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Sector Standard for Environmental Protection of the People's Republic of China

HJ/T 414-2007

Technical requirement for environmental labeling products Solvent-based wood coatings for indoor decoration and refurbishing

Issued on December 21, 2007 Putting into effect as of April 1, 2008

Issued by Ministry of Environmental Protection

Foreword

This standard is developed for the purpose of implementing the Law of the People's Republic of China on Environmental Protection, effectively employing and saving resources and minimizing the adverse impacts of the production and use of solvent-based wood coatings for indoor decoration and refurbishing on the environment and human health and promoting the application of environmental protection products.

This standard presents requirements for the limits of such substances as phthalate, ethanediol ether and its lipids, benzene, methyl benzene, ethyl benzene, dimethyl benzene, methanol, hydrocarbon halide, n-hexane, isophorone, relevant heavy metals and free toluene vulcabond in the solvent-based wood coatings for indoor decoration and refurbishing.

This standard is a guiding standard and applicable to the certification of environmental labeling products in China.

This standard is put forward by the Department of Science, Technology and Standards of the Ministry of Environmental Protection.

Major organizations in charge of the development of this standard: Environment and Development Center of MEP, Beijing Trace Chemicals Institute, National Quality Supervision and Testing Center for Paint, Nippon Paint Langfang Co., Ltd., Guangdong Huarun Coatings Co., Ltd., Eastman Chemicals, ICI Swire Paints (China) Co., Ltd., and The China Paint MFG. Co. (Shenzhen) Ltd.,.

This standard was approved by the Ministry of Environmental Protection on December 21, 2007.

This standard shall be put into effect as of April 1, 2008.

This standard shall be interpreted by the Ministry of Environmental Protection.

Technical Requirement for Environmental Labeling Products

Solvent-based Wood Coatings for Indoor Decoration and Refurbishing

1. Application Scope

This standard specifies the terminology & definition, basic requirements, technical contents and determination method for environmental labeling product — solvent-based wood coatings for indoor decoration and refurbishing.

This standard is applicable to the nitro-, polyurethanes and alkyd solvent face coat and base coat used in indoor decoration and refurnishing. It is not applicable to radiation curable coatings.

2. Standard Quotation Documents

This standard quotes the provisions of the following documents. The effective version of all quotation documents without specific date is applicable to the current standard.

GB/T 16483 General Rules to Drafting Safety Data Sheet for Dangerous Chemicals
GB/T 3186 Sampling of paints and varnishes and their raw materials
GB/T 6750－1986 Paints and varnishes —Determination of density
GB/T 6751－1986 Paints and varnishes —Determination of volatile and non-volatile matter
GB/T18446－2001 Standard test method for unreached tolune diisocyanates (TDI) in urethane prepolymers and coatings solution by gas chromatography
GB 18581－2001 Indoor decorating and refurnishing materials — Limit of harmful substance of solvent coatings for woodenware

3. Terminology and Definitions

The following terminologies are applicable to this standard.

3.1 Volatile Organic Compounds (VOC)

They refer to the organic compounds with primary boiling point less than or equal to 250℃ under 101.3 kPa.

3.2 Nitro-coatings

Nitro-coatings refer to the kind of coats with cellulose nitrate as the dominant film forming substance, which results from esterifying reaction of the mixture of nitric acid and sulphuric acid with cellulose.

3.3 Alkyd coatings

Alkyd coatings refer to the kind of coats with alkyd resins as the dominant film forming substance, which results from the condensation polymerization of polyacid, fatty acid (or vegetable oil) with polybasic alcohol.

3.4 Polyurethane coatings

Polyurethane coatings refer to the kind of coats with polyurethane resin as the dominant film forming substance, which is the product of the reaction of polyisocyanate with compounds containing active hydrogen.

4. Basic Requirements

4.1 The quality of the products shall comply with the quality requirements of each individual product.

4.2 The discharge of pollutants from the enterprises shall comply with the requirements of national or local standards for the discharge of pollutants.

