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AUTOMOTIVE INDUSTRY STANDARD
OF THE PEOPLE'S REPUBLIC OF CHINA

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QC/T 942-2013

**Test methods of hexavalent chromium in
automobiles materials**

汽车材料中六价铬的检测方法

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“*Methyl Butenol Polyether*” and other 811 industrial standards are approved by the Ministry of Industry and Information Technology. In which - Chemical industry standard: 149 items; non-ferrous industry standard: 105 items; gold industry standard: 5 items; metallurgical industry standard: 15 items; building materials industry standard: 3 items; machinery industry standard: 39 items; aviation industry standard: 69 items; shipbuilding industry standard 53 items; automobile industry standard: 42 items; textile industry standard: 63 items; light industry standard: 59 items; petrochemical industry standard: 42 items; civil explosive industry standard: 1 item; electronics industry standard: 50 items; communications industry standard: 116 items.

Above chemical industry standards are published by Chemical Industry Press; textiles, non-ferrous and gold industry standards are published by China Standard Press; metallurgical industry standards are published by Metallurgical Industry Press; building materials industry standards are published by the Building Materials Industry Press; machinery industry standards are published by Machinery Industry Press; the aviation industry standards are published by China Aviation Integrated Technical Institute Organization; the shipbuilding industry standards are published by China Shipbuilding Technology and Economy Institute Organization; the automobile industry standards are published by the China Planning Press; light industry standards are published by China Light Industry Press; the petrochemical industry standard is published by the China Petrochemical Press; civil explosive industry standards are published by China Ordnance Industry Standard Institute Organization; electronic industry standards are published by the Ministry of Industry and Information Technology and electronics industry Standardization Institute organization; communication industry standards are published by the People's Posts and Telecom Press.

Annex: The Standard number, standard name and initial implementation date of 42 automobile industries.

Ministry of Industry and Information Technology of the People's Republic of China

October 17, 2013

Attachment:
Number, Name and Initial Implementation Date of the 42
Automobile Industry Standards

| S/N | Standard No. | Standard Name | Replaced Standard No. | Implementation Date |
|-----|-------------------|--|--------------------------------|---------------------|
| 355 | QC/T 253-2013 | Preparation Methods for Engine Model used in Motorcycles and Mopeds | QC/T 253-1998 | 2014-03-01 |
| 356 | QC/T 682-2013 | Seats Used in Motorcycles and Mopeds | QC/T 682-2002 | 2014-03-01 |
| 357 | QC/T 229-2013 | Technical Conditions for Rotor Pump of Motorcycles and Mopeds | QC/T 229-1997 | 2014-03-01 |
| 358 | QC/T 952-2013 | Disc Wheels for Passenger Car — Dimensional of Attachment on Hub | | 2014-03-01 |
| 359 | QC/T 953-2013 | Commercial Road Vehicles — Flat Attachment Wheel Fixing Nuts | | 2014-03-01 |
| 360 | QC/T 954-2013 | Commercial Vehicles — Flat Attachment Fixing Nuts — Test Methods | | 2014-03-01 |
| 361 | QC/T 258-2013 | Test Methods for the Intensity of Vehicle Wheels and Screw Base | QC/T 258-1998 | 2014-03-01 |
| 362 | QC/T 199-2013 | Vehicle Wheels - Balance Weight | QC/T 199-1995 | 2014-03-01 |
| 363 | QC/T 326-2013 | Numbering Rules for Automobile Standardized Parts | QC/T 326-1999 | 2014-03-01 |
| 364 | QC/T 955-2013 | Auto Leveling Device of Special Purpose Vehicle | | 2014-03-01 |
| 365 | QC/T 956-2013 | Transport Vehicle for Dry-mixed Mortar | | 2014-03-01 |
| 366 | QC/T 957-2013 | Cleaning Sweeper Truck | | 2014-03-01 |
| 367 | QC/T 29104-2013 | Method for Coding the Level of Contamination by Solid Particles of Special Purpose Vehicle Hydraulic System | QC/T 29104-1992 | 2014-03-01 |
| 368 | QC/T 29105.3-2013 | Sampling Methods of Testing Particulate Contamination of Hydraulic Oil of Special Purpose Vehicle Hydraulic System | QC/T 29105.3-1992 | 2014-03-01 |
| 369 | QC/T 718-2013 | Truck Mounted Concrete Pump | QC/T 718-2004 | 2014-03-01 |
| 370 | QC/T 439-2013 | Swept-body Dump Truck | QC/T 439-1999 QC/T 440-1999 | 2014-03-01 |
| 371 | QC/T 935-2013 | Kitchen Garbage Vehicle | | 2014-03-01 |
| 372 | QC/T 939-2013 | Technical Qualifications of Front Discharge Truck | | 2014-03-01 |
| 373 | QC/T 457-2013 | Ambulance | QC/T 457-2002 | 2014-03-01 |
| 374 | QC/T 936-2013 | Detachable Container Garbage Collector | | 2014-03-01 |
| 375 | QC/T 937-2013 | Guardrail Repair Car | | 2014-03-01 |
| 376 | QC/T 940-2013 | Exhibition Vehicle | | 2014-03-01 |
| 377 | QC/T 958-2013 | Performance Requirements and Bench Test Methods of Automobile Vacuum Pump | | 2014-03-01 |
| 378 | QC/T 592-2013 | Performance Requirements and | QC/T 592-1999 | 2014-03-01 |

