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NONFERROUS INDUSTRY STANDARD

OF THE PEOPLE'S REPUBLIC OF CHINA

ICS 71.100.10

CCS H 30

YS/T 492-2021

Replacing YS/T 492-2012

Element additive for aluminum and aluminum alloys

铝及铝合金成分添加剂

Issued on: August 21, 2021

Implemented on: February 01, 2022

Issued by: Ministry of Industry and Information Technology of PRC

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Element additive for aluminum and aluminum alloys

1 Scope

This document stipulates the product classification, quality assurance, requirements, test methods, inspection rules and markings, packaging, transportation, storage, quality certificate and order form (or contract) content of element additives for aluminum and aluminum alloy.

This document applies to additives for formulating or adjusting the chemical composition of aluminum and aluminum alloys (hereinafter referred to as "additives").

2 Normative references

The contents of the following documents constitute essential provisions of this document through normative references in the text. Among them, for dated reference documents, only the version corresponding to the date applies to this document; for undated reference documents, the latest version (including all amendments) applies to this document.

GB/T 3199 Wrought aluminium and aluminium alloy products - Packing, marking, transporting and storing

GB/T 6682 Water for analytical laboratory use - Specification and test methods

GB/T 7999 Optical emission spectrometric analysis method of aluminum and aluminum alloys

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

GB/T 17432 Methods for sampling for analyzing the chemical composition of wrought aluminum and aluminum alloys

GB/T 20975 (all parts) Methods for chemical analysis of aluminium and aluminium alloys

YS/T 491 Flux for aluminium and aluminium alloy

3 Terms and definitions

There are no terms or definitions to be defined in this document.

6.7 Powder content

The content of round cake aluminum type additive powder shall not exceed 1%; it shall not exceed 3% for other types.

6.8 Appearance quality

Additives shall not be deliquescent.

7 Test methods

7.1 Chemical composition

7.1.1 The analysis method for the main element content of additives shall comply with the provisions of Appendix B ~ Appendix G.

7.1.2 The analysis method for the fluorine content of aluminum additives shall be determined, through negotiation between the supplier and the purchaser. The analysis method for the content of other impurity elements shall comply with the provisions of Appendix H.

7.1.3 Routine analysis is only conducted on elements with specified numerical values outside the “Other” column in Tables 2 ~ 7. When it is suspected that the mass fraction of unconventional analysis elements exceeds the limit value of this document, the producer can analyze these elements; the analysis method shall be determined through negotiation between the supplier and the buyer.

7.1.4 The rounding off comparison method is used to determine the analytical value. The numerical rounding rules are in accordance with the provisions of GB/T 8170. The rounding digits are consistent with the limit digits in Tables 2 ~ 7 of this document.

7.2 Moisture

The moisture content of additives shall be determined according to the method specified in Appendix I.

7.3 Density

7.3.1 Weigh the mass of the specimen, recorded it as m_a .

7.3.2 Measure the diameter of the specimen, recorded as D . Measure the thickness of the specimen, recorded as T .

7.3.3 Calculate the density ρ of the specimen according to formula (1), in grams per cubic centimeter (g/cm^3).

take double quantity of samples from the batch of products (including the batch of products represented by the unqualified sample) for repeated testing. If all repeated test results are qualified, the batch of products will be deemed qualified. If there are still sample fractures that are unqualified in the repeated test results, the batch of products will be deemed unqualified.

8.7.6 When the fracture of any sample fails to meet the standard, it shall take double quantity of samples from the batch of products (including the batch of products represented by the unqualified sample) for repeated testing. If all repeated test results are qualified, the batch of products will be deemed qualified. If there are still sample fractures that are unqualified in the repeated test results, the batch of products will be deemed unqualified.

8.7.7 When the powder content of any sample fails to meet the standard, it shall take double quantity of samples from the batch of products (including the batch of products represented by the unqualified sample) for repeated testing. If all repeated test results are qualified, the batch of products will be deemed qualified. If there are still sample fractures that are unqualified in the repeated test results, the batch of products will be deemed unqualified.