5. Technical Contents

5.1 The products are banned to use the substances listed in Table 1.

Table 1 Banned substances in coatings

Banned type	Banned substances
Ethylene glycol ether and its esters	Ethylene glycol monomrthyl ether, ethylene glycol monomrthyl ether acetate, ethyl glycol, ethyl glycol acetate, diethyl-dialcohol butyl ether acetate
Phthalates	DOP and DBP
Alkanes	*n*-hexane
Ketones	3,5,5-trimethyl-2-cyclohexenyl-1-ketone(isophorone)
Hydrocarbon halides	Dichloromethane, dichloroethane, trichloromethane, trichloroethane, carbon tetrachloride
Aromatic hydrocarbon	Benzene
Alcohol	Methanol

5.2 The concentrations of hazardous substances in products shall meet the requirement of Table 2.

Table 2 Limits of Hazardous Substances in Coatings

	Nitro-solvent coatings		Polyurethane solvent based coatings			Alkyd solvent based coatings	
	Face	Base	Face	Face	Base	Colored paint	Varnish
Lustre (incident angle 60°)	—	—	≥80	<80	—	—	—
VOC [a], g/L ≤	700		550	650	600	450	500
Benzene mass fraction [a], % ≤	0.05						
Toluene ＋ xylene ＋ ethylbenzene mass fraction [a], % ≤	25		25			5	
Soluble heavy metals [b], mg/kg ≤							
Soluble Pb	90						
Soluble Cd	75						
Soluble Cr	60						

Soluble Hg	60		
Free toluene vulcabond in hardening agent, (TDI) mass fraction, % ≤	—	0.5	—
Methanol [a], mg/kg ≤	500	—	—
Note: a: Determination will be carried out according to the specified proportion of the product and mixture after dilution. If the application amount of diluent falls into a scope, the analyst shall not carry out the determination until it is diluted in accordance with the recommended maximum dilution amount. b: The determination of soluble heavy metals is only limited to colored paint.			

5.3 The manufacture shall present MSDS in line with the requirements of GB 16483.

6. Determination Method

6.1 The determination of the concentration of free TDI in the hardening agent shall be inline with the requirement of GB/T 18446－2001.

6.2 The determination of the concentration of VOC in the product shall comply with the method specified in Annex A.

6.3 The determination of the concentration of benzene, toluene, xylene, ethyl benzene and methanol in the product shall comply with the method specified in Annex B.

6.4 The determination of the concentration of heavy metals in the products shall comply with the method specified in Annex B of GB 18581－2001.

6.5 Other technical requirements are verified by document review combined with site inspections.

Annex A

(Standard Annex)

Determination of VOC Content in Solvent Coatings

A.1 Method & Principle

The sample is tested by gas chromatography. If no organic compounds with boiling points larger than 250℃ are detected, the tested volatile fraction is VOC concentration of the product. If test results show that there are any organic compounds with boiling points larger than 250℃, then there is the need for the quantitative and qualitative determination of the organic compounds with boiling points larger than 250℃ in the sample and lacquer film after vilatilization. Then calculate the concentration of organic compounds (Cv) with boiling points larger than 250℃ in the volatile amount. Deduct Cv from volatile amount V, you will get the concentration of VOC. See Figure A.1.

