GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

ICS 71.060.50

G 12

GB/T 10500-2009

Replacing GB/T 10500-2000

Sodium Sulfide for Industrial Use

工业硫化钠

Issued on: June 2, 2009 **Implemented on: February 1, 2010**

Issued by: General Administration of Quality Supervision, Inspection and Quarantine of PRC;
Standardization Administration of PRC.

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Sodium Sulfide for Industrial Use

1 Scope

This standard specifies the requirements, testing method, inspection rules, marking, labeling, packing, transporting and storage of sodium sulfide for industrial use.

This standard is applicable to lump, flaky and grainy sodium sulfide for industrial use, which is used in fields like paper making, dyestuff, mineral processing and printing.

2 Normative References

Provisions of the following documents, through reference in this standard, constitute provisions of this standard. For dated reference, subsequent amendments to (excluding correction to), or revisions of, any of these publications do not apply. However, the parties whose enter into agreement according to these specifications are encouraged to research whether the latest editions of these labels are applied or not. For undated references, the latest editions of the normative documents are applicable to this standard.

GB 190-1990 Labels for packages of dangerous goods

GB/T 191-2008 Packaging - Pictorial marking for handling of goods (ISO 780:1997, MOD)

GB/T 3049-2006 Chemical products for industrial use - General method for determination of iron content – 1,10-Phenanthroline spectrophotometric method (ISO 6685:1982, IDT)

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

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

HG/T 3696.1 Inorganic chemical products Preparations of standard volumetric solutions for chemical analysis

HG/T 3696.2 Inorganic chemical products Preparations of standard solutions for chemical analysis

HG/T 3696.3 Inorganic chemical products Preparations of reagent solutions for chemical analysis reference solution, preparation and product used in the tests shall be prepared according to the requirements of HG/T 3696.1, HG/T 3696.2, and HG/T 3696.3.

6.3 Preparation of test solution

6.3.1 Test solution A: with a weighing bottle of a given mass, weigh 30g full-dissolve test sample solution (7.3.1), accurate to 0.01 g; transfer it into a 1000mL volumetric flask; dilute it to the scale with water free of carbon dioxide, and shake up. This solution is test solution A.

The mass (*m*) of sodium sulfide solid sample is calculated according to formula (1):

$$m = \frac{m_2}{m_1} \times m_3 \quad \cdots\cdots(1)$$

Where,

m_1 -- mass value of full-dissolve test sample solution prepared (g);

m_2 -- mass of dissolve sodium sulfide (g);

m_3 -- mass value of the full-dissolve test sample solution weighed by the weighing bottle (g).

6.3.2 Test solution B: weigh about 10g of solid sample, accurate to 0.01 g, place it in a beaker, add with 100mL of water, and heat to dissolving; transfer the solution to a 1000mL volumetric flask; dilute it to the scale with water free of carbon dioxide, and shake up. This solution is test solution B.

Test solution A or B are used for the determination of the contents of sodium sulfide, sodium sulfite, sodium thiosulfate, iron, and sodium carbonate.

6.4 Determination for sodium sulfide content

6.4.1 Method summary

Add superfluous iodine reference solution in a weak acid solution, back-titrate the solution with sodium thiosulfate titrant, and test the total reduction; subtract with the equivalent amount of content of sodium thiosulfate and sodium sulfite, tested by the iodometry. The difference hereof is the sodium sulfide content.

6.4.2 Reagent

6.4.2.1 Acetic acid solution: 1+10;

6.4.2.2 Iodine standard titrant: $c\left(\frac{1}{2}I_2\right) \approx 0.1$ mol/L;

6.4.2.3 Sodium thiosulfate standard titrant: $c(Na_2S_2O_3) \approx 0.1$ mol/L;

6.4.2.4 Starch indicating solution: 5g/L.

6.4.3 Analysis procedure

Take 20mL of iodine standard titrant with a transfer pipet, and transfer to a 250mL iodine measuring flask, add 25mL of water and 10mL of acetic acid solution; while shaking up, add 10mL of test solution A or B with a transfer pipet; titrate with sodium thiosulfate standard titrant to the solution becoming yellow; add 2mL of starch indicating solution; and continue to titrate till the blue color disappears.

