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Standard examination methods for drinking water - Disinfection by-products parameters

生活饮用水标准检验方法

消毒副产物指标

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Foreword

GB/T 5750 "Standard Test Method for Drinking Water" is divided into the following parts:

- General principles;
- Collection and preservation of water samples;
- Water analysis quality control;
- Organoleptic and physical parameters;
- Nonmetal parameters;
- Metal parameters;
- Aggregate organic parameters;
- Organic parameters;
- Pesticides parameters;
- Disinfection by-products parameters;
- Disinfectants parameter;
- Microbiological parameters;
- Radiological parameters.

This standard replaces chloroform in the part II of GB/T 5750-1985 “Standard examination methods for drinking water”.

As compared with GB/T 5750-1985, the main changes are as follows:

- ADJUST the structure in accordance with GB/T 1.1-2000 “Directives for standardization - Part 1: Rules for the structure and drafting of standards” and GB/T 20001.4-2001 “Rules for drafting standards - Part 4: Methods of chemical analysis”;
- REVISE the quantity and measurement unit in accordance with the requirements of national standards;
- CHANGE the equivalent concentration into the molar concentration (the redox part still retains the equivalent concentration);
- CHANGE the mass concentration indication symbol C into ρ, CHANGE the

Standard examination methods for drinking water - Disinfection by-product parameters

1 Trichloromethane

Same as the test method for carbon tetrachloride in Chapter 1 of GB/T 5750.8-2006.

2 Tribromomethane

Same as the test method for carbon tetrachloride in Chapter 1 of GB/T 5750.8-2006.

3 Dichloro-bromomethane

Same as the test method for carbon tetrachloride in Chapter 1 of GB/T 5750.8-2006.

4 Chlorodibromomethane

Same as the test method for carbon tetrachloride in Chapter 1 of GB/T 5750.8-2006.

5 Dichloromethane

5.1 Headspace gas chromatography

5.1.1 Scope

This standard specifies the use of headspace gas chromatography for the determination of dichloromethane, 1,1-dichloroethane and 1,2-dichloroethane in drinking water and its source water.

This method applies to the determination of dichloromethane, 1,1-dichloroethane and 1,2-dichloroethane in drinking water and its source water.

The minimum detection mass concentration of this method: dichloromethane 9 μg/L, 1,1-dichloroethane 8 μg/L, 1,2-dichloroethane 13 μg/L.

Under the operating conditions of this method, other halogenated hydrocarbons do not interfere.

5.1.2 Principle

In a closed headspace bottle, volatile halogenated hydrocarbon molecules escape from the liquid phase into the gas in the headspace. At a certain temperature, the molecules of the halogenated hydrocarbon reach a dynamic equilibrium between the gas and liquid phases, at this time the concentration of the halogenated hydrocarbon in the gas phase is proportional to its concentration in the liquid phase. By measuring the concentration of the halogenated hydrocarbon in the gas phase, it can calculate the mass concentration of the halogenated hydrocarbon in the water sample.

5.1.3 Reagents and materials

5.1.3.1 Carrier gas and auxiliary gas

5.1.3.1.1 Carrier gas: High purity nitrogen (99.999%).

5.1.3.1.2 Gas: Pure hydrogen (> 99.6%).

5.1.3.1.3 Combustion supporting gas: Oil-free compressed air, which is purified by a purge tube containing 0.5 nm molecular sieves.

5.1.3.2 Reagents for preparing standard samples and reagents

5.1.3.2.1 Pure water (fresh deionized water).

5.1.3.2.2 Chromatographic standards (chromatographically pure): dichloromethane, 1,1-dichloroethane, 1,2-dichloroethane.

5.1.3.3 Reagents and materials for preparing columns

5.1.3.3.1 Columns and fillings are as shown in the relevant contents of 5.1.4.1.3.

5.1.3.3.2 The solvent used for the coating fixative solution: chloroform + butanol (1 + 1).

5.1.4 Instruments

5.1.4.1 Gas chromatograph

5.1.4.1.1 Hydrogen flame ionization detector.

5.1.4.1.2 Logger or workstation.

5.1.4.1.3 Column

40 min, which can be used for analysis after gas-liquid equilibrium.

