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# Methods for chemical analysis of titanium sponge, titanium and titanium alloys - Part 3: Determination of silicon content - Molybdenum blue spectrophotometry

海绵钛、钛、及钛合金化学分析方法 第 3 部分：硅量的测定 钼蓝分光光度法

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# Methods for chemical analysis of titanium sponge, titanium and titanium alloys - Part 3: Determination of silicon content - Molybdenum blue spectrophotometry

## 1 Scope

This Part of GB/T 4698 specifies the determination of silicon content, in sponge titanium, titanium, titanium alloys.

This Part applies to the determination of silicon content, in sponge titanium, titanium, titanium alloys. Measurement range: 0.010% ~ 0.70%.

## 2 Method summary

The sample is dissolved in hydrofluoric acid. The fluoride ion is complexed by boric acid. In the sulfuric acid medium, silicon and ammonium molybdate generate silicon molybdenum yellow heteropoly acid. Silicon molybdenum yellow is reduced to silicon molybdenum blue, by ascorbic acid. The absorbance is measured, at a spectrophotometer wavelength of 810 nm.

When the content of vanadium in the color developing solution is more than 2 mg, it will interfere positively with the determination. The corresponding amount of vanadium can be added to the blank test solution, during color development. Its absorbance can be deducted, to eliminate the interference.

## 3 Reagents

Unless otherwise stated, only reagents and grade-2 water confirmed to be superior grade are used in the analysis.

**3.1** Nitric acid (ρ = 1.42 g/mL).

**3.2** Hydrofluoric acid (1 + 5).

**3.3** Boric acid saturated solution. Store in plastic bottles.

**3.4** Ammonia (1 + 3).

**3.5** Sulfuric acid (1 + 11).

**3.6** Potassium permanganate solution (30 g/L): Analytically pure.

**3.7** Ammonium molybdate solution (100 g/L): Stored in a plastic bottle, analytically pure.

**3.8** Sulfuric acid (1 + 3).

**3.9** Ascorbic acid (100 g/L): Prepared at the time of use, analytically pure.

**3.10** Titanium matrix solution: Weigh 0.50 g of metal titanium ($w_{Si}$ < 0.003%, $w_{Ti}$ ≥ 99.99%) into a plastic beaker. Add 20 mL of water. Add 10 mL of hydrofluoric acid (3.2) in stages. Heat and dissolve in a water bath, at about 60 °C. Add 6 ~ 8 drops of nitric acid (3.1) dropwise, until the solution is clear. Add 50 mL of saturated boric acid solution (3.3). Mix well. Transfer to a 100 mL plastic volumetric flask. Use water to dilute it to the mark. Mix well. 1 mL of this solution contains 0.50 mg of titanium.

**3.11** Silicon standard storage solution: Weigh 1.0679 g of silica ($w_{SiO2}$ > 99.99%), that was pre-fired at 1000 °C for 30 min and cooled to room temperature, in a desiccator into a platinum crucible. Add 5 g of sodium carbonate and 5 g of potassium carbonate. Mix well. Melt it in 1000 °C high temperature furnace for 20 min. Take out. Cool it down. The frit is leached by hot water in a polytetrafluoroethylene beaker. Heat to dissolve and make it clear. Cool it down. Transfer to a 500 mL volumetric flask. Use water to dilute it to the mark. Mix well. 1 mL of this solution contains 1 mg of silicon. Store it in plastic bottles.

**3.12** Silicon standard solution: Pipette 10.00 mL of silicon standard storage solution (3.11), into a 200 mL volumetric flask. Use water to dilute it to the mark. Mix well. 1 mL of this solution contains 50 μg of silicon. Store it in plastic bottles.

**3.13** Vanadium standard solution: Weigh 1.785 g of vanadium pentoxide (spectrographically pure), in a 150 mL beaker. Add 50 mL of sodium hydroxide solution (10 g/L). Heat to dissolve it. Cool it down. Add 10 mL of sulfuric acid (1 + 1). Transfer it into a 1000 mL volumetric flask. Use water to dilute it to the mark. Mix well. 1 mL of this solution contains 1 mg of vanadium.

**3.14** 2,4-dinitrophenol solution (1.0 g/L).

# 4 Instruments

Spectrophotometer.

# 5 Specimens

Sampling of titanium and titanium alloys is carried out, in accordance with the corresponding standard methods, that have been issued.

b) If the content of vanadium in the test solution taken is greater than 2 mg, add vanadium standard solution (3.13) to the blank solution, so that the amount of vanadium is the same as the amount of vanadium in the test solution taken. Proceed according to 6.4.2 ~ 6.4.5.

**6.4.7** Move part of the solution into a 1 cm cuvette. Take the blank solution with the sample as a reference. Measure its absorbance, at a wavelength of 810 nm, by a spectrophotometer. The mass of silicon is calculated, from the corresponding working curve.

### 6.5 Drawing of working curve

**6.5.1** Accurately pipette 0 mL, 0.20 mL, 0.40 mL, 0.80 mL, 1.20 mL, 1.60 mL of the silicon standard solution (3.12). Place them in six 100 mL volumetric flasks, respectively. Add the same amount of titanium matrix solution (3.10) as the amount of titanium in the test solution taken. Use water to dilute it to 20 mL. The following shall be carried out according to 6.4.2 ~ 6.4.5.

**6.5.2** Pipette a portion of the test solution into a 1 cm cuvette. Measure its absorbance at a spectrophotometer wavelength of 810 nm, which uses the solution with a silicon mass of "zero" as a reference. Take the mass of silicon as the abscissa AND the absorbance as the ordinate, to draw the working curve.

## 7 Calculation of analysis results

Calculate the mass fraction of silicon according to formula (1):

$$w_{Si} = \frac{m_1 \cdot V_0 \times 10^{-6}}{m_0 \cdot V_1} \times 100\% \qquad \cdots\cdots (1)$$

Where:

$m_1$ - The mass of silicon in the test solution, which is calculated from the working curve, in microgram (μg);

$m_0$ - The mass of the sample, in grams (g);

$V_0$ - The total volume of the test solution, in milliliters (mL);

$V_1$ - The volume of the test solution, in milliliters (mL).

## 8 Precision

### 8.1 Repeatability

For the determined values of the results from two independent tests, which are obtained

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