Designation: D 2352 – 85 (Reapproved 1999)

Standard Test Method for
Sulfur Dioxide in White Pigment Separated from Solvent-Reducible Paints[1]

This standard is issued under the fixed designation D 2352; 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 sulfur dioxide in white pigment separated from solvent-reducible paints.

1.2 This test method is not applicable in the presence of sulfides decomposable under the conditions given.

1.3 *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 1193 Specification for Reagent Water[2]

D 2371 Test Method for Pigment Content of Solvent-Reducible Paints[3]

3. Summary of Test Method

3.1 The extracted pigment is placed in a flask and then sulfur dioxide (SO_2) is released by addition of hydrochloric acid (HCl) and bubbled through an absorption flask containing iodine solution and potassium iodide (KI). The solution is titrated with sodium thiosulfate ($Na_2S_2O_3$) using starch indicator.

3.2 A blank is run as directed in 3.1 with the pigment omitted. The blank is then subtracted from the figure obtained for the sample titration and SO_2 is calculated.

4. Significance and Use

4.1 Calcium sulfate pigments such as titanium-calcium have been used in paints in varying degrees in the past years. As such it is useful to the formulator and the user to be able to monitor the amount of this compound in whole paints.

5. Reagents

5.1 *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.

5.2 Unless otherwise indicated, references to water shall be understood to mean Type II reagent grade water, conforming to Specification D 1193.

5.3 *Hydrochloric Acid* (1 + 3)—Mix 1 volume of concentrated hydrochloric acid (HCl, sp gr 1.19) with 3 volumes of water.

5.4 *Iodine, Standard Solution* (0.05 *N*) (for SO_2)—Place 15 to 20 g of pure potassium iodide (KI) in a 1-L flask, dissolve in as little water as possible, and then add about 6.4 g of resublimed iodine. Shake until the iodine is all dissolved, dilute to the mark with water, and mix. Standardize the solution against 0.05 *N* $Na_2S_2O_3$ solution to obtain its true normality.

5.5 *Potassium Iodide Solution* (100 g/L)—Dissolve 100 g of potassium iodide (KI) in water and dilute to 1 L.

5.6 *Sodium Thiosulfate, Standard* (0.05 *N*) *Solution* (for SO_2)—Dissolve pure sodium thiosulfate $Na_2S_2O_3$ in water (that has been well boiled to free it from carbon dioxide) in the proportion of 12.42 g of $Na_2S_2O_3 \cdot 5H_2O$ to 1 L of the solution. It is best to let this solution stand for about two weeks before standardizing. Standardize[5] with pure resublimed iodine, pure potassium biiodate, or pure potassium iodate. This solution will be approximately 0.05 *N*, and it is best to leave it as it is after determining its exact iodine value, rather than to attempt to

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[1] This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 06.01.

[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.

[5] Buehrer, T. F., and Mason, C. M., "Thiosulfate, Standardization of," *Analytical Chemistry,* Vol 2, 1930, p. 138.

adjust it to exactly 0.05 *N*. Preserve in a stock bottle provided with a guard tube filled with soda lime.

5.7 *Starch Indicator Solution*—Make a homogenous paste of 10 g of soluble starch in cold water. Add to this 1 L of boiling water, stir rapidly, and cool. Salicylic acid (1.25 g/L) may be added to preserve the indicator. If long storage is required, the solution should be kept in a refrigerator at 4 to 10°C (40 to 50°F). Prepare fresh indicator when the end point of the titration from blue to colorless or blue to light green fails to be sharp.

6. Preparation of Sample

6.1 Separate and prepare the pigment for this determination in accordance with Test Method D 2371.

7. Procedure

7.1 Transfer 10 g of the pigment to a suitable flask, insert a stopper fitted with a separatory funnel and a spray trap delivery tube (Note 1), and attach the latter to a condenser. Place about 150 mL of HCl (1 + 3) in the funnel, the stopcock being closed (Note 2), and connect the other end of the condenser with a delivery tube that passes through a two-hole stopper and extends nearly to the bottom of an absorption flask; through the other hole of the stopper connect a tube or flask to serve as a safety device. Place 25 mL of 0.05 *N* iodine solution (5.4) in the absorption flask (dilute with water if necessary) and 20 mL of KI solution (100 g/L) in the safety tube; fit the stopper in the absorption flask. Open the stopcock and allow the acid to slowly enter the flask. Before all of the acid is admitted, force air (washed with sodium hydroxide (NaOH) solution) through the top of the separatory funnel (about 2 bubbles per second in the KI solution). Boil the solution 3 min with air passing through then remove the source of heat and pass air through for 30 min.

NOTE 1—A Knorr CO_2 apparatus is very convenient. In this case, the vertical condenser may be connected with an absorption tower containing the iodine solution, followed by the KI solution in a suitable tube.

NOTE 2—To minimize, if not eliminate, any possible oxidation by the air, add about 1 g (in one piece) of sodium bicarbonate ($NaHCO_3$) to the evolution flask, then add the acid directly to the flask, omitting the separatory funnel and the current of air. Boil the solution until about 50 mL of distillate has passed over.

7.2 Disconnect the absorption vessels, wash the KI solution into the iodine solution, and titrate at once with 0.05 *N* $Na_2S_2O_3$ solution using starch indicator. Run a blank determination in exactly the same manner except for the omission of the pigment. Subtract the figure obtained for the blank determination from the figure obtained for titration of the sample and calculate the final result to SO_2 (1 mL 0.05 *N* iodine = 0.0016 g SO_2).

8. Calculation

8.1 Calculate the percent of sulfur dioxide, *A*, as follows:

$$A = [(V - B)I/S] \times 100$$

where:

V = 0.05 *N* iodine solution required for titration of specimen, mL,
B = 0.05 *N* iodine solution required for titration of the blank, mL,
I = SO_2 equivalent of the 0.05 *N* iodine solution, g/mL, and
S = sample used, g.

9. Precision

9.1 Data are not available to determine the precision of this test method. There are no plans at present to obtain such data. This test method has been in use for several years and is considered acceptable.

10. Keywords

10.1 sulfate analysis; sulfur dioxide; white pigment