5.1.6 Analytical procedures

5.1.6.1 Instrument adjustment

5.1.6.1.1 Gasification chamber temperature: 200 °C.

5.1.6.1.2 Column temperature: 85 °C.

5.1.6.1.3 Detector temperature: 200 °C.

5.1.6.1.4 Gas flow: Carrier gas 50 mL/min; hydrogen 52 mL/min; air 700 mL/min.

5.1.6.1.5 Attenuation: ADJUST the recorder attenuation in accordance with the content of the measured component in the sample.

5.1.6.2 Calibration

5.1.6.2.1 Calibration method in quantitative analysis: external standard method.

5.1.6.2.2 Standard samples

A: Number of uses: Each time the sample is analyzed, USE the new standard use solution to draw the standard curve or USE the response factor to make calculation.

B: Preparation of standard samples: TAKE three 10 mL volumetric flasks, ADD a few milliliters of distilled water, accurately WEIGH it, respectively ADD one drop of dichloromethane, 1,1-dichloroethane, and 1,2-dichloroethane. Accurately WEIGH it, the mass increased is the mass of dichloromethane, 1,1-dichloroethane, and 1,2-dichloroethane. USE pure water to make the volume reach to the mark. After calculating the content, respectively TAKE appropriate amount of this solution, DILUTE it to ρ (dichloromethane) = 5 μg/mL, ρ (1,1-dichloroethane) = 7.5 μg/mL, ρ (1, 2-dichloroethane) = 7.5 μg/mL, PREPARE it before use.

C: Conditions for use of standard solutions in gas chromatography:

a The standard solution injection volume is the same as the specimen injection volume. When measured by the single standard method, the standard solution response value shall be close to the specimen response value.

b When the relative standard deviation is less than 10% within the working range, the instrument is considered to be in a stable state.

c Standard samples and specimen are injected for analysis as far as possible at the same time.

Under selected chromatographic conditions, formaldehyde, propionaldehyde, acetone, and butyraldehyde do not interfere with the determination.

7.1.2 Principle

Acetaldehyde and acrolein in water can be directly separated and measured by gas chromatography with a hydrogen flame ionization detector. The peak order is acrolein and acetaldehyde.

7.1.3 Reagents and materials

7.1.3.1 Carrier gas and auxiliary gas

7.1.3.1.1 Carrier gas: High purity nitrogen (99.999%).

7.1.3.1.2 Combustion gas: Pure hydrogen (> 99.6%).

7.1.3.1.3 Combustion supporting gas: Oil-free compressed air, which is purified by a purge tube fitted with a 0.5 nm molecular sieve.

7.1.3.2 Reagents for preparing standard samples and specimen pretreatments

7.1.3.2.1 Sodium bisulfite solution [c($NaHSO_3$) = 0.05 mol/L]

7.1.3.2.2 Iodine standard solution [c (1/2 I_2) = 0.10 mol/L], to be calibrated.

7.1.3.2.3 Sodium thiosulfate standard solution [c ($Na_2S_2O_3$) = 0.10 mol/L], to be calibrated.

7.1.3.2.4 Starch solution (5 g/L).

7.1.3.2.5 Sulfuric acid solution (1 + 1).

7.1.3.2.6 Standard: Acrolein and acetaldehyde solution [ω(CH_3CHO) = 40%].

7.1.3.3 Reagents and materials for preparing columns

7.1.3.3.1 Columns and fills are described in 7.1.4.1.3.

7.1.3.3.2 Solvent used for coating fixative: dichloromethane.

7.1.4 Instruments

7.1.4.1 Gas chromatograph

7.1.4.1.1 Hydrogen flame ionization detector.

7.1.4.1.2 Logger or workstation.

DRY it naturally, PREPARE for use.

8.1.4.5 PTFE membrane or aluminum foil.

8.1.4.6 Constant temperature water bath: Control temperature ± 1 °C.

8.1.5 Samples

8.1.5.1 Sampling method and storage method: TAKE two headspace bottles containing 0.1 g of sodium thiosulfate to the site, FILL it with water samples, immediately USE overturn rubber plugs which are wrapped with aluminum foil (or PTFE membrane) to seal it, BRING it back to the laboratory. If it cannot be determined immediately, it must be kept in the refrigerator.