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|-----|-----------------|---|---------------|------------|
| | | Bench Test Methods for Hydraulic Brake Caliper Assembly | | |
| 379 | QC/T 959-2013 | Performance Requirements and Bench Test Methods for Mechanical Parking Brake Lever Assembly | | 2014-03-01 |
| 380 | QC/T 960.1-2013 | Road Vehicle- Hydraulic Braking Systems – Part 1: Double – Flare Pipes, Tapped Holes, Male Fittings and Tube Seats | | 2014-03-01 |
| 381 | QC/T 961-2013 | Performance Requirements and Bench Test Methods for Plastic Liquid Storage Tank of Hydraulic Braking Systems | | 2014-03-01 |
| 382 | QC/T 949-2013 | Specification for Audio Player on Board | | 2014-03-01 |
| 383 | QC/T 951-2013 | Circuits-breaker for Automobiles | | 2014-03-01 |
| 384 | QC/T 490-2013 | Drawings for Motor Vehicle Body | QC/T 490-2000 | 2014-03-01 |
| 385 | QC/T 950-2013 | Performance Requirement and Test of Heating Car Cushion | | 2014-03-01 |
| 386 | QC/T 948-2013 | Roof Load Carriers for Road Vehicles | | 2014-03-01 |
| 387 | QC/T 946-2013 | Strength Requirement and Test of Automobile Safe Belt Strap | | 2014-03-01 |
| 388 | QC/T 945-2013 | Passenger Vehicle Air-conditioning Unit | | 2014-03-01 |
| 389 | QC/T 627-2013 | Electronic Locks System for Motor Vehicles | QC/T 627-1999 | 2014-03-01 |
| 390 | QC/T 662-2013 | Vehicle Air Conditioner (HFC-134a) Receiver Dryer | QC/T 662-2000 | 2014-03-01 |
| 391 | QC/T 947-2013 | Technology Standards for Vehicle Auto-Dimming Rearview Mirror | | 2014-03-01 |
| 392 | QC/T 941-2013 | Test Methods of Mercury in Automobiles Materials | | 2014-03-01 |
| 393 | QC/T 943-2013 | Test Methods of Lead and Cadmium in Automotive Materials | | 2014-03-01 |
| 394 | QC/T 942-2013 | Test methods of Hexavalent Chromium in Automobiles Materials | | 2014-03-01 |
| 395 | QC/T 944-2013 | Determination of Polybrominated Biphenyls (PBBs) and Polybrominated Diphenyl Ethers (PBDEs) in Automobiles Material | | 2014-03-01 |
| 396 | QC/T 938-2013 | Test Specification of Protection for Pedestrians in the Event of a Collision | | 2014-03-01 |

