GB

NATIONAL STANDARD OF THE

PEOPLE’S REPUBLIC OF CHINA

ICS 25.160.50
J 33

GB/T 10046-2018

Replacing GB/T 10046-2008

Silver brazing filler metals

银钎料

(ISO 17672:2016, Brazing - Filler metals, MOD)

Issued on: May 14, 2018 **Implemented on: December 01, 2018**

Issued by: State Administration for Market Regulation;

Standardization Administration of the PRC.

Table of Contents

Silver brazing filler metals

1 Scope

This Standard specifies the requirements such as model, technical requirements, test methods, inspection rules, packaging, marking, quality certification of silver brazing filler metals.

This Standard applies to silver brazing filler metals used in brazing methods.

2 Normative references

The following documents are indispensable for the application of this document. For the dated references, only the editions with the dates indicated are applicable to this document. For the undated references, the latest edition (including all the amendments) are applicable to this document.

GB/T 6682 Water for analytical laboratory use - Specification and test methods (GB/T 6682-2008, ISO 3696:1987, MOD)

GB/T 8170 Rules of rounding off for numerical values and expression and judgement of limiting values

GB/T 11363 Test method of the strength for brazed and soldered joint (GB/T 11363-2008, ISO 5187:1985, NEQ)

GB/T 11364 Test method of wettability for brazing filler metals (GB/T 11364-2008, ISO 5179:1983, NEQ)

GB/T 12806 Laboratory glassware - One-mark volumetric flasks (GB/T 12806-2011, ISO 1042:1998, NEQ)

GB/T 12808 Laboratory glassware - One mark pipettes

GB/T 12809 Laboratory glassware - Principles of design and construction of volumetric glassware (GB/T 12809-2015, ISO 384:1978, NEQ)

GB/T 12810 Laboratory glassware - Volumetric glassware - Methods for use and testing of capacity (GB/T 12810-1991, ISO 4787:1984, IDT)

6 Inspection rules

Finished brazing filler metals are inspected by the manufacturer's quality inspection department according to the heat number.

6.1 Heat number division

The definition of heat number of brazing filler metals depends on the method of smelting and refining, as follows:

- In the metal smelting process, slag-metal or gas-metal reactions occur. The heat number refers to the material obtained from the same furnace smelting.

- In the metal smelting process, no obvious chemical reactions occur (such as induction melting or vacuum melting in a controlled atmosphere). The heat number refers to a group of materials obtained by continuous smelting under the same conditions in a smelting furnace, using one lot of metals and alloy elements with controlled burden. And the chemical composition of each furnace of smelting material is in line with the purchaser or brazing filler metal manufacturer's specified range.

6.2 Sampling method

During the inspection of each furnace of brazing filler metals, according to the required quantity, representative samples shall be taken from at least three locations. The sampling method and sampling location shall be recorded.

6.3 Acceptance

The manufacturer of brazing filler metals shall, in writing, provide the main performance parameters such as chemical composition, dimensions, appearance of brazing filler metals; declare that "when tested in accordance with this Standard, the brazing filler metals provided meet the requirements of this Standard".

6.4 Reinspection

When any one of the inspections fails, this item shall be double reinspected. For the chemical composition analysis of brazing filler metals, only those elements that do not meet the requirements are reinspected. The results of the doubled reinspection shall meet the requirements of this item.

If the reinspection result is still unqualified, brazing filler metals in this furnace cannot be delivered as finished products, which meet this Standard.

Annex B

(Normative)

Determination of silver content

B.1 Silver chloride gravimetric method

B.1.1 Scope

This method is suitable for the determination of silver content in silver brazing filler metals. Determination range (mass fraction): 25.0%~99.95%.

B.1.2 Method summary

USE nitric acid to decompose the test portion. If there is precipitation, after filtration, add hydrochloric acid to form silver chloride precipitation; USE a glass crucible to separate it; and, weigh its mass after drying (the filtrate may be used to determine the copper content).

B.1.3 Reagents

B.1.3.1 Nitric acid (1+1).

B.1.3.2 Nitric acid (1+100).

B.1.3.3 Hydrochloric acid (1+9).

B.1.4 Analytical procedures

B.1.4.1 Test portion

WEIGH 1.0 g of sample, accurate to 1 mg.

B.1.4.2 Determination

B.1.4.2.1 PLACE the weighed test portion (B.1.4.1) in a 300 mL tall beaker; ADD 10 mL of nitric acid (B.1.3.1) and cover with a watch glass; slowly heat to decompose; BOIL to allow nitrogen oxides to escape. USE water to rinse the inner wall of the beaker; ADD warm water to 50 mL. At this time, if there is precipitation, it shall let it stand for 1 h in a place where the temperature is slightly higher; then use slow-speed filter paper to filter it. USE hot nitric acid (B.1.3.2) to wash the precipitation.

B.1.4.2.2 ADD water to 150 mL; while stirring, add hydrochloric acid (B.1.3.3) solution after calibration, in moles per liter (mol/L);

V_0 - The value of the volume of ammonium thiocyanate standard titration solution consumed for calibration, in milliliters (mL);

107.868 - The value of the molar mass of silver, in grams per mole (g/mol).

Calibrate three parts in parallel. The range of the volume of the ammonium thiocyanate standard titration solution consumed shall not exceed 0.10 mL. TAKE the average. Otherwise, re-calibrate.

B.2.4 Analytical procedures

B.2.4.1 Test portion

WEIGH 0.50 g of sample, accurate to 1 mg.

