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**NONFERROUS METALS INDUSTRY STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

YS/T 1149.1-2016

**Methods for chemical analysis of zinc concentrate roasting -
Part 1: Determination of zinc content - Na₂EDTA titration
method**

锌精矿焙砂化学分析方法 第1部分：锌量的测定 Na₂EDTA滴定法
(English Translation)

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1 Scope

This Part of YS/T 1149 specifies the method for the determination of zinc content, in zinc concentrate roasting.

This Part applies to the determination of zinc content in zinc concentrate roasting. The measurement range is 30.00% ~ 70.00%.

2 Normative references

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) is applicable to this standard.

GB/T 8151.8 Methods for chemical analysis of zinc concentrates - Part 8:

Determination of cadmium content - The flame atomic absorption spectrometric method

3 Method summary

The sample is dissolved by hydrochloric acid, nitric acid, sulfuric acid; lead in the sulfuric acid medium produces lead sulfate precipitation; it is filtered and separated from coexisting elements; the filtrate is precipitated by ammonia water, to separate coexisting elements, such as iron and manganese. In the filtrate, thiourea is added to mask copper; ammonium fluoride is added to eliminate the interference of a small amount of elements, such as iron and aluminum. In acetic acid-sodium acetate buffer solution of pH5 ~ pH6, use xylenol

orange as indicator; use Na₂EDTA standard titration solution to titrate it, until the solution changes from purple red to bright yellow, which is used as the end point. Calculate the combined amount of zinc and cadmium, according to the volume of the Na₂EDTA standard titration solution consumed. After deducting the amount of cadmium, it is the amount of zinc.

4 Reagents

Unless otherwise specified, the reagents used in this Part are analytical reagents AND distilled water or deionized water or water of equivalent purity, that meet national standards or industry standards.

4.1

Zinc oxide (reference material).

4.2

Ammonium chloride.

4.3

Ammonium fluoride.

4.4

Ascorbic acid.

4.5

Anhydrous sodium acetate.

4.6

Hydrochloric acid (rho1.19 g/mL).

4.7

Sulfuric acid (rho1.84 g/mL).

4.8

Nitric acid (rho1.42 g/mL).

4.9

Ammonia (rho0.90 g/mL).

4.10

Acetic acid (rho1.049 g/mL).

4.11

Hydrochloric acid (1 + 1).

4.12

Sulfuric acid (1 + 1).

4.13

Sulfuric acid (1 + 9).

4.14

Sulfuric acid (2 + 98).

4.15

Ammonia (1 + 1).

4.16

Washing solution

Dissolve 2 g of ammonium chloride (4.2) in 100 mL of water. Add 1 mL of ammonia water (4.9). Mix well.

4.17

Ammonium persulfate solution (200 g/L)

It is prepared on the day when it is used.

4.18

Thiourea saturated solution.

4.19

Ferric sulfate solution (10 g/L)

Weigh 10 g of ferric sulfate; dissolve it in 1000 mL of sulfuric acid (4.13).

4.20 Acetic acid-sodium acetate buffer solution (pH 5.5): Weigh 150 g of sodium

Use water to rinse the watch glass and the wall of the beaker. Dilute to about

50 mL. Heat to a slight boil for 10 min. Cool to room temperature. Let it stand for 1 h. Use slow quantitative filter paper to filter. Use a beaker to collect the filtrate. Use sulfuric acid (4.14) to wash the beaker and the precipitate 5 times each. Use water to rinse the beaker and the precipitate once each. Keep the filtrate. Discard the precipitate.

6.4.4 Add 3 g ~ 5 g of ammonium chloride (4.2) and 5 mL of ammonium persulfate solution (4.17), to the solution of 6.4.2 or the filtrate of 6.4.3. Use ammonia water (4.9) to neutralize it, until the precipitation is complete. Then add another 10 mL. Heat for a slight boiling, for 1 min ~ 2 min. While it is hot, use fast qualitative filter paper to filter it. Use hot washing solution (4.16) to wash the beaker and precipitate 2 ~ 3 times each. Keep the filtrate.

6.4.5 Use hot hydrochloric acid (4.11) to dissolve the precipitation in the original

beaker. Use hot water to wash the filter paper clean. Add 5 mL of ammonium persulfate solution (4.17). Use ammonia water (4.9) to neutralize it, until the precipitation is complete. Add another 10 mL. Heat to slight boiling for 1 min ~

2 min. Remove it. Filter it through the original filter paper in the beaker of the retained solution in 6.4.4. Use hot washing solution (4.16), to wash the beaker and the precipitate for 3 ~ 4 times each.

6.4.6 Boil and concentrate the filtrate (6.4.5) to a volume of about 100 mL.

Completely destroy the excess ammonium persulfate. Remove it for cooling.

6.4.7 Add 0.1 g of ascorbic acid (4.4), 0.2 g of ammonium fluoride (4.3), 5 mL of thiourea saturated solution (4.18). Shake well for each reagent added. Add 1 drop of methyl orange indicator (4.22). Use ammonia water (4.15) and hydrochloric acid (4.11), to adjust the solution, until it just turns red. Add 1 ~ 2 drops of xylenol orange indicator (4.23). Add 20 mL of acetic acid-sodium acetate buffer solution (4.20). Use Na₂EDTA standard titration solution (4.21) to titrate it, until the solution just changes from purple to bright yellow, which is taken as the end point.

7 Calculation of analysis results

The amount of zinc is calculated in terms of the mass fraction of zinc, w_{Zn} ; the value is expressed in %; it is calculated according to formula (2):

Where:

c - The concentration of Na₂EDTA standard titration solution, in moles per milliliter (mol/L);

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

V_3 - The volume of Na₂EDTA standard titration solution, which is consumed by the test solution, in milliliters (mL);

V_2 - The volume of Na₂EDTA standard titration solution, which is consumed by blank test solution, in milliliter (mL);

m_2 - The mass of the sample, in grams (g);

w_{Cd} - The mass fraction of cadmium, which is measured by GB/T 8151.8;

0.5816 - The coefficient of converting the amount of cadmium to the amount of zinc.

The calculation result is expressed to two decimal places.