NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 31604.29-2023

National food safety standard - Food contact materials and products - Determination of migration of acrylic acid and methacrylic acid and their esters

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1 Scope

This Standard specifies the determination method for the migration of acrylic acid and methacrylic acid and their esters in food contact materials and products.

Part 1 - Gas chromatography-mass spectrometry applies to the determination of migration of 18 kinds of acrylate and methacrylate in food contact materials and products, including 2-acrylic acid-2-methylpropyl ester, ethylacrylate, n-butyl acrylate, isopropyl acrylate, acrylic

acid n-propyl ester, 2-propenoic acid, 1,1-dimethylethyl ester, acrylic acid benzyl ester, 2-octyl acrylate, sec-butyl acrylate, ethyl methacrylate, isobutyl methacrylate, butyl methacrylate, 2-methyl-2-acrylic acid-1,1-dimethylethyl ester, phenyl methacrylate, 2-methyl-2-propyl acrylate, benzyl methacrylate, sec-butyl methacrylate and isopropyl methacrylate.

Part 2 - Liquid chromatography applies to the determination of migration of 6 kinds of acrylic acid, methacrylic acid and their esters in food contact materials and products, including acrylic acid, methacrylic acid, 2-hydroxyethyl acrylate, 2-hydroxyethyl-2-methyl-2-acrylate, methyl acrylate and methyl methacrylate.

Part 1 - Gas chromatography-mass spectrometry

2 Principle

The water-based food simulants as well as oil and fat food simulants obtained from the migration test are headspace sampled; the 95% (volume fraction) ethanol and isooctane simulants are directly sample after diluted with ethyl acetate; the target substance is separated in the gas chromatography capillary column, detected by mass spectrometry, and quantified by the external standard method.

3 Reagents and materials

Unless otherwise specified, all the reagents in this method are analytical reagents, and the water is grade-1 water specified by GB/T 6682.

3.1.5 Absolute ethanol (C2H6O).

3.1.6 95% ethanol (C2H6O).

3.2.2 Isooctane-ethyl acetate solution (1+4). Mix 25 mL of isooctane and 100 mL of

ethyl acetate evenly.

3.2.3 95% ethanol-ethyl acetate solution (1+4). Mix 25 mL of 95% ethanol and 100 mL of ethyl acetate evenly.

3.3 Standards

18 kinds of acrylate and methacrylate standards (see Appendix A). purity >=98%, or standard products certified by the country and awarded a standard substance certificate.

3.4.1 Standard stock solutions (1 000 mg/L). Accurately weigh 18 standards (accurate

to 0.1 mg). The weighing mass calculation method is 25 mg divided by the conversion coefficient, which is shown in Appendix A. Dissolve them in ethyl acetate respectively and then transfer them to the 25 mL volumetric flasks; use ethyl acetate to dilute to the mark and mix well. Transfer the solutions to brown glass containers and store them in the dark at 4 °C for 6 months.

3.4.2 Mixture of standard intermediate solution A (50 mg/L). Respectively and

accurately pipette 0.5 mL of the standard stock solutions (1 000 mg/L) into the 10 mL volumetric flasks; add methanol to adjust the volume to the mark; mix well. Transfer the solutions to brown glass containers and store them in the dark at 4 °C for 6 months.

3.4.3 Mixture of standard intermediate solution B (50 mg/L). Respectively and

accurately pipette 0.5 mL of the standard stock solutions (1 000 mg/L) into the 10 mL volumetric flasks; add ethyl acetate to adjust the volume to the mark; mix well. Transfer the solutions to brown glass containers and store them in the dark at 4 °C for 6 months.

3.4.4 Water-based food simulant series standard working solutions. Respectively and

accurately pipette 100 uL, 150 uL, 200 uL, 400 uL and 1 000 uL of the mixtures of standard intermediate solution A (50 mg/L) into the 100 mL volumetric flasks; use 50% (volume fraction) ethanol solution to adjust the volume. The concentrations of each target substance in the obtained standard working solutions are 0.050 mg/L, 0.075 mg/L,

0.10 mg/L, 0.20 mg/L, and 0.50 mg/L, respectively. Respectively pipette 10.0 mL of solutions of each concentration into the 20 mL glass headspace bottles to which 3.0 g of sodium chloride has been added; immediately use a spacer and an aluminum cap to seal them, for later testing. Use the same method to prepare a series of standard working solutions of the same concentration with water, 4% (volume fraction) acetic acid, 10% (volume fraction) ethanol, and 20% (volume fraction) ethanol. Prepare when necessary.

3.4.5 Oil and fat food simulant series standard working solutions. Respectively and

accurately weigh 5.00 g (accurate to 0.01 g) of olive oil simulant into five 20 mL glass headspace bottles; use a pipette to respectively pipette 30 uL, 80 uL, 100 uL, 150 uL, and 300 uL of the mixture of standard intermediate solution B (50 mg/L) into the 20 mL glass headspace bottles; immediately use a spacer and an aluminum cap to seal them;

vortex evenly, for later testing. The concentrations of each target substance in the obtained standard working solutions are 0.30 mg/kg, 0.80 mg/kg, 1.0 mg/kg, 1.5 mg/kg, and 3.0 mg/kg, respectively. Prepare when necessary.

3.4.6 Isooctane series standard working solutions. Respectively pipette 50 uL, 150 uL, 200 uL, 300 uL and 600 uL of the mixture of standard intermediate solution B (50 mg/L)

into the 10 mL volumetric flasks; use isooctane-ethyl acetate solution (1+4) to adjust the volume to the mark; mix well. The concentrations of each target substance in the obtained standard working solution are 0.25 mg/L, 0.75 mg/L, 1.0 mg/L, 1.5 mg/L and

3.0 mg/L, respectively. Prepare when necessary.

3.4.7 95% (volume fraction) ethanol series standard working solutions. Respectively pipette 50 uL, 150 uL, 200 uL, 300 uL, and 600 uL of the mixture of standard intermediate solution B (50 mg/L) into the 10 mL volumetric flasks; use 95% ethanol-ethyl acetate solution (1+4) to adjust the volume to the mark. The concentrations of each target substance in the obtained standard working solution are 0.25 mg/L, 0.75 mg/L, 1.0 mg/L, 1.5 mg/L and 3.0 mg/L, respectively. Prepare when necessary.

4.1 Gas chromatograph-mass spectrometer, equipped with electron impact source (EI)

and headspace sampler.

4.3 Electronic balance. The sensitivity is 0.1 mg and 0.01 g, respectively.

d) Oscillation speed. 500 r/min.

e) Injection volume. 250 uL.

5.4.2 The reference conditions for gas chromatography mass spectrometry

measurement are as follows.

a) Chromatographic column. polyethylene glycol capillary column. column length

30.0 m, inner diameter 0.25 mm, film thickness 0.25 um, or an analytical column with equivalent performance.

b) Programmed temperature rise. initial temperature 35 °C, kept for 8 min; increased to 60 °C at 5 °C/min, kept for 3 min; increased to 160 °C at 20 °C/min, kept for

2 min; increased to 220 °C at 40 °C/min, kept for 3 min.

c) Inlet temperature. 290 °C.

d) Mass spectrometry joint temperature. 280 °C.

e) Ion source temperature. 230 °C;

f) Carrier gas. He (purity >99.999%), constant flow 1.0 mL/min.