GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

ICS 87.040
G 51

GB 24410-2009

Indoor Decorating and Refurbishing Materials - Limit of Harmful Substances of Water based Woodenware Coatings

Issued on: September 30, 2009 **Implemented on: June 1, 2010**

Issued by: General Administration of Quality Supervision, Inspection and Quarantine (AQSIQ);

Standardization Administration (SAC) of the People's Republic of China.

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Foreword

All technical contents of this standard are compulsory.

Appendix A of this standard is normative.

This standard was proposed by China Petroleum and Chemical Industry Association.

This standard shall be under the jurisdiction of the National Technical Committee on Paints & Pigments of Standardization Administration of China.

Drafting organizations of this standard: Changzhou Paint and Coatings Industry Research Institute, Guangdong Huarun Paints Co., Ltd., ICI Swire Paints (China) Ltd., Guangdong Carpoly Chemical Co., Ltd., Nippon Paint (Guangzhou) Co., Ltd., Bayer Material Science Trading (Shanghai) Co., Ltd., BASF (China) Co., Ltd., Jiangsu Elephant East Asia Paint Co., Ltd., Sopel Chemical (Shanghai) Co., Ltd., Skshu Paint Co., Ltd., Nanjing Tianxiang coating Co. Ltd., Shanghai Zhongnan Building Material Company, China Paint (Shenzhen) Co., Ltd., Goldenfish Group Shijiangzhuang Paint Company, Hengchang Petrochemical Industry Co., Ltd., Nankang Austre Paint Co., Ltd., Kunshan Sunmun Science & Technology Development Co., Ltd. and Guangdong Hualong Knitting Co., Ltd.

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Indoor Decorating and Refurbishing Materials – Limit of Harmful Substances of Water based Woodenware Coatings

1 Scope

This standard specifies the requirements, test methods, inspection rules, packaging mark, coating safety and protection for the permissible limits of such substances harmful to human body and environment in the water based woodenware coatings for indoor decorating and refurbishing and water based putty for the woodenware.

This standard is applicable to water based woodenware coatings for indoor decorating and refurbishing and industrial coating, and water based putty for the woodenware.

2 Normative References

The following standards contain the provisions which, through reference in this standard, constitute the provisions of this standard. For dated reference, the subsequent amendments (excluding corrigendum) or revisions of these publications do not apply. However, all parties who enter into an agreement according to this standard are encouraged to study whether the latest editions of these documents apply. For undated references, the latest edition of the normative document referred to applies.

GB/T 1250 Rules for Expression and Judgment of Limiting Values

GB/T 3186 Paints, Varnishes and Raw Materials for Paints and Varnishes - Sampling (GB/T 3186-2006, ISO 15528:2000, IDT)

GB/T 6750 Paints and Varnishes - Determination of Density - Pycnometer Method (GB/T 6750-2007, ISO 2811-1:1997, IDT)

GB/T 9750 Marks for Package of Coating Products

GB 18582-2008 Indoor Decorating and Refurbishing Materials - Limit of Harmful Substances of Interior Architectural Coatings

[b] For the powder putty, except that powder is directly determined for the soluble heavy metal, others are determined according to the specified mixture ratio of the product after mixing the powder with water, adhesive or other liquids. If the mixture ratio is certain range, water shall be determined after mixing according to the minimum mixture ratio of water consumption while other liquids such as adhesive shall be determined after mixing according to the maximum mixture ratio of the consumption.

5 Test Methods

5.1 Sampling

The product shall be sampled according to those specified in GB/T 3186.

5.2 Test methods

5.2.1 Volatile organic compounds content is tested according to those specified in Appendix A of this standard. Test results of coating product are calculated according to those specified in Article A.7.2 of Appendix A; those of putty product are calculated according to those specified in Article A.7.1 of Appendix A.

Note: moisture content and density tests are omitted for all the putty samples.

5.2.2 Content of benzene series (benzene, toluene, ethylbenzene and dimethylbenzene) is tested according to those specified in Appendix A of this standard. The test results are calculated according to those specified in Article A.7.3 of Appendix A.

5.2.3 Content of glycol ether and its esters (ethylene glycol monomethyl ether, ethylene glycol monomethyl ether acetate, ethylene glycol ethyl ether, ethylene glycol ethyl ether acetate and diethylene glycol butyl ether acetate) is tested according to those specified in Appendix A of this standard. Test results are calculated according to those specified in A.7.4 of Appendix A.