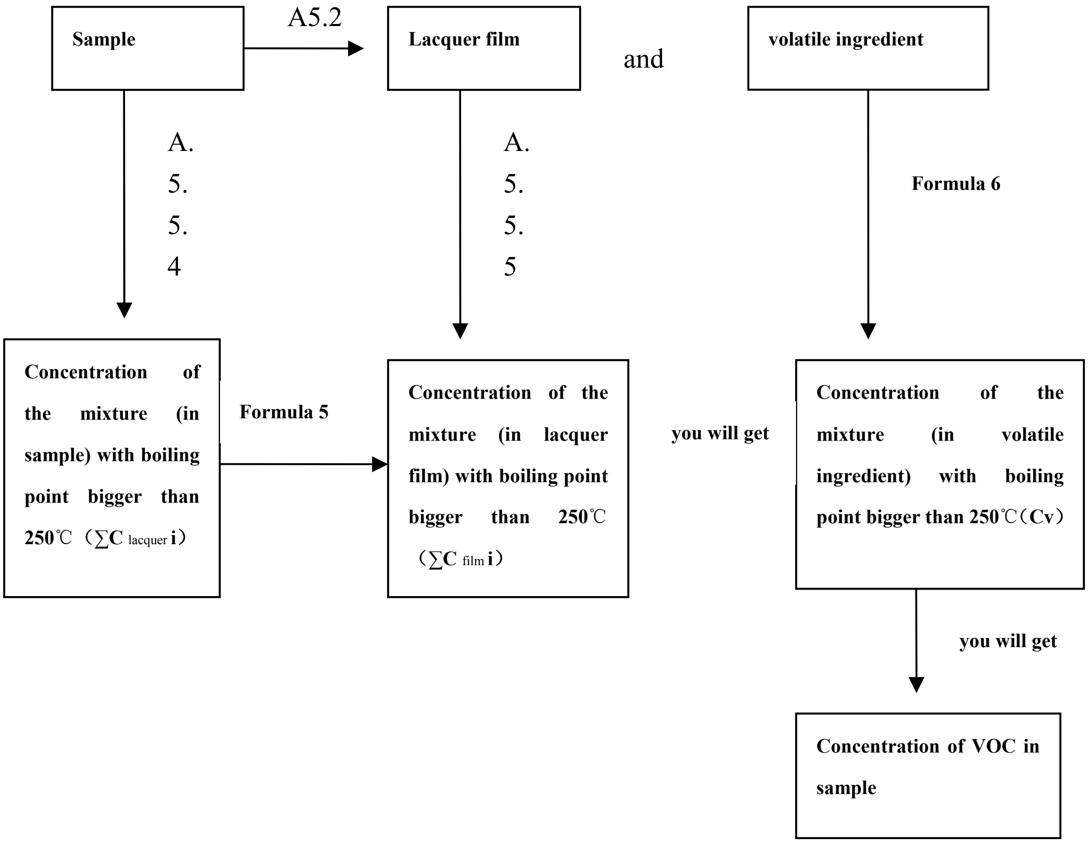


Figure A.1 Principle Flowchart

A.2 Instrument and equipment

A.2.1 Gas chromatograph

Instruments and equipment shall be installed and utilized in line with relevant explanations of the manufacturer. All instrument components exposing to test samples shall be made of the materials (e.g. glass) which tolerate to the sample and shall not have any chemical change in themselves.

A.2.2 Sample introduction system equipped with splitting device

Gas chromatograph shall have a sample inlet equipped with splitting device. The splitting ratio shall be subject to adjustment and supervision. The lining tube of sample inlet shall have silanized glass fiber to retain the non-volatile ingredients. Clean the lining tube and add new glass fiber or replace the tube if necessary to eliminate the error due to viscous substance or pigment residues (e.g. adsorbed compounds). The drag-out tail of the chromatographic peak may indicate possible adsorption, especially low volatile ingredients.

A.2.3 Column oven

The temperature of the column oven shall be subject to isothermal and programmed temperature rising operation from 40℃ to 300℃. The temperature fluctuations of the column oven shall not exceed 1℃. The ultimate temperature of programmed rising temperature shall not exceed the maximum application temperature of the capillary column.

A.2.4 Detector

Any one of the following detectors may be employed in the test.

A.2.4.1 Flame ionization detector (FID)

FID shall be operated under the temperature as high as 300℃. To prevent condensation, the temperature of detector shall be at least 10℃ higher than the maximum application temperature of column oven. Optimized treatment shall be carried out for the gas supply, sample injection amount, splitting ratio and gain setting of the detector so that the message for calculation (peak area) is in proportion with the amount of substance.

A.2.4.2 Mass spectrograph or other mass selection detector subject to calibration and tuning.

A.2.4.3 Calibrated Fourier transform infrared spectrometer (FT-IR).

A.2.5 Capillary column

Capillary column shall be made of glass or quartz glass. The length of the column shall be long enough to separate VOC ingredients. The maximum internal diameter may be 0.32 mm coated with fixed phase with appropriate film thickness, e.g. dimethylpolysiloxane to provide good peak separation.