6.4.4 Result calculation

The sodium sulfide content, expressed by the sodium sulfide (Na_2S) mass fraction w_1 in "%", is calculated according to formula (2):

$$w_1 = \frac{[(V_1c_1 - V_2c_2)/10 - V_3c_1/40] \times M/1\ 000}{m/1\ 000} \times 100 \quad \cdots\cdots\cdots\cdots\cdots\cdots (2)$$

Where,

V_1 -- volume value of iodine standard titrant added, (mL);

V_2 -- volume value of sodium thiosulfate standard titrant consumed in titrating, (mL);

V_3 -- volume value of iodine standard titrant consumed in titrating 6.4.3, (mL);

c_1 -- accurate concentration value of iodine standard titrant (mol/L);

c_2 -- accurate concentration value of sodium thiosulfate standard titrant (mol/L);

m -- mass value of solid sample, (g);

M -- mole mass of sodium sulfide($\frac{1}{2}Na_2S$), (g/mol) (M=39.02).

The arithmetic mean value of the parallel determination results is adopted as the final determination result, and the absolute difference hereof shall not be larger than 0.3%.

6.5 Determination of sodium sulfite content

6.5.1 Method summary

Add zinc carbonate suspension in the test solution, and precipitate sulfur ions; take a portion of filtered liquor, and determinate the sodium thiosulfate and sodium sulfite contents of sodium thiosulfate and sodium sulfite by iodometry; subtract the amount equivalent to the sodium thiosulfate content. The difference between these two is the sodium sulfite content.

6.6 Determination of sodium thiosulfate content

6.6.1 Method summary

Add zinc carbonate suspension in a test solution, precipitate sulfur ions, add formaldehyde solution to shield sodium sulfite, and titrate sodium thiosulfate with iodine standard titrant.

6.6.2 Reagent

6.6.2.1 Formaldehyde;

6.6.2.2 Sodium carbonate solution 100g/L;

6.6.2.3 Zinc sulphate ($ZnSO_4 \cdot 7H_2O$) solution: 100g/L;

6.6.2.4 Acetic acid solution: 1+10;

6.6.2.5 Iodine standard titrant: the concentration is one specified in 6.4.2.2;

6.6.2.6 Starch indicating solution: 5g/L.

6.6.3 Analysis procedure

Take 100mL of filtered liquor (6.5.3) after filtration with a transfer pipet, and transfer it in a 500mL conical beaker; add 5mL of formaldehyde solution, 10mL of acetic acid solution and 2mL of starch indicating solution, and titrate with iodine standard titrant till the solution color becomes blue.

6.6.4 Result calculation

The sodium thiosulfate content, expressed by sodium thiosulfate ($Na_2S_2O_3$) mass fraction w_3 in "%", is calculated according to formula (4):

$$w_3 = \frac{V_4 c \times M/1\ 000}{m \times 200 \times 100/(1\ 000 \times 500)} \times 100 \qquad \cdots\cdots (4)$$

Where,

V_4 -- volume value of iodine reference solution consumed in titrating, (mL);

C -- accurate concentration value of iodine standard titrant (mol/L);

m -- mass value of solid sample, (g);

M -- mole mass value of sodium sulphate ($Na_2S_2O_3$) (g/mol) (M=158.1).

The arithmetic mean value of the parallel determination results is adopted as the final determination result, and the absolute difference hereof shall not be larger than 0.1%.

