Designation: D2448 – 85 (Reapproved 2012)

Standard Test Method for Water-Soluble Salts in Pigments by Measuring the Specific Resistance of the Leachate of the Pigment[1]

This standard is issued under the fixed designation D2448; 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 determination of the specific resistance of the aqueous leachate from a pigment as an index of water-soluble salt content. This test method is applicable to white pigments and colored pigments (organic and inorganic). The water-soluble salts content is a function of the specific resistance of the solution formed by extracting the pigment with water.

1.2 This test method is based on a water to pigment ratio of 9+1. The leachate yield (minimum 160 mL) sufficient for rinsing the cylinder dip cell and thermometer plus the minimum 80 mL required for the measurement to determine the quantity of pigment to be used.

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]

D1193 Specification for Reagent Water

3. Summary of Test Method

3.1 A weighed quantity of pigment is added to water and boiled. For hydrophobic pigments methyl alcohol is used to facilitate wetting. After filtration, the specific resistance of the filtrate is determined using a conductivity bridge.

4. Significance and Use

4.1 This test method provides a reliable means for the determination of the relative amounts of these salts when comparing different lots of the same pigment grade. Water-soluble salts also affect the water resistance and blister resistance of coatings, especially primers for steel. Conductivity measurements, however, cannot be used as the *only* method to determine and compare the amount of water soluble salts of pigments with the same chemical composition but produced by different manufacturing processes.

5. Apparatus

5.1 *Centrifuge* (optional), having a 500-mL capacity per cup.

5.2 *Filter Paper* (Note 1)—For high reliability avoid use of filter paper containing soluble material. Each filter should be washed with reagent water in the funnel until resistance of the filtrate is greater than 200 000 Ω · cm.

NOTE 1—The size of the filter paper will be determined by how voluminous the pigment is. Some organic pigments require at least a 185-mm paper for proper filtering.

5.3 *Filter Aid* (optional)—The use of a filter aid may be desirable with some pigments to improve filtration. However, the filter aid must be treated to meet the same specifications for filter paper as given in 5.2.

5.4 *Ungraduated Cylinders,* approximately 35 mm wide by 125 mm deep.

5.5 *Thermometer,* graduated in 0.2°C intervals.

5.6 *Conductivity Bridge.*[3]

5.7 *Conductivity Cell,*[3] having a cell constant, *K*, of 1. The cell constant recommended for various ranges of electrolytes is as follows:

5.7.1 For specific resistances of less than 250 Ω · cm, use a cell with a constant of 10 or more.

5.7.2 For specific resistances from 250 to 200 000 Ω · cm, use a cell with a constant of 1. This covers the range for most pigments.

[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 D01.31 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] Any commercially produced conductivity bridge and conductivity cell is satisfactory.

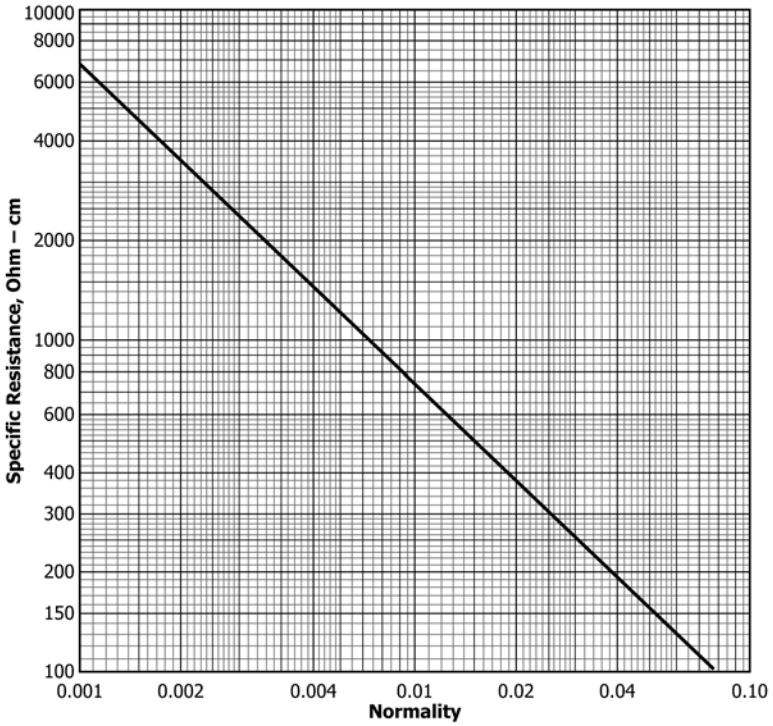

FIG. 1 Specific Resistance of Potassium Chloride at 25°C

5.7.3 For specific resistances of reagent water or of over 200 000 Ω · cm, use a cell with a constant of 0.1.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to 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.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent grade water as defined by Type II of Specification D1193.

