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## SODIUM DECANOATE TESTING METHODS

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**1. PURPOSE:**

- 1.1. To provide the Laboratory personnel with procedures for testing Sodium Decanoate Raw Material, In-Process and Finished Goods at the Bangor, PA facility

**2. SCOPE:**

- 2.1. Applies to the testing of Sodium Decanoate Raw Material, In-Process and Finished Goods in the Laboratory at Biospectra. Methods include testing for all grades of Sodium Decanoate sold by BioSpectra; only the specific tests required for the requested grade must be tested.

**3. RESPONSIBILITIES:**

- 3.1. The Laboratory Manager is responsible for the control, training, maintenance and implementation of this procedure.
- 3.2. The Laboratory Analysts are responsible for compliance with the terms of this procedure. This includes notifying the Quality Assurance and Laboratory Managers, or designees, if any analyses fail to meet their respective specifications.
- 3.3. It is the responsibility of all personnel to read and understand the SDS and don the appropriate PPE for handling and disposing of chemicals in a safe manner.

**4. REFERENCES:**

- 4.1. BSI-ATM-0082, Analytical Method for the Quantification of Sodium by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) in Sodium Decanoate
- 4.2. BSI-ATM-0083, Analytical Method of Analysis: Determination of ICH Q3D Elemental Impurities + Iron by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) in Sodium Decanoate
- 4.3. BSI-RPT-0441, Analytical Method Validation Report: Sodium Decanoate Related Substances Method Validation Report
- 4.4. BSI-SDS-0021, Sodium Decanoate SDS
- 4.5. BSI-SOP-0019, Result Reporting
- 4.6. BSI-SOP-0098, Balance SOP
- 4.7. BSI-SOP-0126, Laboratory Notebooks
- 4.8. BSI-SOP-0133, Blue M Convection Oven Operation and Calibration SOP
- 4.9. BSI-SOP-0143, Metrohm Titrand 907 Auto-Titrator SOP
- 4.10. BSI-SOP-0144, Metrohm 914 pH Conductometer Operation and Calibration
- 4.11. BSI-SOP-0242, Bangor Portable Turbidimeter Operation and Calibration
- 4.12. BSI-SOP-0244, VWR Gravity Convection Oven and Calibration (Model Number 414005-106)
- 4.13. BSI-SOP-0254, Spectrum Two UATR SOP
- 4.14. BSI-SOP-0255, XL200 pH/mV/Conductivity Meter SOP
- 4.15. BSI-SOP-0316, Shimadzu QP2010S GC/MS SOP

**5. EQUIPMENT:**

- 5.1. Analytical Balance
- 5.2. Calibrated Oven
- 5.3. Metrohm 907 Titrand 907 Auto-Titrator
- 5.4. Metrohm 914 pH Conductometer Operation and Calibration
- 5.5. Spectrum Two UATR

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- 5.6. XL200 pH/mV/Conductivity Meter or equivalent
- 5.7. Hach Portable Turbidimeter Model 2100Q
- 5.8. Shimadzu QP2010S GC/MS

## 6. REAGENTS:

- 6.1. **0.1N Perchloric Acid:** Purchased commercially
- 6.2. **15% Potassium Carbonate:** Weigh 15.000 g of Potassium Carbonate and transfer to a 100-mL volumetric flask. Dilute to volume with purified water.
- 6.3. **Composite 5:** Purchased commercially
- 6.4. **Crystal Violet:** Purchased commercially
- 6.5. **Crystal Violet Solution, 1%:** Dissolve 1 g of crystal violet in 100 mL of glacial acetic acid. Store in amber bottle.
- 6.6. **Decanoic Acid:** Purchased commercially
- 6.7. **Ethyl Acetate:** Purchased commercially
- 6.8. **Formamide:** Purchased commercially
- 6.9. **Glacial Acetic Acid:** Purchased commercially
- 6.10. **Methanol:** Purchased commercially
- 6.11. **Nonanoic Acid:** Purchased commercially
- 6.12. **Octanoic Acid:** Purchased commercially
- 6.13. **Potassium Hydrogen Phthalate:** Purchased commercially
- 6.14. **Potassium Carbonate:** Purchased commercially
- 6.15. **Potassium Pyroantimonate TS:** Purchased commercially
- 6.16. **Sodium Sulfate, anhydrous:** Purchased commercially
- 6.17. **Sulfuric Acid (H<sub>2</sub>SO<sub>4</sub>), concentrated:** Purchased commercially
- 6.18. **Sulfuric Acid (H<sub>2</sub>SO<sub>4</sub>), dilute (1N):** Add 1 mL of concentrated sulfuric acid and sufficient water to make 35 mL.