6.7 Determination of iron content

6.7.1 Method summary

Oxidize the sulfide with hydrogen dioxide to sulphate, remove superfluous hydrogen dioxide, and acidulate the solution with hydrochloric acid; reduce the ferric iron with ascorbic acid to ferrous iron; within the pH value of 2~9, ferrous iron and phenanthroline generate red complex, and determinate the absorbance with a spectrophotometer at the absorption wave length maximum of 510nm.

6.7.2 Reagent

6.7.2.1 30% hydrogen dioxide;

6.7.2.2 Anhydrous sodium carbonate solution: 100g/L;

6.7.2.3 Other reagents are same to ones specified in chapter 3 of GB/T 3049-2006.

6.7.3 Instrument and device

Same as chapter 4 of GB/T 3049-2006.

6.7.4 Analysis procedure

6.7.4.1 Drawing of working curve

Draw the working curve according to the requirements of 6.3 in GB/T 3049-2006.

6.7.4.2 Preparation of test solution

Take 10mL of test solution A or B (for low-iron sodium sulfide, weigh 1g of solid test sample, 0.01 g; and add 20mL of water) with a transfer pipet, place it in a 150mL beaker; drop hydrogen dioxide (5 times of the solid sample in addition, and exceed 1.5 mL), shake up, and stay for 5min; add 0.5 mL of anhydrous sodium carbonate solution, and heat and boil for 5min; add 0.5 mL of (1+1) hydrochloric acid solution, continue to heat 1min, cool it, and add a little of water; add the solution to a 100mL volumetric flask (filter the solution in case of deposition); dilute with water to scale, and shake up.

6.7.4.3 Preparation of blank test solution

Expect not adding test sample, all is same to the preparation of the test solutions.

6.7.4.4 Determination

Operate the test solution and the blank test solution from "if necessary, add water to

6.9.2.4 30% hydrogen peroxide solution: 1+ 3;

6.9.2.5 Mixed solvent: ethyl alcohol and acetone (1:1);

6.9.2.6 Potassium hydrate standard titrant: *c* (KOH): about 0.05 mol/L

a) Preparation: weigh and take about 3.5g of potassium hydrate, place it in a beaker, add 150mL of propanetriol, and heat for dissolving; dissolve 0.2 g of thymolphthalein and 0.005 g of thymol blue with a small amount of component solvent, and add it to a beaker; dilute with component solvent to about 1000mL; store it in a brown bottle, and keep it 24h for use.

b) Calibration

Pipette 25mL of anhydrous sodium carbonate reference solution with a transfer pipet, place it in a 250mL round flask, and operated according to the procedure specified in 6.9.4 after connecting the flask to a sodium carbonate analytical equipment system.

The concentration *c* (KOH) of potassium hydrate standard titrant is calculated according to formula (7):

$$c = \frac{c_1 V_1}{V} \qquad \cdots\cdots (7)$$

Where,

c_1 -- accurate concentration value of anhydrous sodium carbonate reference solution (mol/L);

V_1 -- volume value of anhydrous sodium carbonate reference solution pipetted (mL);

V -- volume value of potassium hydrate standard titrant consumed in titrating (mL).

6.9.2.7 Anhydrous sodium carbonate reference solution: $c(\frac{1}{2}Na_2CO_3) \approx 0.04$ mol/L;

Weigh and take 2.1 g of reference anhydrous sodium carbonate ignited at 270℃~ 300℃ to constant mass, accurate to 0.0002 g, place it in a 100mL beaker, dissolve it with water, transfer to a 1000mL measuring flask, dilute with water to the scale, and shake up.

6.9.2.8 Reference solution:

Weigh and take about 0.1g of potassium hydrate, place it in a beaker, add 150mL of propanetriol, and heat for dissolving; with 0.2g of thymolphthalein and 0.005 g of thymol blue dissolved with a small amount of component solvent, transfer to a 1000mL measuring flask, dilute with component solvent to the scale, and shake up.

