



GB 5009.191-2016

National Food Safety Standard

Determination of Chloropropanols and Chloropropanol Fatty Acid Esters in Foods

食品安全国家标准

食品中氯丙醇及其脂肪酸酯含量的测定

National Health and Family Planning Commission
of the People's Republic of China
China Food and Drug Administration

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Foreword

This standard replaces GB/T 5009.191-2006 “Determination of chloropropanols in foods”, GB/T 18782-2002 “Determination of 3-chloro-1,2-propandiol in condiments”, SN/T 0548.1-2002 “Determination of 1,3-dichloro-2-propanol and 2,3-dichloro-1-propanol in soy sauce for export”.

Compared with GB/T 5009.191-2006, the main changes of this standard are as follows:

——Revised the standard name to “National food safety standard Determination of chloropropanols and chloropropanol fatty acid esters in foods”;

——Added D₅-2-MCPD and D₅-2,3-DCP as the internal standard substances in Method II, and revised the purification step from hand-filled diatomite chromatography column purification to diatomite cartridge purification;

——Deleted Method III ;

——Added gas chromatography-mass spectrometry as test method for chloropropanol fatty acid esters content in foods.

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Determination Of Chloropropanols and Chloropropanol Fatty Acid Esters in Foods

1 Scope

This standard specifies the method for determination of chloropropanols and chloropropanol fatty acid esters in foods.

This standard applies to the determination of chloropropanols and chloropropanol fatty acid esters in foods.

This standard includes three test methods, which are the determination of 3-chloro-1,2-propanediol in foods, the determination of multi-component content of chloropropanols in foods, and the determination of chloropropanol fatty acid esters in foods.

Method I Determination of 3-chloro-1,2-propanediol in foods

Isotope dilution-gas chromatography-mass spectrometry

2 Principle

This standard adopts isotope dilution technology with D₅-3-chloro-1,2-propanediol (D₅-3-MCPD) as the internal standard substance. Add the internal standard substance to the test sample, and perform the liquid-liquid extraction supported by solid phase with diatomite as the adsorbent. Wash with n-hexane-anhydrous ethyl ether solution (9+1) to remove the non-polar lipid, and elute 3-chloro-1,2-propanediol (3-MCPD) with anhydrous ethyl ether. Derivatize with heptafluorobutyl imidazole, determine with gas chromatography-mass spectrometry, and quantify with internal standard method.

3 Reagents and Materials

Unless otherwise stated, the reagents used in this method are analytically pure and water is Grade 1 water specified in GB/T 6682.

3.1 Reagents

3.1.1 Ethyl acetate (C₄H₈O₂): chromatographically pure.

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3.1.2 n-hexane (C_6H_{14}): chromatographically pure.

3.1.3 Anhydrous ethyl ether ($C_4H_{10}O$): it needs to be re-distilled before use; add a small amount of reduced iron powder when re-distillation.

3.1.4 Sodium chloride ($NaCl$).

3.1.5 Anhydrous sodium sulfate (Na_2SO_4): bake it at 120 °C for 4 h before use.

3.1.6 Heptafluorobutyl imidazole ($C_7H_3F_7N_2O$, CAS No.: 32477-35-3): with purity of not less than 98%.

3.2 Preparation of reagents

3.2.1 Sodium chloride solution (20%): weigh 20 g of sodium chloride, add 80 mL of water, and stir to dissolve sodium chloride fully.

3.2.2 n-hexane-anhydrous ethyl ether solution (9+1): mix 450 mL of n-hexane and 50 mL of anhydrous ethyl ether well.

3.3 Standard substances

3.3.1 3-chloro-1,2-propanediol (3-MCPD) standard substances ($C_3H_7ClO_2$, CAS No.: 96-24-2): with purity of not less than 98%.

3.3.2 D₅-3-chloro-1,2-propanediol (D₅-3-MCPD) standard substances ($C_3D_5H_2ClO_2$): with purity of not less than 98%.

3.4 Preparation of standard solutions

Note: The well-prepared standard solutions need to be transferred to brown glass containers with very good airtightness and stored at -20 °C.

3.4.1 3-MCPD and D₅-3-MCPD standard stock solution (1000 mg/L): accurately weigh 10 mg (accurate to 0.01 mg) of 3-MCPD and D₅-3-MCPD respectively, dissolve with ethyl acetate, respectively transfer to two 10-mL volumetric flasks, dilute to the scale with ethyl acetate, and mix well. It is valid for 2 years.

3.4.2 3-MCPD standard intermediate solution (10 mg/L): accurately transfer 0.1 mL of 3-MCPD



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