8.1.5.2 Water sample pretreatment: After the water sample is sent to the laboratory, POUR some water sample in the environment without chloroform to make the water sample in the bottle to 50 mL mark, immediately COVER the bottle stopper. PLACE one bottle directly in a constant temperature water bath at 40 °C as the bottle I, USE the injection needle to inject 0.2 mL of sodium hydroxide solution (8.1.3.2.2) into another bottle, SHAKE it uniformly, PLACE it into a constant temperature water bath at 40 °C as a bottle II, both of which are equilibrated in a 40 °C water bath for 2.5 h.

8.1.6 Analytical procedures

8.1.6.1 Instrument adjustment

8.1.6.1.1 Gasification chamber temperature: 200 °C.

8.1.6.1.2 Column chamber temperature: 150 °C.

8.1.6.1.3 Detector temperature: 250 °C.

8.1.6.1.4 Carrier gas flow: 80 mL/min.

8.1.6.2 Calibration

8.1.6.2.1 Calibration method in quantitative analysis: external standard method.

8.1.6.2.2 Standard samples

A: Number of uses: Each time the sample is analyzed, USE the newly prepared standard use solution.

B: Preparation of standard samples

a: Preparation of standard stock solution: WEIGH 0.1000 g of trichloroacetaldehyde (or 0.1120 g of trichloroacetaldehyde hydrate) into a 100 mL volumetric flask, USE distilled water to make the volume reach to the

trichloroacetaldehyde from the working curve, in micrograms per liter (μg/L).

8.1.7.2.2 Precision and accuracy: REPEAT determination in 6 laboratories, the concentration range of trichloroacetaldehyde is 10 μg/L ~ 90 μg/L, the average recovery is 97.8% ~ 101%, the relative standard deviation is 1.0% ~ 3.2%.

9 Dichloroacetic acid

9.1 Liquid-liquid extraction derived gas chromatography

9.1.1 Scope

This standard specifies the use of gas chromatography for the determination of monochloroacetic acid (MCAA), dichloroacetic acid (DCAA) and trichloroacetic acid (TCAA) in drinking water and its source water.

This method applies to the determination of monochloroacetic acid, dichloroacetic acid and trichloroacetic acid in drinking water and its source water.

The minimum detection mass of this method: the monochloroacetic acid (MCAA), dichloroacetic acid (DCAA) and trichloroacetic acid (TCAA) are 0.062 ng, 0.025 ng, 0.012 ng, respectively. If 25 mL of water samples are taken for determination, the minimum detection mass concentrations are 5.0 μg/L, 2.0 μg/L, 1.0 μg/L, respectively.

9.1.2 Principle

In an acidic condition (pH < 0.5), USE the methyl tert-butyl ether containing 1,2-dibromopropane (1,2-DBP) internal standard to extract the water sample, the extract is derivatized with the methanol solution which has been acidified by the sulfuric acid, to make the haloacetic acid in water form the methyl haloacetate, which is separated by a capillary column, determined by electron capture detector (ECD). It is qualitatively determined by the relative retention time, quantified by internal standard method.

9.1.3 Reagents and materials

9.1.3.1 Carrier gas: High purity nitrogen (99.999%).

9.1.3.2 Reagents and materials used in preparation of standard samples and specimen for pretreatment

9.1.3.2.1 Ammonium chloride crystals.

9.1.3.2.2 Anhydrous copper sulfate.

9.1.6.2.1 Calibration method in quantitative analysis: internal standard method.

9.1.6.2.2 Standard samples

A: Every time the sample is analyzed, the standard solution needs to be prepared before use. Standard samples and specimens are analyzed at the same time as much as possible.

B: Preparation of standard samples

a: Standard stock solution: single standard stock solution, TAKE 6.4 μL, 6.4 μL and 6.2 μL of a single standard substance of monochloroacetic acid, dichloroacetic acid and trichloroacetic acid (9.1.3.2.10) with a purity of not less than 99%, respectively ADD it into the 10 mL volumetric flask which contains about 5 mL of methyl tert-butyl ether (9.1.3.9.9), SHAKE it, MAKE its volume reach to the mark, the mass concentration of each solution is 1 mg/mL.

b: Standard use solution: respectively TAKE 1000, 500, 250 μL of standard stock solution (9.1.6.2.2 B a) into a 10 mL volumetric flask containing approximately 5 mL methyl tert-butyl ether. SHAKE it, MAKE its volume reach to the mark; after mixing, the mass concentration of monochloroacetic acid, dichloroacetic acid, and trichloroacetic acid are 100 mg/L, 50 mg/L, and 25 mg/L, respectively.