B.2.4.2 Determination

B.2.4.2.1 PLACE the weighed test portion (B.2.4.1) in a 300 mL conical beaker; ADD 15 mL of nitric acid (B.2.3.1) and cover with a watch glass; slowly heat to decompose; BOIL to allow nitrogen oxides to escape. After cooling, use water to rinse the inner wall of the beaker; then add water to dilute to 150 mL.

B.2.4.2.2 ADD 3 mL of ferric ammonium sulfate solution (B.2.3.2) as an indicator; while shaking, use ammonium thiocyanate standard titration solution (B.2.3.3) to titrate, until the solution just appears reddish-brown, which is the end point.

B.2.5 Expression of analysis results

The silver content is expressed in (Ag). The value is expressed in %, calculated according to formula (B.3):

$$\omega(\mathrm{Ag}) = \frac{c \times V \times 107.868}{m_0 \times 1\,000} \times 100 \qquad \cdots\cdots\cdots\cdots (\mathrm{B.3})$$

Where:

c - The actual concentration value of ammonium thiocyanate standard titration solution after calibration, in moles per liter (mol/L);

V - The value of the volume of ammonium thiocyanate standard titration solution consumed in the titration, in milliliters (mL);

m_0 - The value of the mass of test portion, in grams (g);

107.868 - The value of the molar mass of silver, in grams per mole (g/mol).

Annex C

(Normative)

Determination of copper content - Electrolysis-spectrophotometry

C.1 Scope

This annex applies to the determination of copper content in silver brazing filler metals. Determination range (mass fraction): 14.00%~60.00%.

C.2 Method summary

USE nitric acid to decompose the test portion; ADD hydrochloric acid for precipitation to separate silver; USE sulfuric acid to smoke away hydrochloric acid; after cooling, add water and nitric acid for low-current electrolysis. After the electrolysis is terminated, the platinum cathode is washed using water and absolute ethanol, dried, cooled, and then weighed. USE spectrophotometry to measure the copper content remaining in the solution after electrolysis and correct it to obtain the copper content.

C.3 Reagents

C.3.1 Absolute ethanol.

C.3.2 Nitric acid (1+1).

C.3.3 Nitric acid (1+100).

C.3.4 Hydrochloric acid (1+9).

C.3.5 Sulfuric acid (1+1).

C.3.6 Aqueous ammonia (1+1).

C.3.7 Ammonium citrate solution (500 g/L).

C.3.8 Dicyclohexanone oxalyl dihydrazone (BCO) solution (1 g/L): WEIGH 0.5 g of BCO in a 300 mL beaker; ADD 50 mL of ethanol, 200 mL of warm water to dissolve; TRANSFER to a 500 mL volumetric flask; USE water to dilute to the mark and mix well.

C.3.9 Copper standard stock solution: WEIGH 0.1000 g of pure copper into a 150 mL beaker; ADD 10 mL of nitric acid (C.3.2) and cover with a watch glass; MAKE it completely decomposed at low temperature; BOIL to remove nitrogen replace it with another tall beaker containing 180 mL of water; continue electrolysis for 15 min.

C.5.2.6 Immediately take out the platinum electrode and immerse it in another 250 mL beaker filled with water; MOVE it up and down 3 times. TURN off the power and remove the electrode; PUT it in a beaker containing absolute ethanol (C.3.1); TAKE it out and place it in a 105 °C electrothermal constant-temperature drying oven for drying for 3 min~5 min; then take it out and place it in a desiccator to cool to room temperature.

C.5.2.7 WEIGH the platinum electrode after electrolytic deposition of copper, accurate to 0.0001 g.

C.5.2.8 According to the following method, determine the amount of residual copper in the solution after electrolysis.

C.5.2.8.1 Blank test: TAKE 10 mL of sulfuric acid (C.3.5), 5 mL of nitric acid (C.3.2) in a 500 mL volumetric flask; do a blank test with the test portion.

C.5.2.8.2 COMBINE the electrolyte after electrolysis and a glass of water obtained from electrolysis for 15 min into a 500 mL volumetric flask; USE water to dilute to the mark and mix well.

C.5.2.8.3 PIPETTE 10.00 mL~20.00 mL of solution into a 50 mL volumetric flask; ADD 2 mL of ammonium citrate solution (C.3.7); USE water to dilute to about 25 mL; ADD 2~3 drops of neutral red ethanol solution (C.3.11). USE aqueous ammonia solution (C.3.6) to neutralize, until the red color fades and add 1.0 mL in excess. ADD 8.0 mL of BCO solution (C.3.8); USE water to dilute to the mark and mix well; and let stand for 20 min.

C.5.2.8.4 USE a 2 cm cuvette; USE the blank test as a reference. At a wavelength of 600 nm, measure the absorbance; FIND out the corresponding amount of copper from the working curve.

C.5.2.8.5 Drawing of working curve

PIPETTE 6 parts of 20.00 mL of blank solution (C.5.2.8.1); PLACE them in a set of 50 mL volumetric flasks. ADD 0 mL, 1.00 mL, 2.00 mL, 3.00 mL, 4.00 mL, 5.00 mL of copper standard solution respectively. Then proceed according to C.5.2.8.3 and C.5.2.8.4. USE copper content as the abscissa and absorbance as the ordinate; DRAW the working curve.

C.6 Expression of analysis results

The copper content is expressed in ω(Cu). The value is expressed in %, calculated according to formula (C.1):

Annex D

(Normative)

Determination of zinc and cadmium content - EDTA titration method

D.1 Scope

This annex applies to the determination of zinc content in silver brazing filler metals. Determination range (mass fraction): 4.00%~45.00%.

This annex applies to the determination of cadmium content in silver brazing filler metals. Determination range (mass fraction): 10.00%~30.00%.

