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**Paper, board and paper products - Determination of
chloropropanol content**

纸、纸板和纸制品 氯丙醇含量的测定

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Table of Contents

Foreword.....	3
1 Scope.....	4
2 Normative references.....	4
3 Terms and definitions	4
4 Principle	4
5 Reagents and materials	4
6 Apparatus.....	5
7 Analytical procedure.....	6
8 Result calculation	9
9 Precision	10
10 Detection limit and quantitation limit.....	10
11 Test report	11
Appendix A (Informative) Ion chromatograms and mass spectra of 1,3-DCP and 3-MCPD measured by GC-MS.....	12

Paper, board and paper products - Determination of chloropropanol content

1 Scope

This document describes a method for the determination of content of 1,3-dichloro-2-propanol (1,3-DCP) and 3-chloro-1,2-propanediol (3-MCPD) in paper, board and paper products.

This document is applicable to all kinds of paper, board and paper products; is used as a reference for pulps and papermaking wet strength agents.

2 Normative references

The contents of the following documents, through normative references in this text, constitute indispensable provisions of this document. Among them, for dated references, only the edition corresponding to that date applies to this document. For undated references, the latest edition (including all amendments) applies to this document.

GB/T 462 Paper, board and pulps - Determination of moisture content of analytical sample

GB/T 6682 Water for analytical laboratory use - Specification and test methods

3 Terms and definitions

There are no terms or definitions to be defined in this document.

4 Principle

After the sample is chopped, mixed or diluted, add a deuterium-labeled internal standard solution to the specimen; use sodium chloride solution (20%) to extract it. After concentration, use heptafluorobutyrylimidazole to derivatize it; use gas chromatography-mass spectrometry for detection AND the internal standard method for quantification.

5 Reagents and materials

Unless otherwise specified, use only analytically pure reagents.

6.7 Nylon microporous filter membrane: Pore size is 0.22 μm .

6.8 Vortex mixer.

6.9 Pipette: 100 μL and 1000 μL .

6.10 Other commonly-used laboratory glassware.

7 Analytical procedure

7.1 Preparation of standard solutions

7.1.1 Chloropropanol mixed standard stock solution (1000 mg/L): Respectively weigh 50 mg (accurate to 0.1 mg) of 1,3-DCP standard (5.9) and of 3-MCPD standard (5.10); use ethyl acetate (5.3) to dissolve and transfer to a 50 mL volumetric flask; use ethyl acetate (5.3) to dilute to the mark; seal and store at 0 °C~4 °C. The storage period is 6 months.

7.1.2 Chloropropanol mixed standard intermediate solution (10 mg/L): Accurately pipette 0.1 mL of chloropropanol mixed standard stock solution (7.1.1) into a 10 mL volumetric flask; use ethyl acetate (5.3) to dilute to the mark; seal and store at 0 °C~4 °C. The storage period is 3 months.

7.1.3 Chloropropanol mixed standard intermediate solution (1 mg/L): Accurately pipette 0.1 mL of chloropropanol mixed standard stock solution (7.1.1) into a 100 mL volumetric flask; use ethyl acetate (5.3) to dilute to the mark; seal and store at 0 °C~4 °C. The storage period is 3 months.

7.1.4 Chloropropanol internal standard mixed standard stock solution (1000 mg/L): Respectively weigh 50 mg (accurate to 0.1 mg) of 1,3-DCP-D₅ standard (5.11) and of 3-MCPD-D₅ standard (5.12); use ethyl acetate (5.3) to dissolve and transfer to a 50 mL volumetric flask; use ethyl acetate (5.3) to dilute to the mark; seal and store at 0 °C~4 °C. The storage period is 6 months.

7.1.5 Chloropropanol internal standard mixed standard intermediate solution (10 mg/L): Accurately pipette 0.1 mL of chloropropanol internal standard mixed standard stock solution (7.1.4) into a 10 mL volumetric flask; use ethyl acetate (5.3) to dilute to the mark; seal and store at 0 °C~4 °C. The storage period is 3 months.

7.1.6 Chloropropanol standard working solutions: Respectively pipette 0.01 mL, 0.05 mL, 0.1 mL, 0.2 mL, 0.4 mL, 0.8 mL, 1.6 mL of chloropropanol mixed standard intermediate solution (1 mg/L) (7.1.3) AND 0.32 mL of chloropropanol mixed standard intermediate solution (10 mg/L) (7.1.2) INTO transparent glass tubes with a stopper (cap) of appropriate volume (5 mL or 10 mL) with good airtightness. Respectively add 20 μL of chloropropanol internal standard mixed standard intermediate solution (10

mg/L) (7.1.5); add n-hexane (5.2) to 2 mL and mix well. Prepare a series of standard working solutions containing 10 ng, 50 ng, 100 ng, 200 ng, 400 ng, 800 ng, 1600 ng, and 3200 ng of 1,3-DCP and 3-MCPD respectively; in which the mass of each deuterated chloropropanol is 200 ng. Standard solutions of other concentrations can also be prepared as needed. Prepare the standard working solution at the time of use.

