

JIS

JAPANESE INDUSTRIAL STANDARD

**Methods for chemical analysis
of tungsten powder and
tungsten carbide powder**

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JAPANESE INDUSTRIAL STANDARD

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Methods for chemical analysis of tungsten
powder and tungsten carbide powder

H 1402-1992

1. Scope

This Japanese Industrial Standard specifies the methods for determination of iron, molybdenum, non-volatile contents, oxygen, total carbon and free carbon in tungsten powder and tungsten carbide powder specified in JIS H 2116.

Remarks: The standards cited in this Standard are as follows:

JIS H 2116-Tungsten Powder and Tungsten Carbide Powder

JIS K 0050-General Rules for Chemical Analysis

JIS K 0115-General Rules for Molecular Absorptiometric
Analysis

JIS K 0121-General Rules for Atomic Absorption Spectro-
chemical Analysis

JIS Z 2613-General Rules for Determination of Oxygen in
Metallic Materials

JIS Z 2615-General Rules for Determination of Carbon in
Metallic Materials

JIS Z 8401-Rules for Rounding off of Numerical Values

2. General matters

General matters common to chemical analysis shall be in accordance with JIS K 0050, JIS K 0115, JIS K 0121, JIS Z 2613, and JIS Z 2615.

3. Sampling and treating methods for analysis sample

3.1 Sampling of analysis sample The analysis sample shall be taken in accordance with 4.1 (sampling and preparation of analysis sample) of JIS H 2116.

3.2 Treating of analysis sample An amount necessary for analysis of a sample is taken from the sample taken in 3.1, and is put into a suitable container to prevent oxidation. It is hermetically sealed to prepare an analysis sample.

3.3 Weighing of sample The sample shall be weighed as follows.

- (1) For a test portion, transfer the analysis sample into another container, and allow to stand in a desiccator for 1 h or longer. Thereafter, weigh out the test portion.

Further, for a test portion for determination of oxygen, weigh out especially as soon as possible.

- (2) For weighing out of the test portion, generally use a chemical balance, and read to the nearest 1 mg.

4. Expression of analysis value

4.1 Number of times of analyses As a rule, analyze the same sample two times.

4.2 Blank test At analysis, perform a blank test, and correct the measured value.

4.3 Expression of analysis value The analysis value shall be expressed by mass percentage as follows.

- (1) For a content of tungsten, after subtracting respective content calculated values of iron, molybdenum, and nonvolatile contents from 100, express the residue to one place of decimal and discard the second place and thereafter.
- (2) For a content of tungsten carbide, after subtracting respective content calculated values of iron, molybdenum, nonvolatile contents, and free carbon from 100, express the residue to one place of decimal and discard the second place and thereafter.
- (3) For contents of iron, molybdenum, and nonvolatile contents, calculate to four places of decimals, and round off to three places of decimals according to JIS Z 8401.
- (4) For contents of oxygen, total carbon, and free carbon, calculate to three places of decimals, and round off to two places of decimals according to JIS Z 8401.

5. Cautions to analysis operation

Though cautions shall be always taken for safety and hygiene for performing analysis operation, cares shall be taken especially for the following items.

- (1) For operation using hydrofluoric acid and nitric acid, carefully operate in a ventilation room.
- (2) In an atomic absorption spectrochemical analysis, take care to treating of high pressure gas, ignition of flame and extinguishing fire. Specially, in the case of using nitrogen suboxide - acetylene flame, take care to back fire and explosion.

Further, take care also to toxicity of nitrogen suboxide.

- (3) For treatment of organic solvent and others, have thorough knowledge of its toxicity, inflammability, explosiveness, etc. and consider countermeasures sufficiently.
- (4) When a liquefied hydrogen chloride cylinder is used for determination of nonvolatile content, take cares thoroughly to handling.

Further, discharge exhaust gas of hydrogen chloride after thoroughly treating.

6. Method for determination of iron

6.1 Classification of determination method The method for determination of iron shall be in accordance with either one of the following:

- (1) 1,10-phenanthroline absorptiometry This method shall apply to the sample of 0.001 wt% or over up to and incl. 0.5 wt% iron content.
- (2) Atomic absorption spectrochemical analysis This method shall apply to the sample of 0.001 wt% or over up to and incl. 0.5 wt% iron content.