8.7.8 When the appearance quality of any sample is unqualified, it shall take double quantity of samples from the batch of products (including the batch of products represented by the unqualified sample) for repeated testing. If all repeated test results are qualified, the batch of products will be deemed qualified. If there are still samples that are unqualified in the repeated test results, the batch of products will be deemed unqualified. Upon agreement between the supplier and the buyer, the supplier is allowed to inspect piece by piece and deliver the qualified products.

9 Signs, packaging, transportation, storage, quality certificate

9.1 Signs

9.1.1 Product signs

Print the following content identification on the products that have passed the inspection (OR affix a label containing the following content):

- a) Supplier's name and address;
- b) Product name;
- c) The inspection seal of the supplier's quality inspection department (or the signature or seal of the quality inspection personnel);

Appendix B

(Normative)

Determination of iron content in iron additives

B.1 Summary of methods

In an acidic solution, divalent iron ions are directly titrated with a standard solution of potassium dichromate. The excess potassium dichromate oxidizes the sodium diphenylamine sulfonate indicator to purple, which is the end point. This method is used to measure the iron content. The measuring range is 72% ~ 98%.

B.2 Reagents

Warning - Personnel using this document shall have practical experience in regular laboratory work. This document does not identify all possible safety issues. Users are responsible for taking appropriate safety and health measures and ensuring compliance with the conditions stipulated in relevant national regulations.

Unless otherwise stated, only reagents confirmed to be of analytical grade and grade 3 water complying with GB/T 6682 are used in the analysis.

B.2.1 Hydrochloric acid (1 + 1).

B.2.2 Mixed acid of sulfur and phosphorus: Slowly add 100 mL of phosphoric acid ($\rho = 1.69 \text{ g/mL}$) to 300 mL of sulfuric acid (1 + 3); mix well.

B.2.3 Tin dichloride solution (40 g/L): Weigh 4.0 g of tin dichloride. Add 40 mL of hydrochloric acid (B.2.1). Add water to dilute it to 100 mL. Heat until the solution is clear. Cool and transfer it into a reagent bottle. Put in two tin pellets.

B.2.4 Mercury dichloride (saturated solution): Weigh about 45 g of mercury dichloride. Add 100 mL of warm water (about 60 °C ~ 70 °C), to dissolve it. Cool it slightly. Transfer it into a reagent bottle.

B.2.5 Sodium diphenylamine sulfonate solution (4 g/L).

B.2.6 Standard titration solution of potassium dichromate (0.003 mol/L): Weigh 0.8826 g of standard potassium dichromate (preliminarily dry it at 160 °C for 2 hours; place it in a desiccator to cool to room temperature) into a beaker. Add an appropriate amount of water, to dissolve it. Transfer it into a 1 L measuring flask. Dilute to the mark. Mix well.

B.3 Specimen

After crushing the sample, use the quartering method to reduce it to the required amount.

B.4 Measurement

B.4.1 Sample

Weigh 0.50 g of specimen (B.3), accurate to 0.0001 g, recorded as m_{Fe} .

B.4.2 Parallel test

Make two measurements independently. Take the average value.

B.4.3 Test procedures

B.4.3.1 Place the sample in a 250 mL beaker. Add 30 mL of hydrochloric acid (B.2.1). Heat until complete dissolution. After cooling, filter it into a 250 mL volumetric flask. Dilute to volume. Mix well.

B.4.3.2 Pipette 10.00 mL of the above solution (B.4.3.1) into a 400 mL conical beaker. Add another 15 mL of hydrochloric acid (B.2.1). Heat to nearly boiling. Dropwise add tin dichloride solution (B.2.3), until the yellow color fades away. Add 1 more drop. Cool it down. Add 5 mL of mercuric dichloride solution (B.2.4). Shake the test solution, until filamentous precipitates appear. Let it stand for 3 min ~ 4 min. Add 100 mL of water, 18 mL of thiophosphorus mixed acid (B.2.2), 3 drops of sodium diphenylaminesulfonate solution (B.2.5). Use potassium dichromate standard titration solution (B.2.6) to titrate, until it becomes purple, which is used as the end point.

