NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB/T 2678.2-2021

Paper, board and pulps - Determination of water-soluble chlorides

Issued on: August 20, 2021 **Implemented on: September 1, 2022**

Issued by: SAMR; SAC

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1 Scope

This document specifies the methods for the determination of water-soluble chlorides in paper, paperboard and pulp by mercury nitrate, silver nitrate potentiometric titration and ion chromatography.

This document is applicable to all kinds of paper, cardboard and pulp.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 450, Paper and board - Sampling for testing and identification of machine and cross direction, wire side and felt side (GB/T 450-2008, ISO 186:2002, MOD)

GB/T 462, Paper, board and pulp - Determination of moisture content of analytical sample (GB/T 462-2008, ISO 287:1985 and ISO 638:1978, MOD)

GB/T 740-2003, Pulps - Sampling for testing (ISO 7213:1981, IDT)

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

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1 water-soluble chlorides

The chloride ion content in aqueous extracts of paper, board and pulp specimens measured under specified conditions.

4.1 Principle

The specimen is extracted with boiling water for 1h. Then in a solution containing chloride ions, drop into mercuric nitrate standard titration solution. At this time, mercury ions immediately react with chloride ions to form insoluble mercuric dichloride. Add excess ethanol to the titrant to reduce its solubility. When all the chloride ions in the solution become mercuric dichloride, a slight excess of mercury ions immediately forms purple mercury compounds with diphenylcarbazone added to the solution. Use the concentration and volume of the standard solution of mercury nitrate consumed to calculate the chloride ion concentration in the specimen extract.

4.2 Reagents

Unless otherwise specified, only analytically pure reagents are used.

4.2.4 Nitric acid (HNO3) solution [c(HNO3)=1.0mol/L]. Carefully measure 90mL

[c(HNO3)=14mol/L] of concentrated nitric acid (HNO3 content is about 65%). Add to 500mL of water (4.2.1). Then dilute to 1L.

4.2.6 Sodium chloride standard solution [c(NaCl)=0.01mol/L]. Accurately weigh

0.5846g of the benchmark sodium chloride that has been calcined at 500 degrees C ~ 600 degrees C for 2h and dissolve in water (4.2.1). Transfer to a 1000mL volumetric flask. Use water (4.2.1) to dilute to the scale. Certified standard solutions can also be purchased.

4.2.7 Mercury nitrate standard solution {c[Hg(HNO3)2]=0.01mol/L}. See Annex B for

preparation and calibration.

4.2.8 Nitric acid (HNO3) solution (volume fraction 1:1). Carefully measure 90mL of

concentrated nitric acid c(HNO3)=14mol/L (HNO3 content is about 65%)]. Add into 500mL of water (4.2.1). Then dilute to 1L.

4.2.9 Diphenylcarbazone ($C_{13}H_{12}ON_4$) indicator [$c(C_{13}H_{12}ON_4)=10g/L$]. Weigh 0.25g

of diphenylcarbazone and dissolve it in 25mL of 95% ethanol. Store in a brown bottle.

This solution is valid for one week.

4.3.3 Constant temperature water bath.

4.3.4 1mL microburette: the minimum division is 0.01mL.

4.3.5 150mL, 250mL, 500mL conical flasks.

4.4 Specimen collection and preparation

If it is to evaluate a batch of pulp, the specimen shall be collected according to GB/T 740-2003. If the test is used to evaluate a batch of paper and board, the specimen shall be taken in accordance with GB/T 450. If evaluating other types of samples, it shall be ensured that the samples taken are representative.

Due to the low chloride content in some samples, contamination of the samples shall be avoided during sampling. Clean gloves shall always be worn during specimen preparation. Tear or cut the sample into 5mm x 5mm specimens.

It shall be ensured that the laboratory is free of dust and fumes of chlorine-containing substances such as hydrochloric acid or chlorinated solvents. If the paper mill uses chlorine or chlorine dioxide as a bleaching agent, special attention shall be paid to specimen collection and preparation in the factory's in-house laboratory. Please do it in the on-site laboratory.

Wrap the sample in aluminum foil or plastic bag after specimen preparation until analysis. Store in the wild-mouth bottle with ground glass stopper.

The determination shall be carried out immediately after specimen preparation.

4.5.1 Do two experiments in parallel. Extract each specimen in duplicate. Carry out a

blank test of the reagents in full accordance with the method for the test specimen.

4.5.2 Accurately weigh 5.0g of the air-dried specimen, accurate to 0.001g. At the same

time, take another specimen of the same mass to determine the absolute dry mass m of Extract the sample with boiling water for 1h. Filter the extract. Use hydrogen peroxide heating to reduce the disturbances that may be caused by carbohydrates. Add nitric acid to dissolve and acidify the test solution. Add acetone. Then use potentiometric titration method

to titrate. With the addition of silver nitrate standard solution titrant, the potential of the indicator electrode changes accordingly. Near the end point of the titration, the concentration of the measured ion undergoes a sudden change, causing a sudden jump in the electrode potential. The end point of the titration can be determined from the jump in the electrode potential. From this, the content of chloride ions can be calculated from the volume of the consumed silver nitrate standard solution.

5.2 Reagents

Unless otherwise specified, only analytically pure reagents are used.

5.2.2 Nitric acid solution (1+1). Use water (5.2.1) to dilute 500mL of

[c(HNO3)=14mol/L] concentrated nitric acid (HNO3 content is about 65%) to 1L.

5.2.4 Silver nitrate standard solution [c(AgNO3)=20mmol/L]. Accurately weigh 3.397g

of dried silver nitrate. Use water (5.2.1) to make it completely dissolved. Transfer it into a 1000mL volumetric flask. Use water (5.2.1) to dilute it to the scale. This solution shall be stored in the dark.

5.2.5 Sodium hydroxide solution [c(NaOH)=0.1mol/L]. Weigh 4g of sodium hydroxide.

Use water (5.2.1) to dissolve it. Transfer into a 1000mL volumetric flask. Then use water (5.2.1) to dilute it to the scale.

5.3.1 Potentiometer or other measuring instrument: The measured DC voltage is

0mV~300mV. The accuracy is not less than 2mV. Use a silver electrode (silver ion selective electrode) as the indicator electrode, and a glass electrode as the reference electrode. Use an electrokinetic potentiometer with a motor-driven microburette and graph recording.

5.3.2 Glass microsyringe: 0.100mL, can read to 0.001mL.

5.3.3 500mL conical flask.

5.4 Specimen collection and preparation

Proceed as described in 4.4.

5.5.1 Do two experiments in parallel. Each specimen is extracted in duplicate. Carry

out a blank test according to the procedure for testing specimens.