D.2 Method summary

USE nitric acid to decompose the test portion. The tin is removed by metastannic acid precipitation and filtration. USE silver chloride precipitation method to separate silver, electrolysis method to separate copper. Adjust the solution after separation of silver and copper to ammoniac. Indium and nickel are separated by filtration in the form of hydroxide precipitation and dimethylglyoxime nickel precipitation, respectively. Adjust the pH value within the range of 5.5~6.5; USE thiourea to mask the remaining copper and silver; USE EDTA standard titration solution to titrate the total amount of zinc and cadmium. ADD excess potassium iodide to the test solution to release the EDTA in Cd-EDTA; then use zinc standard solution to back-titrate the released EDTA, to indirectly measure the cadmium content. Zinc is calculated by subtraction method.

D.3 Reagents

D.3.1 Potassium iodide, analytically pure.

D.3.2 Ammonium persulfate, analytically pure.

D.3.3 Nitric acid (1+1).

D.3.4 Nitric acid (3+100).

D.3.5 Hydrochloric acid (1+1).

D.3.6 Hydrochloric acid (1+99).

D.3.7 Sulfuric acid (1+1).

TAKE 3 parts for calibration. The range of the volume of the consumed EDTA standard titration solution does not exceed 0.10 mL. TAKE the average. Otherwise, re-calibrate.

D.3.15 Xylenol orange solution (2.5 g/L).

D.4 Apparatus

D.4.1 Electrolysis device equipped with automatic stirring device, precision ammeter and voltmeter.

D.4.2 Platinum cathode: USE a platinum wire with a diameter of 0.2 mm to weave into a mesh with a mesh size of about 36 μm per square centimeter, to make a mesh cylinder (see Figure C.1 in Annex C).

D.4.3 Platinum anode: Spiral (see Figure C.2 in Annex C).

D.5 Analytical procedures

D.5.1 Test portion

WEIGH 0.40 g of sample, accurate to 1 mg.

D.5.2 Determination

D.5.2.1 PLACE the test portion (D.5.1) in a 250 mL beaker.

D.5.2.2 ADD 5 mL of nitric acid (D.3.3); COVER with a watch glass; slowly heat to decompose the test portion; REMOVE nitrogen oxides, remove and cool.

D.5.2.3 USE water to rinse the watch glass and beaker wall; HEAT and evaporate at a low temperature to make the solution volume about 5 mL. Take off and wash the watch glass and beaker wall. Adjust the volume to about 50 mL; HEAT it at a low temperature for 10 min~20 min; REMOVE it to cool.

D.5.2.4 USE medium-speed double-layer quantitative filter paper to filter by decanting method. USE hot nitric acid (D.3.4) to wash the precipitate in the beaker 3 times; then move the precipitate into the funnel; continue to use nitric acid (D.3.4) to wash the precipitate in the beaker and the funnel 8~10 times. DISCARD the precipitate and adjust the volume of the filtrate to about 120 mL.

D.5.2.5 While stirring, add 2 mL of hydrochloric acid (D.3.5); COVER with a watch glass; HEAT at low temperature to make the solution clear and remove it.

D.5.2.6 USE quantitative filter paper to filter by decanting. USE hot hydrochloric acid (D.3.6) to wash the precipitate in the beaker 3 times; then transfer the precipitate into the funnel; continue to use hot hydrochloric acid (D.3.6) to wash the precipitate in the beaker and the funnel 8~10 times. DISCARD the precipitate.

D.5.2.7 ADD 2 mL of sulfuric acid (D.3.7) to the filtrate; evaporate to smoke, cool; ADD water and 5 mL of nitric acid (D.3.3) to dissolve the salts; adjust the volume of the solution to about 150 mL. INSERT the platinum electrode pre-installed on the electrolysis device into the solution; USE two semicircular watch glasses to cover the beaker; under stirring, electrolyze with 2 A~2.5 A current, until the blue color disappears; rinse the watch glasses and the beaker wall; continue electrolysis for 2 h~3 h. Without disconnecting the current, move the electrode up and leave the liquid surface. USE water to rinse the electrode; then disconnect the power supply. HEAT and evaporate to adjust the volume of the electrolyte to about 120 mL.

D.5.2.8 ADD aqueous ammonia (D.3.8) to the electrolyte, until a strong ammonia smell appears in the test solution; and then add 5 mL in excess. ADD 1 g of ammonium persulfate (D.3.2) and boil for 1 min. COOL to room temperature; TRANSFER the solution into a 200 mL volumetric flask.

D.5.2.9 While shaking, add dimethylglyoxime ethanol solution (D.3.11) (add 7 mL of dimethylglyoxime ethanol solution (D.3.11) per 10 mg of nickel); COOL to room temperature; USE water to dilute to the mark and mix well; and dry filter.

D.5.2.10 Accurately pipette 50.00 mL of filtrate into a 300 mL conical flask; ADD nitric acid (D.3.3) until the solution is acidic (can be tested with pH test paper); and then add 5 mL in excess. ADD 2 mL of thiourea solution (D.3.9), 40 mL of hexamethylenetetramine solution (D.3.10); then add 5 drops of xylenol orange indicator (D.3.15); USE EDTA standard titration solution (D.3.14) to titrate, until the solution turns from purple-red to yellow. RECORD the volume V_1 of the EDTA standard titration solution. This is the volume of the EDTA standard titration solution consumed by zinc and cadmium.

D.5.2.11 ADD about 30 g of potassium iodide (D.3.1) to the test solution and shake well; HEAT and boil for 3 min until the test solution is clear, remove and cool. USE zinc standard solution (D.3.12) to titrate, until the solution becomes stable rose red. RECORD the volume V_2 of the zinc standard solution (D.3.12) consumed. This is the volume of zinc standard solution consumed by EDTA released from Cd-EDTA.

