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# GAIN Report

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Voluntary - Public

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## **China - Peoples Republic of**

**Post:** Beijing

### **National Food Safety Standard-Sodium Hypochlorite**

#### **Report Categories:**

FAIRS Subject Report

#### **Approved By:**

Mark Petry

#### **Prepared By:**

Wu Bugang

#### **Report Highlights:**

On May 5, 2010, China notified the WTO of National Food Safety Standard: Food Additives – Sodium Hypochlorite as SPS/N/CHN/253. This measure applies to the definition and characteristics of the food additive Sodium Hypochlorite. It also specifies the testing methods to detect this chemical. The date for submission of final comments to China was May 20, 2010. The proposed date of entry was May 30, 2010. Contact information on where to send comments is inside the report. This report is an INFORMAL translation of this document.

**Executive Summary:**

On May 5, 2010, China notified the WTO of National Food Safety Standard: Food Additives – Sodium Hypochlorite as SPS/N/CHN/253 This measure applies to the definition and characteristics of the food additive Sodium Hypochlorite. It also specifies the testing methods to detect this chemical. The date for submission of final comments to China was May 20, 2010. The proposed date of entry was May 30, 2010.

Comments can be sent to the China WTO SPS Enquiry Point at: SPS@aqsic.gov.cn.

This report contains an UNOFFICIAL translation of National Standard on Determination of Sodium Hypochlorite in Foods.

**General Information:**

BEGIN TRANSLATION

**National food safety standard****Food Additive Sodium Hypochlorite**

(Draft for soliciting opinions)

Promulgated on XX-2010

Executed from XX-201X

**Foreword**

For revision of this standard (the Standard), "Sodium Hypochlorite" of the 8th version of Japanese standard Official Compilation of Food Additives is adopted.

The technological disparities between the Standard and "Sodium Hypochlorite" of the 8th version of Official Compilation of Food Additives include:

-- Indicators and the measuring method of free alkali, Fe, heavy metal and arsenic items are added;

-- Indicators of available chlorine are adjusted.

Appendix A to the Standard is a normative appendix.

**National Food Safety Standard****Food Additive Sodium Hypochlorite****1. Scope**

The Standard shall be applicable for sodium hypochlorite (NACLO) made from reaction between food additive sodium hydroxide (NaOH) and chlorine.

**2. Cited normative documents**

The documents cited in the Standard are imperative for application of the Standard. For the cited documents with date, only the version of the date shall be applicable for the Standard; for those without date, the latest version (including all revision lists) shall be applicable for the Standard.

### 3. Molecular formula and relative molecular mass

Molecular formula: NaClO

Relative molecular mass: 74.442 (as per international relative atomic mass in 2007)

### 4. Technical requirements

4.1 Requirements for sense evaluation: shall comply with the regulations of Table 1.

Table 1 Requirements for sense evaluation

Item	Requirements	Testing method
Color	No color or straw yellow	Take an amount of sample and put it in a beaker of 50ml, and observe the color and textural state in natural light.
Textural state	Liquid	

4.2 Physical and chemical indicator: shall comply with the regulations of Table 2.

Table 2 Physical and chemical indicator

Item	Indicator	Testing method
Available chlorine (Calculated as per Cl), w/% $\geq$	5.0	A.4 of Appendix A
Free alkali (Calculated as per NaOH), w/%	0.2~ 1.0	A.5 of Appendix A
Fe (Calculated as per Fe), w/% $\leq$	0.00 5	A.6 of Appendix A
Heavy metal (Calculated as per Pb), w/% $\leq$	0.00 1	A.7 of Appendix A
Arsenic (Calculated as per As), w/% $\leq$	0.00 01	A.8 of Appendix A

## Appendix A Testing method (normative appendix)

### A.1 Caution

The reagent applied in this testing method is toxic and corrosive, so care shall be taken in operation! If any reagent spatters on skin, rinse it with water immediately or go to hospital for treatment if serious.

