

# Proposed Standard Operating Procedure

## Uniform formation conditions (UFC)

- pH:  $8.0 \pm 0.2$
- Temperature:  $20.0 \pm 1.0^\circ\text{C}$
- Incubation time:  $24 \pm 1$  h
- Chlorine residual:  $1.0 \pm 0.4$  mg/L as free chlorine after 24 h

## Preliminary study

A 24-h chlorine demand study of the water sample may be required before dosing under uniform formation conditions to determine the applied dosage that will yield a chlorine residual of 1.0 mg/L after 24 h (procedure described below).

## Materials

- Chlorine demand-free glassware
- pH 8 borate buffer
- pH 8 combined hypochlorite-buffer dosing solution

## Methods

- Chlorine demand-free glassware  
Incubation bottles (amber with PTFE-faced caps): soak in detergent\* at least overnight, rinse four times with hot tap water, two times with distilled, deionized (DI) water. Place in 10–20 mg/L chlorine solution (made with DI water) for at least 24 h. Rinse four times with DI water and then one to two times with laboratory clean water (reverse osmosis/ion exchange/granular activated carbon [RO/IX/GAC]); dry in  $140^\circ\text{C}$  oven at least overnight. Store dosing pipettes in ~50 mg/L  $\text{Cl}_2$  (made with laboratory clean water). Rinse three times with dosing solution before use, and store pipettes in chlorine solution after use.
- pH 8 borate buffer  
Before dosing, water samples are buffered to pH 8.0 with 2 mL/L borate buffer: 1.0 M boric acid (American Chemical Society [ACS] grade) and 0.26 M sodium hydroxide (ACS grade) in boiled laboratory clean water.
- pH 8 combined hypochlorite-buffer dosing solution  
A combined hypochlorite-buffer solution (based on the method described in reference 11) is made by buffering the hypochlorite solution to pH 8.0 with pH 6.7 borate buffer.  
To make pH 6.7 borate buffer: 1.0 M boric acid (ACS grade) and 0.11 M sodium hydroxide (ACS grade) in boiled laboratory clean water (RO/IX/GAC).  
Add pH 6.7 borate buffer to chlorine solution (1,000–3,000 mg  $\text{Cl}_2/\text{L}$ ) to yield a pH 8 dosing solution. (A 4–5:1 volume ratio of pH 11.2 hypochlorite solution to pH 6.7 borate buffer yields a pH 8 combined hypochlorite-buffer solution with about a 20 percent drop in chlorine strength.)  
The dosing solution (combined  $\text{OCl}^-$ -buffer) chlorine strength should allow for a dosing volume of < 0.5 percent of the water sample volume (e.g., 2.5 mL dosing solution in a 1-L bottle).
- Preliminary study  
Perform a 24-h chlorine demand study (sample buffered at pH 8.0 and incubated in the dark at  $20.0^\circ\text{C}$  as described in the dosing procedure) using a series of three chlorine dosages based on  $\text{Cl}_2$ :TOC ratios of 1.2:1, 1.8:1, and 2.5:1 after adjusting for inorganic demand. From the results of these tests, the chlorine dose for UFC is selected to yield a 24-h residual of 1.0 mg/L free chlorine.
- Dosing procedure
  1. Add 2.0 mL/L pH 8 borate buffer to water sample.
  2. Adjust to pH 8 with  $\text{H}_2\text{SO}_4/\text{NaOH}$  (if necessary).
  3. Fill incubation bottle three quarters full with buffered water sample.
  4. Dose with combined hypochlorite-buffer solution holding pipette just above water surface.
  5. Cap bottle; invert twice.
  6. Fill to top with buffered water sample and cap headspace-free.
  7. Invert 10 times.
  8. Incubate in dark at  $20.0^\circ\text{C}$  for 24 h.
  9. After incubation, measure chlorine residual and pH, and sample for disinfection by-products.

\*Fisher FL-70, 4 percent, Fair Lawn, N.J.