Designation: D521 – 02 (Reapproved 2012)

Standard Test Methods for Chemical Analysis of Zinc Dust (Metallic Zinc Powder)[1]

This standard is issued under the fixed designation D521; 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 These test methods cover procedures for the chemical analysis of metallic zinc powder in the form commercially known as zinc dust for use as a pigment in paints.

1.2 The analytical procedures appear in the following order:

	Sections
Moisture and Other Volatile Matter	7
Coarse Particles	8
Matter Soluble in Hexane	9 and 10
Total Zinc	11 and 12
Metallic Zinc	13 and 14
Zinc Oxide	15
Calcium	16 and 17
Lead	18
Iron	19
Cadmium	20
Chlorine	21 and 22
Sulfur	23 and 24

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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:*[2]

B214 Test Method for Sieve Analysis of Metal Powders

D185 Test Methods for Coarse Particles in Pigments

D280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments

D1193 Specification for Reagent Water

D6580 Test Method for The Determination of Metallic Zinc Content in Both Zinc Dust Pigment and in Cured Films of Zinc-Rich Coatings

E40 Method for Chemical Analysis of Slab Zinc (Spelter) (Withdrawn 1993)[3]

E68 Method for Polarographic Determination of Lead and Cadmium in Zinc (Withdrawn 1980)[3]

3. Significance and Use

3.1 These test methods compile procedures which can be used to check the composition of purity of metallic zinc powder. This information is useful to both the formulator and users.

4. Treatment of Sample

4.1 Store the laboratory sample in a tightly stoppered bottle to protect it from oxidation. Mix the whole sample thoroughly before taking portions for analysis.

5. Purity of Reagents

5.1 *Purity of Reagents*—Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[4] 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.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type II of Specification D1193.

6. Precision

6.1 Precision statements have not been established.

MOISTURE AND OTHER VOLATILE MATTER

7. Procedure

7.1 Determine moisture and other volatile matter in accordance with Method A of Test Methods D280, except heat the sample for only 1 h.

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[1] These test methods are under the jurisdiction of ASTM Committee D01 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] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

[4] *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 Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

COARSE PARTICLES

8. Procedure

8.1 Determine the percent of coarse particles in the pigment in accordance with Test Methods D185 or B214.

MATTER SOLUBLE IN HEXANE

9. Reagent

9.1 *Hexane*—Pure hexane or commercial hexane or petroleum ether of boiling point not higher than 75°C. Redistill before using.

10. Procedure

10.1 Place 100 g of the pigment in an extraction thimble in a Soxhlet extraction apparatus. Record the tare weight of the receiving flask. Charge the flask with a suitable volume of hexane and extract the sample for 4 h, subjecting the specimen to not less than 20 extractions in this time. Make a blank determination at the same time.

10.2 Remove the receiving flask, evaporate or distill off the hexane on a steam bath, and dry the flask at 105 ± 2°C for 1 h. Cool and weigh.

10.3 Calculate the percent of matter soluble in hexane, allowing for any material found in the blank.

TOTAL ZINC

11. Reagents

11.1 *Potassium Ferrocyanide, Standard Solution*—Dissolve 22 g of potassium ferrocyanide ($K_4Fe(CN)_6 \cdot 3H_2O$) in water and dilute to 1 L. To standardize, transfer 0.2 g of metallic zinc or freshly ignited zinc oxide (ZnO) to a 400-mL beaker. Dissolve in 10 mL of hydrochloric acid (HCl), sp gr 1.19 and 20 mL of water. Drop in a small piece of litmus paper, add ammonium hydroxide (NH_4OH) until slightly alkaline, then add HCl until just acid, and then 3 mL more of HCl. Dilute to about 250 mL with hot water and heat nearly to boiling. Run in the $K_4Fe(CN)_6$ solution slowly from a buret, while stirring constantly, until a drop tested on a white porcelain plate with a drop of the uranyl indicator solution shows a brown tinge after standing 1 min. Do not allow the temperature of the solution to fall below 70°C during the titration. Run a blank using the same amounts of reagents and water as in the standardization. The standardization must be made under the same conditions of temperature, volume, and acidity as obtained when the sample is titrated. Calculate the strength of the $K_4Fe(CN)_6$ solution in terms of grams of zinc as follows:

$$Z = W/(V_1 - B) \tag{1}$$

where:

Z = zinc equivalent of the $K_4Fe(CN)_6$ solution, g/mL,
W = zinc used (or equivalent to the ZnO used), g,
V_1 = $K_4Fe(CN)_6$ solution required for titration of the standard, mL, and
B = $K_4Fe(CN)_6$ solution required for titration of the blank, mL.