## 7. ANALYTICAL PROCEDURES:

- 7.1. **PRE-FILTRATION IN-PROCESS ANALYSIS** :
  - 7.1.1. Perform Visual Inspection and Quantitative NTU result. For NTU result, follow the SOP for Bangor Portable Turbidimeter and Calibration, DCN: BSI-SOP-0242.
- 7.2. **POST-FILTRATION IN-PROCESS SAMPLES** :
  - 7.2.1. Perform Visual Inspection and Quantitative NTU result. For NTU result, follow the SOP for Bangor Portable Turbidimeter and Calibration, DCN: BSI-SOP-0242.
- 7.3. **DRY CRYSTAL KARL FISCHER** :
  - 7.3.1. Weigh ~1 g of sample into a glass weighing spoon and tare the balance.
  - 7.3.2. Transfer the sample to the Karl Fischer vessel by removing the rubber septum and adding the sample into the titration vessel.
    - 7.3.2.1. Do not leave the rubber septum open for long periods of time as this will allow moisture to enter the titration vessel.
  - 7.3.3. 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.
  - 7.3.4. Check to make sure there is no residual sample stuck to the sides of the titration vessel.

- 7.3.4.1. If any sample is stuck to the side, stop the stir bead and swirl the Karl Fischer vessel to rinse the sides.
- 7.3.5. Once the method begins, check to ensure the sample is fully dissolved before the titration begins (i.e. before the stir command completes).
- 7.3.6. The moisture content will then be determined by the Metrohm Auto Titrando 907.

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

7.4. **APPEARANCE** **Refer to Summary Sheet:**

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

7.5. **ASSAY (Dried Basis)** **Refer to Summary Sheet:**

- 7.5.1. **Note: Raw Material Assay analysis requires the Loss on drying analysis and result. Perform LOD on the Raw material sample; there is no raw material specification requirement.**
- 7.5.2. **Standardization of 0.1N Perchloric Acid:**
- 7.5.2.1. The following procedure is performed in triplicate along with the analysis of a single blank (Glacial Acetic Acid).
- 7.5.2.2. **Note:** Before each titration, record the temperature of the 0.1N perchloric acid.
- 7.5.2.2.1. In a well-ventilated area, accurately weigh about 0.7 g of previously dried potassium hydrogen phthalate and transfer into a suitable beaker (150-mL size is ideal), record weight immediately after stabilization.
- 7.5.2.2.2. Ensure stir bar is in the beaker before the addition of the glacial acetic acid to avoid splashing.
- 7.5.2.2.3. Dissolve in 50 mL of glacial acetic acid.
- 7.5.2.2.4. Add 0.1 mL of a 1% Crystal Violet Solution prepared by dissolving 100 mg of crystal violet in 10 mL of glacial acetic acid.
- 7.5.2.2.5. 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.
- 7.5.2.2.6. **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.
- 7.5.2.3. Calculate normality of the titrant utilizing the following equation:

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

- 7.5.2.4. Average the three normalities calculated in step 7.5.2.3.

