STANDARD OPERATING PROCEDURE
STAN MAYFIELD BIOREFINERY PILOT PLANT

TITLE: Bleach Measurement

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APPROVALS: **Process Change Committee** **DATE: September 27th, 2012**

A. Scope

This SOP describes the procedure to indirectly measure hypochlorite concentration by addition of acetic acid and titration with sodium thiosulfate. The addition of acid to the titration flask changes the original chemical form of bleach from NaOCl to HOCl to Cl_2. Under these conditions, sodium thiosulfate can be used to reduce the valence of the chlorine atom from +1 to -1 (to the chloride ion, Cl^-).

B. Safety and Training Requirements

Refer to UF lab safety policies regarding equipment listed in section D below before starting any process work.

Review the location of fire extinguishers, fire blankets, safety showers, spill cleanup equipment and protective gear before beginning any process work.

The following safety gear will be utilized:

- Lab Coat
- Safety Goggles
- Protective Gloves

C. Related Documents and SOPs

1. Bleach Scrubber SOP-7211
2. Sampling SOP-0511
3. MSDS Binder

D. Equipment/Materials

1. Acetic acid solution (50% w/w)
2. Starch solution indicator 0.5% (Ricca Chemical Co., Cat. No. 8000-32)
3. 0.1 N sodium thiosulfate (1 L) - titrant solution
4. Potassium iodide crystals (500 g)
5. Commercial bleach solution (6%)
6. Deionized water

7. Magnetic stirrer
8. Magnetic stir bar
9. Analytical balance (0.001 g)
10. 50 mL titration burette and burette support
11. 50 mL plastic beaker
12. 250 mL Erlenmeyer flask
13. 250 mL volumetric flask
14. 25 mL pipette
15. 10 mL pipette
16. Pipette bulb
17. 50 mL graduated cylinder
18. Weighing bottles

E. Detailed Procedure

1. Measure the density of the sample.
    a. Tare a weighing bottle on the analytical balance.
    b. Pipette 25 ml of the bleach sample into the weighing bottle and weigh to the nearest 1.0 mg (0.001 g).
    c. Calculate the sample density by dividing the weight of the sample (in g) by 25 mL.
2. Transfer 25 mL of sample to a 250 mL volumetric flask.
3. Dilute the sample with deionized water to the 250 mL mark and mix thoroughly.
4. Add 50 ml of deionized water to a 250 mL Erlenmeyer flask.
5. Pipette a 5 mL (for 10 – 16% concentration), a 10 mL (for 3 – 10% concentration), or a 15 mL (for 0.5 – 3%) aliquot of the bleach sample into the Erlenmeyer flask with stirring.
6. Add between 2 to 3 g of potassium iodide crystals and 10 mL of 50% acetic acid (in this order) to the Erlenmeyer flask.
7. Titrate the solution with standardized sodium thiosulfate (0.1N) until the mixture is straw yellow in color.
    a. Before titrating the sample, rinse the titration burette with acid, distilled water, and finally with the titrant.
    b. Fill the burette with the titrant. Open the stopcock to allow the titrant to descend to the 0.00 ml mark. Make sure that the burette tip does not contain an air bubble
    c. Make sure you swirl or thoroughly mix the titrant and sample in the flask during the titration.

d. The sodium thiosulfate must be added by drops and not a steady stream. If this slow addition of sodium thiosulfate is not done, the final results will not be accurate or repeatable.

NOTES: A white sheet of paper should be placed under the Erlenmeyer flask to help see the color change. In addition a small high intensity light placed in the area of the Erlenmeyer flask pointing to the flask and on to the white paper should be used in order to best see the color change and to achieve accurate and repeatable results.

8. Add 5 ml of starch indicator and continue to very slowly, drop by drop, titrate the mixture until the blue color disappears.
9. Calculate the amount of chlorine in the sample (in g/L) using the following formula:

   available chlorine (g/L) = (mL titrant used)*(mM concentration titrant)*(mL of aliquot size*0.1)$^{-1}$ *(0.5 mol/mole)*(0.001 mole/mM)*(70.91 g/mol).

10. Calculate the percentage weight of hypochlorite using the following formula:
    % hypochlorite = 100*(available chlorine g/L)*(sample density g/mL *1000 mL/L)$^{-1}$.

F. Data Archival and Analysis

Record all measurements in the laboratory notebook including the date, time, and batch number of the sample.