# GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

ICS 77.120.10

H 12

# GB/T 20975.17-2020

Replacing GB/T 20975.17-2008

# Methods for chemical analysis of aluminium and aluminium alloys - Part 17: Determination of strontium content

铝及铝合金化学分析方法

第 17 部分：锶含量的测定

**Issued on: September 29, 2020** **Implemented on: August 01, 2021**

**Issued by: State Administration for Market Regulation;**
**Standardization Administration of the People's Republic of China.**

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# Methods for chemical analysis of aluminium and aluminium alloys - Part 17: Determination of strontium content

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

## 1 Scope

This Part of GB/T 20975 specifies the determination of strontium content in aluminum and aluminum alloys by flame atomic absorption spectrometry and $Na_2EDTA$ titration.

This Part applies to the arbitration determination of strontium content in aluminum and aluminum alloys. The determination range of flame atomic absorption spectrometry: 0.020 % ~ 12.00 %; the determination range of $Na_2EDTA$ titration: 3.00 % ~ 22.00 %.

NOTE: When the strontium mass fraction is > 3.00 % ~ 12.00 %, $Na_2EDTA$ titration is used as the arbitration method.

## 2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 8005.2 Terms of aluminium and aluminium alloys - Part 2: Chemical analysis

GB/T 8170-2008 Rules of rounding off for numerical values & expression and judgement of limiting values

## 3 Terms and definitions

For the purpose of this document, the terms and definitions defined in GB/T 8005.2 apply.

## 4 Flame atomic absorption spectrometry

### 4.1 Method summary

Dissolve the sample with hydrochloric acid and hydrogen peroxide, measure the absorbance of strontium using air-acetylene rich flame at the wavelength of 460.7 nm of a flame atomic absorption spectrometer, to determine the strontium content.

**4.2 Reagents**

Unless otherwise stated, only reagents and laboratory grade 2 water confirmed to be of analytical grade are used in the analysis.

**4.2.1** Pure aluminum ($w_{Al} \geq 99.99$ %, $w_{Sr} \leq 0.001$ %).

**4.2.2** Hydrogen peroxide ($\rho$ = 1.10 g/mL).

**4.2.3** Hydrofluoric acid ($\rho$ = 1.14 g/mL).

**4.2.4** Nitric acid ($\rho$ = 1.42 g/mL).

**4.2.5** Hydrochloric acid (1 + 1).

**4.2.6** Lanthanum chloride solution (200 g/L): WEIGH 100 g of lanthanum chloride ($LaCl_3 \cdot 6H_2O$), DILUTE to a 500 mL volumetric flask with water.

**4.2.7** Aluminum solution A (20 mg/mL): WEIGH 20.00 g of pickled pure aluminum (4.2.1) and PLACE it in a 1000 mL beaker, COVER it with a watch glass, and ADD a total of 200 mL of hydrochloric acid (4.2.5) in portions. After the violent reaction stops, ADD a few drops of hydrogen peroxide (4.2.2), HEAT slowly until completely dissolved, and COOL down. TRANSFER the solution into a 1000 mL volumetric flask, DILUTE to the mark with water, and MIX well.

**4.2.8** Aluminum solution B (2 mg/mL): PIPETTE 50 mL of aluminum solution (4.2.7) into a 500 mL volumetric flask, DILUTE to the mark with water, and MIX well.

**4.2.9** Strontium standard storage solution: WEIGH 1.6850 g of strontium carbonate ($w_{SrCO3} \geq 99.99$ %) and DISSOLVE it in an appropriate amount of hydrochloric acid (4.2.5), HEAT to boil, COOL down, DILUTE to 1000 mL with water, and MIX well. 1 mL of this solution contains 1.0 mg of strontium.

**4.2.10** Strontium standard solution A: PIPETTE 50.00 mL of strontium standard storage solution (4.2.9) into a 200 mL volumetric flask, DILUTE to the mark with water, and MIX well. 1 mL of this solution contains 0.25 mg of strontium.