The analyst may choose the above fixed phase and column length to meet the requirement for special separation.

A.2.6 Qualitative analysis equipment

If the identification of the separated ingredient requires mass selection detector or FT-IR, the instrument shall be connected with gas chromatograph and operated in line with relevant instructions of the manufacture.

A.2.7 Syringe

Syringe shall have at least the volume of 2 times of that of sample introduction.

A.2.8 Sample bottle

Sample bottles made of inert chemical materials (e.g. glass) shall be used. The analyst may use appropriate diaphragm (e.g. rubber diaphragm with PTFE coating) stopper to seal up these sample bottles.

A.2.9 Gas filter

Gas filter shall be installed at the connecting tube of gas chromatograph to absorb residual impurity in the gas.

A.2.10 Gas

A.2.10.1 Carrier gas: dry oxygen-free helium gas, nitrogen gas or hydrogen gas with purity at least 99.995% (volume fraction).

A.2.10.2 Detector gas (burning gas and assistant burning gas): They shall be the hydrogen gas and air (prepared) with the purity of at least 99.995% (volume fraction) and be free of any organic compounds.

A.2.10.3 Supporting gas (isolated sweeping gas and tail sweeping gas): nitrogen gas or helium gas with same property of the carrier gas.

A.3 Reagent

A.3.1 Internal standard

Internal standard substance shall be the compound not exiting in the original sample and can be completely separated from other ingredients in the chromatogram map. It shall be a kind of compound inert to the ingredients of the sample (i.e. no chemical reaction with the ingredients of the sample), stable within the testing temperature scope with known purity, e.g. dimethyl (*o*-) phthalate, diethyl (*o*-) phthalate, etc..

A.3.2 Calibration compounds

The compound for calibration shall have the purity of at least 99% (mass fraction) or with known purity.

A.3.3 Dilution solvent

Organic solvent suitable for sample dilution shall have the purity of at least 99% or with known purity. However, it shall not contain any substances that disturb the determination, e.g. substances causing overlap peaks in the chromatogram map. Test on injection solvent shall be conducted separately in order to observe pollutants and any possible disturbing peaks. The diluting solvent may be such solvents as acetic ether, methanol and *n*-hexane.

A.3.4 Labeled substance

It refers to the VOC compounds and non-VOC compounds distinguished according to the definition of VOC with boiling point near (250±3)℃ and known purity. Use diethyl adipate (boiling point at 251℃) as the labeled substance to distinguish the organic compounds with boiling point lower or equal to 250℃.

A.4 Sampling

Referring to GB/T 3186, take representative sample to conduct the determination.

A.5 Test Procedure

A.5.1 Density

The determination of density shall comply with GB/T 6750－1986.

A.5.2 Determination of volatile **(V)**

The determination of volatiles (V) shall comply with GB/T 6751－1986.

In case no organic compounds with boiling point higher than 250℃ after A.5.5 test are found in the product, calculate VOC concentration in the product according to formula (1).

$$VOC = V \times \rho_S \times 1000 \qquad (1)$$

Where VOC —— VOC concentration in the product, g/L;

V —— mass fraction of VOC in the testing sample, %;

ρ_S —— density of the testing sample at 23℃, g/ml;

1000 —— conversion factor.

A.5.3 Gas chromatograph analysis conditions

A.5.3.1 The analysis conditions of the gas chromatograph in use depends on the products ready for analysis. Calibration mixture with known concentration shall be used each time for optimization treatment. See Practical Case 2 of Chromatographic Conditions in Annex C.

A.5.3.2 The injection amount shall match with splitting ratio in order not going beyond the capacity of the column and falling into the linear scope of the detector. Asymmetric peaks indicate that gas chromatograph may be overloaded.

A.5.4 Qualitative analysis of the product

If the product contains unknown organic compounds with boiling point higher than 250℃, qualitative identification is required. Inject the labeled substance into gas chromatograph, determine its retention time on polydimethylsiloxane capillary column, and conduct qualitative identification of each compound with retention time longer than that of the labeled substance. Relatively good method is the combination of gas chromatograph with mass selection detector (A.2.4.2) or FT-IR (A.2.4.3) with chromatographic settings and conditions specified in A.5.3.

