BIOSPECTRA
100 Majestic Way, Bangor, PA 18013 / www.biospectra.us

SODIUM CHLORIDE 5M TESTING METHODS

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1. PURPOSE:

1.1. To provide the Quality Control (QC) Laboratory personnel with a procedure for examining Sodium Chloride 5M samples.

2. SCOPE:

2.1. Applies to examination of Sodium Chloride 5M samples in the QC Laboratory. This document applies to both the Bangor, PA and Stroudsburg, PA BioSpectra sites.

3. RESPONSIBILITIES:

3.1. The Executive Director of Quality Control is responsible for the control, 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 Quality Assurance and Quality Control Managers, or designees, if any analyses fail to meet their respective specifications.

4. REFERENCES:

4.1. BSI-SOP-0098, Balance SOP
4.2. BSI-SOP-0126, Laboratory Notebooks
4.3. BSI-SOP-0140, Standardization of Titrants
4.4. BSI-SOP-0345, Endosafe nexgen-PTS Endotoxin Reader SOP
4.5. BSI-SOP-0350, Anton Paar DMA 35 Portable Density Meter Operation and Calibration
4.6. *ACS, Reagent Chemicals,* current edition
4.7. *Current USP*

5. EQUIPMENT:

5.1. Analytical Balance
5.2. Burette
5.3. Endosafe nexgen-PTS Endotoxin Reader

6. REAGENTS:

6.1. **Acetic Acid (Glacial):** is commercially purchased.
6.2. **0.1 N Silver Nitrate (Silver Nitrate TS):** is commercially purchased.
6.3. **6N Ammonium Hydroxide:** Slowly add 41mL of concentrated 29% ammonium hydroxide to 25mL of purified water in a 100mL volumetric flask. Stopper, and allow to cool to room temperature and dilute to volume with purified water.
6.4. **15% (w/v) Potassium Carbonate:** Dissolve 15 g of potassium carbonate to about 75 mL of water in a volumetric flask. Once dissolved, bring to volume.
6.5. **Eosin Y TS indicator:** Dissolve 50 mg of eosin Y in 10 mL of water.
6.6. **Potassium Pyroantimonate TS**: is commercially purchased
6.7. **Methanol:** is commercially purchased
6.8. **Nitric Acid, Concentrated:** is commercially purchased

7. ANALYTICAL PROCEDURES:

In-Process Analysis:

7.1. **APPEARANCE AND COLOR** **Report:**
7.1.1. Transfer 50mL of the sample in a clean, dry color Nessler tube.
7.1.2. In order to pass, test solution is a clear, colorless solution. 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. **DENSITY @20±1°C** **Report:**

7.2.1. QC or Manufacturing to perform a density check of the material.

7.2.2. Perform a water check on the DMA 35 Density meter before the sample analysis. Record the density of the sample from the DMA 35 Density meter. Clean immediately after use. Refer to DCN: BSI-SOP-0350 for instrument operation, water check, and sample analysis.

7.2.2.1. Density of 5M NaCl is ~1.19g/mL.

7.3. **ASSAY** **4.9-5.1M:**

7.3.1. Standardize or perform a daily check of 0.1N $AgNO_3$ as per Standardization of Titrants.

7.3.2. Transfer 10mL of the sample solution into a 100mL volumetric flask and dilute to volume with purified water.

7.3.3. Accurately transfer 3mL of the diluted solution to a suitable titration vessel. Add 7mL of purified water, 10mL of glacial acetic acid, 75mL of methanol, and Eosin Y TS as the indicator.