Note 1: If there is no tin in the test portion, D.5.2.3~D.5.2.4 can be omitted.

Note 2: If there is no nickel in the test portion, then D.5.2.8~D.5.2.9 and "ADD nitric acid (D.3.3) until the solution is acidic; and then add 5 mL in excess" in D.5.2.10 can be omitted.

Note 3: If there is no manganese in the test portion, then "ADD 1 g of ammonium persulfate (D.3.2) and boil for 1 min" in D.5.2.8 can be omitted.

Note 4: If the test portion contains only zinc, V_1 is the volume of the EDTA standard solution consumed by zinc alone; the next step does not need to be performed.

D.6 Expression of analysis results

The zinc content is expressed in ω(Zn). The value is expressed in %, calculated according to formula (D.2):

$$\omega(\mathrm{Zn}) = \frac{cV_1 \times 65.38 - V_2 \times \rho}{m_0/4 \times 1\ 000} \times 100 \quad \cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots (\mathrm{D.2})$$

Where:

c - The actual concentration value of the EDTA standard titration solution after calibration, in moles per liter (mol/L);

ρ - The value of the concentration of zinc standard solution, in milligrams per milliliter (mg/mL);

V_1 - The value of the volume of EDTA standard titration solution consumed when titrating the total amount of zinc and cadmium, in milliliters (mL);

V_2 - The value of the volume of the zinc standard solution consumed by titrating the EDTA released in Cd-EDTA, in milliliters (mL);

m_0 - The value of the mass of test portion, in grams (g);

65.38 - The value of the molar mass of zinc, in grams per mole (g/mol).

If the test portion contains cadmium, the cadmium content is expressed in ω(Cd). The value is expressed in %, calculated according to formula (D.3):

$$\omega(\mathrm{Cd}) = \frac{V_2 \times \rho \times 1.718\ 9}{m_0/4 \times 1\ 000} \times 100 \quad \cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots (\mathrm{D.3})$$

Where:

V_2 - The value of the volume of the zinc standard solution consumed by titrating the EDTA released in Cd-EDTA, in milliliters (mL);

ρ - The value of the concentration of zinc standard solution, in milligrams per milliliter (mg/mL);

m_0 - The value of the mass of test portion, in grams (g);

E.1.5.3 Determination

E.1.5.3.1 PLACE the test portion (E.1.5.1) in a 100 mL polytetrafluoroethylene beaker; ADD 10 mL of mixed acid (E.1.3.6); and warm it to decompose all the sample. ADD 10 mL of boric acid solution (E.1.3.7); TRANSFER to a 100 mL volumetric flask; USE water to dilute to the mark and shake well. (samples that do not contain tin can be directly dissolved in 10 mL of nitric acid (E.1.3.4); boiled to drive off nitrogen oxides; cooled; transferred to a 100 mL volumetric flask; diluted with water to the mark and shaken well.) For the test solution that does not need to be divided, proceed directly according to E.1.5.3.3.

E.1.5.3.2 PIPETTE 10.00 mL of test solution in a 100 mL volumetric flask; USE water to dilute to the mark and shake well.

E.1.5.3.3 USE an air-acetylene flame; at the wavelength of 232.0 nm of the atomic absorption spectrometer, use water for zero setting; MEASURE the absorbance of the test solution simultaneously with the nickel standard solution series. The absorbance of the blank solution along with the test portion is subtracted from the measured absorbance. The corresponding nickel content is found from the working curve.

E.1.6 Drawing of working curve

E.1.6.1 WEIGH a group or one of silver, copper, and tin equivalent to the test portion in Table E.1; PUT it into a 100 mL polytetrafluoroethylene beaker; ADD 10 mL of mixed acid (E.1.3.6); and warm until the sample is completely decomposed; ADD 10 mL of boric acid solution (E.1.3.7); TRANSFER to a 100 mL volumetric flask [For samples that do not need to be divided, it is possible to directly add 0.00, 2.00, 4.00, 6.00, 8.00, 10.00 mL of nickel standard solution (E.1.3.9) respectively; USE water to dilute to the mark; and proceed according to E.1.6.3.]; USE water to dilute to the mark.

E.1.6.2 PIPETTE 6 portions of 10 mL of test solution (E.1.6.1) into a series of 100 mL volumetric flasks; ADD 0.00 mL, 2.00 mL, 4.00 mL, 6.00 mL, 8.00 mL, 10.00 mL of nickel standard solution (E.1.3.9), respectively; USE water to dilute to the mark.

E.1.6.3 Under the same conditions as the determination of test portion solution, use water for zero setting; MEASURE the absorbance of the standard solution series; subtract the absorbance of the "zero" nickel solution in the standard solution series. USE the nickel content as the abscissa and absorbance as the ordinate; DRAW the working curve.

E.1.7 Expression of analysis results

The nickel content is expressed in ω(Ni). The value is expressed in %, EDTA standard titration solution; in the ammonia solution, use chrome black T as an indicator; USE the zinc standard solution to back-titrate.

E.2.3 Reagents

E.2.3.1 Aqueous ammonia (ρ=0.90 g/mL).

E.2.3.2 Nitric acid (1+1).

E.2.3.3 Nitric acid (1+100).

E.2.3.4 Hydrochloric acid (2+1).

E.2.3.5 Hydrochloric acid (1+1).

E.2.3.6 Hydrochloric acid (1+9).

E.2.3.7 Hydrochloric acid (1+50).

E.2.3.8 Sulfuric acid (1+1).