### A.2 General conditions

Except otherwise specified, reagent and water applied in the Standard shall be pure reagent and water in Class III as specified in GB/T 6682-2008, and the required standard volumetric solution (VS), standard solution of impurity, and agent and product shall be prepared in accordance with GB/T 601-2002, GB/T 602-2002, and GB/T 603-2002.

### A.3 Identification

A.3.1 Reagent and material

A.3.1.1 Phosphate buffer solution: pH $\approx$ 8.0;

Solution I: Take and weigh 23.8g of anhydrous disodium hydrogen phosphate and dissolve in water, and dilute to 1000ml;

Solution II: Take and weigh 9.07g of potassium phosphate crystal and dissolve in water, and dilute to 1000ml;

Mix 50 shares of solution I and 7 shares of solution II, and adjust with solution I and II till the pH value reaches to 8.0.

A.3.1.2 Litmus test paper

A.3.2 Identification method

A.3.2.1 This sample can react like sodium salt and hypochlorite.

A.3.2.2 Take and measure 1ml of the sample, and dilute with water to 25ml; take and measure 4ml of the above test solution, and add 100ml of phosphate buffer solution. The highest absorption value is at the point 291~294nm of the wavelength.

A.3.2.3 When red litmus test paper is submerged into this sample, the color becomes blue and then disappears.

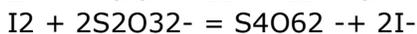
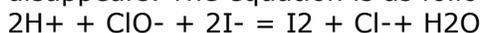
A.3.3 Identification result

This sample can react like sodium salt and hypochlorite, so it can be determined that this sample is NACLO.

#### **A.4 Determination of content of available chlorine**

A.4.1 Method and principle

In acid medium, hypochlorite (ClO<sup>-</sup>) reacts with potassium iodide (KI) and separate out iodine; take starch as the indicating liquid, and titrate with standard VS of sodium thiosulfate till the blue disappears. The equation is as follows:



A.4.2 Reagent and solution

A.4.2.1 KI solution: 100g/l;

A.4.2.2 Sulphuric acid solution: 3 + 100;

A.4.2.3 Standard VS of sodium thiosulfate:  $c(Na_2S_2O_3) = 0.1 \text{ mol/l}$ ;

A.4.2.4 Starch indicating liquid: 10g/l.

A.4.3 Instrument

Instruments in ordinary lab.

A.4.4 Analysis steps

A.4.4.1 Preparation of sample solution

Take and measure about 20ml of the sample and put in a weighed beaker of 100ml with about 20ml of water inside (keep accuracy to 0.01g), and weigh it (keep accuracy to 0.01g); and then, transfer all to a measuring flask of 500ml, and dilute with water to the scale and shake up. This solution is Test Solution A, and it shall be used for determination of the content of available chlorine, free alkali, Fe, heavy metal, and arsenic.

A.4.4.2 Determination

Take out 10.00ml of the sample solution A and put in an iodine measuring flask of iodine of 250ml with 50ml of water; add 10ml of KI solution and 10ml of sulphuric acid solution, cover quickly the bottle stopper followed by water seal, and keep it in shadow for 5min. Titrate with standard VS of sodium thiosulfate till the color becomes pale yellow, add 2ml of starch indicating liquid, and titrate continuously till blue color disappears.

A.4.5 Calculation

The content of available chlorine shall be calculated as per the mass fraction of chlorine ( $w_1$ ) and be expressed by % as shown in Formula A1:

$$w_1 = \frac{V/1000 \cdot cM_1}{m_1 \times 10/500} \times 100 = \frac{5VcM_1}{m_1} \dots\dots\dots (A.1)$$

In which:

c-- Accurate concentration of standard VS of sodium thiosulfate (unit: mol/l)  
 V- Consumed volume of standard VS of sodium thiosulfate (unit: ml)  
 m1- Mass of the sample (unit: g)  
 M1- Molar mass of chlorine (g/mol, M1=35.453)

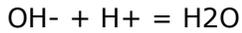
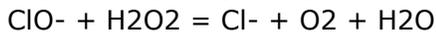
**A.4.6 Allowable difference**

Take arithmetic mean of the parallel determination results as the reported result, and the absolute difference among the parallel determination results shall not be more than 0.2%.