Table of contents

| | |
|---|----|
| Foreword..... | 6 |
| Introduction | 8 |
| 1 Scope | 9 |
| 2 Normative references | 9 |
| 3 X-ray fluorescence spectrometry..... | 10 |
| 4 Qualitative test of hexavalent chromium in metallic anticorrosive coating | 14 |
| 5 Determination of hexavalent chromium in metallic anticorrosive coatings | 18 |
| 6 Determination of hexavalent chromium content in polymeric and electronic materials | 21 |
| 7 Determination of hexavalent chromium content in leather materials | 26 |
| 8 Test report..... | 32 |
| Appendix A (Informative) Calculation method for surface area of fastener coating | 33 |
| Appendix B (Normative) Determination of hexavalent chromium content in polymeric materials and electronic materials - Determination of recovery rate and detection limit..... | 37 |
| Appendix C (Normative) Determination of volatile matter content in leather materials | 40 |
| Appendix D (Normative) Determination of hexavalent chromium content in leather materials - Determination of recovery rate | 42 |

Foreword

This standard was drafted in accordance with the rules given in GB/T 1.1-2009 "Directives for standardization - Part 1: Structure and drafting of standards".

This standard includes five methods.

Method 1 "X-ray fluorescence spectrometry" was drafted with reference to GB/Z 21277-2007 "Rapid screening of lead mercury chromium cadmium and bromine of regulated substances in electrical and electronic equipment - X-ray fluorescence spectrometry".

Method 2 "Qualitative determination of hexavalent chromium in metallic anticorrosive coatings" was drafted with reference to the Appendix B of IEC 62321: 2008 Ed.1.0 "Electro-technical products - Determination of levels of six regulated substances (lead, mercury, cadmium, hexavalent chromium, polybrominated biphenyls, and polybrominated diphenyl ethers).

Method 3 "Determination of hexavalent chromium in metallic anticorrosive coatings" was drafted with reference to ISO 3613:2000 "Chromate conversion coatings on zinc, cadmium, aluminum-zinc alloys, and zinc-aluminum alloys - Test methods".

Method 4 "Determination of hexavalent chromium content in polymer and electronic materials" was drafted with reference to Appendix C of IEC 62321: 2008 Ed.1.0 "Electro-technical products - Determination of levels of six regulated substances (lead, mercury, cadmium, hexavalent chromium, polybrominated biphenyls, and polybrominated diphenyl ethers)". As for its consistency with IEC 62321:2008 Ed.1.0, it is inequivalent. As compared with the Appendix C of international standard, this method has the consistent technical content, BUT has significant difference in standard frame.

Method 5 "Determination of hexavalent chromium content in leather materials" was drafted in accordance with EN ISO 17075:2007 "Leather - Chemical Tests - Determination of chromium (VI) content", BUT it is inequivalent to EN ISO 17075:2007. As compared with the international standard, this method has the same technical content, BUT the standard framework has changed significantly, which details the sample preparation conditions in accordance with the references of the international standard, AND details the determination of the volatile matter content in the sample in accordance with the references of the international standard AND incorporate it in Appendix C of this standard (normative appendix).

Test methods of hexavalent chromium in automobiles materials

1 Scope

This standard specifies the methods for the determination of hexavalent chromium in automobile parts and materials, wherein:

“X-ray fluorescence spectrometry” is applicable to the use of X-ray fluorescence spectroscopy screening and rapid determination of hexavalent chromium content in the automobiles materials.

