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## SODIUM HYDROXIDE 25% (8N) TESTING METHODS

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**1. PURPOSE:**

- 1.1. To provide Laboratory personnel with a procedure for analyzing Sodium Hydroxide 25% In-Process, Stability, and Finished Good samples.

**2. SCOPE:**

- 2.1. Applies to the analysis of Sodium Hydroxide 25% In-Process, Stability, and Finished Goods in the Laboratory. Methods include testing for all grades of Sodium Hydroxide 25% sold by BioSpectra; only the specific tests required for the requested grade must be tested.

**3. RESPONSIBILITIES:**

- 3.1. The Laboratory Manager is responsible for training, maintenance, and implementation of this procedure.
- 3.2. Laboratory personnel are responsible for compliance with the terms of this procedure. This includes notifying the appropriate personnel if any analyses fail to meet their respective specifications.

**4. SAFETY:**

- 4.1. Causes SEVERE skin burns and eye damage. Standard laboratory safety regulations apply. Before working with any chemical, read and understand the Safety Data Sheet (SDS).

**5. REFERENCES:**

- 5.1. BSI-ATM-0074, Analytical Method of Analysis: Sodium Hydroxide via ICP-MS
- 5.2. BSI-ATM-0132, Analytical Method for Determination of Trace Metals in Sodium Hydroxide
- 5.3. BSI-FRM-1437, Sodium Hydroxide 25% (8N) Analytical Procedure
- 5.4. BSI-RPT-0442, Limit of Fe and Pb Verification Report via ICP-MS: Sodium Hydroxide
- 5.5. BSI-SOP-0019, Result Reporting
- 5.6. BSI-SOP-0098, Balance SOP
- 5.7. BSI-SOP-0126, Laboratory Notebooks
- 5.8. BSI-SOP-0135, Laboratory Chemicals
- 5.9. BSI-SOP-0140, Standardization of Titrants
- 5.10. BSI-SOP-0242, Portable Turbidimeter Operation and Calibration
- 5.11. BSI-SOP-0255, XL200 pH mV Conductivity Meter SOP
- 5.12. BSI-SOP-0303, NexION 350X ICP-MS SOP
- 5.13. BSI-SOP-0345, Endosafe Nexgen-PTS Endotoxin Reader SOP
- 5.14. ACS, Reagent Chemicals, current edition.
- 5.15. Current *USP-NF*

**6. EQUIPMENT:**

- 6.1. Analytical Balance
- 6.2. Calibrated Timer
- 6.3. Eppendorf Micropipettes
- 6.4. Nexgen-PTS Endotoxin Reader
- 6.5. NexION 350X ICP-MS
- 6.6. Portable Turbidimeter

## 7. REAGENTS:

- 7.1. **0.02N Hydrochloric Acid:** Dilute 20mL of 0.1N Hydrochloric acid to 100mL with purified water. May be purchased commercially.
- 7.2. **0.1N Silver Nitrate:** Purchased commercially.
- 7.3. **1N Sulfuric Acid:** Purchased commercially.
- 7.4. **1-0.01 EU/mL Endosafe PTS Cartridge:** Purchased commercially.
- 7.5. **Acetic Acid, 1N:** Dilute 57mL of Glacial Acetic Acid to 1L with purified water.
- 7.6. **Ammonium Hydroxide, 10%:** Dilute 35mL of 29% Ammonium Hydroxide to 100mL with purified water.
- 7.7. **Ammonium Peroxydisulfate:** Purchased commercially.
- 7.8. **Ammonium Thiocyanate, 30%:** Dissolve 150g of ammonium thiocyanate in purified water and dilute with purified water to 500mL.
- 7.9. **Buffer Solution:** Purchased commercially.
- 7.10. **Dilute Nitric Acid (1:99):** Dilute 1mL of concentrated Nitric Acid to 100mL with purified water.
- 7.11. **Glycerin Base:** To 200mg of glycerin, add purified water to a total weight of 235g. Add 140mL of 1N NaOH and 50mL purified water and mix.
- 7.12. **Hydrochloric Acid, concentrated:** Purchased commercially.
- 7.13. **Iron Standard (0.01mg of Fe in 1mL):** Dissolve 0.702g of ferrous ammonium sulfate hexahydrate in 10mL of 10% sulfuric acid reagent solution, and dilute with water to 100mL. Immediately before use to 10mL of this solution, add 10mL of 10% sulfuric acid reagent solution and dilute with purified water to 1L. Scale as needed.
- 7.14. **LAL Reagent Water:** Purchased commercially.
- 7.15. **Lead Stock Solution (0.1mg of Pb in 1mL):** Dissolve 0.160g of lead nitrate in 100mL of dilute nitric acid (1:99). Dilute with purified water to 1L. Scale as needed.
- 7.16. **Methyl Orange:** Dissolve 0.10g of methyl orange in 100mL of purified water. Filter if necessary.
- 7.17. **Nitric Acid, concentrated:** Purchased commercially.
- 7.18. **Phenolphthalein TS:** Dissolve 1.0g of phenolphthalein in 100mL of reagent grade alcohol.
- 7.19. **Potassium Carbonate (15%):** Weigh 15.000g of Potassium Carbonate and transfer to a 100mL volumetric flask. Dissolve and dilute to volume with purified water.
- 7.20. **Potassium Pyroantimonate TS:** Purchased commercially.
- 7.21. **Purified Water:** In-House.
- 7.22. **Sulfuric Acid Reagent Solution, 10%:** Slowly add 30mL of 96% sulfuric acid to 375mL of purified water. Cool and dilute with water to 500mL.
- 7.23. **Thioacetamide:** Dissolve 4g of thioacetamide in purified water to make 100mL.