c: Internal standard extract: TAKE 7.8 μL of internal standard substance 1,2-DBP (9.1.3.2.5) into a 50 mL volumetric flask which contains about 20 mL of methyl tert-butyl ether (9.1.3.2.9), SHAKE it, MAKE its volume reach to the mark, the mass concentration of internal standard stock solution is 300 mg/L; then TAKE 50 μL of this stock solution, ADD it into a 50 mL volumetric flask containing about 20 mL of methyl tert-butyl ether in advance, SHAKE it, MAKE its volume reach to the mark, the internal standard extract concentration is 300 μg/L.

9.1.6.2.3 Preparation of working curve

Respectively TAKE 0, 5, 10, 20, 40 μL of standard use solution (9.1.6.2.2 B b) into an extraction flask containing 25 mL of pure water. The mass concentration of the working curve after preparation: MCAA is 0, 25, 50, 100, 200 μg/L, DCAA is 0, 12.5, 25, 50, 100 μg/L, TCAA is 0, 6.25, 12.5, 25, 50 μg/L. PERFORM extraction, derivation and analysis in accordance with the methods of 9.1.5.3.2. USE the ratio of the peak area of the standard substance to the peak area of the internal standard substance as the ordinate, the mass concentration as the abscissa, to draw the working curve.

9.1.6.3 Test

9.1.7.1 Qualitative results

In accordance with the retention time of each component of the standard chromatogram, DETERMINE the name of the component and the number of components in the water sample.

9.1.7.2 Quantitative results

9.1.7.2.1 Expression of content: CALCULATE the mass concentration of each component in accordance with the formula (6), expressed in micrograms per liter (μg/L).

9.1.7.2.2 Precision and accuracy: The relative standard deviations MCAA, DCAA, and TCAA determined in 5 laboratories are 4.6%, 5.4%, 3.8%, respectively. As for the spiked recovery test results of the three concentrations (high, medium, low) from 5 laboratories, when the dichloroacetic acid is at low concentration (4.0 μg/L~20 μg/L), the average recovery is 93.0%. At medium concentrations (40 μg/L ~ 90 μg/L), the average recovery is 96.0%. At high concentrations (100 μg/L ~ 200 μg/L), the average recovery is 92.0%. At low concentrations of trichloroacetic acid (2.0 μg/L ~ 10 μg/L), the average recovery is 98.0 %. At medium concentrations (10 μg/L~40 μg/L), the average recovery is 91.0%. At high concentrations (40 μg/L~100 μg/L), the average recovery is 98.0 %.

10 Trichloroacetic acid

See Chapter 9: Dichloroacetic acid.

11 Cyanogen chloride

11.1 Isonicotinic acid-barbituric acid spectrophotometry

11.1.1 Scope

This standard specifies the use of isonicotinic acid-barbituric acid spectrophotometry for the determination of cyanogen chloride in drinking water.

This method applies to the determination of cyanogen chloride in drinking water (chloride disinfected).

The minimum detection mass of this method is 0.10 ug. If 10.0 mL of water sample is taken for determination, the minimum detected mass concentration is 0.01 mg/L.

11.1.2 Principle

chromatographic interference peaks before application.

12.2.3.2.2 Hydrochloric acid solution [c(HCl)=1 mol/L]: TAKE 83 ml of hydrochloric acid (ρ_{20} = 1.84 mg/L), ADD pure water to dilute it to 1 L.

12.2.3.2.3 Sodium hydroxide solution [c (NaOH) = 1 mmol/L]: WEIGH 0.04 g of sodium hydroxide, DISSOLVE it in 1 L of pure water.

12.2.3.2.4 Sodium hydroxide solution [c(NaOH) = 0.1 mol/L]: WEIGH 4 g of sodium hydroxide, DISSOLVE it in 1 L of pure water.

12.2.3.2.5 2,4,6-trichlorophenol and pentachlorophenol standard substances, chromatographically pure.