The “qualitative test of hexavalent chromium in metallic anticorrosive coatings” is applicable to determine the presence of hexavalent chromium in automobiles anticorrosive coating by drip test and boiling water extraction method. “Determination of hexavalent chromium in metallic anticorrosive coatings” is applicable to quantitatively determine the hexavalent chromium content in automotive anticorrosive coatings by boiling water extraction and colorimetric method. The “qualitative test of hexavalent chromium in metallic anticorrosive coatings” and the “Determination of hexavalent chromium in metallic anticorrosive coatings” are applicable to the coatings without additional coverings (for example, oil film, water based or solvent based polymer film or wax film).

The “Determination of hexavalent chromium in polymer and electronic materials” is applicable to the determination of hexavalent chromium in automobile polymer materials and automobile electronic materials by alkaline solution extraction and colorimetric method. It is not applicable to polyethylene (PE) and ethylene - vinyl acetate copolymer (EVAC) material. The determination of hexavalent chromium content of other automobiles materials, such as glass, ceramic, fabric and paint coatings can make reference to this method.

“Determination of hexavalent chromium in leather materials” is applicable to the determination of hexavalent chromium content in automobiles leather materials by phosphate solution extraction and colorimetric method.

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 Standard.

GB/T 1839 Test method for gravimetric determination of mass per unit area of galvanized coatings of steel products (GB/T 1839-2008, ISO 1460:1992, MOD)

GB/T 8170 Rules for rounding off for numerical values & expression and judgment of limiting values

GB/T 20017-2005 Metallic and other inorganic coatings - Measurement of mass per unit area - Review of gravimetric and chemical analysis methods (GB/T 20017-2005, ISO 10111:2000, IDT)

GB/T 30512-2014 Requirements for prohibited substances on automobiles

GB/Z 21277-2007 Rapid screening of lead mercury chromium cadmium and bromine of regulated substances in electrical and electronic equipment - X-ray fluorescence spectrometry

QB/T 2262-1996 Terms of leather industry

QB/T 2706 Leather - Chemical, physical, mechanical and fastness test - Sampling location (QB/T 2706-2005, ISO 2418:2002, MOD)

IEC 62321: 2008 Ed.1.0 Electro-technical products - Determination of levels of six regulated substances (lead, mercury, cadmium, hexavalent chromium, polybrominated biphenyls, and polybrominated diphenyl ethers)

ISO 3613:2000 Chromate conversion coatings on zinc, cadmium, aluminum-zinc alloys, and zinc-aluminum alloys - Test methods

EN ISO 17075:2007 Leather - Chemical Tests - Determination of chromium (VI) content

3 X-ray fluorescence spectrometry

3.1 Principles

PLACE the prepared sample in the X-ray fluorescence spectrometer sample chamber; based on the selected analysis mode, CONDUCT X-ray fluorescence spectrum analysis on the chromium content in the sample; and based on the screening limit, JUDGE whether the chromium content in the sample is qualified AND whether it is required for accurate measurement.

AND it shall pay special attention to the consistency of the sample thickness and the uniformity of composition. During analysis, to make the sample spread flatly, it may add lining materials as a support; AND it shall select the lining materials of low background as far as practicable.

As for the samples of various unshaped blocks and plates as well as the oversized samples, it may use a cutting machine or grinder to machine them to a suitable size. Small samples (such as plastic particles, etc.) will be made into the analysis samples through liquid nitrogen freezing, mechanical crushing, and then pressed into tablets by tableting machine.

As for the fragile samples such as glass and ceramics, firstly CRUSH them into small pieces; then GRIND it into powder less than 200 sieve (0.074 mm); MIX it uniformly; USE the boric acid (3.2.1) substrate to press it into tablet sample, which is not less than 1 mm of thickness; OR otherwise USE the anhydrous lithium tetraborate (3.2.2) to make it into glass frit analysis sample.

As for the non-homogeneous sample consisting of heterogeneous material, which is not required or difficult to be further mechanically resolved, CUT and CRUSH the sample, FRIZE it by liquid nitrogen, USE the grinder to grind the crushed sample into powdery sample of particle size not more than 1 mm, MIX it uniformly; USE boric acid (3.2.1) substrate to press it into tablet sample, which is not less than 1 mm of thickness; OR otherwise USE the anhydrous lithium tetraborate (3.2.2) to make it into glass frit analysis sample.