A.5.5 Calibration

A.5.5.1 If the compounds suitable for calibration are available in the market, the following method shall be employed to determine the relative response factor.

A.5.5.1.1 Weigh certain amount (with accuracy to 0.1 mg) the identified compound in A.5.4 and put it into a sample bottle. The weighed amount shall be at the same order of magnitude with the individual concentration in the subject product.

Weigh the internal standard (A.3.1) with similar mass to the subject compound and put it into the same sample bottle, use diluting solvent (A.3.3) to dilute the mixture. Then carry out the separation and determination under then same conditions of the test sample.

A.5.5.1.2 Optimize instrument's parameters in line with A.5.3.

A.5.5.1.3 Re-inject appropriate amount of calibration mixture into the gas chromatograph, use the following formula to calculate relative response factor of each compound:

$$f_i = \frac{m_{ci} \times A_{is}}{m_{is} \times A_{ci}} \qquad (2)$$

Where f_i — relative response factor of compound *i*;

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

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

A_{is} — peak area of the internal standard;

A_{ci} —peak area of compound *i*.

The relative error of two testing results shall be no more than 5％.

A.5.5.2 In case that there is unidentified chromatographic peak or calibration compound not available, it shall be assumed that relative response factor is 1.0.

A.5.5.3 Inject the labeled substance into the gas chromatograph, determine its retention time at polydimethylsiloxane capillary column in order to identify the integral starting point of the chromatographic map.

A.5.5.4 Determination of the organic compounds with boiling point higher than 250℃ in the paint: accurately weigh appropriate amount of paint sample into a 10 ml sample bottle, employ diluting solvent to dilute it, add the internal standard substance with mass similar to the analyte into the bottle, shake it until the solution is homogeneous. Determine the concentration according to the setting and conditions of gas chromatograph specified in A.5.3, then use formula (3) for calculation.

$$C_{\text{漆} i} = \frac{A_i \times m_s}{A_s \times f_i \times S_{\text{漆}}} \times 100 \qquad (3)$$

Where $C_{\text{漆}i}$—— mass fraction of organic compound i (in the coat) with boiling point higher than 250℃, %;

f_i—— relative response factor of the analyte i;

A_i—— peak area of the analyte i;

$S_{\text{漆}}$—— sample mass, g;

A_s—— peak area of the internal standard;

m_s—— mass of the internal standard, g.

The relative error of two test results shall be no more than 10%.

A.5.5.5 Determination of the organic compounds with boiling point higher than 250℃ in the lacquer film: accurately weigh dried lacquer film specified in A.5.2 into a 10 ml sample bottle, employ diluting solvent to dilute, add the internal standard substance with mass similar to the analyte into the bottle, shake it until the solution is homogeneous. Determine the concentration according to the setting and conditions of gas chromatograph specified in A.5.3, then use formula (4) to calculate the concentration of organic compounds (C) with boiling point higher than 250℃ in the lacquer film.

$$C_{膜i} = \frac{A_i \times m_s}{A_s \times f_i \times S_{膜}} \times 100 \qquad (4)$$

Where $C_{膜i}$—— mass fraction of organic compound i (in the lacquer film) with boiling point higher than 250℃, %;
f_i——relative response factor of the analyte i;
A_i—— peak area of the analyte i;
$S_{膜}$—— sample mass, g;
A_s——peak area of the internal standard;
m_s——mass of the internal standard, g.
The relative error of two test results shall be no more than 10%.

A.6 Calculating VOC Concentration in the Product

Calculate the volatile amount of organic compound (Cv) with boiling point higher than 250℃ during the determination of volatile ingredients according to formula (5).

$$C_V = \sum_{i=1}^{i=n} C_{漆i} - \sum_{i=1}^{i=n} C_{膜i} \times (1 - V) \qquad (5)$$

Where Cv——mass fraction of volatile organic compound with boiling point higher than 250℃, %.