B.5 Test data processing

B.5.1 The iron content is recorded as the mass fraction of iron w_{Fe} , which is calculated according to formula (B.1):

$$w_{\text{Fe}} = \frac{c_{\text{K}_2\text{Cr}_2\text{O}_7} V_{\text{K}_2\text{Cr}_2\text{O}_7} M_{\text{Fe}} \times 6 \times 25}{1000 m_{\text{Fe}}} \times 100\% \quad \dots\dots\dots (\text{B.1})$$

Where:

$c_{\text{K}_2\text{Cr}_2\text{O}_7}$ - The concentration of potassium dichromate standard titration solution, in moles per liter (mol/L);

$V_{\text{K}_2\text{Cr}_2\text{O}_7}$ - The volume of potassium dichromate standard solution as consumed in titration, in milliliters (mL);

M_{Fe} - The molar mass of iron, in grams per mole (g/mol) ($M_{\text{Fe}} = 55.85$);

6 - 1 mol of potassium dichromate can reduce 6 mol of ferrous sulfate;

Appendix C

(Normative)

Determination of manganese content in manganese additives

C.1 Summary of methods

Use sulfuric acid and hydrogen peroxide solution to dissolve the specimen. Hydroxylamine hydrochloride keeps manganese divalent, whilst sodium fluoride masks the elements such as aluminum and iron. In an ammoniacal solution, use EDTA standard solution to complex with manganese; use chrome black T as an indicator; use zinc standard solution to titrate the excess EDTA standard solution, until the solution turns purple, which is taken as the end point. This method is used to determine the manganese content. The measuring range is 72% ~ 96%.

C.2 Reagents

Warning - Personnel using this document shall have practical experience in regular laboratory work. This document does not identify all possible safety issues. Users are responsible for taking appropriate safety and health measures and ensuring compliance with the conditions stipulated in relevant national regulations.

Unless otherwise stated, only reagents confirmed to be of analytical grade and grade 3 water complying with GB/T 6682 shall be used in the analysis.

C.2.1 Hydrogen peroxide (1.10 g/mL).

C.2.2 Sulfuric acid (1 + 4).

C.2.3 Hydroxylamine hydrochloride solution (100 g/L).

C.2.4 Sodium fluoride (saturated solution): Weigh about 55 g of sodium fluoride. Add 1000 mL of water. Heat and boil for 30 minutes, until it is completely dissolved. After cooling, transfer the upper solution into the reagent bottle.

C.2.5 Zinc standard solution (0.025 mol/L): Weigh 1.6348 g of metallic zinc ($w_{\text{Zn}} \geq 99.99\%$); add 15 mL of concentrated hydrochloric acid ($\rho = 1.19 \text{ g/mL}$); dissolve and transfer it into a 1 L volumetric flask; dilute to volume; mix well.

C.2.6 EDTA standard solution ($c_{\text{EDTA}} \approx 0.05 \text{ mol/L}$):

- Preparation: Weigh 18.6120 g of EDTA into a 1 L beaker. Add an appropriate amount of water. Heat to dissolve. After cooling, use absorbent cotton to filter it

Appendix D

(Normative)

Determination of copper content in copper type additives

D.1 Summary of methods

In a weakly acidic solution, sodium pyrophosphate is used to mask iron and hydroxylamine hydrochloride is used to eliminate nitrogen oxides. Potassium iodide is added; copper ions react with iodide ions to precipitate an equivalent amount of iodine. Use starch as an indicator; use sodium thiosulfate standard solution to titrate the precipitated iodine, to determine the copper content. The measuring range is 72% ~ 93%.

D.2 Reagents

Unless otherwise stated, only reagents confirmed to be of analytical grade and grade-3 water complying with GB/T 6682 are used in the analysis.

D.2.1 Ammonium fluoride.

D.2.2 Sodium pyrophosphate (solid).

D.2.3 Nitric acid (1 + 1).

D.2.4 Potassium iodide solution (150 g/L).

D.2.5 Hydroxylamine hydrochloride solution (100 g/L).

D.2.6 Potassium thiocyanate solution (100 g/L).