3.4.2.2 Liquid samples. PIPETTE a certain volume of liquid sample into the special liquid sample cup (sample thickness of at least 15 mm); at the bottom of the cup, USE a 6 mm thickness polyester film for support; and COVER the cup by a cover with holes.

3.5 Analysis steps

3.5.1 Instrument preparation

In accordance with the instrument operation instructions, TURN on the instrument, and PRE-HEAT the instrument until it runs stably.

3.5.2 Analysis spectral lines

As for the analysis of chromium element by the X-ray fluorescence spectrometry, it is recommended to select $K\alpha$ analysis line.

3.5.3 Drawing of working curve

SELECT the standard substance matched with the substrate of the sample to be measured; in accordance with the measurement condition of the X-ray

4.2.7 Hexavalent chromium standard solution: PIPETTE 0.50 mL of hexavalent chromium stock solution (4.2.6) into a 250mL volumetric flask, and DILUTE it to the mark. The hexavalent chromium content of this solution is 1 µg/mL. Tightly COVER the vessel, AND this solution shall be used within 24 h after preparation. Or otherwise USE the national standard solution to prepare the hexavalent chromium standard solution of hexavalent chromium content 1 µg/mL.

4.2.8 Coloring solution A: WEIGH 0.4 g of diphenylcarbazide (4.2.1) and DISSOLVE it in the mixture of 20 mL of acetone (4.2.3) and 20 mL of ethanol (4.2.4); After dissolved, ADD 20 mL of orthophosphoric acid solution (4.2.5) and 20 mL of water. This solution shall be used within 8 h after preparation.

4.2.9 Coloring solution B: WEIGH 0.5 g of diphenylcarbazide (4.2.1) and DISSOLVE it into 50 mL of acetone (4.2.3); slowly ADD 50 mL of water to dilute it while stirring (rapid mixing may produce diphenylcarbazide precipitation). This solution shall be refrigerated in a brown glass bottle, AND it shall not be used if there is any color change.

4.3 Instruments and equipment

4.3.1 Analytical balance: Accuracy of 0.1 mg.

4.3.2 Heating device which can maintain the boiling state of the extract.

4.3.3 Spectrophotometer: detection wavelength is 540 nm, with 1 cm or 1 cm above absorption vessel. It may also use other colorimeters that meet the accuracy requirements.

4.4 Sample preparation

4.4.1 Before the test, it shall place the sample in an environment of temperature not higher than 35 °C and relative humidity not higher than 75%.

4.4.2 During the test, the sample surface shall be free from contamination, fingerprints or other foreign stains. If the sample surface is coated with an oil film, it shall use detergent, soft cloth or suitable solvent to remove them at room temperature (≤ 35 °C). Note that the sample shall neither be forced to dry at temperatures above 35 °C nor be treated in alkaline solutions.

4.4.3 If there is polymer coating on the sample surface, it may select an effective method to remove the surface coating, BUT pay attention not to remove the anticorrosive coating from the sample surface.

4.5 Test

two beakers in an 1 cm absorption vessel; USE a spectrophotometer (4.3.3) to measure the absorbance at 540 nm for 3 times; TAKE the average value.

4.5.3.5 PIPETTE 1.0 mL of hexavalent chromium standard solution (4.2.7); DILUTE it to 50 mL. ADD 1 mL of orthophosphoric acid solution (4.2.8); MIX it uniformly; ADD 2.0 mL of coloring solution A (4.2.8) or coloring solution (B); MIX it uniformly; USE a spectrophotometer (4.3.3) to measure the absorbance at 540 nm for 3 times; TAKE the average value.