E.2.3.9 Ammonium chloride solution (250 g/L).

E.2.3.10 Tartaric acid solution (250 g/L).

E.2.3.11 Dimethylglyoxime ethanol solution (10 g/L).

E.2.3.12 Zinc standard solution [c(Zn)=0.02 mol/L]: WEIGH 1.3076 of pure zinc (purity above 99.99%) into a 250 mL beaker; ADD 35 mL of hydrochloric acid (E.2.3.5); HEAT to decompose and remove nitrogen oxides, cool; TRANSFER to 1000 mL volumetric flask; USE water to dilute to the mark and shake well.

E.2.3.13 EDTA standard titration solution [$c(C_{10}H_{14}N_2O_8Na_2 \cdot 2H_2O)$=0.02 mol/L].

E.2.3.13.1 Preparation: WEIGH 7.45 g of ethylene diamine tetraacetic acid disodium in a 300 mL beaker; ADD water to dissolve; TRANSFER to a 1000 mL volumetric flask; USE water to dilute to the mark and mix well.

E.2.3.13.2 Calibration: PIPETTE 25.00 mL of EDTA standard titration solution; ADD 10 mL of ammonium chloride solution (E.2.3.9) and 2~3 drops of chrome black T indicator (E.2.3.14); USE water to dilute to 100 mL; ADD aqueous ammonia (E.2.3.1) dropwise, until the solution turns blue; USE zinc standard solution (E.2.3.12) to titrate, until the solution turns purple-red as the end point.

According to formula (E.2) and formula (E.3), calculate the actual concentration of EDTA standard titration solution:

nitric acid (E.2.3.2) and cover with a watch glass; slowly heat until the test portion is dissolved, boil, and remove nitrogen oxides. USE water to rinse the watch glass and the inner wall of the beaker (if there is precipitation at this time, it shall add warm water to a volume of 50 mL; and let it stand for 1 h~2 h in a place with higher temperature; then use slow-speed filter paper to filter; wash the beaker and the precipitate; discard the precipitate.). Adjust the volume of the solution to 150 mL; while stirring, add hydrochloric acid (E.2.3.6) dropwise to generate silver chloride precipitation; ADD hydrochloric acid (E.2.3.6) dropwise, until no precipitation is formed; then add 1 mL of hydrochloric acid (E.2.3.6) in excess, to make it saturated. After fully stirring, heat and boil for 5 min; COOL and stand for 2 h, filter; USE nitric acid (E.2.3.3) to wash several times; and then use water to wash thoroughly. The filtrate and washing liquid are combined in a 500 mL conical cup.

E.2.5.2.2 Then, add 10 mL of sulfuric acid (E.2.3.8) to the filtrate; HEAT and evaporate to make the sulfuric acid smoke. After standing and cooling, add 50 mL of water and 5 mL of nitric acid (E.2.3.2); HEAT to dissolve the soluble salts; and boil for 1 min~2 min to allow nitrogen oxides to escape. REMOVE it and slightly cool; TRANSFER to a 300 mL tall beaker; USE water to wash the wall of the cup; and dilute the solution volume about 150 mL.

E.2.5.2.3 INSERT the platinum electrode pre-installed on the electrolysis device into the solution; USE two semicircular watch glasses to cover the beaker; under stirring, electrolyze with 2 A~2.5 A current, until the blue color disappears; rinse the watch glasses and the beaker wall; continue electrolysis for 2 h~3 h. Without disconnecting the current, move the electrode up and leave the liquid surface. USE water to rinse the electrode; then disconnect the power supply.

E.2.5.2.4 TRANSFER the electrolyte into a 500 mL beaker; ADD 20 mL of tartaric acid solution (E.2.3.10) and 20 mL of ammonium chloride solution (E.2.3.9). USE methyl red solution (E.2.3.15) as an indicator; USE aqueous ammonia (E.2.3.1) to neutralize, until the solution turns from red to yellow, with an excess of 3 mL; adjust the volume of the solution to 300 mL.

E.2.5.2.5 HEAT the solution to 90 °C; while stirring, add dimethylglyoxime ethanol solution (E.2.3.11) (add 7 mL of dimethylglyoxime ethanol solution (E.2.3.11) per 10 mg of nickel). After stirring well, cool to room temperature and stand for 30 min. USE fast quantitative filter paper to filter; USE water to wash the beaker and the precipitate. Then, use 10 mL of hot hydrochloric acid (E.2.3.4) to dissolve the precipitate in portions into the original beaker; USE warm water and warm hydrochloric acid (E.2.3.7) to wash the filter paper; and merge the lotion into the original beaker.

E.2.5.2.6 USE a burette to accurately add the EDTA standard titration solution (E.2.3.13) (add 10 mL of EDTA standard titration solution (E.2.3.13) per 10 mg

Annex F

(Normative)

Determination of tin content - Potassium iodate titration method

F.1 Scope

This annex applies to the determination of tin content in silver brazing filler metals. Determination range (mass fraction): 2.00%~12.00%.

F.2 Method summary

USE nitric acid to decompose the test portion, to make tin precipitate metastannic acid. After separation, use nitric acid and sulfuric acid to dissolve the precipitate; in a hydrochloric acid medium, use antimony trichloride and lead or nickel to reduce tin (IV) to tin (II). USE starch solution as indicator; USE potassium iodate standard titration solution to titrate.

F.3 Reagents

F.3.1 Lead (granular).

F.3.2 Nickel wire, which can be used repeatedly.

F.3.3 Potassium iodide, solid.

F.3.4 Hydrochloric acid (ρ=1.19 g/mL).

F.3.5 Hydrochloric acid (1+1).

