

ENVIRO TECH CHEMICAL SERVICES STANDARD OPERATING PROCEDURE

Original SOP Effective Date: 4/10/12	Superseded SOP Dated: N/A	Effective Date: 6/11/12	Procedure No.: ETQC44
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Review Frequency: 2 years	Approval Title: VP Technology		Page 1 of 2
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Revised Section(s): IV. Time limitation between drawing sample and performing analysis			

- I. TITLE:** PROCEDURE FOR DETERMINATION OF BROMINE AND CHLORINE IN SOLUTIONS OF GREATER THAN 450 PPM AS Br₂ AND LESS THAN 900 PPM AS Br₂ APPLIED TO MEAT AND POULTRY PRODUCTS (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 solution applied to the meat and poultry products e.g. the carcass wash, head wash, pre-evisceration or OLR cabinet. It is recommended that this procedure be followed at least on a weekly basis (or at each plant's discretion, more frequently) because this method can distinguish between hypobromous acid and chlorine bleach.
- III. EQUIPMENT / REAGENTS:**
- HACH DR/890 Colorimeter- Catalog number 48470-00 (or equivalent)
or: HACH Colorimeter 2 Catalog number 58700-00 (or equivalent)
 - Distilled water
 - 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)
 - 100 ml graduated cylinder
 - 5 ml syringe
 - 1 ml syringe
- IV. PROCEDURE:**

It should be noted that all readings are reported as available chlorine (Cl₂) and are converted to available bromine (Br₂) after the procedure is performed.

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

Note: The following analytical procedure should be performed within 2 minutes of drawing the sample at the point of use

1. Into the 100 ml graduated cylinder, add 97.5 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 0.5 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 0.5 ml marking with a knife or scissors.
2. Fill both 10 ml colorimeter sample cells with 10 mls of the sample prepared in Step 1. 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.
3. 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.
4. Add the contents of one DPD **FREE** Chlorine pillow packet to the prepared sample cell. Cap and shake vigorously. A pink color will develop.
5. Place the sample cell in the compartment with the diamond mark facing you, close the cover and press READ.
6. The instrument display will show "--" followed by the results in ppm total chlorine. Call this reading "B" for Bromine. For increased accuracy, most readings should be between 0.5 and 2.0 ppm (as Cl₂) on the meter reading.
7. Note: if the instrument display is blinking, the sample concentration is too high and needs to be diluted further before resuming with step #8.
8. Remove the sample cell from the compartment and add a small amount of potassium iodide (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 potassium iodide (KI).
9. 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 (Total Halogen).

Ideally, results from step 6 and step 9 should be the same, $\pm 2\%$: $TH = B$.

If not, then some of the halogen is present as Chlorine (Cl)*: Therefore,

$$\text{ppm Cl}_2 = TH - B$$

Calculations:

$$\text{ppm as Bromine} = B \times 2.25 \times 200 \text{ (dilution factor)}$$

$$\text{ppm as Chlorine} = TH - B \times 200 \text{ (dilution factor)}$$

****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 recommended as it has been found to be less susceptible to interferences than Reverse Osmosis (RO) or De-ionized (DI) water.
- (4) It is recommended to re-adjust the hypobromous acid generation system to yield no free chlorine, if the available chlorine exceeds 1% of the available bromine value.