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AMERICAN SOCIETY FOR TESTING AND MATERIALS
100 Barr Harbor Dr., West Conshohocken, PA 19428
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Standard Test Method for Arsenic in Paint[1]

This standard is issued under the fixed designation D 2348; 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 less than 0.5 % arsenic in whole paint.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of whoever uses this standard to consult and 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]

3. Summary of Test Method

3.1 The specimen of liquid paint is prepared for analysis by acid-digestion. The arsenic content of a distillate from the acid-digested sample is determined by titration with $KBrO_3$ solution.

4. Significance and Use

4.1 The permissible level of toxic elements in certain coatings is specified by governmental regulatory agencies. This test method provides a documented procedure for determining low concentrations of arsenic present in whole paint to determine compliance.

5. Apparatus

5.1 *Kjeldahl Flask*, 800-mL, with standard-taper 24/40 joint.

5.2 *Condenser Delivery Tube*, with standard-taper 24/40 joint.

5.3 *Mechanical Shaker*.

5.4 *Gas Burner*, suitable for heating Kjeldahl flask.

5.5 *Glass or Disposable Syringes*, 2 mL capacity.

5.6 *Erlenmeyer Flasks*, 250 and 500 mL capacity.

5.7 *Pipets*, 10 mL capacity.

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.[3] Other grades may be used, provided it is 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 conforming to Type II of Specification D 1193.

6.3 *Arsenous Oxide, Standard Solution*—Dissolve 0.3241 g of arsenous oxide (As_2O_3) in 25 mL of sodium hydroxide (NaOH) solution (100 g/L). Make slightly acid with sulfuric acid (H_2SO_4, 1 + 8), and dilute to 1.0 L with water.

6.4 *Hydrazine Sulfate-Potassium Bromide Solution*—Dissolve 20 g of hydrazine sulfate ($N_2H_4 \cdot H_2SO_4$) and 23 g of potassium bromide (KBr) in 200 mL of concentrated hydrochloric acid (HCl, sp gr 1.19) (**Precaution**—see 7.1) and dilute to 1 L.

6.5 *Methyl Orange Indicator Solution*—Dissolve 0.1 g in 100 mL water.

6.6 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3) (**Precaution**—see 7.1).

6.7 *Potassium Bromate, Standard Solution*—Dissolve 0.18 g of potassium bromate ($KBrO_3$) in water and dilute to 1 L with water. Standardize as follows: Transfer 10.0 mL of standard arsenous oxide solution by pipet to a 250-mL flask. Add 100 mL of water and 25 mL of HCl (sp gr 1.19). Heat to 90°C and titrate with $KBrO_3$ as described in 8.5. Calculate the standardization factor, *F*, as follows:

$$F = (0.0032410 \times 0.7574)/V \tag{1}$$

where:

0.0032410 = As_2O_3 in 10.0 mL of solution, g,
0.7574 = factor converting As_2O_3 to As, and
V = $KBrO_3$ required for the titration, mL.

6.8 *Sodium Chloride*(NaCl).

6.9 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid (H_2SO_4) (**Precaution**—see 7.1).

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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 Material.

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

[3] *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.

7. Hazards

7.1 Handling concentrated acids can be hazardous. Refer to their individual Material Safety Data Sheets (MSDS).

8. Procedure

8.1 Mix the sample until it is homogeneous, preferably on a mechanical shaker.

8.2 Weigh to 1 mg from a glass syringe, 1.0 g of whole paint directly into the 800-mL Kjeldahl flask, being careful not to get any on the sides of the flask. Add approximately 10 boiling aids.

8.3 Digest by adding the following: 40 mL of HNO_3 (allow to stand 20 min) and 25 mL of H_2SO_4 (allow to stand 5 min). Boil; when charring occurs, add HNO_3 cautiously until the solution becomes clear and begins to reflux within the flask. The flask should be at about a 45° angle while digesting. Cool, carefully add 150 mL of water, and boil to sulfur trioxide (SO_3) fumes.

8.4 Add 100 mL of water and a thermometer to an Erlenmeyer flask. After the solution in the Kjeldahl flask has cooled, add 25 mL of water, 25 g of NaCl, and 25 mL of the hydrazine sulfate-potassium bromide solution, and connect the distillation tube so that the delivery end is immersed in the receiving water. Quickly heat the Kjeldahl flask over a small, well-protected flame, and distill into the Erlenmeyer flask (Note 1). Adjust the flame so that the temperature of the distillate solution will rise to 80°C in 9 to 11 min, and then discontinue distillation by first removing the thermometer, lifting the distillation tube out of solution slightly and turning the heat off.

NOTE 1—Although it may be necessary to boil the solution, heating is primarily to bring about evolution of hydrogen chloride (HCl) gas, which carries over the arsenic trichloride ($AsCl_3$). Absorption of evolved HCl gas by water causes a rise in temperature which indicates progress of distillation. If distillation proceeds further, or if a larger quantity of H_2SO_4 than specified is used in digestion, sulfur dioxide (SO_2) will be distilled and titrated as arsenic.

8.5 Titrate the distillate at once with $KBrO_3$ solution, using 3 drops of methyl orange indicator solution. Toward the end of the titration add the $KBrO_3$ solution very slowly with constant agitation to prevent a local excess. The end point is reached when a single drop of the $KBrO_3$ just destroys the final tinge of red. Correct the results for the volume of $KBrO_3$ used in a blank determination (digest 5 g of pure sucrose, using the same reagents and procedure).

NOTE 2—The blank titration should not exceed 0.7 mL of $KBrO_3$ solution. The method is accurate down to variations in the blank, which should not exceed 0.1 mL when reagents from the same lot are used.

9. Calculation

9.1 Calculate the percent of arsenic in the sample as follows:

$$\text{Arsenic, \%} = [(V - B) \times F)/S] \times 100 \tag{2}$$

where:

V = $KBrO_3$ solution required for the sample, mL,
B = $KBrO_3$ solution required for the blank, mL,
S = sample used, g, and
F = factor as calculated in 6.7.

10. Precision and Bias

10.1 Precision and bias of this test method have not been determined.

11. Keywords

11.1 arsenic; paint