**8. ANALYTICAL PROCEDURES:****8.1. IN-PROCESS TESTING:****8.1.1. ASSAY**

- 8.1.1.1. Perform a manual standardization or titrant check of 1 N Sulfuric Acid per Standardization of Titrants.
- 8.1.1.2. Accurately weigh 6g of sample and add 40 mL of purified water in a clean flask. Stopper the flask and allow to cool to room temperature.
- 8.1.1.3. Add 150 $\mu$ L of Phenolphthalein as the indicator and titrate using previously standardized 1N Sulfuric Acid to a colorless endpoint (V1).
- 8.1.1.4. Add 150 $\mu$ L of Methyl Orange as the indicator.
- 8.1.1.5. Titrate using previously standardized 1N Sulfuric Acid to a pink endpoint (V2).
- 8.1.1.6. Calculate the percentage of Sodium Hydroxide using the following equation:

$$\%NaOH = \frac{(V_2) \times N \text{ } H_2SO_4 \times 4.00}{\text{Sample Weight (g)}}$$

**8.2. FINISHED GOOD TESTING:****8.2.1. APPEARANCE AND COLOR**

- 8.2.1.1. Transfer 50 mL of the sample into a 50 mL a Nessler tube.
- 8.2.1.2. In order to pass, test solution is complete, clear, and colorless. Verify the solution appearance against a clear and colorless reference solution, such as purified water, and view against a color comparison plate with suitable lighting.

**8.2.2. ASSAY**

- 8.2.2.1. Perform a manual standardization or titrant check of 1 N Sulfuric Acid per Standardization of Titrants.
- 8.2.2.2. Accurately weigh 6g of sample and add 40 mL of purified water in a clean flask. Stopper the flask and allow to cool to room temperature.
- 8.2.2.3. Add 150 $\mu$ L of Phenolphthalein as the indicator and titrate using previously standardized 1N Sulfuric Acid to a colorless endpoint (V1).
- 8.2.2.4. Add 150 $\mu$ L of Methyl Orange as the indicator.
- 8.2.2.5. Titrate using previously standardized 1N Sulfuric Acid to a pink endpoint (V2).
- 8.2.2.6. Calculate the percentage of Sodium Hydroxide using the following equation:

$$\%NaOH = \frac{(V_2) \times N \text{ } H_2SO_4 \times 4.00}{\text{Sample Weight (g)}}$$

**8.2.3. CHLORIDE**

- 8.2.3.1. Thoroughly rinse Nessler Tubes and glassware using purified water prior to use.
- 8.2.3.2. **Sample Analysis Preparation:**
  - 8.2.3.2.1. Weigh 10.0g of sample into a clean beaker or Nessler tube.
  - 8.2.3.2.2. Dilute to ~20 mL with purified water.
  - 8.2.3.2.3. Slowly, using extreme caution, acidify with Nitric Acid, testing with litmus paper.
  - 8.2.3.2.4. Dilute to ~40 mL with purified water.
- 8.2.3.3. **Standard Preparation:**
  - 8.2.3.3.1. Dilute 70.5 $\mu$ L of 0.02N HCl to ~40 mL with purified water in a Nessler tube.

**8.2.3.4. Analysis procedure:**

- 8.2.3.4.1. To both the standard and sample solutions, add 1 mL of concentrated Nitric Acid and 1 mL of 0.1N Silver Nitrate TS.
- 8.2.3.4.2. Dilute both the sample and standard solutions to 50 mL with purified water.
- 8.2.3.4.3. Mix and allow to sit for 5 minutes using a calibrated timer.
- 8.2.3.4.4. Acceptance Criteria: After 5 minutes, the turbidity in the sample solution does not exceed the turbidity produced by the standard when viewed against a dark background. If the sample cannot be determined visually, analyze turbidity utilizing the turbidimeter and record the NTU result. The sample NTU must be less than the standard NTU in order to be considered acceptable.
- 8.2.3.4.5. If utilized, follow the appropriate SOP to measure and record the turbidity of the standard and the blank. Any turbidity in the solution of the sample should not exceed that in the standard.