## 7.5.3. Sample preparation:

- 7.5.3.1. Note: Due to the nature of non-aqueous titrations minimize sample and titrant exposure to the environment (i.e. prepare samples one at a time and only immediately before titration).
- 7.5.3.2. Note: Before each titration, record the temperature of the 0.1N Perchloric acid.
- 7.5.3.3. Accurately weigh 0.5 g of sodium decanoate and transfer to a suitable beaker.
- 7.5.3.4. Dissolve in ~50 mL of glacial acetic acid.
- 7.5.3.5. Add 0.1 mL of a 1% Crystal Violet Solution prepared by dissolving 100 mg of crystal violet in 10 mL of glacial acetic acid.
- 7.5.3.6. 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.
- 7.5.3.7. 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.
- 7.5.3.8. To calculate the result: Reference the LOD result from section 7.9, calculate the temperature correction refer to section 7.5.4. and apply the calculation in section 7.5.5.

## 7.5.4. Temperature Correction:

- 7.5.4.1. 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 ( $T_s$ ).
- 7.5.4.1.1. To Calculate the Temperature Correction Factor Coefficient ( $C_f$ ) use the following equation:

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

## 7.5.4.2. Where:

- 7.5.4.2.1. A = Thermal expansion coefficient of titrant (Acetic Acid =  $1.07 \times 10^{-3}$ )
- 7.5.4.2.1.1.  $T_i$  = Temperature at sample titration
- 7.5.4.2.1.2.  $T_s$  = Temperature at titrant standardization.

## 7.5.5. Assay Result Calculation:

- 7.5.5.1. Calculate the result using the following equation:

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

## 7.5.5.2. Where:

- 7.5.5.2.1.  $C_f$  = Temperature Correction Factor Coefficient (Refer to 7.5.4.2.1.)
- 7.5.5.2.2. EP1 = Sample Titration End Point (mL)
- 7.5.5.2.3. EPb = Blank Titration End Point (mL)
- 7.5.5.2.4. N = Normality of Titrant as Standardized
- 7.5.5.2.5. W = Sample Weight (g)
- 7.5.5.2.6. LOD = Loss on Drying (%)

- 7.6. **ELEMENTAL IMPURITIES IRON, AND SODIUM QUANTIFICATION** \_\_\_\_\_ :  
 7.6.1. Primary Method: Refer to Analytical Method of Analysis: Sodium in Sodium Decanoate by ICP-OES, BSI-ATM-0082, and Analytical Method of Analysis: Elemental Impurities in Sodium Decanoate via ICP-MS, BSI-ATM-0083.  
 7.6.2. Alternate Method: Package and send no less than 10 g of sample to an approved outside testing facility for Elemental Impurities, Iron, and Sodium Quantification.
- 7.7. **IDENTIFICATION (IR)** \_\_\_\_\_ **Refer to Summary Sheet:**  
 7.7.1. For UATR analysis, follow Spectrum Two UATR SOP.
- 7.8. **IDENTIFICATION (GC)** \_\_\_\_\_ **Refer to Summary Sheet:**  
 7.8.1. Refer to Single Impurities and Total Impurities Analysis.
- 7.9. **LOSS ON DRYING** \_\_\_\_\_ **Refer to Summary Sheet:**  
 7.9.1. Tare an LOD vial that has been previously dried for 30 minutes under the same conditions to be employed in the determination.  
 7.9.2. Allow to come to room temperature in a desiccator for at least 15 minutes before weighing.  
 7.9.3. 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.  
 7.9.4. Place the LOD vial containing the sample into the oven.  
 7.9.5. Dry the sample at  $105^{\circ}\text{C} \pm 2^{\circ}\text{C}$  for 3 hours.  
 7.9.6. Allow to come to room temperature in a desiccator for at least 15 minutes before weighing.  
 7.9.7. Calculate result using the equation below:
- $$\% \text{LOD} = \frac{\text{Initial Sample Weight}(g) - \text{Final Sample Weight}(g)}{\text{Initial Sample Weight}(g)} \times 100$$
- 7.10. **pH 10%** \_\_\_\_\_ **Refer to Summary Sheet:**  
 7.10.1. Accurately weigh 2.5 g of sample.  
 7.10.2. Dissolve the sample in 25 mL of freshly boiled and cooled water.  
 7.10.3. Measure the pH of the solution @  $25^{\circ}\text{C} \pm 2^{\circ}\text{C}$ .  
 7.10.4. Follow the appropriate SOP for pH calibration and measurement.
- 7.11. **RESIDUAL SOLVENTS** \_\_\_\_\_ **Refer to Summary Sheet:**  
 7.11.1. Package and send no less than 30 g of sample to an approved outside testing facility to test for Ethanol.
- 7.12. **SINGLE IMPURITIES AND TOTAL IMPURITIES** \_\_\_\_\_ **Refer to Summary Sheet:**  
 7.12.1. Dilute Sulfuric Acid: Add 1 mL of concentrated sulfuric acid and sufficient water to make 35 mL (1N equivalent).  
 7.12.2. Sample Solution (10 mg/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.

