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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 8151.1-2012

**Methods for chemical analysis of zinc concentrates -- Part 1: Determination of
zinc content -- Precipitate separation-Na₂EDTA titrimetric method and extractive
separation -- Na₂EDTA titrimetric method**

(English Translation)

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1 Scope

This Part of GB/T 8151 specifies the determination of zinc content in zinc concentrates.

This Part is applicable to the determination of zinc content in zinc concentrates. The

Method-1 determination range: 30.00% ~ 60.00%; while the Method-2 determination range: 11.00%~62.00%.

2 Method 1 -- Precipitate Separation-Na₂EDTA

Titrimetric Method

2.1 Method summary

The testing material was dissolved by hydrochloric acid, nitric acid and sulfuric acid, and precipitated and separated coexisting elements such as iron, manganese and lead.

A masking agent is added to the filtrate to mask small amounts of interfering elements.

In the acetic acid-sodium acetate buffer solution with pH 5~6, using xylenol orange as an indicator, titrate with Na₂EDTA standard titration solution. The measured result is the total content of zinc and cadmium; and the content of cadmium is deducted, which remains the content of zinc.

2.2 Reagents

Unless otherwise specified, only use reagents and distilled water or deionized water or equivalent purity water confirmed to be analytically pure in the analysis.

2.2.1

Metal zinc (wZn ≥ 99.99%) has no oxidation on the surface before use. Zinc is washed by hydrochloric acid (1+9) for 1 min; then rinsed by water and acetone; and dried in an oven at 50°C.

2.2.2

Ammonium chloride.

2.4.3 Blank test

Do the blank test with the testing material.

2.4.4 Determination

2.4.4.1

Place the testing material (2.4.1) in a 400mL beaker; add a small amount of water for wetting (if the sample contains a high content of silica, 1g of ammonium fluoride shall be added); add 10mL of hydrochloric acid (2.2.5); cover the watch glass, dissolve at a low temperature to expel the hydrogen sulfide for 5min ~ 10min. Add 5mL of nitric acid (2.2.6) to complete decomposition of the testing material; take off the watch glass.

2.4.4.2

When the lead content is less than 4%, add 5 mL of sulfuric acid (2.2.12); and continue to heat until it is wet and salty [if the testing material contains high content of carbon, it may be taken off when it is steamed to emit white smoke and left to cool.

Add 1mL~2mL of perchloric acid (2.2.8) and continue heating until it is wet and salty], take off and let cool. Add 20mL of sulfuric acid (2.2.13); cover the watch glass; heat to dissolve the salt; cool slightly; rinse the watch glass and the wall of the beaker with water; and dilute the volume to about 60 mL [if the iron content in the solution is low, add iron sulfate stock solution (2.2.21) makes the solution contain about 20mg of iron].

2.4.4.3

When the lead content is greater than 4%, add 5mL of sulfuric acid (2.2.7) to heat it up to thick white smoke appears; and steam to a volume of about 2mL; take off it, and cool. Rinse the watch glass and the wall of the beaker with water; and dilute the volume to about 50mL; heat it to boil for 10min; cool to room temperature; and stand for 1h. Filter with slow quantitative filter paper; take the filtrate with a beaker; wash the beaker and precipitate for 5 times by sulfuric acid (2.2.14), respectively; wash the beaker and precipitate for 1 time by water, respectively; retain the filtrate.

2.4.4.4 Add 3g~5g of ammonium chloride (2.2.2) and 5mL of ammonium persulfate

solution (2.2.17) to 2.4.4.2 or 2.4.4.3; neutralize with ammonia water (2.2.9) until precipitation is complete, and then excess 10mL. Heat to boil slightly for 1min~2min;

filter with fast qualitative filter paper while it is hot; wash the beaker and the precipitate with hot washing liquid for 2 ~ 3 times, respectively; and retain the filtrate.

2.4.4.5 Wash the precipitate by hot washing solution into the beaker of the original precipitate; add hydrochloric acid (2.2.11) to dissolve the precipitate; add 5 mL of ammonium persulfate solution (2.2.17); and neutralize it with ammonia (2.2.9) till the precipitate is complete; and then excess 10 mL. Heat to boil slightly for 1min~2min; take off; filter through the original filter paper in the beaker of the retentate; wash the beaker and the precipitate with hot washing liquid for 3 ~ 4 times, respectively.

2.4.4.6 Boil the filtrate (2.4.4.5) and concentrate to a volume of about 100mL; and completely destroy the excessive ammonium persulfate; take off it and let it cool. use iodide ion to mask cadmium; and titrate zinc with Na₂EDTA solution at pH 5.5.

3.2 Reagents

During the analysis, use the analytical reagents and Class-II water specified in GB/T 6682.

3.2.1

Metal zinc ($w_{Zn} \geq 99.99\%$) has no oxidation on the surface before use. Zinc is cleaned by hydrochloric acid (3.2.2) (1+9) for 1 min; then rinsed by water and acetone; and dried in an oven at 50°C.

3.2.2

Hydrochloric acid ($\rho 201.16\text{g/mL} \sim 1.19\text{g/mL}$).

3.2.3

Liquid bromine.

3.2.4

Nitric acid ($\rho 201.42\text{g/mL}$).

3.2.5

Hydrofluoric acid ($\rho 201.13\text{g/mL} \sim 1.15\text{g/mL}$).

3.2.6

Hydrochloric acid (1+4).

3.2.7

Sulfuric acid (1+1).

3.2.8

Ammonia ($\rho 200.89\text{g/mL}$).

3.2.9

Methyl isobutyl ketone (4-methyl-2-pentanone).

3.2.10

Anhydrous ethanol.

3.2.11

Masking agent

Dissolve 60g of thiourea, 100g of diammonium hydrogen citrate, 200g of ammonium thiocyanate in water and dilute to 1L; and filter if necessary.

3.2.12

Sodium fluoride solution (20g/L).

3.2.13

Thiourea solution (100g/L).

3.2.14

Potassium iodide solution (1000g/L), prepared on the same day.

3.2.15 Buffer solution: dissolve 250g of hexamethylenetetramine (methenamine) in

water; add 60mL of acetic acid ($\rho_{20} 1.05\text{g/mL}$); and then dilute to 1L.

3.2.16 Iron stock solution (45g/L): dissolve 45g of iron nitrate $[\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}]$ in water

and dilute to 1L.

3.2.17 Xylenol orange: evenly mix 1g of xylenol orange and 99g of potassium nitrate crystals, and grind it in a ceramic vessel with a pestle. When the color is consistent, it is considered that the mixture is uniform.

3.2.18 Disodium ethylenediaminetetraacetic acid (Na_2EDTA) standard titration solution

Take 2.50g of specimen, accurate to 0.0001g.

3.4.2 The number of determinations

Perform 2 determinations independently and take the average value.