D.2.7 Sodium thiosulfate stock solution (1 mol/L): Weigh 130 g of sodium thiosulfate pentahydrate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$) into a conical flask. Add an appropriate amount of newly boiled and cooled distilled water to dissolve it. Add 5 g of sodium carbonate. After it is completely dissolved, filter it through absorbent cotton into a 500 mL volumetric flask. Use the newly boiled and cooled distilled water to dilute it to 500 mL. Mix it evenly. Put it in a brown reagent bottle. Leave it for two weeks before use.

D.2.8 Starch solution (10 g/L). Weigh 1.00 g of starch. Add a little water to soak it evenly. Add 100 mL of boiling water. Heat until the solution is clear.

D.2.9 Copper standard solution (0.02 mol/L): Weigh 1.2709 g of metallic copper ($w_{\text{Cu}} \geq 99.99\%$). Add 40 mL of nitric acid (D.2.3). Boil to remove nitrogen oxides. Cool to room temperature and transfer to 1 L volumetric flask. Use water to dilute it to the mark. Mix well (or dilute the copper standard storage solution to a certain mark).

Appendix E

(Normative)

Determination of chromium content in chromium type additives

E.1 Atomic absorption spectrometry

E.1.1 Summary of methods

In an acidic solution, it is sprayed directly and is excited by air-acetylene combustion, to produce a ground state atomic vapor layer, which absorbs the characteristic radiation wave emitted by the chromium hollow cathode lamp. The intensity of absorption is proportional to the amount of chromium, by which the chromium content is determined. The measuring range is 72% ~ 83%. This method is an arbitration method.

E.1.2 Reagents

Unless otherwise stated, only reagents confirmed to be of analytical grade and grade-3 water complying with GB/T 6682 are used in the analysis.

E.1.2.1 Hydrogen peroxide (1.10 g/mL).

E.1.2.2 Hydrochloric acid (1 + 1).

E.1.2.3 Chromium standard solution (1.0 mg/mL): Weigh 1.00 g of metal chromium powder ($w_{Cr} \geq 99.99\%$). Add 20 mL of hydrochloric acid (E.1.2.2). Heat until complete dissolution. Cool it. Transfer it into a 1000 mL volumetric flask. Use water to dilute it to the mark. Mix well. (or chromium standard storage solution)

E.1.3 Instruments

Atomic absorption spectrometer, with chromium hollow cathode lamp.

E.1.4 Specimen

After crushing the sample, use the quartering method to reduce it to the required amount.

E.1.5 Determination

E.1.5.1 Sample

Weigh 0.50 g of specimen (E.1.4). The specimen is accurate to 0.0001 g, recorded as m_{Cr} .

E.1.5.2 Parallel test

E.2 Ferrous ammonium sulfate titration method

E.2.1 Summary of methods

In an acidic solution, use silver nitrate as a catalyst; use ammonium persulfate to oxidize chromium (III) to chromium (VI). Use hydrochloric acid to reduce the simultaneously oxidized manganese (VII). Use anthranilic acid as an indicator. Use ferrous ammonium sulfate standard solution to make titration. The measuring range is 72.0% ~ 83.0%,

E.2.2 Reagents

Warning - Personnel using this document shall have practical experience in regular laboratory work. This document does not identify all possible safety issues. Users are responsible for taking appropriate safety and health measures and ensuring compliance with the conditions stipulated in relevant national regulations.

Unless otherwise stated, only reagents confirmed to be of analytical grade and grade-3 water complying with GB/T 6682 are used in the analysis.

E.2.2.1 Phosphoric acid ($\rho = 1.69 \text{ g/mL}$)

E.2.2.2 Nitric acid ($\rho = 1.42 \text{ g/mL}$).

E.2.2.3 Nitric acid (1 + 1).

E.2.2.4 Hydrochloric acid (1 + 3).

E.2.2.5 Sulfuric acid (1 + 4).

E.2.2.6 Sulfuric acid-phosphoric acid mixed acid: In 300 mL of water, slowly add 100 mL of phosphoric acid (E.2.2.1) and 200 mL of sulfuric acid ($\rho = 1.84 \text{ g/mL}$), under constant stirring. Mix well.

E.2.2.7 Silver nitrate solution (10 g/L): Store in brown bottle.