6.3 *Methanol.*

6.4 *Potassium Chloride, Standard Solution* (0.02 *N*)— Prepare a 0.02 *N* potassium chloride (KCl) solution with water by dissolving 1.491 g of KCl (heated before weighing for 1 h at 105°C) in water, followed by dilution to 1 L.

7. Determination of Cell Constant

7.1 Prepare a 0.002 *N* solution of KCl by diluting the 0.02 *N* KCl solution described in 6.4 with water. Cool and measure the specific resistance, R_m, at 25 ±0.5°C as described in 8.2.2.

7.2 Calculate the cell constant, *K*, as follows:

$$K_{25} = (R_m/R_s)$$

where:

R_m = specific resistance at 25°C (see 8.2.2), and
R_s = specific resistance in ohm centimetres of an 0.002 *N* KCl solution at 25°C (Table 1) = 3427 Ω · cm.

NOTE 2—In general the "cell constant" is not greatly affected by variations in the strength of the KCl solution, but for greatest accuracy, measurements should be made at or near the specific resistance of the solution to be measured and at values that utilize the medium range of the scale of the conductivity bridge, using the same multiplier tap.

7.3 The specific resistances of KCl solutions are shown for concentrations from 0.001 *N* to 0.073 *N* at 25 C in Fig. 1. This curve, almost a straight line, was made from published values of specific conductances and equivalent conductances of KCl solutions at 25 C. Table 1 gives values of the specific resistances of KCl solutions for those concentrations useful for pigment testing.

8. Procedure

8.1 *Hydrophobicity Test*—Test a small amount of pigment with boiling water to see if it is water-wettable. Pigments that do not wet well with water are probably hydrophobic and should be treated as described in 8.3. If the pigment wets easily, proceed as described in 8.2.

8.2 *Hydrophilic Pigments:*

8.2.1 A 20.0-g specimen weight is usually sufficient for pigments easily wet with water. Add 20.0 g of the pigment to 180 g of boiling water in a tared, 400-mL beaker with stirring rod. (Usually a 250-mL beaker is sufficient for white pigments. Some white pigment, because of tendency to foam and crawl,

[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.

TABLE 1 Specific Resistances of Solutions of Potassium Chloride at 25°C

Normality	Ω · cm
0.001	6802 **(1)**
0.002	3427 **(1)**
0.005	1393 **(1)**
0.01	707.3 **(1,2)**
0.02	361.5 **(3)**

can be handled better in a larger beaker. Usually 20-g specimens of organic pigments require a 600-mL beaker to allow adequate room for foaming. Boil slowly for 5 min with occasional stirring. Cool to about 60°C and add water to bring the net weight back to 200 g. Stir thoroughly. Filter directly through fine-texture paper (Note 1) or separate the solids by centrifuge using clean, dry cups, or cups washed with some of the slurry, followed by decanting the supernatant liquid through a filter. In either case discard the first 10 mL through the filter.

8.2.2 Cool the filtrate to about 20°C. Rinse the footed cylinder and the conductivity cell, previously rinsed with water, with the leachate. Fill the footed cylinder with the leachate to be measured and place the conductivity cell into the leachate. Move the dip cell up and down to remove all air bubbles. Adjust the temperature slowly to 25°C and, with the cell submerged so that the vent is ½ in. (12.7 mm) below the surface of the liquid and centered upright in the cylinder, make at least five measurements of the specific resistance at 25 ± 0.5°C, using the conductivity bridge with the multiplier set to give a reading near the center of the scale, following the instructions supplied with the instrument to obtain a balance.

8.3 *Hydrophobic Pigments*—A modification of the procedure given in 8.2 is necessary for organic pigments that are not easily wet with water. Wet 30.0 g of pigment with 5 to 20 g of alcohol, as required, to produce a smooth wet paste. Complete the addition of water to bring the net weight to 300 g by diluting with boiling water in a tared beaker (1000 mL has been found satisfactory), with a stirring rod. Boil, cool, filter, and determine the reading as outlined in 8.2.2.

9. Calculation

9.1 Calculate the specific resistance, R_{25}, in ohm centimetres, of the pigment at 25°C by taking the mean R_a, of the five or more readings made and divide by the cell constant, K, determined in accordance with Section 7.

10. Precision and Bias

10.1 On the basis of a laboratory study of this test method, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

10.1.1 *Repeatability*—Two results obtained by the same operator should be considered suspect if they differ by more than 5 % for white pigments and 7 % for colored organic pigments.

10.1.2 *Reproducibility*—Two results obtained by operators in different laboratories should be considered suspect if they differ by more than 10 % for white pigments and 15 % for colored organic pigments.

10.2 *Bias*—Bias has not been determined for this test method.

11. Keywords

11.1 inorganic pigments; leachate; organic pigments; water soluble salts; white pigments