BIOSPECTRA

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SODIUM HYDROXIDE 5N
TESTING METHODS

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1. PURPOSE:

1.1. To provide Laboratory personnel with a procedure for analyzing Sodium Hydroxide 5 N In-Process, Stability, and Finished Good samples.

2. SCOPE:

2.1. Applies to the analysis of Sodium Hydroxide 5N In-Process, Stability, and Finished Goods in the Laboratory. Methods include testing for all grades of Sodium Hydroxide 5N sold by BioSpectra; only the specific tests required for the requested grade must be tested.

3. RESPONSIBILITIES:

3.1. The Laboratory Manager, or qualified, designee 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 Laboratory Management 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-MEM-0130, Endosafe NexGen PTS Endotoxin Reader: Qualified Products
5.2. BSI-SOP-0019, Result Reporting
5.3. BSI-SOP-0098, Balance SOP
5.4. BSI-SOP-0126, Laboratory Notebooks
5.5. BSI-SOP-0135, Laboratory Chemicals
5.6. BSI-SOP-0140, Standardization of Titrants
5.7. BSI-SOP-0242, Bangor Portable Turbidimeter and Calibration SOP
5.8. BSI-SOP-0244, VWR Gravity Convection Oven Operation and Calibration (Model Number 414005-106)
5.9. BSI-SOP-0303, NexION 350X ICP-MS SOP
5.10. BSI-SOP-0350, Anton Paar DMA 35 Portable Density Meter Operation and Calibration
5.11. BSI-SOP-0345, Endosafe nexgen-PTS Endotoxin Reader SOP
5.12. *ACS Reagent Chemicals*, current edition
5.13. *USP-NF* current edition

6. EQUIPMENT:

6.1. Analytical Balance
6.2. Hach Portable Turbidimeter Model 2100 Q, or equivalent
6.3. Calibrated Oven
6.4. Anton Paar DMA 35 Portable Density Meter
6.5. Endosafe PTS Endotoxin Reader, or equivalent
6.6. NexION 350X ICP-MS

7. REAGENTS:

7.1. **0.02N Hydrochloric Acid (HCl):** Slowly add 20 mL of 0.1N Hydrochloric Acid to 80 mL of purified water to make a total volume of 100 mL.
7.2. **Concentrated Hydrochloric Acid:** purchased commercially.
7.3. **Endosafe PTS Cartridge 1-0.01 EU/mL:** purchased commercially.
7.4. **LAL Reagent Water:** purchased commercially.
7.5. **Litmus Paper:** purchased commercially
7.6. **Nitric Acid (HNO_3):** purchased commercially
7.7. **Phenolphthalein:** purchased commercially.
7.8. **Phenolphthalein Indicator:** Dissolve 1.0 g of phenolphthalein in 100 mL of reagent grade alcohol.
7.9. **pH 9-10 Buffer (0.25M Tris Base):** purchased commercially.
7.10. **Potassium Carbonate (15%):** Weigh 15.000g of Potassium Carbonate and transfer to a 100mL volumetric flask. Dissolve and dilute to volume with purified water.
7.11. **Potassium Hydrogen Phthalate (KHP):** Crush and dry a suitable amount of KHP at 120°C for 2 hours. Allow to cool to ambient temperature in a desiccator.
7.12. **Potassium Pyroantimonate TS:** purchased commercially.
7.13. **Purified Water: g**enerated in-house
7.14. **0.1N Silver Nitrate ($AgNO_3$) TS:** purchased commercially

8. ANALYTICAL PROCEDURES:

8.1. IN-PROCESS TESTING:

8.1.1. NORMALITY (CONFIRMATION 1 AND 2) REFER TO BATCH RECORD:

8.1.2. **KHP (Potassium Hydrogen Phthalate) Preparation:**

8.1.2.1. Crush and dry a suitable amount of KHP at 120°C for 2 hours. Allow to cool to ambient temperature in a desiccator.

8.1.3. **Burette Preparation:**

8.1.3.1. Fill a 25-mL volumetric flask with sample. Quantitatively transfer the aliquot to a 250-mL volumetric flask with purified water. Rinse the 25-mL flask by filling the flask halfway with purified water, shaking it, then transferring the rinse to the 250-mL volumetric flask. Perform the rinse procedure in duplicate. Fill the 250-mL volumetric flask to volume with purified water. Mix well and cool to 25° ± 2°C. QS the sample solution to 250 mL after cooling is complete.

8.1.3.2. Prime the 50-mL burette by filling it with the diluted sample solution. Empty the burette and repeat.

8.1.3.3. Fill the burette to the required volume with the prepared sample solution.

8.1.4. **Sample Preparation:**

8.1.4.1. Weigh 4.0 – 4.2 g of the previously dried KHP into a 250-mL beaker.

8.1.4.2. Add 100 mL of purified water down the sides of the beaker to avoid the loss of KHP.

8.1.5. **Analysis Procedure:**

8.1.5.1. To the KHP solution, add 150 µL phenolphthalein indicator.

8.1.5.2. Titrate the KHP using the sample solution in the burette, to a pink endpoint.

8.1.5.3. Calculate the normality using the following equation:

$$N = \frac{KHP\ Weight\ (g) \times KHP\ Purity\ \times 10}{0.20423\ \times mL\ of\ NaOH\ Sample\ Solution}$$