12.2.3.2.6 Sodium chloride.

12.2.4 Instruments

12.2.4.1 Gas chromatograph

12.2.4.1.1 Electron capture detector, Ni-63.

12.2.4.1.2 Logger or workstation.

12.2.4.2 Column: HP-5 capillary column (30 m x 0.32 mm x 0.25 μm), SE-30 or column of equivalent polarity.

12.2.4.3 Solid phase microextraction device

12.2.4.3.1 Sampling station, stirring and constant temperature heating, control temperature at 60 °C ± 1 °C.

12.2.4.3.2 Extraction handle.

12.2.4.3.3 Polyacrylate extraction head, with a film thickness of 85 μm.

12.2.4.3.4 Headspace bottle, 15 mL, with silicone rubber seal cap. When used for the first time, USE hydrochloric acid solution (12.2.3.2.2) to boil it for 20 min, after being boiled in pure water for 20 min, finally USE oven to bake it for 30 min. When it is used later, it shall be rinsed clean and baked at 120 °C for 30 min.

12.2.4.4 100 mL stoppered test tube (or colorimetric tube).

12.2.5 Samples

12.2.5.1 Stability of the sample: The components to be determined in the sample are not stable, they shall be determined as soon as possible.

12.2.5.2 Sample collection and storage method: ADD 1 mL of sodium hydroxide solution (12.2.3.2.4) to a 100 mL stoppered test tube, BRING it to the site, FILL in 100 mL of water sample, SEAL it. FINISH determination within 24 h after collection.

12.2.6 Analytical procedure

12.2.6.1 Instrument conditions

12.2.6.1.1 Gasification chamber temperature: 280 °C.

12.2.6.1.2 Column temperature (programmed temperature increase): 40 °C (maintained for 3 min), rise to 120 °C at 10 °C/min, and rise to 240 °C at 15 °C/min (maintained for 2 min).

12.2.6.1.3 Detector temperature: 300 °C.

12.2.6.1.4 Carrier gas flow rate: 2.0 mL/min.

12.2.6.2 Calibration

12.2.6.2.1 Calibration methods in quantitative analysis: external standard method.

12.2.6.2.2 Standard samples

A: Number of uses: For each sample analyzed, USE the new standard solution to draw the standard curve.

B: Standard sample preparation:

a: Standard stock solution: Accurately WEIGH 0.1000 g of 2,4,6-trichlorophenol and 0.1000 g of pentachlorophenol standard substance (12.2.3.2.5), respectively USE 0.1 mol/L sodium hydroxide solution (12.2.3.2.4) to dissolve it, MAKE its volume reach to 100 mL. The concentration of this solution p (chlorophenols) = 1.0 mg/mL. It can be preserved at 4 °C for 1 month.

b: Mixed standard use solution: Respectively TAKE 5.00 mL and 1.00 mL of 2,4,6-trichlorophenol and pentachlorophenol standard stock solution (12.2.6.2.2 B a), ADD it into a 100 mL volumetric flask, USE 1 mmol/L sodium hydroxide solution (12.2.3.2.3) to make its volume reach to the mark. Then TAKE another 10.00 mL of this solution, USE 1 mmol/L sodium hydroxide solution (12.2.3.2.3) to make its volume reach to 100 mL. The contents of 2,4,6-trichlorophenol and pentachlorophenol in this mixed standard solution are 0.5 μg/mL and 0.1 μg/mL, respectively. PREPARE it before use.

12.2.6.2.3 Preparation of the standard series: In a laboratory that does not

recovery range is 86.0% ~ 116%; when the 2,4,6-trichlorophenol is spiked at 0.5 μg/L ~ 50 μg/L, the relative standard deviation ranges from 2.1% to 8.9%, the average recovery range is 90.3% to 111%; when the pentachlorophenol is spiked at 1 μg/L~50 μg/L, the relative standard deviation ranges from 2.0% to 8.5%, the average recovery ranges from 87.7% to 111%.

13 Chlorite

13.1 Iodometric method

13.1.1 Scope

This standard specifies the use of iodometric method for the determination of chlorite and chlorate in drinking water.

This method applies to the determination of chlorite and chlorate content in drinking water.

The minimum detection mass of this method: chlorite, 0.004 mg; chlorate, 0.004 mg. If taking 100 mL of water sample for determination, the minimum detectable concentration of chlorite is 0.04 mg/L. If taking 15 mL of water sample for determination, the minimum detectable concentration of chlorate is 0.23 mg/L.