6.2 1,10-phenanthroline absorptiometry

6.2.1 Summary Decompose a sample with a suitable reagent, add tartaric acid to make complex salts with tungsten, iron, etc. Thereafter, adjust pH, reduce iron (III) to iron (II) with L (+) - ascorbic acid, add 1,10-phenanthroline to generate complex of 1,10-phenanthroline and iron (II), and measure the absorbance with a photoelectric photometer.

6.2.2 Reagents The reagents shall be as follows.

- (1) Boric acid solution (50 g/l)
- (2) Mixed acid A (1 part nitric acid, 1 part hydrofluoric acid, and 3 parts water)
- (3) Mixed acid B (15 parts hydrochloric acid, 5 parts nitric acid, and 2 parts sulfuric acid)
- (4) Aqueous ammonia
- (5) Sodium hydroxide solution (100 g/l)
- (6) Hydrogen peroxide
- (7) Acetic acid
- (8) Tartaric acid solution (500 g/l)
- (9) L (+) - Ascorbic acid solution (50 g/l)

Prepare at each time of use.

- (10) 1,10-phenanthroline solution

Dissolve 0.2 g of 1,10 phenanthroline hydrochloride monohydrate in water, and dilute the amount of solution to 100 ml with water.

(11) Standard iron solution (20 $\mu\text{gFe/ml}$)

Decompose 0.200 g of iron (99.9 wt% or over) with 20 ml hydrochloric acid (1+1) by heating, add 1 ml of hydrogen peroxide thereto, and oxidize iron by boiling. Also, decompose excess hydrogen peroxide. After cooling to ordinary temperature, transfer into a 1000 ml one mark volumetric flask by using water, and dilute to the mark with water to make stock solution (200 $\mu\text{gFe/ml}$). Dilute a necessary amount of the stock solution accurately 10 times each service to make standard iron solution.

(12) p-Nitrophenol solution (4 g/l)

6.2.3 Weighing out amount of sample The weighing out amount of a sample shall be 1.0 g.

6.2.4 Operation

6.2.4.1 Preparation of sample solution Prepare sample solution in accordance with any one of the following procedures.

(1) Decomposition with hydrogen peroxide⁽¹⁾

- (a) Weigh out a sample, transfer into a beaker (200 to 300 ml), and moisten the sample with several ml of water. Cover with a watch glass, add 5 to 15 ml of hydrogen peroxide, and decompose by standing or heating.
- (b) After evaporating by heating until the amount of solution becomes about 5 ml, dilute to about 20 ml the amount of solution with water. After cooling to room temperature, add 15 ml of sodium hydroxide solution, and allow to stand for several min. After violent effervescence has ended, boil until the amount of solution becomes 10 to 20 ml, and decompose excess hydrogen peroxide. Add 10 ml of tartaric acid solution, and boil. After cooling to room temperature, wash the bottom surface of the watch glass with water, remove the watch glass, and dilute to about 60 ml the amount of solution with water.

(2) Decomposition with mixed acid A

- (a) Weigh out a sample, and transfer into a platinum pan (No. 75 or 90), a polytetrafluoroethylene beaker (100 to 200 ml) or a polyethylene beaker (100 to 200 ml).
- (b) Cover with a platinum lid, a polytetrafluoroethylene watch glass or a polyethylene watch glass, add little by little 10 ml of mixed acid A, and decompose by gently heating⁽²⁾. Expel nitrogen oxide and others by successively heating. After cooling to room temperature, wash the bottom surface of the lid or watch glass with water, and remove the lid or watch glass.

Add 10 ml of tartaric acid solution and 20 ml of boric acid solution while stirring⁽³⁾ and dilute to about 60 ml the amount of solution with water.

(3) Decomposition with hydrogen peroxide and mixed acid B

- (a) Weigh out a sample, transfer into a beaker (200 to 300 ml), and moisten the sample with several ml of water. Cover with a watch glass, add 5 to 15 ml of hydrogen peroxide, decompose by standing or heating, and evaporate by heating until tungstic acid is deposited.
- (b) Add little by little 20 ml of mixed acid B, and heat. After ending effervescence, wash the bottom surface of the watch glass with water, remove the watch glass, and continue to heat almost to drying up. After cooling to room temperature, add about 5 ml of water and 20 ml of sodium hydroxide solution, cover with the watch glass, and dissolve salts by heating. Add 10 ml of tartaric acid solution, and boil⁽³⁾. After cooling to room temperature, wash the bottom surface of the watch glass with water, remove the watch glass, and dilute to about 60 ml the amount of solution with water.

