Designation: D 5517 – 03

Standard Test Method for Determining Extractability of Metals from Art Materials[1]

This standard is issued under the fixed designation D 5517; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the extraction of metals from art materials using an extractant that simulates the acid potential of gastric juice. This test method is similar to the extraction method noted in Specification F 963 but involves conducting extraction steps at body temperature instead of at room temperature. The extraction procedure specified in this test method is more rigorous than that noted in Specification F 963.

1.2 This test method is adapted from the European Toy Safety Standard, EN 71-3:1988.

1.3 This test method differs from EN 71-3:1988 in that a solvent extraction step is not required for processing waxes or oil-based products. The rationale for this test method is discussed in Appendix X1.

1.4 This test method does not specify any specific acceptable metal level.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 4236 Practice for Labeling Art Materials for Chronic Health Hazards[2]

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals[3]

E 456 Terminology Relating to Quality and Statistics[4]

F 963 Consumer Safety Specification on Toy Safety[5]

2.2 *International Standards:*[6]

EN 71-3:1988 Safety of Toys

ISO 3696 Water for Laboratory Use—Specifications

ISO 3856 Paints and Varnishes—Determination of "Soluble" Metal Content Part 1: Determination of Lead Content—Flame Atomic Absorption Spectrometric Method and Dithiazone Spectrophotometric Method

Part 2: Determination of Antimony Content—Flame Atomic Absorption Spectrophotometric Method and Rhodamine B Spectrophotometric Method

Part 3: Determination of Barium Content—Flame Atomic Emission Spectrometric Method

Part 4: Determination of Cadmium Content—Flame Atomic Absorption Spectrometric Method and Polarographic Method

Part 5: Determination of Hexavalent Chromium Content of the Pigment Portion of the Liquid Paint or the Paint in Powder Form—Diphenylcarbazide Spectrophotometric Method

Part 6: Determination of Total Chromium Content of the Liquid Portion of Paint—Flame Atomic Absorption Spectrometric Method

2.3 *USEPA Standards:*[7]

USEPA Test Method SW-846

6010 Test Method for antimony, arsenic, barium, beryllium, cadmium, chromium, cobalt, copper, lead, manganese, molybdenum, nickel, selenium, silver, thallium, vanadium, and zinc

6020 Test Method for aluminum, antimony, arsenic, barium, beryllium, cadmium, chromium, cobalt, copper, lead, manganese, nickel, silver, thallium, and zinc

7040 Test Method for antimony

7041 Test Method for antimony

7060 Test Method for arsenic

7061 Test Method for arsenic

7080 Test Method for barium

7090 Test Method for beryllium

7091 Test Method for beryllium

7130 Test Method for cadmium

7131 Test Method for cadmium

7190 Test Method for chromium

7191 Test Method for chromium

7200 Test Method for cobalt

7201 Test Method for cobalt

[1] This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.57 on Artist Paints and Related Materials.

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[2] *Annual Book of ASTM Standards*, Vol 06.02.

[3] *Annual Book of ASTM Standards*, Vol 15.05.

[4] *Annual Book of ASTM Standards*, Vol 14.02.

[5] *Annual Book of ASTM Standards*, Vol 15.07.

[6] Available from the Comite European de Normalisation, Central Secretariat, Rue Brederode 2, B–100 Brussels.

[7] Available from USEPA, Environmental Protection Agency, Alexander Drive, Research Triangle Park, NC 27709.

7210 Test Method for copper
7420 Test Method for lead
7421 Test Method for lead
7460 Test Method for manganese
7470 Test Method for mercury
7471 Test Method for mercury
7480 Test Method for molybdenum
7481 Test Method for molybdenum
7520 Test Method for nickel
7550 Test Method for osmium
7740 Test Method for selenium
7741 Test Method for selenium
7760 Test Method for silver
7840 Test Method for thallium
7841 Test Method for thallium
7870 Test Method for tin
7910 Test Method for vanadium
7911 Test Method for vanadium
7950 Test Method for zinc

3. Terminology

3.1 *Definitions:*

3.1.1 For formal definitions of statistical terms see Terminology E 456.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *bioavailability*, *n*—The extent that a substance can be absorbed in a biologically active form.

