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NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA

GB 31604.27-2016

**National Food Safety Standard - Food contact
materials and products - Determination of ethylene
oxide and epoxy propane in plastics**

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National Food Safety Standard - Food contact materials and products - Determination of ethylene oxide and epoxy propane in plastics

1 Scope

This standard specifies the methods of determination of ethylene oxide and epoxy propane in food contact materials and products.

This standard is applicable to the determination of ethylene oxide and epoxy propane in plastic food contact materials and products.

The first method -

Headspace gas chromatography-mass spectrometry

2 Principle

After ethylene oxide or epoxy propane being extracted by N, N-dimethyl acetamide (DMAC), introduce it in the sample by headspace injection. Separate the ethylene oxide or epoxy propane from the internal standard diethyl ether and other components in the chromatographic column. Detect with spectrum detector, determine the quantity by internal standard method.

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

N, N-dimethyl acetamide (DMAC, C_4H_9NO , CAS: 127-19 -5): color spectrum pure. The area of the impurity peak with the same retention time of ethylene oxide or epoxy propane shall not exceed 1%.

3.2 Standard product

3.2.1 Ethylene oxide (C_2H_4O , CAS: 75-21-8): purity $\geq 99\%$, or a standard

2.0µg/mL, 4.0µg/mL; the concentrations of epoxy propane are respectively 0.05µg/mL, 0.1µg/mL, 0.2µg/mL, 0.5µg/mL, 1.0µg/mL, 2.0µg/mL. The storage condition of the solution is the same as 3.3.1.

3.3.5 Diethyl ether internal standard reserve solution

Weigh 50 mg (precision to 0.0001 g) of diethyl ether, put into a 50mL volumetric flask containing 5mL of DMAC. Meter with DMAC and obtain the reserve solution with concentration of 1 mg/mL. The storage condition of the solution is the same as 3.3.1.

3.3.6 Diethyl ether internal standard intermediate solution

Weigh 5mL of DMAC and put into a 10mL volumetric flask, then draw with graduated pipette 2.5mL of the diethyl ether internal standard reserve solution (3.3.5) and put into the volumetric flask. Meter with DMAC and obtain the diethyl ether internal standard intermediate solution. The concentration is 250.0µg/mL. The storage condition of the solution is the same as 3.3.1.

4 Instruments and equipment

4.1 Gas chromatography-mass spectrometer: equipped with electron bombardment ion source (EI) and headspace automatic sampler.

4.2 Micro-syringe: 10µL, 100µL, 1000µL;

4.3 Headspace bottle: 20mL, equipped with aluminum cap and butadiene rubber or silicone rubber spacer. The spacer shall be coated with polytetrafluoroethylene on the side contacting sample.

4.4 Analytical balance: sensitivity 0.0001g.

4.5 Mechanical oscillator.

5 Analytical steps

5.1 Sample treatment

The sample soluble in DMAC shall be weighed directly. The sample which is not soluble in DMAC shall be crushed to a particle size smaller than 1mm×1mm by cutting tools such as cryogenic grinder or scissors, and be weighed as soon as possible. When cutting the sample, shall not make it heated and soft.

5.2 Preparation of sample solution

e) Carrier gas: helium.

5.4.3 Mass spectrometry conditions

The mass spectrum conditions are as follows:

- a) Mass spectrum interface temperature: 280°C;
- b) Quadrupole rod temperature: 150°C;
- c) Ionization mode: EI;
- d) Ionization energy: 70 eV;
- e) Determination method: select ion monitoring mode, monitor ion range: m/z 15 ~ m/z 100. The qualitative ions of ethylene oxide are 15, 29, 43, 44, of which 44 is quantitative ion; the qualitative ions of epoxy propane are 28, 31, 43, 58, of which 58 is quantitative ion; the qualitative ions of diethyl ether are 31, 45, 59, 74, of which 74 is quantitative ion.

5.5 Making of standard curve

Draw with graduated pipette 5.0 mL of ethylene oxide and epoxy propane mixed standard solution (3.3.4) to 6 headspace bottles respectively. Add respectively with micro-syringe 10 μ L internal standard intermediate solution of diethyl ether (3.3.6) and seal with cover as soon as possible. Under the conditions provided in 5.4, put the above solutions respectively through headspace gas chromatography-mass spectrometer to determine. Use the concentrations of ethylene oxide (or epoxy propane) in the solution as abscissa in unit of microgram per milliliter (μ g/mL) and the peak area ratio of ethylene oxide (or epoxy propane) to diethyl ether as ordinate to draw standard curves respectively. See A.1 for standard chromatogram.

5.6 Determination of sample solution

Inject the sample solution and blank solution into the headspace gas chromatography-mass spectrometer to determine the peak area ratio of ethylene oxide (or epoxy propane) to diethyl ether. Obtain the concentration of ethylene oxide (or epoxy propane) in sample solution from the standard curve.

5.7 Qualitative

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 $\pm 2.5\%$ of the chromatographic peak retention time of the standard substance; and all the selected ions

10.2.1 Ethylene oxide (C₂H₄O, CAS: 75-21-8): purity ≥99%, or a standard substance certified by the State and granted the standard substance certificate.

10.2.2 Epoxy propane (C₃H₆O, CAS: 200-879-2): purity ≥99%, or a standard substance certified by the State and granted the standard substance certificate.

10.2.3 Diethyl ether (C₄H₁₀O, CAS: 60-29-7): purity ≥99.5%, or a standard substance certified by the State and granted the standard substance certificate. The area of the impurity peak with the same retention time of ethylene oxide or epoxy propane shall not exceed 1%.

10.3 Standard solution preparation

10.3.1 Ethylene oxide standard reserve solution

Weigh 50 mg (precision to 0.0001 g) of ethylene oxide, dissolve with DMAC, meter the volume to 50 mL, obtain the reserve solution with concentration of 1mg/mL. Store the solution at 4°C sealed and keep away from light. The validity period is 2 months.

10.3.2 Epoxy propane standard reserve solution

Weigh 50 mg (precision to 0.0001 g) of epoxy propane, dissolve with DMAC, meter the volume to 50 mL, obtain the reserve solution with concentration of 1mg/mL. Store the solution at 4°C sealed and keep away from light. The validity period is 2 months.

10.3.3 Mixed standard intermediate solution of ethylene oxide and epoxy propane

Draw 5.0mL of DMAC and put into 6 10mL volumetric flasks respectively. Draw with micro-syringe respectively 0μL, 40μL, 100μL, 200μL, 300μL, 400μL of the ethylene oxide standard reserve solution (10.3.1) and put into the above-mentioned 6 volumetric flasks respectively. Draw with micro-syringe 0μL, 40μL, 100μL, 200μL, 300μL, 400μL of the epoxy propane standard reserve solution (10.3.2) and put into the above-mentioned 6 volumetric flasks respectively. Meter with DMAC to the scale. Thus, obtain the mixed standard intermediate solution of ethylene oxide and epoxy propane. The concentrations of ethylene oxide are respectively 0μg/mL, 4μg/mL, 10μg/mL, 20μg/mL, 30μg/mL, 40μg/mL; the concentrations of epoxy propane are respectively 0μg/mL, 4μg/mL, 10μg/mL, 20μg/mL, 30μg/mL, 40μg/mL. The storage condition of the solution is the same as 10.3.1.

10.3.4 Diethyl ether internal standard reserve solution

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