



Standard Test Method for Screening of Oxidizers in Waste¹

This standard is issued under the fixed designation D 4981; 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 is intended for use prior to preparation of waste samples for organic analysis. Waste samples that have oxidizing compounds may react with certain reagents in the laboratory (for example, organic solvents).

1.2 This test method is applicable to the analysis of waste liquids, sludges, and solids.

1.3 This test method can neither identify specific oxidizing compounds nor measure concentrations. Since no acid or base is added in this test method, potential oxidizers that require the presence of acid or base will not be detected by this test method.

1.4 It is recommended that, prior to this test, waste samples be screened for water compatibility; see Test Methods D 5058.

1.5 This test method is designed and intended as a preliminary test to complement quantitative analytical techniques that may be used to determine the presence of oxidizers in wastes. This test method offers the ability to screen waste for potentially hazardous reactions due to oxidizer content when the more sophisticated techniques are not available or the total waste composition is unknown.

1.6 *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.* Specific hazard statement is given in Section 8 and 11.2.

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

D 4980 Test Method for Screening of pH in Waste³

D 5058 Test Methods for Compatibility of Screening Analysis of Waste³

¹ This test method is under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.01.05 on Screening Methods.

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

³ *Annual Book of ASTM Standards*, Vol 11.04.

3. Terminology

3.1 Description of Term Specific to This Standard:

3.1.1 *screening analysis*—A preliminary qualitative or semiquantitative test that is designed to efficiently give the user specific information about a waste that will aid in determining waste identification, process compatibility, and safety in handling.

4. Summary of Test Method

4.1 A small portion of the sample is placed onto a strip of potassium iodide (KI) starch paper. The blue color which is the result of oxidizing the potassium iodide to iodine (I_2) in the presence of starch, indicates a positive test for oxidizers.

5. Significance and Use

5.1 This test method is intended for use by those in waste management industries to avoid potentially harmful reactions due to oxidizing compounds in wastes.

6. Interferences

6.1 Materials that mask the KI starch paper, (for example, oils, syrups, etc.) prevent reaction with the test paper or visual detection of a color change.

6.2 Samples or slurries of samples that are already dark colored prior to applying a test portion to the KI starch paper can give false results.

6.3 Oxidizers such as ferric salts may not oxidize organics; however, they may show positive reactions with KI.

6.4 Oxidizers that require the presence of acid or base will give a false negative result. To overcome this problem, run the oxidizer test at acid, base, and neutral pH conditions.

7. Reagents and Materials

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

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type III of Specification D 1193.

7.3 *Potassium Iodide (KI) Starch Paper Strips*.

7.4 *Beakers*.

7.5 *Pipet or Droppers*.

7.6 *Hydrogen Peroxide Solution (3 % H₂O₂)*, commercial grade.

7.7 *Nitric Acid (1 + 9)*—Add 1 volume of nitric acid (HNO₃, sp. gr 1.42) to 9 volumes of water.

8. Hazards

8.1 Avoid inhalation, skin or eye contact, or both, with all hazardous material.

8.2 This analysis must always be performed in a fume hood.

9. Sampling

9.1 Collect a representative sample of the waste in a container with a sealed lid.

9.2 The sample should be analyzed as soon as possible.

9.3 Allow the samples to stabilize to room temperature.

10. Procedure

10.1 *Aqueous Samples*—Using a clean pipet or dropper, place a drop sample on a strip of KI starch paper and note color change.

10.2 *Non-Aqueous Samples (for example, solid, oil, or solvents)*:

10.2.1 In a beaker, prepare a slurry by adding 1 to 5 g of sample to an equal amount of reagent water.

10.2.2 Using a clean pipet or dropper, place a drop of slurry on a strip of KI starch paper.

10.2.3 Note any color change.

10.3 All sample results shall be checked against the quality control and blank test strips to verify positive/negative readings (see Section 11).

10.4 All positive results (a blue color) shall be reported immediately to prevent potential hazardous reactions that may occur.

NOTE 1—Analyst should be aware that the blue color on the reference strip and any positive sample test strip will slowly fade with time.

11. Quality Control

11.1 Quality control check samples, reference blanks and duplicates should be performed at an action level specified by the laboratory and at an appropriate frequency.

11.1.1 Place a drop of reagent water on a KI starch test strip to serve as a reference blank.

11.1.2 Place a drop of 3 % H₂O₂ solution (or 10 % HNO₃) on a KI starch test strip to obtain a dark blue reference color.

11.2 (**Warning**—Fortifications (spikes) are not to be prepared due to possible adverse reactions.)

11.3 Method detection limits should be determined by each laboratory by using a test oxidizer (7.6 or 7.7).

12. Precision and Bias

12.1 *Precision and Bias*—No information is presented about either the precision or bias of Test Method D 4981 for measuring oxidizers since the test result is nonquantitative.

13. Keywords

13.1 oxidizers; screening analysis; starch-iodide paper

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 Pharmacopoeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

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