E.2.2.8 Manganese sulfate solution (10 g/L).

E.2.2.9 Ammonium persulfate solution (200 g/L): Prepare it before use.

E.2.2.10 Potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) standard solution (0.003 mol/L): Weigh 0.8826 g of the standard potassium dichromate (preliminarily dried at 160 °C for 2 h and placed in a desiccator to cool to room temperature). Place it in the beaker. Add an appropriate amount of water to dissolve it. Transfer it into a 1000 mL measuring flask. Use water to dilute it to the mark. Mix well.

E.2.2.11 Ferrous ammonium sulfate standard solution ($\text{C}_{(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2} \approx 0.02 \text{ mol/L}$)

titanium.

H.2.19 A80Cu mixed standard solution for aluminum type copper additive: pipette 7.5 mL of silicon standard storage solution (H.2.7), 7.5 mL of iron standard storage solution (H.2.12), 5 mL of lead standard storage solution (H.2.13), 5 mL of manganese standard storage solution (H.2.9), 5 mL of chromium standard storage solution (H.2.10), 5 mL of nickel standard storage solution (H.2.11), 5 mL of titanium standard storage solution (H.2.15) in a 100 mL volumetric flask. Add 10 mL of nitric acid (H.2.2). Use water to dilute it to the mark. Mix well. 1 mL of this solution contains 75 µg of silicon, 75 µg of metallic iron, 50 µg of metallic lead, 50 µg of metallic manganese, 50 µg of metallic chromium, 50 µg of metallic nickel, 50 µg of metallic titanium.

H.2.20 A80Cr mixed standard solution for aluminum type chromium additive: Pipette 20 mL of silicon standard storage solution (H.2.7), 35 mL of iron standard storage solution (H.2.12), 7.5 mL of manganese standard storage solution (H.2.9), 5 mL of copper standard storage solution (H.2.8), 5 mL of nickel standard storage solution (H.2.11), 5 mL of titanium standard storage solution (H.2.15) in a 100 mL volumetric flask. Add 10 mL of hydrochloric acid (H.2.1). Use water to dilute it to the mark. Mix well. 1 mL of this solution contains 200 µg of silicon, 350 µg of metallic iron, 75 µg of metallic manganese, 50 µg of metallic copper, 50 µg of metallic nickel, 50 µg of metallic titanium.

H.2.21 A80Ti mixed standard solution for aluminum type titanium additive: Pipette 20 mL of silicon standard storage solution (H.2.7), 15 mL of magnesium standard storage solution (H.2.14), 50 mL of iron standard storage solution (H.2.12), 7.5 mL of copper standard storage solution (H.2.8), 7.5 mL of nickel standard storage solution (H.2.11), 7.5 mL of manganese standard storage solution (H.2.9), 7.5 mL of chromium standard storage solution (H.2.10), in 100 mL volumetric flask. Mix well. 1 mL of this solution contains 200 µg of silicon, 150 µg of metallic magnesium, 500 µg of metallic iron, 75 µg of metallic copper, 75 µg of metallic nickel, 75 µg of metallic manganese, 75 µg of metallic chromium.

H.2.22 A80Ni mixed standard solution for aluminum type nickel additive: Pipette 7.5 mL of iron standard storage solution (H.2.12), 7.5 mL of cobalt standard storage solution (H.2.16), 5 mL of silicon standard storage solution (H.2.7), 5 mL of copper standard storage solution (H.2.8), 5 mL of manganese standard storage solution (H.2.9), 5 mL of chromium standard storage solution (H.2.10), 5 mL of titanium standard storage solution (H.2.15), in a 100 mL volumetric flask. Add 10 mL of mixed acid 2 (H.2.4). Use water to dilute it to the mark. Mix well. 1 mL of this solution contains 75 µg of metallic iron, 75 µg of metallic cobalt, 50 µg of silicon, 50 µg of metallic copper, 50 µg of metallic manganese, 50 µg of metallic chromium, 50 µg of metallic titanium.

H.2.23 Metallic aluminum ($w_{\text{Al}} \geq 99.99\%$).

H.2.24 Metallic iron ($w_{\text{Fe}} \geq 99.99\%$).

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