F.3.6 Nitric acid (ρ=1.40 g/mL).

F.3.7 Nitric acid (1+1).

F.3.8 Nitric acid (1+50).

F.3.9 Sulfuric acid (ρ=1.84 g/mL).

F.3.10 Sodium hydroxide solution (5 g/L).

F.3.11 Saturated solution of sodium bicarbonate.

F.3.12 Antimony trichloride solution (10 g/L): TAKE 5 g of antimony trichloride and put it in 500 mL of hydrochloric acid (3.1); HEAT to dissolve it.

Annex G

(Normative)

Determination of lithium content - Atomic absorption spectrometry

G.1 Scope

This annex applies to the determination of lithium content in silver brazing filler metals. Determination range (mass fraction): 0.02%~1.00%.

G.2 Method summary

USE nitric acid to decompose the test portion, to prepare the test solution to be measured. USE air-acetylene flame; at 670.7 nm wavelength of atomic absorption spectrometer, measure the absorbance.

G.3 Reagents

G.3.1 Nitric acid (1+1).

G.3.2 Lithium standard stock solution: WEIGH 0.9218 g of lithium sulfate ($Li_2SO_4 \cdot H_2O$ analytically pure) into a 250 mL beaker; ADD 50 mL of water to dissolve it; then transfer it to a 1000 mL volumetric flask; USE water to dilute to the mark and mix well. 1 mL of this solution contains 100 μg of lithium.

G.3.3 Lithium standard solution: PIPETTE 25.00 mL of lithium standard stock solution (G.3.2) into a 100 mL volumetric flask; USE water to dilute to the mark and mix well. 1 mL of this solution contains 25 μg of lithium.

G.4 Apparatus

Atomic absorption spectrometer, with lithium hollow cathode lamp.

Under the best condition of the apparatus, those which can reach the following indexes can be used.

Sensitivity: In a solution that is basically the same as the measurement test solution, the characteristic concentration of lithium shall not be greater than 0.025 μg/mL.

Precision: USE the highest concentration of standard solution to measure the absorbance 10 times; the standard deviation shall not exceed 1.0% of the average absorbance. USE the lowest concentration of standard solution (not the "zero" standard solution) to measure the absorbance 10 times; the standard

Annex H

(Normative)

Determination of indium content

H.1 Atomic absorption spectrometry

H.1.1 Scope

This method is suitable for the determination of indium content in silver brazing filler metals. Determination range (mass fraction): 0.50%~2.00%.

H.1.2 Method summary

USE nitric acid to decompose the test portion. In a dilute nitric acid medium, use an air-acetylene flame; at a wavelength of 303.9 nm of an atomic absorption spectrometer, measure the absorbance of indium.

H.1.3 Reagents

H.1.3.1 Nitric acid (1+1).

H.1.3.2 Indium standard stock solution: WEIGH 0.2500 g of pure indium (purity>99.99%) into a 200 mL tall beaker; ADD 20 mL of nitric acid (H.1.3.1) and cover with a watch glass; HEAT at low temperature until it is completely decomposed; BOIL to remove nitrogen oxides, cool; USE water to rinse the watch glass and beaker wall. TRANSFER to a 500 mL volumetric flask; USE water to dilute to the mark and mix well. 1 mL of this solution contains 500 μg of indium.

H.1.3.3 Indium standard solution: PIPETTE 20.00 mL of indium standard stock solution (H.1.3.2); PLACE it in a 50 mL volumetric flask; USE water to dilute to the mark and mix well. 1 mL of this solution contains 200 μg of indium.

H.1.4 Apparatus

Atomic absorption spectrometer, with indium hollow cathode lamp.

Under the best condition of the apparatus, those which can reach the following indexes can be used.

Sensitivity: In a solution that is basically the same as the measurement test solution, the characteristic concentration of indium shall not be greater than 0.5 μg/mL.

H.2 Aqueous ammonia separation - EDTA complexometric titration method

H.2.1 Scope

This method is suitable for the determination of indium content in silver brazing filler metals. Determination range (mass fraction): 2.00%~16.00%.

H.2.2 Method summary

USE nitric acid to decompose the test portion. In a medium containing ammonium nitrate, use aqueous ammonia as a precipitant, to precipitate indium, which is filtered and washed to separate from elements such as silver, copper, zinc. The precipitate and filter paper are decomposed by heating with nitric acid and perchloric acid and evaporated to near dryness. ADD excess ethylene diamine tetraacetic acid disodium standard solution to boil and cool. Adjust the pH between 1.5~2.0; USE xylenol orange as an indicator; USE bismuth standard titration solution to titrate.

H.2.3 Reagents

H.2.3.1 Ammonium nitrate (solid).

H.2.3.2 Perchloric acid (ρ=1.67 g/mL).

H.2.3.3 Nitric acid (ρ=1.42 g/mL).

H.2.3.4 Nitric acid (1+1).

H.2.3.5 Sulfuric acid (1+10).

H.2.3.6 Aqueous ammonia (1+1).

H.2.3.7 Ammonium nitrate-aqueous ammonia washing solution: ADD 3 g of ammonium nitrate and 5 mL of aqueous ammonia (H.2.3.6) to 100 mL of water, and mix well.

H.2.3.8 Saturated sodium bicarbonate solution.

H.2.3.9 Bismuth standard solution [c(Bi)=0.02 mol/L]: WEIGH 4.1798 g of pure bismuth (purity above 99.99%) into a 150 mL tall beaker; ADD 20 mL of nitric acid (H.2.3.4) and heat at low temperature to decompose; ADD 10 mL of perchloric acid (H.2.3.2); HEAT to emit perchloric acid fumes, and cool to room temperature. RINSE the inner wall of the beaker; adjust the volume to about 70 mL~80 mL; HEAT to dissolve the salts, cool; TRANSFER to a 1000 mL volumetric flask; USE water to dilute to the mark and mix well.