6.9.2.9 Absorbing solution:

By the analysis procedure specified in 6.9.4, and add reference solution (about 80mL) to 2/3 volume of the absorption pipet (7); add 10mL of anhydrous sodium carbonate reference solution with a transfer pipet in a round flask; titrate it with potassium hydrate reference solution to show same color with the reference solution (the solution shall be replaced after using continuously absorbing solution if the color shows dark) when the solution color shows yellow after the carbon dioxide is absorbed.

6.9.3 Instrument and device

The sodium carbonate content determination device is shown in Figure 1.

6.9.4 Analysis procedure

Install the sodium carbonate determination device as shown in Figure 1; pipette 100mL of test solution A or B with a transfer pipet, place it in a 250mL round flask (1), add 15mL of hydrogen peroxide solution, and connect it with the device; heat it, and open the boiling-water vacuum for degassing; control the raising rate of the air bubbles in the absorption pipet; after a while, add 10mL of sulfuric acid solution in the separatory funnel (3), and transfer the generated carbon dioxide in a absorption pipet (7) contrasting to white background with lighting; titrate it with potassium hydrate standard titrant till it shows same color with the reference solution and no color change within 3 min; if the color of the absorption pipet (8) has obviously different color from the reference solution, it means carbon dioxide has not be absorbed by the solution in the absorption pipet (7) fully, and the determination shall be re-conducted.

6.9.5 Result calculation

The sodium carbonate content, based on sodium carbonate (Na_2CO_3) mass fraction w_7, expressed in percentage, is calculated according to formula (8):

$$w_7 = \frac{cV \times M/1\ 000}{m \times 100/1\ 000} \times 100 \qquad \cdots\cdots (8)$$

Where,

c -- accurate concentration value of potassium hydrate standard titrant (mol/L);

V -- volume value of potassium hydrate standard titrant consumed in titrating (mL);

M -- mole mass value of sodium carbonate($\frac{1}{2}Na_2CO_3$), (g/ mol)(M=53.0);

7 Testing Rules

7.1 All items specified by this standard are delivery inspection items.

7.2 The products of same material and basically same production condition produced continuously or by same shift shall be regarded as a lot, and the mass of the products in a lot shall not exceed 60t.

7.3 The sampling method, different by solid sample and liquid sample, is conducted according to the following procedures:

7.3.1 For lump product in drum, randomly select a drum from each lot, cut open the drum shell, take 100g of sample respectively from upper, middle and lower position, weigh the mass of sodium sulfide, and add water for dissolving; heat for accelerating the dissolution; continue to add water after complete dissolution, prepare the solution with the mass fraction of 20%, and weigh the mass; mix uniformly and stir continually, and take out 30g of the liquid sample for test use on the same day.

For flaky and grainy sodium sulfide in bags, 3 bags (50kg) or 6 bags (25kg) shall be selected randomly from a lot, and sampled by inserting into 20cm under the sample surface. Not less than 50g of sample is selected from each bag, and dissolved by the above method.

7.3.2 The manufacturer may sample representative liquid sample in product packaging procedure, and made as solid sample after cooling; prepare the test solution as required in 6.3.2. If there is dispute about quality between the supply and demand parties, the inspection result of sampling method specified in 7.3.1 shall prevail.

7.4 Sodium sulfide for industrial use shall be inspected by the quality supervision & inspection department of the manufacturer according to the requirements of this standard. The manufacturer shall ensure the products of each lot meet the requirements of this standard. In case one in the results cannot meet the requirement of this standard, doubled packages of samples shall be sampled and inspected; in case one in re-inspection results cannot meet the requirement of this standard, the product of whole lot shall be de-rated or rejected.

7.5 In case one in the results cannot meet the requirement of this standard, doubled packages of samples shall be inspected; in case one in re-inspection results cannot meet the requirement of this standard, the product of whole lot shall be rejected.

7.6 The rounding value comparison method specified in GB/T 8170 is adopted to judge whether the test results meet the standard.

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

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