- 7.12.3. Working Standard: Prepare a 0.75 mg/mL solution of decanoic acid in ethyl acetate, record precise sample weight and dilution factor. (e.g. 100-mL volumetric flask is equal to 100-dilution factor, 25-mL volumetric flask is equal to a 25-dilution factor).
- 7.12.4. Related compound solution (0.1 mg/mL): Prepare a related compound standard solution containing 0.1 mg/mL of octanoic acid, 0.1 mg/mL nonanoic acid, and 0.2 mg/mL of decanoic acid in ethyl acetate.
- 7.12.4.1. Accurately weigh 100 mg of each related substance and 200 mg of decanoic acid and transfer each quantitatively to a 25-mL volumetric flask. Dissolve and dilute to volume with ethyl acetate.
- 7.12.4.2. Pipette 0.25 mL of the solution into a 10-mL volumetric flask, mix and QS to volume.
- 7.12.5. Blank: Pipette 5 mL of water, add 1 mL of dilute sulfuric acid (1N) 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.
- 7.12.6. The GC-MS is used with the following parameters:
- 7.12.6.1. Method File: Sodium Decanoate Single Impurity Quantitative.qgm
- 7.12.6.2. Column: 0.25-mm x 30-m fused silica; coated with a 0.25-mm layer of phase G25
- 7.12.6.3. Injection Port: 250 °C
- 7.12.6.4. Detector: 250 °C
- 7.12.6.5. Flow rate: 1.5 mL/min
- 7.12.6.6. Carrier gas: Helium
- 7.12.6.7. Injection volume: 1 µL
- 7.12.6.8. Autosampler: AOC-20i
- 7.12.6.9. Injection type: Split
- 7.12.6.10. Split ratio: 5
- 7.12.6.11. Oven Temperature: See Table 1

**TABLE 1: OVEN TEMPERATURE GRADIENT**

Initial Temperature (°C)	Temperature Ramp(°C/min)	Final Temperature(°C)	Hold Time at Final Temperature (min)
100	-	100	1
100	5	220	10

- 7.12.7. Determine the peak responses for system suitability.
- 7.12.8. System Suitability: The system suitability is determined using *related compound solution (0.1 mg/mL)* and the signal-to-noise ratio shall be not less than 14.5 for each related substance.
- 7.12.8.1. Open Post Run Software
- 7.12.8.2. Open associated suitability file.
- 7.12.8.3. Click Qualitative
- 7.12.8.4. Select Peak Integrate for TIC (All Group)
- 7.12.8.4.1. Set Min/Area to 100,000 and width to 5 seconds.
- 7.12.8.4.2. Click *Program* and Set Program to:
- 7.12.8.4.2.1. T=0 Integration off



- 7.12.8.4.2.2. T=10 Integration on
- 7.12.8.4.2.3. T=22 Integration off
- 7.12.8.4.2.4. (These times may change if a new column is used and retention times change, ensure the suitability peak is bracketed by integration)
- 7.12.8.4.3. Click OK
- 7.12.8.4.4. Click OK
- 7.12.8.4.5. Click *OK*
- 7.12.8.5. Click Compound Table
  - 7.12.8.5.1. Select Wizard (New...)
  - 7.12.8.5.2. Click Next (1)
  - 7.12.8.5.3. Click Next (2)
  - 7.12.8.5.4. Click Next (3)
  - 7.12.8.5.5. Click Next (4)
  - 7.12.8.5.6. Click Next (5)
  - 7.12.8.5.7. Click Next (6)
  - 7.12.8.5.8. Click Finish
- 7.12.8.6. Select “View” from the right hand side of the screen.
- 7.12.8.7. Assign peak ID based upon area count and retention time. The largest peak will be assigned as decanoic acid. Assign nonanoic acid as the first adjacent impurity with approximate RRT 0.89 to the decanoic acid peak. Assign octanoic acid as the first adjacent impurity peak to nonanoic acid with an approximate RRT of 0.79.
- 7.12.9. Click Quantitative
  - 7.12.9.1. Select “Peak Integrate for all IDs”
- 7.12.10. Click “Report” from the left hand side bar.
- 7.12.11. Click “Open”
- 7.12.12. Open “System Suitability Report” from data files sodium decanoate OI folder.
- 7.12.13. Ensure the S/N ratio is displayed for octanoic acid and nonanoic acid.
  - 7.12.13.1. Print report.
  - 7.12.13.2. Sample analysis:
    - 7.12.13.2.1. Prepare a sequence including:
    - 7.12.13.2.2. Blank Sample Single Injection (1)
    - 7.12.13.2.3. System Suitability Single Injection (1)
    - 7.12.13.2.4. Inject Working Standard Solution A
    - 7.12.13.2.5. Inject Working Standard Solution B
    - 7.12.13.2.6. Inject Working Standard Solution A
    - 7.12.13.2.7. Inject Working Standard Solution B
    - 7.12.13.2.8. Inject Sample(s)
    - 7.12.13.2.9. Inject Working Standard Solution A
    - 7.12.13.2.10. Inject Working Standard Solution B
    - 7.12.13.2.11. Blank Sample Single Injection
  - 7.12.13.3. % RSD of the working standards should not exceed 12.0%

7.12.14. Calculate the percentage of each impurity using the equation below.

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

7.12.14.1. Where

- 7.12.14.1.1. Au = Peak response of the individual impurity
- 7.12.14.1.2. Du = Dilution Factor of Sample Solution (10 mL)
- 7.12.14.1.3. RFAve = Average response factor
- 7.12.14.1.4. RF = Response Factor = ((As)(Ds))/(Wstd)(P)
- 7.12.14.1.5. As = Peak area of decanoic acid in working standard solution
- 7.12.14.1.6. Ds = Dilution Factor of Working Standard (mL)
- 7.12.14.1.7. Wstd = Weight of Decanoic Acid Reference Material (mg)
- 7.12.14.1.8. P = Purity of Decanoic Acid Reference Material Expressed as a Percentage
- 7.12.14.1.9. Wsample = Weight of Sodium Decanoate Sample (mg)

7.12.15. To perform the integration with the GC-MS software follow the steps below:

- 7.12.15.1. Open Post Run Software
- 7.12.15.2. Open associated sample file
- 7.12.15.3. Click Qualitative
  - 7.12.15.3.1. Select Peak Integrate for TIC (All Group)
  - 7.12.15.3.2. Set Min/Area to: (As\* 0.0013) and width to 10 seconds.
    - 7.12.15.3.2.1. Take the average peak area for decanoic acid from the 6 working standard injection and multiply by 0.0013.
  - 7.12.15.3.3. 0.01% Reporting Threshold: 0.75 mg/mL \* 0.0013 = < 0.01% of 10 mg/mL
  - 7.12.15.3.4. Calculation for Reporting Threshold: (0.75\*0.0013) = 0.000975 (0.000975/10)\*100= 0.00975 or 0.01%
  - 7.12.15.3.5. Click Program and set program to:
    - 7.12.15.3.5.1. T=0 Integration off
    - 7.12.15.3.5.2. T=3 Integration on (These times may change if a new column is used and retention times change, ensure the suitability peak is bracketed by integration)
  - 7.12.15.3.6. Click OK
  - 7.12.15.3.7. Click OK
  - 7.12.15.3.8. Click OK
- 7.12.15.4. Click Compound Table
  - 7.12.15.4.1. Select Wizard (New...)
  - 7.12.15.4.2. Click Next (1)
  - 7.12.15.4.3. Click Next (2)
  - 7.12.15.4.4. Click Next (3)
  - 7.12.15.4.5. Click Next (4)
  - 7.12.15.4.6. Click Next (5)
  - 7.12.15.4.7. Click Next (6)
  - 7.12.15.4.8. Click Finish