Annex I

(Normative)

Determination of aluminum content - Sodium fluoride replacement - EDTA titration method

I.1 Scope

This annex applies to the determination of aluminum content in silver brazing filler metals. Determination range (mass fraction): 1.00%~7.00%.

I.2 Method summary

After the sample is dissolved in nitric acid, silver is precipitated using hydrochloric acid and removed by filtration. ADD excess EDTA to the filtrate and heat it, to completely complex aluminum and metal ions with EDTA; USE zinc standard solution to titrate the excess EDTA. ADD sodium fluoride to replace the EDTA in the aluminum-EDTA complex; and then use zinc standard solution to titrate the released EDTA.

I.3 Reagents

I.3.1 Ascorbic acid.

I.3.2 Sodium fluoride.

I.3.3 Nitric acid (1+1).

I.3.4 Hydrochloric acid (1+1).

I.3.5 Hydrochloric acid (1+2).

I.3.6 Hydrochloric acid (2+98).

I.3.7 Aqueous ammonia (1+1).

I.3.8 Sodium hydroxide solution (300 g/L).

I.3.9 Hexamethylenetetramine solution (300 g/L).

I.3.10 EDTA standard solution [$c(C_{10}H_{14}N_2O_8Na_2 \bullet 2H_2O)=0.03$ mol/L]: Dissolve 11.168 g of ethylene diamine tetraacetic acid disodium in about 300 mL of water; TRANSFER it into a 1000 mL volumetric flask; USE water to dilute to the mark and mix well. STORE it in polyethylene container.

Annex J

(Normative)

Determination of manganese content

J.1 Potassium periodate spectrophotometry

J.1.1 Scope

This method is suitable for the determination of manganese content in silver brazing filler metals. Determination range (mass fraction): 0.50%~2.00%.

J.1.2 Method summary

USE a mixed acid of hydrofluoric acid, boric acid and nitric acid to decompose the test portion. ADD potassium periodate to oxidize manganese to permanganic acid. USE the base color solution obtained by selective reduction of permanganic acid with sodium nitrite as a reference. At the wavelength of 530 nm of the spectrophotometer, measure the absorbance.

J.1.3 Reagents

J.1.3.1 Hydrofluoric acid (ρ=1.13 g/mL).

J.1.3.2 Nitric acid (ρ=1.42 g/mL).

J.1.3.3 Nitric acid (1+3).

J.1.3.4 Sulfuric acid (ρ=1.84 g/mL).

J.1.3.5 Sulfuric acid (1+3).

J.1.3.6 Boric acid solution (40 g/L).

J.1.3.7 Dissolving agent: MIX 300 mL of boric acid solution (J.1.3.6), 30 mL of hydrofluoric acid (J.1.3.1), 500 mL of nitric acid (J.1.3.2) and 150 mL of water. STORE in a plastic bottle.

J.1.3.8 Diluent: ADD 1 mL of sulfuric acid (J.1.3.4) to 100 mL of boric acid solution (J.1.3.6). When boiling, add a few grains of potassium periodate to oxidize the organic matter that may reduce permanganic acid.

J.1.3.9 Potassium periodate solution (50 g/L): USE nitric acid (J.1.3.3) to prepare.

K.3 Principles

K.3.1 USE nitric acid and sulfuric acid to decompose the sample. In an acidic medium, use an inductively coupled plasma atomic emission spectrometer, to measure the mass concentration of each element at the corresponding wavelength.

K.3.2 When the mass fraction of nickel, zinc, cadmium, bismuth, tin, and manganese is not more than 0.001%, and the mass fraction of lead is not more than 0.002%, silver is separated by hydrochloric acid precipitation; copper is separated and enriched by electrolysis.

K.4 Reagents and materials

K.4.1 Pure silver (the mass fraction of silver is not less than 99.99%; the mass fraction of the element to be tested is not more than 0.00005%).

K.4.2 Pure copper (the mass fraction of copper is not less than 99.99%; the mass fraction of the element to be tested is not more than 0.00005%).

K.4.3 Pure zinc (the mass fraction of zinc is not less than 99.99%; the mass fraction of the element to be tested is not more than 0.00005%).

K.4.4 Pure cadmium (the mass fraction of cadmium is not less than 99.99%; the mass fraction of the element to be tested is not more than 0.00005%).

K.4.5 Absolute ethanol.

K.4.6 Nitric acid (ρ=1.42 g/mL), guaranteed reagent.

K.4.7 Nitric acid (1+1).

K.4.8 Nitric acid (1+5).

K.4.9 Nitric acid (1+100).

K.4.10 Hydrochloric acid (ρ=1.19 g/mL), guaranteed reagent.

K.4.11 Hydrochloric acid (1+1).

K.4.12 Hydrochloric acid (1+10).

K.4.13 Sulfuric acid (ρ=1.84 g/mL), guaranteed reagent.

K.4.14 Sulfuric acid (1+1).

K.4.15 Standard stock solutions of copper, nickel, zinc, manganese and lead: WEIGH 1.0000 g of pure copper, pure nickel, pure zinc, pure manganese, and

K.4.22 Tin standard stock solution: WEIGH 1.0000 g of pure tin (the mass fraction of tin is not less than 99.99%) into a 150 mL beaker. ADD 30 mL of hydrochloric acid (K.4.10), a few drops of nitric acid (K.4.6); HEAT at low temperature to dissolve; TRANSFER to a 1000 mL volumetric flask; ADD 70 mL of hydrochloric acid (K.4.10); USE water to dilute to the mark and mix well. 1 mL of this solution contains 1 mg of tin.

