GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 31604.28-2016

National Food Safety Standard -

Food contact materials and products -

Determination of di (2-ethylhexyl) adipate and migration

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Foreword

This standard replaces GB/T 20499-2006 "Method for the determination of di (2-ethylhexyl) adipate migrating from polyvinyl chloride film in contact with foodstuffs", “Method for the determination of di (2-ethylhexyl) adipate” in GB/T 20500-2006 “Method for the determination of di (2-ethylhexyl) adipate and di-n-octyl adipate in polyvinyl chloride film” and “Method for the determination of di (2-ethylhexyl) adipate migration” in SN/T 2826-2011 “Food contact materials for export. Polymers. Determination of adipate plasticizers in food stimulants. Gas chromatography-mass spectrometry”.

The main differences of this standard in comparison with GB/T 20499-2006 and GB/T 20500-2006 are as follows:

- Changed the name of the standard to "National Food Safety Standard - Food contact materials and products - Determination of di (2-ethylhexyl) adipate and migration";

- Deleted the gas chromatography-mass spectrometry for determination of di (2-ethylhexyl) adipate.

National Food Safety Standard - Food contact materials and products - Determination of di (2-ethylhexyl) adipate and migration

1 Scope

This standard specifies the methods of determination of di (2-ethylhexyl) adipate (DEHA) and migration in food contact materials and products.

This standard is applicable to the determination of di (2-ethylhexyl) adipate and migration in polyvinyl chloride products.

Determination of di (2-ethylhexyl) adipate (DEHA)

2 Principle

Dissolve the sample with tetrahydrofuran and precipitate the polymer by methanol. After filtration, DEHA remains in the filtrate. Determine by gas chromatography-mass spectrometer and quantify by external standard method.

3 Reagents and materials

3.1 Reagent

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.

3.1.1 Tetrahydrofuran (C_4H_8O, CAS No.:109-99-9)

3.1.2 Methanol (CH_4O,CAS No.:67-56-1)

3.1.3 N-hexane (C_6H_{12},CAS No.:110-54-3): Chromatographic pure

3.2 Standard product

di (2-ethylhexyl) adipate ($C_{22}H_{42}O_4$, CAS No.: 103-23-1): purity≥99.8%, or a standard substance certified by the State and granted the standard

The mass spectrum conditions are as follows:

a) Mass spectrum interface temperature: 250°C;

b) Temperature of ion source: 250°C;

c) Ionization mode: EI;

d) Ionization energy: 70 eV;

e) Determination method: select the ion monitoring mode, the monitoring ion range(m/z) of DEHA: 40~370, the DEHA characteristic ions of are: 129, 147, 112, 71, of which 129 is quantitative ion;

f) Solvent delay by: 5 min.

5.5 Making of standard curve

According to the determination conditions listed in 5.4, inject the standard working solution (3.3.3) into the gas chromatography-mass spectrometer in turn. The standard curve was drawn with the concentration of the standard working solution as the horizontal coordinate with the unit of mg per liter (mg/L), and the corresponding peak area of DEHA as the vertical coordinate. See figure A.1. for a standard chromatogram.

5.6 Determination of sample solution

5.6.1 Qualitative determination

In accordance with the determination conditions listed in 5.4, inject the sample solution (5.2) and the blank solution (5.3) into the gas chromatography-mass spectrometer for determination. When determine the sample under the same experimental conditions, if the deviation of the chromatographic peak retention time of the substance to be determined in the sample solution is within ±2.5% of the chromatographic peak retention time of the standard substance; and all the selected ions appear in the sample mass spectrometry after background subtraction; and the deviation of the relative abundance of the qualitative ion in the sample spectrum compared with the relative abundance of the qualitative ion in the standard solution with similar concentration does not exceed the range specified in Table 1, then it can be determined that there is such substance in the sample. The characteristic ions and their abundance ratios of DEHA are shown in Table 2. For DEHA chromatogram see figure A.2.

Table 1 -- Maximum allowable deviation of relative ion abundance in qualitative confirmation

Relative ion abundance K /%	>50	20~50	10~20	≤10

DEHA concentration of 2mg/mL is obtained. Store the solution in the refrigerator for validity period of 1 month.

