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Replacing GB/T 33372-2016

Limit of volatile organic compounds content in adhesive

胶黏剂挥发性有机化合物限量

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Standardization Administration of the People's Republic of China.

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Limit of volatile organic compounds content in adhesive

1 Scope

This Standard specifies the limit requirements, test methods, inspection rules and packaging marks of volatile organic compounds (hereinafter referred to as VOC) content in adhesive under specified conditions.

This Standard applies to the limitation of volatile organic compounds content in solvent-based, water-based and bulk adhesives.

This Standard does not apply to:

-- adhesives that are used as intermediates or as raw materials for production without entering the circulation field;

-- adhesives for testing or evaluation in any research and development, quality assurance or analytical laboratory;

-- urea formaldehyde, phenolic formaldehyde, melamine formaldehyde adhesives;

-- special functional surface treatment agent that is applied when materials are bonded.

2 Normative references

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

GB/T 2793, The method for determination of nonvolatile content of adhesives

GB/T 2943, Terms of adhesive

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

GB/T 13354, Test method for density of adhesives in fluid form - Method of weight cup

GB 19340, Adhesives for footwear and case and bag

6 Test method

6.1 Sampling

Adhesive product sampling shall be carried out in accordance with GB/T 20740.

6.2 Determination of VOC content

6.2.1 The determination of VOC content in solvent-based adhesive shall be performed according to Appendix A.

6.2.2 The VOC content in water-based adhesive shall be determined in accordance with the provisions of Appendix D.

6.2.3 The VOC content in bulk adhesive shall be determined in accordance with the provisions of Appendix E.

6.2.4 The VOC content of ethyl α-cyanoacrylate instantaneous adhesives shall be determined according to the method that is specified in Appendix B of HG/T 2492-2018.

6.2.5 The VOC content can also be calculated according to the composition of the adhesive. When the calculation is not possible or the calculation and measurement results do not match, the measurement result shall prevail.

7 Inspection rules

7.1 Inspection items

7.1.1 All requirements that are listed in this Standard are type inspection items.

7.1.2 Under normal production conditions, type inspection shall be carried out at least once a year.

7.1.3 Type inspection shall be carried out in any of the following cases:

-- when a new product is initially finalized;

-- when the product is produced off-site;

-- when there are major changes in production formulas, processes, and key raw material sources;

-- when production is resumed after 3 months of suspension.

7.2 Product sampling

Appendix A

(Normative)

Determination of VOC content in solvent-based adhesive

A.1 Overview

Place an appropriate amount of the adhesive in a blast drying oven at a constant temperature; measure the amount of the volatiles in the adhesive within a prescribed time. Use the gas chromatography to determine the content of low photochemical reaction compounds; use Karl Fischer method or gas chromatography to determine the water content in the adhesive; subtract the water content and the acetone, methyl acetate and dimethyl carbonate amount from the amount of the volatiles in the adhesive, to obtain the VOC content in the adhesive.

A.2 Test steps

A.2.1 General

Perform two parallel determinations for all tests.

A.2.2 Density

Prepare the mixed sample according to the matching requirements that are expressed by the adhesive product; after stirring well, measure the density of the sample according to the method that is specified in GB/T 13354, at a test temperature of (23 ± 2) °C.

A.2.3 Volatile content of the sample

A.2.3.1 One-component sample

Determine the non-volatile content of the sample according to the method that is specified in GB/T 2793.

A.2.3.2 Multi-component sample

According to the matching requirements that is expressed by the adhesive product, take about 2 g of the mixed sample; stir it well quickly; then, measure the non-volatile content of the sample within 5 minutes according to the method that is specified in GB/T 2793.

A.2.3.3 Volatile content of the sample

Calculate the volatile content of the sample according to Formula (A.1):

B.3.1.4 Dropping bottle: 30 mL.

B.3.1.5 Magnetic stirrer.

B.3.1.6 Beaker: 100 mL.

B.3.1.7 Petri dish.

B.3.2 Reagent

B.3.2.1 Distilled water: in accordance with the requirements of grade-3 water in GB/T 6682.

B.3.2.2 Karl Fischer reagent: use appropriate reagents (for samples that do not contain aldehyde and ketone compounds, the main components of the reagents are iodine, sulfur dioxide, methanol, and organic bases. For samples that contain aldehyde and ketone compounds, use special reagents for aldehyde and ketone, whose main components are iodine, imidazole, sulfur dioxide, 2-methoxyethanol, 2-chloroethanol and trichloromethane.

B.3.3 Test steps

B.3.3.1 Concentration calibration of Karl Fischer titrant

Add fresh Karl Fischer solvent into the titration cup of the titrator until the liquid surface covers the electrode tip; use Karl Fischer titrant to titrate to the end point (drift value <10 μg/min). Use a micro-syringe to inject 10 μL of distilled water into the titration cup; use the weight reduction method to obtain the mass of the water (accurate to 0.1 mg); enter the mass into the titrator; use Karl Fischer titrant to titrate to the end point; record the instrument displayed calibration results.

