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SODIUM HYDROXIDE 5N

TESTING METHODS

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1. PURPOSE:

1.1. To provide the Quality Control (QC) 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 QC 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 Executive Director of Quality Control is responsible for training, maintenance and implementation of this procedure.
3.2. The QC Analysts are responsible for compliance with the terms of this procedure. This includes notifying the Executive Director of Quality Control 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. *ACS Reagent Chemicals*, current edition
5.2. *USP-NF* current edition
5.3. Anton Paar DMA 35 Portable Density Meter Operation and Calibration
5.4. Bangor Portable Turbidimeter and Calibration SOP
5.5. Laboratory Chemicals
5.6. Balance SOP
5.7. Laboratory Notebooks
5.8. NexION 350X ICP-MS SOP
5.9. Result Reporting
5.10. Sodium Hydroxide 5N Analytical Procedure
5.11. Standardization of Titrants
5.12. VWR Gravity Convection Oven Operation and Calibration (Model Number 414005-1016)

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. ANALYTICAL PROCEDURES:

7.1. IN-PROCESS TESTING:

7.1.1. SPECIFIC GRAVITY MONITOR:

7.1.1.1. QC or Manufacturing is to perform a density check of the sample.

7.1.1.2. Perform a water check on the DMA 36 Density Meter before the sample analysis. Refer to DCN:19-002946 for instrument operation and water check analysis.
7.1.1.3. Analyze the sample at 20°C ± 1°C. Refer to DCN:19-002946 for instrument operation and sample analysis. Record the density in the appropriate Laboratory Documentation.
7.1.1.4. Clean the instrument immediately after use following DCN: 19-002946.

7.1.2. **CHLORIDE ≤ 5 PPM:**

7.1.2.1. **Note: Record <5ppm or >5ppm in the batch record.**
7.1.2.2. **Sample preparation**:
7.1.2.2.1. Thoroughly rinse Nessler tubes using purified water prior to use.
7.1.2.2.2. Weigh 2.0g of sample and quantitatively transfer to 50mL Nessler Color Comparison Tube using purified water.
7.1.2.2.3. Dilute sample to ~20mL with purified water.
7.1.2.2.4. Slowly, using extreme caution, acidify the sample with nitric acid to litmus.
7.1.2.2.5. Dilute to 40mL with purified water.
7.1.2.3. **Standard Preparation**: Standard preparation for internal reporting only.
7.1.2.3.1. 2ppm Limit: Dilute 5.64µL of 0.02N HCl to ~40mL with purified water.
7.1.2.3.2. 5ppm Limit: Dilute 14.1µL of 0.02N HCl to ~40mL with purified water.
7.1.2.3.3. 10ppm Limit: Dilute 28.2µL of 0.02N HCl to ~40mL with purified water.
7.1.2.3.4. 50ppm Limit: Dilute 141µL of 0.02N HCl to ~40mL with purified water.

Procedure:

7.1.2.3.5. To the standard and sample solutions prepared above, add 1mL of concentrated nitric acid and 1mL of 0.1N Silver Nitrate TS.
7.1.2.3.6. Dilute each tube to 50mL with purified water.
7.1.2.3.7. Mix and allow to sit for 5 minutes, using a calibrated timer.
7.1.2.3.8. 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.

7.1.3. **NORMALITY REPORT:**

7.1.3.1. Refer to Section 7.2.5 for sample preparation and analysis.

7.2. FINISHED GOOD TESTING:

7.2.1. APPEARANCE AND COLOR CLEAR, COLORLESS LIQUID:

7.2.1.1. Transfer 50mL of sample into a Nessler Color Comparison tube.

7.2.1.2. 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.

7.2.2. CHLORIDE ≤ 5PPM:

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

7.2.2.2. **Sample Preparation:**

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

7.2.2.2.2. Dilute to ~20mL with purified water.

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

7.2.2.2.4. Dilute to 50mL with purified water.

7.2.2.3. **5 ppm Standard Preparation:**

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

7.2.2.4. **Analysis:**

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

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

7.2.2.4.3. 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.

7.2.3. HEAVY METALS (PB) ≤ 1PPM:

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

7.2.4. IRON ≤ 0.5 PPM:

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

7.2.5. NORMALITY 4.95-5.05 N:

7.2.5.1. KHP (Potassium Hydrogen Phthalate) preparation:

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

7.2.5.2. Burette preparation:

7.2.5.2.1. Fill a 25mL volumetric flask with sample. Quantitatively transfer the aliquot to a 250mL volumetric flask with purified water. Rinse the 25mL flask by filling the flask halfway with purified water, shaking it, then transferring the rinse to the 250mL volumetric flask. Perform the rinse procedure in duplicate. Fill the 250mL volumetric flask to

volume with purified water. Mix well and cool to 25°±2°C. QS the sample solution to 250mL after cooling is complete.

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

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

7.2.5.3. Sample preparation:

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

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

7.2.5.4. Analysis Procedure:

7.2.5.4.1. To the KHP solution, add phenolphthalein indicator.

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

7.2.5.4.3. Calculate the normality using the following equation:

$$N = \frac{(KHP\ weight\ g)(KHP\ Purity)(10)}{(0.20423)(mL\ of\ NaOH\ sample\ solution)}$$

$$KHP\ Purity\ =\ Assay\ percent\ of\ KHP/100\ (from\ manufacturer's\ CoA)$$

$$0.20423\ =\ Formula\ weight\ of\ KHP/1000$$

$$10\ =\ Dilution\ Factor$$