**8.2.4. ENDOTOXINS :**

- 8.2.4.1. Pipette 0.200 mL of sample into a sterile vial and add 1.600 mL of LAL reagent water.
- 8.2.4.2. Add 0.160 mL of concentrated Hydrochloric acid to acidify.
- 8.2.4.3. Check the pH of the solution with pH paper; solution must be acidic.
  - 8.2.4.3.1. If basic, add HCl in increments until acidic.
    - 8.2.4.3.1.1. Add approximately 0.02 mL of HCl.
- 8.2.4.4. Once acidic, add sufficient buffer of a pH range ~9-10 until the solution is between pH 6-8.
  - 8.2.4.4.1. Add approximately 1.0 mL of buffer.
- 8.2.4.5. Dilute with LAL reagent water to a final volume of 10 mL.
- 8.2.4.6. Follow the Endosafe Nexgen PTS Endotoxin Reader SOP for sample analysis.
  - 8.2.4.6.1. The dilution factor is 50.

**8.2.5. HEAVY METALS (Pb) :**

- 8.2.5.1. Refer to Analytical Method of Analysis: Sodium Hydroxide via ICP-MS, BSI-ATM-0074.

*Alternate Method:*

- 8.2.5.2. Standard and Solution Preparation:
  - 8.2.5.2.1. Lead Standard Solution (0.01 mg of Pb in 1 mL): Dilute 10 mL of lead stock solution to 100 mL with purified water. This must be prepared at the time of use.
  - 8.2.5.2.2. Thioacetamide-glycerin base: Thoroughly mix 1 mL of thioacetamide with 5 mL of Glycerin base. Heat in a boiling bath for 20 seconds. Prepare immediately before use.
- 8.2.5.3. Procedure:
  - 8.2.5.3.1. Note: Prepare in hood, and use caution for standard and sample preparation to avoid spattering of sample.
  - 8.2.5.3.2. Sample Preparation: Weigh 30 g of sample into a suitable beaker and carefully add 18 mL of concentrated nitric acid.
  - 8.2.5.3.3. Standard Preparation: Weigh 10 g of sample and add 5 mL of concentrated nitric acid. Add 2 mL of 0.01 mg Lead Standard Solution (0.01mg/ml Pb).

- 8.2.5.3.4. Place both the standard and sample on a hot plate and evaporate to dryness. Cool and dissolve each residue with 20 mL of purified water. Adjust the pH to between 3 and 4 utilizing a pH meter, with 1N acetic acid, or 10% ammonium hydroxide.
- 8.2.5.3.5. Transfer the solutions to separate Nessler Color Comparison Tubes. Add 1.2 mL of freshly prepared thioacetamide-glycerin base to each of the solutions and mix. QS each tube to 50 mL and mix.
- 8.2.5.3.6. Any brown color produced in the sample solution must not exceed that in the standard solution.
- 8.2.6. **IDENTIFICATION (SODIUM)** :
- 8.2.6.1. Pipette 1mL of sample into a test tube containing 25 mL of purified water.
- 8.2.6.2. Add 2mL of 15% Potassium Carbonate and heat to boiling.
- 8.2.6.3. Allow to cool in an ice bath and as necessary, rub the inside of the test tube with a glass rod to initiate precipitation.
- 8.2.6.4. Add 4 mL of Potassium Pyroantimonate TS and heat to boiling.
- 8.2.6.5. Allow to cool in an ice bath and necessary, rub the inside of the test tube with a glass rod to initiate precipitation.
- 8.2.6.6. A dense precipitate must form in order to pass test.
- 8.2.7. **IRON** :
- 8.2.7.1. Refer to Analytical Method of Analysis: Sodium Hydroxide via ICP-MS, BSI-ATM-0074.
- Alternate Method:*
- 8.2.7.2. Procedure:
  - 8.2.7.2.1. Thoroughly rinse glassware with purified water prior to use.
  - 8.2.7.2.2. Sample Preparation: To 10 g of sample, add 0.1 mL of phenolphthalein indicator solution, neutralize with hydrochloric acid (solution will turn from pink to clear) and dilute with water to 40 mL in a graduated cylinder. Transfer to a Nessler Color Comparison Tube.
  - 8.2.7.2.3. 0.500ppm Iron Standard Preparation: Pipette 0.5mL of Iron standard (0.01 mg/mL Fe) into a graduated cylinder and dilute to 40 mL with purified water. Transfer to a Nessler Color Comparison Tube.
  - 8.2.7.2.4. To the sample and standard solutions, add 30-50 mg of ammonium peroxydisulfate crystals, 3 mL of Hydrochloric acid, and 3 mL of 30% Ammonium Thiocyanate reagent solution, and mix.
  - 8.2.7.2.5. Any red color in the sample must not exceed the standard solution.