Designation: D 4980 – 89 (Reapproved 2003)

Standard Test Methods for Screening of pH in Waste[1]

1. Scope

1.1 These test methods are used to determine the pH of a hazardous waste liquid, sludge, semisolid and solid.

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1.2 Test Method A uses a wide-range pH paper for a rapid indication of pH to within about 1 pH unit.

1.3 Test Method B uses a pH meter to measure within about 0.1 pH unit.

1.4 These test methods are designed and intended as a preliminary test to complement the more sophisticated quantitative analytical techniques that may be used to determine pH. These test methods offer, to the user, the option and the ability to screen waste for potentially hazardous levels of acidity and alkalinity when the more sophisticated techniques are not available and the total waste composition is unknown.

1.5 *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 information is given in Section 6.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]
D 1293 Test Methods for pH of Water[2]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *screening analysis*—a preliminary qualitative or semi quantitative test developed from classical qualitative and quantitative techniques 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. Significance and Use

4.1 These test methods are intended for use by those in the waste management industries to characterize waste streams by pH. These methods will identify those waste materials that may dictate a specific waste management procedure due to high acidity or alkalinity.

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 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 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 as defined by Type III of ASTM Specification D 1193.

6. Hazards

6.1 Avoid inhalation and skin and eye contact of all hazardous materials.

6.2 All measurements shall be done in a laboratory fume hood.

7. Sampling

7.1 Collect a representative sample of the waste.

7.2 Samples should be analyzed as soon as possible after collection.

8. Report

8.1 The report shall include at a minimum:

8.1.1 Sample identification,

8.1.2 Date of test,

8.1.3 Reference to the procedure applied, that is, test method and if applicable, dilution ratio,

8.1.4 Analytical results, and

8.1.5 Identification of the analyst.

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[1] These test methods are 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] *Reagent Chemicals, American Chemical Society Specifications*, Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Reagent Chemicals and Standards*, by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the *United States Pharmacopeia.*

TEST METHOD A—pH SCREENING BY pH PAPER

9. Summary of Test Method A

9.1 *pH Paper*—A small portion of the sample is introduced onto full range pH paper. The results are visually compared to the appropriate color chart and reported to ±1 pH unit.

10. Interferences

10.1 Materials that mask the pH paper, for example, oils, syrups, paint, etc. cause the visual detection of color on the paper to be difficult.

10.2 Strong dyes and solutions of deep colors can give false results.

10.3 Oxidizers can bleach the pH paper.

11. Apparatus

11.1 *Stirring Rod, Spatula, and Disposable Pipet or Eye Dropper*, for transferring sample to test paper.

11.2 *Disposable Beaker, Test Tube*, etc.

11.3 *Vortex Mixer* (optional).

11.4 *Separatory Funnel*.

12. Reagents and Materials

12.1 Full range pH paper with a stated precision of 1 pH unit and with a corresponding color chart.

12.2 Standard Buffer Solutions.

13. Procedure

13.1 *Aqueous Liquids*—Introduce a representative portion of the sample onto a strip of pH paper.

13.2 *Solids, Sludges, and Non-Aqueous Liquids*:

13.2.1 Place approximately 10 mL of water in a disposable beaker or test tube.

13.2.2 Add approximately 1 mL of waste sample and agitate for 10 s or until well mixed.

13.2.3 Let suspension settle and measure the pH of the supernatant as described in 13.1.

13.3 Visually compare the pH paper to the color chart and record the pH to the nearest whole unit. See 8.1.4.

14. Quality Control

14.1 Standard buffer solutions are used to check each new lot or shipment of pH paper.

15. Precision and Bias

15.1 Precision and Bias statements shall be supplied at a later date.

METHOD B—pH SCREENING BY ELECTROMETRIC MEASUREMENT

16. Summary of Test Method B

16.1 *pH Meter*—The pH of a sample is determined electrometrically using a pH meter with a combination pH electrode. Prior to the analysis of a sample, the instrument must be calibrated by using at least two standard buffer solutions. The results should be reported to the nearest 0.1 pH unit.

17. Interferences

17.1 The glass electrode, in general, is not subject to solution interferences from color, turbidity, colloidal matter, oxidants, reductants, or high salinity.