4.5.3.6 If the absorbance value obtained in 4.5.3.4 is equal to or greater than 4.5.3.5, the sample coating shall be considered to contain hexavalent chromium (positive). Otherwise, it is considered that the sample coating does not contain hexavalent chromium (negative).

4.5.4 If the results of the above-mentioned drip test and boiling water extraction test are negative, it indicates that the coating does not contain hexavalent chromium. Otherwise, USE the following “Determination of hexavalent chromium content in metallic anticorrosive coating” to measure the hexavalent chromium content in the coating.

5 Determination of hexavalent chromium in metallic anticorrosive coatings

5.1 Principles

USE the boiling water extraction method to extract the hexavalent chromium from the sample; ADJUST the pH value of the extract; under acidic conditions, ADD diphenylcarbazide solution; AND the hexavalent chromium in the extract reacts with the diphenylcarbazide, generating purple complex; at 540 nm wavelength, CONDUCT the quantitative colorimetric determination.

5.2 Reagents and materials

Unless otherwise stated, in the analysis only the reagents identified as analytically pure and distilled or deionized water or water of equivalent purity are used.

5.2.1 Sulfuric acid (1 +3).

5.2.2 Sodium dihydrogen phosphate.

5.2.3 Phosphate buffer solution: WEIGH 47.8 g of sodium dihydrogen phosphate (5.2.2) and DISSOLVE it in 100 mL of water.

with the diphenylcarbazide, generating purple complex; at 540 nm wavelength, CONDUCT the quantitative colorimetric determination.

6.2 Reagents and materials

Unless otherwise stated, in the analysis only the reagents identified as analytically pure and distilled or deionized water or water of equivalent purity are used.

6.2.1 Sulfuric acid (1 +9): excellent grade pure.

6.2.2 Nitric acid (ρ is 1.42 g/mL): excellent grade pure.

6.2.3 Anhydrous sodium carbonate.

6.2.4 Sodium hydroxide.

6.2.5 Anhydrous magnesium chloride.

6.2.6 Dipotassium hydrogen phosphate ($K_2HPO_4 \cdot 3H_2O$).

6.2.7 Potassium dihydrogen phosphate.

6.2.8 Phosphate buffer solution: WEIGH 87.09 g of dipotassium hydrogen phosphate (6.2.6) and 68.04 g of potassium dihydrogen phosphate (6.2.7); DISSOLVE them in 700 mL of water; TRANSFER it to a 1000 mL volumetric flask; DILUTE it to the mark. The pH of this phosphate buffer solution is 7, containing 0.5 mol/L of dipotassium hydrogen phosphate and 0.5 mol/L of potassium dihydrogen phosphate.

6.2.9 Alkaline extraction solution: WEIGH 20.0 g \pm 0.05 g of sodium hydroxide (6.2.4) and 30.0 g \pm 0.05 g of anhydrous sodium carbonate (6.2.3); DISSOLVE it in water; TRANSFER it into a 1000 mL volumetric flask; DILUTE it to the mark. SEAL and PRESERVE this extract at 20 °C ~ 25 °C in a polyethylene bottle; it is needed to re-prepare it every month. Before use, TEST the pH value, which shall be \geq 11.5.

6.2.10 Coloring solution C: WEIGH 0.5 g of diphenylcarbazide (4.2.1); DISSOLVE it in 100 mL acetone (4.2.3). PRESERVE this solution in a brown bottle; if its color changes, it shall not be used.

6.2.11 Liquid nitrogen: Industrial grade.

6.3 Instruments and equipment

6.3.1 Pulverizer.

6.5.2.2 Respectively PIPETTE a certain volume of the standard solution into the absorption vessel; USE the 0 mL hexavalent standard solution (6.5.2.1) as the blank; USE the spectrophotometer (4.3.3) to measure the absorbance at 540 nm. Based on the absorbance values and the concentration of hexavalent chromium in the standard solution, DRAW and FIT the calibration curve. The correlation coefficient of the calibration curve shall be ≥ 0.99 ; otherwise it shall be redrawn.