10.4.2 DEHA standard intermediate solution (Water, acidic, alcohol based food simulants analysis)

Draw 2.5mL DEHA standard reserve solution (10.4.1) with graduated pipette to 50mL volumetric bottle, dilute to the scale with n-hexane. The standard intermediate solution of DEHA is obtained with the concentration of 100.0mg/L. The storage method of the solution is the same as that of 10.4.1.

10.4.3 DEHA standard working solution (for water, acidic, alcohol based food simulants analysis)

Draw with graduated pipette and micro-syringe respectively 50μL, 0.2mL, 0.5mL, 1.0mL, 2.0mL, 5.0mL of the DEHA standard intermediate solution (10.4.2), place them in 6 10mL volumetric bottles, dilute with n-hexane, meter the volume and shake to mix well. The standard working solution of DEHA is obtained with concentration of 0.5mg/L, 2.0mg/L, 5.0mg/L, 10.0mg/L, 20.0mg/L, 50.0mg/L. The storage method is the same as that of 10.4.1.

10.4.4 DEHA standard reserve solution (for oil-based food simulants analysis)

Weigh DEHA 50mg (precision to 0.0001g) and put it into the 50mL beaker. Add oil-based food simulant 50g (precision to 0.0001g), dissolve and mix evenly. The concentration of the standard reserve solution is 1000mg/kg. Store the solution in the refrigerator for validity period of 1 month.

10.4.5 DEHA standard intermediate solution (for oil-based food simulants analysis)

Weigh 1.0g (precision to 0.01g) of DEHA standard reserve solution (10.4.4), put into the 10mL volumetric bottle. Weigh accurately 9.0g (precision to 0.0001g) of oil-based food simulant and put into the same bottle. Mix to obtain the DEHA standard intermediate solution with the concentration of 100mg/kg. The storage method of the solution is the same as that of 10.4.4.

10.4.6 DEHA standard working solution (for oil-based food simulants analysis)

Weigh respectively 0.2g, 0.5g, 1.0g, 2.0g, 4.0g, 5.0g (precision to 0.01g) of the DEHA standard intermediate solution (10.4.5), place them into 6 10mL volumetric bottles, add respectively 9.8g, 9.5g, 9.0g, 8.0g, 6.0g, 5.0g (precision to 0.0001g) of oil-based food simulant, shake to mix and obtain the standard working solution of DEHA with concentration of 2mg/kg, 5mg/kg, 10mg/kg, 20mg/kg, 40mg/kg, 50mg/kg respectively. Weigh respectively the

e) Determination method: select the ion monitoring mode, the monitoring ion range(m/z) of DEHA: 40~370, the DEHA characteristic ions of are: 129, 147, 112, 71, of which 129 is the quantitative ion;

f) Solvent delay by: 5 min.

12.5 Making of standard curve

In accordance with the determination conditions listed in 12.4, determine respectively the water, acid, alcohol, oil-based food simulant standard working solution (10.4.3, 10.4.6). Draw the standard curve using the DEHA concentration in the standard working solution as the horizontal coordinate, in unit of “milligram per liter(mg/L) (water, acid, alcohol-based food simulant), or milligrams per kilogram (mg/kg) (oil-based food simulant)", and the peak area of corresponding DEHA as the vertical coordinate.

12.6 Determination of sample solution

12.6.1 Qualitative determination

In accordance with the determination conditions listed in 12.4, inject the sample solution (12.2) and the blank solution (12.3) into gas chromatography mass spectrometer for determination. The qualitative method is the same as 5.6.1.

12.6.2 Quantitative determination

When determine the sample and blank solution, deduct the blank value to obtain the peak area of DEHA.

13 Expression of the analytic result

Obtain the concentration of DEHA in the sample solution from the standard curve. Calculate according to GB 5009.156 to obtain the migration amount of DEHA in food contact materials and products. Retain two-digit valid numbers for the results of calculation.

14 Precision

The absolute difference between the two independent measurements obtained under repetitive conditions shall not exceed 10% of the arithmetic average.