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NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA

GB 31604.26-2016

**National Food Safety Standard - Food contact
materials and products - Determination of
epichlorohydrin and determination of migration**

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Republic of China**

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Foreword

This standard replaces GB/T 23296.7-2009 "Food contact materials. Determination of epichlorohydrin in plastics. High performance liquid chromatography" and SN/T 2897-2011 "Food contact materials for export. Metal materials. Determination of epichlorohydrin in coating. Gas chromatography".

In comparison with GB/T 23296.11-2009, the main differences of this standard are as follows:

- Changed the name of the standard to "National Food Safety Standard - Food contact materials and products - Determination of epichlorohydrin and its migration";
- Added applicable scope;
- Revised the sample preparation method of coating;
- Added the determination method of the migration of epichlorohydrin in food contacting materials and products.

National Food Safety Standard - Food contact materials and products - Determination of epichlorohydrin and determination of migration

1 Scope

This standard provides the determination method of epichlorohydrin in food contact materials and products (plastics, paints, paper and cardboard) and the determination method of migration.

This standard is applicable to the determination of epichlorohydrin in food contact materials and products (plastics, paints, paper and cardboard) and the determination of the migration of epichlorohydrin. The high performance liquid chromatography (HPLC) is suitable for the determination of epichlorohydrin in food contact materials and products (plastics, coatings, paper products). The gas chromatography-mass spectrometry is suitable for the determination of the migration of epichlorohydrin in food contact materials and products (plastics, coatings, paper and cardboard).

Determination of epichlorohydrin

2 Principle

After extraction of epichlorohydrin from the sample by 1,4-dioxane, the extract solution is distilled by a microdistillation unit. The epichlorohydrin in the distilled liquid is derived and its derivatives are separated by liquid chromatography. The content of epichlorohydrin derivatives is determined by fluorescence detector and the content of epichlorohydrin in the sample can be calculated. The external standard method is used for quantitative analysis.

3 Reagents and materials

Unless otherwise stated, the reagents used in this method shall be analytical pure. The water shall be the primary water specified in GB/T 6682.

3.1 Reagents

3.1.1 Acetonitrile (C_2H_3N): chromatographic pure.

3.1.2 9,10-Sodium dimethoxanthrone-2-sulfonate ($C_{16}H_{13}NaO_5S$), or DAS-Na.

3.1.3 1,4-Dioxane ($C_4H_8O_2$): purity $\geq 99\%$ and the water content $< 0.01\%$.

3.1.4 Methanol (CH_4O): chromatographic purity.

3.1.5 Strong acid cation exchange resin.

3.1.6 Nitrogen: purity greater than or equal to 99.9%.

3.1.7 Ice.

3.2 Reagent preparation

3.2.1 Methanol solution (80%, volume fraction)

Take 400mL of methanol and put in 500ml volumetric bottle. Meter the volume with water.

3.2.2 9,10-Sodium dimethoxanthrone-2-sulfonate reagent (DAS reagent)

Use 200 mL methanol solution (80%, volume fraction) to activate about 50g pre-treated strong acid cation exchange resin. Then filled resin to a glass column with inner diameter of 1cm to 20cm high. Put aside to be used. Weigh 100mg of DAS-Na add to a beaker containing 20ml methanol, heat to dissolve. Pour the solution through the strong acid cation exchange resin. Complete all the solutions in 2 times or 3 times. Elute with methanol and make the flow rate of the effluent at about 0.5mL/min, collect the eluent at pH 1~2 until the pH begins to rise. Dry the obtained eluent with nitrogen blower to get the DAS reagent. The DAS reagent should be kept away from light and validity period is 1 year.

3.2.3 DAS-Acetonitrile solution (5mg/mL)

Weigh 20mg (precision to 0.1mg) DAS reagent and dissolve in 4mL acetonitrile. Prepare when needed; keep away from light.

3.3 Standard product

Epichlorohydrin (C_3H_5ClO , CAS No.: 106-89-8): purity $> 99\%$, or a standard substance certified by the State and granted the standard substance certificate.

3.4 Standard solution preparation

3.4.1 Epichlorohydrin standard reserve solution (1mg/mL)

Take 10.0mL of 1,4-dioxane that is not in contact with plastic, paper or coating samples and put it into a 20mL sample bottle, add magnetic stirring rod, seal with spacer and aluminum cap and mark the sample bottle as A₀.

5.4 Distillation of 1,4- dioxane extract

Use the micro-distillation unit in figure A.1 to distill 1,4-dioxane extract and collect the partial distillate. In figure A.1, bottle A are the sample bottles in 5.2.1, 5.2.2, 5.3 and 3.4.4, which contain 10mL sample extract solution, blank solution and standard working solution respectively.

Take another 20mL sample bottles and mark at the volume of 3mL, then seal with spacer and aluminum cap. Weigh the sealed sample bottle (precision to 0.1mg) and label as bottle B.

As shown in figure A.1, bottle A and bottle B are connected by a stainless steel tube with polytetrafluoroethylene on the outside. The depth of the pipe pierced into bottle A is 1cm~2cm and in bottle B is 3cm~4cm. To prevent the inserted pipe from being blocked, prick a small hole on the bottle plug of the sample bottle so that the steel pipe can quickly pierce into the bottle. Insert a syringe needle on the bottle B's cover to avoid excessive pressure in the bottle.