**A.5 Determination of content of free alkali**

**A.5.1 Method and principle**

Decompose ClO<sup>-</sup> with hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), take phenolphthalein as indicating liquid, titrate with standard VS of HCl till the color becomes reddish. The equation is as follows:



**A.5.2 Reagent and material**

A.5.2.1 H<sub>2</sub>O<sub>2</sub> solution: 1 + 5;

A.5.2.2 Standard VS of HCl: c (HCl)≈0.1mol/l;

A.5.2.3 Phenolphthalein indicating liquid: 10g/l;

A.5.2.4 Starch-KI test paper.

**A.5.3 Instrument**

Instruments in ordinary lab.

**A.5.4 Analysis steps**

Take out 50.00ml of sample solution A and put in a cone flask of 250ml, and add H<sub>2</sub>O<sub>2</sub> solution till there is not any ClO<sup>-</sup> (the starch-KI test paper does not becomes blue); and 2~3 drops of phenolphthalein indicating liquid, and titrate with standard VS of HCl till the color becomes reddish.

**A.5.5 Calculation**

The content of free alkali shall be calculated as per the mass fraction of NaOH (w<sub>2</sub>) and be expressed by % as Formula A2:

$$w_2 = \frac{V/1000 \cdot cM_2}{m_2 \times 50/500} \times 100 = \frac{VcM_2}{m_2} \dots\dots\dots (A.2)$$

In which:

c-- Accurate concentration of standard VS of HCl (unit: mol/l)  
 V-- Consumed volume of standard VS of HCl (unit: ml)  
 m2-- Mass of the sample (unit: g)  
 M2-- Molar mass of NaOH (unit: g/mol, M2=40.00)

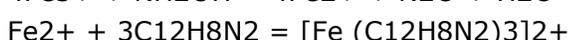
**A.5.6 Allowable difference**

Take arithmetic mean of the parallel determination results as the reported result, and the absolute difference among the parallel determination results shall not be more than 0.04%.

**A.6 Determination of content of Fe**

**A.6.1 Method and principle**

In medium without ClO<sup>-</sup>, oxammonium hydrochloride reduce Fe<sup>3+</sup> to Fe<sup>2+</sup>; in buffer solution system, of which the pH value is 4~4.5, Fe<sup>2+</sup> and 1 10-Phenanthroline monohydrate make orange red complex; determine the absorbance with a spectrophotometer. The equation is as follows:



**A.6.2 Reagent and solution**

A.6.2.1 H<sub>2</sub>O<sub>2</sub> solution: 1 + 5;

A.6.2.2 Acetic acid-sodium acetate buffer solution: pH≈4.5;

A.6.2.3 Oxammonium hydrochloride solution: 10g/l;

A.6.2.4 Take and weigh 1g of oxammonium hydrochloride and dissolve in water, and dilute to 100ml;

A.6.2.5 Standard solution of Fe: in 1ml of the solution, Fe content is 0.1mg;

A.6.2.6 Standard solution of Fe: in 1ml of the solution, Fe content is 0.01mg;

Use a pipette and take out 25.00ml of standard solution of Fe (A.6.2.5), and put in a measuring flask of 250ml; dilute to the scale and shake up. This solution shall be prepared just before use.

A.6.2.7 1,10-Phenanthroline monohydrate solution: 2g/l;

A.6.2.8 Starch-KI test paper

A.6.3 Instrument

Instrument and spectrophotometer in ordinary lab.

A.6.4 Analysis steps

A.6.4.1 Drawing of working curve

A.6.4.1.1 Take out 0.0ml, 1.0ml, 2.0ml, 3.0ml, 4.0ml, 6.0ml, 8.0ml, and 10.0ml of standard solution of Fe (A.6.2.6) and put in 8 measuring flasks of 100ml respectively; add 5ml of oxammonium hydrochloride solution, 10ml of acetic acid-sodium acetate buffer solution, and 5ml of 1,10-Phenanthroline monohydrate solution into each measuring flask, dilute with water to the scale and shake up, and keep for 10min.

