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Feed additive - Benzoic acid

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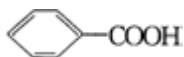
Feed additive – Benzoic acid

1 Scope

This Standard specifies the requirements, test methods, inspection rules, marking, packaging, transport, storage and guarantee period of feed additive benzoic acid.

This Standard applies to feed additive benzoic acid which is made through catalytic oxidation and purification using petroleum toluene as the raw material. This product is mainly used as the agents for corrosion resistance, mould resistance and acidification of feed.

Molecular formula: $C_7H_6O_2$

Structural formula: 

Relative molecular mass: 122.12 (based on the International Relative Atomic Mass 2001)

2 Normative references

The provisions in following documents become the provisions of this Standard through reference in this Standard. For dated references, the subsequent amendments (excluding corrigendum) or revisions do not apply to this Standard, however, parties who reach an agreement based on this Standard are encouraged to study if the latest versions of these documents are applicable. For undated references, the latest edition of the referenced document applies.

GB/T 601, *Chemical reagent – Preparations of standard volumetric solutions*

GB/T 602, *Chemical reagent – Preparations of standard solutions for impurity determination*

GB/T 603, *Chemical reagent – Preparations of reagent solution for use in test method*

GB/T 606, *Chemical reagent – General method for the determination of water – Karl Fischer method*

GB/T 617, *Chemical reagent – General method for the determination of melting*

V – the volume of sodium standard volumetric solution consumed by sample, in mL;

C – the concentration of sodium standard volumetric solution, in mol/L;

m – the weight of sample, in g;

M – the molar mass of benzoic acid, in g/mol ($M = 122.12$).

The result of calculation is rounded off to two significant digits.

Take the arithmetic mean value of two results determined in parallel as the result of determination; the absolute difference of the two results determined in parallel shall not be greater than 0.2%.

4.3 Melting point determination

As specified in GB/T 617.

4.4 Easy oxide test

4.4.1 Principle

The easy oxide in sample reacts with potassium permanganate; after reaction, it is the end of reaction when the solution appears the pink colour of potassium permanganate solution; use the volume of potassium permanganate standard volumetric solution consumed to control the amount of easy oxide.

4.4.2 Reagents

4.4.2.1 Sulfuric acid.

4.4.2.2 Potassium permanganate standard volumetric solution: $c(1/5\text{KMnO}_4) = 0.02$ mol/L.

4.4.3 Analytical procedures

Add 1.5 mL of sulfuric acid to 100 mL of water; add dropwise potassium permanganate standard volumetric solution until pink while boiling; maintain for 30 without fading. Weigh 1.0 g of sample, accurate to 0.001 g; dissolve in the above-mentioned solution while hot; at about 70°C, use potassium permanganate standard volumetric solution for titration until pink; maintain for 15 s without fading; potassium permanganate standard volumetric solution used in sample titration shall not exceed 0.5 mL.

4.5 Determination of heavy metal (in terms of Pb) content

4.5.1 Principle

Use ethanol to dissolve sample; heavy metal and sodium sulfide generate a brown

4.6.2.4 Potassium iodide.

4.6.2.5 Stannous chloride: 400 g/L.

4.6.2.6 Arsenic standard stock solution: 0.1 mg/mL.

4.6.2.7 Arsenic standard working solution: 1.0 µg/mL. Prepare immediately prior to use. Use a pipette to transfer 1.0 mL of arsenic standard stock solution (4.6.2.6); place in a 100 mL volumetric flask before using water to dilute to scale; mix up.

4.6.2.8 Lead acetate cotton.

4.6.2.9 Mercury bromide test paper.

4.6.3 Apparatus

Arsenic determination instrument.

4.6.4 Analytical procedures

Weigh 1 g of sample, accurate to 0.001 g; place in a 30 mL porcelain crucible; after low-temperature carbonization to smokeless, transfer to a high-temperature furnace for carbonization at 550°C for 3.5 ~ 4 h. Take out for cooling; slowly add 10 mL of hydrochloric acid solution; after violent reaction, boil the solution and transfer to an arsenic determination bottle; add water to dilute to about 40 mL. Add 6 mL of hydrochloric acid; shake up; add 1 g of potassium iodide and 5 drops of stannous chloride solution; shake up; place aside for 10 min. Add 3 g of arsenic-free zinc granule to the arsenic determination bottle; immediately put stopper on the arsenic determination tube loaded with lead acetate cotton and mercury bromide test paper and store in a dark place for 1 h. Take out mercury bromide test paper to observe and the colour of arsenic stains of sample shall not be darker than that of the standard.