7.3.4. While stirring, titrate with 0.1N Silver Nitrate TS to a pink endpoint. Each mL of 0.1N Silver Nitrate is equivalent to 5.844mg of Sodium Chloride.

$$M\ NaCl = \frac{(mL\ of\ titer)(N\ of\ titer)(10)}{3}$$

7.3.4.1. **If required for adjustment Assay (w/w%) ~24.6%: either method can be used.**

Manual titration

7.3.4.1.1. Accurately weight 0.5g of sample to a suitable titration vessel.

7.3.4.1.2. Add ~50mL of purified water.

7.3.4.1.3. Add 7mL of purified water, 10mL of glacial acetic acid, 75mL of methanol, and Eosin Y TS as the indicator.

7.3.4.1.4. While stirring, titrate with 0.1N Silver Nitrate TS to a pink endpoint. Each mL of 0.1N Silver Nitrate is equivalent to 5.844mg of Sodium Chloride.

$$\%NaCl = \frac{(mL)(\ N\ of\ 0.1N\ AgNO3)(5.844)}{Sample\ Weight\ (g)}$$

Potentiometric titration

7.3.4.1.1. Accurately weight 0.5g of sample to a suitable titration vessel.

7.3.4.1.2. Add ~100mL of purified water.

7.3.4.1.3. Titrate with 0.1N silver nitrate to a potentiometric endpoint.

7.3.4.1.4. Utilize Same calculation above.

Finished Good Analysis:

7.4. **APPEARANCE AND COLOR** **Clear Colorless Solution:**

7.4.1. Transfer 50mL of the sample in a clean, dry color Nessler tube.

7.4.2. In order to pass, test solution is a clear, colorless solution. 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.

7.5. **ASSAY** **4.9-5.1M:**

Standardize or perform a daily check of 0.1N $AgNO_3$ as per Standardization of Titrants.

7.5.1. Transfer 10mL of the sample solution into a 100mL volumetric flask and dilute to volume with purified water.

7.5.2. Accurately transfer 3mL of the diluted solution to a suitable titration vessel. Add 7mL of purified water, 10mL of glacial acetic acid, 75mL of methanol, and Eosin Y TS as the indicator.

7.5.3. While stirring, titrate with 0.1N Silver Nitrate TS to a pink endpoint. Each mL of 0.1N Silver Nitrate is equivalent to 5.844mg of Sodium Chloride.

$$M\ NaCl = \frac{(mL\ of\ titer)(N\ of\ titer)(10)}{3}$$

7.6. **ENDOTOXIN (USP/EP) ≤2.5EU/mL:**

7.6.1. Add 0.34mL of sample into a sterile capped conical tube or other suitable sterile vessel.

7.6.2. Dilute to 10mL with LAL reagent water.

7.6.2.1. The sample preparation may be scaled to desired volume.

7.6.3. Enter specification of 2.5EU/mL.

7.6.4. Ensure all data is accurate before proceeding to load sample.

7.6.5. Load 25µL of sample in to each well and run test.

7.6.6. Ensure all suitability requirements meet specification.

7.6.7. Report result directly from instrument printout.

7.7. **IDENTIFICATION SODIUM Meets Requirements:**

7.7.1. Transfer 0.34mL of sample solution into a suitable beaker.

7.7.2. Add 2mL of purified water.

7.7.3. Add 2mL of 15% potassium carbonate and heat to a boil. No precipitate is formed.

7.7.4. Add 4mL of Potassium Pyroantimonate TS and heat to a boil.

7.7.5. Allow to cool in an ice bath, and if necessary, rub the inside of the test tube with a glass rod. A dense precipitate is formed to pass test.

7.8. **IDENTIFICATION CHLORIDE Meets Requirements:**

7.8.1. Transfer 5mL of sample solution to a beaker.

7.8.2. To the sample solution add 1mL of 0.1N silver nitrate.

7.8.3. Mix thoroughly.

7.8.4. A curdled, white precipitate is formed.

7.8.5. The precipitate is insoluble when 1 mL of concentrated Nitric acid is added but soluble when 6 mL of 6N Ammonium hydroxide is added.

7.9. **MICROBIAL CONTENT TAMC ≤50CFU/g TYMC ≤150CFU/g:**

7.9.1. Package ~20g into a sterile container and send to MPL Laboratories. The analysis request form should include TAMC and TYMC. In order to pass the Total Aerobic Microbial Count must be ≤50CFU/g, the Total Yeast and Mold Count must be ≤150CFU/g.

7.10. **TRACE METALS As, Cu, Fe, and Pb NMT 2ppm:**

7.10.1. Refer to NexION ICP-MS for sample preparation and analysis.