Repeat the calibration until the difference between the two adjacent calibration values is less than 0.01 mg/mL; find the average value of the two calibrations; enter the calibration results into the titrator.

When the relative humidity of the test environment is less than 70%, it shall be calibrated once a week; when the relative humidity is greater than 70%, it shall be calibrated twice a week; if necessary, calibrate at any time.

B.3.3.2 Sample processing

If the viscosity of the to-be-tested sample is large and cannot be dispersed well in Karl Fischer solvent, perform an appropriate dilution for the sample. In the beaker, weigh 20 g (accurate to 1 mg) of well-stirred sample; then, add about 20% of distilled water to the beaker; accurately record the sample amount and water addition. Cover a petri dish on the beaker; stir on a magnetic stirrer for 10 min ~ 15 min. Pour the diluted sample into a dropping bottle for later use.

Appendix C

(Normative)

Determination of acetone, methyl acetate and dimethyl carbonate

C.1 Principle

Directly inject the sample into the gas chromatograph after dilution; use chromatographic column to separate it; then, use a hydrogen flame ionization detector to detect; use the internal standard method to quantify.

C.2 Materials and reagents

C.2.1 Carrier gas: nitrogen, purity ≥ 99.995%.

C.2.2 Fuel gas: hydrogen, purity ≥ 99.995%.

C.2.3 Combustion-supporting gas: air.

C.2.4 Auxiliary gas (separate purge and makeup gas): nitrogen that has the same properties as the carrier gas.

C.2.5 Internal standard: a compound that does not exist in the sample; the compound can be completely separated from other components on the chromatogram; the purity is at least 99% or known. Such as: n-heptane, n-pentane and so on.

C.2.6 Calibration compounds: acetone, methyl acetate, dimethyl carbonate, whose purity is at least 99% or known.

C.2.7 Dilution solvent: the organic solvent that is used to dilute the sample (it does not contain any substance that interferes with the test); the purity is at least 99% or known. Such as: ethyl acetate, n-hexane, etc.

C.3 Instruments and apparatuses

C.3.1 Gas chromatograph, which has the following configuration:

-- inlet of the split device; the lining of the vaporization chamber can be replaced;

-- programmed heating controller;

-- detector, flame ionization detector;

f) injection volume: 1.0 μL.

D.5.2 Chromatographic condition 2:

a) chromatographic column (confirmation column): polyethylene glycol capillary column, 30 m × 0.25 mm × 0.25 μm;

b) temperature of the sample injector: 240 °C;

c) detector: FID, temperature of 250 °C;

d) column temperature: programmed heating; keep the initial temperature of 60 °C for 1 min; then, increase to 240 °C at 20 °C /min; keep it for 20 min;

e) split ratio: split injection, split ratio can be adjusted;

f) injection volume: 1.0 μL.

D.6 Test steps

D.6.1 General

Perform two parallel determinations for all tests. Multi-component samples are the same as A.2.3.2.

D.6.2 Density determination

Perform according to the method that is specified in GB/T 13354.

D.6.3 Optimization of chromatographic parameters

According to the gas chromatography conditions, a known calibration compound shall be used to optimize the instrument each time, so as to make the instrument's sensitivity, stability and separation effect at the best state.

D.6.4 Qualitative analysis

Qualitatively identify the presence of calibration compounds in D.3.6.

Preferentially select GC-MS or GC- (FT-IR); perform the determination according to the given gas chromatography test conditions. The column that is specified by GC-FID and D.4d) can also be used. Respectively record the chromatogram of calibration compounds on the two chromatographic columns (the polarity of the two selected columns shall be as large as possible, such as 6% cyanopropylphenyl and 94% polydimethylsiloxane capillary column, polyethylene glycol capillary) according to the given gas chromatography test conditions. Under the same chromatography test conditions, after making the chromatogram of the test sample, compare and characterize it.

Appendix E

(Normative)

Determination of VOC content in bulk adhesive

E.1 Overview

This appendix specifies the determination of the VOC content in bulk adhesives by the oven method.

E.2 Principle

E.2.1 Place an appropriate amount of the adhesive in a blast drying oven at a constant temperature; measure the amount of the volatiles of the adhesive within a prescribed time.

E.2.2 For reactive bulk adhesives (such as acrylates, etc.), give the specified reaction time (provided by the product supplier); then, measure the VOC content of the adhesive as E.2.1, so as not to count the reactive monomer into the VOC content.

E.2.3 For thermoplastic (or thermosetting) bulk adhesive, weight the sample; operate according to the actual sizing (or vulcanization) conditions that are provided by the product supplier; then, measure the VOC content in the adhesive as E.2.1.

E.3 Test steps

E.3.1 General

Perform two parallel determinations for all tests.

E.3.2 Volatile content of the sample

E.3.2.1 Volatile content in general bulk adhesive

same as A.2.3.

E.3.2.2 Volatile content in reactive bulk adhesive

E.3.2.2.1 For reactive bulk adhesive product, take about 2 g of the sample; after curing according to the curing conditions of the product, determine the nonvolatile content of the sample according to the method that is specified in GB/T 2793; calculate the volatile content same as A.2.3.3.

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