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Limit of Harmful Substances of Industrial Protective Coatings

工业防护涂料中有害物质限量

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Limit of Harmful Substances of Industrial Protective Coatings

1 Scope

This Standard specifies the classification, requirements, test methods, inspection rules, packaging marks, and standard implementations for the products involved in the allowable limits for substances harmful to humans and the environment in industrial protective coatings.

This Standard is applicable to all types of industrial protective coatings (excluding marine coatings) for the protection of metal, concrete, plastic and other surfaces except putty.

This Standard is not applicable to aerospace coatings, nuclear power coatings, coatings for military equipment and facilities.

2 Normative References

The following documents are essential to the application of 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) are applicable to this document.

GB/T 1725-2007 Paints Varnishes and Plastics - Determination of Non-Volatile-Matter Content

GB/T 3186 Paints Varnishes and Raw Materials for Paints and Varnishes - Sampling

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

GB/T 6750-2007 Paints and Varnishes - Determination of Density - Pycnometer Method

GB/T 8170-2008 Rules of Rounding off for Numerical Values & Expression and Judgement of Limiting Values

GB/T 9750 Marks for Package of Coating Products

GB/T 9758.5-1988 Paints and Varnishes - Determination of “Soluble” Metal

Content – Part 5: Determination of Hexavalent Chromium Content of the Pigment Portion of the Liquid Paint and the Paint in Power Form - Diphenylcarbazide Spectrophotometric Method

GB/T 9760-1988 Paints and Varnishes - Preparation of Acid Extracts from Paints in Liquid or Powder Form

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

GB/T 23986-2009 Paints and Varnishes - Determination of Volatile Organic Compound (VOC) Content - Gas-Chromatographic Method

GB/T 23990-2009 Determination of the Contents of Benzene Toluene Ethylbenzene and Xylene in Coatings by Gas Chromatography

GB/T 23992-2009 Gas chromatography of chlorinated hydrocarbons content in paint

GB/T 30647-2014 Determination of Harmful Elements - Total Content of Coatings

GB/T 34675-2017 Determination of Volatile Organic Compound (VOC) - Content in Radiation Curable Coatings

GB/T 34682-2017 Determination of Volatile Organic Compound (VOC) - Content in Reactive Diluent Containing Coatings

GB/T 36488-2018 Determination of Polycyclic Aromatic Hydrocarbons in Coatings

3 Terms and Definitions

For the purpose of this document, the following terms and definitions apply. ‘

3.1 Engineering machinery

Working machinery and equipment used in the comprehensive mechanized construction of earthmoving works, stone works, concrete works and various types of building installation engineering.

NOTE: For example, industrial vehicles, construction machinery, line machinery, municipal sanitation machinery, elevators and escalators, pneumatic tools, etc.

3.2 Agricultural machinery

Various machines used in crop planting and animal husbandry production, as well as in the initial processing and processing of agricultural and livestock products.

A kind of equipment for transportation of cargoes, which shall meet the following conditions:

a) It has sufficient strength and rigidity and can be used repeatedly for a long time;

b) It is suitable for carrying by one or more modes of transportation. When transferring in the road, the package for cargoes in the box need not be replaced;

c) It shall be equipped with devices that facilitate fast loading and unloading and handling, especially when transferring from one mode of transport to another;

d) It facilitates the filling or emptying of the cargo;

e) It has a volume of 1m^3 and above;

f) It is a freight vehicle designed in accordance with the requirements to ensure security and has the ability to prevent easy access by unrelated personnel.

[GB/T 1992-2006, definition 3.1]

3.8 Package

The general name of the container, material, and auxiliary used in accordance with certain technical methods in order to protect the product in the circulation process, facilitate storage and transportation, and promote sales.

NOTE: For example, beverage cans, food cans, chemical drums, steel drums, non-stick pans, etc.

3.9 Profiles

Objects with a certain geometric shape made of aluminum, iron or steel and materials with a certain strength and toughness through rolling, extrusion, casting and other processes.

NOTE: For example, aluminum profiles, plastic profiles, etc.

3.10 Electrical and electronic product

Equipment and ancillary products that operates by relying on current or electromagnetic fields or aims at generating, transmitting, and measuring current and electromagnetic fields; whose rated operating voltage is no more than 1500V for direct current and 1000V for alternating current. The equipment that involves the production, transmission and distribution of electrical energy is excluded.

3.11 Pre-coated coil

Organic material/metal composite sheet material that is sold in coil or sheet after

protective coatings for buildings and constructions, container coatings, pre-coated coil coatings, packaging coatings, profile coatings (including metal substrate curtain wall coatings), and electrical and electronic coatings. The radiation-curable coatings are divided into water-based and non-water-based coatings.

