is as follows: Place the sample in a platinum dish, add 10 mL of water and 5 mL of hydrofluoric acid (5.10), and cover the platinum dish with a polypropylene or platinum watch glass; heat to about 80 degrees C, add nitric acid (5.11)

dropwise until the sample is completely dissolved, and then cool it. Add 10 mL of sulfuric acid (5.7) and 5 g of ammonium sulfate (5.2), heat until smoking (SO_3) occurs, and cool.

If any undissolved particles or carbonaceous materials remain, cool the solution to less than 100 °C, carefully add 1 mL of perchloric acid (5.6), heat until perchloric acid fumes emerge, and then cool to room temperature.

WARNING: Avoid contact with organic materials when using perchloric acid.

8.4 Preparation of analytical solutions Add 1 mL of hydrogen peroxide (5.9) in portions and shake well. While shaking, add

30 mL of citric acid solution (5.5) in portions, and add 40 mL of water.

8.4.1 Using a 250 mL volumetric flask Transfer the analysis solution into a 250 mL volumetric flask, wash it thoroughly with water, add 25 mL of sulfuric acid (5.8), dilute the solution with water to close to the mark, mix well, and cool. Add 1 mL of hydrogen peroxide (5.9), mix well, dilute to volume with water, and mix well.

8.4.2 Using a 500 mL volumetric flask Transfer the analysis solution into a 500 mL volumetric flask, wash it thoroughly with water, add 30 mL of citric acid solution (5.5) and 50 mL of sulfuric acid (5.8), dilute the solution with water to close to the mark, mix well, and cool. Add 2 mL of hydrogen peroxide (5.9), mix well, dilute to volume with water, and mix well.

8.5 Preparation of reference solutions Place about 30 mL of the color-developed solution (8.4) into a 50 mL beaker, and add about 0.2 g of sodium bisulfite (5.3) to make the solution fade. Add more sodium bisulfite if necessary.

8.6 Measurement of absorbance Choose a suitable absorption dish. Use the same or

paired absorption dishes to measure the absorbance of the color-developed solution and the reference solution at a wavelength of 420 nm.

9 Elimination of interference

9.1 Vanadium Place 30 mL of the color-developed solution (8.4) into a 50 mL beaker, and add about

0.1 g of ammonium hydrogen fluoride (5.1) to destroy the colored complex of titanium.

After 3 minutes, the color of the solution will be the color of vanadium.

Measure the absorbance of vanadium and subtract the absorbance of vanadium from the absorbance of the combined vanadium and titanium.

9.2 Molybdenum The color of the molybdenum peroxide complex is light and cannot be eliminated by chemical methods, but the correction value can be calculated based on the content of molybdenum. Usually at 420 nm, 1% of molybdenum in the sample is equivalent to approximately 0.08% of titanium. However, a spectrophotometer must be used to determine the correction value.

10 Drawing of working curve

10.1 Titanium standard solution Titanium standard solution can be prepared with metallic titanium or titanium dioxide (5.4).

10.1.1 Preparation of titanium standard solution from titanium metal Weigh about 250 mg of titanium metal (5.4) and place it in a 200 mL Erlenmeyer flask.

Add 30 mL of water and 20 mL of sulfuric acid (5.8), cover the Erlenmeyer flask, heat slowly and continuously add water to keep the volume constant until the metal is completely dissolved, and then cool. Add about 1 mL of hydrogen peroxide (5.9) to oxidize titanium, heat to evaporate the water until smoking, and cool. Add 10 g of ammonium sulfate (5.2), slowly heat until all sediments on the wall of the Erlenmeyer flask are dissolved, and then cool. Add about 50 mL of water, and transfer to a 250 mL volumetric flask; add 25 mL of sulfuric acid (5.8), cool, dilute with water to the mark, and mix well. Calculate the content of titanium per liter of solution.