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# Test Methods for Harmful Substances in Building Waterproofing Materials

建筑防水材料有害物质试验方法

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# Test Methods for Harmful Substances in Building Waterproofing Materials

Warning - This Document does not point out all possible safety issues, the user is responsible for taking appropriate safety and health measures and ensuring compliance with the relevant regulations.

# 1 Scope

This Document specifies the classification and the test items of harmful substances, test methods and test reports of test methods for harmful substances in building waterproofing materials.

This Document applies to construction waterproofing sheets, waterproofing coatings and other waterproofing materials.

### 2 Normative References

The provisions in following documents become the essential provisions of this Document through reference in this Document. For the dated documents, only the versions with the dates indicated are applicable to this Document; for the undated documents, only the latest version (including all the amendments) is applicable to this Document.

GB/T 328.4-2007 Test Methods for Building Sheets for Waterproofing - Part 4: Bitumen Sheets for Waterproofing - Thickness and Mass per Unit Area

GB/T 1725 Paints Varnishes and Plastics - Determination of Non-Volatile-Matter Content

GB/T 6682-2008 Water for Analytical Laboratory Use - Specification and Test Methods

GB/T 6750 Paints and Varnishes - Determination of Density - Pycnometer Method

GB/T 8928-2008 Standard Test Method for Density of Semi-Solid and Solid Asphalt

GB/T 18446 Binders for Paints and Varnishes - Determination of Monomeric Diisocyanates in Isocyanate Resins

GB 18582-2020 Limit of Harmful Substances of Architectural Wall Coatings

GB/T 23985-2009 Paints and Varnishes - Determination of Volatile Organic Compound (VOC) Content - Difference Method

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it is a sheet-like waterproof material that is processed by calendering or extrusion molding with rubber as the main base material, adding fillers, plasticizers, vulcanizing agents, antioxidants, stabilizers and other additives.

NOTE: There may be structures such as reinforcing materials, backing materials and surface bonding materials, etc.

#### 3.4 Water-borne waterproof coating

It is a waterproof material that uses water as the dispersion medium, and the water volatilizes and solidifies into a film.

NOTE: Including single-liquid type, double-liquid type, liquid-powder type.

#### 3.5 Reactive waterproof coating

A waterproof material that reacts and cures with moisture in the air or cures to form a film by reaction between product components.

NOTE 1: Including single-component type and multi-component type.

NOTE 2: The solid content of reactive waterproof coatings is generally no less than 85%.

#### 3.6 Solvent-borne waterproof coating

A waterproof material that is cured into a film by solvent evaporation or by reaction.

NOTE: The solid content of solvent-based waterproof coatings is generally less than 85%.

#### 3.7 Hot melt waterproof coating

The waterproof materials of single-component type, solid or viscous body at room temperature, heating and melting for construction.

NOTE: The solid content of hot melt waterproof coatings is generally no less than 98%.

#### 3.8 Dry powder waterproof material

The product is a dry powder, a waterproof material that is used after adding water and mixing evenly.

#### 3.9 Target volatile organic compounds

Single volatile organic compound that is selected and analyzed in the product.

NOTE: It can be formaldehyde, benzene, toluene, o-xylene, m-xylene, p-xylene, styrene, ethylbenzene, n-butyl acetate, n-undecane and n-tetradecane, etc.

[Source: JG/T 528-2017, 3.3, with modifications]

 $M_{\rm i}$  – mass fraction of component *i*.

- **5.2.3** The reactive waterproof coating and solvent-borne waterproof coating shall be tested according to the provisions of GB/T 23985-2009; and the results shall be calculated according to 8.3 of GB/T 23985-2009. The non-volatile matter content is tested according to the provisions of GB/T 1725. The sample is mixed uniformly according to the proportion and weighs about 1 g of the sample. The high-temperature treatment test condition keeps the constant temperature at  $(105\pm2)$  °C for 1 h; thereof, after the specimen preparation and sampling weighing of the non-volatile matter content of the reactive waterproof coating, then take the high temperature treatment after standing for 24h under the conditions of  $(23\pm2)$  °C and relative humidity of  $(50\pm5)$  %. The density shall be tested according to the provisions of GB/T 6750. Moisture is not measured, and the moisture content is set to zero. The fast-curing reactive waterproof coatings are tested for each component separately, and the results are calculated according to the Formula (1) in combination with the ratio.
- **5.2.4** The hot melt coating shall be tested according to the provisions of GB/T 23985-2009, and the results shall be calculated according to 8.3 of GB/T 23985-2009. The non-volatile matter content is tested according to the provisions of GB/T 1725. Take about 1 g of the specimen, and the high temperature treatment test condition is that keep constant temperature at  $(130\pm2)$  °C for 3h. The density shall be tested at 23°C according to the requirements for semi-solid petroleum asphalt specified in GB/T 8928-2008. Moisture is not measured, and the moisture content is set to zero.
- **5.2.5** The dry powder waterproof material shall be tested according to the putty method in 6.2.1 of GB 18582-2020; only the powder shall be tested, and it shall be taken as the test result.