Calculate the amount of volatile organic compounds (VOC), g/L, with boiling point lower than 250℃ according to formula (6).

$$VOC = (V - C_v) \times \rho_S \times 1000 \qquad (6)$$

Where V — mass fraction of VOC in test sample, %;
ρ_S—— density of test sample at 23℃, g/ml.
When calculating VOC value, whole number shall be taken.

A.7 Accuracy

A.7.1 Repeatability

Under the same conditions, the relative error of test results on the same testing substance by the same operator shall comply with Table A.1.

A.7.2 Reproducibility

The relative error of test results on the same testing substance by different operator in different labs shall comply with that in Table A.1.

Table A.1 Test Results of Several Labs

Parameter	Polyurethane coatings	Alkyd coatings	Nitro-coatings
Amount of labs	5	5	5
Number of repeated tests	4	3	4
Relative deviation of	1	1	3

repeatability, %			
Relative deviation of reproducibility, %	5	2	5

Annex B

(Standard Annex)

Determination of Methanol, Benzene, Toluene， Ethyl Benzene and Xylene in Coatings — Gas Chromatography

B.1 Application Scope

This method is applicable to the determination of the concentrations of methanol, benzene, toluene, ethyl benzene and xylene in coatings.

B.2 Method and Principle

After dilution by diluting solvent, the sample is directly injected into a gas chromatograph. The subject ingredients are separated by capillary column, employ hydrogen flame ionization detector for determination with quantity determined by internal standard method.

B.3 Instrument and Equipment

B.3.1 Gas chromatograph

The instrument and equipment shall be installed and utilized according to relevant instructions of the manufacturer. All instrument components exposing to testing sample shall be made of materials (e.g. glass) which tolerate the sample and shall not have any chemical change.

B.3.2 Sample introduction system with splitting device

Gas chromatograph shall be equipped with a sample inlet for splitting device. The splitting ratio shall be adjustable and subject to supervision and control. The lining tube of sample inlet shall have silanized glass fiber to retain the non-volatile ingredients. Clean the lining tube and add new glass fiber or replace the tube if necessary to eliminate the error due to viscous substance or pigment residues (e.g. adsorbed compounds). The dragged tail of the chromatographic peak may indicate possible adsorption, especially low volatile ingredients.

B.3.3 Column oven

The temperature of the column oven shall be subject to isothermal and programmed temperature rising operation from 40℃ to 300℃. The temperature fluctuations of the column oven shall not exceed 1℃. The ultimate temperature of programmed rising temperature shall not exceed the maximum application temperature of the capillary column.

B.3.4 Detector

Flame ionization detector (FID) can has the application temperature as high as 300℃. To prevent condensation, the temperature of detector shall be at least 10℃ higher than the application temperature of the column. Optimized treatment shall be carried out for the gas flow rate, sample injection amount, splitting ratio and gain setting of the detector so that the message for calculation (peak area) is in proportion with the amount of substance.

B.3.5 Capillary column

Capillary column shall be made of glass or quartz glass. The length of the column shall be long enough to separate VOC ingredients. The maximum internal diameter may be 0.32 mm coated with dimethylpolysiloxane with appropriate film thickness to provide good peak separation.

The analyst may choose the fixed phase and column length to meet the requirement for special separation.
Refer to Case 1 of Chromatographic Conditions in Annex C.

B.3.6 Syringe
The capacity of syringe shall be at least 2 times of the sample introduction amount.
B.3.7 Sample bottle
Sample bottle made of inert chemical materials (e.g. glass) shall be employed and sealed up by appropriate stopper (e.g. rubber film with PTFE coating).

B.3.8 Gas filter
Gas filter shall be installed at the connecting tube of gas chromatograph to absorb residual impurities in the gas.

B.3.9 Gas
B.3.9.1 Carrier gas: dry oxygen-free helium gas, nitrogen gas or hydrogen gas with purity at least 99.995% (volume fraction).
B.3.9.2 Detector gas (burning gas and assistant burning gas): They shall be the hydrogen gas and air (prepared) with the purity of at least 99.995% (volume fraction) and be free of any organic compounds.
B.3.9.3 Supporting gas (isolated sweeping gas and tail sweeping gas): nitrogen gas or helium gas with same property of the carrier gas.

