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# SODIUM HYDROXIDE 2N

# 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 2N In-Process, Stability, and Finished Good samples.

## 2. SCOPE:

2.1. Applies to the analysis of Sodium Hydroxide 2N In-Process, Stability, and Finished Goods in the QC Laboratory. Methods include testing for all grades of Sodium Hydroxide 2N 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. Bangor Portable Turbidimeter and Calibration SOP
5.4. Laboratory Chemicals
5.5. Balance SOP
5.6. VWR Gravity Convection Over Operation and Calibration (Model Number 414005-106)
5.7. Laboratory Nexgen-PTS Endotoxin Reader SOP, DCN:18-002735
5.8. Laboratory Notebooks
5.9. NexION 350X ICP-MS SOP
5.10. Result Reporting
5.11. Sodium Hydroxide 2N Analytical Procedure
5.12. Standardization of Titrants

## 6. EQUIPMENT:

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

## 7. ANALYTICAL PROCEDURES:

### 7.1. IN-PROCESS TESTING:

7.1.1. **APPEARANCE** **REPORT:**

7.1.1.1. Transfer 50mL of sample into a Nessler tube.

7.1.1.2. Inspect the sample to ensure it is particulate free.

7.1.2. **NORMALITY (CONFIRMATION 1 AND 2)** **1.990 – 2.010N:**

7.1.2.1. KHP (Potassium Hydrogen Phthalate) preparation:

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

7.1.2.2. Burette preparation:

7.1.2.2.1. Allow the NaOH 2N sample to come to 25°C ±2°C.

7.1.2.2.2. Prime the 50 mL burette by filling it with the NaOH 2N sample solution. Empty the burette and repeat.

7.1.2.2.3. Fill the burette to the required volume with the NaOH 2N sample solution. .

7.1.2.3. Sample preparation:

7.1.2.3.1. Weigh 12.0g of the previously dried KHP into a 250mL beaker.

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

7.1.2.4. Analysis Procedure:

7.1.2.4.1. To the KHP solution, add phenolphthalein indicator.

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

7.1.2.4.3. Calculate the normality using the following equation:

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

$$KHP\ Purity\ =\ Assay\ percent\ of\ KHP/100\ (from\ manufacturer's\ CoA)$$
$$0.20423\ =\ Formula\ weight\ of\ KHP/1000$$

**NOTE:**

- **If Confirmation 1 sample is in specification, perform Normality on the Confirmation 2 sample. Both must be in specification to release for packaging.**
- **If Confirmation 1 sample is out of specification perform Assay % (w/w) and inform appropriate QC management of both results to make adjustment to the blend.**

7.1.3. **ASSAY** **REPORT:**

7.1.3.1. **NOTE: only required if Normality Confirmation sample is out of specification.**

7.1.3.2. Perform a manual standardization or titrant check of 1N Sulfuric Acid per Standardization of Titrants.

7.1.3.3. Accurately weigh 10-15g of sample and add 100mL of purified water in a clean flask. Stopper the flask and allow to cool to room temperature.

7.1.3.4. Add Phenolphthalein as the indicator and titrate using previously standardized 1N Sulfuric Acid to a colorless endpoint ($V_1$).
7.1.3.5. Add Methyl Orange as the indicator.
7.1.3.6. Titrate using previously standardized 1N Sulfuric Acid to a pink endpoint ($V_2$).
7.1.3.7. Calculate the percentage of Sodium Hydroxide using the following equation:

$$\% \text{ NaOH} = \frac{(V2)(N\ H2SO4)(4.00)}{Sample\ Weight\ (g)}$$

7.2. **FINISHED GOOD TESTING:**

7.2.1. **APPEARANCE AND COLOR REFER TO SUMMARY SHEET:**
7.2.1.1. Transfer 50mL of sample into a Nessler tube.
7.2.1.2. In order to pass, the test solution is complete, clear and colorless, and free from particulate matter.
7.2.1.3. 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 REFER TO SUMMARY SHEET:**
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 40mL 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. Dilute each tube to 50mL with purified water.
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. **ENDOTOXINS REFER TO SUMMARY SHEET:**
7.2.3.1. Pipet 0.200mL of sample into a sterile vial and add 1.600 mL of LAL reagent water.
7.2.3.2. Add concentrated Hydrochloric acid to acidify.
7.2.3.3. Check the pH of the solution with pH paper: solution must be acidic.
7.2.3.3.1. If basic add HCl in increments until acidic.
7.2.3.3.1.1. Add approximately 1-2µL of HCl.

7.2.3.4. Once acidic add sufficient buffer of a pH range ~9-10 until the solution is between pH 6-8.
7.2.3.4.1. Add approximately 0.3mL of buffer.
7.2.3.5. Dilute with LAL reagent water to a final volume of 10mL.
7.2.3.6. Follow the Endosafe Nexgen PTS Endotoxin Reader SOP for sample analysis.
7.2.3.6.1. The dilution factor is 50.

7.2.4. **HEAVY METALS (PB) REFER TO SUMMARY SHEET:**
7.2.4.1. Refer to NexION 350X ICP-MS SOP.

7.2.5. **IDENTIFICATION (SODIUM) PASSES TEST:**
7.2.5.1. Pipette 1mL of sample into a test tube containing 25mL of purified water.
7.2.5.2. Add 2mL of 15% Potassium Carbonate and heat to boiling
7.2.5.3. Allow to cool in ice bath and as necessary, rub the inside of the test tube with a glass rod to initiate precipitation
7.2.5.4. No precipitate should be formed at this stage of analysis.
7.2.5.5. Add 4mL Potassium Pyroantimonate TS and heat to boiling.
7.2.5.6. Allow to cool in ice bath and as necessary, rub the inside of the test tube with a glass rod to initiate precipitation.
7.2.5.7. A dense precipitate must form in order to pass test.

7.2.6. **IRON REFER TO SUMMARY SHEET:**
7.2.6.1. Refer to NexION 350X ICP-MS SOP.

7.2.7. **NORMALITY REFER TO SUMMARY SHEET:**
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