**4.2.11** Strontium standard solution B: PIPETTE 50.00 mL of strontium standard storage solution (4.2.9) into a 500 mL volumetric flask, DILUTE to the mark with water, and MIX well. 1 mL of this solution contains 0.1 mg of strontium.

**4.3 Instruments and equipment**

Atomic absorption spectrometer with strontium hollow cathode lamp. The instrument shall meet the following conditions:

- Characteristic concentration: in a solution consistent with the matrix of the tested sample solution, the characteristic concentration of strontium shall not be greater than 0.25 μg/mL;

- Precision: use the highest-concentration standard solution to measure the absorbance 10 times, the standard deviation shall not exceed 1.0 % of the average absorbance; use the lowest-concentration standard solution (not a "zero" concentration solution) to measure the absorbance 10 times, the standard deviation shall not exceed 0.5 % of the average absorbance of the highest-concentration standard solution;

- Working curve linearity: divide the working curve into five equal segments according to concentration. The ratio of the absorbance difference of the highest segment to the absorbance difference of the lowest segment shall not be less than 0.7.

**4.4 Sample**

Process the sample into chips with a thickness of no more than 1 mm.

**4.5 Analysis steps**

**4.5.1 Sample**

Weigh the sample (4.4) with a mass ($m_0$) of 0.50 g, accurate to 0.0001 g.

**4.5.2 Parallel test**

Carry out two tests in parallel and take the average value.

**4.5.3 Blank test**

Weigh pure aluminum (4.2.1) according to Table 1 to replace the sample (4.5.1), and carry out a blank test along with the sample.

**4.5.4 Determination**

**4.5.4.1** PLACE the sample (4.5.1) into a 250 mL beaker, ADD 15 mL of hydrochloric acid (4.2.5), after the violent reaction stops, ADD 3 ~ 5 drops of hydrogen peroxide (4.2.2), BOIL and STEAM until salt precipitates, COOL down slightly, ADD 3 mL of hydrochloric acid (4.2.5), HEAT until the salts are dissolved, then REMOVE the beaker and COOL down.

**4.5.4.2** If there is insoluble matter, filter and wash. PLACE the residue together with the filter paper in a platinum crucible, ASH it (do not burn the filter paper). BURN at

**5.2.3** Ammonia water ($\rho$ = 0.89 g/mL).

**5.2.4** Hydrochloric acid (1 + 1).

**5.2.5** Hexamethylenetetramine solution (250 g/L).

**5.2.6** Copper reagent solution (50 g/L): WEIGH 5 g of copper reagent (sodium diethyl aminothiocarbamate) and DISSOLVE it in 25 mL of absolute ethanol (5.2.2), DILUTE to 100 mL with water. (Prepare before use.)

**5.2.7** Ammonia-ammonium chloride buffer solution (pH ≈ 10): WEIGH 54 g of ammonium chloride and DISSOLVE it in 200 mL of water, ADD 350 mL of ammonia water (5.2.3), and DILUTE to 1000 mL.

**5.2.8** Zinc standard solution ($c'$ = 0.02 mol/L): WEIGH 1.3076 g of zinc ($w_{Zn} \geq 99.9$ %) and PLACE it in a 300 mL beaker, ADD 20 mL of hydrochloric acid (5.2.4), HEAT to dissolve completely and then EVAPORATE to 10 mL, COOL down, TRANSFER to a 1000 mL volumetric flask, DILUTE to the mark with water, and MIX well.

**5.2.9** Magnesium standard solution (0.02 mol/L): WEIGH 0.4861 g of metallic magnesium ($w_{Mg} \geq 99.9$ %) and PLACE it in a 300 mL beaker, ADD 30 mL of hydrochloric acid (5.2.4), HEAT to dissolve completely, COOL down, TRANSFER into a 1000 mL volumetric flask, DILUTE to the mark with water, and MIX well.