5.2.4 Content of free formaldehyde is tested according to those specified in Appendix C of GB 18582-2008.

5.2.5 Content of soluble heavy metal (lead, cadmium, chromium and mercury) is tested according to those specified in Appendix D of GB I8582-2008 and its results are expressed in milligram of the soluble heavy metal containing in the film per kilogram. For the powder putter, it is directly tested with the powder.

Note: content of soluble lead, cadmium, chromium and mercury in the treated test solution may be determined with other appropriate analysis instruments, such as inductively coupled plasma atomic emission spectrometric method (ICP-OES); operation and test are carried out according to relevant instructions of the instrument manufacturer. Moreover, the adopted analysis instrument shall be indicated in the test report.

Appendix A

(Normative)

Test for Content of Volatile Organic Compound, Benzene Series, Glycol Ether and Its Esters - Gas Chromatography

A.1 Scope

This appendix specifies test methods for the content of volatile organic compounds (VOC), benzene series (benzene, toluene, ethylbenzene and dimethylbenzene), glycol ether and its esters (ethylene glycol monomethyl ether, ethylene glycol monomethyl ether acetate, ethylene glycol ethyl ether, ethylene glycol ethyl ether acetate and diethylene glycol butyl ether acetate) in water based woodenware coatings and water based putty.

A.2 Principle

After the sample is diluted, volatile organic compounds in the sample are separated through the gas chromatographic analysis technology; after qualitative identification of the measured compound, its content is tested by internal standard method.

A.3 Materials and reagents

A.3.1 Carrier gas: nitrogen, with purity greater than or equal to 99. 995%.

A.3.2 Gas: hydrogen, with purity greater than or equal to 99. 995%.

A.3.3 Combustion-supporting gas: air.

A.3.4 Auxiliary gas (septum purging and make-up gas): nitrogen of same nature with the carrier gas.

A.3.5 Internal standard substances: compounds absent in the sample; the compounds can be completely separated from other components on the chromatogram. The purity is at least 99% (mass percent) or the purity is known. For example: isobutanol, ethylene glycol butyl ether and diethylene glycol dimethyl ether.

A.3.6 Calibration compounds

In this standard, calibration compounds cover benzene, toluene, ethylbenzene, dimethylbenzene, ethylene glycol monomethyl ether, ethylene glycol monomethyl ether acetate, ethylene glycol ethyl ether, ethylene glycol ethyl ether acetate, diethylene glycol butyl ether acetate, acetone, ethanol, isopropanol, triethylamine,

isobutanol, 1-butanol, propylene glycol monomethyl ether, dipropylene glycol monomethyl ether, acetic acid n-butyl ester, dimethylethanolamine, methyl isoamyl ketone, propylene glycol n-butyl ether, ethylene glycol butyl ether, 1,2-propylene glycol, ethylene glycol, N-methyl pyrrolidone, dipropylene glycol n-butyl ether, diethylene glycol monobutyl ether, propylene glycol phellyl ether, diethylene glycol and ethylene glycol phellyl ether. Purity of the calibration compound is at least 99% (mass fraction) or the purity is known.

A.3.7 Dilution solvent: organic solvent for diluting the sample; it doesn't contain any substance interfering the test. The purity is at least 99% (mass fraction) or the purity is known. For example, solvents such as acetonitrile, methanol or tetrahydrofuran.

A.3.8 Marker: compound that distinguishes VOC and non-VOC according to the definition of VOC. In this standard, it is diethyl oxalate (251°C boiling point).

A.4 Instruments and equipment

A.4.1 Gas chromatograph; it is configured as follows:

A.4.1.1 Injection port of diverter; in addition, the lining of the vaporizing chamber is replaceable;

A.4.1.2 Temperature programmed controller;

A.4.1.3 Detector

Any one of the following detectors may be used:

A.4.1.3.1 Flame ionization detector (FID);

A.4.1.3.2 Calibrated or tuned mass spectrometer or other mass selective detectors;

A.4.1.3.3 Calibrated Fourier transform infrared spectrometer (FT-IR spectrometer).

Note: if the detector described in Article A.4.1.3.2 or A.4.1.3.3 is selected to carry out qualitative identification on the separated component, the instrument shall be connected with the gas chromatograph and operated according to relevant instructions of the instrument manufacturer.

A.4.1.4 Chromatographic column: 6% nitrile propyl benzene/94% polydimethyl siloxane (PDMS) capillary column, polyethylene glycol capillary column;

A.4.2 Sample injector: the capacity at least shall be twice the sample size;

A.4.3 Blending bottle: about 20mL glass bottle; it is provided with sealable bottle cap;

A.4.4 Balance: 0.1mg accuracy.