Notes ⁽¹⁾ This procedure does not apply to tungsten carbide powder.

⁽²⁾ In the case of using a polyethylene beaker, decompose while heating on a water bath or in a water bath not to cause deformation of a container by heating.

⁽³⁾ When free carbon is recognized, filter with filter paper (class 5 A), wash 3 or 4 times with water, and joint the filtrate and washings.

6.2.4.2 Coloration Carry out the coloration according to the following procedure.

- (1) Transfer the solution obtained in (1)(b), (2)(b) or (3)(b) of 6.2.4.1 into a 100 ml one mark volumetric flask with water⁽⁴⁾.
- (2) After adding several drops of p-nitrophenol solution as an indicator, add aqueous ammonia until the color of solution turns yellow while mixing by shaking⁽⁵⁾, then drip acetic acid until the solution turns colorless, and further add several drops in excess.
- (3) After cooling to ordinary temperature, add 2 ml of L (+) - ascorbic acid solution [6.2.2 (9)] and 10 ml of 1,10-phenanthroline solution [6.2.2 (10)], mix by shaking, dilute to the mark with water, and allow to stand for about 10 min.

Notes (4) In the case of 500 μg or over the amount of iron in this solution, transfer this solution into a 100 ml one mark volumetric flask with water. After diluting to the mark with water, partially take the solution into another 100 ml one mark volumetric flask to become iron content less than 500 μg .

(5) In the case of decomposition in 6.2.4.1 (2), white turbidity occasionally occurs. In that case, the decomposition shall be performed in accordance with (1) or (3) of 6.2.4.1.

6.2.4.3 Measurement of absorbance Take a portion of the solution obtained in 6.2.4.2 (3) into the absorption cell (10 ml) of a photometer, and measure the absorbance near 510 nm wavelength by using water as contrast solution.

6.2.5 Blank test The absorbance of the solution without addition of standard iron solution, which is obtained in the preparation of working curve of 6.2.6, is taken as the absorbance of a blank test.

6.2.6 Preparation of working curve Prepare a working curve in accordance with any one of the following procedures.

- (1) In the case of preparation of sample solution by 6.2.4.1 (1)
Take several ml of water and the same amount of hydrogen peroxide as the amount joined in 6.2.4.1 (1)(a) into several beakers (200 to 300 ml), and cover with watch glasses. After operation of 6.2.4.1 (1)(b), transfer the solution respectively into a 100 ml one mark volumetric flask with water⁽⁶⁾. Deal out step by step 0 to 25.0 ml of standard iron solution [6.2.2 (11)] (0 to 500 μg as iron). Hereafter operate according to the procedure of 6.2.4.2 (2) to 6.2.4.3, prepare the relation curve between the obtained absorbance and the amount of iron, and parallel transfer the relation curve so as to pass through the original point to make a working curve.
- (2) In the case of preparation of sample solution by 6.2.4.1 (2)
Prepare several platinum pans (No. 75 or 90), polytetrafluoroethylene beakers (100 to 200 ml) or polyethylene beakers (100 to 200 ml). After operation of 6.2.4.1 (2)(b), transfer the solution respectively into a 100 ml one mark volumetric flask with water⁽⁶⁾. Deal out step by step 0 to 25.0 ml of standard iron solution [6.2.2 (11)] (0 to 500 μg as iron). Hereafter operate according to the procedure of 6.2.4.2 (2) to 6.2.4.3, prepare the relation curve between the obtained absorbance and the amount of iron, and parallel transfer the relation curve so as to pass through the original point to make a working curve.

- (3) In the case of preparation of sample solution by 6.2.4.1 (3) Take several ml of water and the same amount of hydrogen peroxide as the amount joined in 6.2.4.1 (3)(a) into several beakers (200 to 300 ml), and cover with watch glasses. After operation of 6.2.4.1 (3)(b), transfer the solution respectively into a 100 ml one mark volumetric flask with water⁽⁶⁾. Deal out step by step 0 to 25.0 ml of standard iron solution [6.2.2 (11)] (0 to 500 µg as iron). Hereafter operate according to the procedure of 6.2.4.2 (2) to 6.2.4.3, prepare the relation curve between the obtained absorbance and the amount of iron, and Parallel transfer the relation curve so as to pass through the original point to make a working curve.