13.1.2 Principle

After the chlorine dioxide-disinfected water sample is purged with pure nitrogen to remove chlorine dioxide, it is first reacted with iodine at pH7 to determine the non-volatile residual chlorine. Then the chlorite is determined at pH2. The water sample after nitrogen purging is treated with potassium bromide to avoid the interference of potassium iodide oxidation by dissolved oxygen. After the treatment, chlorate is determined.

13.1.3 Reagents

The pure water used for preparing reagents, diluting standard solutions, and washing glass instruments in this method is chlorine-free water.

Chlorine-free water preparation method: ADD 5 mg of free chlorine into each liter of pure water, PLACE it in the dark for two days, the free residual chlorine shall be at least > 2 mg/L. BOIL the pure water which is placed after chlorination, LET it be irradiated in the sun or UV lamp to decompose the residual chlorine. USE it after checking for no residual chlorine.

13.1.3.1 Phosphate buffer solution (pH7): DISSOLVE 25.4 g of anhydrous potassium dihydrogen phosphate and 33.1 g of anhydrous sodium phosphate monohydrate in 1000 mL of pure chlorine-free water. If any precipitate is present, it shall be filtered before use.

This method applies to the determination of chlorite, chlorate, and bromide ions in drinking water and source water.

The minimum detection mass concentrations for this method are: ClO_2^- 2.4 μg/L; ClO_3^- 5.0 μg/L; Br^- 4.4 μg/L.

If high concentrations of ClO_2 is existed in the water sample, it has an impact on the analysis, the effect of ClO_2 on the analysis can be eliminated by blowing in nitrogen and adding ethylenediamine as a protective agent.

When there is relatively high concentration of low molecular weight organic acids in the water sample, it may cause interference due to similar retention times. USE post-spiking measurements to help identify such interference. If the concentration of NO_3^- in the water is too high, it can cause serious interference to the determination of ClO_3^-. Such interference can be improved by diluting the water sample and changing the leaching conditions.

Due to the small injection volume, it shall strictly prevent the contamination of pure water and utensils in the pretreatment of the water sample during the operation, to ensure the accuracy of the analysis.

To prevent plugging of the protective column and separation column system, the sample shall be first filtered through a 0.20 μm filter. In order to prevent high hardness water from being precipitated in the carbonate eluent, the water sample is first passed through a strongly acidic cation exchange column if necessary.

The mutual interference when the different ions are analyzed at the same time or the interference from other components can be eliminated by such methods as water sample pre-condensation, gradient elution, or sample injection after collecting the flowing out; however, it shall confirm the precision and deviation of the method used.

13.2.2 Principle

The anions to be determined in the water sample enter the ion exchange system (composed of the guard column and the separation column) together with the carbonate eluent, and are separated in accordance with the affinity of the separation column for different ions, the separated anions flow through the suppressor system and are converted into a strong acid with high conductance, while the eluent is converted into a weakly conductive carbonic acid. The conductance of the various ionic components is measured by a conductivity detector, which is subject to qualitative determination by relative retention time, and quantitative determination by peak area or peak height.

13.2.3 Reagents and materials

n=6) is: 5.1%, 2.7%, 1.2%; 2.8%, 3.3%, 1.7%; 5.8%, 5.4%, 3.9%, respectively. The drinking water is spiked at 50, 200, 400 μg/L, respectively, the recovery rates are: 83.9%, 85.5%, 92.1%; 97.7%, 95.6%, 95.3%; 109%, 106%, 106%, respectively.

Bromide (Br^-): Three laboratories determine 50, 200, 400 μg/L bromide ion (Br^-) standard solution, respectively, the relative standard deviation (RSD, n=6) is: 6.7%, 2.1%, 0.8%; 5.6%, 3.4%, 0.9%; 8.4%, 6.6%, 2.4%. The drinking water is spiked at 50, 200, and 400 μg/L respectively, the recovery rates are: 105%, 95.0%, 98.5%; 113%, 102%, 105%; 101%, 105%, 106%, respectively.

Note: High-purity sodium chlorite is extremely explosive and can only use industrial sodium chlorite as a standard product. The $NaClO_2$ content of industrial products is only about 80%, and it always contains a small amount of ClO_3^- (3% ~ 4%). Therefore, sodium chlorite can only be used after accurate calibration of $NaClO_2$ content and impurity $NaClO_3$ content. The ClO_3^- contained therein will also affect the ClO_3^- concentration in the mixed standard solution.