#### 5.3 Content of benzene series

- **5.3.1** The water-borne waterproof coating shall be tested according to the provisions of Method-B in GB/T 23990-2009; and the results shall be calculated according to Formula (1) in combination with the ratio. The double-liquid fast-curing waterproof coating (rubber asphalt, water-borne non-curing) is only tested for the main component, which is the test result.
- **5.3.2** The reactive, solvent-borne and hot melt coatings shall be tested according to the provisions of Method-A in GB/T 23990-2009; and the results shall be calculated according to Formula (1) in combination with the ratio.
- **5.3.3** The dry powder waterproof material shall be tested according to the provisions of Method-B in GB/T 23990-2009; only the powder shall be tested, which shall be taken as the test result.

#### 5.4 Total content of glycol ethers and their ether esters

The total content of glycol ethers and their ether esters (ethylene glycol methyl ether, ethylene glycol methyl ether acetate, glycol ether, ethylene glycol monoethyl ether acetate, dimethoxyethane, ethylene glycol diethyl ether, diethylene glycol dimethyl ether, triethylene glycol monomethyl ether) shall be tested according to the provisions of GB/T 23986-2009; and

calculated according to 10.2 of GB/T 23986-2009. The multi-component waterproof coatings shall be tested for each component separately; and the results shall be calculated according to Formula (1) in combination with the ratio. The double-liquid fast-curing waterproof coating (rubber asphalt, water-based non-curing) is only tested for the main component, which is the test result.

#### 5.5 Content of phthalates compounds

- **5.5.1** For the waterproof sheet, the sampling part of the product with multi-layer structure is the main material on the upper surface; and the overall thickness shall be maintained when sampling homogeneous products. After sampling, use a liquid nitrogen freezer grinder to pulverize the samples. For samples that cannot be pulverized, clean scissors can be used to chop them as much as possible, with a particle size of about 1 mm. Carry out the test according to the provisions of GB/T 30646.
- **5.5.2** The waterproof coating shall be tested according to the provisions of GB/T 30646. The multi-component waterproof coatings are tested for each component separately; and the results are calculated according to Formula (1) in combination with the ratio. The double-liquid fast-curing waterproof coating (rubber asphalt, water-borne non-curing) is only tested for the main component, which is the test result.

#### 5.6 Content of polycyclic aromatic hydrocarbon

- **5.6.1** For the waterproof sheet, the sampling part of the product with multi-layer structure is the main material on the upper surface; and the overall thickness shall be maintained when sampling homogeneous products. After sampling, use a liquid nitrogen freezer grinder to pulverize the samples. For samples that cannot be pulverized, clean scissors can be used to chop them as much as possible, with a particle size of about 1 mm. Carry out the test according to the provisions of GB/T 36488.
- **5.6.2** The waterproof coating shall be tested according to the provisions of GB/T 36488. The multi-component waterproof coatings are tested for each component separately, and the results shall be calculated according to Formula (1) in combination with the ratio.

#### **5.7 Phenol content**

Carry out the test as specified in 5.6. Phenol is used as the calibration compound; the relative molecular mass is 94; the characteristic fragment ions were 95, 94, 66; and the quantitative ions are 94.

#### 5.8 Formaldehyde content

- **5.8.1** When the waterproof coating needs to be mixed and applied, it shall be mixed evenly according to the requirements provided by the manufacturer. When the mix ratio has a range, take the middle value.
- **5.8.2** The water-borne waterproof coating shall be tested according to the provisions of GB/T

Test according to the provisions of Appendix G in GB 36246-2018. Multi-component products are tested for each component separately, and the results shall be calculated according to Formula (1) in combination with the ratio.

#### 5.15 4,4'-Methylene-bis-(2-chloroaniline) (MOCA) content

Carry out the test according to the provisions of Appendix H in GB 36246-2018; only test the curing agent component to obtain the test result of this component; and calculate the content in the sample in combination with the ratio of this component.

# 5.16 Release amount of target volatile organic compounds, release amounts of total volatile organic compounds, and release amount of formaldehyde

#### 5.16.1 Sample pretreatment

For waterproofing sheet and spray molding coatings, prepare test pieces with a single-sided area material/cabin load ratio of 1m<sup>2</sup>/m<sup>3</sup>, and place them under the conditions of temperature (23±2) °C and relative humidity (50±5) % for 24h; and remove the anti-stick isolation material from the surface; surface sand and mineral particles are not removed; prepare 2 test pieces and put them into the environmental test chamber for testing.

The coating weight of waterproof coating should be 1/3 of the actual coating weight; or it can be tested according to the agreed coating weight. Spread the materials on the glass plate or flat plate tray; prepare the test pieces with the material/cabin load ratio of 1m<sup>2</sup>/m<sup>3</sup>. Prepare 2 test pieces, and put them into the environmental test chamber respectively for testing.