11.2 *Uranyl Nitrate Indicator Solution*—Dissolve 5 g of uranyl nitrate ($UO_2(NO_3)_2 \cdot 6H_2O$) in 100 mL of water.

12. Procedure

12.1 Transfer 0.25 g of the sample to a 400-mL beaker, moisten with alcohol, and dissolve in 10 mL of HCl (sp gr 1.19) and 20 mL of water.

12.2 Continue with the procedure used in standardizing the $K_4Fe(CN)_6$ solution as described in 10.1, beginning with the addition of the litmus paper and the adjustment of the acidity with NH_4OH and HCl.

12.3 *Calculation*—Calculate the percent of total zinc, T, as follows:

$$T = [(V_2 - B_2)Z/S_1] \times 100 \tag{2}$$

where:

V_2 = $K_4Fe(CN)_6$ solution required for titration of the specimen, mL,
B_2 = $K_4Fe(CN)_6$ solution required for titration of the blank, mL,
Z = zinc equivalent of the $K_4Fe(CN)_6$ solution, g/mL, and
S_1 = sample used, g.

METALLIC ZINC

NOTE 1—Sections 12 and 13 cover a rapid method for determining metallic zinc, intended for routine analysis. The results are inclined to be somewhat low, and for highest accuracy and particularly for referee tests, the hydrogen evolution method should be used.[5]

NOTE 2—Metallic zinc may also be determined in accordance with Test Method D6580.

13. Reagents

13.1 *Ferric Chloride Solution*—Prepare a solution containing 20 g of ferric chloride ($FeCl_3 \cdot 6H_2O$) and 20 mL of 20 % sodium acetate ($NaC_2H_3O_2$) solution per 100 mL. It is advisable to make up only one day's supply at a time (50 mL are required for each determination).

13.2 *Potassium Permanganate, Standard Solution* (0.1 *N*, 1 mL = 0.008 g TiO_2)—Dissolve 3.16 g of $KMnO_4$ in water and dilute to 1 L. Let stand 8 to 14 days, siphon off the clear solution (or filter through a medium porosity fritted disk), and standardize against the National Institute of Standards and Technology (NIST) standard chemical No. 40 of sodium oxalate ($Na_2C_2O_4$) as follows: In a 400-mL beaker dissolve 0.2500 to 0.3000 g of the NIST sodium oxalate in 250 mL of hot water (80 to 90°C) and add 15 mL of H_2SO_4 (1+1). Titrate at once with the $KMnO_4$ solution, *stirring the liquid vigorously and continuously.* The $KMnO_4$ must not be added more rapidly than 10 to 15 mL/min, and the last 0.5 to 1 mL must be added dropwise with particular care to allow each drop to be fully decolorized before the next is introduced. The solution shall not be below 60°C by the time the end point has been reached. (More rapid cooling may be prevented by allowing the beaker to stand on a small hot plate during the titration. The use of a small nonmercury type thermometer as a stirring rod is most

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[5] Wilson, L. A., "The Evaluation of Zinc Dust: A Proposed Method of Analysis," *Proceedings*, ASTEA, Am. Soc. Testing and Mats., Vol 18, Part II, 1918, p. 220.

convenient.) Keep the $KMnO_4$ solution in a glass-stoppered bottle painted black to keep out light, or in a brown glass bottle stored in a dark place.

13.3 *Sodium Acetate Solution (200 g/L)*—Dissolve 200 g of sodium acetate ($NaC_2H_3O_2$) or 332 g of $NaC_2H_3O_2{\cdot}3H_2O$, in water and dilute to 1 L.

