



Designation: D 1301 – 91 (Reapproved 1999)

Standard Test Methods for Chemical Analysis of White Lead Pigments¹

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This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 These test methods cover procedures for the chemical analysis of basic carbonate white lead and basic sulfate white lead.

NOTE 1—If it is necessary to separate these pigments from others, refer to Test Methods D 215.

1.2 The analytical procedures appear in the following order:

	Section
Preparation of Sample	6
<i>Basic Carbonate White Lead:</i>	
Small Amounts of Iron	7
Total Lead	8
Moisture and Other Volatile Matter	9
Carbon Dioxide (Evolution Method)	10
Carbon Dioxide and Combined Water (Combustion Method)	11
Lead Carbonate	12
Total Matter Insoluble in Acetic Acid	13
Total Matter Insoluble in Acid Ammonium Acetate	14
Total Impurities Other Than Moisture	15
Coarse Particles	16
<i>Basic Sulfate White Lead:</i>	
Small Amounts of Iron	17
Total Lead	18
Moisture and Other Volatile Matter	19
Total Sulfate	20
Zinc Oxide	21
Basic Lead Oxide	22
Total Impurities	23
Coarse Particles	24

1.3 *This standard does not purport to address 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:

C 25 Test Methods for Chemical Analysis of Limestone, Quicklime, and Hydrated Lime²

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.31 on Pigment Specifications.

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² *Annual Book of ASTM Standards*, Vol 04.01.

D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints³

D 215 Practice for Chemical Analysis of White Linseed Oil Paints³

D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments³

D 1193 Specification for Reagent Water⁴

D 2371 Test Method for Pigment Content of Solvent-Reducible Paints⁵

D 2372 Practice for Separation of Vehicle from Solvent-Reducible Paints⁵

D 3280 Test Methods for Analysis of White Zinc Pigments³

E 11 Specification for Wire-Cloth Sieves for Testing Purposes⁶

3. Significance and Use

3.1 These test methods are suitable for determining the level of purity and for determining the levels of various impurities. They may be used to establish compliance with specification requirements.

4. Reagents

4.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁷ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

4.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type II of Specification D 1193.

³ *Annual Book of ASTM Standards*, Vol 06.03.

⁴ *Annual Book of ASTM Standards*, Vol 11.01.

⁵ *Annual Book of ASTM Standards*, Vol 06.01.

⁶ *Annual Book of ASTM Standards*, Vol 14.02.

⁷ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopoeia and National Formulary*, U.S. Pharmacopoeial Convention, Inc. (USPC), Rockville, MD.

4.3 Concentration of Reagents:

4.3.1 Concentrated Acids and Ammonium Hydroxide—

When acids and ammonium hydroxide are specified by name or chemical formula only, it shall be understood that concentrated reagents of the following specific gravities or concentrations are intended:

Acetic acid, CH ₃ COOH	99.5 %
Hydrochloric acid, HCl	sp gr 1.19
Hydrofluoric acid, HF	48 %
Nitric acid, HNO ₃	sp gr 1.42
Sulfuric acid, H ₂ SO ₄	sp gr 1.84
Ammonium hydroxide, NH ₄ OH	sp gr 0.90

The desired specific gravities or concentrations of all other concentrated acids are stated whenever they are specified.

Warning—See Section 5.

4.3.2 Diluted Acids and Ammonium Hydroxide—

Concentrations of diluted acids and ammonium hydroxide, except when standardized, are specified as a ratio stating the number of volumes of concentrated reagent to be diluted with a given number of volumes of water, as in the following example: HCl (1 + 99) means 1 volume of concentrated HCl (sp gr 1.19) diluted with 99 volumes of water.

5. Hazards

5.1 The concentrated acids bases and other reagents used in these test methods can be dangerous. Check their Material Safety Data Sheets, (MSDS) before use.

6. Preparation of Sample

6.1 Grind dry pigments, if lumpy or not finely ground, to a fine powder for analysis. Large samples may be thoroughly mixed and a representative portion taken and powdered if lumpy or not finely ground. Mix the sample in all cases thoroughly and comminute before taking specimens for analysis.

6.2 In cases of pastes in oil, extract the oil from the pigment as described in Test Methods D 2371 or D 2372, but without straining.