The standard solution is prepared by using a pipette to transfer 2 mL of arsenic standard working solution (4.6.2.7), starting from “add water to dilute to about 40 mL” and processing using the same method and at the same time as sample.

4.7 Determination of chloride (in terms of Cl) content

4.7.1 Principle

Sample contains organic chlorides (aromatic chlorides) and inorganic chlorides; organic chlorides are converted into calcium chloride by addition of calcium carbonate and burning at high temperature, before it is dissolved into the sample solution along with inorganic chlorides. Under acidic conditions, chloride ion in the sample solution reacts with silver nitrate solution to generate a silver chloride precipitation and the turbidity generated shall not be greater than that of the standard turbidimetric solution.

standard substance; dissolve in 30 mL of methanol before adding 1% acetic acid dropwise to 100 mL.

4.9.2.5 Phthalic acid standard working solution, 1.0 µg/mL: take 1.0 mL of phthalic acid standard stock solution (4.9.2.4); add the mixed solution of methanol and 1% acetic acid dropwise to 100 mL.

4.9.3 Apparatus

Liquid chromatograph, which is equipped with an ultraviolet detector.

4.9.4 Analytical procedures

Weigh 1 g of sample, accurate to 0.0001 g; dissolve in 20 mL of methanol before adding acetic acid dropwise to 50 mL as the sample test solution. Take respectively 20 µL of phthalic acid standard working solution and sample test solution as sample.

4.9.5 Operating conditions for apparatus

Chromatographic column: C₁₈ column, 4.6 mm × 250 mm.

Column temperature: 40°C.

Moving phase: mixed solution of methanol + 1% acetic acid = 3 + 7.

Determination wavelength: 228 nm.

Flow velocity: 1 mL/min.

4.9.6 Result calculation

The content of phthalic acid of sample W_2 , which is expressed as mass fraction (mg/kg), is calculated in accordance with Equation (2):

$$W_2 = \frac{C_{st} \times A_s \times V}{A_{st} \times m} \dots\dots\dots (2)$$

where:

A_s – the peak area of the sample test solution;

A_{st} – the peak area of the phthalic acid standard solution;

C_{st} – the concentration of the phthalic acid standard solution, in µg/mL;

V – the volume of the sample test solution, in mL;

m – the weight of sample, in g.

The total content of biphenyl substances in sample to be tested, W_3 , which is expressed as mass fraction (mg/kg), is calculated in accordance with Equation (5):

$$W_3 = W_{BP} + W_{2MBP} + W_{3MBP} + W_{4MBP} + W_{BB} \dots\dots\dots (5)$$

where:

W_{BP} – the content of component BP of biphenyl substances in the sample test solution, in mg/kg;

W_{2MBP} – the content of component 2MBP of biphenyl substances in the sample test solution, in mg/kg;

W_{3MBP} – the content of component 3MBP of biphenyl substances in the sample test solution, in mg/kg;

W_{4MBP} – the content of component 4MBP of biphenyl substances in the sample test solution, in mg/kg;

W_{BB} – the content of component BB of biphenyl substances in the sample test solution, in mg/kg.

The result of calculation is rounded off to three significant digits.

5 Inspection rules

5.1 Inspection types

Product inspection includes exit-factory inspection and type inspection.

5.2 Batch and sampling

1,000 kg is deemed as an inspection batch: take 2% of the bag number of each batch as sample, not less than 3 bags for a small batch. From the bags selected, use a sampling tool to stretch $\frac{3}{4}$ into each bag and take not less than 100 g of sample. Mix up sample taken; reduce sample using the quartering method; load in a clean, dry, ground-glass wide-mouth bottle (with the capacity not less than 250 mL); use wax or sealing film to seal tight. Put on label; indicate manufacturer name, manufacturing date, product name, batch number and sampling date; deliver to laboratory for analysis on a timely basis.

5.3 Exit-factory inspection

5.3.1 Before delivery, exit-factory inspection shall be carried out for each batch; products can only be delivered after they pass the inspection.

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