13.4 *Zimmerman-Reinhardt Solution*—Prepare a solution containing 67 g of manganese sulfate ($MnSO_4{\cdot}4H_2O$) 130 mL of H_2SO_4 (sp gr 1.84), and 138 mL of phosphoric acid (H_3PO_4) (85 %) per L.

14. Procedure

14.1 Weigh 0.2 g of the sample, transfer immediately to a dry, 600-mL heavy-wall Erlenmeyer flask, and add 50 mL of the $FeCl_3$ solution. Tightly stopper the flask and agitate constantly for approximately 15 min. As soon as the zinc dust is all dissolved, add 50 mL of the Zimmerman-Reinhardt solution and 250 mL of water. Titrate with 0.1 *N* $KMnO_4$ solution.

NOTE 3—If preferred, the reduced iron may be titrated with 0.1 *N* potassium dichromate ($K_2Cr_2O_7$) solution, using sodium diphenylamine sulfonate in the presence of phosphoric acid (H_3PO_4) as an internal indicator.[6]

14.2 *Blank*—Make a blank determination, following the same procedure and using the same amounts of all reagents prior to the titration. (The blank is usually 0.1 to 0.2 mL of 0.1 *N* $KMnO_4$ solution.)

14.3 *Calculation*—Calculate the percent of metallic zinc, *M*, as follows:

$$M = \left[(V_3 - B_3)(N \times 0.0327)/S_2\right] \times 100 \quad (3)$$

where:

V_3 = $KMnO_4$ solution required for titration of the specimen, mL,
B_3 = $KMnO_4$ solution required for titration of the blank, mL,
N = normality of the $KMnO_4$ solution,
S_2 = sample used, g, and
0.327 = milliequivalent weight of Zn.

ZINC OXIDE

15. Calculation

15.1 Calculate the percent of zinc oxide (ZnO), *Z*, as follows:

$$Z = (A - C) \times 1.2447 \quad (4)$$

where:

A = total zinc, % (Section 10), and
C = metallic zinc, % (Section 12).

CALCIUM

16. Reagents

16.1 *Ammonium Oxalate, Saturated Solution*—Mix 50 g of ammonium oxalate and 1 L of water.

16.2 *Ammonium Oxalate Solution (10 g/L)*—Dissolve 10 g of ammonium oxalate in water and dilute to 1 L.

16.3 *Potassium Permanganate, Standard Solution* (0.1 *N*)—See 13.2.

17. Procedure

17.1 Transfer 10 g of the sample, weighed to 0.1 g, to a 400-mL beaker. Dissolve in 30 mL of HCl (1 + 1), keeping the beaker covered. Dilute to 200 mL. The solution at this point should be clear and transparent. Neutralize with NH_4OH. (Some zinc, because of its high concentration, will precipitate out as hydroxide at this point.) Add NH_4OH (sp gr 0.90) dropwise, stirring until the solution becomes clear. Heat nearly to boiling and add 75 mL of saturated ammonium oxalate solution. Boil until the precipitate assumes a dense crystalline appearance. Allow to settle on a steam bath for 20 min and then cool. Filter on close-grained paper and wash five times with a cold ammonium oxalate solution (10 g/L).

17.2 Carefully dissolve the precipitate from the filter paper, using alternately hot HCl (1+3) and hot water. Catch in a clean 400-mL beaker, dilute (if necessary) to a volume of about 250 mL, and add 25 mL of saturated ammonium oxalate solution. Bring to a boil, and add NH_4OH in slight excess. Boil until the precipitate becomes crystalline and dense. Let stand 1 h on a steam bath. Allow to cool. Filter and wash with *small* amounts of hot water until all ammonium oxalate is removed.

17.3 Remove the paper from the funnel and spread it out on the inside of a 600-mL beaker above 300 mL of warm water to which has been added 20 mL of H_2SO_4 (1+1). Rinse the precipitate off the paper with water from a wash bottle and warm the solution to 80°C. Titrate at this temperature with $KMnO_4$ solution. As soon as a persistent pink end point is obtained, drop the paper into the liquid, rinse the side of the beaker, and quickly complete the titration.

17.4 *Calculation*—Calculate the percent of calcium, *C*, as CaO, as follows:

$$C = \left[(V_4 N \times 0.02804)/S_3\right] \times 100 \quad (5)$$

where:

V_4 = $KMnO_4$ solution required for titration of the sample, mL,
N = normality of the $KMnO_4$ solution, and
S_3 = specimen used, g.

LEAD

18. Procedure

18.1 Determine the lead content in accordance with Section 6 of Test Method E40.

NOTE 4—Lead may also be determined in accordance with Test Method E68.

IRON

19. Procedure

19.1 Determine the iron content in accordance with Section 22 of Test Method E40.

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[6] Kolthoff, I. M., and Sandell, E. B., *Textbook of Quantitative Inorganic Analysis*, 1945, p. 608.

CADMIUM

20. Procedure

20.1 Determine the cadmium content in accordance with 18.1 and 18.3 of Test Method E40.

NOTE 5—Cadmium may also be determined in accordance with Test Method E68.

CHLORINE

21. Reagents

21.1 *Silver Nitrate Solution (3.5 g/L)*—Dissolve 3.5 g of silver nitrate ($AgNO_3$) in water and dilute to 1 L.

21.2 *Sodium Chloride, Standard Solution* (0.01 *N*)—Dissolve 0.5850 g of pure sodium chloride (NaCl) in water and dilute to 1 L.

22. Procedure

22.1 Transfer 1.000 g of the sample to a 200-mL electrolytic beaker. Add 20 mL of water and then cautiously add 5 mL of nitric acid (HNO_3) (sp gr 1.42). Cover with a watch glass and heat on a steam bath with frequent stirring for 5 min, or until a clear solution results. Add 70 mL of water and cool to room temperature.

22.2 To the specimen and to a blank (prepared in similar fashion) add 5.0 mL of $AgNO_3$ solution (3.5 g/L) and stir. To the blank, add dropwise from a 10-mL buret, with thorough mixing, enough NaCl solution to develop a turbidity matching that of the sample. Keep the contents of both beakers well stirred and view against a black background in equal illumination.

22.3 *Calculation*—Calculate the percent of chlorine, *C*, as follows:

$$C = 0.035\,V_5 \tag{6}$$

where V_5 = 0.01 *N* NaCl solution added to the blank, mL.

SULFUR

23. Reagents

23.1 *Barium Chloride Solution (100 g/L)*—Dissolve 117 g of barium chloride ($BaCl_2 \cdot 2H_2O$) in water and dilute to 1 L.

23.2 *Bromine Water (saturated).*

24. Procedure

24.1 Transfer 20 g of the specimen, weighed to the nearest 0.1 g, to a 600-mL beaker. Cover the sample with 50 mL of saturated bromine water and then cautiously add HNO_3 (sp gr 1.42) until solution is complete.

24.2 Add 1 g of anhydrous sodium carbonate (Na_2CO_3) and boil down until salts just begin to separate, then add 75 mL of HCl (sp gr 1.19) and again boil down until salts begin to separate. Repeat this operation and, finally, dilute to 100 mL, heat until solution is complete, and filter into a 400-mL beaker through close-texture paper.

24.3 Wash the paper with hot water, make the filtrate alkaline with NH_4OH, and then just acid with HCl. Heat to boiling and hold at boiling temperature at least 5 to 10 min to drive out CO_2 then slowly add with stirring 5 mL of $BaCl_2$ solution. Allow to stand at least 5 h (preferably overnight).

24.4 Filter on a weighed Gooch crucible, wash free from chlorides with hot water, dry, and ignite carefully at 900°C. Cool and weigh. The difference between the original and final weight is $BaSO_4$.

24.5 *Blank*—Make a blank determination, following the same procedure and using the same amounts of all reagents.

24.6 *Calculation*—Calculate the percent of sulfur, *S*, as follows:

$$S = [((W_1 - B_4) \times 0.1374)/S_4] \times 100 \tag{7}$$

where:

W_1 = $BaSO_4$, g,
B_4 = correction for blank, g,
S_4 = sample used, g, and
0.1374 = $S/BaSO_4$ = 32.06/233.42.

25. Keywords

25.1 calcium; chlorine; potassium ferrocyanide; zinc dust; zinc powder