3.2.2 *detection limit*, *n*—Three times the standard deviation of the blank value.

3.2.3 *base material*, *n*—Material upon which coatings are deposited or formed.

3.2.4 *coating*, *n*—All layers of material covering the base material.

3.2.5 *scraping*, *v*—Removal of a coating down to the base material without removing any portion of the base material. The use of solvents is not permitted.

4. Summary of Test Method

4.1 A powdered, liquid, comminuted or ground art material is mixed with a 0.07 *N* hydrochloric acid solution and, after adjusting the pH to 1.5, is shaken for 1 h and then allowed to sit for an additional hour. These extraction steps are conducted at 37 ± 2°C. Solids are separated from the extractant by centrifugation and filtration through a 0.45-µm filter. The resultant eluant is then analyzed for the metal(s) of interest.

5. Significance and Use

5.1 This acid extraction method is intended to indicate the solubility of metals from art materials in a weak acid medium. This test method may be useful as one indicator of the amount of metal that is readily available for absorption[8]. It is not meant as a replacement for *in vivo* tests of the absorption of a metal.

5.2 Maximum levels of metal extraction are seen with this method when results are 250 ppm or less. If results are greater than 250 ppm, the extractant volume should be increased to 100 mL or greater, to keep metal levels in the eluant at a level of 5 ppm or less[9].

6. Apparatus

6.1 *Metal Sieve*, of aperture 0.5 mm.

6.2 *pH meter*, with an accuracy of ±0.1 pH units.

6.3 *Membrane Filter*, with a pore size of 0.45 µm.

6.4 *Centrifuge*, able to centrifuge at a minimum of 13 600 g.

6.5 *Precision Reciprocal Shaker*, 150 oscillation/min with 1 in. stroke length or *wrist-action shaker* capable of controlling the shaking amplitude to 4 ± 2 mm and the frequency to 9 ± 2 Hz.

6.6 *Constant Temperature Water Bath*, at 37 ± 2°C.

7. Reagents

7.1 *Hydrochloric Acid* (0.07 *N*)—Add 2.55 g concentrated hydrochloric acid (HCl) to Grade 3 purity water and dilute to 1 L with Grade 3 purity water.

7.2 *Hydrochloric Acid* (0.14 *N*)—Add 5.10 g concentrated hydrochloric acid (HCl) to Grade 3 purity water and dilute to 1 L with Grade 3 purity water.

7.3 *Hydrochloric Acid* (2.0 *N*)—Add 72.9 g concentrated hydrochloric acid (HCl) to Grade 3 purity water and dilute to 1 L with Grade 3 purity water.

7.4 *Hydrochloric Acid* (6.0 *N*)—Add 218.8 g concentrated hydrochloric acid (HCl) to Grade 3 purity water and dilute to 1 L with Grade 3 purity water.

7.5 *Water*, of at least Grade 3 purity in accordance with ISO 3696.

8. Preparation of Test Portions

8.1 A test portion is approximately 100 mg.

8.2 Art materials that are in the form of a liquid, dust or comminuted solid or are metals are tested without further preparation.

8.3 Scraped coatings of art materials are prepared by comminuting the sample sufficiently to pass through a 0.5-mm sieve.

8.4 Films, textiles, and paper are prepared by cutting into approximately 6 by 6-mm squares.

8.5 Solids are comminuted, ground or scraped to prepare a sample sufficient to pass through a 0.5-mm sieve.

9. Procedure

9.1 Mix the test portion with 50 times its mass of an aqueous solution of 0.07 *N* hydrochloric acid at 37 ± 2°C. In case there is less than a 100-mg test portion, mix the portion with 5.0 mL of this solution at the given temperature. Shake for 1 min.

9.2 Check the acidity of the mixture. If the pH is greater than 1.5, add drop wise with shaking an aqueous solution of 2 *N* hydrochloric acid until the pH is 1.5. Protect the mixture from light. Continuously, shake the mixture efficiently for 1 h and then allow the mixture to stand for 1 h at 37 ± 2°C.

[8] Supporting data are available from ASTM International Headquarters. Request Document RR D01-1120.

[9] Supporting data are available from ASTM International Headquarters. Request Document RR D01-1121.