5 Requirements

5.1 The limit values of VOC content in various types of industrial protective coatings other than special functional coatings shall meet the requirements of Tables 1, 2, 3, and 4.

NOTE: Special functional coatings refer to insulating coatings, fingerprint-resistant coatings for touch screens and optical plastic sheets, and polytetrafluoroethylene coatings (chemical media resistance, wear resistance, lubrication, non-stick and other special functions) that are sintered and formed at high temperatures above 150°C, fluorosilicone coatings for elastomers, silver-plated effect paints (radiation-curable), marking paints, protective coatings for electronic components (special functions such as anti-acid fog, dustproof, and moisture-proof), etc.

The limit value of VOC content in water-based coatings shall meet the requirements of Table 1. The limit value of VOC content in solvent-based coatings shall meet the requirements of Table 2. The limit value of VOC content in solvent-free coatings shall meet the requirements of Table 3. The limit value of the VOC content in radiation-curable coatings shall meet the requirements of Table 4. When the coating product is expressly suitable for multiple uses, it shall meet the strictest limit value requirements among each requirement.

Water-based coatings and water-based radiation-curable coatings do not consider the dilution ratio of water. Other types of coatings are measured after mixing according to the construction ratio of the product in the specified construction state. If the usage among of a component among a multi-component is in a certain range, it shall be measured after mixing according to the maximum ratio specified in the construction ratio of the product under construction.

chromatographic column (6% cyanopropyl-phenyl/94% polydimethylsiloxane capillary column), and the label was diethyl adipate. VOC content is calculated according to 10.4 of GB/T 23986-2009.

If the moisture content in the coating is less than 70% (mass fraction), it shall be carried out according to the provisions of GB/T 23985-2009. The content of non-volatile matter is carried out according to the provisions of GB/T 1725-2007; take about 1g of the specimen; and the baking condition is (105±2) °C/1h. VOC content is calculated according to 8.4 of GB/T 23985-2009.

6.2.1.3 VOC content in solvent-based coatings

Solvent-based coatings that do not contain reactive diluent and water are carried out in accordance with the provisions of GB/T 23985-2009. The content of non-volatile matter is carried out in accordance with the provisions of GB/T 1725-2007. Take about 1g of the specimen; and the baking condition is (105±2) °C/1h. The moisture content is not measured and the moisture content was set to zero. The calculation of VOC content in solvent-based coatings without reactive diluent and water shall be carried out according to 8.3 of GB/T 23985-2009.

Solvent-based coatings containing reactive diluents are carried out in accordance with the provisions of 6.2.1.4.

Solvent-based coatings with intentionally-added water shall be carried out in accordance with the provisions of GB/T 23985-2009. The content of non-volatile matter is carried out in accordance with the provisions of GB/T 1725-2007. Take about 1g of specimen; and the baking condition is (105±2) °C/1h; the determination of the moisture content is performed according to the provisions of Appendix A. The calculation of VOC content is carried out according to 8.4 of GB/T 23985-2009.

6.2.1.4 VOC content in solvent-free coatings

It shall be performed according to the provisions of GB/T 34682-2017. The storage time for the determination of non-volatile matter content is 24h under the standard test environment [temperature (23±2) °C; relative humidity (50±5)%], or the time specified in the product manual; but the storage time is no greater than 7d. If the moisture is not determined, the moisture content shall be set to zero.

The calculation of VOC content is carried out according to 8.3 of GB/T 34682-2017.

6.2.1.5 VOC content in radiation-curable coatings

It shall be performed according to the provisions of GB/T 34675-2017.

The calculation of VOC content in water-based radiation-curable coatings shall be carried out in accordance with 8.4 of GB/T 34675-2017; the determination of moisture

a year, and the type inspection items include all the requirements listed in this Standard.

7.1.2 Type inspection shall be carried out in one of the following cases:

--- When the new product is initially finalized;

--- When the product is produced off-site;

--- When the production formula, process, key raw material sources and construction ratio of the product under construction have changed greatly;

--- When production resumes after three months of suspension.

7.2 Determination of inspection results

7.2.1 The test results shall be determined according to the rounding-off comparison method in GB/T 8170-2008.

7.2.2 When reporting the inspection results, the construction ratio in the construction state explicitly stated by the product shall be indicated at the same time.

7.2.3 When the inspection results of all items meet the requirements of this Standard, the product meets the requirements of this Standard.

8 Packaging Mark

8.1 In addition that the product packaging mark conforms to the provisions of GB/T 9750, the products that pass the inspection according to this Standard may be clearly indicated on the packaging mark.

8.2 The construction ratio in the construction state shall be clearly stated on the packaging mark or in the product manual.