6.5.3 Extraction by alkaline extraction method

6.5.3.1 WEIGH 2.5 g of sample, accurate to 0.001 g. PLACE the sample in an extraction vessel. If the concentration of hexavalent chromium of the sample is too high or too low, it may adjust the mass of the weighed sample.

6.5.3.2 MEASURE 50 mL of the alkaline extract (6.2.9) and ADD it into the extraction vessel; then ADD 0.4 g of magnesium chloride (6.2.5) and 0.5 mL of the phosphate buffer solution (6.2.8); fully SHAKE it uniformly; COVER the watch glass.

Note:

1. Adding magnesium chloride can inhibit the oxidation reaction of chromium.
2. As for the polymer which easily floats on the surface of the extract, it may add 1 or 2 drops of wetting agent (e.g. Triton X), in order to increase the wettability of the sample.

6.5.3.3 HEAT the sample solution to 90 °C ~ 95 °C; MAKE it at constant temperature for at least 3 h before cooling to room temperature. In the heating, constant temperature, and cooling process, it shall continue stirring (6.3.4).

6.5.3.4 REMOVE the stirrer (6.3.4); USE the 0.45 μm filter (6.3.6) to filter the sample solution. USE water to rinse the extraction vessel for three times; RINSE the stirrer; FILTER the rinsing solution. If the 0.45 μm filter (6.3.6) is blocked, it may use a larger pore size filter to pre-filter the sample.

6.5.3.5 USE the water to rinse the filter bottle and filter (6.3.6); TRANSFER the filtrate and rinsing solution into a 250 mL vessel. RETAIN the filter cake on the filter membrane (6.3.6), and PRESERVE it at 4 °C \pm 2 °C for the purposes of evaluation when the hexavalent chromium substrate tracer recovery is low.

6.5.3.6 Under stirring, slowly DRIP the nitric acid (6.2.2) into this 250 mL vessel; ADJUST the pH of the solution to 7.5 \pm 0.5. If the solution is turbid AND there is flocculent precipitation at this time, USE the 0.45 μm filter (6.3.6) or slow filtration paper to filter it; as for the solution with color, USE the C18SPE

X - Measured hexavalent chromium concentration in the extract, $\mu\text{g/mL}$;

N - Dilution factor of extract (6.5.4.2);

V - Constant volume of extract, mL;

m - Weighed mass of sample, g.

The hexavalent chromium content is expressed in mass fraction (%) or milligrams per kilograms (mg/kg). When expressed in mass fraction (%), KEEP at least two significant figures; when expressed in milligrams per kilograms (mg/kg), ROUND it off to single digit in accordance with GB/T 8170.

6.6.2 Correction of detection results and detection limits

6.6.2.1 The determination of recoveries is in accordance with Appendix B.1, AND the determination of detection limits is in accordance with Appendix B.2.

6.6.2.2 If the recovery is greater than 75% and less than 125%, the sample detection results and detection limits do not need not be corrected.

6.6.2.3 If the recovery rate is greater than or equal to 10% and less than or equal to 75%, the sample detection results and detection limits shall be corrected based on the recovery rate. Based on the 100% recovery rate, CALCULATE the correction factor; and USE the correction factor to correct the detection results and detection limits.

6.6.2.4 If the sample detection result as obtained in accordance with 6.6.2.3 is greater than the detection limit of the sample, REPORT the corrected detection results; otherwise, REPORT the sample detection limit as the sample detection results.

6.7 Precision

The absolute difference between two parallel test specimens shall not exceed 20% of its arithmetical mean value; otherwise it shall conduct re-measurement.

7 Determination of hexavalent chromium content in leather materials

7.1 Principles

USE the phosphate buffer of pH 7.5 ~ 8.0 as the extraction solution to extract the hexavalent chromium from leather samples. If necessary, it may use the

7.5.1.1 USE the hexavalent chromium standard solution (4.2.7) to prepare the solution used for calibration curve. Within the range of 0.50 mL ~ 15 mL, USE at least 6 hexavalent chromium standard solutions to draw an appropriate calibration curve. The concentration range of the hexavalent chromium of the solution used for the calibration curve shall include the expected range of measured concentrations.