(ethylene glycol monomethyl ether, ethylene glycol monomethyl ether acetate, ethylene glycol ethyl ether, ethylene glycol ethyl ether acetate and diethylene glycol butyl ether acetate)

A.6.3.1 Parameter optimization of chromatograph

According to chromatographic condition described in Section A.5, known calibration compound shall be used every time to carry out the optimization treatment so that the sensitivity, stability and separation effect are in the optimal state.

A.6.3.2 Qualitative analysis

It is identified whether there is calibration compound described in Article A.3.6 through qualitative analysis. For the preferred method, gas chromatograph and mass selective detector (A.4.1.3.2) or FT-IR spectrometer (A.4.1.3.3) are combined and test conditions for the gas chromatography given in Section A.5 are used. Alternatively, gas chromatograph is used, flame ionization detector (FID) (A.4.1-3.1) and chromatographic column (A.4.1.4) are adopted, and test conditions for the gas chromatography given in Section A.5 are used; chromatograms of the calibration compounds (A.3.6) on two pieces of chromatographic columns (the polarity difference of the two selected columns shall be large as much as possible, for example, 6% nitrile propyl benzene/94% polydimethyl siloxane (PDMS) capillary column and polyethylene glycol capillary column) are recorded respectively; under the same test conditions for the chromatogram, the measured sample is contrasted to be qualitative after the chromatogram is made for it.

A.6.3.3 Calibration

A.6.3.3.1 Preparation of calibration sample: a certain amount (accurate to 0.1mg) of identified calibration compounds described in Article A.6.3.2 is weighed respectively and put into the blending bottle (A.4.3); the weighed mass and the respective content in the sample to be tested shall be in the same order of magnitude; then internal standard substance (A.3.5) in the same order of magnitude with the compounds to be tested is weighed and put into the same blending bottle (A.3.5), the mixture is diluted with dilution solvent (A.3.7), the blending bottle is sealed and well shaken.

A.6.3.3.2 Test for relative correction factor: under the same chromatogram test condition as the test sample, the parameter is optimized according to those specified in Article A.6.3.1. Adequate amount of calibration compound is filled into the gas chromatograph and the chromatogram is recorded. Relative correction factor of each compound is respectively calculated according to Formula (A.1):

$$R_i = \frac{m_{ci} \times A_{is}}{m_{is} \times A_{ci}} \quad \text{..... (A.1)}$$

Where,

R_i - the relative correction factor of the component *i*;

m_{ci} - the mass of the compound *i* in the calibration mixture, g;

m_{is} - the mass of the internal standard substance in the calibration mixture, g;

A_{is} - the peak area of internal standard substance;

A_{ci} - the peak area of the compound *i*.

Average of the two test results are taken as R_i and its relative deviation shall be less than 5% with three significant figures retained.

A.6.3.3.3 If any compound chromatogram peak beyond the calibration compounds described in Article A.3.6, it shall be assumed that its correction factor relative to isobutanol is 1.0.

A.6.3.4 Test for test sample

A.6.3.4.1 Preparation of sample: 1g well-mixed sample (accurate to 0.1mg) and internal standard substance (A.3.5) whose amount is approximately equal to that of the tested substances are weighed and put into the blending bottle (A.4.3), 10mL dilution solvent (A.3.7) is added to dilute the sample, the blending bottle (A.4.3) is sealed and shaken well.

A.6.3.4.2 The parameter is set according to the optimized condition during the calibration.

A.6.3.4.3 The marker (A.3.8) is filled into the gas chromatograph and its retention time on the 6% nitrile propyl benzene/94% polydimethyl siloxane (PDMS) capillary column is recorded so that the end point of the integral in the chromatogram is determined according to the definition of VOC given in Section 3.1.

A.6.3.4.4 1μL is filled into the gas chromatograph according to the sample blended in Article A.6.3.4.1, the chromatogram and the peak areas (except dilution solvent) of the compounds whose retention time is less than that of the marker are recorded; and then mass frictions of compounds contained in the sample are respectively calculated according to the following formula:

$$\omega_i = \frac{m_{is} \times A_i \times R_i}{m_s \times A_{is}} \quad \text{..... (A.2)}$$

Where,

ω_i - the mass fraction of the measured compound *i* in the test sample, g/g;

R_i - the relative correction factor of the measured component *i*;

A.8 Accuracy

A.8.1 Repeatability

Relative deviation of two test results by the same operator is less than 10%.

A.8.2 Reproducibility

Relative deviation of test results in different laboratories is less than 20%.

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