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

To Whom It May Concern,

INTRODUCTION: The following analyses are conducted for Sodium Decanoate, product code ND3220, in accordance with the Sodium Decanoate Testing Methods DCN: 18-002419 v.5.0 and Certificate of Analysis DCN: 18-002529 v.6.0. Specific details for the procedures were also obtained from Spectrum Two UATR SOP DCN: 16-001330 v.3.0.

1. APPEARANCE — White to off-white Powder:

1.1. Place a suitable amount of the sample in a clean, dry glass beaker.
1.2. In an area with sufficient lighting, view the sample from all sides.
1.3. The sample should be white to off-white in color and characteristic of powder.
1.4. Report any foreign matter or nonconformity of sample immediately to the appropriate personnel.

2. ASSAY (Dried Basis) — 97.0% - 103.0%:

2.1. **Standardization of 0.1N Perchloric Acid :**

2.1.1. Note: Before each titration, record the temperature of the 0.1N Perchloric acid.
2.1.2. In a well ventilated area, accurately weigh about 0.7g of previously dried Potassium hydrogen phthalate and transfer into a suitable beaker (150mL size is ideal), record weight immediately after stabilization.
2.1.3. Ensure stir bar is in the beaker before the addition of the glacial acetic acid to avoid splashing.
2.1.4. Dissolve in 50mL of Glacial Acetic Acid.
2.1.5. Add 0.1mL of a 1% crystal violet solution prepared by dissolving 100mg of crystal violet in 10mL of glacial acetic acid.
2.1.6. Prepare a burette with 0.1N Perchloric Acid in glacial acetic acid and titrate with 0.1N Perchloric Acid to a Blue-Green end point.
2.1.7. Note: The crystal violet solution will turn blue before blue green, the end point is immediately AFTER the blue end point when the first appearance of a green color is indicated.
2.1.8. Calculate normality of the titrant utilizing the following equation:

$$N\ Perchloric\ Acid = \frac{g\ KHP(\frac{Reference\ Material\ Assay}{100})}{0.20423\ x\ (Perchloric\ Acid\ Volume\ (mL) - Blank\ (mL))}$$

2.1.9. Perform standardization in triplicate with a single blank.

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2.2. **Sample preparation:**

2.2.1. Note: Before each titration, record the temperature of the 0.1N Perchloric acid.

2.2.2. Accurately weigh 0.5g of Sodium Decanoate and transfer to a suitable beaker.

2.2.3. Dissolve in ~50mL of Glacial Acetic Acid.

2.2.4. Add 0.1mL of a 1% crystal violet solution prepared by dissolving 100mg of crystal violet in 10mL of glacial acetic acid.

2.2.5. Prepare a burette with 0.1N Perchloric Acid in glacial acetic acid and titrate with 0.1N Perchloric Acid to a Blue-Green end point.

2.2.6. Note: The crystal violet solution will turn blue before blue green; the end point is immediately AFTER the blue end point when the first appearance of a green color is indicated.

2.2.7. To calculate the result: Reference the LOD result, calculate the temperature correction refer to section 2.3. and apply the calculation in section 2.4.

2.3. **Temperature Correction:**

2.3.1. 0.1N Perchloric acid in glacial acetic acid has a high coefficient of expansion. Due to the high coefficient of expansion of the titrant and inherent variability of temperature, care should be taken to correct for the difference in temperature from when the titration is carried out (T_i) and from when the titrant was standardized (T_s).

2.3.1.1. To Calculate the Temperature Correction Factor Coefficient (C_f) use the following equation:

$$C_f = [1 + a(T_s - T_i)]$$

2.3.1.2. Where:

2.3.1.2.1. α= Thermal expansion coefficient of titrant (Acetic Acid= 1.07×10^{-3})

2.3.1.2.2. T_i= temperature at sample titration

2.3.1.2.3. T_s= temperature at titrant standardization.

2.4. **Assay Result Calculation:**

2.4.1. Calculate the result using the following equation:

$$Result\ (\%) = \frac{(Cf)(EP1 - EPb)(\ N)\ (19.425)}{W} x \left(\frac{100}{100 - LOD}\right)$$

2.4.2. Where:

2.4.2.1. C_f= Temperature Correction Factor Coefficient

2.4.2.2. *EP1*=Sample Titration End Point (mL)

2.4.2.3. *EPb*=Blank Titration End Point (mL)

2.4.2.4. *N*= Normality of Titrant as Standardized

2.4.2.5. *W*= Sample Weight (g)

2.4.2.6. *LOD* =Loss on Drying (%)

3. IDENTIFICATION (IR) Passes Test:

3.1. For UATR analysis, follow Spectrum Two UATR SOP for Instrument Set-Up and Use.

3.1.1. Perform a background scan prior to use each day and after every ten samples.

3.1.2. Each analyst must run a Reference Standard prior to analyzing a product. A Reference Standard may be compared to multiple lots of the corresponding product on that day.

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3.1.3. Enter the Lot Number, Expiration Date, Date of Analysis, and Analyst Initials in the Sample ID.
3.1.4. Place the Sample on the UATR crystal using a static free scoop.
3.1.5. Align the swinging arm with the crystal and apply force by turning the green arm clockwise.
3.1.6. Press "Scan" on the top Toolbar. The program will preview the sample. Turn the green arm until the Force Gauge is approximately 125, or until the noise has subsided.
3.1.7. Once the Force Gauge is adjusted, press "Scan".
3.1.8. Once the scan is complete, release the swinging arm by turning it counterclockwise.
3.1.9. Clean the UATR crystal and the swinging arm with methanol and a Kim Wipe.
3.1.10. If the correlation is above 0.95. the comparison will be reported with Pass as the result.

4. LOSS ON DRYING 3.0% max.:

4.1. Tare an LOD vial that has been previously dried for 30 minutes under the same conditions to be employed in the determination.
4.2. Allow to come to room temperature in a desiccator for at least 15 minutes before weighing.
4.3. Transfer approximately 2 g of the sample to be tested to the LOD vial, and accurately weigh the vial and contents. By gentle, sidewise shaking, distribute the sample as evenly as possible in the weighing bottle.
4.4. Place the LOD vial containing the sample into the oven.
4.5. Dry the sample at 105°C ± 2°C for 3 hours.
4.6. Allow to come to room temperature in a desiccator for at least 15 minutes before weighing.
4.7. Calculate result using the equation below:

$$\%LOD = \frac{Initial\ Sample\ Weight(g) - Final\ Sample\ Weight(g)}{Initial\ Sample\ Weight(g)}\ x\ 100$$

5. pH (10%) 9.0-11.0:

5.1. Accurately weigh 2.5 g of sample.
5.2. Dissolve the sample in 25 mL of freshly boiled and cooled water.
5.3. Measure the pH of the solution @ 25°C±2°C.
5.4. Follow the appropriate SOP for pH calibration and measurement.

6. SINGLE IMPURITIES (GC) ≤ 1.0%:

6.1. Dilute Sulfuric acid: Add 1mL of concentrated sulfuric acid and sufficient water to make 35mL (1N equivalent).
6.2. Sample solution (10mg/mL Decanoic Acid): Dissolve 113 mg of Sodium Decanoate in 5 mL of water, add 1 mL of dilute sulfuric acid and extract with 10 mL of ethyl acetate. Separate and collect the organic layer, dry organic layer over anhydrous sodium sulfate and then filter before use.
6.3. Working Standard: Prepare a 0.75mg/mL solution of decanoic acid in ethyl acetate, record precise sample weight and dilution factor. (e.g. 100mL volumetric flask is equal to 100 dilution factor, 25mL volumetric flask is equal to a 25 dilution factor).

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6.4. Related compound solution (0.1mg/mL): Prepare a related compound standard solution containing 0.1mg/mL of octanoic acid, 0.1mg/mL nonanoic acid, and 0.2mg/mL of decanoic acid in ethyl acetate.
  6.4.1. Accurately weigh 100mg of each related substance and 200mg of decanoic acid and transfer each quantitatively to a 25mL volumetric flask. Dissolve and dilute to volume with ethyl acetate.
  6.4.2. Pipette 0.25mL of the solution into a 10mL volumetric flask, mix and QS to volume.
6.5. Blank: Pipette 5mL of water, add 1mL of dilute sulfuric acid (1N) and extract with 10mL of ethyl acetate. Separate and collect the organic layer, dry organic layer over anhydrous sodium sulfate and then filter before use.
6.6. Determine the peak responses for system suitability.
6.7. System suitability: The system suitability is determined using *related compound solution (0.1mg/mL)* and the signal-to-noise ratio shall be not less than 14.5 for each related substance.
6.8. Sample analysis:
  6.8.1. Prepare a sequence including:
    6.8.1.1. Blank Sample Single Injection (1)
    6.8.1.2. System Suitability Single Injection (1)
    6.8.1.3. Inject Working Standard Solution A
    6.8.1.4. Inject Working Standard Solution B
    6.8.1.5. Inject Working Standard Solution A
    6.8.1.6. Inject Working Standard Solution B
    6.8.1.7. Inject Sample(s)
    6.8.1.8. Inject Working Standard Solution A
    6.8.1.9. Inject Working Standard Solution B
    6.8.1.10. Blank Sample Single Injection
  6.8.2. % RSD of the working standards should not exceed 2.0%
6.9. Calculate the percentage of each impurity using the equation below.

$$Result\ \% = \frac{(A_u)(D_u)}{(RF_{Ave})(W_{Sample})}\ x\ 100\%$$

  6.9.1. Where:
    6.9.1.1. A_u = peak response of the individual impurity
    6.9.1.2. Du= Dilution Factor of Sample Solution (10mL)
    6.9.1.3. RFAve = Average response factor
    6.9.1.4. RF= Response Factor = ((As)(Ds))/(Wstd)(P))
    6.9.1.5. As= Peak area of decanoic acid in working standard solution
    6.9.1.6. Ds= Dilution Factor of Working Standard (mL)
    6.9.1.7. Wstd= Weight of Decanoic Acid Reference Material (mg)
    6.9.1.8. P = Purity of Decanoic Acid Reference Material Expressed as a Percentage
    6.9.1.9. Wsample = Weight of Sodium Decanoate Sample (mg)

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7. SODIUM **Passes Test:**

7.1. Dissolve 0.1 g of sample in 2 mL of water.
7.2. Add 2 mL of 15% potassium carbonate and heat to boiling. No precipitation is formed.
7.3. Add 4 mL of potassium pyroantimonate TS and heat to boiling.
7.4. Allow to cool in ice water and, if necessary, rub the inside of the glass vessel with a glass rod. A passing sample will produce a dense precipitation.

8. SOLUBILITY IN WATER **Passes Test:**

8.1. Weight at least 2g of sample directly in to a clean and dry beaker. Dissolve in 40mL of purified water by using a magnetic stir bar at a low to medium stir speed at room temperature, do not heat.
8.2. Inspect the sample solution for complete solubility and any foreign matter.
8.2.1. Note: the following steps are for Monitoring only:
8.2.1.1. The sample is expected to be slightly turbid and not completely colorless.
8.2.1.2. Measure NTU result and document. Follow the SOP for Bangor Portable Turbidimeter and Calibration DCN: 16-001317. Monitor specification is <50NTU.
8.2.1.3. Filter the solution through a white round filter paper, inspect for insoluble matter.
8.2.1.4. After this solution is analyzed for Solubility, analyze for particulates. Inspect the magnetic stir bar and verify that no particulates are present on the stir bar. If present notify the appropriate personnel immediately.

9. WATER (KARL FISCHER) **1.5% - 3.0%:**

9.1. Weigh ~1g of sample into a glass weighing spoon and tare the balance.
9.2. Transfer the sample to the Karl Fischer vessel by removing the rubber septum and adding the sample into the titration vessel.
9.2.1. Do not leave the rubber septum open for long periods of time as this will allow moisture to enter the titration vessel.
9.3. Return the weighing spoon to the balance, making sure not to lose any sample that was left behind. Once the weight stabilizes, Record the sample weight and transfer to instrument.
9.4. Check to make sure there is no residual sample stuck to the sides of the titration vessel.
9.4.1. If any sample is stuck to the side, stop the stir bead and swirl the Karl Fischer vessel to rinse the sides.
9.5. Once the method begins, check to ensure the sample is fully dissolved before the titration begins (i.e. before the stir command completes).
9.6. The moisture content will then be determined by the Metrohm Auto Titrando 907.

$$\%\ Moisture = \frac{(mL\ of\ Composite\ 5)(\frac{mg}{mL}\ of\ Composite\ 5)(0.1)}{Sample\ Weight\ (g)}$$

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If there are any questions or concerns, please feel free to contact ra@biospectra.us.

Sincerely,

Cassie Baun

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