7.5.1.2 PIPETTE a given volume (volume range is 0.5 mL ~ 15 mL) of the hexavalent chromium standard solution (4.2.7) into a 25 mL volumetric flask. In each flask, ADD 0.5 mL of phosphate solution (7.2.4) and 0.5 mL of coloring solution D (7.2.6); then ADD the phosphate buffer solution (7.2.5) and DILUTE it to the mark; MIX it uniformly and LET it standing for 15 min \pm 5 min.

7.5.1.3 Using the blank solution (7.5.2) as reference, USE the spectrophotometer and a 4 cm absorption vessel (7.3.7) to measure the absorbance of the solution at 540 nm. Based on the absorption value and the hexavalent chromium standard solution concentration, DRAW the calibration curve.

7.5.2 Preparation of blank solution

ADD phosphate buffer solution (7.2.5) to three-quarters of the volume of the 25 mL volumetric flask; ADD 0.5 mL of phosphoric acid solution (7.2.4) and 0.5 mL of coloring solution D (7.2.6); then use the phosphate buffer solution (7.2.5) to dilute it to the mark; MIX it uniformly. This solution shall be prepared every day AND stored in the dark.

7.5.3 Extraction by phosphate extract

7.5.3.1 WEIGH 2 g \pm 0.01 g prepared sample, accurate to 0.001 g.

7.5.3.2 PIPETTE 100 mL of phosphate buffer solution (extract) (7.2.5) into a 250 mL conical flask. INSERT the airway (7.3.2) (airway does not touch the liquid surface); USE argon or nitrogen (7.2.7) into the conical flask to remove oxygen; VENT for 5 min at the flow rate of 50 mL/min \pm 10 mL/min.

7.5.3.3 REMOVE the airway (7.3.2); ADD the weighed sample; COVER the stopper; READ the extract volume V_0 .

7.5.3.4 USE the mechanical oscillator (7.3.1) to shake the leather power suspension for 3 h \pm 5 min, in order to extract the hexavalent chromium. Stably SHAKE the suspension to avoid sample powder from adhering to the vessel wall; AVOID shaking too fast.

7.5.3.5 After extraction for 3 h, immediately USE the membrane filter (7.3.6) to filter the solution into a screw-caped glass vial, in order to check the pH of the

Appendix D

(Normative)

Determination of hexavalent chromium content in leather materials -

Determination of recovery rate

D.1 Effect of substrate on recovery rate

D.1.1 Determination of recovery rate is important, which can provide information about whether the potential substrate effect affects the test results.

Note:

1. If the hexavalent chromium added is not detected, it may indicate that the leather may contain a reducing agent. In this case, if the recovery rate as obtained in accordance with D.2 is greater than 90%, it is concluded that the leather does not contain hexavalent chromium (below the detection limit).
2. Recovery rate is an indicator of whether the method is effective or whether the substrate effect affects the outcome. Usually the recovery rate is greater than 80%.

D.1.2 Determination of recovery rate

D.1.2.1 ADD the hexavalent chromium standard solution (4.2.7) of appropriate volume of to the 10 mL test solution as extracted in accordance with 7.5.3.5, so that the concentration of hexavalent chromium in the extract is approximately 2 times the original concentration ($\pm 25\%$). When selecting the concentration of the added solution, it shall ensure that the final volume after addition does not exceed 11 mL. USE the method same as sample preparation to treat this solution (absorbance indicated as A_{1s} and A_{2s}) (SEE 7.5.4).

D.1.2.2 The absorbance of the solution shall be within the range of the calibration curve. Otherwise, it shall reduce the transferred volume AND re-test it.

D.1.2.3 Recovery rate calculation

USE the equation (D.1) to calculate the recovery rate: