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Surface active agents - Determination of quinoline content in dispersing agents

表面活性剂 分散剂中喹啉含量的测定

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Surface active agents - Determination of quinoline content in dispersing agents

1 Scope

This Standard specifies the determination method for quinoline content in surface active dispersing agents.

This Standard is applicable to various surface-active dispersing agents that are produced from coal chemical raw materials.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 6682, *Water for analytical laboratory use - Specification and test methods*

GB/T 8170, *Rules of rounding off for numerical values & expression and judgement of limiting values*

GB/T 9722-2006, *Chemical reagent - General rules for the gas chromatography*

3 Principle

Disperse and dissolve the sample in water. Use ethyl acetate to extract the quinoline in it. Use gas chromatography or gas chromatography-mass spectrometry to detect. Use peak area external standard method to quantify.

4 Reagents

4.1 General provisions

Unless otherwise stated, use analytical reagents that meet national standards. The test water shall meet the specifications for grade three water in GB/T 6682.

5.7 Organic phase needle filter

The specification is 13mm × 0.45μm.

5.8 Pipette

The capacity is 20mL.

6 Determination methods

6.1 Sample pre-treatment

Weigh 0.5g of sample (to the nearest of 0.0001g). Place in the extractor (see 5.6). Add 20mLof water. Shake vigorously till dissolved or fully wetted and dispersed. Use the pipette (see 5.8) to accurately add 20mL of ethyl acetate (see 4.2). Shake the extractor for 0.5min to make the aqueous phase and the organic phase fully contact. It may also use ultrasonic-assisted extraction. Conduct static layering (if the emulsification is serious and the layering is slower, it can be centrifuged at 3000r/min for 10min). Take the supernatant. For the sample solution that is filtered through the organic phase needle filter (see 5.7), use gas chromatograph or GC/MS to analyze. If the sample concentration exceeds the linear range of the instrument, it needs to dilute the sample to the linear range.

6.2 Gas chromatography

6.2.1 Gas chromatography analysis conditions

Since the test results depend on the instrument used, it is not possible to give general parameters for chromatographic analysis. It may choose the best analysis conditions according to different equipment. Use the following parameters that have been proved suitable for the test. The parameters are as follows:

- Chromatographic column: HP-5, 30m × 0.32mm × 0.25μm, or equivalent;
- Carrier gas: nitrogen, constant flow, flow rate of 1mL/min;
- Inlet temperature: 270°C;
- Detector (FID) temperature: 300°C;
- Hydrogen flow: 40mL/min;
- Air flow: 400mL/min;
- Compensation gas: nitrogen, 30mL/min;

milligrams per kilogram (mg/kg):

$$X = \frac{(A - b) \times V}{a \times m} \times F \qquad \cdots\cdots (2)$$

Where,

A - Peak area of quinoline in sample solution;

V - Volume of sample solution, in milliliters (mL);

a, b - Standard curve constants;

m - Sample mass, in grams (g);

F - Dilution factor.

The calculation result is rounded off to the integer according to GB/T 8170.

6.3 Gas chromatography-mass spectrometry (arbitration method)

6.3.1 GC-MS analysis conditions

Since the test results depend on the instrument used, it is not possible to give general parameters for chromatographic analysis. It may choose the best analysis conditions according to different equipment. Use the following parameters that have been proved suitable for the test. The parameters are as follows:

- Chromatographic column: HP-5MS, 30m × 0.25mm × 0.25μm, or equivalent;
- Carrier gas: nitrogen, constant flow, flow rate of 1mL/min;
- Inlet temperature: 260°C;
- Auxiliary interface temperature: 280°C;
- Injection volume: 1μL;
- Injection method: injection without flow division;
- Heating program: maintain at 80°C for 2min, rise at 10°C/min to 180°C for 2min;
- Solvent delay: 4min;
- Ion source temperature: 230°C;

Where,

A - Quinoline quantitative ion peak area;

c - Quinoline concentration, in milligrams per liter (mg/L);

a, b - Standard curve constants.

After each instrument maintenance or replacement of parts, it shall redraw the standard curve. It is recommended that before each test of sample solution, use the quinoline standard solution with a known concentration to verify the curve. When the error exceeds 5%, it shall redraw the standard curve.

6.3.4 Sample determination steps

Analyze the sample solution that has been well processed according to the chromatographic conditions (see 6.3.1). Record the quinoline quantitative ion peak area A in the sample solution.

6.3.5 Result calculation

The quinoline content in surface active agent is calculated according to formula (4).

The quinoline content X in surface active agent is calculated according to formula (4), in milligrams per kilogram (mg/kg):

$$X = \frac{(A - b) \times V}{a \times m} \times F \qquad \cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots (4)$$

A - Peak area of quinoline quantifier ion in sample solution;

V - Volume of extract liquid, in milliliters (mL);

a, b - Standard curve constants;

m - Sample mass, in grams (g);

F - Dilution factor.

The calculation result is rounded off to the integer according to GB/T 8170.

7 Detection limit, recovery rate and precision

7.1 Detection limit

GC-FID detection limit is 20mg/kg. GC-MS detection limit is 5mg/kg.

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