A.6.4.1.2 Adjust the spectrophotometer to zero with blank solution, i.e. standard solution with out Fe; use appropriate colorimetric tube at the point 510nm of the wavelength 510nm to determine the absorbance.

A.6.4.1.3 Take the content of Fe in 100ml of colorimetric solution as abscissa and the relevant absorbance as the ordinate, and draw working curve or equation of linear regression.

A.6.4.2 Blank test

For blank test, the solution shall be without sample, but the analysis steps, reagent, and use level shall be the same as that in the test of sample solution.

A.6.4.3 Determination

Take out 50.00ml of sample solution A and put in a measuring flask of 100ml, and add H<sub>2</sub>O<sub>2</sub> solution till there is not ClO<sup>-</sup> (the starch-KI test paper does not becomes blue); and then, add 5ml of oxammonium hydrochloride solution, 10ml of acetic acid-sodium acetate buffer solution, and 5ml of 1,10-Phenanthroline monohydrate solution, dilute with water to the scale and shake up, and keep for 10min. The subsequent steps shall be taken in accordance with A.6.4.1.2.

A.6.5 Calculation

The content of Fe shall be calculated as per the mass fraction of Fe (w<sub>3</sub>) and be expressed by % as shown in Formula A3:

$$w_3 = \frac{m_4/1000}{m_3 \times 50/500} \times 100 = \frac{m_4}{m_3} \dots\dots\dots (A.3)$$

In which:

m<sub>3</sub>-- Mass of the sample (unit: g)

m<sub>4</sub>-- Mass of Fe in sample material, found out from working curve or calculated as per equation of linear regression (unit: mg).

A.6.6 Allowable difference

Take arithmetic mean of the parallel determination results as the reported result, and the absolute difference among the parallel determination results shall not be more than 0.001%.

## A.7 Determination of content of heavy metal

A.7.1 Method and principle

Under the condition of sub acidity (pH is 3~4), heavy metal ion and sulfur ion create brownish black deposit; compare it with the standard solution of Pb treated by the same method, and make limit test.

A.7.2 Reagent and material

A.7.2.1 HCL

A.7.2.2 H<sub>2</sub>O<sub>2</sub> solution: 1 + 5;

A.7.2.3 Acetic acid-sodium acetate buffer solution: pH≈3;

A.7.2.4 Saturated solution of hydrogen sulphide

Send hydrogen sulphide gas into water without CO<sub>2</sub> till it is saturated. This solution shall be prepared just before use.

A.7.2.5 Standard solution of Pb: in 1ml of the solution, Pb content is 0.1mg;

A.7.2.6 Standard solution of Pb: in 1ml of the solution, Pb content is 0.01mg;

Use a pipette to take out 10.00ml of standard solution of Pb (A.7.1.5) and put in a measuring flask of 100ml, and dilute to the scale and shake up. This solution shall be prepared just before use..

A.7.2.7 Phenolphthalein indicating liquid: 10g/l;

A.7.2.8 Starch-KI test paper

A.7.3 Instrument

Instruments and Nessler tube of 50ml in ordinary lab.

Submerge all glass apparatus in nitric acid (10~20%) for 24h at least; and then rinse with tap water repeatedly till clear.

A.7.4 Analysis steps

A.7.4.1 Tube A: Take out 1.0ml of standard solution of Pb (A.7.2.6) and put in a Nessler tube of 50ml, add water to 25ml, and add 5ml of acetic acid-sodium acetate buffer solution shake up for use.

A.7.4.2 Tube B: take one Nessler tube matching with Tube A, take out 25.0ml of sample solution A and put in a Nessler tube of 50ml, and add water to 25ml; add H<sub>2</sub>O<sub>2</sub> solution till there is not hypochlorite (the starch-KI test paper does not becomes blue). Add 1 drop of phenolphthalein indicating liquid, and adjust with HCl to reddish color; add 5ml of acetic acid-sodium acetate buffer solution, and shake up and for use.

