ENVIRO TECH CHEMICAL SERVICES STANDARD OPERATING PROCEDURE

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Facility: Modesto	Approval Name & Signature: Jonathan Howarth Ph.D		Revision No.: 4
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Revised Section(s): Time period in which to perform analysis after withdrawing sample			

I. TITLE: HOBr: PROCEDURE FOR DETERMINATION OF BROMINE AND CHLORINE AT THE HB2 FEED TANK (Modified DPD Method)

II. PURPOSE: This document is to be used by any lab personnel to determine the amount of bromine and chlorine in activated HOBr solutions using the HACH DR/890 Colorimeter or equivalent. This test is performed on the concentrated solution taken from the HB2 feed tank. It is recommended that this procedure be followed at least on a weekly basis (or at each plant's option, more frequently) because this method can distinguish between hypobromous acid and chlorine bleach. Since the activated HB2 solution is concentrated, a 1:10 dilution of the product is performed. Then, the test normally performed on the carcass wash water is used.

III. EQUIPMENT / REAGENTS:

-HACH DR/890 Colorimeter- Catalog number 48470-00 (or equivalent)
or: HACH Colorimeter 2 Catalog number 58700-00 (or equivalent)
- 10% Glycine solution (Enviro Tech #D-10015-03) or equivalent.
-DPD ***FREE*** Chlorine Reagent Pillow Packets (for 10 mL)- HACH # 21055-69
-Potassium Iodide Crystals (KI) – SHAPE Products catalog #8118 (or equivalent)
-Two 100 ml graduated cylinders
- Distilled Water
-5 ml syringe
-1 ml syringe

IV. PROCEDURE:

Before testing make sure the instrument is in the low (LO) range mode by checking that the display reads to the hundredths (0.00).

<u>The following analytical procedure should be performed with 2 minutes of drawing the sample from the HB2 feed tank.</u>

1. Into the first graduated cylinder, add 10 ml of concentrated solution from the HB2 feed tank. Make up to the 100 ml mark with distilled water.

2. Into the second graduated cylinder, add 97 ml of distilled water. Add 2 ml of 10 % glycine solution using the 5 ml syringe, cover and invert the cylinder to mix, then add 1 ml of test solution using the 1 ml syringe. (The glycine masks the chlorine concentration at this point). Invert the cylinder several times to mix. The markings on the 1 ml syringe will rub off very easily, so it is recommended to score the 1 ml marking with a knife or scissors.

3. Fill both 10 ml sample cells with 10 mls of the sample prepared in Step 2. Designate one of these to be the blank and the other to be the prepared sample. Make sure the cells are not wet and they are free of fingerprints or smudges.

4. Cap the blank cell and place it in the cell holder with the diamond mark facing you. Cover the cell compartment and press ZERO. The instrument will display 0.00. Remove the blank.

5. Add the contents of one DPD **FREE** Chlorine pillow packet to the prepared sample cell. Cap and shake vigorously. A pink color will develop.

6. Place the sample cell in the compartment with the diamond mark facing you, close the cover and press READ.

7. The instrument display will show "--" followed by the results in ppm total chlorine. Call this reading "B" for Bromine.

8. Note: if the instrument display is blinking, the sample concentration is too high and needs to be diluted further before resuming with step #9.

9. Remove the sample cell from the compartment and add a small amount of KI crystals (two crystals) to the sample cell still containing the sample. Vigorously shake for 15 seconds, this step allows the glycine-combined chlorine to react with the KI.

10. Place the sample cell in the compartment with the diamond mark facing you, close the cover and press READ. This reading is ppm Total Halogen. Call this reading TH.

    Ideally, results from step 7 and step 10 should be the same, ±5%: TH = B.
    If not, then some of the halogen is present as Chlorine (Cl)*: Therefore,

    $$\text{ppm Cl} = \text{TH} - \text{B}$$

    Calculations:

    $$\text{ppm Bromine (at the HB2 feed tank)} = \text{B} \times 2.25 \times 1000 \text{ (dilution factor)}$$

    $$\text{ppm Chlorine (at the HB2 feed tank)} = \text{TH-B} \times 1000 \text{ (dilution factor)}$$

**Reminders:

(1) Multiply colorimeter reading by dilution factor used.
(2) Sample cell and cell cap must be washed thoroughly between replicates to remove KI residues, which will interfere with the accuracy of the subsequent samples.
(3) Distilled water is preferred over Reverse Osmosis (RO) or deionized (DI) water because it has been found to be less prone to interferences.