8.3 The classification, product category and product type (or application method) in accordance with this Standard shall be indicated on the packaging mark or in the product manual.

8.4 Solvent-based coatings containing reactive diluents shall be clearly indicated on the packaging mark or in the product manual.

8.5 Solvent-based coatings with intentionally-added water shall be clearly indicated on the packaging mark or in the product manual.

8.6 For polyurethane, epoxy and other multi-component cured coatings, the applicable period shall be indicated on the packaging mark or in the product manual.

Appendix A

(Normative)

Determination of Moisture Content – Gas Chromatography

A.1 Reagents and materials

A.1.1 Distilled water: meets the requirements of Grade-III water in GB/T 6682-2008.

A.1.2 Dilution solvent: organic solvent used to dilute the sample and dried by molecular sieve, does not contain any substances that interfere with the test. Purity is at least 99% (mass fraction), or known purity. For example: dimethylformamide, etc.

A.1.3 Internal standard substance: a compound that is not present in the specimen and dried by molecular sieve; and the compound can be completely separated from other components on the chromatogram. Purity is at least 99% (mass fraction), or known purity. For example: isopropanol, etc.

A.1.4 Molecular sieve: the pore size is 0.2nm ~ 0.3nm, and the particle size is 1.7mm ~ 5.0mm. Molecular sieves shall be used after regeneration.

A.1.5 Carrier gas: hydrogen or helium, purity ≥99.995%.

A.2 Apparatus

A.2.1 Gas chromatograph: equipped with thermal conductivity detector and programmed temperature-rise controller.

A.2.2 Chromatographic column: capillary column of styrene-divinylbenzene porous polymer.

NOTE: Other chromatographic columns that meet the inspection requirements can also be used.

A.2.3 Injector: micro-syringe, 10μL.

A.2.4 Sample bottle: about 10mL glass bottle with sealable bottle cap.

A.2.5 Balance: actual division value d = 0.1mg.

A.3 Gas chromatography test conditions

A.3.1 Chromatographic column: capillary column of styrene-divinylbenzene porous polymer, 25m × 0.53mm × 10μm.

Appendix B

(Normative)

Determination of Hexavalent Chromium (Cr^{6+}) Content – Spectrophotometry

Caution: All samples and reagents that potentially contain hexavalent chromium (Cr^{6+}) in the test method shall be prevented by appropriate measures. Solutions and wastes containing hexavalent chromium (Cr^{6+}) shall be properly disposed of.

B.1 Principle

If the total chromium content in the sample is less than 8mg/kg; the result of hexavalent chromium (Cr^{6+}) content shall be reported as "not detected"; and the detection limit shall be 8mg/kg. If the total chromium content in the sample is greater than or equal to 8mg/kg, after the specimen (with simultaneous matrix spikes) is ultrasonically dispersed, a hexavalent chromium (Cr^{6+}) compound is extracted from the sample by an alkaline digestion solution. Hexavalent chromium (Cr^{6+}) in the extraction solution reacts with diphenyl carbazide in an acidic solution to form a purple-red complex. The content of hexavalent chromium (Cr^{6+}) in the test solution is determined spectrophotometrically (at a wavelength of 540nm). At the same time, the nonvolatile matter content of the specimen is measured, and the final result is reported as the hexavalent chromium (Cr^{6+}) content in the dry film.

B.2 Reagents and materials

Only the reagents identified as analytically pure were used in the analysis test, and the used water met the requirements of Grade-III water in GB/T 6682-2008.

B.2.1 N-Methyl pyrrolidone (NMP): the reagent is stored in a brown bottle at 20°C ~ 25°C, avoiding direct sunlight. Before use, 10g of active molecular sieve shall be added to each 100mL of reagent and store for more than 12h. After the container is opened, the storage period is one month.

B.2.2 Nitric acid: about 65% (mass fraction); density is about 1.40 g/mL; yellowed nitric acid shall not be used.

B.2.3 Sulfuric acid: about 98% (mass fraction); density is about 1.84 g/mL.

B.2.4 Sodium hydroxide.

B.2.5 Anhydrous sodium carbonate.

specimen. One to two drops of wetting agent (anhydrous ethanol) can be added to increase the wettability of the specimen. Cover the digester (B.3.5) by a stopper, place it in an ultrasonic water bath (B.3.3); and sonicate at 60°C ~ 65°C for 1h.

Take out the digester (B.3.5) from the ultrasonic water bath (B.3.3); gradually cool to room temperature; and transfer the solution (do not filter the solution even if the solution is turbid or flocculent) in the digester (B.3.5) to a clean beaker (B.3.9); add nitric acid (B.2.11) dropwise to the beaker as stir; test by an acidity meter (B.3.4); adjust the pH of the solution to 7.5±0.5; and get extracting solution. The extracting solution shall be tested for color development as soon as possible.

