



Standard Test Method for Determining Extractability of Metals from Art Materials¹

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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 Hazards²

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals³

E 456 Terminology Relating to Quality and Statistics⁴

F 963 Consumer Safety Specification on Toy Safety⁵

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

7210 Test Method for copper

7420 Test Method for lead

7421 Test Method for lead

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 Comité Européen de Normalisation, Central Secretariat, Rue Brederode 2, B-100 Brussels.

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

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*—The extent that a substance can be absorbed in a biologically active form.

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

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

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

3.2.5 *scraping*—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 absorption⁸. 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 less⁹.

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

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

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.

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.

NOTE 1—It has been shown that the extraction of soluble cadmium can show a 2 to 5-fold increase when extraction is carried out in the light rather than in the dark. Using brown flasks or aluminum foil-covered flasks will eliminate light effects.

9.3 If necessary, centrifuge the mixture and separate the solids from the mixture by filtration through a membrane filter with a pore size of 0.45 µm.

NOTE 2—Some fine pigments may break through a standard 0.45-µm filter. In these cases centrifugation of the eluate at 13 600 g for 15 min will remove remaining suspended pigment particles.

9.4 Paper products are prepared by first macerating the test portion with 25 times its mass of Grade 3 purity water at

37 ± 2°C so that the resulting mixture is uniform in color and texture. This mixture is then transferred quantitatively to a conical flask. Add 25 times the mass of the test portion of an aqueous solution of 0.14 N hydrochloric acid at 37 ± 2°C and shake for 1 min. Complete the extraction as in 9.2 and 9.3.

9.5 If a test portion contains large quantities of alkaline materials, generally in the form of calcium carbonate, adjust the pH to 1.5 with 6 N hydrochloric acid in order to avoid over dilution.

9.6 Determine metal(s) concentration in eluate with the appropriate analytical procedure (see Test Method ISO 3856 or USEPA Test Method SW-846).

10. Calculation

10.1 Determine the extractable metal level in the test sample in parts per million (ppm) (milligrams of metal per kilograms of test material) as follows:

Metal concentration in sample (ppm)

$$= \text{metal concentration in eluate (ppm)} \times 50. \quad (1)$$

11. Precision and Bias¹⁰

11.1 *Precision*—The precision estimates are based on an interlaboratory study in which one operator in 17 different laboratories analyzed identically prepared test materials in duplicate using this test method. Precision of the extraction was estimated by analyses of a comminuted paint sample and the acid extract of this sample. Repeatability and reproducibility standard deviations and 95 % limits are determined as specified in Practice E 180.

¹⁰ Supporting data are available from ASTM Headquarters. Request Document RR D01-1084.

TABLE 1 Comminuted Paint Sample

	Barium	Cadmium	Chromium
Mean value, mg/kg	101.0	22.7	27.2
Degrees of freedom, df	12.0	14.0	13.0
Repeatability, standard deviation	6.0	0.9	1.66
Repeatability, 95 % limits	±17.0	±2.5	±4.6
Reproducibility, standard deviation	21.0	4.9	3.74
Reproducibility, 95 % limits	±48.0	±14.0	±10.0

TABLE 2 Acid Extract of Comminuted Paint Sample

	Barium	Cadmium	Chromium
Mean value, mg/L	1.90	0.431	0.513
Degrees of freedom, df	12.0	14.0	13.0
Repeatability, standard deviation	0.06	0.01	0.029
Repeatability, 95 % limits	±0.17	±0.028	±0.081
Reproducibility, standard deviation	0.36	0.051	0.062
Reproducibility, 95 % limits	±1.0	±0.13	±0.17

11.1.1 *Repeatability*—The within-laboratory standard deviations and 95 % repeatability limits for testing three metals are reported in Table 1 and Table 2.

11.1.2 *Reproducibility*—The between-laboratory standard deviations and 95 % reproducibility limits for testing three metals are reported in Table 1 and Table 2.

11.1.3 *Bias*—No systematic errors were detected between measured and true values on either intralaboratory or between laboratory testing.

12. Keywords

12.1 art materials; bioavailability; bioequivalency; metals

APPENDIX

(Nonmandatory Information)

X1. RATIONALE

X1.1 Practice D 4236 requires that a toxicologist shall take into account the bioavailability of potentially toxic components of an art material in making a determination whether or not the

art material would require labeling for a chronic health hazard. This test method may provide an estimate of the bioavailability after ingestion of selected metals in art materials.

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