7.2 Sample processing

For the sample of paper, board, paper products and pulp, weigh about 10 g of the sample; cut it into small pieces of about 5 mm×5 mm. After mixing well, accurately weigh 1.0 g (accurate to 0.0001 g) of the specimen into a glass test tube (6.5). At the same time, take another specimen from the sample; measure the moisture content according to GB/T 462.

For the sample of papermaking wet strength agents, if the chloropropanol content is high, in order to ensure the representativeness of the sampling, use water (5.1) to dilute the sample appropriately step by step. Then accurately weigh 1.0 g (accurate to 0.0001 g) of the diluted and mixed specimen into a glass test tube (6.5), so that the concentration of chloropropanol in the weighed specimen is within the range of the standard working solution. If the chloropropanol content of the papermaking wet strength agent sample is low, it is possible to directly weigh 1.0 g (accurate to 0.0001 g) of the sample into a glass test tube (6.5).

7.3 Test procedure

7.3.1 Extraction: Add 15 mL of sodium chloride solution (20%) (5.6) AND 20 µL of chloropropanol internal standard mixed standard intermediate solution (10 mg/L) (7.1.5) into the test tube containing the sample. After thorough mixing, use an ultrasonic cleaner (6.3) to extract at 40 °C for 30 min, to obtain the extraction solution. During the ultrasonic extraction process, the test tube mouth should be kept sealed until purification.

Note: If the sample contains water-absorbing resin, increase the volume of sodium chloride solution (20%) appropriately.

7.3.2 Purification: Weigh the macroporous diatomite (5.7) whose mass is equivalent to half the mass of the extraction solution (7.3.1); load it into a chromatographic column (5.8). Pour the extraction solution (7.3.1) into the diatomite chromatographic column and balance for 10 min. Use 10 mL of n-hexane (5.2) to rinse and discard the rinsing solution. Then use 18 mL of ethyl acetate (5.3) to elute; collect the eluent in a test tube. Add 3 g of anhydrous sodium sulfate (5.4) into the test tube; after letting it stand for 10 min, transfer the eluent (discard the anhydrous sodium sulfate part) to another clean stoppered test tube. Concentrate the eluent to nearly dryness by blowing nitrogen; add 2 mL of n-hexane (5.2) to reconstitute; wait for derivatization.

Note: The volume of the eluent after concentration by nitrogen blowing is approximately 0.5 mL.

7.3.3 Derivatization: Quickly add 0.04 mL of heptafluorobutyrylimidazole (5.5) to the purification solution (7.3.2); immediately cover the test tube plug; mix by vortex for 30 s; incubate in a 70 °C thermostat water bath (6.4) for 20 min; remove and cool to room temperature. Add 2 mL of sodium chloride solution (20%) (5.6); mix by vortex for 1 min to separate the water phase and n-hexane phase. Let it stand for a while until the water phase becomes clear; transfer the n-hexane phase; add about 0.3 g of anhydrous sodium sulfate (5.4) to dry it. Pass the dried n-hexane phase test solution through a nylon microporous filter membrane (6.7); then transfer it to the injection bottle for gas chromatography-mass spectrometry. At the same time, derivatize the chloropropanol standard working solution (7.1.6).

7.3.4 Blank test: Except that no sample is added, the blank test is carried out in accordance with 7.3.1~7.3.3.

7.4 Instrument reference conditions

7.4.1 Reference conditions for gas chromatography

The following operating conditions have been found to be suitable for testing:

- a) Chromatographic column: Weakly polar capillary gas chromatography-mass spectrometry column containing 5% phenyl-methylpolysiloxane (column length is 30 m; inner diameter is 0.25 mm; film thickness is 0.25 μm); or one with equivalent performance;
- b) Carrier gas: Helium (purity>99.999%); flow rate is 1.0 mL/min;
- c) Inlet temperature: 250 °C;
- d) Injection volume: 1 μL ; splitless injection. Splitless time is 0.5 min. Solvent delay time is 5 min;
- e) Programmed temperature rise: Keep at 50 °C for 1 min; raise to 90 °C at 2 °C/min; then raise to 270 °C at 40 °C/min; maintain for 5 min.

7.4.2 Reference conditions for mass spectrometry

The following operating conditions have been found to be suitable for testing:

- a) Ion source: Electron impact ion source (EI);
- b) Ionization energy: 70 eV;
- c) Ion source temperature: 250 °C;
- d) Transmission line temperature: 280 °C;

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