17.2 Sodium error at pH levels >10 can be reduced or eliminated by using a low sodium error electrode.

17.3 Coatings of oily material or particulate matter can impair electrode response. These coatings can usually be removed by gentle wiping or detergent washing, followed by dilute water rinsing. An additional treatment with hydrochloric acid 1 + 9, (1 volume HCl added to 9 volumes of H_2O) may be necessary to remove any remaining film.

17.4 Temperature effects on the electrometric determination of pH arise from two sources. The first is caused by the change in electrode output at various temperatures. This interference can be controlled with instruments having temperature compensation or by calibrating the electrode instrument system at the temperature of the samples. The second source of temperature effects is the change of pH due to changes in the sample as the temperature changes. This error is sample dependent and cannot be controlled. It should, therefore, be noted by reporting both the pH and temperature at the time of analysis.

18. Apparatus

18.1 *pH Meter, Laboratory of Field Model*—A wide variety of instruments are commercially available with various specifications and optional equipment.

18.2 *Glass Electrode*.

18.3 *Reference Electrode*—A silver-silver chloride or other reference electrode of constant potential may be used.

NOTE 1—Combination electrodes incorporating both measuring and reference functions are convenient to use and are available with solid, gel type filling materials that require minimal maintenance.

18.4 *Magnetic Stirrer and TFE-Fluorocarbon-Coated Stirring Bar*.

18.5 *Thermometer or Temperature Sensor*, for automatic compensation.

19. Reagents and Materials

19.1 *Primary Standard Buffer Salts*, are available from NIST and should be used in situations where extreme accuracy is necessary. Preparation of reference solutions from these salts requires some special precautions and handling such as low conductivity dilution water, drying ovens, and carbon-dioxide-free purge gas. These solutions should be replaced at least once each month.

19.2 *Secondary Standard Buffers*, may be prepared from NIST salts or purchased as solutions from commercial vendors. These commercially available solutions have been validated by comparison to NIST standards and are recommended for routine use.

20. Calibration and Standardization

20.1 Each instrument/electrode system must be calibrated at two points that bracket the expected pH of the samples and are approximately three pH units or more apart. Various instrument designs may involve use of a balance or standardize dial or slope adjustment as outlined in the manufacturer's instructions.

Repeat adjustments on successive portions of the two buffer solutions until readings are within 0.05 pH units of the buffer solution values.

NOTE 2—Wide variation in the stirring speed may cause fluctuations in pH measurements, but the variations will not affect test accuracy.

20.2 Place the liquid sample or buffer solution in a glass beaker using a sufficient volume to cover the sensing elements or the electrodes and to give adequate clearance for the magnetic stirring bar. If field measurements are being made, the electrodes may be immersed directly in the sample stream to an adequate depth and moved in a manner to ensure sufficient sample movement across the electrode-sensing element as indicated by drift free (less than 0.1 pH) readings during the analysis.

20.3 If the sample temperature differs by more than 2°C from the buffer solution, the measured pH values must be corrected. Instruments are equipped with automatic or manual compensators that electronically adjust for temperature differences. Refer to manufacturer's instructions.

21. Procedure

21.1 Because of the wide variety of pH meters and accessories, detailed operation procedures cannot be incorporated into this test method. Each analyst must be acquainted with the operation of each system and familiar with all instrument functions. Special attention to care of the electrodes is recommended.

21.2 Thoroughly rinse and gently wipe the electrodes prior to measuring pH of each sample. Immerse the electrodes into the sample beaker or sample stream and gently stir at a constant rate to provide homogeneity and suspension of solids (see Note 3). Note and record sample pH and temperature. Repeat measurement on successive volumes of sample until values differ by less than 0.1 pH unit. Two or three volume changes are usually sufficient.

NOTE 3—Wide variation in stirring speed may cause fluctuations in pH measurements but the variations will not adversely affect test applicability.

21.3 *Aqueous Liquid*—Insert the electrode into the aqueous portion of the waste and report the instrument reading.

21.4 *Solids, sludges, and non-aqueous liquids*:

21.4.1 Mix approximately a 10 % slurry of waste in water and take measurement on the aqueous portion. (See 8.1.4.)

21.4.2 For pH determination, refer to 21.2.

22. Quality Control

22.1 Instrument performance standards (where applicable), quality control check samples of appropriate matricies, and duplications, should be performed at an action level specified by the laboratory at an appropriate frequency.

23. Precision and Bias

23.1 The data are being accumulated and will be added to the test method when completed.