- 7.12.16. Click “Report” from the left hand side bar.
- 7.12.17. Click “Open”
- 7.12.18. Open “Sodium Decanoate Report” from data files sodium decanoate OI folder.
- 7.12.19. Ensure all impurities are visible in the report chart; refer to each by retention time.
- 7.12.20. Ensure that the integration of the minimum area applies to all samples and not just the standards by verifying the same Min/Area is applied throughout integration process and ensure that the integration is performed on the samples.
  - 7.12.20.1. The 0.01% threshold will change based on the area count of the decanoic acid peak, which will differ from run to run.
  - 7.12.20.2. Reviewers should also verify that minimum area is correct based on area counts and that integration is applied to each standard and sample apart of the injection sequence.
- 7.12.21. Print report
- 7.12.22. Calculate each integrated impurity as %w/w using the calculation in 7.12.14 and report total impurity as the sum of all calculated impurities.
- 7.12.23. In the case where an unknown impurity is present above the 0.01% threshold and requires identification, the sample can be run under the “Sodium Decanoate Single Impurity.qgm” GC-MS method and a qualified analyst can use the MS library for choosing a compound that shows the best match.

**7.13. SODIUM Refer to Summary Sheet:**

- 7.13.1. Dissolve 0.1 g of sample in 2 mL of water.
- 7.13.2. Add 2 mL of 15% potassium carbonate and heat to boiling. No precipitation is formed.
- 7.13.3. Add 4 mL of Potassium Pyroantimonate TS and heat to boiling.
- 7.13.4. 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.

**7.14. SOLUBILITY IN WATER Refer to Summary Sheet:**

- 7.14.1. Weigh at least 2 g of sample directly in to a clean and dry beaker. Dissolve in 40 mL of purified water by using a magnetic stir bar at a low to medium stir speed at room temperature, do not heat.
- 7.14.2. Inspect the sample solution for complete solubility and any foreign matter.
  - 7.14.2.1. Note: the following steps are for Monitoring only:
    - 7.14.2.1.1. The sample is expected to be slightly turbid and not completely colorless.
    - 7.14.2.1.2. Measure NTU result and document. Follow the SOP for Bangor Portable Turbidimeter and Calibration DCN: BSI-SOP-0242. Monitor specification is < 50 NTU.
    - 7.14.2.1.3. Filter the solution through a white round filter paper, inspect for insoluble matter.
    - 7.14.2.1.4. After this solution is analyzed for Solubility, analyze for particulates.
    - 7.14.2.1.5. Inspect the magnetic stir bar and verify that no particulates are present on the stir bar. If present notify the appropriate personnel immediately.

**7.15. WATER (KARL FISCHER)****Refer to Summary Sheet:**

- 7.15.1. Weigh ~1 g of sample into a glass weighing spoon and tare the balance.
- 7.15.2. Transfer the sample to the Karl Fischer vessel by removing the rubber septum and adding the sample into the titration vessel.
  - 7.15.2.1. Do not leave the rubber septum open for long periods of time as this will allow moisture to enter the titration vessel.
- 7.15.3. 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.
- 7.15.4. Check to make sure there is no residual sample stuck to the sides of the titration vessel.
  - 7.15.4.1. If any sample is stuck to the side, stop the stir bead and swirl the Karl Fischer vessel to rinse the sides.
- 7.15.5. Once the method begins, check to ensure the sample is fully dissolved before the titration begins (i.e. before the stir command completes).
- 7.15.6. The moisture content will then be determined by the Metrohm Auto Titrando 907.

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