Where:

$$KHP\ Purity = \frac{Assay\ percent\ of\ KHP}{100} \quad (from\ manufacturer's\ CoA)$$

$$0.20423 = \frac{Formula\ weight\ of\ KHP}{1000}$$

$$10 = Dilution\ Factor$$

8.2. **FINISHED GOOD TESTING:**

8.2.1. **APPEARANCE AND COLOR:**

8.2.1.1. Transfer 50 mL of sample into a Nessler Color Comparison 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. **CHLORIDE:**

8.2.2.1. Thoroughly rinse Nessler tubes using purified water prior to use.

8.2.2.2. **Sample Preparation:**

8.2.2.2.1. Weigh 2.0 g of sample and quantitatively transfer to a 50-mL Nessler Color Comparison Tube using purified water.

8.2.2.2.2. Dilute to ~20 mL with purified water.

8.2.2.2.3. Slowly, using extreme caution, acidify the sample with nitric acid to litmus.

8.2.2.2.4. Dilute to 50 mL with purified water.

8.2.2.3. **5 ppm Standard Preparation:**

8.2.2.3.1. Dilute 14.1 µL of 0.02N HCl to ~40 mL with purified water.

8.2.2.4. **Analysis:**

8.2.2.4.1. To both the sample and standard solutions, add 1 mL of concentrated nitric acid and 1 mL of 0.1N Silver Nitrate TS.

8.2.2.4.2. Mix and allow solutions to sit for 5 minutes using a calibrated timer.

8.2.2.4.3. After 5 minutes, the turbidity in the sample solution does not exceed the turbidity produced by the standard when viewed against a dark background. Analyze turbidity utilizing the turbidity meter and record the sample NTU results.

8.2.3. **ENDOTOXIN:**

8.2.3.1. Pipette 0.200 mL of sample into a sterile vial and add 1.600 mL of LAL reagent water.

8.2.3.2. Check the pH of the solution with pH paper.

8.2.3.2.1. If the solution is basic, add HCl in small increments until the solution is acidic.

8.2.3.3. Once acidic, add sufficient pH 9-10 buffer solution until the pH is between 6-8.

8.2.3.4. Dilute to 10 mL with LAL reagent water.

8.2.3.5. Follow the Endosafe Nexgen PTS Endotoxin Reader SOP for sample analysis.

8.2.3.5.1. The dilution factor is 50.

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

8.2.4.1. Refer to NexION 350X ICP-MS SOP.

8.2.5. **IDENTIFICATION (SODIUM):**

8.2.5.1. Pipette 1 mL of sample into a test tube containing 25 mL of purified water.

8.2.5.2. Add 2 mL of 15% Potassium Carbonate and heat to boiling.

8.2.5.3. Allow to cool in an ice batch and as necessary, rub the inside of the test tube with a glass rod to initiate precipitation.

8.2.5.4. Add 4 mL of Potassium Pyroantimonate TS and heat to boiling.

8.2.5.5. Allow to cool in an ice bath and as necessary, rub the inside of the test tube with a glass rod to initiate precipitation.

8.2.5.6. A dense precipitate must form in order to pass test.

8.2.6. **IRON (Fe)**:

8.2.6.1. Refer to NexION 350X ICP-MS SOP.

8.2.7. **NORMALITY**:

8.2.7.1. **KHP (Potassium Hydrogen Phthalate) Preparation:**

8.2.7.1.1. Crush and dry a suitable amount of KHP at 120°C for 2 hours. Allow to cool to ambient temperature in a desiccator.

8.2.7.2. **Burette Preparation:**

8.2.7.2.1. Fill a 25-mL volumetric flask with sample. Quantitatively transfer the aliquot to a 250-mL volumetric flask with purified water. Rinse the 25-mL flask by filling the flask halfway with purified water, shaking it, then transferring the rinse to the 250-mL volumetric flask. Perform the rinse procedure in duplicate. Fill the 250-mL volumetric flask to volume with purified water. Mix well and cool to 25° ± 2°C. QS the sample solution to 250 mL after cooling is complete.

8.2.7.2.2. Prime the 50-mL burette by filling it with the diluted sample solution. Empty the burette and repeat.

8.2.7.2.3. Fill the burette to the required volume with the prepared sample solution.

8.2.7.3. **Sample Preparation:**

8.2.7.3.1. Weigh 4.0 – 4.2 g of the previously dried KHP into a 250-mL beaker.

8.2.7.3.2. Add 100 mL of purified water down the sides of the beaker to avoid the loss of KHP.

8.2.7.4. **Analysis Procedure:**

8.2.7.4.1. To the KHP solution, add 150 µL phenolphthalein indicator.

8.2.7.4.2. Titrate the KHP using the sample solution in the burette, to a pink endpoint.

8.2.7.4.3. Calculate the normality using the following equation:

$$N = \frac{KHP\ Weight\ (g) \times KHP\ Purity\ \times 10}{0.20423\ \times mL\ of\ NaOH\ Sample\ Solution}$$

Where:

$$KHP\ Purity = \frac{Assay\ percent\ of\ KHP}{100} \quad (from\ manufacturer's\ CoA)$$

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