



Designation: D 2110 – 9600

Standard Test Method for pH of Water Extractions of Halogenated Organic Solvents and Their Admixtures¹

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1. Scope

1.1 This test method covers the measurement of the pH of water extractions of halogenated organic solvents and admixtures thereof. This test method is applicable for the determination of the pH of water extractions of virgin, reclaimed, or used solvents.

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 the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Summary of Test Method

2.1 The solvent sample is shaken with freshly boiled neutral distilled or deionized water and the pH of the water extract determined by one of two procedures:

2.1.1 *Procedure A*, by comparing the color formed upon the addition of Gramercy Universal Indicator with that of a calibrated Gramercy pH Indicator Color Chart, or

2.1.2 *Procedure B*, using a glass electrode pH meter.

3. Significance and Use

3.1 The pH of halogenated solvents varies according to the nature of stabilizers in the particular solvent. Solvents with alkaline stabilizers (amine-types) generally have pHs in the 7 to 11 range, whereas solvents containing neutral stabilizers (epoxide-types) generally have pHs in the 5 to 7 range.

3.2 Virgin and reclaimed solvents should have pHs within these ranges, dependent upon the claimed method of stabilization. These values should be within the accepted limits as proposed by the producers of the original virgin solvent.

3.3 A lowering of pH from the above values, on receipt or use, is cause for concern. In this event, solvents having epoxide stabilizers should be tested for acid acceptance; solvent having alkaline stabilizers should be tested for alkalinity.

¹ This test method is under the jurisdiction of ASTM Committee D-26 on Halogenated Organic Solvents and Fire Extinguishing Agents and is the direct responsibility of Subcommittee D26.04 on Test Methods.

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4. Apparatus

- 4.1 ~~Gramercy Universal~~ Universal Indicator² and ~~Gramercy~~ pH Indicator Color Chart (Procedure A),² or
 4.2 ~~Glass Electrode~~ pH Meter (Procedure B).

5. Water

5.1 Neutral Distilled or Deionized Water shall be prepared as follows: Boil 1 L of distilled or deionized water for 5 min in a borosilicate glass or stainless steel container, then cover and cool to room temperature. ~~Titrate temperature.~~

5.2 Procedure A—To a 50-mL aliquot of the cooled, boiled water, add 2 mL of Universal Indicator. Titrate the aliquot to a pH of ~~7.0~~ 7 (as indicated by comparing the color of the aliquot to ~~7.3~~ the color chart per manufacturer's instructions) with either 0.01 N sodium hydroxide (NaOH) solution HCl if the pH is above 7 or 0.01 N hydrochloric acid (HCl), depending upon whether NaOH if the initial pH is above or below 7, and record the titer. From this titer, calculate and add the volume of 0.01 N NaOH solution or 0.01 N HCl required to adjust the pH of the remaining 950 mL of the boiled distilled or deionized water to a pH of ~~7.0 to 7.3~~. Add the calculated volume 7. The amount of 0.01 N NaOH solution or 0.01 N HCl together with needed to neutralize the 950 mL water will be 19 X titer. Determine the pH of a second aliquot. If further adjustment is necessary to obtain a pH of 7, repeat the above procedure.

5.3 Procedure B—Using a pH meter, titrate a 50-mL aliquot to a pH of ~~neutralized water~~ 7.0 to 7.3 with either 0.01 N HCl if the ~~950-mL stock~~ pH is above 7.0 or 0.01 N NaOH if the pH is 7.0 and record the titer. From ~~this~~ this titer, calculate and add the volume of 0.01 N NaOH or 0.01 N HCl required to adjust the remaining 950 mL of the boiled distilled or deionized water to a pH of 7.0 to 7.3. The amount of 0.01 N NaOH or 0.01 N HCl needed to neutralize the 950 mL water will be 19 X titer. Determine the pH of a second aliquot. If further adjustment is necessary to obtain a pH of 7.0 to 7.3, repeat the above procedure.

6. Procedure

6.1 Transfer 50 mL of the halogenated organic solvent to a 250-mL separatory funnel containing 50 mL of freshly boiled, neutral, distilled or freshly boiled, neutral, deionized water. Shake the mixture for 2 min; then let stand and allow the layers to separate. Drain and discard the halogenated solvent layer and transfer the water extract to a 100-mL beaker. Determine the pH of the water extract by either Procedure A or B.

6.2 Procedure A—Pipet 2 mL of ~~Gramercy~~ Universal Indicator into the water extract, stir, and compare the resultant color of a 10-mL aliquot in a 13-mm outside diameter test tube with those of to the Gramercy Color Chart, color chart per manufacturer's instructions. The pH (± 0.25 pH unit) of the water extract is that of the same as the pH of the closest matching color on the Gramercy Color Chart, color chart.

6.3 Procedure B—Determine the pH of the water extract to the nearest 0.1 pH unit with a ~~calibrated glass electrode~~ pH meter.

7. Precision and Bias³

7.1 The precision for Procedure A, Universal Indicator, is ± 0.5 pH unit; for Procedure B, pH meter, ± 0.2 pH unit; and for multilaboratory, ± 0.9 pH unit.

7.2 Bias has not been determined.

8. Keywords

8.1 acidity; alkalinity; halogenated organic solvents; pH; solvents

² Supporting data have been filed at ASTM headquarters and may be obtained by requesting Research Report RR D26-1007.

² Available from suppliers of reagent chemicals such as Fisher Scientific, Houston, TX or Aqua Solutions, Deer Park, TX.

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