A.7.4.3 Tube C: take one Nessler tube matching with Tube A and B, add isometric sample solution with Tube B, and add isometric standard solution of Pb with Tube A (A.7.2.6), and add water to 25ml; titrate H<sub>2</sub>O<sub>2</sub> solution till there is not ClO<sup>-</sup> (the starch-KI test paper does not becomes blue). Add 1 drop of phenolphthalein indicating liquid, and adjust with HCl to reddish color; add 5ml of acetic acid-sodium acetate buffer solution, and shake up for use.

A.7.4.3 Add 10ml of freshly prepared hydrogen sulphide saturated solution into each tube, add water to 50ml, mix up evenly and put in shadow for 5min; observe in white background, the degree of color in Tube B shall not be deeper than that in Tube A, and that in Tube C shall be equivalent to or deeper than that in Tube A.

## **A.8 Determination of content of y**

A.8.1 Reagent and material

A.8.1.1 HCL

A.8.1.2 H<sub>2</sub>O<sub>2</sub> solution: 1 + 5;

A.8.1.3 KI solution: 150g/l;

Take and weigh 15.0g of KI and dissolve in water, and dilute with water to 100ml.

A.8.1.4 Stannous chloride solution: 400g/l;

A.8.1.5 Standard solution of arsenic: in 1ml of the solution, the content of arsenic is 0.1mg;

A.8.1.6 Standard solution of arsenic: in 1ml of the solution, the content of arsenic is 0.001mg;

Use a pipette to take out 1.00ml of standard solution of arsenic (A.8.2.5), and put in a measuring flask of 100ml; dilute to 100ml and shake up. This solution shall be prepared just before use..

A.8.1.7 Lead acetate cotton

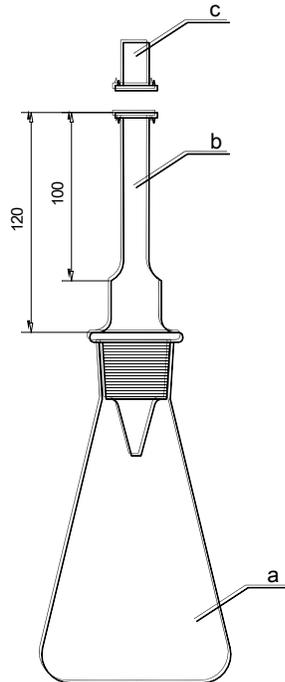
A.8.1.8 Mercuric bromide test paper

A.8.1.9 Starch-KI test paper

A.8.1.10 Zinc granule

A.8.2 Instrument

Instruments and arsenic apparatus in ordinary lab (See Figure A.1).



Unit: mm

a-- Cone flask of 100ml;

b-- Absorption tube

c-- Absorption tube cap

Figure A.1 Sketch map of arsenic apparatus

A.8.3 Analysis steps

A.8.3.1 Take out 1.0ml of solution of arsenic (A.8.1.6) and put in the cone flask of arsenic apparatus, add 5ml of HCl and water to 30ml, and additionally 5ml of KI solution and 5 drops of stannous chloride solution; shake up and keep for 10min.

A.8.3.2 Take out 25.00ml of sample solution A and put in the cone flask of arsenic apparatus, add H<sub>2</sub>O<sub>2</sub> solution till there is not any ClO<sup>-</sup> (the starch-KI test paper does not becomes blue). Adjust with HCl till the sample solution reaches to neutral state and 5ml of HCl is excess, and add water to 30ml and additionally 5ml of KI solution and 5 drops of stannous chloride solution; shake up and keep for 10min.

A.8.3.3 Add 2g of zinc granule in each of the above cone flasks, connect immediately with the absorption tube having been with lead acetate cotton and mercuric bromide test paper, keep in shadow at 25°C for 1h.

A.8.3.4 Take out arsenic stain, and the sample solution arsenic stain shall not be deeper than the limit standard; for the determination each time, standard arsenic stain shall be prepared simultaneously.

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END TRANSLATION