13.2.8 Determination of sodium chlorite content and sodium chlorate content in sodium chlorite

13.2.8.1 Determination of sodium chlorite content

13.2.8.1.1 Reagents and solutions

A: Sulfuric acid solution (1 + 8): PIPETTE 20 mL of sulfuric acid, slowly ADD it into 160 mL of water, STIR it constantly.

B: Potassium iodide solution (100 g/L): WEIGH 20 g of potassium iodide, DISSOLVE it in 200 mL of water, PREPARE it before use.

C: Starch indicator solution (5 g/L): WEIGH 0.5 g starch, DISSOLVE it in 100 mL boiling water, PREPARE it before use.

D: Sodium thiosulfate standard solution [$c(Na_2S_2O_3)$ = 0.10 mol/L]: WEIGH 26 g of sodium thiosulfate and 0.2 g of sodium carbonate, ADD appropriate amount of cold water which is just boiled to dissolve it, DILUTE it to 1000 mL, MIX it uniformly, TRANSFER it into a brown reagent bottle, FILTER it after let it be standing for one month, PREPARE for use after accurate calibration.

a: Calibration of the sodium thiosulfate standard solution: WEIGH accurately about 0.15 g of potassium dichromate (national standard substance GB W 06105c) which has been dried at 120 °C to a constant weight, PLACE it in a 500 mL iodine volumetric flask, ADD 50 mL of water to dissolve it. ADD 2 g of potassium iodide, SHAKE gently to dissolve it, then ADD 20 mL of sulfuric acid solution (13.2.8.1.1 A), SEAL it, SHAKE it uniformly. PLACE it in the dark for 10 minutes, USE 250 mL of water to dilute it. USE sodium thiosulfate

WEIGH 10.60 g of anhydrous sodium carbonate (excellent grade pure), USE pure water (14.2.3.1) to dissolve it, MAKE its volume reach to the mark in a 100 mL volumetric flask. PLACE it in a 4 °C refrigerator to prepare for use, which can be preserved for 6 months.

14.2.3.9 Sodium hydroxide stock solution [c(NaOH) = 1.0 mol/L]: Accurately WEIGH 4.00 g of sodium hydroxide (excellent pure), USE pure water (14.2.3.1) to dissolve it, MAKE its volume reach to the mark in a 100 mL volumetric flask. PLACE it in a 4 °C refrigerator to prepare for use, which can be preserved for 6 months.

14.2.3.10 Sodium bicarbonate stock solution [c(HCO_3^-) = 1.0 mol/L]: Accurately WEIGH 8.40 g of sodium bicarbonate (excellent pure), USE pure water (14.2.3.1) to dissolve it, MAKE its volume reach to the mark in a 100 mL volumetric flask. PLACE it in a 4 °C refrigerator to prepare for use, which can be preserved for 6 months.

14.2.3.11 Eluent use solution: PIPETTE appropriate amount of sodium carbonate stock solution (14.2.3.8) and sodium hydroxide stock solution (14.2.3.8), or sodium bicarbonate stock solution (14.2.3.10), USE pure water (14.2.3.1) to dilute it, PREPARE it daily before use.

14.2.3.12 Regeneration solution [c(H_2SO_4) = 50 mmol/L]: PIPETTE 6.80 mL of concentrated sulfuric acid into a 1000 mL volumetric flask which has been filled with 800 mL of pure water (14.2.3.1), MAKE its volume reach to the mark (applicable to chemical suppressors).

14.2.4 Instruments

14.2.4.1 Ion chromatograph.

14.2.4.2 Conductance detector.

14.2.4.3 Chromatography workstation

14.2.4.4 Supporting gas: high purity nitrogen, purity 99.99%.

14.2.4.5 Sample injector: 2.5 mL ~ 10 mL syringe.

14.2.4.6 0.45 μm microporous membrane filter.

14.2.4.7 Ion chromatography instrument parameters (example)

Analytical system 1: Anion guard column: IonPac AG9-HC or equivalent guard column; anion analysis column: IonPac AS9-HC or equivalent analytical column; anion suppressor: AAES suppressor or equivalent suppressor; suppressor current: 53 mA; eluent: 7.2 mmol/L Na_2CO_3 +2.0 mmol/L NaOH; eluent flow rate: 1.00 mL/min,

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