B.4 Reagents
B.4.1 Calibration compounds
The compound for calibration shall have the purity of at least 99% (mass fraction) or with known purity.

B.4.2 Dilution solvent
Organic solvent suitable for sample dilution shall have the purity of at least 99% or with known purity. However, it shall not contain any substances that disturb the determination of benzene series substance, e.g. substances causing overlap peaks of benzene series substance in the chromatogram map. Test on injection solvent shall be conducted separately in order to observe pollutants and any possible disturbing peaks. The diluting solvent may be such solvents as methanol, *n*-hexane, acetic ether for example.

B.4.3 Internal standard
Internal standard substance shall be the compound not exiting in the original sample and can be completely separated from other ingredients in the chromatogram map. It shall be a kind of inert compound with respect to the ingredients of the sample, stable within the testing temperature scope with known purity, e.g. *n*-dodecane and dimethyl (*o*-) phthalate, etc.

B.5 Sampling
Take representative samples to conduct the determination (the sample taken from

multi-ingredients coatings shall be representative for each ingredient).

B.6 Test Procedures

B.6.1 Gas chromatograph analysis conditions

B.6.1.1 The analysis conditions of the gas chromatograph in use depends on the products ready for analysis. Calibration mixture with known concentration shall be used each time for optimization treatment. See Practical Case 1 of Chromatographic Conditions in Annex C.

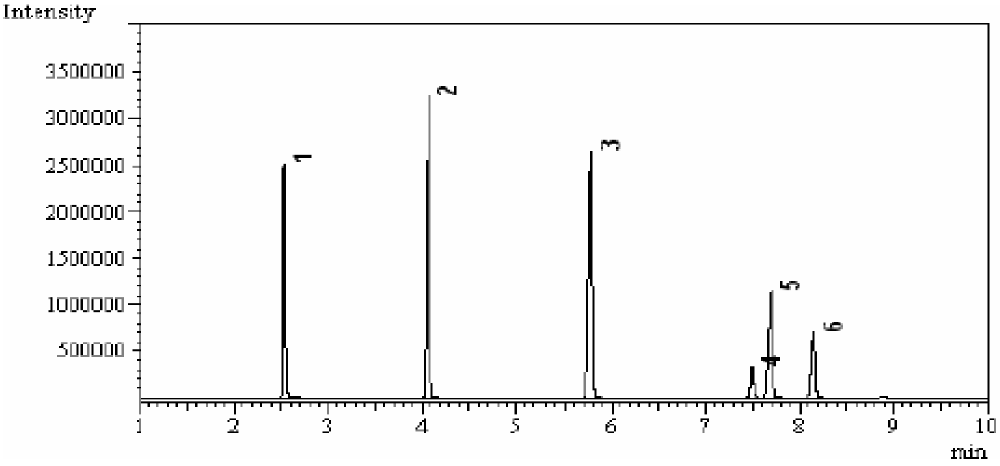
B.6.1.2 The sample injection amount shall match with splitting ratio in order not to go beyond the capacity of the column and to fall into the linear scope of the detector. Asymmetric peaks indicate that gas chromatograph may be overloaded.

B.6.2 Qualitative analysis of the product

B.6.2.1 Optimize the parameters of instruments according to B.6.1.

B.6.2.2 Determination of retention time of the analyte: inject 1 μl solution containing the standard substance of the analyte specified in B.4.1. Record the retention time of the standard ingredient of each analyte. The peak sequence of standard ingredient of each analyte is shown in Figure B.1.

B.6.2.3 Qualitative analysis of the ingredient of the sample: take appropriate amount of sample, employ diluting solvent (B.4.2) and dilute it into a 10 ml volumetric flask, inject 1 μl of the solution into gas chromatograph and determine if there is any subject substance from Figure B.1.