6.3 Dry pigments separated from paints or pastes in oil in an oven at 203 to 210°F (95 to 98°C) for 2 h, grind to a fine powder, pass through a No. 80 (180-μm) sieve (Note 2) to remove skins, and mix thoroughly. Such pigments, after weighing, should be moistened with a little ethyl alcohol (95 %) before adding reagents for analysis.

NOTE 2—Detailed requirements for this sieve are given in Specification E 11.

6.4 Preserve all samples in stoppered bottles or containers.

BASIC CARBONATE WHITE LEAD

7. Small Amounts of Iron

7.1 Reagents:

7.1.1 *Ammonium Hydroxide* (sp gr 0.90). **Warning**—See 5.1.

7.1.2 *Hydrofluoric Acid* (48 %). **Warning**—See 5.1.

7.1.3 *Nitric Acid* (sp gr 1.42). **Warning**—See 5.1.

7.1.4 *Sulfuric Acid* (sp gr 1.84). **Warning**—See 5.1.

7.2 Procedure:

7.2.1 Weigh to 10 mg about 1 g of specimen into a 400-mL beaker. Treat the sample with 10 mL of HNO₃ (1 + 1) and

dilute to about 200 mL with water. If insoluble matter remains following treatment with HNO₃ and dilution, filter and wash the residue with hot water until lead free. Evaporate the filtrate and washings to about 200 mL. Add 20 mL of H₂SO₄ (1 + 1) to precipitate the bulk of the lead (it is unnecessary to evaporate down). Cool, filter, and wash with diluted H₂SO₄ (1 + 99). Save the precipitate for determination of total lead (Section 8).

7.2.2 Ignite the HNO₃-insoluble matter and treat with HF and H₂SO₄. Bring into solution, filter (any precipitate is probably BaSO₄), and add to the PbSO₄ filtrate.

7.2.3 Colorimetrically determine iron in the combined filtrates by the thiocyanate method,⁸ using the same amounts of reagents in preparing the reference standards. If copper is present in the filtrate, as shown by the characteristic blue-green or yellow color, remove it by precipitating the iron with NH₄OH, filtering, washing, redissolving the Fe(OH)₃ in 10 mL of HNO₃(1 + 1), and diluting to about 200 mL before proceeding with the thiocyanate method.

8. Total Lead

8.1 Apparatus:

8.1.1 *Gooch Crucible*, prepared prior to use.

8.2 Reagents:

8.2.1 *Acetic Acid* (glacial)—**Warning**—See 5.1.

8.2.2 *Ammonium Hydroxide* (sp gr 0.90)—**Warning**—See 5.1.

8.2.3 *Ethyl Alcohol* (95 volume %)—**Warning**—See 5.1.

8.2.4 *Potassium Dichromate Solution* (100 g K₂Cr₂O₇/L)—**Warning**—See 5.1.

8.3 Procedure:

8.3.1 Ignite the PbSO₄ precipitate and filter paper from 7.2.1 at or below 1020°F (550°C), and transfer the residue to a 400-mL beaker. (If preferred, a new 1-g specimen of pigment may be weighed to 10 mg into a 400-mL beaker. Proceed to 8.3.2.)

8.3.2 Moisten with water and add 5 mL of glacial acetic acid. Warm to dissolve the material and dilute to about 200 mL with water. Neutralize the solution with NH₄OH and then make slightly acid with acetic acid, adding about 3 mL excess. Filter off any insoluble residue and wash thoroughly with hot water.

8.3.3 Unite the filtrate and washings, heat to boiling, and add 15 mL of K₂Cr₂O₇ solution. Stir and heat until the yellow precipitate assumes an orange color. Let settle and filter on a weighed Gooch crucible. Wash by decantation with hot water until the washings are colorless. Finally transfer all the precipitate from the beaker to the crucible and wash with ethyl alcohol (95 %). Dry at 220 ± 4°F (105 ± 2°C) for 1 h. Cool in a desiccator and weigh as PbCrO₄.

8.4 Calculation:

$$\text{PbO, \%} = (P \times 0.691 / S) \times 100$$

where:

P = PbCrO₄ precipitate, g,

S = specimen, g, and

⁸ Described in Scott, *Standard Methods of Chemical Analysis*, Fifth Edition, D. Van Nostrand Co., New York, NY, 1939, p. 486.