Note ⁽⁶⁾ In the case where Note ⁽⁴⁾ is applied to 6.2.4.2 (1), after diluting those solutions respectively to the mark, partially take the same amount thereof as the aliquot amount of sample solution respectively into a 100 ml one mark volumetric flask.

6.2.7 Calculation The calculation is carried out according to either one of the following procedures.

- (1) In the case of no aliquot in 6.2.4.2 (1) Obtain the amount of iron from the absorbance to be obtained by subtracting the absorbance obtained in 6.2.5 from the absorbance obtained in 6.2.4.3 and the working curve prepared in 6.2.6, and calculate the content of iron in a sample according to the following formula.

$$\text{Iron wt\%} = \frac{A_1}{m} \times 100$$

where, A_1 : detection amount of iron in sample solution (g)

m : weighing out amount of sample (g)

- (2) In the case of aliquot in 6.2.4.2 (1) Obtain the amount of iron from the absorbance to be obtained by subtracting the absorbance obtained in 6.2.5 from the absorbance obtained in 6.2.4.3 and the working curve prepared in 6.2.6, and calculate the content of iron in a sample according to the following formula.

$$\text{Iron wt\%} = \frac{A_2}{m \times \frac{B}{100}} \times 100$$

where, A_2 : detection amount of iron in aliquot sample solution (g)

m : weighing out amount of sample (g)

B : aliquot amount of sample solution and blank test solution (ml)

6.3 Atomic absorption spectrochemical analysis

6.3.1 Summary After decomposing a sample by a suitable reagent, spray into an air and acetylene flame, and measure the absorbance by using an atomic absorption photometer.

6.3.2 Reagents The reagents shall be as follows.

- (1) Phosphoric acid
- (2) Boric acid solution (50 g/l)
- (3) Mixed acid (1 part nitric acid, 1 part hydrofluoric acid and 3 parts water)
- (4) Tungsten powder Class 1 specified in JIS H 2116, of which the iron content is low.
- (5) Tungsten carbide powder Class 1 specified in JIS H 2116, of which the iron content is low.
- (6) Hydrogen peroxide
- (7) Standard iron solution (20 $\mu\text{gFe/ml}$) As specified in 6.2.2 (11).

6.3.3 Weighing out amount of sample The weighing out amount of a sample shall be 2.0 g.

6.3.4 Operation

6.3.4.1 Preparation of sample solution Prepare a sample solution in accordance with either one of the following procedures.

(1) Decomposition with hydrogen peroxide⁽¹⁾

- (a) Weigh out a sample, transfer into a beaker (200 to 300 ml), moisten with several ml of water, and cover with a watch glass.
- (b) Add 1 ml of phosphoric acid and 10 to 15 ml of hydrogen peroxide, and decompose by standing or heating. Evaporate by heating until the amount of solution becomes about 10 ml. After cooling to ordinary temperature, wash the bottom surface of the watch glass with water, and remove the watch glass.
- (c) Transfer the solution into a 100 ml one mark volumetric flask with water, and dilute to the mark with water⁽⁷⁾.

(2) Decomposition with mixed acid

- (a) Weigh out a sample, and transfer to a platinum pan (No. 75 or 90) or a polytetrafluoroethylene beaker (100 to 200 ml) or a polyethylene beaker (100 to 200 ml).
- (b) Cover with a platinum lid, a polytetrafluoroethylene watch glass or a polyethylene watch glass, add 1 ml of phosphoric acid, further add little by little 15 ml of mixed acid, and decompose by gently heating⁽²⁾. Expel nitrogen oxides or the like by successively heating. After cooling to room temperature, wash the bottom surface of the lid or the watch glass with water, and remove the lid or the watch glass. Add 30 ml of boric acid while stirring, and cool to ordinary temperature⁽³⁾.

- (c) Transfer the solution into a 100 ml one mark volumetric flask with water, and dilute to the mark with water⁽⁷⁾.

(⁷) In the case where the amount of iron in the solution is 300 µg or over, partially take the solution into another 100 ml one mark volumetric flask so that the amount of iron becomes under 300 µg, and dilute to the mark with water.