1-methanol, 2-benzene, 3-toluene, 4-ethyl benzene, 5-xylene (*p*-,*m*-), 6-xylene (*o*-)

Figure B.1 Peak Sequence of the Analytes at Dimethylpolysiloxane Capillary Column

B.6.3 Calibration

B.6.3.1 Separately weigh the calibration compound. The weighed amount shall be close to the corresponding concentration in tested product with accuracy of 0.1 mg, inject it into a sample bottle (B.3.7).

Weigh similar amount of internal standard (B.4.3) and inject it into a sample bottle. Employ diluting solvent (B.4.2) to dilute the mixture, inject it into gas chromatograph at the same conditions with that of the test sample.

B.6.3.2 Re-inject appropriate amount of calibration mixture into the gas chromatograph and use the following formula to calculate the response factor of the analyte.

$$r_i = \frac{m_{ci} \times A_{is}}{m_{is} \times A_{ci}} \qquad (1)$$

Where r_i—— response factor of the analyte i ;
m_{is}—— mass of the internal standard in the standard mixture, g;
m_{ci}—— mass of compound i in the standard mixture, g;
A_{is}—— peak area of the internal standard;
A_{ci}——peak area of the analyte i.
r shall take the average of the two test results with relative error less than 5%. It shall retain three significant figures.

B.6.4 Preparation of sample
Weigh 1 g sample (with accuracy to 0.1 mg), inject the internal standard with mass similar to the mass of analyte that is injected into the sample bottle. Employ appropriate amount of diluting solvent to dilute the sample, shake the solution until it is homogeneous.

B.6.5 Determination of the concentration of compounds
B.6.5.1 When doing calibration, the analyst shall set the instrument parameter in optimum way.
B.6.5.2 Inject 1 μl sample into gas chromatograph, record the chromatographic map. Determine the peak area of analyte, use formula (2) to calculate methanol and benzene concentrations in the coatings, use formula (3) to calculate benzene, toluene and xylene concentrations in the coatings.

$$m_{\text{methanol, benzene}} = (r_i \times A_i \times m_{is}) \times 1000 \div (m_s \times A_{is}) \qquad (2)$$

$$m_{\text{hazardous substance}} = (r_i \times A_i \times m_{is}) \times 1000 \div (m_s \times A_{is}) \qquad (3)$$

Where
m_{benzene}—— mass fraction of methanol and benzene in the product, %;
$m_{\text{hazardous substance}}$—— mass fraction of toluene, ethyl benzene and xylene in the product, %;
r_i—— response factor of compound i ;
A_i—— peak area of compound i;
A_{is}—— peak area of the internal standard;
m_{is}—— mass of the internal standard in test sample, μg;
m_s—— mass of test sample, g.
Take the average value of two test results as the determination value with relative error less than 5%. The calculated result shall retain to first number .

Annex C

(Information Annex)

Practical Case of Gas Chromatograph Testing Conditions

C.1 Case 1 —— gas chromatographic conditions for the determination of methanol, benzene, toluene , ethyl benzene and xylene

Chromatographic column: capillary column with dimethylpolysiloxane coat, 30 m × 0.25 mm (internal diameter)× 0.25 µm film thickness;

Sample inlet temperature: 240℃;

Detector temperature: 280℃;

Column temperature: programmed temperature rise, keep 36℃ for 5 min, raise it to 280℃ at the rate of 10℃/min and maintain at 280℃ for 5 min；

Carrier gas: highly pure nitrogen gas;

Pre-column pressure: 35 kPa；

Splitting ratio: the injecting sample is split with adjustable splitting ratio;

Sample introduction volume: 1.0 µl.

C.2 Case 2 —— gas chromatographic conditions for the determination of VOC

Chromatographic column: capillary column with dimethylpolysiloxane coat, 30 m×0.25 mm (internal diameter) × 0.25 µm film thickness;

Sample inlet temperature: 300℃；

Detector temperature: 300℃；

Column temperature: programmed temperature rise to 160℃ and keep that temperature for 1min，then raise to 290℃ at the rate of 10 /min ℃ and maintain that temperature for 15 min;

Carrier gas: highly pure nitrogen;

Flow-rate of carrier gas: 1.2ml/min;

Splitting ratio: the injecting sample is split with adjustable splitting ratio;

Sample introduction volume: 1.0 µl.

—