B.4.3 Test

B.4.3.1 Preparation of chromogenic solution

Slowly add the sulfuric acid solution (B.2.12) to the extracting solution in each beaker (B.3.9); test by an acidity meter (B.3.4); adjust the pH of the solution to 2.0±0.5; and mix well. Then use a pipette (B.3.7) to accurately transfer 2.0mL of diphenyl carbazide developer (B.2.15) and mix well. Then transfer it all to a 100mL volumetric flask (B.3.6) and dilute it to the mark with water to obtain a test solution. The test solution shall be measured as soon as possible after standing for 5~10min; and the test on the machine shall be completed within 30 minutes.

B.4.3.2 Preparation of serial standard working solutions

Use a pipette (B.3.7) to transfer 0.0mL, 2.0mL, 4.0mL, 6.0mL, 8.0mL, 10.0mL, 20mL of hexavalent chromium (Cr^{6+}) standard solution (B.2.17) to a 100 mL volumetric flask, respectively. Separately add 50 mL of water by the measuring cylinder (B.3.8); separately titrate the sulfuric acid solution (B.2.12); and test by a pH meter (B.3.4); adjust the pH value of the solution to 2.0±0.5; use a pipette (B.3.7) to transfer 2.0mL of diphenyl carbamide developer (B.2.15); separately dilute with water to the mark, and mix well. After standing for 5min~10min, complete the test as soon as possible within 30min. The mass concentration of hexavalent chromium (Cr^{6+}) in this serial standard working solutions is 0.0 mg/L, 0.1 mg/L, 0.2 mg/L, 0.3 mg/L, 0.4 mg/L, 0.5 mg/L, 1.0 mg/L.

B.4.3.3 Determination of hexavalent chromium (Cr^{6+}) content in the specimen

An appropriate amounts of serial standard working solutions are moved into a 10mm colorimetric cell, respectively; measure its absorbance on a spectrophotometer (B.3.2) at a wavelength of 540nm. Draw a calibration curve by the mass concentration value corresponding to absorbance value. The correction coefficient of the calibration curve shall be ≥0.99. Otherwise, a new calibration curve shall be made.

Under the same conditions, test the absorbance of the test solution (B.4.3.1) filtered through a 0.45μm syringe filter (B.3.10); and calculate the mass concentration of

Where:

SR – matrix spike recovery, in %;

SS - hexavalent chromium (Cr^{6+}) content in the specimen (by dry film) after spike, in mg/kg;

US - hexavalent chromium (Cr^{6+}) content in the specimen (by dry film) without spike, in mg/kg;

SA - hexavalent chromium (Cr^{6+}) content calculated by specimen dry film, which is converted into the hexavalent chromium (Cr^{6+}) content in the spike solution, in mg/kg.

Example:

If 0.5mL of hexavalent chromium (Cr^{6+}) standard stock solution (100 mg/L) is added, the non-volatile matter content in the specimen is 0.50 g/g. Take about 0.1g of the specimen; then SA = 0.5mL × (100 mg/L) / (0.1g × 0.50 g/g) = 1000 mg/kg.

According to the hexavalent chromium (Cr^{6+}) content of the tested specimen, other suitable amount of spike solution can be selected to ensure that the mass concentration after spike is within the appropriate curve range.

B.4.4.3 Calibration of results and detection limits

The acceptable range of matrix spike recovery shall be ≥50% and ≤125%.

When the matrix spike recovery is <50%, double amount of the spike solution shall be re-added for test. When the matrix spike recovery is> 125%, the same amount of spike solution shall be re-added for the test. If the matrix spike recovery for repeated test is still in the range of ≥50% and ≤125%, and the alkaline digestion method is not applicable to the tested sample; the content of hexavalent chromium (Cr^{6+}) in the specimen shall, according to the provisions of Clause 6, 8.1, 8.2.3, and 8.4 of GB/T 9760-1988, prepare the acid extracting solution (the weighing amount of the prepared pigment is about 0.5g); and then measure the hexavalent chromium (Cr^{6+}) content according to GB/T 9758.5-1988. The results are reported by the hexavalent chromium (Cr^{6+}) content in the dry film after dividing by the non-volatile matter content.

If the matrix spike recovery rate is >75% and ≤125%, no calibration result is required, and the detection limit is 8 mg/kg.

If the matrix spike recovery rate is within the range of ≥50% and ≤75%, the results and detection limits shall be corrected according to the matrix spike recovery, that is: the result multiplied by the ratio of 100% spike recovery and the actual matrix spike recovery; and detection limit shall be corrected in the same way.

Example:

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