6.3.4.2 Measurement of absorbance Spray a portion of the solution obtained in (1)(c) or (2)(c) of 6.3.4.1 into the air and acetylene flame of an atomic absorption photometer of which the zero point is adjusted by using water, and measure the absorbance at 248.3 nm wavelength.

6.3.5 Blank test Carry out the same operation as for a sample in parallel with the sample without using the sample⁽⁸⁾.

Note (⁸) In the case where the sample solution is partially taken by applying Note (⁷) in (1)(c) or (2)(c) of 6.3.4.1, the blank test solution is also partially taken by the same amount as for the sample solution.

6.3.6 Preparation of working curve Prepare a working curve in accordance with either one of the following procedures.

- (1) In the case of preparation of sample solution by 6.3.4.1 (1)
Weigh out several pieces of each 2.00 g of tungsten powder [6.3.2 (4)], transfer into several beakers (200 to 300 ml), moisten the tungsten powder with several ml of water, and cover with a watch glass. After operation of 6.3.4.1 (1)(b) in parallel with a sample, transfer the solution respectively into a 100 ml one mark volumetric flask with water⁽⁹⁾. Deal out step by step 0 to 15.0 ml of standard iron solution [6.3.2 (7)] (0 to 300 µg as iron), and dilute to the mark with water. Spray a portion of the solution into the air and acetylene flame of an atomic absorption photometer of which the zero point is adjusted with water, measure the absorbance at 248.3 nm wavelength in parallel with the sample, prepare the relation curve between the obtained absorbance and the amount of iron, and transfer the relation curve parallel so as to pass through the original point to make a working curve.
- (2) In the case of preparation of sample solution by 6.3.4.1 (2)
Weigh out several pieces of each 2.00 of tungsten powder [6.3.2 (4)] or tungsten carbide powder [6.3.2 (5)]⁽¹⁰⁾, and transfer to several platinum pans (No. 75 or 90), polytetrafluoroethylene beakers (100 to 200 ml), or polyethylene beakers (100 to 200 ml). After operation of 6.3.4.1 (2)(b) in parallel with the sample, transfer the solution respectively into a 100 ml one mark volumetric flask with water⁽¹¹⁾. Deal out step by step 0 to 15.0 ml of standard iron solution [6.3.2 (7)] (0 to 300 µg as iron), and dilute to the mark with water. Spray a portion of the solution into the air and acetylene flame of an atomic absorption photometer of which the zero point is adjusted with water, measure the absorbance at 248.3 nm wavelength in parallel with the sample, prepare the relation curve between the obtained absorbance and the amount of iron, and transfer the relation curve parallel so as to pass through the original point to make a working curve.

Notes (⁹) In the case where Note (⁷) is applied in 6.3.4.1 (1)(c), after diluting those solutions respectively to the mark with water, partially take the same amount as the aliquot amount of sample solution respectively into a 100 ml one mark volumetric flask.

(¹⁰) In the case where the sample is tungsten powder, use the tungsten powder [6.3.2 (4)]. In the case where the sample is tungsten carbide powder, use the tungsten carbide powder [6.3.2 (5)].

(¹¹) In the case where Note (⁷) is applied in 6.3.4.1 (2)(c), after diluting those solutions respectively to the mark with water, partially take respectively the same amount as the aliquot amount of sample solution into a 100 ml one mark volumetric flask.

6.3.7 Calculation The calculation shall be in accordance with either one of the following procedures.

- (1) In the case of no aliquot in (1)(c) or (2)(c) of 6.3.4.1 Obtain the amount of iron from the absorbance to be obtained by subtracting the absorbance obtained in 6.3.5 from the absorbance obtained in 6.3.4.2 and the working curve prepared in 6.3.6, and calculate the content of iron in a sample according to the following formula.

$$\text{Iron wt\%} = \frac{A_1}{m} \times 100$$

where, A_1 : detection amount of iron in sample solution (g)

m : weighing out amount of sample (g)

- (2) In the case of aliquot in (1)(c) or (2)(c) of 6.3.4.1 Obtain the amount of iron from the absorbance to be obtained by subtracting the absorbance obtained in 6.3.5 from the absorbance obtained in 6.3.4.2 and the working curve prepared in 6.3.6, and calculate the content of iron in a sample according to the following formula.

$$\text{Iron wt\%} = \frac{A_2}{m \times \frac{B}{100}} \times 100$$

where, A_2 : detection amount of iron in aliquot sample solution (g)

m : weighing out amount of sample (g)

B : aliquot amount of sample solution and blank test solution (ml)

7. Methods for determination of molybdenum

7.1 Classification of determination method The method for determination of molybdenum shall be in accordance with either one of the following methods.