**5.2.10** $Na_2EDTA$ standard titration solution ($c_1 \approx 0.02$ mol/L):

- Preparation: WEIGH 7.4 g of disodium ethylenediaminetetraacetate ($C_{10}H_{14}N_2O_8Na_2 \cdot 2H_2O$) and DISSOLVE it in 200 mL of hot water, COOL down, TRANSFER into a 1000 mL volumetric flask with water, and MIX well.

- Calibration: PIPETTE 20.00 mL ($V'$) of zinc standard solution (5.2.8) into a 250 mL Erlenmeyer flask, ADD 50 mL of water and 15 mL of ammonia-ammonium chloride buffer solution (5.2.7), ADD 4 ~ 5 drops of chrome black T indicator (5.2.11), TITRATE the solution with $Na_2EDTA$ standard solution (5.2.10) until the solution turns pure blue, which is the end point, and RECORD the volume ($V$) of the $Na_2EDTA$ standard titration solution (5.2.10) consumed. CALIBRATE three copies in parallel, and the difference in volume between the two calibration copies shall not be greater than 0.10 mL.

- Calculation: CALCULATE the concentration of $Na_2EDTA$ standard solution according to formula (2):

$$c_1 = \frac{c'V'}{V} \qquad \cdots\cdots (2)$$

where:

$c'$ - the concentration of zinc standard solution, in moles per liter (mol/L);

$V'$ - the volume of the zinc standard solution pipetted, in milliliters (mL);

$V$ - the volume of the $Na_2EDTA$ standard titration solution consumed, in milliliters (mL).

The calculation results retain four significant figures, and rounding off of numerical values is performed in accordance with 3.2 and 3.3 of GB/T 8170-2008.

**5.2.11** Chrome black T indicator: WEIGH 0.2 g of chrome black T and DISSOLVE it in 10 mL of ammonia water (5.2.3) and 40 mL of absolute ethanol (5.2.2). After complete dissolution, store it in a brown dropper bottle.

**5.3 Sample**

Process the sample into chips with a thickness of no more than 1 mm.

**5.4 Analysis steps**

**5.4.1 Sample**

Weigh the sample (5.3) with a mass ($m$) of 0.20 g, accurate to 0.0001 g.

**5.4.2 Parallel test**

Carry out two tests in parallel and take the average value.

**5.4.3 Determination**

**5.4.3.1** PLACE the sample (5.4.1) into a 250 mL beaker, ADD 6 mL of hydrochloric acid (5.2.4), ADD a few drops of hydrogen peroxide (5.2.1), HEAT, after the sample is completely dissolved, COOL down.

**5.4.3.2** DILUTE to about 20 mL with water, NEUTRALIZE with ammonia water (5.2.3) until the pH of the solution is about 6 (precision pH test paper), ADD 15 mL of hexamethylenetetramine solution (5.2.5), CONTROL the pH of the solution to 6. 5 ~ 7.5, HEAT and BOIL for 1 min, then COOL down to room temperature. ADD 10 mL of copper reagent solution (5.2.6) while stirring, TRANSFER it to a 200 mL volumetric flask ($V_5$), DILUTE to the mark with water, and MIX well. LET STAND for 30 min and carry out dry filtration.

**5.4.3.3** PIPETTE 50.00 mL of filtrate ($V_6$) into a 250 mL Erlenmeyer flask, ADD water to approximately 100 mL, ADD 10 mL of ammonia-ammonium chloride buffer solution (5.2.7), ADD 10.00 mL ($V_0$) of magnesium standard solution (5.2.9), ADD 3 ~ 5 drops of chrome black T indicator (5.2.11), TITRATE with $Na_2EDTA$ standard titration solution (5.2.10) until the color change nears the end point, ADD 7 mL to 8 mL of absolute ethanol (5.2.2), continue to TITRATE until the solution turns pure blue, which is the end point. RECORD the volume of $Na_2EDTA$ standard solution consumed ($V_4$).

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