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**Test Method of Adsorption Resin
for Food Industrial Use**

食品工业用吸附树脂产品测定方法

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Test Method of Adsorption Resin for Food Industrial Use

1 Scope

This Standard specifies the test method of adsorption resin for food industrial use.

This Standard applies to the determination of appearance, heavy metals and organic residues in adsorption resin for food industrial use that is copolymerized under the action of initiator, of which, the polymeric monomer is styrene, divinylbenzene, acrylics, acrylonitrile, and the pore-foaming agent is toluene or liquid paraffin.

2 Method summaries

2.1 Heavy metal detection

In the weak-acidic solution, sodium sulfide precipitates heavy metal ions into the corresponding sulfides; the resulting color is visually compared to the standard color.

2.2 Organic residue detection

The organic residue in the sample is ultrasonically extracted by absolute ethanol; it is injected into the gas chromatograph and carried by the carrier gas into the column for separation; the effluent is detected by a hydrogen flame ionization detector; the content of organic residue is calculated according to the external standard method.

3 Reagents and materials

WARNING: Some of the reagents which are used in this test method are toxic or corrosive; therefore, some test procedures may cause dangerous conditions, and the operator shall take appropriate safety and health measures.

3.1 Hydrochloric acid: analytical reagent.

3.2 Acetic acid solution: 1 + 4.

3.3 Sodium sulfide solution: 100 g/L.

Weigh 2.0 g of sample (dry basis) which is dried at $105^{\circ}\text{C} \pm 2^{\circ}\text{C}$ for 2 h; accurate to 0.01 g; place it in the evaporating dish; carbonize it and then burn it at $500^{\circ}\text{C} \pm 25^{\circ}\text{C}$ until the complete ashing; cool it; then, add 2 mL of hydrochloric acid; evaporate it to dryness on a water bath; cool it; then, use 4 mL of acetic acid solution and 20 mL of water to dissolve the residue. Carry out filtration as necessary; use 5 mL of water to wash the residue on the filter paper each time, and wash it for three times in total. Combine the filtrate and the washing solution; dilute to 50 mL as the test solution. Take 25 mL of test solution to a 50 mL colorimetric tube, which is the tube A.

5.2.2 Preparation of lead (Pb) standard colorimetric solution

Transfer 2 mL of acetic acid solution into a 50 mL colorimetric tube (matching tube A); add 1.50 mL of lead standard solution; add water to 25 mL, which is the tube B.

5.2.3 Determination

Add two drops of freshly-prepared sodium sulfide solution to tubes A and B respectively; mix quickly; shake well; place them in the dark for 5 min; observe under a white background. If the dark color of the tube A is not deeper than the tube B, they are judged as qualified.

5.3 Determination of organic residue

5.3.1 Preparation of samples

Place 10 mL of the sample in a centrifuge tube; centrifuge at $2000\text{ r/min} \pm 200\text{ r/min}$ for 5 min. Take the upper layer of the sample from which the surface free water has been removed; weigh 2 g (wet weight, accurate to 0.000 1 g); place it in a 50 mL brown volumetric flask; add 10 mL of absolute ethanol; cover with a stopper; use a sealing film to seal; ultrasonically oscillate (add an appropriate amount of ice cubes to the ultrasonic oscillator to reduce the temperature) for 20 min; let stand for 10 min; take the supernatant; use a filter to filter for later use.

5.3.2 Preparation of standard solution

5.3.2.1 Preparation of mixed standard solution mother solution

According to the order of styrene, o-xylene, m-xylene, p-xylene, chlorobenzene, toluene, methyl methacrylate, divinylbenzene, 1,2-dichloroethane, benzene, acrylonitrile, weight 0.1 g of each of the above standards (accurate to 0.000 1 g); place in a 100 mL brown volumetric flask; use absolute ethanol to dilute to the mark; shake well.

Appendix A

(Informative)

Organic residue determination reference gas chromatography operating conditions

According to the used gas chromatograph, adjust the appropriate temperature and flow rate to achieve the proper separation effect. Reference chromatographic conditions are shown as below:

- a) column: polyethylene glycol 20M column (30 m × 0.25 mm × 0.25 μm);
- b) temperature of the injector: 250°C;
- c) temperature of the detector: 280°C;
- d) column temperature: 35°C (7 min) $\xrightarrow{10^{\circ}\text{C}/\text{min}}$ 150°C (4 min);
- e) carrier gas: high-purity nitrogen; line speed of 30 cm/s (that is, column flow of 1.25 mL/min);

Injection method: split injection, split ratio: 1 : 50;

Injection volume: 1.0 μL.

The gas chromatogram of 11 organic residues is shown in Figure A. 1.

Note: Since the separation effect that is achieved by different instruments under the same chromatographic conditions cannot be exactly the same, the chromatographic conditions can be adjusted according to the actual instrument used, as long as the appropriate separation effect is achieved.

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