K.4.23 Aluminum standard stock solution: WEIGH 1.0000 g of pure aluminum (the mass fraction of aluminum is not less than 99.99%) into a 150 mL polytetrafluoroethylene beaker. ADD 10 mL of water, 5 g of sodium hydroxide; HEAT to dissolve at low temperature, cool; USE hydrochloric acid (K.4.11) to slowly neutralize, until precipitation appears. Then, add 20 mL of hydrochloric acid (K.4.11); TRANSFER it into a 1000 mL volumetric flask; USE water to dilute to the mark and mix well. 1 mL of this solution contains 1 mg of aluminum.

K.4.24 Silicon standard stock solution: WEIGH 0.4279 g of silicon dioxide (guaranteed reagent), which has been burned to constant weight at 1000 °C in advance and cooled to room temperature in a desiccator; PLACE it in a platinum crucible with 3 g of anhydrous sodium carbonate in advance. COVER 1 g~2 g of anhydrous sodium carbonate; HEAT it at a low temperature first; then melt it at 950 °C until transparent; continue to melt for 3 min; TAKE it out and cool it; USE water to leach it into a polytetrafluoroethylene beaker. TRANSFER to a 200 mL volumetric flask; USE water to dilute to the mark and mix well; and store in a plastic bottle. 1 mL of this solution contains 1 mg of silicon.

K.4.25 Silver standard stock solution: WEIGH 1.0000 g of pure silver (the mass fraction of silver is not less than 99.99%) into a 150 mL beaker. ADD 20 mL of nitric acid (K.4.7); HEAT to dissolve; BOIL to remove nitrogen oxides, and cool. TRANSFER to a 1000 mL volumetric flask; ADD 80 mL of nitric acid (K.4.7); USE water to dilute to the mark and mix well. 1 mL of this solution contains 1 mg of silver.

K.4.26 Silver standard stock solution: WEIGH 5.0000 g of pure silver (the mass fraction of silver is not less than 99.99%) into a 150 mL beaker. ADD 20 mL of nitric acid (K.4.7); HEAT to dissolve; BOIL to remove nitrogen oxides, and cool. TRANSFER to a 1000 mL volumetric flask; ADD 80 mL of nitric acid (K.4.7); USE water to dilute to the mark and mix well. 1 mL of this solution contains 5 mg of silver.

K.4.27 Standard solution A: DILUTE the standard stock solution (K.4.15~K.4.24) to 10 μg per milliliter and maintain the same acidity as the standard stock solution.

K.4.28 Standard solution B: DILUTE the standard stock solution (K.4.15~K.4.24) volumetric flasks; respectively add 0 mL, 1.00 mL, 5.00 mL, 10.00 mL of standard solution A (K.4.27), 5.00 mL, 10.00 mL of standard solution B (K.4.28); USE water to dilute to the mark and mix well. (According to the range of the mass fraction of the measured elements, select appropriate 3~4 points from the working curve for analysis.)

Note: For standard solution A (K.4.27) and standard solution B (K.4.28), the standard solution of hydrochloric acid medium needs to be prepared separately.

K.6.4.3 Working curve III - The mass fraction of the element to be tested is greater than 0.05% to the upper limit of determination in Table 1 (except silver)

ADD 0 mL, 5.00 mL, 10.00 mL of standard solution A (K.4.27) and 0.50 mL, 1.00 mL, 3.00 mL, 6.00 mL, 12.00 mL of standard stock solution (1 mL contains 1 mg in K.4.16~K.4.24) INTO one set of 100 mL volumetric flasks. ADD 10 mL of nitric acid (K.4.7) respectively; USE water to dilute to the mark and mix well. (According to the range of the mass fraction of the measured elements, select appropriate 3~4 points from the working curve for analysis.)

K.6.4.4 Working curve IV - Silver curve

ADD 0 mL, 2.50 mL, 5.00 mL, 10.00 mL of silver standard stock solution (K.4.25) and 4.00 mL, 6.00 mL, 8.00 mL of silver standard stock solution (K.4.26) INTO a set of 100 mL volumetric flasks. ADD 10 mL of nitric acid (K.4.7) respectively; USE water to dilute to the mark and mix well. (According to the range of the mass fraction of the measured elements, select appropriate 3~4 points from the working curve for analysis.)

K.6.4.5 Working curve V - The mass fraction of copper, zinc, cadmium and palladium is greater than 15%

ADD 0 mL, 5.00 mL, 15.00 mL, 20.00 mL, 25.00 mL, 30.00 mL of standard stock solutions (K.4.15), (K.4.20), (K.4.21) INTO a set of 100 mL volumetric flasks. ADD 10 mL of nitric acid (K.4.7) respectively; USE water to dilute to the mark and mix well. (According to the range of the mass fraction of the measured elements, select appropriate 3~4 points from the working curve for analysis.)

K.6.4.6 Standard sample series solution

Choose certified standard samples with the same matrix and similar mass fraction as the samples to be tested; WEIGH the same amount as the sample to be tested; prepare a series of standard sample solutions along with the test portion.

K.6.5 Determination

Translated by: Field Test Asia Pte. Ltd. (Incorporated & taxed in Singapore. Tax ID: 201302277C)

About Us (Goodwill, Policies, Fair Trading…): https://www.chinesestandard.net/AboutUs.aspx

Contact: Wayne Zheng, Sales@ChineseStandard.net

Linkin: https://www.linkedin.com/in/waynezhengwenrui/

------ The End ------