NOTE: The material/chamber load ratio refers to the ratio of the surface area of the material to the volume of the environmental test chamber during the test, and the unit is  $m^2/m^3$ .

#### **5.16.2 Test procedures**

Carry out the test according to the provisions of JG/T 528. The temperature in the environmental test chamber is controlled to be  $(23\pm1)$  °C, the relative humidity is  $(50\pm5)$  %, the number of air changes is once per hour, and the sampling time is  $(72\pm1)$  h after the specimen is placed in the environmental test chamber. The release amount of the organic matter is sampled at a sampling flow rate of 200 mL/min for 30 min; and the release amount of formaldehyde is sampled at a sampling flow rate of 400 mL/min for 25 min. When calculating the results, the chamber blank release amount shall be deducted. The sampling volume can be adjusted according to the situation; and other temperature and humidity conditions of the environmental test chamber can also be selected, such as: temperature  $(60\pm2)$  °C, relative humidity  $(5\pm2)$  %.

# **6 Test Report**

The test report shall give at least the following aspects:

# Appendix B

#### (Normative)

#### **Determination of Ammonia Content - Post-Distillation Titration**

#### **B.1** Principle

Ammonia was distilled from the alkaline solution, which is absorbed by excessive sulfuric acid solution; and the excessive sulfuric acid is titrated by sodium hydroxide standard solution using methyl red-methylene blue mixed indicator as indicator.

After mixing evenly according to the ratio, take samples for testing. The double-liquid fast-curing waterproof coating (rubber asphalt, water-borne non-curing) is only tested for the main component, which is the test result. The double-liquid fast-curing waterproof coating (acrylate) test each component separately; and then convert the results according to the ratio requirements. Dry powder waterproofing material only tests the powder, which shall be taken as the test result.

#### **B.2** Reagents and materials

- **B.2.1** The water involved in this method meets the requirements of Class-III water in GB/T 6682-2008.
- **B.2.2** The chemical reagents involved in this method are all analytically pure chemical reagents unless otherwise specified.
- **B.2.3** Hydrochloric acid solution: 1+1.
- **B.2.4** Sulfuric acid solution:  $c(1/2 \text{ H}_2\text{SO}_4)=0.1\text{mol/L}$ .
- **B.2.5** Sodium hydroxide standard solution: c(NaOH)=0.1 mol/L.
- **B.2.6** Sodium hydroxide solution: about 1mol/L.
- **B.2.7** Methyl red-methylene blue mixed indicator: Mix 50mL of methyl red ethanol solution (2g/L) and 50mL of methylene blue ethanol solution (1g/L).

#### **B.3** Instruments and utensils

- **B.3.1** Analytical balance: accuracy of 0.001g.
- **B.3.2** pH meter, accuracy of  $\pm 0.1$ .
- **B.3.3** Electric furnace.
- B.3.4 500mL glass distillation flask.

- **B.3.5** 300mL beaker.
- B.3.6 500mL conical flask.
- **B.3.7** 20mL pipette.
- B.3.8 50mL basic burette.
- **B.3.9** Straight condenser tube.

#### **B.4** Test procedures

#### **B.4.1 Treatment of specimens**

Stir the specimen evenly; and take about 5g of the specimen in a 300mL beaker (B.3.5), accurate to 0.001g.

For water-soluble specimen, add an appropriate amount of water to the 300mL beaker (B.3.5) containing the specimen; and transfer it into a 500mL glass distillation flask (B.3.4); control the total volume to 200mL; and prepare for distillation.

For water-insoluble specimen that may contain the remained ammonia, add 20 mL of water and 10 mL of hydrochloric acid solution (B.2.3) to the 300 mL beaker (B.3.5) containing the specimen; stir evenly; stand for 20 min; filter; and collect the filtrate to a 500mL glass distillation flask (B.3.4). Add an appropriate amount of water to control the total volume of 200mL; and prepare for distillation.

#### **B.4.2 Distillation**

Add sodium hydroxide solution (B.2.6) to the distilled solution; test with a pH meter (B.3.2); adjust the pH value to (12±0.2); and add a few explosion-proof glass beads. If the initial pH value is greater than 11.8, no adjustment is required.

Accurately pipette 20 mL of sulfuric acid solution (B.2.4) into a 500 mL conical flask (B.3.6); add  $3 \sim 4$  drops of mixed indicator (B.2.7); and insert the glass tube of the effluent outlet of the distiller into the bottom of the flask containing sulfuric acid solution. Heat and distill until the distillate volume reaches 180mL; then stop heating. Remove the distillation flask; wash the condenser tube with distilled water; and collect the washing liquid in a conical flask.

#### **B.4.3 Titration**

Titrate the excessive sulfuric acid solution with sodium hydroxide standard solution (B.2.5) until the indicator changes from bright purple to gray-green, and does not change color in 30s; and the volume of consumed sodium hydroxide standard solution is  $V_1$ .

#### **B.4.4 Blank test**

At the same time of determination, according to the same test procedures, reagents and dosage,

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