- (1) Thiocyanic acid absorptiometry This method shall apply to the sample of 0.001 wt% or over up to and including 0.16 wt% molybdenum content.
- (2) Atomic absorption spectrochemical analysis This method shall apply to the sample of 0.001 wt% or over up to and including 0.7 wt% molybdenum content.

7.2 Thiocyanic acid absorptiometry

7.2.1 Summary Decompose a sample with a suitable reagent, add citric acid and hydrochloric acid to make acidic solution, add potassium thiocyanate and ethyl acetate, reduce molybdenum (IV) with tin (II) chloride, extract molybdenum thiocyanate complex to be generated in ethyl acetate, and measure the absorbance by using a photoelectric photometer.

7.2.2 Reagents The reagents shall be as follows.

- (1) Hydrochloric acid
- (2) Nitric acid
- (3) Mixed acid A (1 part nitric acid, 1 part hydrofluoric acid, and 3 parts water)
- (4) Mixed acid B (15 parts hydrochloric acid, 5 parts nitric acid, and 2 parts sulfuric acid)
- (5) Sodium hydroxide solution (100 g/l)
- (6) Tungsten powder Class 1 specified in JIS H 2116 of which the molybdenum content is low.
- (7) Tungsten carbide powder Class 1 specified in JIS H 2116 of which the molybdenum content is low.
- (8) Hydrogen peroxide
- (9) Iron (III) solution Dissolve 0.87 g of ammonium iron (III) sulfate dodecahydrate in 100 ml of sulfuric acid (0.1 mol/l). 1 ml of this solution contains about 1 mg as iron.
- (10) Potassium thiocyanate solution Dissolve 150 g of potassium thiocyanate in 350 ml of water.
- (11) Tin (II) chloride solution After dissolving 150 g of tin (II) chloride dihydrate in 100 ml of hydrochloric acid by heating, add 250 ml of water. Add a small amount of tin and preserve in a brown bottle.

- (12) Citric acid solution Dissolve 500 g of citric acid monohydrate in 800 ml of water.
- (13) Ethyl acetate
- (14) Standard molybdenum solution (10 $\mu\text{gMo/ml}$) Decompose 0.150 g of molybdenum (VI) trioxide (99.5 wt% or over) with 10 ml sodium hydroxide solution (100 g/l) by heating. After cooling to ordinary temperature, transfer into a 1000 ml one mark volumetric flask with water, and dilute to the mark with water to make a stock solution (10 $\mu\text{gMo/ml}$). Dilute a necessary amount of the stock solution accurately 10 times each service to make standard molybdenum solution.

7.2.3 Weighing out amount of sample The weighing out amount of a sample shall be as given in Table 1.

Table 1. Weighing out amount of sample

Molybdenum content wt%	Weighing out amount of sample g
0.001 or over to and excl. 0.015	1.00
0.015 or over up and incl. 0.16	0.10

7.2.4 Operation

7.2.4.1 Preparation of sample solution Prepare a sample solution in accordance with any one of the following procedures.

(1) Decomposition with hydrogen peroxide⁽¹⁾

- (a) Weigh out a sample, and transfer into a beaker (200 to 300 ml). Moisten with several ml of water, and cover with a watch glass.
- (b) Add 5 to 15 ml of hydrogen peroxide, and decompose by standing or heating. After evaporating by heating until the amount of solution becomes about 5 ml, and dilute to about 20 ml the amount of solution with water. After cooling to room temperature, add 15 ml of sodium hydroxide solution, and allow to stand for several min. After ending violent effervescence, boil until the amount of solution becomes 10 to 20 ml, and decompose excess hydrogen peroxide.
- (c) After cooling to ordinary temperature, wash the bottom surface of a watch glass with water, remove the watch glass, transfer the solution into a 100 ml one mark volumetric flask with water, and dilute to the mark with water.