Place bottle A on a heating plate and put bottle B in a beaker containing a mixture of ice water. Heat bottle A to boil the 1,4-dioxane extract, continue to heat mildly and move the distilled 1,4-dioxane extract to bottle B until the liquid level reaches 3mL mark in bottle B. Remove bottle A from the heater and remove the syringe needle and stainless steel tube from bottle B. Wipe dry bottle B carefully with filter paper. Reweigh bottle B (precision to 0.1mg). Calculate the volume of distillate according to the difference between the two weights and the density of 1,4-dioxane.

The distillate can be stored for 2 weeks at 0°C~ 4°C.

5.5 Derivatization of epichlorohydrin

Open the aluminum cover of bottle B containing distillate solution, add 50μL of DAS-acetonitrile solution (5mg/mL), seal with spacer and aluminum cap. Place the sample bottle in 75°C oven for 17h±1h, then cool the derivative solution in room temperature and filter with 0.22μm microporous membrane. Set aside the filtrate to be tested.

The Das derivatives of epichlorohydrin are very sensitive to light. The derivatization shall be performed under the condition away from light. The derivatives can be stored in room temperature away from light for 2 days.

5.6 Instrument reference condition

Accurately weigh the standard material of epichlorohydrin 50 mg (precision to 0.1mg/g), transfer to 50mL volumetric flask with dichloromethane and dilute to the scale. Seal to store at 0°C~ 4°C.

10.4.2 Epichlorohydrin standard intermediate for water-based, acidic and alcohol food simulants (50mg/L)

Accurately move 5mL of epichlorohydrin standard reserve solution (1000mg/L) into 100mL volumetric flask, dilute to scale with dichloromethane. Seal to store at 0°C~ 4°C.

10.4.3 Epichlorohydrin standard intermediate for water-based, acidic and alcohol food simulants (10mg/L)

Accurately move 1mL of epichlorohydrin standard reserve solution (1000mg/L) into 100mL volumetric flask, dilute to scale with dichloromethane. Seal to store at 0°C~ 4°C.

10.4.4 Epichlorohydrin standard intermediate for water-based, acidic and alcohol food simulants (1mg/L)

Accurately move 5mL of epichlorohydrin standard reserve solution (10mg/L) into 50mL volumetric flask, dilute to scale with dichloromethane. Seal to store at 0°C~ 4°C.

10.4.5 Epichlorohydrin standard working solution for water-based and acidic food simulants

Add 2.5mL and 5.0mL of epichlorohydrin standard intermediate for water-based, acidic and alcohol food simulants (1mg/L) and 10 mg/L, 2.5mL and 5.0mL of epichlorohydrin standard intermediate for water-based, acidic and alcohol food simulants (10mg/L) to 5 50mL volumetric flasks respectively. Use the same food stimulants to dilute to scale. The concentrations of epichlorohydrin in the series of the standard working solutions are 0.05mg/L, 0.1mg/L, 0.2mg/L, 0.5mg/L, 1.0mg/L respectively.

10.4.6 Epichlorohydrin standard intermediate for alcohol food simulants

Add 1mL, 2mL, 4mL, 10mL, 20mL of Epichlorohydrin standard intermediate for water-based, acidic and alcohol food simulants (50mg/L) to 5 50mL volumetric flasks respectively. Meter to scale with methanol. The concentrations of this series of standard intermediates are 1.0mg/L, 2.0mg/L, 4.0mg/L, 10.0mg/L, 20.0mg/L.

10.4.7 Epichlorohydrin standard working solution for alcohol food simulants

the droplets in the funnel neck of the separating funnel, release the lower layer of dichloromethane extract liquid into a centrifuge tube and plug it. According to the above steps, add again the dichloromethane 5mL accurately, extract the simulant, and combine the extract liquid in the same centrifuge tube. Record the volume of the extract liquid as V. Reserve to be used.

12.2.2 Preparation of alcohol and oil based food simulants

Accurately take 5mL of alcohol food simulants obtained from the migration test or weigh 10g of the oil based simulants, place into 20mL headspace bottles respectively, seal with spacer and aluminum cap, and set aside to be used.

Note: When the alcohol content in the alcohol food simulants exceeds 10%, dilute the ethanol in the simulant solution to 10%. Operate as per 12.2.2.

12.2.3 Preparation of blank test solution

Treat the food simulants not in contact with food contact materials in accordance with 12.2.

12.3 Dynamic headspace conditions

The options are set out in Appendix A.

Note: Refer to these conditions for the determination of epichlorohydrin in alcohol and oil-based food simulants.

12.4 Reference conditions for Gas Chromatography-Mass Spectrometry

The gas chromatography-mass spectrometry reference conditions are as follows:

- a) Chromatographic column: polyethylene glycol quartz capillary column, column length 30m, inner diameter 0.25mm, film thickness 0.25 μ m, or equivalent;
- b) Carrier gas: helium, purity $\geq$ 99.999%;
- c) Carrying gas velocity: 1.0mL/min;
- d) Temperature increase program: initial temperature at 35°C, remain for 2 minutes; raise the temperature at a rate of 10°C/min to 240°C, then maintain for 15min;
- e) Temperature of injection port: 280°C;

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Contact: Wayne Zheng, Sales@ChineseStandard.net

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